

Thermal Characterization of Functionally Graded Materials: Design of Optimum Experiments

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An optimum experimental design applied to the measurement of thermal properties in functionally graded materials is studied. As a first step, a material with linearly varying thermal properties is analyzed, and several different transient experimental designs are discussed. An optimality criterion, based on sensitivity coefficients, is used to identify the best experimental design. Simulated experimental results are analyzed to verify that the identified best experimental design has the smallest errors in the estimated parameters. This procedure is general and can be applied to design of experiments for a variety of materials.

Nomenclature

b_k	=	k th parameter
C	=	specific heat per unit volume, $\text{J}(\text{m}^3 \text{K})^{-1}$
D	=	optimality condition, Eq. (12)
e	=	slope of spatial variation
k	=	thermal conductivity, $\text{W}(\text{mK})^{-1}$
L	=	sample thickness, m
n	=	number of time steps
p	=	number of parameters
q_0	=	applied heat flux, $\text{W} \cdot \text{m}^{-2}$
s	=	number of sensors
T	=	temperature, K
t	=	time, s
X	=	sensitivity coefficient, Eq. (9)
\mathbf{X}	=	sensitivity matrix, $sn \times p$
x	=	spatial coordinate, m
α	=	thermal diffusivity, $\text{m}^2 \cdot \text{s}^{-1}$
ϵ	=	small value for finite difference
ρ	=	density, $\text{kg} \cdot \text{m}^{-3}$
θ	=	dimensionless time

Subscripts

h	=	heater
i	=	index for time step
j	=	index for sensors
k	=	index for parameters
0	=	fixed value

Superscripts

$-$	=	spatial average quantity
$+$	=	dimensionless quantity

Introduction

FUNCTIONALLY graded materials are being studied as possible components of aerospace thermal protection systems. Functionally graded (FG) materials include composites with epoxy and metal matrices, metal foams, or any structure with properties de-

signed to vary with position. In the future when FG materials are specified as part of a vehicle program, part of the procurement process will involve certification that the material meets the specifications.

To date there has been little research on accurate thermal characterization of FG materials. The present research is intended to close this gap in the procurement cycle by developing accurate methods to measure the thermal properties of FG materials.

A review of the pertinent literature is given next. The focus here is on FG materials described by macroscopic or effective properties, rather than on microscopic structures. Several researchers have found exact analytical solutions for thermal response by representing an FG material as composed of multiple layers each with different, spatially uniform, thermal properties,^{1,2} or with exponential-function variation of thermal properties along one spatial direction.^{3,4} Still others have used Galerkin's method to find temperature in materials with arbitrary property distributions (see Ref. 5). The primary motivation for these studies of temperature has been to determine the thermal stresses. There has also been some work to find the distribution of thermal properties that optimizes the thermal stress distribution.^{6,7}

There is only one research group that has reported experiments to measure thermal properties in an FG material. Makino and Noda have used transient theory applied to an FG material with exponential variation of thermal properties.^{8,9} They have measured the single parameter that describes the thermal property variation in the material with a transient heating experiment. Their data analysis combines a single temperature datum with their transient theory to provide a single value for the parameter. Although simple in concept, this approach is sensitive to measurement noise.

The approach used in the present work is parameter estimation, a statistics-based method of property measurement, that has been applied to transient experiments for many years.¹⁰ In this method the desired parameters are found by nonlinear regression between the experimental data (temperatures in this case) and a computational model of the experiment. Parameter estimation concepts have recently been applied to optimum experimental design for thermal characterization of uniform materials^{11,12} and for materials with temperature-varying properties.¹³

The focus of this paper is to develop optimum experiments to characterize thermally FG materials with low thermal conductivity. To the author's knowledge, this is the first study of optimum experiments for thermal properties in FG materials.

Next a brief overview of the paper is given. In the next section, a heat transfer model of a one-dimensional FG material is described. The sensitivity coefficients and sensitivity matrix are then defined, and their use in the design of optimum experiments is described. Several experimental designs are investigated for a material with linearly varying thermal properties under specific (simulated) experimental conditions. The best experimental design and the optimum operating conditions are identified. The results are also verified with

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simulated experiments for estimation of thermal parameters from noise-containing data.

