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# DESIGN AND VALIDATION OF OPTIMAL EXPERIMENTS FOR ESTIMATING THERMAL PROPERTIES OF COMPOSITE MATERIALS

By

Ramsis Taktak

# A THESIS

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### ABSTRACT

# DESIGN AND VALIDATION OF OPTIMAL EXPERIMENTS FOR ESTIMATING THERMAL PROPERTIES OF COMPOSITE MATERIALS

By

# Ramsis Taktak

Composite materials have gained an unprecedented interest in the last twenty years. Their superior strength-to-weight ratios have made them very popular with aerospace, automotive, boat, biomedical, and even the sporting goods industry. The radarabsorbing quality of composite materials, together with their strength-to-weight ratios, make these materials attractive for military applications.

When used in air and space vehicles, composite materials are exposed to harsh thermal loads. An understanding of the thermal behavior of these materials became necessary. The thrust of this work is the estimation of two thermal parameters: thermal conductivity and volumetric heat capacity. There are three main parts to the current research; each part describes a different aspect of estimating these thermal properties.

The first part compares two experimental research paradigms pertaining to the effect of the temperature rise on the estimated parameters during transient parameter estimation experiments.

The second part of this research relates to optimal experiments. Two onedimensional experiments for finite and semi-infinite geometries are shown to be superior to some previously published ones. The two experimental parameters of heating time and cooling time are especially of interest for these experiments. An optimal value is obtained for each parameter using a selected optimality criterion. A two-dimensional case is studied following the same methodology. Design curves relating the optimality criterion to the geometry and directional properties of the material of interest are obtained. The analytical results of the optimal experiments pertaining to the finite one-dimensional geometry are verified using an experimental technique developed by Garnier and Beck.

In the third part of this research, the thermocouple-induced errors, which are usually small quantities, are quantified using an analytical approach. Finite difference and finite element methods are supplemented because of their limitations in calculating small differences. Some experiments are then designed and implemented in order to verify these analytical results.

The results of the three different parts of this research have great potential for improving the estimation of composite material thermal properties.

to my parents, Lamjed and Saida Taktak

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# LIST OF SYMBOLS

x,y	: cartesian coordinates (m)
x <sub>0</sub>	: cartesian coordinate used with the semi-infinite geometry (m)
r	: cylindrical coordinate (m)
a	: radius of the thermocouple wire, and width of the two-dimensional geometry (m)
L	: thickness of the finite geometry, and thermocouple in-depth location (m)
x*,y*	: non-dimensional spatial position
н	: thermocouple radius-to-depth ratio
A	: area (m <sup>2</sup> )
v	: volume (m <sup>3</sup> )
t	: time (s)
t <sup>+</sup>	: non-dimensional time
t <sub>h</sub> +	: non-dimensional heating time
t"+	: non-dimensional duration of the experiment
u	: non-dimensional time used with Green's functions
k	: thermal conductivity (W/m°C)
k <sub>1</sub> , k <sub>2</sub>	: thermal conductivities at temperatures $T_1$ and $T_2$ (W/m°C)

 $k_x, k_y$ : directional thermal conductivities (W/m°C)

 $\rho c$  : volumetric heat capacity (W/m<sup>3</sup>°C)

 $\rho c_1$ ,  $\rho c_2$ : volumetric heat capacities at temperatures T<sub>1</sub> and T<sub>2</sub> (W/m<sup>3</sup>°C)

 $\zeta_0,\zeta_1$  : alternative intercept and slope of the thermal conductivity as a function of temperature (W/m°C) and (W/m°C²)

 $\zeta_{2},\zeta_{3}$  : alternative intercept and slope of the volumetric heat capacity as a function of temperature (W/m<sup>3o</sup>C) and (W/m<sup>3o</sup>C<sup>2</sup>)

 $\rho c_{w}$  : volumetric heat capacity of the thermocouple wire (W/m<sup>3</sup>°C)

 $\Delta \rho c$  : difference between  $\rho c$  and  $\rho c_w$ 

 $\alpha$  : thermal diffusivity (m<sup>2</sup>/s)

$$q_0$$
 : heat flux (W/m<sup>2</sup>)

q : average heat flux  $(W/m^2)$ 

T : temperature (°C)

 $T_0$  : initial temperature (°C)

T : average temperature (°C)

 $T_w$  : temperature of the wire (°C)

 $T_L$  : temperature at x=L (°C)

 $T_{-}$ : temperature at the wire location had the wire not been there (°C)

T<sup>+</sup> : non-dimensional temperature

 $T_{Max}^{+}$ : maximum non-dimensional temperature reached between the start and the end of the experiment

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X<sup>+</sup><sub>i</sub> : i-th dimensionless sensitivity coefficient

 $\lambda_n, \lambda_m$  : eigenvalues

p : number of parameters

m : number of sensors used

n : number of measurements by each thermocouple

P : power (W)

U : voltage (V)

- R : electrical resistance of the heater (Ohm)
- $Y_i$  : average temperature at time  $t_i$  from m sensors (°C)
- Y<sub>ii</sub> : measured temperature at time t<sub>i</sub> corresponding to the j-th measurement

s : standard deviation

- S : weighted sum of squares
- Y : (mn×1) column vector of transient measured temperatures
- W : (mn×mn) weighting matrix
- T : (mn×1) column vector of transient calculated temperatures
- $\beta$  : (p×1) parameter vector
- **b** : (p×1) vector estimator of  $\beta$
- $\nabla_{\beta}$  : derivative with respect to vector  $\beta$
- $X(\beta)$  : (mn×p) sensitivity coefficient matrix

 $X_{jk}(i)\;$  : partial derivative of the temperature T at time  $t_i$  by the j-th sensor with respect to the k-th parameter

D : determinant of the product of the sensitivity coefficient matrix and its transpose

 $C_{ij}^{+}$ : i-th row, j-th column entry of the product of the sensitivity coefficient matrix and its transpose

 $\sigma_k$ ,  $\sigma_q$ ,  $\sigma_L$ : variances on k, q, and L

 $\phi(q_0,t)$  : exact temperature at time t for the constant heat flux  $q_0=1$ 

 $\Delta \phi$  : temperature disturbance (°C)

K(t) : thermocouple function kernel

# CHAPTER 1

### INTRODUCTION

Special interest in composite materials has been growing in the last twenty years. This interest has been fueled by the superior dynamic performance of these materials, by their light weight, and by their radar absorbing quality. The high strength and low weight properties make composite materials ideal for use in space vehicles (National Aerospace Plane), airplanes (commercial and military), ships, and in mass-produced goods such as cars, boats, motorcycles, tennis rackets, and baseball bats. The use of composite materials in most of these applications means reduced fuel consumption, which in turn leads to reduced emissions, and eventually to cleaner air, and a better environment.

Composite materials are much more complex than ordinary metallic materials because they are non-homogeneous. They are made of two ingredients: fibers and matrix. The fibers can be carbon fibers, aluminum fibers, glass fibers, kevlar fibers, titanium fibers, etc. The matrix is usually an epoxy (and there are different kinds) made of a resin and a curing agent. When one or more types of fibers are combined with the epoxy to obtain the composite material, the strength-to-weight ratio of the composite is higher than that of the bulk fibers. The use of composite materials in aerospace applications and airplanes subjects them to harsh thermal conditions, which can lead to disasters if not looked at carefully. To account for these thermal conditions, the study of the thermal behavior of composite materials became a significant parameter of the evaluation process of these materials. An important part of the thermal studies of composite materials pertains to the estimation of their thermal properties: conductivity and volumetric heat capacity.

The process of estimating the thermal properties of materials (or parameter estimation) combines the two classical approaches usually performed separately: experiments and theory. This parameter estimation process was the driving force behind the current research. This research has three main objectives: the investigation of two experimental paradigms, the analytical investigation and experimental verification of some optimal thermal parameter estimation experiments, and the analytical investigation and experimental verification of thermocouple errors.

The first objective pertains to the investigation of two competing experimental paradigms which relate to the temperature rise achieved in the parameter estimation experimental procedure. The effects of low and large temperature rises on the confidence regions of the estimated temperature dependent thermal properties are investigated using an experimental approach.

The second objective of the current research is to design optimal alternative thermal parameter estimation experiments and implement them in the laboratory. To meet this objective, three experiments are investigated: two one-dimensional experiments (finite and semi-infinite), and one two-dimensional experiment. The durations of heating and

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cooling are the main parameters of interest for the one-dimensional experiments. A nondimensional group containing geometry and directional thermal properties of the twodimensional case is the main parameter of interest. The experimental implementation is only applied to the finite one-dimensional experiment. The confidence regions are compared for the experimental data and are shown to confirm the theoretical results.

The third and last objective of the current research is to quantify the errors caused by the thermocouples embedded in the specimens to measure internal temperatures. This was done by Pfahl and Dropkin in 1966 using finite differences. In the current research however, finite differences and finite elements are avoided because of their limitations in calculating small differences (such as thermocouple errors); instead, a pure analytical method is followed. The method employs the Unsteady Surface Element (USE) method, and is shown to be more flexible than methods using finite elements or differences. The results of this USE method are also compared with those of a previous investigation by Beck (1968) based on the evaluation of some convolution kernels.

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# CHAPTER 2

# BACKGROUND

# 2.1 Introduction

In order to provide a framework for the several analyses in this dissertation, it is necessary to provide a background outlining the present practice. This chapter provides this background. These analyses involve the investigation of an experimental paradigm, the investigation of optimal experiments (Chapters 3 and 6), and the investigation of measurement errors (Chapters 4 and 5). In each case, the analyses have been supplemented by experiments.

## 2.1.1 Motivation

Composite materials are important for many advanced applications. They are much more complex than ordinary metallic materials because they contain fibers and a matrix supporting the fibers. Fine fibers (10 to 15 microns in diameter) typically have much improved strength-to-weight ratios compared to the bulk properties of the fiber material, which is commonly carbon or glass. The combination of the fibers and matrix makes the composite material.

The thermal properties (thermal conductivity and volumetric heat capacity) of

composite materials are needed for a number of reasons. One reason is that such materials can be subjected to hostile thermal environments during service. Examples are the proposed National Aerospace Plane (NASP), military airplanes such as the Stealth bomber, advanced heat exchangers, and jet engines. These properties are also needed to model the curing and molding of composite materials.

The thermal conductivity of composite materials can be directionally dependent, as a result of the orientation of the fibers. It can also be affected by the thermal contact between the fibers and matrix (Cha and Beck, 1989, Beck and Osman, 1987). These factors make the determination of the thermal properties more varied and difficult to obtain.

#### 2.1.2 Meaning of Effective Thermal Properties

The meaning of the "effective" thermal properties of composite materials must be defined. Effective thermal properties are usually needed and also usually measured in the research to be described. By effective thermal conductivity is meant:

$$k_{\text{eff}} = -\frac{\bar{q}}{(\frac{\partial \bar{T}}{\partial x})}$$
(2.1)

where  $\partial \overline{\mathbf{T}} / \partial \mathbf{x}$  is the average temperature gradient and  $\overline{\mathbf{q}}$  is the average heat flux. Both averages are over the area normal to the direction of flow of heat. The effective volumetric heat capacity is defined by

$$\rho c_{\text{eff}} = \frac{1}{V} \int \rho c \, dv \tag{2.2}$$

where V is volume. Notice that the definitions of these properties in Equations (2.1) and

(2.2) are quite different.

The basic equation in which these effective properties are used is (for temperaturedependent properties)

$$\frac{\partial}{\partial x} \left( k_{\text{eff}}(T) \frac{\partial \bar{T}}{\partial x} \right) = \rho c_{\text{eff}}(T) \frac{\partial \bar{T}}{\partial t}$$
(2.3)

where the average temperature is defined by

$$\overline{T}(\mathbf{x},\mathbf{t}) = \frac{1}{A} \int_{-\infty}^{A} T(\mathbf{x},\mathbf{x},\mathbf{t}) \, d\mathbf{x}$$
(2.4)

where æ and A are the areas normal to x. In the remainder of this dissertation, the subscript "eff" is dropped.

### 2.1.3 Carbon Fiber/Epoxy Material

The composite material of interest in this research is a carbon fiber/epoxy material. The effective thermal properties of a Hercules carbon fibers/Epon 828-DDS epoxy material were estimated in our laboratory at an early stage of the current research. The results of the corresponding experiments are not reported in this dissertation however. The effective thermal properties of such a material are functions of the thermal properties of the carbon fibers and of the epoxy, as well as the volume fraction of the fibers in the composite (Ziebland, 1974, Han and Cosner, 1981). One approach for estimating the effective thermal properties is based on Equations (2.1) and (2.2), and considers the material to be homogeneous (no inclusions, just one component). Another way of estimating these effective thermal properties is to obtain the thermal conductivities and volumetric heat capacities of the different constituents (carbon fibers and epoxy in this case), and to calculate numerically the effective thermal properties.

of Tł ļ st th fi а S \$ S i 5 Initial average values of the specific heats of the two constituents of the composite of interest can be obtained by Differential Scanning Calorimetry (DSC) (Barton, 1985). The densities, on the other hand, can be obtained by following the guidelines of the standard test method for density of glass by buoyancy. This method was described by the American Society for Testing and Materials (ASTM, 1990).

Estimation of the thermal conductivity of carbon fibers is very difficult. The fibers are 10 to 15 microns in diameter, and come in tows of 3,000 to 12,000. The tows are supplied on spools several hundred yards to a couple of miles long. However, the samples of use in our laboratory are rigid solids of surface area much larger than a few square microns. A finite heat flux and/or temperature are usually imposed at both surfaces of the solid sample. Thermocouples are usually instrumented at the surface and inside the samples. The cured epoxy samples used, which are rigid solids of macroscopic surface area, were analyzed in our laboratory as described below.

## 2.1.4 Boundary Conditions

The current parameter estimation problem is based on the heat diffusion differential Equation (2.3) with the following boundary conditions:

$$-k\frac{\partial T}{\partial x}\Big|_{x=0} = q_0(t)$$

$$T\Big|_{x=L} = T_L(t)$$
(2.5)

where T is temperature, x is position, and t is time. The heat flux  $q_0(t)$  is a measured heat flux at one surface, and  $T_L(t)$  is the known temperature at the other surface. The choice of a heat flux boundary condition was partly made because of the practicality and ease of imposing such a boundary condition. Even more important was the optimal experiment. Such analyses are an important part of this dissertation (see Chapter 3), and are the second analysis indicated in the first paragraph of this chapter. A heat flux was generated by applying a voltage across a finite-resistance electrical heater. The power generated by the heater is obtained using Ohm's law as

$$\mathbf{P} = \mathbf{U}^2 \mathbf{R} \tag{2.6}$$

where P is power, R is the electrical resistance of the heater, and U is the voltage imposed across the heater.

The other boundary condition of the sample was a temperature one. This boundary condition was selected because composite materials tend to have low thermal conductivities. By attaching these low conductivity materials to high conductivity materials (such as metals), the temperature boundary condition is easily achieved.

### 2.1.5 Outline of the Remainder of the Chapter

A brief outline of the remainder of this chapter is given. The next section of this chapter describes how the problem was implemented experimentally and gives some experimental data. The third part of this chapter briefly explains the theory and implementation of program PROP1D used to estimate the thermal properties. Finally, the results of PROP1D are shown and discussed.

### 2.2 Description of the Experiment

#### 2.2.1 Experimental Setup

The experimental setup described here was constructed in two major stages. The first stage consisted of making two epoxy specimens from a commercial epoxy and a

commercial curing agent. In the second stage, the specimens were instrumented. Thermocouples were installed in the specimens, one heater was sandwiched between the specimens, and a metallic block on the opposite surfaces of the specimens (see Figure 2.1). Finally, the instrumented specimens were placed in an oven, and connected to the data acquisition system.

#### 2.2.1a Fabrication of the Specimens

The first step of the experimental investigation was to build two epoxy specimens. For this purpose, the curing agent Diamino Diphenyl Sulfone (DDS) was added to DER332 resin in a beaker at a weight ratio of 35.8%. The beaker was then placed in an oven at 140°C, and the resin was mixed every 5 to 10 minutes until all of the DDS was diluted. A clean rubber mold was then placed in a vacuum oven along with the beaker containing the epoxy. The vacuum pump was turned on until all the bubbles in the epoxy were gone, and new bubbles started to form very slowly. The beaker and the mold were then taken out of the vacuum oven, and the epoxy was poured in the mold. The mold was then placed in a programmable oven allowing the control of the curing cycle.

The curing cycle was originally similar to the one usually used to cure dog-bone shaped DER332/DDS samples that are no more than 1 cm thick. Such samples are usually used in the Composite Materials and Structures Center at Michigan State University for structural analysis or optical tests. The curing cycle consists of imposing a constant temperature of 180°C for 3 hours. This same curing cycle caused our relatively thick (3 cm) circular samples to burn inside because of the excessive exothermic reactions taking place during the curing process.

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The samples were chosen that thick for the following reasons. The first reason was to allow the installation of the thermocouples at various distances from the heated surface. The second reason for making the samples relatively thick was to allow them to behave as semi-infinite regions for only one part of the experiment, then as finite bodies.

