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Optimal experimental design for estimating thermal properties of composite materials

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Abstract—Design of optimal transient experiments is needed for the efficient estimation of thermal conductivity and volumetric heat capacity of composite materials. One criterion for optimal experiments is the minimization of the area (or volume) of the confidence region. The experimental designs are transient and involve both finite and semi-finite geometries with finite duration heating. Two cases are considered for the finite body: one-dimensional heat conduction within cured composite materials and one-dimensional heat conduction within composite materials undergoing curing. The optimal dimensionless heating and experimental times at the heated boundary and the optimal location of the temperatures sensors are determined.

INTRODUCTION AND LITERATURE REVIEW

IN PERFORMING experiments, the researcher would like to gain as much insight and information from the results as possible. To reach this goal, experiments have to be designed properly. The design of experiments has been the topic of a number of papers in the fields of statistics [1–9], chemical engineering [10], and mechanical engineering [11–17].

The design of optimal heat conduction experiments for determining thermal conductivity, k, and volumetric heat capacity, c, was considered by Beck and Arnold [13, chap. 8]; in this book, two one-dimensional rectangular geometries were analyzed: a finite geometry insulated on one boundary and subject to a heat flux on the other boundary and a semi-infinite geometry with a heat flux boundary condition. The optimal experiments were defined to be those that minimized the confidence region for k and c for certain statistical assumptions and certain given conditions, such as for a semi-infinite body and for a finite body insulated on one surface. For the finite geometry with one surface insulated, the best experiment corresponded to a prescribed *heat flux* producing a step change in temperature on one boundary, with the temperature sensors placed at both boundaries. The use of an insulated boundary is appropriate for metals (which have relatively high thermal conductivities); polymer based composite materials, on the other hand, tend to have low thermal conductivities and are difficult to insulate. However, it is not difficult to approximate an isothermal boundary condition at the unheated surface by placing the composite specimens in intimate thermal contact with a high conductivity material. Similarly, for the semi-infinite geometry, the best experiment resulted from a prescribed time-variable heat flux producing a step change in temperature and two temperature sensors, with one at the heated surface and the second at an internal location determined by a particular dimensionless time. It is not easy to achieve a step change in temperature by applying a time-variable heat flux. However, a finite-duration constant heat flux can be readily accomplished by applying a constant voltage across a heater of known resistance for a finite time period.

This paper focuses on the analytical design of optimal transient heat conduction experiments performed in our laboratory on orthotropic materials (composite materials can be modeled as such). These experiments have been designed to estimate k and c of carbon-fiber/epoxy-matrix composite materials. Three different cases were considered: one-dimensional heat conduction experiments in cured composite materials of finite thickness, one-dimensional heat conduction experiments in thick cured composites which can be approximated by a semi-infinite geometry, and one-dimensional heat conduction in finite composite materials undergoing curing. For the two finite geometries, the optimal heating durations were considered for one-dimensional plates having an isothermal condition opposite to the heated surface. The results of this analysis were applied in our laboratory on composite materials; however, this analysis can also be applied to metals with and without chemical reactions, provided the $x = L$ surface is nearly isothermal.

THEORETICAL PROCEDURE

The choice of an optimal design must be based on some criteria. Three optimality criteria are given by Beck and Arnold [13, p. 432]; all of these criteria are

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NOMENCLATURE

- c volumetric heat capacity [J m^{-3} °C^{-1}]
- C_{ij}^+ i-th row j-th column entry of the product of the sensitivity coefficient matrix and its transpose
- D' determinant of the sensitivity coefficient matrix and its transpose
- D dimensionless determinant of the sensitivity coefficient matrix and its transpose
- g_0 volumetric heat generation [W m^{-3}]
- k thermal conductivity [W m^{-1} °C^{-1}]
- L finite thickness of the material of interest [m]
- m number of sensors used
- n number of measurements by each thermocouple
- p number of parameters
- q^0 heat flux [W m^{-2}]
- t time [s]
- t_h^+ nondimensional heating time
- t_n^+ nondimensional duration of the experiment
- T temperature [°C]
- $\mathbf{T}$ $(mn \times 1)$ column vector of transient calculated temperatures
- T_0 initial temperature [°C]
- T_{Max} maximum nondimensional temperature reached between the start and the end of the experiment
- x spatial position [m]
- x_0 sensor location inside the semi-infinite body [m]
- $\mathbf{X}$ $[mn \times p]$ sensitivity coefficient matrix
- $X_{jk}(i)$ partial derivatives of T at t_i by the j-th sensor with respect to the k-th parameter
- $X_{i,1}^+$ i-th sensitivity coefficient associated with k
- $X_{i,2}^+$ i-th sensitivity coefficient associated with c.

Greek symbols

- α thermal diffusivity [m^2 s^{-1}]
- β_1 unknown parameter
- λ_m eigenvalue.

Superscript

- $+$ non-dimensional.

related to the sensitivity coefficient matrix, $\mathbf{X}$, which is described below. The entries of this matrix are the dimensionless sensitivity coefficients. There are two parameters under consideration: thermal conductivity, k, and volumetric heat capacity, c. The two dimensionless sensitivity coefficients associated with these parameters, $X_{i,1}^+$ and $X_{i,2}^+$ are defined as

$$X_{i,1}^+ = \frac{k}{q_0 L/k}\frac{\partial T_i}{\partial k} \quad i = 1,2,\ldots,n \tag{1}$$

$$X_{i,2}^+ = \frac{c}{q_0 L/k}\frac{\partial T_i}{\partial c} \quad i = 1,2,\ldots,n \tag{2}$$

where q_0 is a constant heat flux, T is temperature, L is the sample thickness, i is the time index, and n is the number of measurements.