Model

In this section, a one-dimensional heat conduction model of an FG material is discussed. This model is used to both simulate the experiments and to construct the sensitivity coefficient matrix.

Consider the one-dimensional heat conduction in a slab of thickness L . The thermal conductivity and (volume) specific heat vary with coordinate x . The material properties are constant with respect to temperature (or small changes in temperature are assumed). The following dimensionless variables will be used to describe the heat conduction problem:

$$T^+ = \frac{T - T_0}{q_0 L / \bar{k}}, \quad x^+ = \frac{x}{L}, \quad \theta = \frac{\bar{\alpha} t}{L^2} \quad (1)$$

$$k^+ = \frac{k(x)}{\bar{k}}, \quad C^+ = \frac{C(x)}{\bar{C}}, \quad \bar{\alpha} = \frac{\bar{k}}{\bar{C}} \quad (2)$$

$$\bar{k} = \frac{1}{L} \int_0^L k(x) dx, \quad \bar{C} = \frac{1}{L} \int_0^L C(x) dx \quad (3)$$

Here \bar{k} and \bar{C} are the spatially averaged properties over the slab body, T_0 is a fixed temperature, and q_0 is the applied surface heat flux. The use of spatially averaged properties to normalize the problem facilitates comparisons between different experiments and between different materials.

Based on the author's previous experience with low-conductivity materials,¹⁴ there are several elements that every experiment should contain: rapid heating on one side of the material for some period, continued data collection during a zero-heating period, and a fixed temperature at the other side of the material. (If active cooling is not practical, a large thermal mass at the nonheated face can be used.)

When the preceding dimensionless values are used, this type of experiment can be simulated by solving the following equations:

$$\frac{\partial}{\partial x^+} \left(k^+ \frac{\partial T^+}{\partial x^+} \right) = C^+ \frac{\partial T^+}{\partial \theta}, \quad 0 < x^+ < 1 \quad (4)$$

At $x^+ = 0$,

$$-k^+(0) \frac{\partial T^+}{\partial x^+} = \begin{cases} 1, & \theta < \theta_h \\ 0, & \theta > \theta_h \end{cases} \quad (5)$$

At $x^+ = 1$,

$$T^+(1, \theta) = 0 \quad (6)$$

At $\theta = 0$,

$$T^+(x^+, 0) = 0 \quad (7)$$

Heating takes place at surface $x^+ = 0$ until time θ_h after which the heating ends. This problem is solved by a finite difference procedure. The time derivative is treated with the Crank–Nicholson method with uniform time steps. The spatial nodes are crowded toward $x = 0$ with a sine-squared scheme so that the early-time temperature can be accurately computed with a reasonable number of nodes. Properties $k^+(x)$ and $C^+(x)$ are evaluated in a subroutine so that different property distributions may be easily studied. The numerical solution was verified by comparison with two exact solutions: a constant-property transient solution¹⁵ and a steady-state solution for a material with linearly varying properties.¹⁶ These comparisons show that 40 spatial nodes are adequate and that the maximum time step should be about $\delta\theta = 0.005$ for 0.1% accuracy in the surface temperature value.

Optimum Experimental Design

Sensitivity coefficients and the sensitivity matrix are needed in the design of optimum experiments for thermal property evaluation. The sensitivity coefficients are defined by

$$X_{jk}(i) = b_k \frac{\partial T_j^+}{\partial b_k} \quad (8)$$

which is the sensitivity for the k th parameter, the j th temperature sensor, and the i th time step. Parameters b_k may include conductivity, specific heat, density, etc. In this research, the sensitivity matrix may encompass several heating events considered together as one experiment, in which case additional heating events are treated as additional sensors.

The sensitivity coefficients were computed with a finite difference procedure to approximate the derivative, as follows:

$$X_{jk}(i) \approx b_k \frac{\{T_{ij}^+[(1 + \epsilon)b_k] - T_{ij}^+(b_k)\}}{\epsilon b_k} \quad (9)$$

Here T_{ij}^+ is the temperature at the i th time step for the j th sensor. The value of $\epsilon = 0.001$ was found to give well-behaved values for X .