A new curing cycle was then suggested (by M. Rich of the Composite Materials and Structures Center at Michigan State University) to get around the damaging effect of the exothermic cure reactions. The new curing cycle was as follows: constant temperature of 140°C for 2 hours, ramp for 20 minutes to 160°C, constant temperature of 160°C for 2 hours, ramp for 20 minutes to 180°C, constant temperature of 180°C for 2 hours, and a ramp down to 40°C for one hour. The specimen was then taken out of the oven and allowed to cool in ambient temperature. Finally, a second identical specimen was made, following the same methodology used to make the first one. The thickness of this second sample was also made equal to that of the first sample.

The choice to make the two specimens identical has an experimental justification. By making the samples identical in composition and dimensions, then placing a flat heater between them, a symmetry condition is achieved. The power generated by the heater is equally shared by the two half-domains made of the identical specimens. For this particular reason, the measured power of Equation (2.7) is divided by twice the area of the heater to give the heat flux at each specimen's surface as

$$q = \frac{U^2 R}{2A}$$
(2.7)

This of course assumes no side heat losses from the heater to the surroundings, which is

a reasonable assumption for our setup.

To reduce the contact resistances between the heater and the specimens, as well as that between the metallic blocks and the samples, the specimens were polished. Different grades of sanding paper were used until a nice gloss was achieved. Silicone grease was then carefully spread between the heater and the samples, and between the metallic blocks and the samples. By minimizing the contact resistances between the specimens and the heater, the temperature drop across the surface of contact is minimized, and most of the heat flux leaving the heater reaches the surface of the specimens. Minimizing the contact resistance between the specimens and the metallic blocks also makes the temperatures at the surfaces of contact closer to the temperatures of the metallic blocks.

### 2.2.1b Instrumentation of the Specimens

The next step of the experimental investigation was to instrument the cured samples. The first part of the instrumentation of the samples was to install the thermocouples needed for temperature measurement. The temperature range to which these wires are sensitive coincides with the range of temperatures of our experiments. The plastic insulation was stripped off the Chromel-Constantan wires, which were then electrically insulated using a ceramic (alumina) sheath; this sheath made the installation of the thermocouples in the holes drilled in the samples possible and practical.

The next part of the instrumentation of the specimens was to place a heater between them. A circular Kapton heater (made by Minco of Minneapolis) was sandwiched between the two identical cured epoxy specimens. The heater was 7.5 cm in diameter, and about 0.025 cm thick. A high conductivity silicone grease (DOW Corning 340) was used between the heater and each side of the samples to maximize thermal contact. The non-heated side of each sample was attached to an aluminum block having a relatively high thermal conductivity compared to that of the samples. As mentioned above, the temperature of the aluminum block did not change significantly under the influence of the heat flux imposed by the heater, and thus simulated a constant temperature boundary condition. This was possible because the low thermal conductivity of epoxy permits only a small heat flow to the aluminum blocks. During the experiment, the heat flow to the aluminum blocks was so small that their temperature rise was small. Any temperature rise is treated in program PROP1D which is described in Section 2.3.

The themocouples were connected to the data acquisition system made of a VAX station II GPX computer. The heater terminals were connected to a Hewlett Packard 6024A DC power supply which was controlled by the same computer. This VAX computer is equipped with a data acquisition board that allows simultaneous temperature measurements. Indeed, it takes only 10<sup>-6</sup> second to scan two consecutive channels of the board, making the simultaneous measurements assumption a good one. At this stage, the experimental setup was ready to operate as explained in the next section.

#### 2.2.2 Some Experimental Results

Two series of experiments were initially considered to investigate the effect of the heating regime on the estimated thermal properties. This relates to the experimental paradigm mentioned in the first paragraph of this chapter. Each series consisted of running four independent experiments. In the first series, each experiment consisted of applying a large heat flux (4750 W/m<sup>2</sup> on the average) to the samples at uniform room temperature (25°C). In the second series, a much smaller heat flux (425 W/m<sup>2</sup>) was applied to the samples starting at four different uniform temperatures (23°C, 52°C, 78°C, and 101°C). In the first series of experiments, the heat flux was made large to cover the total temperature range of interest (20 to 100°C) in an experiment. In the second series, the same temperature range was covered but each experiment covered a small portion of it.

Some of the transient temperatures and heat flux data collected for the two series of experiments are shown in Figures 2.2 and 2.3. Figure 2.2 corresponds to large heat pulse experiments, and is discussed first. Figure 2.2a shows the transient measured experimental heat flux. The non-zero values of the heat flux oscillate between 4634.0  $W/m^2$  and 4853.1  $W/m^2$  making the fluctuation in heat flux between -2.44% and 2.17%.

Figure 2.2b shows eight curves corresponding to the transient experimental temperatures measured by two thermocouples (one at x=0, and the other at 3.9 mm), and corresponding to four experiments. The upper curves in Figure 2.2b are in fact four curves corresponding to the surface thermocouple. The two curves indicated by the continuous line are almost identical. The two other curves indicated by the dashed line are also almost identical. These upper four curves in Figure 2.2b would have merged into a single curve if the experiments were all started at exactly 25°C; however, as indicated by the temperatures measured between time zero and time 10 seconds, the initial uniform temperature was not exactly the same for all four cases.

Notice that the surface temperatures start rising almost instantaneously


(a) Heat Flux



# (b) Temperatures

Figure 2.2 Experimental Transient Heat Flux and Temperatures (at two thermocouples) on DER332/DDS Epoxy for Large Heat Pulse

with heat flux, then behave approximately as the square root of time. This behavior is characteristic of semi-infinite domains subjected to a heat pulse. Based on this observation, it can be concluded that the heated surface of the specimen does not "see" the constant temperature boundary during most of the experiment. After the power is turned off, at about 160 seconds, the heated surface temperature drops abruptly.

The lower curves of Figure 2.2b show a smoother drop than the upper curves after time 160 seconds. There are again four curves, and they would all have been nearly identical if the experiments were started at exactly 25°C. These curves correspond to the internal thermocouple. The delay in the response of this thermocouple is a result of the distance between the thermocouple and the heated surface. Note also that this set of curves and the curves described above are nearly parallel to each other for times between 60 and 160 seconds.

There are several purposes in running experiments such as shown in Figures 2.2a and 2.2b. One relates to replication. Another relates to the investigation of measurement errors. Yet another relates to the experimental paradigm. These are now briefly discussed.

Replication must be assured in these experiments (the epoxy material does not change in composition over the experimental temperature range shown in Figure 2.2b). The replication is remarkably good since it is within 0.1284°C over the entire range, if the initial temperatures are adjusted to be the same. The value 0.1284°C is the largest standard deviation of the experimental measurements calculated as shown later in this section (see Equation (2.9)). In calculating this standard deviation, the temperature data

obtained from the series of the four large heat flux experiments is considered as data obtained in one experiment from four identical thermocouples at exactly the same location. Notice that this type of replication is that termed of the first order by Moffat (Moffat, 1982, 1985). The same thermocouples and the same specimen are used. A more rigorous examination would be to run another experiment with different types of temperature sensors, and a new set of specimens. This was not practical; however, there are two specimens, each with its own thermocouples.

Running one experiment with those two specimens is similar to a replication of the second order. In this second order replication, the specimen is changed, and the same experiment is repeated using the same instruments. Unfortunately though, the thermocouples of the two specimens were not at exactly the same locations. This "pseudo" replication of the second order could not be achieved then.

An inspection of the results of Figure 2.2b and also the individual thermocouples leads to a partial understanding of the measurement errors (all thermocouple readings at the same time by the same thermocouple could have similar biased errors for the different experiments). The average temperature at a particular time,  $t_i$ , for m temperature values can be calculated using

$$\overline{\mathbf{Y}}_{i} = \frac{1}{m} \sum_{j=1}^{m} \mathbf{Y}_{ij}$$
(2.8)

where  $\overline{\mathbf{Y}}_{i}$  is the average at time  $t_{i}$ , and  $\mathbf{Y}_{ij}$  is the measured temperature at  $t_{i}$  corresponding to the j-th measurement. The j-th measurement could come from the same experiment (different specimens) or different experiments. The estimated standard deviation of the error in each  $Y_{ii}$  at a given  $t_i$  is

$$s_{Y_{i}} = \left[\frac{1}{m-1} \sum_{j=1}^{m} (Y_{ij} - \bar{Y}_{i})^{2}\right]^{1/2}$$
(2.9)

The values of  $s_y$  obtained for the large heat flux experiments range between 0.0427°C and 0.1284°C as shown in Tables 2.2 and 2.4.

The data of Tables 2.2 and 2.4 was obtained as follows. First the temperatures of Tables 2.1 and 2.3 corresponding to the different thermocouples were averaged at time zero. Next, the initial temperature offset from this average was calculated for each thermocouple. Finally, the temperatures at other times were corrected with the obtained offset, and Tables 2.2 and 2.4 were obtained. The averaging of the temperature at time zero explains the zero value for the standard deviation at that time. On the average, the standard deviations of Tables 2.2 are larger than those of Table 2.4. This was expected based on the knowledge that surface thermocouples cause more disturbance than internal thermocouples.

The final aspect is related to the experimental paradigm. This is also related to one of the significant analyses of this dissertation. The underlying question is: is it better to cover "small" temperature ranges in an experiment or "large" ones? Small and large are defined with respect to the temperature dependence of the thermal properties. If the properties change less than some small value (such as 2%) in the temperature range of the individual experiment, then the experiment has a small temperature range. On the other hand, if the thermal properties change more than 10% in a given experiment, the temperature range is said to be large. Figure 2.2 represents a large temperature range, and

t(sec)	Y <sub>i1</sub>	Y <sub>i2</sub>	Y <sub>i3</sub>	Y <sub>i4</sub>	Avg Y
0	23.54	22.58	22.33	23.88	23.08
40	71.33	70.40	70.08	71.54	70.84
80	96.08	95.18	94.90	96.28	95.61
120	113.71	112.79	112.56	113.84	113.23
160	122.92	122.05	121.78	123.08	122.46
200	91.26	90.43	90.12	91.50	90.83

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Table 2.1 Some High Heat Pulse Experimental Temperature Data for the Surface Thermocouple

Table 2.2 Some Corrected High Heat Pulse Experimental Temperature Data and the Corresponding Standard Deviation for the Surface Thermocouple

t(sec)	Y <sub>i1,corr</sub>	Y <sub>i2,corr</sub>	Y <sub>i3,corr</sub>	Y <sub>i4,corr</sub>	Avg Y <sub>corr</sub>	S <sub>Y</sub>
0	23.08	23.08	23.08	23.08	23.08	0
40	70.87	70.90	70.83	70.74	70.84	0.0695
80	95.62	95.68	95.65	95.48	95.61	0.0884
120	113.25	113.29	113.31	113.04	113.22	0.1242
160	122.46	122.55	122.53	122.28	122.46	0.1229
200	90.80	90.93	90.87	90.70	90.83	0.0988

t(sec)	Y <sub>i1</sub>	Y <sub>i2</sub>	Y <sub>i3</sub>	Y <sub>i4</sub>	Avg Y
0	23.54	22.58	22.33	23.88	23.08
40	28.67	27.78	27.56	29.05	28.27
80	42.84	42.01	41.75	43.13	42.43
120	56.20	55.34	55.16	56.44	55.79
160	68.22	67.34	67.13	68.39	67.77
200	72.34	71.50	71.29	72.58	71.93

Table 2.3 Some High Heat Pulse Experimental Temperature Data for the Internal Thermocouple

Table 2.4 Some Corrected High Heat Pulse Experimental Temperature Data and Corresponding Standard Deviation for the Internal Thermocouple

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t(sec)	Y <sub>i1,corr</sub>	Y <sub>i2,corr</sub>	Y <sub>i3,corr</sub>	Y <sub>i4,corr</sub>	Avg Y <sub>corr</sub>	S <sub>Y</sub>
0	23.08	23.08	23.08	23.08	23.08	0
40	28.21	28.28	28.31	28.25	28.26	0.0427
80	42.38	42.51	42.50	42.33	42.43	0.0891
120	55.74	55.84	55.91	55.64	55.78	0.1179
160	67.76	67.84	67.88	67.59	67.77	0.1284
200	71.88	72.00	72.04	71.78	71.93	0.1182

Figure 2.3 represents a small temperature range. The estimation techniques are sufficiently powerful so the temperature dependence can be found from the analysis of a single experiment. Even though the properties can be found as functions of temperature for a given experiment, it does not follow that this is the best procedure.

The competing experimental research paradigms of small and large temperature variations in experiments have been investigated. Such an investigation was one of the objectives of this research. Unfortunately, the best way to proceed depends greatly upon the characteristics of the measurement errors, which have not been completely understood. The investigation of the measurement errors is the third major analysis area in this dissertation.

Figure 2.3 is now analyzed. This figure is similar to Figure 2.2, and most of the ideas discussed above apply here. Figure 2.3 corresponds to a much smaller heat flux (425 W/m<sup>2</sup> on the average) imposed at the four different initial temperatures 23°C, 52°C, 78°C, and 101°C. The top part of Figure 2.3 shows that the non-zero values of the heat flux fluctuate between 396.5 W/m<sup>2</sup> and 453 W/m<sup>2</sup> making the fluctuations in heat flux between -6.7% and 6.6%. These fluctuations are larger than the ones corresponding to Figure 2.2. The possible causes of this behavior are noise in the power supply used to impose the voltage of Equation (2.7), and/or the behavior of the Minco heater which might be more accurate for higher voltages.

Figure 2.3b shows the temperatures measured by the same thermocouples used to obtain Figure 2.2b. The only difference here is that the specimens were initially at different uniform temperatures, and were then subjected to a smaller heat pulse.



(a) Heat Flux



(b) Temperatures

Figure 2.3 Experimental Transient Heat Flux and Temperatures (at two thermocouples) on DER332/DDS Epoxy for Small Heat Pulse

A check was performed for the replication of each one of the experiments of Figure 2.3. This was done by running each experiment twice and checking if the temperature curves were identical or not. The results of this check are not shown in Figure 2.3b, but were as good as those of Figure 2.2.

The experimental data of Figures 2.2 and 2.3 was next used to estimate the thermal properties of the epoxy samples. The computer program PROP1D (Beck, 1989) was used for this purpose. The logic and theory implemented in PROP1D are briefly described in the next section.

#### 2.3 Description of PROP1D

The parameter estimation program PROP1D was used to estimate simultaneously the thermal conductivity and volumetric heat capacity. PROP1D permits the estimation of any combination of a total of four parameters as functions of temperatures. The program uses the Gauss minimization method, and the parameters are found by minimizing the scalar weighted sum of squares S defined by

$$\mathbf{S} = [\mathbf{Y} - \mathbf{T}(\boldsymbol{\beta})]^{\mathrm{T}} \mathbf{W} [\mathbf{Y} - \mathbf{T}(\boldsymbol{\beta})]$$
(2.10)

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where Y is the column vector of size  $(mn\times 1)$ , and whose entries correspond to transient measured temperatures for each thermocouple used. The dimension m stands for the number of thermocouples, while the dimension n stands for the number of measurements obtained by each thermocouple. W is a symmetric  $(mn\times mn)$  weighting matrix; however, PROP1D has the capability of treating a diagonal W with the different weights only for different temperature sensors. The entries of the  $(mn\times 1)$  column vector T are the calculated temperatures obtained using the Crank-Nicolson finite difference numerical method;  $\beta$  is the (p×1) parameter vector (p being the number of estimated parameters).

The set of estimated thermal properties is obtained by minimizing S with respect to the parameters of interest. This is done by setting the matrix derivative of S with respect to  $\beta$  equal to zero:

$$\nabla_{\mathbf{\beta}} \mathbf{S} = 2[-\mathbf{X}(\boldsymbol{\beta})] \mathbf{W} [\mathbf{Y} - \mathbf{T}(\boldsymbol{\beta})] = 0$$
(2.11)

where  $X(\beta)$  is the (mn×p) sensitivity coefficient matrix. Equation (2.11) can be rearranged to solve a set of p equations for the vector estimator **b** of  $\beta$  using an iterative procedure needed because of the non-linearity of the problem (indicated by the temperature (and thus  $\beta$ ) dependence of the sensitivity coefficient matrix X):

$$b^{(i+1)} = b^{(i)} + (X^{T^{(0)}}WX^{(i)})^{-1} [X^{T^{(0)}}W(Y - T^{(i)})]$$
(2.12)

The sensitivity matrix X is computed by PROP1D, and is defined by:

$$\mathbf{X}(\boldsymbol{\beta}) = \begin{bmatrix} \nabla_{\boldsymbol{\beta}} \mathbf{T}^{\mathsf{T}}(\boldsymbol{\beta}) \end{bmatrix}^{\mathsf{T}} = \begin{bmatrix} \mathbf{X}(1) \\ \mathbf{X}(2) \\ \vdots \\ \vdots \\ \mathbf{X}(n) \end{bmatrix}$$
(2.13)

where

The entries  $X_{jk}(i)$  of this matrix are the partial derivatives of the dependent variable T

measured at time t<sub>i</sub> 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 referred to as sensitivity coefficients. These sensitivity coefficients are important when analyzing experiments; they provide much insight into the data as shown later in Chapter 3.

The entries of the diagonal weighting matrix W are either specified by the user in the input file of PROP1D, or automatically calculated by the program itself; however, the weighting is varied only for a given temperature sensor. These weighting coefficients are larger for thermocouples with smaller residuals than thermocouples with larger residuals. Smaller residuals imply smaller differences between the measured and the calculated temperatures; therefore, thermocouples with such residuals measure temperatures closer to the exact model, and consequently have smaller errors than other thermocouples with larger residuals.