The optimality criterion [13, p. 432] is based on the maximization of the determinant, D', of the sensitivity coefficient matrix and its transpose. It is subject to a *maximum* temperature rise, a *fixed* number of measurements, and the eight standard statistical assumptions. These assumptions are summarized as additive uncorrelated normal errors with zero mean and constant variance, with errorless independent variables, and no prior information. This criterion was selected because it minimizes the hypervolume of the confidence region of the parameter estimates.

In equation form, the determinant, D', for the case of two parameters is

$$D' = \det(\mathbf{X}^T\mathbf{X}) \tag{3}$$

where the sensitivity matrix $\mathbf{X}$ is defined by:

$$\mathbf{X} = [\nabla_\beta T^T(\beta)]^T = \begin{bmatrix} \mathbf{X}(1) \\ \mathbf{X}(2) \\ \vdots \\ \mathbf{X}(n) \end{bmatrix},$$

$$\mathbf{X}(i) = \begin{bmatrix} X_{11}(i) & X_{12}(i) & \ldots & X_{1p}(i) \\ X_{21}(i) & X_{22}(i) & \ldots & X_{2p}(i) \\ \vdots & \vdots & \ldots & \vdots \\ X_{m1}(i) & X_{m2}(i) & \ldots & X_{mp}(i) \end{bmatrix}. \tag{4a,b}$$

The entries $X_{jk}(i)$ of this matrix are the partial derivatives of the dependent variable T measured at time t_i by the j-th sensor with respect to the k-th parameter (here there are two parameters: thermal conductivity, and volumetric heat capacity). These partial derivatives are the sensitivity coefficients defined by equations (1) and (2). They are defined for uniformly spaced measurements in time between 0 and t_n^+, and for a fixed large number of time steps. The constraint of a fixed large number of equally spaced measurements and the same maximum temperature rise can be incorporated in D' by defining a dimensionless D for two parameters by [13, p. 434]

$$D = C_{11}^{+}C_{22}^{+} - (C_{12}^{+})^2 \quad (5)$$

where

$$C_{ij}^{+} = \left(\frac{1}{T_{\max}^{+2}}\right)\left(\frac{1}{mt_{n}^{+}}\right)\sum_{r=1}^{m}\int_0^{t_n^+}\left(\beta_i\frac{\partial T_r^+}{\partial \beta_i}\right)\left(\beta_j\frac{\partial T_r^+}{\partial \beta_j}\right)\mathrm{d}t^+ \quad (6)$$

where $\beta_1 = k$ and $\beta_2 = c$. The term C_{ij}^{+} is the time average of the temperature for all sensors divided by the square of the dimensionless T-rise; this incorporates in D the complete sensitivity for the entire experiment, over all relevant times and over all sensors.

Numerical values for the temperature distribution in the geometry of interest are needed; for the investigated cases, these solutions are derived by classical methods [18]; finite element or finite difference methods could also be used. The sensitivity coefficients are then computed by differentiating the temperature solutions with respect to thermal conductivity or volumetric heat capacity. The determinant, D, is calculated from equation (5). The optimal experimental conditions are then established through the comparison of the values of D obtained for different experimental parameters, such as the duration of the heating time, the duration of the experiment (or equivalently, the duration after heating), and the sensor placement within the composite.

ANALYSIS

Three cases were considered in this study. First, one-dimensional heat transfer was considered in a finite cured carbon/epoxy composite, with the heat transfer in the direction perpendicular to the fiber axis. In the second case, one-dimensional heat transfer was studied in a thick cured carbon/epoxy composite thermally behaving as a semi-infinite body. Finally, the finite composite was considered during curing, and a heat generation term is added in the analysis to account for the exothermic chemical reactions of the epoxy matrix.

Finite cured composite (X21B50T0)

Carbon-fiber/epoxy-matrix composite materials tend to have low thermal conductivities for which an isotherm can readily be approximated at the unheated surface. The experiment shown in Fig. 1 and analyzed here allows for the measurement of this relatively low thermal conductivity. The specimen is a slab of finite thickness, L, with one boundary subjected to a heat flux produced by a heater (constant for a prescribed time and then zero), and the second boundary having a constant temperature (the number for this case is X21B50T0; see Beck *et al.* [18]). The advantages of this experiment include the ability to obtain the heat flux experimentally, the simplicity of

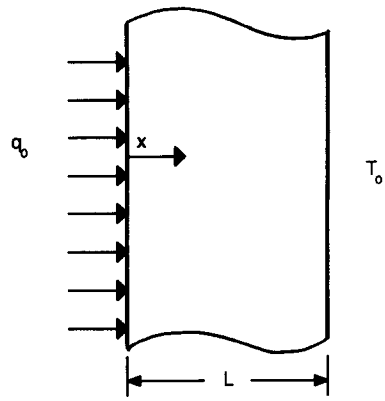

FIG. 1. Cured composite geometry.