Much can be gained from studying the sensitivity coefficients to guide the design of an experiment, and there are two specific requirements that the sensitivity coefficients must satisfy. First, the sensitivity coefficients should be as large as possible. Generally any change in the experiment that increases the size of the sensitivity coefficient is an improvement. Second, when two or more parameters are to be measured in the same experiment, the sensitivity coefficients must be linearly independent. That is, the shape of the sensitivity coefficients must be different. A formal procedure to quantify these two requirements is given next.

The sensitivity coefficients are assembled into a sensitivity matrix X , defined by

$$X = \begin{bmatrix} X(1) \\ X(2) \\ \vdots \\ X(n) \end{bmatrix}, \quad X(i) = \begin{bmatrix} X_{11}(i) & X_{12}(i) & \cdots & X_{1p}(i) \\ X_{21}(i) & X_{22}(i) & \cdots & X_{2p}(i) \\ \vdots & \vdots & \ddots & \vdots \\ X_{s1}(i) & X_{s2}(i) & \cdots & X_{sp}(i) \end{bmatrix} \quad (10)$$

Optimum experimental design is based on maximization of a quantity constructed from the sensitivity matrix multiplied by its transpose, given formally by $X^T X$. The elements of $[p \times p]$ matrix $X^T X$ are given by

$$(X^T X)_{lm} = \sum_{j=1}^s \sum_{i=1}^n X_{ji}(i) X_{jm}(i) \quad (11)$$

The optimality criterion selected for this study is the (normalized) determinant of matrix $X^T X$ given by

$$D = [sn(T_{\max}^+)^2]^{-1} \det(X^T X) \quad (12)$$

Note that the optimality criterion D is normalized by the maximum temperature rise (squared), the number of sensors, and the number of time steps. This is important so that the optimality criterion D may be used to compare different experiments. The determinant is computed for any value of p with well-known matrix methods.¹⁷ The optimality criterion ensures that the sensitivity coefficients will be large and linearly independent.

The optimality criterion is subject to the following standard statistical assumptions: additive, uncorrelated errors with zero mean and constant variance; errorless independent variable; and no prior information. Maximizing the optimality criterion minimizes the hypervolume of the confidence region of the parameter estimates.¹³

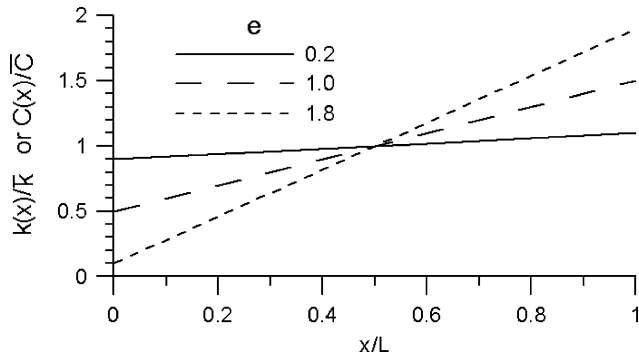


Fig. 1 Spatial variation of thermal properties studied.

Experimental Designs Considered

The focus of this research is to explore the design of experiments for thermal characterization of FG materials, that is, materials with spatially varying thermal properties.

As a first step, a material with linearly varying properties was analyzed. Consider a one-dimensional slab body ($0 < x < L$) of this material. The thermal conductivity and volume specific heat are given by

$$k(x) = \bar{k} \left[1 + e \cdot \left(x/L - \frac{1}{2} \right) \right] \quad (13)$$

$$C(x) = \bar{C} \left[1 + e \cdot \left(x/L - \frac{1}{2} \right) \right] \quad (14)$$

Here the parameters are the spatially averaged thermal conductivity \bar{k} , the spatially averaged specific heat \bar{C} , and the dimensionless slope e . The same slope is used for both k and C to represent the effect of density variation on thermal properties in a metal foam, for example.

A finite difference computer routine was written to compute temperature, sensitivity coefficients, and optimality criterion D . Three levels of spatial-property spatial variation were studied, $e = 0.2, 1.0$, and 1.8 , representing property variation of $\pm 10\%$, $\pm 50\%$, and $\pm 90\%$, respectively, from the mean value, as shown in Fig. 1. Different mean values for \bar{k} and \bar{C} were not studied because the normalized results are valid for any conductivity and specific heat.

Several combinations of simulated experimental conditions were studied, including the number of and location of sensors, heating on one side or the other, heating duration, experiment duration, and the number of parameters.