Sequential computation of the entries of vector **b** obtained by PROP1D is now described. The data over the whole time interval of interest is used with initial guesses (of the parameters) included in the input file to estimate the thermal properties for the first iteration. These estimates are used as initial guesses in the next iteration which again uses the data of the whole time interval. Once the convergence criterion (difference between consecutive estimated parameters smaller than a specified constant) is met, the last estimates are used as initial guesses in the last iteration; unlike previous iterations, this last one uses the data one time step at a time to estimate and print the parameters as functions of time. The maximum number of iterations is specified in the input file used

with the program. Limiting the number of iterations avoids unnecessary computations that might be due to errors in the input file; however, the parameter estimates usually converge within five to six iterations. It should be noted that this type of sequential analysis is not the same as sequential over experiments, which is described in the last paragraph of Section 2.3 and in Section 2.4.

The equations used for each parameter as a function of temperature are

$$k = k_1 + \frac{k_2 - k_1}{T_2 - T_1} (T - T_1)$$
(2.15a)

$$\rho c = \rho c_1 + \frac{\rho c_2 - \rho c_1}{T_2 - T_1} (T - T_1)$$
(2.15b)

instead of

$$\mathbf{k} = \zeta_0 + \zeta_1 \mathbf{T} \tag{2.16a}$$

$$\rho c = \zeta_2 + \zeta_3 T \tag{2.16b}$$

Such a choice (Equations (2.15a) and (2.15b)) gives the parameters the same units as the property of interest, for which the user has a better feel; furthermore, such a choice allows the natural extension of the model to more complex ones with still some feel for the values of the parameters.

Another feature of PROP1D is that the program gives confidence intervals in addition to parameter values. PROP1D computes two types of confidence intervals of the estimated parameters: a 95% ellipsoid confidence region, and the Bonferroni square approximate confidence region (Seber and Wild, 1989). The 95% confidence region is an ellipsoid; the end points of the major and minor axes of the ellipse obtained by projection on the plane are also computed.

The 95% confidence intervals computed by PROP1D are based on the assumption that temperature is the only quantity having significant errors. This in itself assumes that the errors in all other measured quantities are negligible compared to the error in temperature. Consider for example the steady state case:

$$q = k \frac{\delta T}{L} \rightarrow k = \frac{qL}{\delta T}$$
(2.17)

where q is the constant heat flux, L is the location of the sensor of interest, and T is the temperature measured by that same sensor. Assume that q, L, and  $\delta T$  all have random errors in them, the error in k is approximately

$$\Delta \mathbf{k} = \frac{\partial}{\partial q} \left( \frac{qL}{\delta T} \right) \Delta q + \frac{\partial}{\partial L} \left( \frac{qL}{\delta T} \right) \Delta L + \frac{\partial}{\partial \delta T} \left( \frac{qL}{\delta T} \right) \Delta \delta T$$
  
$$= \frac{L}{\delta T} \Delta q + \frac{q}{\delta T} \Delta L - \frac{qL}{(\delta T)^2} \Delta \delta T$$
(2.18)

Assume independent errors so the covariances of the different quantities are zero. Then we have

$$\sigma_{k}^{2} = V(\Delta k) = \left(\frac{L}{\delta T}\right)^{2} \sigma_{q}^{2} + \left(\frac{q}{\delta T}\right)^{2} \sigma_{L}^{2} + \left(\frac{qL}{(\delta T)^{2}}\right)^{2} \sigma_{T}^{2}$$
(2.19)

where  $\sigma_k^2$  is the variance of k (also denoted V( $\Delta k$ )),  $\sigma_q^2$  is the variance of q,  $\sigma_L^2$  is the variance of the thermocouple location L, and  $\sigma_T^2$  is the variance of the measured temperature T. PROP1D handles only errors in the measured temperatures. The other sources of errors such as the measured heat flux, and the thermocouples locations are not accounted for in PROP1D; therefore, PROP1D assumes  $\sigma_L^2, \sigma_q^2 << \sigma_T^2$ .

Finally, another one of the many features of PROP1D is that the program permits

the use of sequential-over-experiments analysis. This type of analysis allows the user to combine information gained from one experiment (as prior information) to estimate the thermal properties using the data of the next experiment (Beck, Hollister, and Osman, 1990). The added information coming from more experiments improves the quality of the estimated parameters by reducing their confidence regions. This is exactly what is needed to analyze the series of experiments of Section 2.2.

#### 2.4 **PROP1D Experimental Results**

Some of the results of program PROP1D are shown in Figures 2.4, 2.5, and 2.6, and are discussed in this section.

## 2.4.1 Large Pulse Experiments

When the data of Figure 2.2 (for large heat pulse) was analyzed with PROP1D, the transient thermal properties were calculated as functions of temperature. They were determined at two temperatures. At 20°C, the thermal conductivity k was found to be  $0.1657 \pm 0.0139$  W/m°C, and  $\rho c$  to be  $(0.1179 \pm 0.0082) \times 10^7$  J/m<sup>3</sup>°C. At 120°C, k was found to be  $0.2395 \pm 0.0107$  W/m°C, and  $\rho c$  to be  $(0.2240 \pm 0.0205) \times 10^7$  J/m<sup>3</sup>°C.

The transient residuals were also computed by PROP1D, and are shown in Figure 2.4 for two thermocouples. The surface thermocouple (sensor 1) shows residuals as large as 3°C for a maximum temperature rise of 104°C. The corresponding error is then 2.9%, which is considered large for our purposes. Note that 3°C is much larger than the 0.1284°C found in connection with Table 2.4. The curves of the same Figure show that the errors are not zero on the average (they do not oscillate around zero), indicating a





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bias. Finally, the residuals of Figure 2.4 seem to be highly correlated. This correlation is observed by looking at consecutive portions of each curve, and noting that the data follows an increasing (or decreasing) trend. If the errors were uncorrelated, the behavior of a portion of the data could not be predicted from an adjacent portion.

The estimated confidence region of the thermal conductivity corresponding to the four large pulse experiments is shown by the dashed lines of Figure 2.5. Each one of the four experiments has information about the whole temperature range. In the process of using the sequential-over-experiments analysis, the data sets of the four experiments were combined together. This combination of the data sets of the four experiments was needed to compare the parameter estimates of the two sets of large and small pulse experiments; it was necessary to have the same amount of information (same number of measurements) for the large and small pulse experiments. The combination process minimally improved the confidence regions at the different temperatures (see Table 2.5).

#### 2.4.2 Small Pulse Experiments

The experimental data of Figure 2.3 (for small heat flux) was next analyzed using PROP1D. The transient thermal properties were calculated as functions of temperature. They were estimated at two temperatures. At 20°C, k was found to be 0.1977  $\pm$  0.0095 W/m°C, and  $\rho c$  to be (0.1327  $\pm$  0.0019)×10<sup>7</sup> J/m<sup>3</sup>°C. At 120°C, k was found to be 0.2316  $\pm$  0.0113 W/m°C, and  $\rho c$  to be (0.1861  $\pm$  0.0027)×10<sup>7</sup> J/m<sup>3</sup>°C.

The transient residuals were also computed by PROP1D and are shown in Figure 2.6. These residuals were as large as 0.3°C for a maximum temperature rise of 12.6°C. The corresponding error is then 2.4%, which is of the same order as that of Figure 2.4



Figure 2.5 Estimated Thermal Conductivity of DER332/DDS Epoxy versus Temperature for High and Low Heat Pulse Heating Regimes

data of exp.	k(20°C)	k(120°C)	ρC(20°C)	ρC(120°C)
1	0.1651±	0.2389±	(0.1173±0.0166)×	(0.2240±0.0406)×
	0.0278	0.0207	10 <sup>7</sup>	10 <sup>7</sup>
2	0.1661±	0.2392±	(0.1179±0.0115)×	(0.2239±0.0288)×
	0.0196	0.0149	10 <sup>7</sup>	10 <sup>7</sup>
3	0.1659±	0.2396±	(0.1180±0.0094)×	(0.2241±0.0236)×
	0.0159	0.0123	10 <sup>7</sup>	10 <sup>7</sup>
4	0.1657±	0.2395±	(0.1179±0.0082)×	(0.2240±0.0205)×
	0.0139	0.0107	10 <sup>7</sup>	10 <sup>7</sup>

Table 2.5 Calculated Confidence Intervals for the Large Pulse Experiments

Table 2.6 Calculated Confidence Intervals for the Small Pulse Experiments

data of exp. at	k(20°C)	k(120°C)	ρC(20°C)	ρC(120°C)
20°C	0.1575±	0.6599±	(0.1129±0.0338)×	(0.5210±0.5859)×
	0.0444	0.4632	10 <sup>7</sup>	10 <sup>7</sup>
20, 50°C	0.1970±	0.2318±	(0.1315±0.0023)×	(0.1925±0.0096)×
	0.0117	0.04348	10 <sup>7</sup>	10 <sup>7</sup>
20, 50,	0.1965±	0.2350±	(0.1321±0.0020)×	(0.1886±0.0047)×
75°C	0.0101	0.0188	10 <sup>7</sup>	10 <sup>7</sup>
20, 50,	0.1977±	0.2316±	(0.1327±0.0019)×	(0.1861±0.0027)×
75, 100°C	0.0095	0.0113	10 <sup>7</sup>	10 <sup>7</sup>

(for large heat pulse). Note that 0.3°C is of the same order of magnitude as the 0.1284°C found in connection with Table 2.4. Finally, the same correlated and biased behaviors of the errors observed for Figure 2.4 apply to Figure 2.6.

When a small heat pulse was used, the temperatures inside the specimens increased by no more than 10°C from the initial steady state temperature. PROP1D, however, estimated the thermal properties at temperatures between 20°C and 120°C. The confidence intervals of the thermal properties at the temperature closest to the initial one were the smallest (see Table 2.6). This is true because most of the experimental information was available at and around that particular initial temperature. The thermal properties estimated at the initial temperatures were then expected to be more accurate than the other properties at higher temperatures. The knowledge gained from one experiment was used as prior information (Beck, Hollister, and Osman, 1990) in analyzing the next experiment. In doing so for the first experiment of Figure 2.3, the confidence region obtained at 25°C was much smaller than that at 125°C. The knowledge gained at 25°C was then used (as prior information) in the analysis of the next experiment. Using this prior information with data of the experiment at 50°C helped estimate the parameters at both 25°C and 50°C. The same idea was used for 75°C, 100°C, and 125°C. The confidence region for the thermal conductivity was then obtained as shown by the continuous lines of Figure 2.5.

The possible sources of the residuals of Figures 2.4 and 2.5 are errors in temperature measurements, errors in the times at which the heat flux starts and ends, errors in the measurement of the positions of the thermocouples, non-uniform initial



Figure 2.6 Transient Residuals Calculated by PROP1D for DER332/DDS Epoxy for Small Heat Pulse (at surface thermocouple)

temperature, side heat losses, and the existence of contact resistance between the thermocouple and the surrounding material, and between the heater and the samples (even with the use of silicone grease). Most of the possible sources of errors were kept in mind when designing the experiment, and setting it up. Some of these sources could not be tightly controlled for a number of reasons. One of the reasons was that temperature measurement, for example, involves the thermocouple wires, the alumina sheath, the drilled holes, and the contact resistance between the junction and the surrounding material. The possibility of controlling all these experimental conditions, and others not identified, was not practically possible.

The above focused on the thermal conductivity as indicated by Figure 2.5, and ignored volumetric heat capacity. The reason for doing so is that volumetric heat capacity is not as sensitive to temperature as thermal conductivity. It should be noted though that program PROP1D estimates both thermal properties, and provides the same information for both of them.

Each one of the two confidence regions of Figure 2.5 is, in reality, made up of five confidence intervals at 25°C, 50°C, 75°C, 100°C, and 125°C computed by PROP1D. The two confidence regions of Figure 2.5 are similar in size. These confidence regions were considered large, i.e., the parameter estimates were not accurate enough. Using different heat pulse magnitudes then proved to be insufficient in improving the transient estimation of thermal properties, and another strategy was needed. This other strategy consists of designing optimal experiments, and is applied in Chapter 3. By the same token, since the residuals of Figures 2.4 and 2.5 seemed large, it was necessary to analyze

the errors due to thermocouples; this is done in Chapters 4 and 5. Chapter 4 represents an analytical study of the transient behavior of thermocouple errors, while Chapter 5 is the experimental investigation corresponding to this analytical study. If such analyses did not show the possibility of reducing these residuals, then another temperature measurement technique was needed. Chapter 6 investigates an alternative method of measuring temperatures developed by Garnier, Delaunay, and Beck (1991).

## **CHAPTER 3**

#### **OPTIMAL EXPERIMENTS**

#### 3.1 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 (Biggers, 1961, Box and Lucas, 1961, Draper and Hunter, 1966, Draper and Hunter, 1967, Kenward and Stone, 1969, Hill and Hunter, 1974, Atkinson, 1981, Atkinson, 1982, Seber and Wild, 1989), chemical engineering (Hunter et al., 1969), and mechanical engineering (Beck, 1966, Beck, 1969, Beck and Arnold, 1977, Balakovskii et al., 1988, Artyukhin et al., 1988, Artyukhin, 1989, Vigak et al., 1989).

This chapter 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 specifically designed to estimate the thermal properties of cured carbon-fiber/epoxy-matrix composite materials. The thermal properties of interest are conductivity and volumetric heat capacity for the onedimensional case, and directional thermal conductivities for the two-dimensional case.

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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 semiinfinite geometry, and two-dimensional heat conduction in finite cured composite materials. The optimal heating durations were considered for all three cases. The results of this analysis were applied in our laboratory on composite materials; however, this analysis can also be applied to metals.

A brief outline of this chapter is given. In the first section of the chapter, the theory pertaining to the design of optimal experiments for the estimation of thermal properties is reviewed. The temperature solutions and the sensitivity coefficients are derived and plotted for the three cases mentioned above. The optimality criterion is calculated and plotted for different experimental parameters. The results are discussed and compared with those of previous studies. Finally, some conclusions are stated.

### **3.2 Theoretical Procedure**

The choice of an optimal design must be based on some criteria. Three optimality criteria are given by Beck and Arnold (1977); all of these criteria are related to the sensitivity coefficient matrix, 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,  $\rho c$ . The two dimensionless sensitivity coefficients associated with these parameters,  $X_1^+$  and  $X_2^+$ , are defined as:

$$X_{i,1}^{+} = \frac{k}{q_0 L/k} \frac{\partial T_i}{\partial k}, \qquad i=1,2,...,n$$
(3.1)

$$X_{i,2}^{+} = \rho \frac{c}{qL/k} \frac{\partial T_{i}}{\partial \rho c} \qquad i=1,2,...,n \qquad (3.2)$$

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

The optimality criterion chosen in this study is discussed by Beck and Arnold (1977); it 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 (Beck and Arnold, 1977). 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 of maximizing D 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:

$$\mathbf{D} = \det(\mathbf{X}^{\mathsf{T}}\mathbf{X}) = \begin{vmatrix} \mathbf{C}_{11}^{+} & \mathbf{C}_{12}^{+} \\ \mathbf{C}_{12}^{+} & \mathbf{C}_{22}^{+} \end{vmatrix} = \mathbf{C}_{11}^{+}\mathbf{C}_{22}^{+-}(\mathbf{C}_{12}^{+})^{2}$$
(3.3)

where  $C_{11}^{+}$ ,  $C_{22}^{+}$ , and  $C_{12}^{+}$  are defined for uniformly spaced measurements in time between 0 and  $t_n^{+}$ , and for large number of time steps as (Beck and Arnold, 1977)

$$\mathbf{C}_{ij}^{+} = \left(\frac{1}{\mathbf{T}_{\max}^{+2}}\right) \left(\frac{1}{\mathbf{mt}_{n}^{+}}\right) \sum_{r=1}^{m} \int_{0}^{t_{n}} \left(\beta_{i} \frac{\partial \mathbf{T}_{r}^{+}}{\partial \beta_{i}}\right) \left(\beta_{j} \frac{\partial \mathbf{T}_{r}^{+}}{\partial \beta_{j}}\right) dt^{+}$$
(3.4)

where  $\beta_1 = k$ , and  $\beta_2 = c$ .

Numerical values for the temperature distribution in the geometry of interest are needed; for the investigated cases, these solutions are derived by the method of separation of variables or obtained from (Carslaw and Jaeger, 1959); finite element (FE) or finite difference (FD) 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. Finally, the determinant, D, is calculated from Equation (3.3). 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.

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 with the boundary heat flux imposed on half of the surface causing a heat flow both parallel and normal to the fiber axis.