the experimental procedures (for example, the heat flux is controlled by simply turning it on and off) and the relative ease of the composite sample preparation due to its simple geometry. The heat conduction equation, boundary conditions, and initial conditions in nondimensional form are

$$\frac{\partial^2 T^+}{\partial x^{+2}} = \frac{\partial T^+}{\partial t^+}, \quad 0 < x^+ < 1, \quad t^+ > 0 \quad (7a)$$

$$\frac{\partial T^+}{\partial x^+} = \begin{cases} -1 & 0 < t^+ \leqslant t_h^+ \\ 0 & t_h^+ < t^+ \leqslant t_n^+ \end{cases}, \quad \text{at} \quad x^+ = 0 \quad (7b)$$

$$T^+ = 0, \quad \text{at} \quad x^+ = 1, \quad t^+ > 0 \quad (7c)$$

$$T^+ = 0, \quad \text{for} \quad 0 \leqslant x^+ \leqslant 1, \quad t^+ = 0. \quad (7d)$$

The thermal properties are assumed to be T-independent. The dimensionless time t_h^+ is the dimensionless heating duration. The dimensionless variables are defined as

$$T^+ = \frac{T - T_0}{q_0 L/k}, \quad t^+ = \alpha t/L^2, \quad t_h^+ = \alpha t_h/L^2, \quad x^+ = x/L. \quad (8a,b,c,d)$$

Equation (7b) gives the heat flux condition, and equation (7c) gives the isothermal condition.

One method for the solution to this problem involves the use of the method of superposition. Up to time t_h^+, the temperature solution of equation (7a) is obtained for a heat flux condition starting at time zero using the method of separation of variables [18, p. 171]:

$$T^+(x^+, t^+) = (1 - x^+) - 2\sum_{m=1}^{\infty}\frac{1}{\lambda_m^2} \times \cos(\lambda_m x^+)\,\mathrm{e}^{-\lambda_m^2 t^+}, \quad 0 < t^+ \leqslant t_h^+ \quad (9a)$$

where λ_m is equal to $(2m-1)\pi/2$. Note that the maximum value of T^+ occurs at steady state and is at $x^+ = 0$; the maximum value is $T^+(0, \infty) = 1$. For $t > t_h^+$, superposition is employed, and the solution for a heat flux condition starting at dimensionless time t_h^+ is subtracted from the solution for the heat flux condition starting at time zero, shown above in equation (9a). The resulting solution is:

$$T^+(x^+,t^+) = -2\sum_{m=1}^{\infty}\frac{1}{\lambda_m^2}\times\cos(\lambda_m x^+) \times[e^{-\lambda_m^2 t^+} - e^{-\lambda_m^2(t^+-t_h^+)}], \quad t_h^+ < t^+ \leqslant t_n^+ \quad (9b)$$

where t_n^+ is the final time for taking measurements.

The next step is to compute the dimensionless sensitivity coefficients defined by equations (1) and (2). The differentials in these equations are found by differentiating the temperature solutions shown in equations (9a,b) with respect to the parameters, k and c. The i subscript used in equations (1) and (2) is dropped in the remainder of the paper for simplicity of notation. The resulting sensitivity coefficients for the thermal conductivity are:

$$X_1^+ = \frac{k}{q_0 L/k}\frac{\partial T}{\partial k} = -(1-x^+)+2\sum_{m=1}^{\infty}\frac{1}{\lambda_m^2} \times\cos(\lambda_m x^+)\,e^{-\lambda_m^2 t^+}(1+\lambda_m^2 t^+), \quad 0 < t^+ \leqslant t_h^+ \quad (10a)$$

$$X_1^+ = 2\sum_{m=1}^{\infty}\frac{1}{\lambda_m^2}\cos(\lambda_m x^+)[e^{-\lambda_m^2 t^+}(1+\lambda_m^2 t^+) - e^{-\lambda_m^2(t^+-t_h^+)}(1+\lambda_m^2(t^+-t_h^+))], \quad t_h^+ < t^+ \leqslant t_n^+. \quad (10b)$$

Likewise, the sensitivity coefficients for the volumetric heat capacity are:

$$X_2^+ = \frac{c}{q_0 L/k}\frac{\partial T}{\partial c} = -2\sum_{m=1}^{\infty} \times\cos(\lambda_m x^+)\,t^+\,e^{-\lambda_m^2 t^+}, \quad 0 < t^+ \leqslant t_h^+ \quad (11a)$$

$$X_2^+ = 2\sum_{m=1}^{\infty}\cos(\lambda_m x^+)[-t^+\,e^{-\lambda_m^2 t^+} + (t^+ - t_h^+)\,e^{-\lambda_m^2(t^+-t_h^+)}], \quad t_h^+ < t^+ \leqslant t_n^+. \quad (11b)$$

The sensitivity coefficients for thermal conductivity and volumetric heat capacity are shown as functions of time for $t_h^+ = t_n^+$ in Fig. 2. The magnitude of the thermal conductivity sensitivity coefficient is about equal to that of T^+, while the sensitivity coefficients for the volumetric heat capacity are smaller but still on the order of T^+ for some times. Also note that the shapes (except at early times) of the thermal conductivity and volumetric heat capacity sensitivity coefficient curves are quite different. These observations verify that the two sensitivity coefficients, X_1^+, and X_2^+, are 'large' (i.e. on the order of T^+), and uncorrelated (different shapes), which are desirable conditions for parameter estimation. Since the k sensitivity coefficient shown in Fig. 2 is larger in magnitude than the c sensitivity coefficient, on the average k should be estimated more accurately than c for such experiments. For the insulation conditions discussed in ref. [13], the opposite is true. Hence, the isothermal condition is preferred over the insulation condition if k is of primary interest.