Results for Optimality

The results for optimality condition D are given in this section. The special case when the thermal properties are spatially constant is considered first, for comparison with earlier work. There are only two parameters present, \bar{k} and \bar{C} . Consider an experiment with a single on-off heating event and with one temperature sensor located on the heated surface. The normalized optimality condition for this case is plotted vs dimensionless time in Fig. 2 for several different heater-off times. Figure 2 reproduces the results of Taktak et al.¹¹ to provide verification of the code used for the present research. Note in Fig. 2 that continuous heating creates a single baseline value for each case and, then when heating stops, the D value jumps above this baseline by a factor of two or so. The optimum experiment for a uniform-property material involves a heating duration of $\theta_h = 2.25$ and experiment duration about 3.0 where $D_{\max} = 0.02$. In the next sections materials with linearly varying properties will be discussed.

One Heating Event, One Sensor

In this section, a material with linear properties described by Eqs. (13) and (14) is studied for which the three parameters are \bar{k} , \bar{C} , and e . Consider a simulated experiment containing a single heating event and a single sensor at the heated surface. The other surface is maintained at ambient temperature. Consider first continuous heating to investigate the baseline values of D . In Fig. 3, optimality condition D is plotted vs time for three values of slope

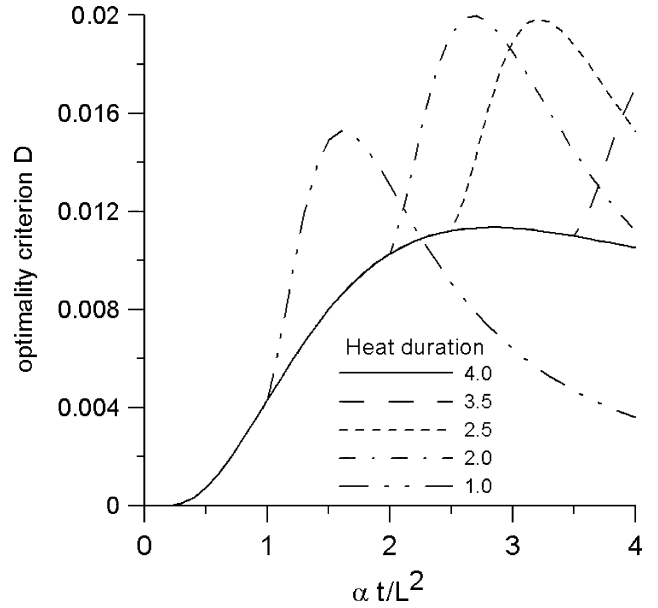


Fig. 2 Optimality condition D for a uniform property material for estimating parameters \bar{k} and \bar{C} for several values of heater-off time θ_h ; one temperature sensor at $x=0$ and heating at $x=0$.

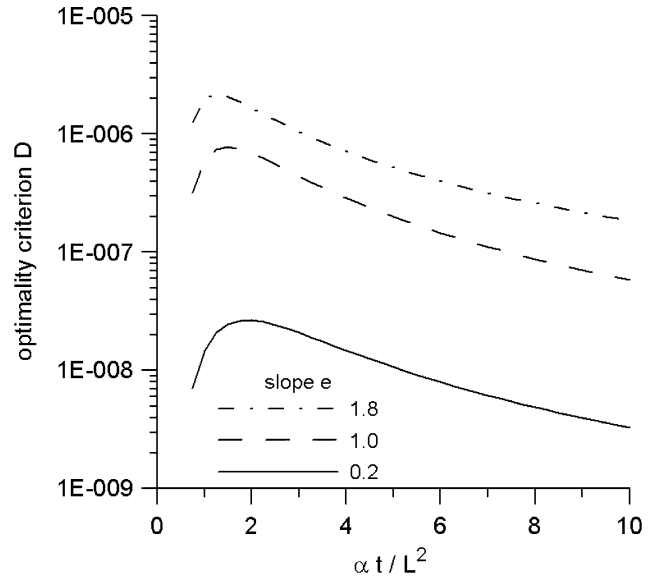


Fig. 3 Optimality condition D for a linearly varying property material with continuous heating at $x=0$ and one sensor at $x=0$.

e for a material heated at $x=0$ (the low- k side). The central result shown in Fig. 3 is that the baseline D values for three parameters are orders of magnitude less than for two parameters as shown in Fig. 2. Clearly it is more difficult to estimate three parameters compared with two. Another observation is that the magnitude of D is smaller for smaller values of slope e . Thus, it is more difficult to estimate small values of e for which the properties are nearly uniform.