#### 3.2.1 Finite One-Dimensional Composite (X21B50T0) (Case 1)

Carbon-fiber/epoxy-matrix composite materials tend to have low thermal conductivities for which an isothermal condition can be readily approximated at the unheated surface. The experiment shown in Figure 3.1 and analyzed here allows for the measurement of this relatively low thermal conductivity. The specimen is a slab of finite





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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., 1992). The advantages of this experiment include the ability to obtain the heat flux, experimentally, the simplicity of the experimental procedures, and the relative ease of the composite sample preparation due to its simple geometry; for example, the heat flux is controlled by simply turning it on and off. The heat conduction equation, boundary conditions, and initial conditions in non-dimensional form are:

$$\frac{\partial^2 T^+}{\partial x^{+2}} = \frac{\partial T^+}{\partial t^+}, \ 0 < x^+ < 1, \ t^+ > 0$$
(3.5a)

$$\frac{\partial T^{+}}{\partial x^{+}} = \begin{cases} -1 & 0 < t^{+} \le t_{h}^{+} \\ 0 & t_{h}^{+} < t_{h}^{-} \end{cases}, \text{ at } x^{+} = 0$$
(3.5b)

$$T^{+} = 0$$
 at  $x^{+} = 1$ ,  $t^{+} > 0$  (3.5c)

$$T^{+} = 0$$
, for  $0 \le x^{+} \le 1$ ,  $t^{+} = 0$  (3.5d)

The dimensionless time  $t_h^+$  is the dimensionless heating duration. The dimensionless variables are defined as:

$$T^{+} = \frac{T - T_0}{q_0 L/k}, \quad t^{+} = \alpha t/L^2, \quad t_h^{+} = \alpha t_h/L^2, \quad x^{+} = x/L$$
 (3.6a,b,c,d)

Equation (3.5b) gives the heat flux condition, and Equation (3.5c) gives the isothermal condition.

One method for the solution to this problem involves the use of the method of superposition. Up to time  $t_{b}^{+}$ , the temperature solution of Equation (3.5) is obtained for

a heat flux condition starting at time zero using the method of separation of variables (Carslaw and Jaeger, 1959, Ozisik, 1980):

$$T^{*}(x^{*},t^{*}) = (1-x^{*}) - 2 \sum_{n=0}^{\infty} \frac{(-1)^{n}}{\lambda_{n}^{2}} \sin(\lambda_{n}(1-x^{*})) e^{-\lambda_{n}^{2}t^{*}}, \quad 0 < t^{*} \le t_{h}^{*}$$
(3.7a)

where  $\lambda_n$  is equal to  $(2n+1)\pi/2$ . 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 (3.7a). The resulting solution is

$$T^{*}(x^{*},t^{*}) = -2\sum_{n=0}^{\infty} \frac{(-1)^{n}}{\lambda_{n}^{2}} \sin \left(\lambda_{n}(1-x^{*})\right) \left[e^{-\lambda_{n}^{2}t^{*}} - e^{-\lambda_{n}^{2}(t^{*}-t_{h}^{*})}\right], \quad t_{h}^{*} < t^{*} \le t_{n}^{*} \quad (3.7b)$$

where  $t_n^+$  is the final time for taking measurements. This solution (Equations (3.7a,b)) is shown in Figure 3.2 as a function of time for four different  $x^+$  values.

The next step is to compute the dimensionless sensitivity coefficients defined by Equation (3.1) and Equation (3.2). The differentials in these equations are found by differentiating the temperature solutions shown in Equations (3.7a,b) with respect to the parameters, k and  $\rho c$ . The i subscript in Equations (3.1) and (3.2) is dropped in the remainder of the chapter for simplicity of notation. The resulting sensitivity coefficients for thermal conductivity are

$$X_{1}^{\star} = \frac{k}{q_{0}L/k}\frac{\partial T}{\partial k} = -(1-x^{\star}) + 2\sum_{n=0}^{\infty}\frac{(-1)^{n}}{\lambda_{n}^{2}}\sin(\lambda_{n}(1-x^{\star})) e^{-\lambda_{n}^{2}t^{\star}}(1+\lambda_{n}^{2}t^{\star})$$
(3.8a)

for  $0 < t^+ \leq t_h^+$ , and



Figure 3.2 Dimensionless Temperature Solution for the Finite X21B50T0 Case

$$X_{1}^{*} = 2\sum_{n=0}^{\infty} \frac{(-1)^{n}}{\lambda_{n}^{2'}} \sin(\lambda_{n}(1-x^{*})) \left[ e^{-\lambda_{n}^{2}t^{*}} (1+\lambda_{n}^{2}t^{*}) - e^{-\lambda_{n}^{2}(t^{*}-t_{n}^{*})} (1+\lambda_{n}^{2}(t^{*}-t_{n}^{*})) \right]$$
(3.8b)

for  $t_h^+ < t^+ \le t_n^+$ . Likewise, the sensitivity coefficients for the volumetric heat capacity are:

$$X_{2}^{*} = \frac{\rho c}{q_{0} L/k} \frac{\partial T}{\partial \rho c} = -2 \sum_{n=0}^{\infty} (-1)^{n} \sin(\lambda_{n} (1-x^{*})) t^{*} e^{-\lambda_{n}^{2} t^{*}}, \qquad 0 < t^{*} \le t_{h}^{*}$$
(3.9a)

$$X_{2}^{+} = 2\sum_{n=0}^{\infty} (-1)^{n} \sin(\lambda_{n}(1-x^{+})) \left[ -t^{+}e^{-\lambda_{n}^{2}t^{+}} + (t^{+}-t_{h}^{+})e^{-\lambda_{n}^{2}(t^{+}-t_{h}^{+})} \right], \quad t_{h}^{+} < t^{+} \le t_{n}^{+} \quad (3.9b)$$

The sensitivity coefficients for thermal conductivity and volumetric heat capacity are shown as functions of time for  $t_h^+ = t_h^+$  in Figures 3.3 and 3.4. The magnitude of the thermal conductivity sensitivity coefficient is about equal to that of T<sup>+</sup>, while the sensitivity coefficients for the volumetric heat capacity are smaller, and they approach zero for t<sup>+</sup> greater than 2. Also note that the shapes (except at very early times) of the thermal conductivity and volumetric heat capacity sensitivity coefficient curves are quite different. Finally, note that the sum of the dimensionless sensitivity coefficients X<sub>1</sub><sup>+</sup> and X<sub>2</sub><sup>+</sup> is equal to the negative of T<sup>+</sup>. These observations verify that the two sensitivity coefficients, X<sub>1</sub><sup>+</sup>, and X<sub>2</sub><sup>+</sup>, are "large" (i.e. on the order of T<sup>+</sup>), and uncorrelated (different shapes), which are desirable conditions for parameter estimation.

The final step of the analysis requires the determination of the determinant, D, shown in Equation (3.3). The maximum temperature rise,  $T_{max}^{+}$ , is first determined from Equations (3.7a,b) with x<sup>+</sup>=0, and the sensitivity coefficients, X<sub>1</sub><sup>+</sup> and X<sub>2</sub><sup>+</sup>, are found from Equations (3.8a,b) and Equations (3.9a,b). The C<sub>ij</sub> matrix coefficients are then found from



Figure 3.3 Dimensionless Thermal Conductivity Sensitivity Coefficients for the Finite X21B50T0 Case



Figure 3.4 Dimensionless Volumetric Heat Capacity Sensitivity Coefficients for the Finite X21B50T0 Case

Equation (3.4), using the calculated values of  $T_{max}^+$ ,  $X_1^+$ , and  $X_2^+$  and integration. Finally, the determinant, D, is calculated from Equation (3.3). The determinant was found and compared using different heating times  $(t_h^+)$  and different experiment durations  $(t_n^+)$  (Figure 3.5), and different sensor locations (Figure 3.6).

#### 3.2.2 Semi-infinite One-Dimensional Composite (X20B5T0) (Case 2)

The second case considered is similar to the first case, with the exception that the sample is thick; thus, it behaves as a semi-infinite body with a constant heat flux at its surface (the number in this case is X20B5T0; see Beck et al., 1992). In this case, the heat conduction equation, boundary conditions, and initial conditions are

$$k\frac{\partial^2 T}{\partial x^2} = \rho c \frac{\partial T}{\partial t} , \ 0 < x < \infty , \ t > 0$$
(3.10a)

$$-k\frac{\partial T}{\partial x} = \begin{cases} q_0 & 0 < t \le t_h \\ 0 & t > t_h \end{cases} \quad \text{at } x = 0$$
 (3.10b)

$$T = T_0$$
, at  $x \ge 0$ ,  $t = 0$  (3.10c)

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

$$T^{+} = \frac{T - T_0}{q_0 x_0 / k}, \quad t^{+} = \frac{\alpha t}{x_0^2}, \quad x^{+} = \frac{x}{x_0}$$
 (3.11a,b,c)

where  $x_0$  can be any given location inside the body (not on the surface). If the temperature at the surface is of interest, then T is non-dimensionalized with respect to the position of the internal thermocouple.

The temperature solution for this problem was obtained from Carslaw and Jaeger (1959). The dimensionless temperature is given by


Figure 3.5 Determinant, D, for Different Heating Times for the Finite X21B50T0 Case



Figure 3.6 Determinant, D, at Different Sensor Locations for the Finite X21B50T0 Case

$$T^{+}(t^{+}) = 2\sqrt{t^{+}} \quad \text{ierfc}\left[\frac{x^{+}}{2\sqrt{t^{+}}}\right], \quad 0 < t^{+} \le t_{h}^{+}$$
(3.12a)

$$T^{+}(t^{+}) = 2\sqrt{t^{+}} \operatorname{ierfc}\left[\frac{x^{+}}{2\sqrt{t^{+}}}\right] - 2\sqrt{t^{+}-t_{h}^{+}} \operatorname{ierfc}\left[\frac{x^{+}}{2\sqrt{t^{+}-t_{h}^{+}}}\right], \quad t_{h}^{+} < t^{+} < t_{h}^{+}$$
(3.12b)

Upon differentiation of T (not T<sup>+</sup>) with respect to the parameter k, the thermal conductivity sensitivity coefficient  $X_1^+$  can be written as

$$X_{1}^{+} = \frac{k}{q_{0}x_{0}/k} \frac{\partial T}{\partial k} = -\sqrt{\frac{t^{+}}{\pi}} \exp\left(-\frac{x^{+^{2}}}{4t^{+}}\right) + \operatorname{erfc}\left[\frac{x^{+}}{2\sqrt{t^{+}}}\right], \quad 0 < t^{+} \le t_{h}^{+} \quad (3.13a)$$

$$X_{1}^{*} = -\sqrt{\frac{t^{*}}{\pi}} \exp\left(-\frac{x^{*^{2}}}{4t^{*}}\right) + \operatorname{erfc}\left(\frac{x^{*}}{2\sqrt{t^{*}}}\right) - \left[\sqrt{\frac{t^{*}-t_{h}^{*}}{\pi}} \exp\left(-\frac{x^{*^{2}}}{4(t^{*}-t_{h}^{*})}\right) + \operatorname{erfc}\left(\frac{x^{*}}{2\sqrt{t^{*}-t_{h}^{*}}}\right)\right], \quad t_{h}^{*} < t^{*} < t_{h}^{*}$$
(3.13b)

In a similar way, the volumetric heat capacity sensitivity coefficient  $X_2^+$  is

$$X_{2}^{*} = \frac{\rho c}{q_{0} x_{0} / k} \frac{\partial T}{\partial \rho c} = -\sqrt{\frac{t^{*}}{\pi}} \exp\left(-\frac{x^{*^{2}}}{4t^{*}}\right), \qquad 0 < t^{*} \le t_{h}^{*}$$
(3.14a)

$$X_{2}^{+} = -\sqrt{\frac{t^{+}}{\pi}} \exp\left(-\frac{x^{+2}}{4t^{+}}\right) + \sqrt{\frac{t^{+}-t_{h}^{+}}{\pi}} \exp\left(-\frac{x^{+2}}{4(t^{+}-t_{h}^{+})}\right), \qquad t^{+}>t_{h}^{+} \qquad (3.14b)$$

The determinant, D, is again calculated from Equation (3.3) as described for the first case involving a finite geometry. The solutions corresponding to different heating times  $t_h^+$  are found and shown in Figure 3.7.



Figure 3.7 Determinant, D, for Different Heating Times for the Semi-Infinite X20B5T0 Case

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# 3.2.3 Finite Two-Dimensional Composite (X22B00Y12B0x5T0) (Case 3)

The third case considered is that of two-dimensional heat conduction through a carbon- fiber/epoxy-matrix composite material. The experimental set-up and boundary conditions used for the first case are also considered here, with the exception that the boundary heat flux is imposed on half of the surface instead of all of it as shown in Figure 3.8. This type of boundary condition causes the heat to flow both parallel and normal to the axis of the fibers. The specimen then behaves as an orthotropic material with directional thermal conductivities  $k_x$  and  $k_y$ ; the subscripts x and y refer to the directions parallel and normal to the fibers respectively. The volumetric heat capacity is assumed known in this study; it could have been obtained from the above-described finite one-dimensional cured composite study, or from Differential Scanning Calorimetry (DSC) (to get the specific heat), and the standard test method for density of glass by buoyancy (the American Society for Testing and Materials (ASTM, 1990)) (to get density). The energy equation, boundary conditions, and initial conditions for this case are then

$$\frac{\partial^2 T^+}{\partial x^{*^2}} + \frac{\partial^2 T^+}{\partial y^{*^2}} = \frac{\partial T^+}{\partial t^+}$$
(3.15a)

$$T^{+}=0$$
 at  $y^{+}=0$  (3.15b)

$$\frac{\partial T^+}{\partial x^+} = 0$$
 at  $x^+ = 0$ ,  $x^+ = \frac{a}{b} \sqrt{\frac{k_y}{k_x}}$  (3.15c)

$$\frac{\partial T^{+}}{\partial y^{+}} = 1 \qquad \text{at } y^{+} = 1, \ 0 \le x^{+} \le \frac{a_{1}}{b} \sqrt{\frac{k_{y}}{k_{x}}}$$
(3.15d)





$$\frac{\partial T^{+}}{\partial y^{+}} = 0 \quad \text{at } y^{+} = 1, \ \frac{a_{1}}{b} \sqrt{\frac{k_{y}}{k_{x}}} \le x^{+} \le \frac{a}{b} \sqrt{\frac{k_{y}}{k_{x}}}$$
(3.15e)

$$T^{+}=0$$
 at  $t^{+}=0$  (3.15f)

where

$$T^{+} = \frac{T - T_{0}}{q_{0}b/k_{y}} , \quad t^{+} = \left(\frac{k_{y}}{\rho c}\right)\frac{t}{b^{2}} , \quad x^{+} = \frac{x}{b}\sqrt{\frac{k_{y}}{k_{x}}} , \quad y^{+} = \frac{y}{b}$$
(3.16a,b,c,d)

The temperature solution to this problem is made up of two components: a steady state one, and a transient one. The steady state part of the temperature solution is given for  $x^*=x/a$  and  $y^*=y/b$  by

$$T_{s.s.}^{*}(\mathbf{x}^{*}, \mathbf{y}^{*}) = \mathbf{a}_{1}^{*}\mathbf{y}^{*} + \frac{2}{\pi^{2}} \frac{\mathbf{a}}{\mathbf{b}} \sqrt{\frac{\mathbf{k}_{y}}{\mathbf{k}_{x}}} \sum_{n=1}^{*} \frac{1}{n^{2}} \sin(n\pi \mathbf{a}_{1}^{*}) \cos(n\pi \mathbf{x}^{*}) \tanh\left(n\pi \frac{\mathbf{b}}{\mathbf{a}} \sqrt{\frac{\mathbf{k}_{x}}{\mathbf{k}_{y}}} \mathbf{y}^{*}\right)$$
(3.17)

The transient part of the temperature solution is given by

$$T_{t}^{+}(x^{+},y^{+},t^{+}) = 2a_{1}^{*}\sum_{n=1}^{\infty} \frac{(-1)^{n}}{\lambda_{n}^{2}} \sin(\lambda_{n}y^{*}) e^{-\lambda_{n}^{2}t^{+}} + \frac{4}{\pi} *$$

$$\sum_{m=1}^{\infty}\sum_{n=1}^{\infty} \frac{(-1)^{n}}{m\left(\lambda_{n}^{2}\frac{b^{2}}{a^{2}}\frac{k_{y}}{k_{x}} + \lambda_{n}^{2}\right)} \cos(\lambda_{m}x^{*}) \sin(\lambda_{m}a_{1}^{*}) \sin(\lambda_{n}y^{*}) e^{-\left(\lambda_{m}^{2}\frac{b^{2}}{a^{2}}\frac{k_{y}}{k_{x}} + \lambda_{n}^{2}\right)t^{+}}$$
(3.18)

where  $\lambda_n = (2n-1)\pi/2$ ,  $\lambda_m = m\pi$ . The transient temperature solution is shown in Figure 3.9 for different values of x at the heated surface (y=b). Examination of Figure 3.9 reveals



Figure 3.9 Dimensionless Temperature Solution for the Two-Dimensional X22B00Y12B0x5T0 Case

that the sum of the non-dimensional temperatures at steady state at x=0, x=a, and y=b is unity; the same result is obtained when temperatures at locations on opposite sides of the midpoint (x=a/2) are added. This is a check on the validity of the solution; the problem can be looked at as the sum of two "half" problems, one insulated and the other heated.