The final step of the analysis requires the determination of the determinant, D. The maximum temperature rise, T_{max}^+, is first determined from equations (9a,b) with $x^+ = 0$, and the sensitivity coefficients, X_1^+ and X_2^+, are found from equations (10a,b) and (11a,b). The C_{ij}^+ matrix coefficients are then found from equation (6), using the calculated values of T_{max}^+, X_1^+, and X_2^+ and integration. Finally, the determinant, D, is calculated from equation (5). The determinant was found and compared using different heat-

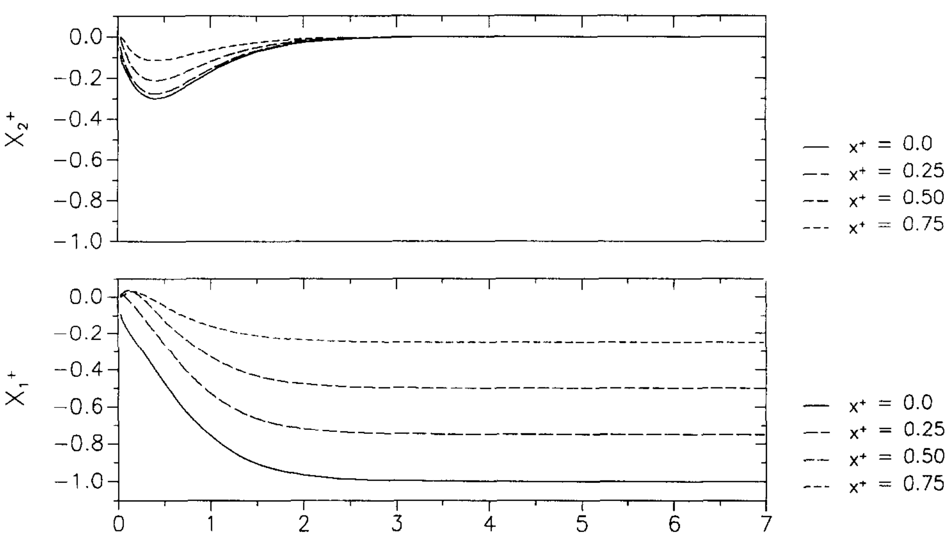

FIG. 2. Dimensionless sensitivity coefficients, X_1^+ and X_2^+, as functions of dimensionless spatial position, x^+, for cured composite with finite geometry.

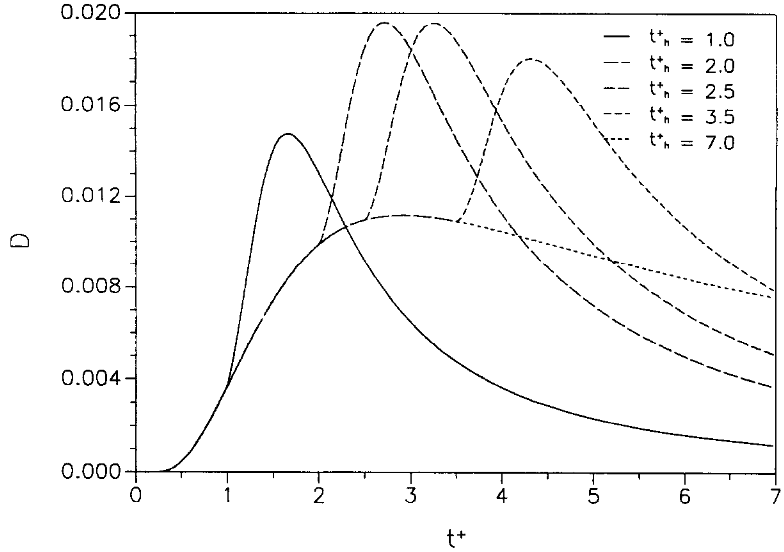

FIG. 3. Effect of the dimensionless heating time, t_h^+, on the determinant, D, for the cured composite with finite geometry.

ing times (t_h^+) and different experiment durations (t_n^+) (Fig. 3), and the different sensor locations (Fig. 4).

Semi-infinite cured composite (X20B5T0)

The second case considered is similar to the first case, with the exception that the sample is thermally thick; thus, it behaves as a semi-infinite body with a constant heat flux at its surface (the number in this case is X20B5T0; [18, chap. 2]). In this case, the heat conduction equation, boundary conditions, and initial conditions are:

$$k\frac{\partial^2 T}{\partial x^2} = c\frac{\partial T}{\partial t}, \quad 0 < x < \infty, \quad t > 0 \tag{12a}$$

$$-k\frac{\partial T}{\partial x} = \begin{cases} q_0 & 0 < t^+ \leqslant t_h \\ 0 & t > t_h \end{cases}, \quad \text{at} \quad x^+ = 0 \tag{12b}$$

$$T = T_0, \quad \text{at} \quad x \geqslant 0, \quad t = 0. \tag{12c}$$

For convenience, the following dimensionless groups are defined for this case:

$$T^+ = \frac{T - T_0}{q_0 x_0 / k}, \quad t^+ = \frac{\alpha t}{x_0^2}, \quad x^+ = \frac{x}{x_0} \tag{13a,b,c}$$

where x_0 can be any given location inside the body (not at the surface).