Next consider Fig. 4 in which D values are shown for the same materials with parameters \bar{k} , \bar{C} , and e , but with the experiment reversed: continuous heating at $x=L$ (the high- k side), a single temperature sensor at $x=L$, and a fixed temperature at $x=0$. For $e=0.2$, the D values are similar in size and shape to Fig. 3. For $e=1.0$, the peak D value occurs at a slightly later time because of the greater thermal mass near $x=L$. (Recall that the time axis is normalized by the spatial-average thermal diffusivity.) Finally for $e=1.8$, not only is the peak D value delayed, but the peak value is about 10 times greater than for heating at $x=0$. At this point, one might conclude that heating at the high- k side is best, at least for large- e materials.

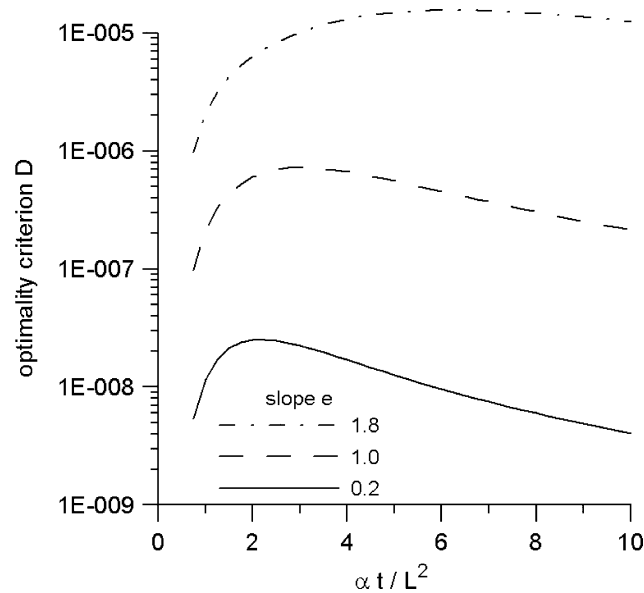


Fig. 4 Optimality condition D for a linearly varying property material with continuous heating at $x=L$ and one sensor at $x=L$.

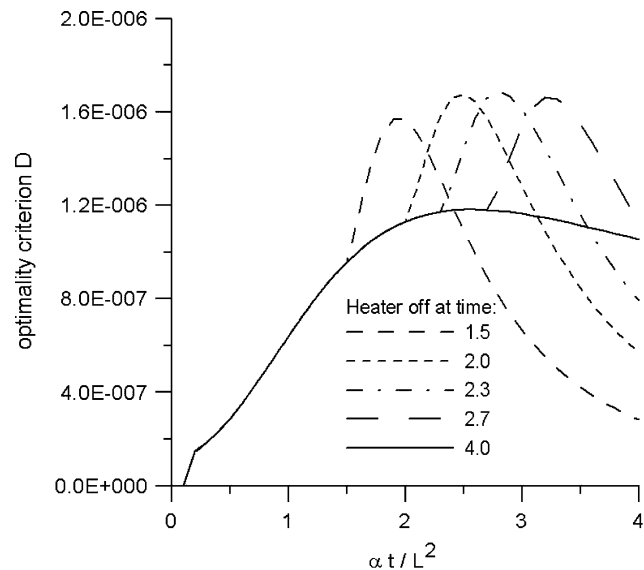


Fig. 5 Optimality condition D for experiment with two temperature sensors at $x/L=0$ and 0.25 and heating at $x=0$ for several cases with different heating duration.

However, larger D values than these are presented in the following sections by the use of interior temperature sensors and by combining two heating events in a single experiment.

One Heating Event, Two Sensors

In this section simulated experiments were analyzed with two sensors, one at the heated surface and one sensor inside the sample. Generally a second temperature sensor located in the range $0.2 < x/L < 0.4$ gave the largest D values. If the second sensor is too close to the surface sensor, it offers little new information, and if the second sensor is too close to $x=L$, its response will be limited by the fixed-temperature boundary there.