The steady state and transient components of the sensitivity terms in Equation (3.2) were found by differentiating the dimensional forms of Equations (3.17) and (3.18) with respect to the parameters thermal conductivity,  $k_x$ , and thermal conductivity  $k_y$ , then premultiplying by  $k_x/(q_0b/k_y)$  and  $k_y/(q_0b/k_y)$  respectively. The resulting expressions for the steady state components are

$$X_{1}^{+} = \frac{k_{x}}{q_{0}b/k_{y}} \frac{\partial T}{\partial k_{x}} = \frac{-1}{\pi^{2}} \frac{a}{b} \sqrt{\frac{k_{y}}{k_{x}}} \sum_{n=1}^{\infty} \frac{1}{n^{2}} \sin(n\pi a_{1}^{*}) \cos(n\pi x^{*}) \tanh\left(n\pi \frac{b}{a} \sqrt{\frac{k_{x}}{k_{y}}}y^{*}\right) + \frac{1}{\pi^{2}} \sum_{n=1}^{\infty} \frac{1}{n} \sin(n\pi a_{1}^{*}) \cos(n\pi x^{*}) \operatorname{sech}^{2}\left(n\pi \frac{b}{a} \sqrt{\frac{k_{x}}{k_{y}}}y^{*}\right)$$
(3.19a)

$$X_{2}^{*} = \frac{k_{y}}{q_{0}b/k_{y}} \frac{\partial T}{\partial k_{y}} = \frac{-1}{\pi^{2}} \frac{a}{b} \sqrt{\frac{k_{y}}{k_{x}}} \sum_{n=1}^{\infty} \frac{1}{n^{2}} \sin(n\pi a_{1}^{*}) \cos(n\pi x^{*}) \tanh\left(n\pi \frac{b}{a} \sqrt{\frac{k_{x}}{k_{y}}}y^{*}\right) - \frac{1}{\pi} \sum_{n=1}^{\infty} \frac{1}{n} \sin(n\pi a_{1}^{*}) \cos(n\pi x^{*}) \operatorname{sech}^{2} \left(n\pi \frac{b}{a} \sqrt{\frac{k_{x}}{k_{y}}}y^{*}\right) - a_{1}^{*}y^{*}$$
(3.19b)

The transient components of the sensitivity coefficients are

$$X_{1}^{*} = 4\pi \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{m(-1)^{n+1}}{\left(\lambda_{m}^{2} \frac{b}{a} \sqrt{\frac{k_{x}}{k_{y}}} + \lambda_{n}^{2} \frac{a}{b} \sqrt{\frac{k_{y}}{k_{x}}}\right)^{2}} \cos(\lambda_{m} x^{*}) \sin(\lambda_{m} a_{1}^{*}) \sin(\lambda_{n} y^{*}) e^{-\left(\lambda_{m}^{2} \frac{b^{2}}{a^{2}} \frac{k_{x}}{k_{y}} + \lambda_{n}^{2}\right) t^{*}}$$
(3.20a)

$$X_{2}^{*} = 2\mathbf{a}_{1}^{*}\sum_{n=1}^{\infty} (-1)^{n+1} \sin(\lambda_{n}y^{*}) e^{-\lambda_{n}^{2}t^{*}} \left(\frac{1}{\lambda_{n}^{2}} + t^{*}\right) + \frac{4}{\pi} *$$

$$\sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{(-1)^{n+1}}{m\left(\lambda_{m}^{2}\frac{b^{2}}{a^{2}}\frac{k_{x}}{k_{y}} + \lambda_{n}^{2}\right)} \cos(\lambda_{m}x^{*}) \sin(\lambda_{m}a_{1}^{*}) \sin(\lambda_{n}y^{*}) e^{-\left(\lambda_{m}^{2}\frac{b^{2}}{a^{2}}\frac{k_{x}}{k_{y}} + \lambda_{n}^{2}\right)t^{*}} *$$

$$\left(1 - \frac{\lambda_{m}^{2}}{\lambda_{m}^{2}} + \lambda_{n}^{2}\frac{a^{2}}{b^{2}}\frac{k_{y}}{k_{x}} + \lambda_{n}^{2}t^{*}\right)$$

$$(3.20b)$$

These sensitivity coefficients are plotted in Figure 3.10 and Figure 3.11. The zero value of the  $k_x$  sensitivity coefficient at the midpoint of the flux boundary implies that no information is gained by placing the sensors there. Placing the sensors at x=a/2 on the heated surface will then decrease the value of the determinant D.

The determinant, D, is then calculated from Equation (3.3). The matrix coefficients defined by Equation (3.4) are determined using the maximum temperature rise,  $T_{max}^{+}$ , found from Equations (3.17) and (3.18) with  $x^{+} = 0$ , and the sensitivity coefficients,  $X_{1}^{+}$  and  $X_{2}^{+}$ , are found from Equations (3.19a,b and 3.20a,b). In this case, the solutions resulting from different heating times,  $t_{h}^{+}$ , as well as different thermocouple combinations were found and are shown in Figures 3.12 and 3.13 respectively. Finally, six global design curves (Figure 3.14) were generated for different values of the ratio of the heated surface to the whole surface; these curves involve the geometry and the thermal properties of the sample to be tested.

#### 3.4 Results and Discussion

The determinant, D, was compared for different experimental conditions, such as



Figure 3.10 X-axis Directional Thermal Conductivity Sensitivity Coefficient for the X22B00Y12B0x5T0 Case



Figure 3.11 Y-axis Directional Thermal Conductivity Sensitivity Coefficient for the X22B00Y12B0x5T0 Case



Figure 3.12 Determinant, D, for Different Heating Times for the Finite X22B00Y12B0x5T0 Case



Figure 3.13 Determinant, D, for Different Heated Surface Thermocouple Combinations for the X22B00Y12B0x5T0 Case



Figure 3.14 Global Design Curves for the X22B00Y12B0x5T0 Case

different heating times  $(t_h^+)$ , total measurement time  $(t_h^+)$ , and sensor locations, to determine the optimal experimental conditions. In the first case, a finite one-dimensional composite was considered, and the experimental variables included heating time, total experimental time, and sensor location. In the case of the thick composite, the experiment was optimized with respect to heating time and total experimental time. In the third case, a finite two-dimensional composite was considered; the experimental parameters of interest for that case were the heating time as well as the sensor locations. Finally, some optimal solutions were obtained for four simple experiments.

#### 3.4.1 Finite One-Dimensional Composite (X21B50T0) (Case 1)

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 3.3 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 3.4 shows that the volumetric heat capacity sensitivity coefficient,  $X_2^+$ , becomes essentially zero shortly after the dimensionless time, t<sup>+</sup>, equals 2. This indicates that little additional information is obtained using values of t<sup>+</sup> greater than two for the estimation of the volumetric heat capacity. The sensitivity coefficients, as shown in Figures 3.3 and 3.4 are not linearly dependent on each other; consequently, k and  $\rho c$ can be simultaneously and independently estimated. 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 Figure 3.5 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 results in a slightly higher maximum value of D; in this case, the maximum value is approximately equal to 0.020 which occurs at 3.

An interesting aspect of Figure 3.5 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 time does not have to be precise. Notice that the choice of the duration of the experiment after heating,  $t_n^+ - t_n^+$ , 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 pc (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 dimensionless 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 (if the same number of measurements is spread over a large 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 seconds rather than 100, both with time steps of one second. 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 that the finite duration of heating experiment gives poorer results than if heating occurs over the total experiment (see for example Figure 3.5 for  $t_h^+=2$  and  $t^+$  greater than 5).

The next factor considered was the sensor location. Figure 3.6 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 Figure 3.3, 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. Placing a sensor at the heated surface can have its disadvantages in practice; it may be difficult to place a sensor at the heated surface, and doing so can magnify errors in the sensor's reading caused by contact resistance and temperature disturbances resulting from the sensor's size.

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#### 3.4.2 Semi-Infinite One-Dimensional Composite (X20B5T0) (Case 2)

For the semi-inifinite body with a constant surface heat flux, 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 Figure 3.7. This figure shows that the optimal dimensionless heating time is approximately 1.5 which corresponds to a value of D of 0.0055. 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 0.22, making the optimal dimensionless total time equal to 1.7. The abrupt increase after heating stops, and rapid decrease after the maximum illustrate the importance of plots such as Figure 3.7. 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.

### 3.4.3 Comparison of One-Dimensional Results

At this point, a comparison of the current study with other published results is needed. Two geometries are described by Beck and Arnold (1977), a finite and a semiinfinite geometry. These two geometries are subjected to boundary conditions different from the ones used in the current study. Table 3.1 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 Beck and Arnold (1977), while cases II, VI,

	Geom	Boundary Condition(s)	Sensor Location(s)	Max. D	Opt t <sub>b</sub> ⁺	Opt t <sub>n</sub> +	
Ι	semi- inf.	(X20B1T0)	heated surface and in-depth	0.00263	t <sub>n</sub> +=1.5	_t_*=1.5	a
п	semi- inf.	(X20B5T0)	heated surface and in-depth	0.0055	1.5	1.72	b
ш	finite	(X22B10T0)	heated surface	0.00098	t <sub>n</sub> +=1.2	t <sub>n</sub> +=1.2	a
IV	finite	(X22B10T0)	x = 0 and 1	0.0058	t <sub>n</sub> <sup>+</sup> =0.65	t_+=0.65	a
v	finite	(X22B50T0)	x = 0 and 1	0.0088	0.4	0.6	a
VI	finite	(X12B05T0)	heated surface	0.020	2.25	2.98	b
VII	finite	(X12B01T0)	heated surface	0.012	t <sub>n</sub> +=7	t <sub>n</sub> +=7	b

· ...

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Table 3.1 Comparison of the Maximum Determinant, D, Values for 7 Cases.

a. Beck and Arnold (1977) b. This study

and VII are from the present study. For the finite geometry, cases VI and VII show a larger value of D (0.02 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 0.22 after the end of the heat flux (with D=0.0055) makes a better experiment than a continuous heat flux that lasts during the whole experiment (where D is 0.00263).

#### 3.4.4 Finite Two-Dimensional Composite

The experimental variables considered for the two-dimensional composite were the duration  $t_h^+$  of the heat pulse, as well as the locations of the thermocouples. The determinant, D, was calculated for five different heating times using five different thermocouples spread evenly along the surface that is half-heated and half-insulated. This determinant was then plotted as shown in Figure 3.12. The time dependence of this determinant is very different from that of the one-dimensional cases. For the two-dimensional case, the time to achieve the optimal experiment corresponds to steady state, and the optimal duration of the experiment is the same as the heating time period.

The next factor of interest was the locations of two thermocouples. Figure 3.13 shows that two thermocouples are best placed at the two ends of the flux boundary surface; i.e. x=0 and x=a. This Figure also shows that the determinant D is larger when the thermocouples are placed at the insulated half rather than at the heated half. Finally, some global design curves were developed for two-dimensional experiments with setups

similar to the one described above. The curves were obtained for different values of the ratio of the heated surface to the whole surface. These curves involve the geometry and the thermal properties of the sample to be tested; the curves are shown in Figure 3.14, and represent the determinant, D, as a function of the non-dimensional quantity (a/b)sqrt $(k_y/k_x)$ . The quantity (a/b)sqrt $(k_y/k_x)$  combines the composite's geometry parameters, a and b, as well as the directional properties of interest,  $k_x$  and  $k_y$ . The curves show that for the design of an optimal experiment, (a/b)sqrt $(k_y/k_x)$  needs to be around 2.25. In other words, to achieve an optimal two-dimensional experiment on an orthotropic material, the specimen needs to be cut in such a way that (a/b)sqrt $(k_y/k_x)$  is around 2.25 and three quarters of its surface need to be heated.

# **CHAPTER 4**

### THERMOCOUPLE ERROR ANALYSIS

# 4.1 Introduction and Literature Review

With the development of new materials for automotive and aerospace industries, power generation, and medicine, there is a need for the quantification of the thermal behavior of these materials and various transient thermal phenomena. By studying this thermal behavior, knowledge about the safety and feasibility of the materials and about thermal processes can be gained. These thermal studies might involve the estimation of the thermal properties thermal conductivity and volumetric heat capacity. Another related problem is the inverse heat conduction problem (IHCP), which involves the estimation of the surface heat flux history from interior transient temperatures. In problems such as estimation of thermal properties, investigation of chemical reactions in solids and the inverse heat conduction problem, the results can be extremely sensitive to measurement errors. This chapter presents a study of the measurement errors motivated, at this point by the estimation of thermal properties from transient measurements (Scott and Beck, 1991, Loh and Beck, 1991, and Garnier, Delaunay, and Beck, 1991). Transient measurements were used in part because of characterizing composite materials during the cure cycle.

Thermocouples can be placed either parallel or normal to the heated surface as shown in Figure 4.1. In both cases, the temperatures measured by thermocouples are those of the junction and not of the specimen. In this chapter, the sensor is parallel to the heated surface (Figure 4.1b). For the case of low conductivity composite materials, the thermocouple has a relatively high thermal conductivity and volumetric heat capacity resulting in a temperature measurement error. Certain systematic errors were noted.

A number of papers dealt with thermocouple errors; for example Beck (1962) used finite differences to analyze the case of a thermocouple normal to the heated surface, and showed that the disturbances can be excessively large. Pfahl and Dropkin (1966) used an implicit finite difference approach to study the case of a thermocouple parallel to the heated surface in low conductivity materials; the authors assumed the thermocouple to have a square cross-section and considered different parameters affecting the thermocouple disturbance. This configuration inherently introduces smaller errors than that normal to the heated surface, provided the location of the sensor is known. Beck (1968) also computed some correction kernels in a form of Duhamel's superposition integral to determine the undisturbed temperatures from the thermocouple measurements; the case analyzed was that of a thermocouple normal to the heated surface. Larrain and Bonilla (1968) calculated the error due to leakage current flowing through the electrical insulation between thermocouple wires for metal-sheathed swaged thermocouples used at high temperatures; the authors considered temperature errors for sheathed and unsheathed thermocouples with step change and linear temperatures. Dutt and Stickney (1969)



Figure 4.1 Thermocouple Placement Relative to the Heated Surface

considered the error due to conduction through the wire when measuring fluid or solid temperatures; their analysis used fin theory and energy balance ideas. Yoshida, Yamamoto, and Yorizane (1982) performed a finite difference calculation to determine the error due to the radial insertion of the thermocouple in a thermal conductivity measuring device; they found that the insertion of the thermocouple led to a higher temperature at that location as compared to without the thermocouple. Balakovskii (1987) considered the inverse problem of estimating the surface heat flux using thermocouple temperature measurements. He studied the sensitivity of the estimated heat flux to errors in the thermocouple location; he showed that location uncertainties cause an error in the estimated heat flux mostly at early times. Balakovskii and Baranovskii (1987) performed a similar study; they considered a thermocouple normal to the heated surface, and included the thermocouple effect in their model for the estimation of the surface heat flux.

The goal of the current study is to extend the analysis of Pfahl and Dropkin (1966). Longer dimensionless times are investigated, larger thermocouple depths are considered, and the circular cross-section is treated. The circular cross-section was treated using the finite element (FE) method; however, the FE or finite difference (FD) method is not completely satisfactory for this problem. For the problem of small disturbances (some less than one percent), the FE and FD methods can have difficulty. Even though the errors may be small, they are not random and have amplified effect on the estimation of thermal properties, and upon the IHCP. For these and other reasons, this study utilizes the Unsteady Surface Element (USE) method with a single element. The method is closely related to the Boundary Element Method (BEM) (Brebbia, 1978).

The USE method was introduced by Keltner and Beck (1981), and used by Litkouhi and Beck (1986), by Beck and Keltner (1987), and by Sobolik, Keltner, and Beck (1989). One advantage of the USE method is that only the interface nodes need to be considered initially and later the temperature at any interior or boundary location can be obtained using a convolution equation; as a result of the much smaller grid, the USE method uses less computer time than the FE or FE method. Another advantage is that the USE method treats semi-infinite bodies more readily than the two other methods. FE and FD methods are potentially much more accurate for determining the temperature level than for determining small differences in temperature at two locations (such as at the thermocouple and far away from it). The USE method is also more powerful than the two above-mentioned methods when the quantity of interest is a small difference in temperature (which is what the thermocouple disturbance is). A further very important characteristic of the USE method is that many times it leads to relatively simple algebraic solutions which cover a wide range of conditions; this permits greater insight, such as the behavior for infinite times. Such simple solutions are given in this chapter.

An outline of the chapter follows. First, a general formulation of the problem is given. Second, a Green's function solution to the problem is provided, and its results are compared to those of the FE and FD methods' solutions. Third, the form of the solution is compared to that of obtaining the undisturbed temperatures using thermocouple kernels as shown by Beck (1967). Finally, some conclusions are given.

# **4.2 Theoretical Procedure**

### 4.2.1 General Formulation

The thermocouple wire is assumed to extend infinitely parallel to the heated surface of a semi-infinite solid representing the surrounding material (Figure 4.1b). Perfect contact is assumed to exist between the thermocouple and the surrounding material, but the method can also readily treat the case of imperfect contact. The describing differential equation and its boundary conditions are:

$$\frac{\partial}{\partial x}\left(k\frac{\partial T}{\partial x}\right) + \frac{\partial}{\partial y}\left(k\frac{\partial T}{\partial y}\right) = \rho c \frac{\partial T}{\partial t} \qquad -\infty < x < \infty, 0 \le y < \infty, t \ge 0$$
(4.1a)

$$-k\frac{\partial T}{\partial y} = q_0 \qquad \qquad y = 0, t > 0 \qquad (4.1b)$$

$$T(x,y,0) = 0$$
 (4.1c)

$$T(x,y,t) \to 0 \qquad x \to \infty, x \to -\infty, y \to \infty \qquad (4.1d)$$

. . . ..

where T is the space-dependent temperature, k and  $\rho c$  are the constant thermal properties outside the thermocouple and different but constant inside the circular region of radius, a, centered at y=L and x=0 (Figure 4.1b), and  $q_0$  is the imposed constant boundary heat flux.