The basic dimensionless temperature is given by [18, p. 151]

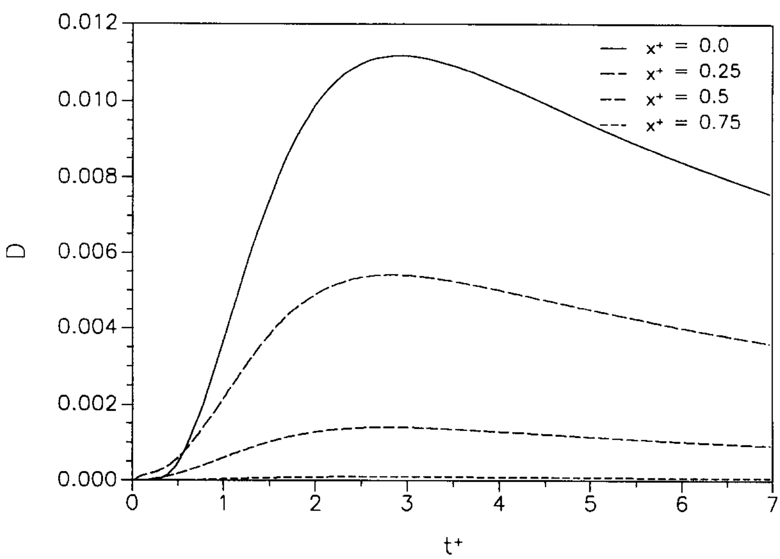

FIG. 4. Effect of a single sensor's location, x^+, on the determinant, D, as a function of the dimensionless time, t^+, for the cured composite with finite geometry.

$$T^+(x^+,t^+) = 2\sqrt{t^+}\ \mathrm{ierfc}\left[\frac{x^+}{2\sqrt{t^+}}\right], \quad 0 < t^+ \leqslant t_h^+. \tag{14}$$

Equation (14) is used in a similar manner as for case X21B50T0 to obtain the equation for $t^+ > t_h^+$, and the equations for X_1^+ and X_2^+.

The determinant, D, is again calculated from equation (5) as described for the first case involving a finite geometry. The solutions corresponding to different heating times t_h^+ are found and shown in Fig. 5 for a sensor at the heated surface and another one at an in-depth location. (Since there are two locations, m in equation (6) is 2.)

Composite undergoing curing (X21B50G1T0)

The third case considered is that of finite-body one-dimensional heat conduction through a carbon-fiber/epoxy-matrix composite material undergoing curing. The experimental set-up and boundary conditions used for the cured composite case are also considered here. The solution procedure is similar to that shown for the cured composite; however, in this case, heat is generated from the chemical reactions occurring during the curing of the epoxy matrix. This results in an extra term appearing in the one-dimensional heat conduction equation. For the purpose of this study, this heat generation term was considered to be constant, and the resulting energy equation in dimensionless form is

$$\frac{\partial^2 T^+}{\partial x^{+2}} + g_0^+ = \frac{\partial T^+}{\partial t^+}, \quad g_0^+ \equiv g_0 L/q_0. \tag{15}$$

The dimensionless terms, T^+, t^+, and x^+ are given by equations (8a,b,d), and the boundary and initial conditions are given by equations (7b–d).

The temperature solution for the energy equation shown by equation (15) and the initial and boundary conditions given in equations (7b–d) is

$$T^+(x^+,t^+) = g_0^+(1-x^+)\left(\frac{x^+ + 1}{2}\right) + (1-x^+) - 2\sum_{m=1}^{\infty} \times \frac{\cos(\lambda_m x^+)}{\lambda_m^2} e^{-\lambda_m^2 t^+}\left(1-(-1)^m \frac{g_0^+}{\lambda_m}\right), \quad 0 < t^+ \leqslant t_h^+ \tag{16a}$$

$$T^+(x^+,t^+) = -2\sum_{m=1}^{\infty} \frac{\cos(\lambda_m x^+)}{\lambda_m^2}\left[e^{-\lambda_m^2 t^+} - e^{-\lambda_m^2 (t^+ - t_h^+)}\right] \times \left[1-(-1)^m \frac{g_0^+}{\lambda_m}\right], \quad t_h^+ < t^+ \leqslant t_n^+. \tag{16b}$$

The sensitivity terms in equations (1) and (2) were found by solving equations (16a,b) for T, and differentiating with respect to the parameters thermal conductivity, k, and volumetric heat capacity, c, respectively.

The determinant, D', is then calculated from equation (3). The matrix coefficients shown in equation (3) and defined by equation (4) are determined from the maximum temperature rise, $T_{\max}^+$, found from equations (16a,b) with $x^+ = 0$, and the sensitivity coefficients, X_1^+ and X_2^+, are found as described above. In this case, the solutions resulting from different heating times, t_h^+, were found and compared. Two different values for the dimensionless heat generation term, g_0^+, were used in this analysis. See Figs. 6 and 7.