In all of the cases reported here, the second sensor is located at $x_2 = 0.25L$. Results for $e = 0.2$ are typical and are presented in Fig. 5. Again the parameters are \bar{k} , \bar{C} , and e . Note that the baseline values for D for two sensors, with the heater continuously energized, are about 60 times larger than for one sensor as shown in Fig. 3 (for case $e = 0.2$). That is, use of an additional sensor inside the body greatly improves the experiment. In Fig. 5, four cases are

Table 1 Maximum value of optimality condition D , and the conditions under which it occurs, for three experiments

Experiment	Parameters	Slope e	Time θ_h	Time at D_{\max}	D_{\max}
1 ^a	\bar{k}, \bar{C}, e	0.2	2.3	2.8	$1.7(10^{-6})$
1 ^a	\bar{k}, \bar{C}, e	1.0	1.5	1.8	$8.7(10^{-5})$
1 ^a	\bar{k}, \bar{C}, e	1.8	1.5	1.7	$1.2(10^{-3})$
2 ^b	\bar{k}, \bar{C}, e	0.2	1.3	1.9	$9.5(10^{-6})$
2 ^b	\bar{k}, \bar{C}, e	1.0	1.7	2.2	$24.(10^{-5})$
2 ^b	\bar{k}, \bar{C}, e	1.8	1.3	1.5	$2.2(10^{-3})$
3 ^c	\bar{k}, \bar{C}	—	2.25	3.0	0.02

^aOne heating event and two sensors at $x/L = 0, 0.25$.

^bTwo heating events, from each side, with one sensor on the heated side.

^cSpatially uniform properties (for comparison), one heating event, one sensor at $x = 0$.

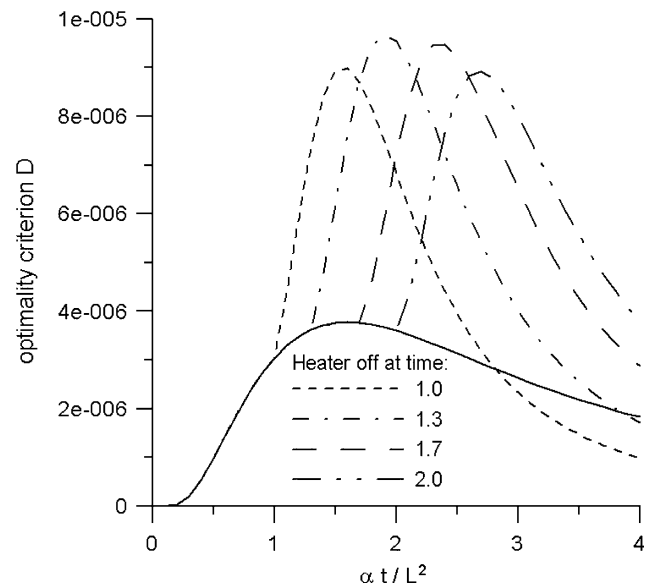


Fig. 6 Optimality condition D for two heating events, $x=0$ and $x=L$, combined into one experiment; temperature sensor located at heated surface.

also presented for which the heater is shut off before $\theta = 4.0$. The best experiment is that for which the heater is shut off at $\theta_h = 2.3$ and the experiment continues until $\theta = 2.8$. The same trend of improvement is present for other values of e . Results for $e = 1.0$ and $e = 1.8$ for two sensors and one heating event are listed as experiment 1 in Table 1. Table 1 shows that the optimum heating times and experiment durations are shorter for larger values of e . For all e values the choice of heater shutoff time causes only small changes in the maximum D value, that is, the peak D value is insensitive to heating duration when an interior sensor is used.

Two Heating Events, One Sensor Each

In this section results are presented for an experiment composed of data from two heating events, each involving one surface-mounted temperature sensor. In one heating event the sample is heated at $x = 0$ and the sensor is located at $x = 0$. In the other heating event the sample is heated at $x = L$ and the sensor is located at $x = L$. In the laboratory the second heating event could be accomplished with the same heater and sensor by reversing the sample. This experimental design takes advantage of the different conductivity values on each side of the body and the fact that surface-mounted sensors are simpler to install than interior sensors.