For simplicity, the thermal properties are assumed to be independent of temperature. This is an important case; certainly thermal properties vary with temperature, but the main error is caused by local conditions. Hence for small sensors, these results may also have some validity for temperature-variable properties. It is also true that small errors are being considered. An inaccuracy of say ten percent in an error which itself is only two percent leads to an error in the correction of only 0.2%, which

is much smaller than the uncorrected value of two percent.

The temperature disturbance caused by the presence of the thermocouple is mainly due to the difference in volumetric heat capacities of the wire and the surrounding material. The thermal conductivity of the wire is very large compared to that of the surrounding material, making the lumped capacitance assumption an acceptable one for the wire. A control volume approach is then used to obtain an expression for the driving force behind the disturbance. The procedure is based on three observations. First, the relatively high thermal conductivity, k<sub>w</sub>, of the wire (or thermocouple assembly) causes it to be nearly uniform in temperature; it can however vary with time. Second, the heat capacity of the wire minus that of the material that would be there (if the wire were not) is the main cause of the temperature disturbance. Third, the effect of lumped wire is the same as that of a volume heat sink, which is proportional to the rate of change of its temperature. In other words, a control volume just outside the wire should have the same temperature and same net integrated heat flux for the actual problem of a relatively high thermal conductivity wire as for a volume energy sink. This USE analysis permits easy extension to larger dimensionless time and provides an analytical form that yields insight more readily than purely numerical solutions.

The excess energy entering a control volume about the wire of radius a is  $\int_{0}^{2\pi} (-\vec{q} \cdot \hat{n}) a d\theta = \int_{0}^{a} (\rho c - \rho c_w) \frac{\partial T_w(t)}{\partial t} 2\pi r dr$ (4.2)

where a is the radius of the thermocouple wire,  $\mathbf{q}$  is the inward pointing heat flux, and  $\mathbf{\hat{n}}$  is the outward pointing normal.  $\rho c$  and  $\rho c_w$  are respectively the volumetric heat capacities of the region surrounding the thermocouple, and of the thermocouple wire.  $T_w$  is the temperature of the wire. Since the temperature of the wire is independent of position (lumped capacitance), Equation (4.2) becomes

$$\int_{0}^{2\pi} (-\vec{q}.\hat{n}) \mathbf{a} d\theta = (\rho c - \rho c_w) \frac{dT_w(t)}{dt} \pi \mathbf{a}^2$$
(4.3)

If both sides of Equation (4.3) are divided by the cross-sectional area  $\pi a^2$  of the wire, the average volumetric sink is found to be

$$\mathbf{g}(\mathbf{t}) = (\rho \mathbf{c} - \rho \mathbf{c}_{\mathbf{w}}) \frac{\mathrm{d} \mathbf{T}_{\mathbf{w}}(\mathbf{t})}{\mathrm{d} \mathbf{t}} \quad 0 < \mathbf{r} < \mathbf{a}$$
(4.4)

The derivative of temperature with respect to time is a total one rather than a partial one because of the lumped capacitance assumption. The describing partial differential equation then becomes

$$k\left[\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2}\right] + g(x, y, t) = \rho c \frac{\partial T}{\partial t}, \qquad -\infty < x < \infty, 0 \le y < \infty, t \ge 0$$
(4.5)

where g(x,y,t) = g(t) as given by Equation (4.4); g(t) is non-zero only at the location of the wire which is located at x=0, y=L (see Figure 4.1b).

# 4.2.2 Green's Function Approach

In this analysis, Green's functions are a natural tool because the disturbance is caused by a heat source. The current Green's function analysis is based on the work of Beck (1983), and on the book by Beck, et al. (1992).

The Green's function corresponding to an instantaneous, cylindrical source surrounded by an infinite medium is denoted  $G_{R00}(r,t|r',\tau)$ . The R subscript denotes the radial coordinate, the first zero indicates a natural boundary condition at r=0, and the

second zero is for the condition  $r \rightarrow \infty$ . This notation is described more fully by Beck and Litkouhi (1988), and Beck, et al. (1992).

The temperature of the wire is assumed uniform with position, i.e., lumped, due to the relatively large thermal conductivity of the wire. At early dimensionless times, the temperature of the wire is affected by the temperature, time, and space variations near the wire and this is better investigated using the FE and FD methods. The dimensionless time period of interest can be conveniently broken into three parts. The first part is before the temperature penetrates from the heated surface to the thermocouple and shortly thereafter; this corresponds to dimensionless times less than about  $\alpha t/L^2 \approx 0.06$ . In such times, the assumption of uniform temperature in the wire may not be accurate, compared with the temperature rise. However, the temperature rise is extremely small at these times. It is not difficult to analyze times of  $\alpha t/L^2 \approx 0.06$  using the FE and FD methods. The second part of the dimensionless time period is the period of  $\alpha t/L^2$  between 0.06 and 0.31; in this time period, the heated surface at y=0 does not cause any thermal "reflections" at y=L. During this period of time, the disturbance caused by the thermocouple is similar to that caused by a heat sink in an infinite medium. This can be modelled by using the  $G_{R00}(r,t|r',\tau)$  Green's function. The third part of the dimensionless time period is for  $\alpha t/L^2 > 0.31$ . In this time period, the effect of the surface at y=0 is important at the location of the thermocouple. The effect of this boundary is equivalent to having a second fictitious source a distance twice the depth of the thermocouple from the wire, 2L. The Green's function contribution describing this second source is given by  $G_{x_{00}y_{00}}(0,2L,t|0,0,\tau)$  as

$$G_{X00Y00}(0,2L,t|0,0,\tau) = \frac{1}{4\pi\alpha(t-\tau)} e^{\frac{-(2L)^2}{4\alpha(t-\tau)}}$$
(4.6)

where the notation X00Y00 denotes a line source in Cartesian coordinates.

We first obtain the average wire temperature as (Beck, et al., 1992):

$$T_{w}(t) = T_{w}(t) + \frac{\alpha}{k} \int_{\tau=0}^{\tau=t} \left[ \frac{1}{\pi a^{2}} \int_{r=0}^{r=a} \int_{r'=0}^{r'=a} g(\tau) G_{R00}(r,t|r',\tau) 2\pi r' dr' 2\pi r dr \right] d\tau, \quad (0.06 < \alpha t/L^{2} < 0.31)$$
(4.7)

This equation has two basic parts. One of these is  $T_{-}(t)$ , which is the undisturbed temperature at the same y=L location as the wire but a large x distance away. The other term in Equation (4.7) is caused by the volume energy.

There are two integrations over space in Equation (4.7) because one (on r') is needed for integrating over the annular source, and the other (on r) is for finding the average temperature from r=0 to a. It should be noted here that the analysis assumes the whole wire as a cylindrical source of uniform temperature and not just one line; this leads to averaging the temperature over the cross sectional area of the wire which, in turn, leads to the use of the average Green's function  $G_{R00}(t-\tau)$  as

$$\overline{G_{R00}}(t-\tau) = \frac{1}{\pi a^2} \int_{r=0}^{r=a} \left[ \int_{r'=0}^{r'=a} G_{R00}(r,t|r',\tau) 2\pi r' dr' \right] 2\pi r dr$$

$$= \frac{1}{\pi a^2} \left\{ 1 - e^{-\frac{1}{2u}} \left[ I_0(\frac{1}{2u}) + I_1(\frac{1}{2u}) \right] \right\}, \quad u = \frac{\alpha}{a^2} (t-\tau)$$
(4.8)

where  $I_0$  and  $I_1$  are the modified Bessel functions of the first kind of orders 0 and 1. Equation (4.8) is denoted  $\overline{G_{R00}}(t-\tau)$  and is the average of the integrated  $G_{R00}$  over r' (weighted with r'). This relation was derived by D. Amos of Sandia National Laboratories and is contained in Beck, et al. (1992). The quantity  $\pi a^2 \overline{G_{R00}}(t-\tau)$  starts at the value of 1 for u=0 and decreases gradually to zero with increasing u. Using the definition given by Equation (4.8), Equation (4.7) can be written as

$$T_{w}(t) - T_{a}(t) = \frac{\alpha}{k} \int_{\tau=0}^{\tau=t} \overline{G_{R00}}(t-\tau)g(\tau)\pi a^{2}d\tau$$
(4.9)

The expression for the wire temperature disturbance for times  $\alpha t/L^2 > 0.31$  then becomes

$$T_{w}(t) - T_{m}(t) = \frac{\alpha}{k} \int_{\tau=0}^{\tau=t} \left[ \frac{\overline{G}_{R00}(t-\tau)\pi a^{2} + \pi \frac{a^{2}}{4\pi\alpha(t-\tau)}}{\frac{1}{4\pi\alpha(t-\tau)}} e^{-\frac{(2L)^{2}}{4\alpha(t-\tau)}} \right] g(\tau) d\tau$$

$$= \frac{\Delta \rho c}{\rho c} \int_{\tau=0}^{\tau=t} \left[ \frac{\overline{G}_{R00}(t-\tau)\pi a^{2} + \frac{a^{2}}{4\alpha(t-\tau)}}{\frac{1}{4\alpha(t-\tau)}} e^{-\frac{L^{2}}{\alpha(t-\tau)}} \right] \frac{dT_{w}(\tau)}{dt} d\tau$$
(4.10)

Notice that Equation (4.10) can be considered as an integral equation for the unknown function  $T_w(t)$  which is valid for any time greater than say  $\alpha t/L^2 \approx 0.1$ .

One major simplification to the solution of this equation is the result of small temperature disturbances. For such cases, the rate of change of the wire temperature is nearly equal to the rate of change of the undisturbed temperature,  $T_{\infty}(t)$ ; in equation form, we have

$$\frac{\mathrm{d}T_{w}}{\mathrm{d}t} \approx \frac{\mathrm{d}T_{w}}{\mathrm{d}t}$$
(4.11)

This undisturbed temperature,  $T_{\omega}(t)$ , is the temperature distribution in a semi-infinite solid subjected to a constant heat flux. At the thermocouple location x=L, it is given by

$$T_{\mathbf{x}}(t) = 2\frac{q_0}{k}\sqrt{\alpha t} \ \operatorname{ierfc}(\frac{L}{2\sqrt{\alpha t}}) = 2\frac{q_0}{k}\sqrt{\alpha t} \left\{ \frac{1}{\sqrt{\pi}} e^{-\frac{L^2}{4\alpha t}} - \frac{L}{2}\operatorname{erfc}(\frac{L}{2\sqrt{\alpha t}}) \right\}$$
(4.12)

where ierfc is the integral of the complementary error function. Then,

$$\frac{dT_{\bullet}(t)}{dt} = \frac{q_0}{k} \sqrt{\frac{\alpha}{\pi}} \left[ \frac{1}{\sqrt{t}} e^{-\frac{L^2}{4\alpha t}} \right]$$
(4.13)

This simplifies the solution of Equation (4.10) in the sense that the equation becomes an explicit expression of  $T_w(t)$ ; it is no longer an integral equation. Equation (4.13) is then used to obtain the normalized temperature disturbance  $\Delta \phi$  such that

$$T_{w}(t) - T_{\omega}(t) = \frac{\Delta \rho c}{\rho c} \int_{\tau=0}^{\tau=t} \left[ \frac{\overline{G_{R0}}(t-\tau)\pi a^{2} + \frac{a^{2}}{4\alpha(t-\tau)}e^{-\frac{L^{2}}{\alpha(t-\tau)}} \right] \frac{q_{0}}{k} \sqrt{\frac{\alpha}{\pi\tau}} e^{-\frac{L^{2}}{4\alpha\tau}} d\tau$$
(4.14)

or

$$\Delta \phi = \frac{\Delta \rho c}{\rho c} \int_{\tau^{+}=0}^{t^{+}} \left[ \frac{\overline{G}_{R00}(t-\tau)\pi a^{2} + \frac{H^{2}}{4(t^{+}-\tau^{+})} e^{\frac{-1}{t^{+}-\tau^{+}}} \right] \frac{1}{\sqrt{\pi \tau^{+}}} e^{\frac{-1}{4\tau^{+}}} d\tau^{+}$$
(4.15a)

where

$$\Delta \phi = \frac{k}{q_0 L} [T_w(t^+) - T_u(t^+)], \quad t^+ = \frac{\alpha t}{L^2}, \quad \tau^+ = \frac{\alpha \tau}{L^2}, \quad H = \frac{a}{L} \quad (4.15b, c, d, e)$$

Equation (4.15a) reveals that the temperature disturbance is directly proportional to the ratio  $\Delta \rho c/\rho c$ , which represents the difference of volumetric heat capacities between the thermocouple wire and the surrounding material, normalized with respect to that of the surrounding material. As a result, dividing both sides of Equation (4.15a) by  $C = \Delta \rho c/\rho c$  eliminates the explicit dependence of the disturbance on the difference in volumetric heat capacities. This fact is not apparent from FE and FD analyses. The numerical evaluation

of Equation (4.15a) was done using the IMSL library.

The normalized temperature disturbance is computed for non-dimensional times as large as 1000, and for thermocouple radius to depth ratios, H, of 0.25, 0.125, 0.0625, and 0.03125; numerical integration using some IMSL subroutines was performed for this purpose. The temperature disturbance is shown in Figure 4.2 as a function of dimensionless time. The data generated by Pfahl and Dropkin (1966) (Figure 8) for the case of H = 0.25, k/k<sub>w</sub>=0.025, and  $\Delta \rho c/\rho c$ =-9, as well as the data obtained using the FE code TOPAZ2D are also shown in Figure 4.2 which is discussed below.

In order to decide which method was more accurate, the competing results were compared to the FE extrapolated results corresponding to a grid size equal to zero. This was done by setting up three different FE grids, one with 374 nodes, another one with 825 nodes, and a third one with 1452 nodes. A  $(\Delta x)^2$  extrapolation was then used to obtain the expected FE results corresponding to  $(\Delta x)^2=0$ . For reasonably sized grids, the dimensionless temperatures varied in a linear fashion with  $(\Delta x)^2$ . The results of the extrapolation are shown in Figure 4.2 along with the FD results and the USE ones. That figure shows that, for dimensionless times greater than 3, the thermocouple disturbances calculated by the USE method are the same as the FE extrapolated results. For dimensionless times less than 3, the USE and FE results are different; this difference was expected since, as mentioned above, the USE method does not apply to early times.

It should be noted here that, in generating the FE results, the calculated undisturbed temperatures were up to 1.6% different from the exact temperatures obtained by Equation (4.12). This limitation, added to the fact that the FE-calculated temperatures



Figure 4.2 Theoretical, FE, and FD Thermocouple Disturbances

depend on the grid size, made the FE method not reliable for our purposes of estimating thermocouple disturbances.

The USE curves of Figure 4.2 show linear dependence on  $H^{\beta}$ ; a least squares fit showed that  $\beta$  is about 1.9. Larger values of H lead to larger values of the temperature disturbance. This allowed the normalization of the curves with respect to  $H^{1.9}$ , and Figure 4.3 was then obtained.

#### 4.2.3 The Unsteady Surface Element Method Versus Inverse Convolution

The above method of obtaining the thermocouple induced temperature disturbance is now compared to the method described by Beck (1968). The latter uses thermocouple function kernels, K(t), which need to be computed by numerical inversion of a convolution integral. That inversion method employs a least squares procedure similar to the inverse problem of estimating the surface heat flux from internal temperature measurements. The expression for the disturbance given by that method is

$$T_{w}(t) - T_{w}(t) = -\int_{\tau=0}^{\tau=t} K(t-\tau) \frac{\partial T_{w}(\tau)}{\partial t} d\tau$$
(4.16)

where  $T_w(t)$  is the measured function of time. Notice that this equation does not require any inverse convolution calculations to find the error in the measurements, with K(t) known. Note also that K(t- $\tau$ ) is a universal function in the sense that it is independent of time variation of  $T_w(t)$ ; it does however depend upon the geometry.

Comparing Equation (4.16) to Equation (4.10), the kernel  $K(t-\tau)$  is related to Green's functions; more precisely, for the present problem, we have


Figure 4.3 Normalized Theoretical Thermocouple Disturbance

$$K(t-\tau) = \frac{\Delta \rho c}{\rho c} \left[ \frac{\overline{G}_{R00}(t-\tau)\pi a^2 + \frac{a^2}{4\alpha(t-\tau)}e^{-\frac{L^2}{\alpha(t-\tau)}} \right]$$
(4.17)

which is shown in Figure 4.4 along with  $\overline{G_{R00}}(t-\tau)$  for different a/L ratios. The kernel  $K(t-\tau)$  in Equation (4.16) then represents a Green's function. This is an important observation and has significant applications; it means, for example, that the error using Equation (4.16) can be found provided the Green's function K(t) can be found. Since K(t) represents a Green's function, it is not necessary to solve the complete problem as has been done herein; instead, one can concentrate on obtaining K(t). No inverse convolution is now needed to obtain the kernel  $K(t-\tau)$ ; instead, this kernel can now be computed from known Green's functions which can be obtained using exact expressions available in the literature such as (Beck, et al., 1992).