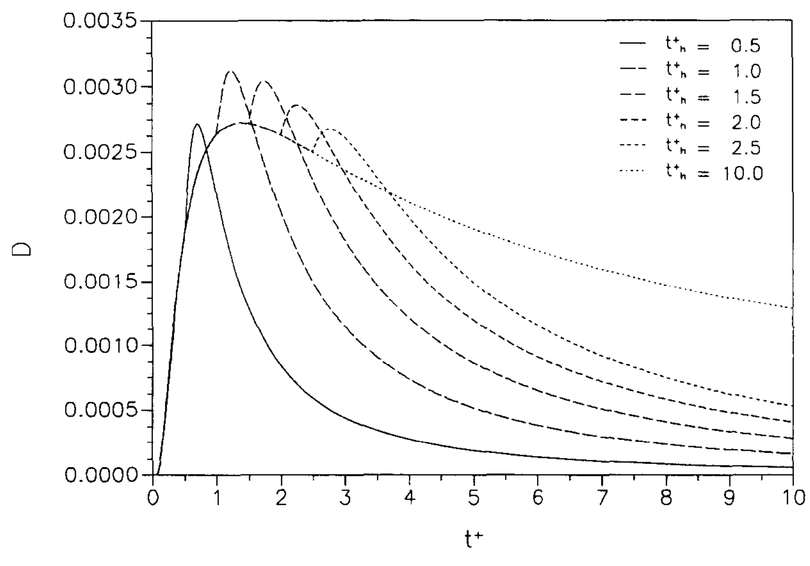

FIG. 5. Effect of the dimensionless heating time, t_h^+, on the determinant, D, for the cured composite with semi-infinite geometry, with a thermocouple at the heated surface and another one at an in-depth location.

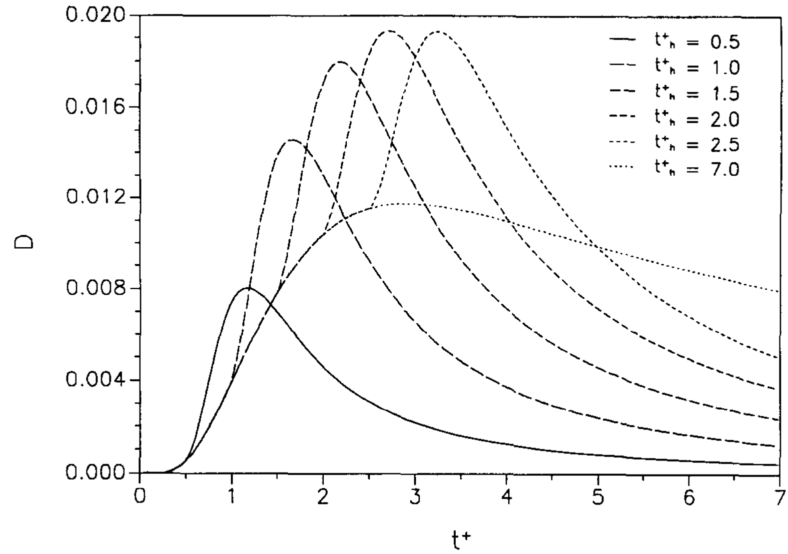

FIG. 6. Effect of the dimensionless heating time, t_h^+, on the determinant, D, for the composite undergoing curing with the dimensionless heat generation term, g_0^+, equal to 0.1.

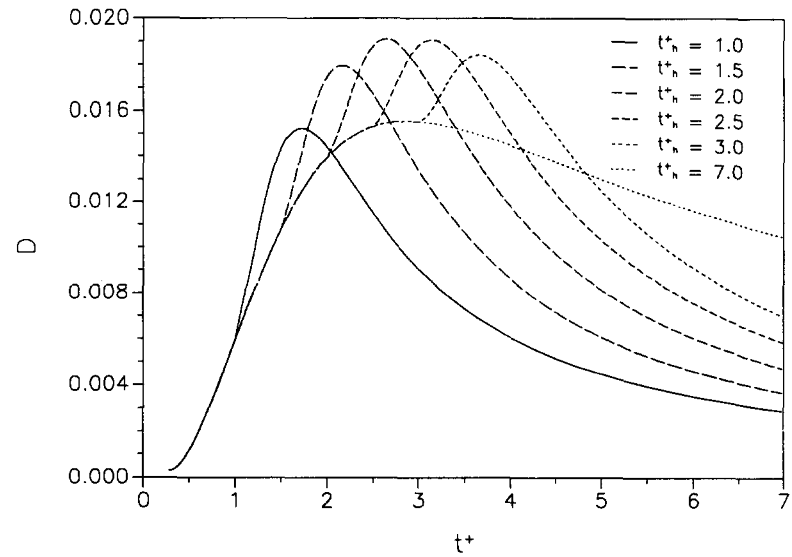

FIG. 7. Effect of the dimensionless heating time, t_h^+, on the determinant, D, for the composite undergoing curing with the dimensionless heat generation term, g_0^+, equal to 1.0.

RESULTS AND DISCUSSION

The determinant, D, was compared for different experimental conditions, such as different heating times (t_h^+), total measurement times (t_n^+), and sensor locations, to determine the optimal experimental conditions. In the first case, a cured composite was considered, and the experimental variables included heating time, total experimental time, and sensor location. In the cases of the cured thick composite and the composite undergoing curing, the experiment was optimized with respect to heating time and total experimental time.

Finite cured composite (X21B50T0)

The optimal criterion used in this study is based on the determinant, D, which involves the sensitivity coefficients, X_1^+ and X_2^+. Therefore, investigation of the sensitivity coefficients can be useful in providing insight into the optimization procedure. For this investigation, the heating time, t_h^+, is considered to be equal to the total experimental time. Figure 2 corresponds to the transient change of the thermal conductivity sensitivity coefficient, X_1^+. Each curve starts at zero and goes to a non-zero negative, steady state value; the magnitude of the sensitivity coefficients is largest at the heated surface. Figure 2 also shows that the volumetric heat capacity sensitivity coefficient, X_2^+, becomes essentially zero shortly after the dimensionless time, t^+, equals 2. This indicates that little additional information is obtained using values of t^+ greater than 2 for the estimation of the volumetric heat capacity. The sensitivity coefficients, as shown in Fig. 2, are not linearly dependent on each other, consequently, k and c can be simultaneously and independently estimated; see ref. [13, p. 349].