For simplicity both heating events involve the same heating duration θ_h and the same data duration. In Fig. 6, with the same parameters, D values are plotted vs time for four different heater-off times, again for $e = 0.2$. Each curve represents the combination of data from two heating events into a single experiment. Once again the familiar shape occurs with a baseline value for continuous heating,

with heater-off cases providing an additional boost to the maximum D value. The distinguishing feature, once again, is the magnitude of D_{\max} compared to earlier experiment designs. This case with two heating events gives a D_{\max} nearly six times higher than for the interior sensor case (Fig. 5). The best experiment from the $e = 0.2$ results shown in Fig. 5 is for heating off at $\theta_h = 1.3$, which provides $D_{\max} = 9.5(10^{-6})$ at experiment duration $\theta = 1.9$.

A summary of results for $e = 1.0$ and $e = 1.8$ for two heating events are listed as experiment 2 in Table 1. The same general trends are exhibited for these e values; however, the amount of improvement in D_{\max} is less for higher e values when comparing two heating events with an interior sensor.

Simulated Experiments

The purpose of optimum experimental design is to provide meaningful assistance in thermal characterization and in analyzing experimental data. In this section simulated thermal-characterization experiments are carried out for two experimental designs.

Simulated experiments are carried out by adding errors to exact temperature values (computed from the model) and analyzing this error-containing data to estimate parameters. The added errors are normally distributed with zero mean and an adjustable variance. The error variance was set to either 1 or 5% of the maximum temperature values. The added error values are found with a computer routine that requires a seed number, and different seed numbers can be used to produce different sequences of error values. (These are sometimes called pseudorandom numbers.) The simulated data are analyzed with a Marquardt regression scheme, which systematically compares the simulated data with values computed from the model based on iteratively improved guesses for the parameters (see Refs. 14 and 17). Iteration ceases when the improvements in the parameters become small.

Figure 7 shows a set of simulated data with added error variance 5%. The regression fit for this data is also shown in Fig. 7. The 5% variance in the added error is much larger than would be tolerated in reasonable experimental practice, but it is useful as a test of the estimation scheme. In this case the estimation scheme converges without incident.

The regression scheme was applied to a variety of simulated experiments, and it was found that the parameter estimates varied somewhat for different error sequences (different seed numbers in the random-number generator), even though the variance of the errors was identical. This suggested that a single simulated experiment may be misleading as to the precision of the estimates. One estimate

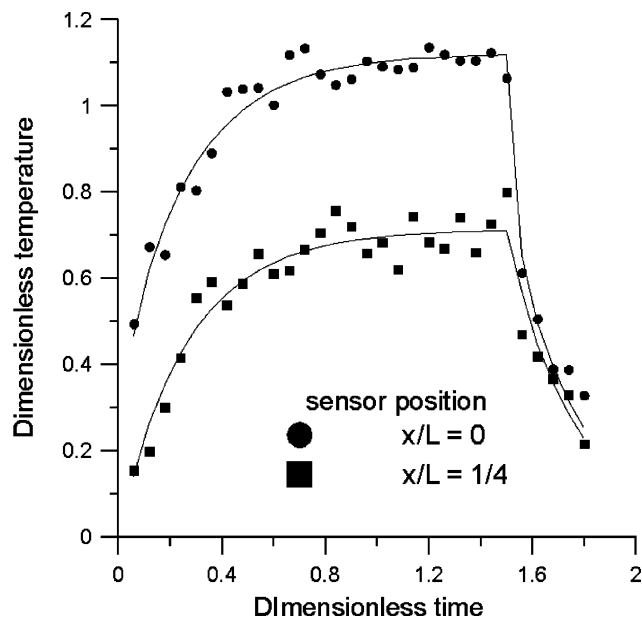


Fig. 7 Simulated data (with noise) and regression fit for experiment 1 (one heat event, two sensors) for $e = 1.8$ and error variance 5%.