## 4.4 Results and Discussion

The restriction to relatively large values of thermal conductivity of the thermocouple compared to that of the surrounding material is not severe for measurements in low conductivity materials. For example, a surrounding material might be epoxy or composite material whose thermal conductivities are about 0.2 W/m°C. The thermocouple assembly can be an alumina sheath through which two wires pass; the thermal conductivities is about 40 W/m°C. The ratio of the two thermal conductivities is about 200, which justifies the assumption of uniform temperature inside the wire.

The major simplification used with the Green's function solution (Equation (4.11))



Figure 4.4 Kernel K(t-t) for Different Thermocouple-to-Depth Ratios

was checked using FE computations (TOPAZ2D). The transient temperatures at the wire location and at a "distant" location were obtained; the rates of change of the temperatures at the two locations with time were then found to be the same.

Figure 4.3 shows the temperature disturbance normalized with respect to  $C = \Delta \rho c / \rho c$ and H<sup>1.9</sup>. The dimensionless disturbance is of the order of 0.4 for dimensionless times between 0 and 100. In some experiments performed in our laboratory on a DER332/DDS epoxy sample, a heat flux of 4700 W/m<sup>2</sup> causes a maximum temperature rise of about 100°C. The thermal properties of this epoxy are about 0.2 W/m°C for k and 1.5×10<sup>6</sup>  $J/m^{3\circ}C$  for pc. Using this information along with Equation (4.15a), the dimensionless temperature disturbance of 0.4 corresponds to about 1°C. Since Equation (4.15a) was derived for an infinitely long thermocouple, it can be induced (by linearity of the problem) that the disturbance for the finite length thermocouple is only 0.5°C. The maximum temperature disturbance is therefore about 0.5 percent of the maximum temperature rise. This is a small value; however, as shown in Chapter 2, when estimating the thermal properties from transient measurements of temperature and heat flux, the residuals (difference between the calculated and measured temperatures) for this case were about 2%; this means that the disturbance caused by the thermocouple is 25% of the discrepancy between the model and the experiment.

## **CHAPTER 5**

# EXPERIMENTAL INVESTIGATION OF THE THERMOCOUPLE ERROR ANALYSIS

# **5.1 Introduction**

The process of estimating thermal properties, and finding ways to improve transient parameter estimation experiments is a combination of two interrelated tasks: an analytical one and an experimental one. The analytical task was presented earlier in this dissertation in Chapters 3 and 4. Part of the experimental task is presented in this chapter. This experimental task consists of "measuring" the temperature disturbance caused by the measuring device (here a thermocouple). To achieve this goal, a series of experiments was designed and then performed in the Laboratoire de Thermocinetique-ISITEM Nantes-France. The material of interest is a cured epoxy, similar to the one of Chapter 2. The specimen has an imposed finite-duration constant heat flux on one side and a 40°C constant temperature on the other side.

This chapter describes the design, implementation, and results of the series of experiments. The first part of the chapter briefly describes some criteria used in designing the experiments, and making the specimen. The next part of this chapter

explains the procedure followed. The third part of the chapter describes the setup used. The fourth part of the chapter describes the experiments performed on the specimen, and compares the results to those of Chapter 4. In that same part, the thermocouple locations are estimated, and the corresponding disturbances are calculated. Unfortunately, the position errors seemed to mask much of the errors caused by the thermocouples themselves. A sensitivity analysis on the importance of different parameters was also performed. The last part of this chapter discusses the results of the experimental investigation.

#### 5.2 Some Design Criteria of the Experiment

In designing the thermocouple error analysis experiment, four criteria were satisfied. First, the thermocouple-induced disturbance had to be large. Second, the different experimental parameters such as heat flux, temperature, and thermocouple locations had to be known as accurately as possible. Next, the sample had to be thick enough to behave as a semi-infinite body during the experiments; in other words, none of the thermocouples "sees" the back boundary of the sample. Finally, the thermocouples had to be placed far enough from each other to minimize thermal interaction, and to not disturb each other.

To satisfy these criteria, thermocouples of three diameter sizes were selected: 0.16mm, 0.5mm, and 2mm and were numbered as shown in Figure 5.1. The 0.16mm and 0.5mm-diameter wires were used because those were the largest diameter wires available in the laboratory. The 2mm-thermocouple was a little different. It was made from a



\* distances between thermocouples not shown to scale

Figure 5.1 Thermocouple Numbering for the Estimation of Thermocouple Disturbance

Chromel-Alumel male plug. The leads of the plug were cut, then welded tip-to-tip at the junction. A platinum gage was used to measure the reference temperature that was needed in the measurement of all the experimental temperatures. The 140mm×140mm DER332 epoxy-based specimen (described with more detail in the next section) was 18mm thick, insuring its semi-infinite thermal behavior. The thermocouples were placed 10mm away from each other to make sure they do not interact during the experiments.

## 5.3 Experimental Procedure

The first step of this experimental investigation was to build the specimen with the chosen themocouples. The mold shown in Figure 5.2 was used to make this specimen with the installed thermocouples. The spring system shown in that same figure kept the smallest thermocouples in tension to improve the accurate positioning of the corresponding junctions. The mold was sprayed with a mold-release compound to facilitate the separation of the sample from the mold after the epoxy was cured. The thermocouples were cleaned with an acid-based solution to eliminate any impurities that might introduce some contact resistance between the thermocouples and the epoxy. These thermocouples were then stretched across the mold as shown in Figures 5.2 and 5.3. The DER332-based epoxy was mixed with the curing agent, then poured inside the mold. The mold was then placed in a vacuum chamber to eliminate all the air bubbles in the epoxy. The mold was then placed inside an oven to cure the epoxy. Later, the cured sample was removed from the mold and allowed to cool.

The next step of the experimental investigation was to polish both surfaces of the



Figure 5.2 Mold and Different Methods Used in Stretching the Thermocouples



Figure 5.3 Thermocouple Layout in the Mold

sample. In doing so, thermal contact between the specimen and the bounding heater on one side, and between the specimen and the rear plate on the other side was improved. After polishing the specimen, it was clearly visible that most of the thermocouples had moved inside the sample. This movement was due to the release of the tension that was imposed by the spring system. The locations of the thermocouple junctions were thus altered. The new locations were estimated as described in Section 5.5.6.

The third step of this experimental study was to use the finished sample with a setup already in place at the Laboratoire de Thermocinetique-ISITEM Nantes-France. This setup is described in the following section.

#### 5.4 Experimental Setup

The experimental setup was previously built for estimating the thermal conductivities of low conductivity materials. A schematic of the setup is shown in Figure 5.4. It mainly included a Hewlett Packard (HP)-based data acquisition system, a pair of Lauda temperature control units, and a hydraulic unit in which the finished sample was placed.

The HP-based data acquisition system consisted of an HP terminal, an HP scanner (model 3455A), an HP digital voltmeter (model 3490A), and a computer controlled power supply unit. The next part of the setup was a pair of Lauda temperature control machines pumping oil into a closed loop to maintain a constant boundary temperature. The oil leaves the reservoir of the Lauda machine through a rubber hose to reach the plates shown in Figure 5.5, then returns back through another rubber hose to the reservoir.



Figure 5.4 Experimental Setup for the Estimation of Thermocouple Disturbance



Figure 5.5 Placement of the Epoxy Specimen for the Estimation of Thermocouple Disturbance

Finally, the hydraulic unit of Figure 5.5 consisted of two plates (labeled hot source and cold source in the figure) through which the constant temperature oil passes, an electric heater embedded in an aluminum block, an electronic caliper, a compressed air system, and finally some side insulation. The electronic caliper allowed the measurement of the average thickness of the sample. The compressed air system was needed to raise and lower the upper constant temperature plate. This air system also held the specimen, the heating plate, and the constant temperature plate together. Good contact was thus maintained by the constant pressure applied throughout the experiment.

The scanner had thirteen channels and each temperature was read at a different time. One channel recorded voltage for the power input. Another channel recorded the current (which is really voltage converted to current by means of a 10-ohm resistor). A third channel allowed the recording of the reference temperature measured by a platinum gage (which is an accurate temperature measurement device). A fourth channel was connected to the electronic caliper, allowing the measurement of the average thickness of the sample. Two other channels recorded the temperatures behind the heating plate (TC3 and TC8 of Figure 5.5). Two more channels recorded the temperatures of the specimen's boundaries (TC0 and TC1 of Figure 5.5). Finally, six more channels acquired the temperatures inside the specimen. The time was recorded every time a channel was scanned. In doing so, it was possible to circumvent the limitation of sequential reading of the channels and to minimize any possible timing errors.

A thin layer of silicone grease was applied to the aluminum plate covering the heating element, and to the back surface of the specimen. The specimen was then

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carefully placed on top of the heating element. Insulating material of the same thickness as the sample was placed all around to eliminate side losses. The upper part of the unit was lowered hydraulically. The thermocouples were then connected to the data acquisition system, and four successful experiments were run.

# 5.5 Experimental Results

The thermal properties were needed in the estimation of the thermocouple disturbance, and had to be obtained first. The thermal conductivity of the specimen was estimated by running a steady state experiment. The volumetric heat capacity was assumed known as indicated in Chapter 2. In that chapter, the volumetric heat capacity of DER332/DDS epoxy was found to be about  $1.5 \times 10^6$  J/m<sup>3o</sup>C.

The first one of the four successful experiments was steady state and allowed the estimation of the thermal conductivity of the specimen. The three other experiments were transient ones performed in order to investigate thermocouple disturbance. More than one experiment was needed (three here) because the specimen had eleven thermocouples and the data acquisition system had only six channels available for thermocouple hookup.

#### 5.5.1 Steady State Experiment

The steady state experiment allowed the system to reach a linear temperature distribution. One boundary was at 30°C and the other at 40°C. The 30°C boundary was possible by the cold plate of Figure 5.5. The 40°C boundary was achieved by the heating element and the hot plate of the same figure. The heater was turned on and off by the data acquisition system every time the temperature difference between TC3 and TC8 of

Figure 5.5 was larger than 0.025°C (which is really  $1\mu$ V). The thermal conductivity, k, of the sample was calculated by

$$\mathbf{k} = \overline{\mathbf{q}} \frac{\Delta \mathbf{x}}{\Delta \overline{\mathbf{T}}} \tag{5.1}$$

where L is the thickness of the specimen,  $\Delta T$  is the difference in average temperature between the two boundaries of the specimen, and q is the average heat flux imposed on the specimen. The software for this experiment was previously developed in the laboratory; a brief description is given here. The data acquisition system kept sampling continuously in time; when the difference between the readings of thermocouples TC3 and TC8 of Figure 5.5 was less than 0.025°C (which is really 1µV), the current, voltage, and temperatures were stored. The process of sampling and storing the different quantities was repeated sixty times. An average of the stored data was taken and the thermal conductivity was calculated using Equation (5.1). It was found to be about 0.237W/m°C at 40°C; this value is 12% larger than the upper limit of the confidence region of Figure 2.5 obtained for a DER332-based epoxy with a different curing agent.

## 5.5.2 Transient Experiments

At this point, the thermal properties of the epoxy were known, and the temperatures recorded by the different size thermocouples were needed to estimate the disturbance. These temperatures were obtained in a series of three experiments. First, the system was allowed to reach the constant uniform initial temperature of 40°C. A lower temperature was not chosen because of the difficulty of controlling temperatures near the ambient temperature. Once an initial steady state was reached, data collection started. The heater was turned on around time 33 seconds to impose a constant heat flux.

At time 153 seconds, the heater was turned off, and data collection continued until the end of the experiment (at time 219 seconds).

The temperatures recorded during the three experiments are shown in Figures 5.6, 5.7, and 5.8. The two temperature curves of Figure 5.6 labeled  $T_3$  and  $T_8$  correspond to thermocouples TC3 and TC8 at the back of the heater (recall Figure 5.5). These temperatures  $T_3$  and  $T_8$  increased with time from 39.5°C to about 47°C and 43°C respectively. The increase in  $T_3$  and  $T_8$  is an indication that it was incorrect to assume that 100% of the heat supplied by the heating plate goes into the specimen. The heat flux at the heated surface of the specimen was then unknown. Knowledge of this boundary heat flux was crucial for the estimation of the thermocouple temperature disturbance. It was then necessary to estimate the heat flux. This is done in the following section.

The temperatures recorded by thermocouples 05 (of Figure 5.7) and 06 (of Figures 5.6 and 5.8) are inaccurate. The curves  $T_{05}$  and  $T_{06}$  corresponding to these thermocouples are not as smooth as the other curves; curves  $T_{05}$  and  $T_{06}$  show some unusual oscillations throughout the whole experiment, even at early times before the heating starts. These early time oscillations between time 0 and time 33 seconds indicate that thermocouples 05 and 06 are not reliable ones. As a result of the non-reliability of those two thermocouples, the data they collected had to be ignored in the remaining part of the dissertation.

Figure 5.6 shows that the experimental heated surface temperature curve,  $T_1$ , and the curve  $T_{exact}$  for the calculated surface temperature (calculated as shown later in this chapter) are similar. The maximum difference between the two curves is less than 1°C,





Figure 5.7 Measured Temperatures of Experiment 3



Figure 5.8 Measured Temperatures of Experiment 4

which is of the same size as the temperature error at the surface caused by a 10% error in heat flux.

Notice that thermocouples 04, 09, and 10 were used in more than one experiment. Thermocouple 04 was used in experiments 2, 3, and 4. Thermocouple 09 was used in experiments 2 and 4, and thermocouple 10 was used in experiments 2 and 4. This repeated use of the same thermocouples was needed to check for replication. The replication sought here was of the first order (recall Chapter 2); it was remarkably good for all three thermocouples 04, 09, and 10.

## 5.5.3 Calculation of the Surface Heat Flux and the Thermocouple Disturbance

The calculation of the surface heat flux was motivated by the existence of a heat flow at the insulated back of the heater. This back heat flow implied that the heat flux at the surface of the specimen was unknown, not as thought initially to be known from voltage, current, and surface area (recall Chapter 2). This surface heat flux was expected to be a constant finite duration pulse. The experiments of interest here were the ones that led to Figures 5.6, 5.7, and 5.8. All three experiments had the same boundary conditions; the only difference was that different thermocouples were used. One boundary of the specimen (finite body) was heated by the unknown heat flux, and the other boundary was held at a constant 40°C temperature. Ideas of the inverse heat conduction problem (IHCP) (Beck, Blackwell, and St Clair, 1985) were implemented in order to obtain this heat flux. The software IHCP1D (Beck, 1990) was used for that purpose, and the transient heat flux was estimated from the data of Figure 5.6, using the surface thermocouples TC0 and TC1 of Figure 5.5. The calculated heat flux is shown in Figure



Figure 5.9 Estimated Surface Heat Flux Using IHCP1D

5.9. This figure shows that the heat flux going into the sample varied with time, and was not constant as thought initially.

To account for the time dependence of the calculated surface heat flux, a convolution was needed in the calculation of the exact surface temperature. This convolution is written as

$$T(t) = \int_{0}^{t} q(\tau) \frac{\partial \phi(q_{0}, t-\tau)}{\partial t} d\tau = \sum_{i=1}^{N} q_{i}(\phi(q_{0}, t_{N-i+1}) - \phi(q_{0}, t_{N-i}))$$
(5.2)

where the time interval t was divided into N equal time steps, and each time step was labeled  $t_i$ ;  $q_i$  is the constant heat flux at time  $t_i$ , N is the index corresponding to the time of interest t, and  $\phi(q_0, t_i)$  is the exact temperature calculated at time  $t_i$  for the constant heat flux  $q_0$  equal to unity. This temperature  $\phi(q_0, t_i)$  was calculated for a semi-infinite geometry for the following reason. For dimensionless times less than 0.3 (based on the thickness of the specimen), the heated surface of the finite thickness specimen does not "see" the constant temperature boundary, and behaves as the heated surface of a semi-infinite region. The dimensionless time for the specimen based on the thickness of the specimen (0.018m) and the assumed known thermal properties (k=0.2W/m°C,  $\rho c=1.5 \times 10^6 J/m^{30}$ C) for the maximum time of the experiment (233 seconds) was only 0.095 (which is smaller than 0.3).

The temperature for a semi-infinite geometry with constant heat flux is

$$T_{\infty}(q_0,t) = T_0 + \frac{2q_0}{k}\sqrt{\alpha t} \ \text{ierfc}(\frac{x}{2\sqrt{\alpha t}})$$
(5.3)

where  $T_0$  is the initial temperature,  $q_0$  is the constant surface heat flux, x is the distance from the heated surface. The corresponding temperature T(q(t)) for a time dependent heat flux was computed at the heated surface with the convolution of Equation (5.2), then plotted in Figure 5.6 (as  $T_{exact}$ ) with the experimental temperatures.