The first experimental variable investigated was the heating time, t_h^+, for the heat flux boundary condition at $x^+ = 0$. Five different dimensionless heating times were considered, and the results for the determinant, D, are shown in Fig. 3 for a single sensor at $x^+ = 0$. The curve having the highest peak represents the maximum value of the determinant, D; this value of D equal to 0.0195 corresponds to a dimensionless heating time of about 2.5, and a 'cooling' time of 0.73. A heating time of 2.25 (not shown) results in a slightly higher maximum value of D; in this case, the maximum value is approximately equal to 0.020 which occurs at dimensionless time of 2.983.

An interesting aspect of Fig. 3 is that the optimal heating time curve obtained by joining the peaks of the four different adjoining curves has a rather flat peak between dimensionless heating times of 1.5 and 3.5; this implies that any values used within this range will be close, in terms of the optimum, to the optimal value. Hence, the choice of the optimal heating time does not have to be precise. Notice that the choice of the duration of the experiment after heating, $t_n^+ - t_h^+$, is crucial. The four high-peak curves show a sudden drop in the value of D, which means that taking the data longer than the time at which D is maximum lowers the value of D and degrades the quality of the sought thermal properties k and c (it is assumed that the same number of measurements is taken regardless of the experiment duration). The maximum value of D occurs a constant 0.73 dimensionless time interval after the heating time; this implies that the total duration of the optimal experiment is about 3. Note also that for a given heating time, an error of say 10% in the chosen optimal duration of the experiment has less effect on driving the value of D away from the maximum one than a 10% error in an experiment lasting longer, and for which the value of the determinant is away from the peak. Note that information regarding the rapid degradation in the value of D (if the same number of measurements is spread over a larger time) is lost if an optimizing program is used and only the maximum is found.

Another clarification might help. More measurements invariably contain more information if the same size time step is taken, such as going to time 125 s rather than 100, both with time steps of 1 s. In such cases, the confidence interval should decrease with increasing number of measurements. That is not what is being held constant in this analysis; the number of equally spaced measurements is held constant. If the total duration of the experiment is allowed to become large for a fixed number of measurements, then it is possible for the finite duration of heating experiment to be poorer than the heating over the total experiment (see for example Fig. 3 for $t_h^+ = 2$ and t^+ greater than 5).

The next factor considered was the sensor location. Figure 4 shows four curves corresponding to four different sensor locations. The maximum value of the determinant, D, corresponds to the sensor at the heated surface. This is because, as shown in Fig. 2, the sensitivity coefficients at the heated surface have the greatest magnitude. It is then concluded that, when using a single sensor, it is best to place it as close to the heated surface as possible, provided the measurement errors are independent of location.

Semi-infinite cured composite (X20B5T0)

The experimental variable investigated was the duration of the heat pulse referred to as the heating time, t_h^+. The determinant, D, with one sensor at the heated surface and a second one at an internal location, x_0, was calculated for six different dimensionless heating times. The resulting curves are shown in Fig. 5. This figure shows that the optimal dimensionless heating time is approximately 1.0 which corresponds to a value of D of 0.0031. The optimal duration of the experiment is shorter for this geometry than for the finite body geometry; indeed, the total optimal experiment lasts only a heating time period plus about 0.2, making the optimal dimensionless total time equal to about 1.2. The abrupt increase after heating stops, and rapid decrease after the maximum illustrate the importance of plots such as Fig. 5. If the duration of a fixed number of equally-spaced measurements is extended, the value of the determinant decreases so much as to become smaller than that one corresponding to collecting measurements only over the heated period.

Comparison of results

At this point, a comparison of the current study with other published results is needed. Two geometries are described in ref. [13, Chap. 8], a finite and a semi-infinite body. These two geometries are subjected to boundary conditions different from the ones used in the current study. Table 1 lists the boundary conditions, the locations of the temperature sensors, and the values of the determinant D for the different cases. Cases I, III, IV, and V come from ref. [13, Chap. 8], while cases II, VI, and VII are from the present study. For the finite geometry, cases VI and VII show a larger value of D (0.020 and 0.012 respectively) than cases III, IV, or V. This implies that, for a finite geometry, to estimate the thermal properties of interest, it is better to have a constant temperature boundary than to insulate one side with a finite duration heat pulse on the other side. For the semi-infinite geometry, a finite duration heat flux boundary condition with measurements lasting 0.2 after the end of the heat flux (with $D = 0.0031$) makes a better

Table 1. Comparison of the maximum of the determinant, D, values for 7 cases

Case	Geom.	Boundary condition(s)	Sensor location(s)	Max. D	Opt. t_h^+	Opt. dur.	
I	semi-infinite	(X20B1T0)	heated surface and in-depth	0.00263	$t_n^+ = 1.5$	$t_n^+ = 1.5$	a
II	semi-infinite	(X20B5T0)	heated surface and in-depth	0.0031	1.0	1.2	b
III	finite	(X22B10T0)	heated surface	0.00098	$t_n^+ = 1.2$	$t_n^+ = 1.2$	a
IV	finite	(X22B10T0)	$x^+ = 0$ and 1	0.0058	$t_n^+ = 0.65$	$t_n^+ = 0.65$	a
V	finite	(X22B50T0)	$x^+ = 0$ and 1	0.0088	0.4	0.6	a
VI	finite	(X21B50T0)	heated surface	0.020	2.25	2.98	b
VII	finite	(X21B10T0)	heated surface	0.012	$t_n^+ = 7$	$t_n^+ = 7$	b

a. Beck and Arnold [13, Chap. 8].
b. This study.

experiment than a continuous heat flux that lasts during the whole experiment (where D is 0.00263).