Table 2 Percent error in parameter estimates from simulated experiments^a

Percent variance in data	Slope e	Ensemble average error in parameters, %		
		\bar{k}	\bar{C}	e
		<i>Experiment 1, $x/L = 0, 0.25$</i>		
1	0.2	0.2435	2.1681	10.9687
1	1.0	0.3100	1.9259	1.2855
1	1.8	0.2936	1.5820	0.1461
5	0.2	0.9914	10.6676	59.2553
5	1.0	2.2731	13.4547	18.2089
5	1.8	1.9463	4.7742	0.9925
<i>Experiment 2</i>				
1	0.2	0.3249	1.0254	7.4094
1	1.0	0.3706	0.8316	1.3456
1	1.8	0.8085	0.5851	0.3114
5	0.2	1.5017	6.8977	23.6133
5	1.0	1.9755	4.0834	6.3749
5	1.8	3.1122	1.7011	1.4480

^aReported values are ensemble averages over 10 repetitions of the data analysis for each experiment design.

might by chance be particularly close to the actual value, and another estimate might be particularly far from its actual value. To deal with this uncertainty, each simulated experiment was repeated 10 times, and an ensemble average error was computed. For each repetition n , the error for parameter b_i is given by

$$\text{err}_i(n) = [(b_i - \hat{b}_i)/b_i] \times 100\% \quad (15)$$

Here the exact value is b_i and the estimated value is \hat{b}_i . The ensemble average error for parameter b_i is given by

$$\overline{\text{err}}_i = \frac{1}{10} \sum_{n=1}^{10} |\text{err}_i(n)| \quad (16)$$

Note that the absolute value of each error is used in the ensemble average. The purpose here is to compare the errors produced by different experiments, and the absolute value reduces the variability in the results without biasing the error values toward zero. In contrast, in the analysis of laboratory data from several identical experiments, a simple average of the parameter estimates (no absolute value) would be appropriate to find the most precise estimates.

Table 2 shows the summary of results of the ensemble average error found from 10 repetitions of each simulated experiment. Two experiment designs are compared in Table 2, and the operating conditions for each experiment are taken from the optimum conditions given earlier. The values in Table 2 show that experiment 2 tends to provide smaller error in the estimates for the high-noise data and when the material distribution slope e is small. Under these difficult conditions, the conclusion is that experiment 2 is best for estimating parameters. For less difficult conditions, for example, with low-noise data and for e large, experiments 1 and 2 provide comparable-size errors in the estimated parameters.

In all cases listed in Table 2, the simulated experiments were carried out with 30 simulated data points (extracted from the many time steps required in the model calculation), and the initial guesses for the parameters in the regression scheme were taken to be 0.9 times the correct parameter values. To explore the effect of these arbitrary choices on the parameter estimates, additional simulated experiments were carried out. Additional cases included 60 simulated data points, initial guesses of 0.5 times the correct parameter values, and, initial guesses of 1.5 times the correct parameter values. In all of these additional cases, the results were comparable to those in Table 2.

Summary

Optimum experiments are sought for the measurement of thermal properties in FG materials with spatially varying thermal properties. As a first step, one-dimensional transient experiments were studied

for a material with linearly varying thermal properties described by parameters \bar{C} , \bar{k} , and slope e . Variation of properties with temperature was not treated.

Several transient experimental designs were considered, including single or multiple heating events and including surface and/or interior temperature sensors. An optimality condition based on sensitivity coefficients was used to find the best operating conditions for each experiment and to find the best experimental design among those studied. The best experiment has the smallest hypervolume of the confidence region of the parameter estimates.

The results of the optimality study show that it is more difficult to obtain accurate values of slope e when e is small compared to when e is large, that is, it is easier to "see" large spatial variations in properties. The best experimental design involves analysis of combined data from two separate heating events, one with heating on one side of the body and one with heating on the other side, each time with the sensor located on the heated side and the unheated side maintained at a fixed temperature. This conclusion is also supported by a series of simulated experiments, carried out with regression analysis of error-containing temperature values.

The optimum operating conditions for the experiment are somewhat dependent on the property slope e . For the specific case $e = 0.2$, the optimum conditions are heating duration of $\theta_h = 1.3$ and with data recorded until $\theta = 1.9$, where θ is dimensionless time.

The method of optimum experimental design discussed has been demonstrated for a class of FG materials; however, the approach is completely general and applies to any material. Work in progress includes optimal experimental design for thermal characterization of porous materials under conditions where both conduction and radiation heat transfer are present.

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