The results of four experiments conducted in our laboratory on a finite slab were found consistent with the analytical optimal experiments study. Two experiments were optimal and two were not. One optimal experiment ended at the optimal dimensionless time of 2.5, while the other optimal experiment ended at a dimensionless time of 0.73 after the heating ends. The non-optimal experiments were similar to the optimal ones except for their short durations of 0.5 and 1.23. The areas of the square confidence regions corresponding to the four experiments were compared. The area of the confidence region corresponding to the first optimal experiment was 16.32 (W m^{-1} °C^{-1}) (J m^{-3} °C^{-1}) as opposed to the non-optimal one of 2883.58, while the area of the confidence region corresponding to the second optimal experiment was 11.43 as opposed to the non-optimal one 2291.53. Better experiments (reflected by smaller confidence region areas) were hence achieved when the dimensionless heating time is about 2.5; these experiments were further improved by taking measurements after the end of the heating period for a dimensionless time of 0.73.

Finite composite undergoing curing (X21B50G1T0)

The experimental variable considered for the composite undergoing curing was the heating time, t_h^+. Results for the determinant, D, with a single sensor at the heated surface and six different dimensionless heating times are shown in Fig. 6 for a dimensionless heat generation term g_0^+, equal to 0.1 and in Fig. 7 for g_0^+ equal to 1.0. The curves shown in both of these figures indicate that the highest value for the determinant, D, resulted from a dimensionless heating time, t_h^+, of 2 and occurs approximately a constant 0.66 dimensionless time interval after the heating ends. Further analysis of the case with $g_0^+ = 0.1$ indicated that the maximum value of D of 0.0205 is slightly higher with $t_h^+ = 2.125$. This heating time is only 5.5% less than the optimal value of 2.25 found for the cured composite sample ($g_0^+ = 0$). The optimal heating time of 2 found for the case with $g_0^+ = 1.0$ is 11% less than the optimal value for the $g_0^+ = 0$ case.

Comparing Figs. 6 and 7 with Fig. 3 ($g_0^+ = 0$), the magnitude of the maximum determinant value decreases as the value of g_0^+ increases. In addition, the peaks of the curves obtained by joining the maximum determinant values in Figs. 3, 6, and 7 flatten as the value of g_0^+ increases indicating that any heating time close to the optimum will produce similar results. A third interesting point is that the maximum determinant value for the case in which the heating time is equal to the experimental time increases as the value of g_0^+ increases.

SUMMARY AND CONCLUSIONS

This paper focused on the optimization of one-dimensional transient experiments for the determination of the thermal conductivity and the volumetric heat capacity of carbon-fiber/epoxy-matrix composite materials. An optimal criterion was presented, and the optimization procedure for three cases, including finite geometry cured composites, finite composites undergoing curing, and semi-infinite cured composites, was derived. The effects of three experimental parameters (heating time, duration of the experiment, and the placement of sensors) on the optimal criterion were then considered. The analysis was performed for the linear case where the thermal properties are constant; however, a similar procedure can be applied to non-linear problems corresponding to temperature dependent properties.

From this analysis, it was concluded that the optimal dimensionless heating time for a fixed number of measurements and for one-dimensional experiments with a heat flux on one boundary and a constant temperature at the second boundary of cured composite samples is approximately the dimensionless time of 2.25 with an additional dimensionless time of 0.73 after the heating; the optimal location of sensors in the case is the heated surface. In addition, the analysis of the experimental design for the finite geometry in this study resulted in a higher value of the optimal criterion (the determinant, D) than was found in ref. [13] for an insulated surface (X22B10T0). This indicates that the experimental design presented here, with one boundary at a constant temperature and the second boundary with a finite duration heat flux (X21B10T0), is a better one for estimating the thermal properties than with an insulated boundary. Also, since composite materials tend to have low thermal conductivity values are are consequently difficult to insulate, this X21B10T0 design is much easier to implement experimentally than the X22B10T0 design [13]. It should also be noted that the X21B10T0 case produces more accurate (on the average) parameter estimates than the X22B10T0 case; this is particularly true for the thermal conductivity.

For a thick composite behaving as a semi-infinite body with a finite duration constant heat flux (Case II), the value of the optimal criterion found in this study is larger than that of Case I shown in Table 1. Imposing a finite-duration heat flux for a dimensionless time 1.0, and taking measurements for a nondimensional period of 0.2 after that is better than heating throughout the entire data collection process.

Finally, in the case of the composite undergoing curing, dimensionless heat generation terms of 0.1 and 1.0 reduced the optimal dimensionless heating time 5.5% and 11%, respectively, from the value obtained with no heat generation. In addition, it was found that increasing the dimensionless heat generation term slightly reduced the optimal criterion (the maximum value of the determinant, D), which indicates that the potential parameter estimates would be slightly less accurate compared with those obtained without heat generation.

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