As mentioned above, the temperatures used in the computation of the discretized convolution integral are evenly spaced in time. The data acquisition system did not allow this even time spacing, and a linear interpolation was used to compute experimental temperatures,  $T_{exp,interp}$ , for each thermocouple at every two seconds. These interpolated temperatures,  $T_{exp,interp}$ , were then used to compute the finite difference convolution of Equation (5.2). The calculated exact temperature,  $T_{exact}(q(t))$ , for the experimental time-dependent heat flux was obtained at the specified locations of the thermocouples. Finally, the temperature disturbance was computed as

$$\Delta T(t) = \int_{0}^{t} q(\tau) \frac{\partial}{\partial t} (\Delta \phi(q_0, t-\tau)) d\tau - \sum_{i=1}^{N} q_i (\Delta \phi(q_0, t_{N-i+1}) - \Delta \phi(q_0, t_{N-i}))$$
(5.4)

where  $\phi(q_0, t_i)$  is the exact temperature calculated at time  $t_i$  for the constant heat flux  $q_0$  equal to unity. This equation gives the experimental temperature disturbance for a time dependent heat flux. This disturbance was next compared with the analytical temperature disturbance of Chapter 4. This comparison is done in the next section.

#### 5.5.4 Comparison of the Experimental and Analytical Temperature Disturbances

Unfortunately, the values and sign of the experimental temperature disturbances were not in agreement with those obtained using the USE method (recall Chapter 4). The possible sources of this disagreement had to be determined. The first one of these sources was the error in thermocouple locations. The cause of this error was the movement of the thermocouples noted after releasing the tension on the wires. The error in thermocouple locations was found to affect the temperature disturbance a great deal. Knowledge of the exact location of the thermocouple junctions then became crucial. Xray could have been used to measure the thermocouple locations. This was not done though because the importance of the thermocouple location was quantified only later when the specimen was not available. A numerical estimation of the thermocouples locations was then the only method to use. This estimation was carried out as described in the next section.

#### 5.5.5 Estimation of Thermocouple Locations

The calculation of the thermocouple locations was made possible, in part, by the parameter estimation program PROP1D (see Chapter 2). The first step in the estimation of the junctions' locations was to calculate the analytical disturbance using the USE method as shown above for a constant surface heat flux, then to apply the convolution of Equation (5.5) to account for the time dependence of the heat flux. At each time step, the value  $q_i$  of the heat flux was assumed constant, and the difference of disturbances  $\Delta \phi(q_0, t_{N-i+1})$  and  $\Delta \phi(q_0, t_{N-i})$  was multiplied by that constant heat flux. The analytical disturbance for the time dependent heat flux was thus obtained.

Ideally, the experimental temperatures used in the least squares minimization of PROP1D should not have thermocouple-induced errors (recall Equation (2.19) of Chapter 2); however, based on the current study, experimental temperatures are known to have such errors (which are in fact calculated by the USE method and the convolution of Equation (5.4)). To accommodate for these thermocouple-induced errors in PROP1D (when estimating thermocouple locations), the experimental temperatures were corrected

before being used with PROP1D. This was accomplished by subtracting the errors calculated by the USE from the experimental temperatures.

Recall from Chapter 2 that PROP1D allows the estimation of the thermal properties k and  $\rho c$ . To estimate the thermocouple depth d, the heat equation was transformed as:

$$k\frac{\partial^2 T}{\partial x^2} = \rho c \frac{\partial T}{\partial t} \rightarrow k \frac{\partial^2 T}{\partial \left(\frac{x}{d}\right)^2} = (d^2 \rho c) \frac{\partial T}{\partial t}$$
(5.5a)

$$T(x=0,t) = T_1(t)$$
 (5.5b)

$$T(x=d,t) = T_2(t)$$
(5.5c)

$$T(x,t=0) = T_0$$
 (5.5d)

where k is the known thermal conductivity, and  $\rho cd^2$  is the unknown "new" volumetric heat capacity. The new space variable is not just x, but x/d; the thickness of any region was thus divided by an initial guess of the thermocouple depth, d. The weighted volumetric heat capacity estimated by PROP1D was divided by the original one to get  $d^2$ . This new thermocouple location was used to compute the undisturbed temperature and then the disturbance (by the USE method). The whole process was repeated until the calculated thermocouple location stopped changing. This took no more than two or three iterations.

The new estimated thermocouple locations are listed for the thermocouples of interest in Table 5.1 along with the locations specified before making the sample. The absolute error in the location of the thermocouples was as high as 26% (for thermocouple

Thermocouple	Nominal Location (mm)	Estimated Location (mm)	Approx. Error (mm)
00	1	1.021	0.02
02	1	0.739	-0.26
04	2	2.251	0.25
06	2	1.823	-0.18
07	2	1.851	-0.15
08	2	2.120	0.12
09	4	3.523	-0.43
10	4	4.336	0.33

Table 5.1 Nominal Versus Estimated Thermocouple Locations

02). One should not be misled by this large value; it should be kept in mind that the nominal thermocouple depths were only 1, 2, and 4mm. Taking into account all the errors involved with preparing the mold, the degree to which the thermocouples were stretched across the mold, and the effect of the release of the tension on the thermocouples, an error of 0.26mm out of 1mm (for thermocouple 02) was not too surprising. The same conclusion can be drawn for the other thermocouples.

#### 5.5.6 Sensitivity Analysis

The goal of the final part of this experimental investigation was to quantify analytically the importance of the effect of the possible sources of disagreement between the experimental and analytical studies. Some of the possible sources of discrepancy are the thermocouple location, the thermal conductivity of the epoxy, and its volumetric heat capacity. To reach the goal of quantifying the importance of these sources, a sensitivity analysis was performed. The first step of this analysis consisted of differentiating the exact undisturbed temperature  $T_{\omega}$  given by Equation (5.3) with respect to the parameters of interest (here the sources of discrepancy) one at a time; next, this derivative was multiplied by the expected error of each parameter as well as the parameter. Finally, the resulting quantity was compared to the disturbance at the same depth. For the thermocouple location, the sensitivity gives

$$\Delta T_{\bullet} = x \frac{\partial T_{\bullet}}{\partial x} \frac{\Delta x}{x} = \left[ \frac{-xq_0}{k} \operatorname{erfc}\left(\frac{x}{2\sqrt{\alpha t}}\right) \right] \frac{\Delta x}{x}$$
(5.6)

The sensitivity to thermal conductivity results in

$$\Delta T_{a} = k \frac{\partial T_{a}}{\partial k} \frac{\Delta k}{k} = q_{0} \left[ -\sqrt{\frac{t}{k\rho c}} \operatorname{ierfc}\left(\frac{x}{2\sqrt{\alpha t}}\right) + \frac{x}{2k} \operatorname{erfc}\left(\frac{x}{2\sqrt{\alpha t}}\right) \right] \frac{\Delta k}{k}$$
(5.7)

and the sensitivity to volumetric heat capacity provides

$$\Delta T_{n} = \rho c \frac{\partial T_{n}}{\partial \rho c} \frac{\Delta \rho c}{\rho c} = q_{0} \left[ -\frac{1}{\rho c} \sqrt{\frac{t}{k\rho c}} \operatorname{ierfc}\left(\frac{x}{2\sqrt{\alpha t}}\right) - \frac{x}{2k\rho c} \operatorname{erfc}\left(\frac{x}{2\sqrt{\alpha t}}\right) \right] \frac{\Delta \rho c}{\rho c}$$
(5.8)

Three values of radius-to-depth ratio, H, were considered (H=0.0625, H=0.125, and H=0.25) at two dimensionless times 5 and 10. The results are shown in Tables 5.2, 5.3, and 5.4. Table 5.2 was generated for a 10% error in thermocouple location. Table 5.3 was generated for a 2.4% (not 10%) error in thermal conductivity. This was done because, as reported by Garnier (1991), the error in the estimation of the thermal conductivity using the same setup was less than 2.4%. Finally, Table 5.4 was generated for a 2% error in volumetric heat capacity; this was based on the results of Chapter 2. The confidence intervals of the estimated volumetric heat capacities in that chapter reflect an error less than 2%.

t <sup>+</sup>	Н	<b>▲</b> T_(x) (°C)	$\Delta T_{ans}(x) (°C)$	$\Delta T_{ans} / \Delta T_{ans}(x)$
	0.0625	-0.075	-0.003	25
5	0.125	-0.075	-0.009	8.3
_	0.25	-0.075	-0.030	2.5
	0.0625	-0.083	-0.0023	36
10	0.125	-0.083	-0.0075	11.1
	0.25	-0.083	-0.028	3

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Table 5.2 Sensitivity of Temperature Disturbance to a 10% Error in Thermocouple Location

Table 5.3 Sensitivity of Temperature Disturbance to a 2.4% Error in Thermal Conductivity

t <sup>+</sup>	Н	<b>△</b> T <b>_</b> (x) (°C)	$\Delta T_{ana}(x) (°C)$	$\Delta T_{ans}(x)$
	0.0625	-0.011	-0.003	3.67
5	0.125	-0.011	-0.009	1.22
	0.25	-0.011	-0.030	0.367
	0.0625	-0.022	-0.0023	7.334
10	0.125	-0.022	-0.0075	2.44
	0.25	-0.022	-0.028	0.733

t <sup>+</sup>	Н	<b>△</b> T <b>_</b> (x) (°C)	$\Delta T_{ana}(x) (^{\circ}C)$	$\Delta T_{\Delta} / \Delta T_{ana}(x)$
	0.0625	-0.0239	-0.003	7.987
5	0.125	-0.0239	-0.009	2.662
	0.25	-0.0239	-0.030	0.799
	0.0625	-0.0351	-0.0023	15.261
10	0.125	-0.0351	-0.0075	4.68
	0.25	-0.0351	-0.028	1.254

Table 5.4 Sensitivity of Temperature Disturbance to a 2% Error in Volumetric Heat Capacity

### **5.6 Discussion of the Results**

The estimated heat flux obtained by IHCP1D was based on measured temperatures. These measured temperatures have thermocouple-induced errors as shown in Chapter 4, which means that the estimation of the heat flux should have been an iterative procedure. The iterative procedure would first involve estimating the heat flux, the thermocouple locations, the temperature disturbance, then correction of the measured temperatures using the obtained locations and disturbance values, and finally estimation of the heat flux. The same steps are repeated until the heat flux stops changing from one iteration to the next one. This whole iterative process on the heat flux was avoided because, even with the assumption of an error-free heat flux, the experimental thermocouple disturbance was found to be quite different from the analytical one.

Tables 5.2, 5.3, and 5.4 show that thermocouple location affects the temperature disturbance calculations more than the thermal properties. The third column of Table 5.2

indicates that a 10% error in thermocouple location causes errors in the exact undisturbed temperature from 2.5 to 36 times in the value of the calculated temperature disturbance. The thermocouple-induced temperature disturbance (as shown in Chapter 4) is less than 5% for dimensionless times larger than 3. A 10% error in the thermocouple location then causes a net error between 12.5% (2.5×5%) for H=0.25, and 180% (36×5%) for H=0.0625 on the measured temperature. Such an error is too large because it is much greater than the residuals of Chapter 2. The stretching of the thermocouple before curing the epoxy and the release of the tension after the epoxy was cured proved to be an inefficient technique when estimating thermocouple temperature disturbance. Larger thermocouples proved to be more consistent with analytical results than smaller thermocouples. To insure the future success of this experimental technique, the specimen should be x-rayed to determine the exact locations of the thermocouples. The specific heat of the specimen should be obtained by DSC (Chapter 2), and its density should be obtained by the standard method for density of glass by buoyancy (Chapter 2). The setup could be modified to have the heater sandwiched between two identical specimens as in Chapter 2, so that the imposed heat flux is equally shared by the two identical specimens.

# **CHAPTER 6**

## **EXPERIMENTAL VERIFICATION OF OPTIMAL EXPERIMENTS**

#### **6.1 Introduction**

A unique component of this dissertation is the development of optimal experiments, as discussed in Chapter 3. The statistical assumptions in that chapter are quite idealized, including no bias and uncorrelated errors. It is known that these conditions are not satisfied in our experiments. Nevertheless, the general guidelines provided in that chapter should lead to better experiments than if they were not used. It is extremely important that the optimal experiments concepts have practical application and thus can be experimentally verified.

The objective of this chapter is to demonstrate that the optimal experiment designs lead to better parameter estimates. One indication that one experiment is better than another experiment is that the better experiment has a smaller confidence interval. Fortunately, program PROP1D can provide such intervals. However, a number of the basic assumptions in Chapter 3 are not experimentally satisfied. One of these is that the errors are uncorrelated; PROP1D models these errors as a first order autoregressive process. There are also some other conditions that are not satisfied. Nevertheless, it is

believed that the recommended optimal experiments in Chapter 3 are superior to other related experiments. This can only be demonstrated through some experiments.

The experimental verification of the analytical results of Chapter 3 was originally made possible by a setup different from the one described in Chapter 2. This setup was developed by Garnier, Delaunay, and Beck (1991). The setup uses resistance thermometer devices (RTD) and thermocouples to measure temperatures. Using this setup, the X21B50T0 case of Chapter 3 was implemented in our laboratory. The material of interest was DER332/DDS epoxy. The thermal properties of the sample were estimated for four different experimental conditions, then confidence intervals were obtained and compared.

## 6.2 Experimental Setup

The first step of this experimental investigation was to cut two specimens out of the circular DER332/DDS epoxy specimens of Chapter 2; each of the specimens was 5.62mm thick. Both specimens were polished to a high gloss, and a RTD/heater combination was sandwiched between them as shown in Figure 6.1. Silicone grease was used to achieve good contact between the RTD/heater combination and the samples. The back surface of each specimen was in contact with an aluminum block (simulating a constant temperature boundary condition because of its higher conductivity relative to epoxy) to which a thermocouple was attached. The whole assembly of epoxy specimens, RTD/heater, and aluminum blocks was placed in an oven, and the thermocouples and RTD/heater were connected to the data acquisition system described in Chapter 2.



Figure 6.1 Experimental Setup for the Investigation of Optimal One-Dimensional Designs

Two sets of experiments were run to show the effect of optimal heating, and the effect of the duration of the experiment on the optimality of the experiments. Each set consisted of two experiments, and all four experiments were based on the ideas of Chapter 3. The first experiment was an optimal one ending when the heating was turned off (at  $t_h^+=2.5$ ). The second experiment was a non-optimal one with a short heating time ( $t_h^+=0.5$ ). The third experiment was chosen optimal with a duration longer than the end of heating (at  $t_h^+=2.5$ ). Finally, the fourth experiment was non-optimal; it was similar to the third one except for its shorter heating time ( $t_h^+=0.5$ ). The same number of measurements (250) was taken for all four experiments, and for each optimal/non-optimal combination, the same maximum temperature rise was achieved as required by the constraints of the analytical study.

#### **6.3 Experimental Results**

The different experimental parameters corresponding to the four experiments mentioned above are summarized in Table 6.1. The calculated voltage was deduced from the boundary heat flux which was, in turn, calculated from a preselected maximum temperature rise. Table 6.1 shows also the measured heat flux which is calculated from the measured voltage and current, and from the heated surface area. Notice that the column of non-dimensional heating times shows numbers slightly different from the ones indicated above. This difference is due to the round off in the calculated experimental times needed to satisfy the same number of measurements condition.

The parameter estimation program PROP1D (Beck, 1989) described in Chapter 2
exp.	time step (sec)	time heat off (sec)	non- dim. t <sub>h</sub> +	time exp. off (sec)	non- dim cooling time	meas. volt. (V)	meas. curr. (A)	meas. heat flux (W/m <sup>2</sup> )
1	2.2	550	2.8	550	0	17.5	0.176	337.7
2	0.44	110	0.7	110	0	20.3	0.204	452.9
3	2.7	558.9	2.7	675	0.59	22.4	0.223	547.7
4	0.53	109.7	0.7	132.5	0.14	26.4	0.262	758.4

Table 6.1 Experimental Parameters of the Four Experiments Performed for the Investigation of Optimal One-Dimensional Designs

was used to estimate the thermal properties, the corresponding 95% confidence intervals, the sensitivity coefficients, and the residuals. The sensitivity coefficients and residuals are examined now at the heated surface. The magnitudes and relative shapes of the curves of the sensitivity coefficients of the different parameters of interest are indicators of how good a particular experiment is to estimate the parameters (recall Chapter 3). The two sets of sensitivity coefficients for the two sets of experiments described above are shown here in Figures 6.2 and 6.3. The "k" in the labels of the curves stands for thermal conductivity, and the "c" stands for volumetric heat capacity. The curves of Figure 6.2 labeled "k1" and "c1" correspond to the first experiment, while the curves labeled "k3" and "c3" correspond to the third experiment, while the curves labeled "k4" and "c4" correspond to the fourth experiment. The shapes of the optimal thermal conductivity curves ("k1" and "k3") are very similar to those of the curves of Figure 3.3 of Chapter



Figure 6.2 Sensitivity Coefficients for the First Set of Optimal One-Dimensional Experiments



Figure 6.3 Sensitivity Coefficients for the Second Set of Optimal One-Dimensional Experiments

3. The same is true for the volumetric heat capacity curves and those of Figure 3.4.

To be able to compare all four curves ("k1", "c1", "k3", and "c3") to those of Figures 3.3 and 3.4, time is non-dimensionalized using the estimated thermal properties, and with respect to the thickness of the specimens. Furthermore, the sensitivity coefficients of Figures 6.2 and 6.3 (which are really  $k(\partial T/\partial k)$ ) and  $\rho c(\partial T/\partial \rho c)$  as calculated by PROP1D) are non-dimensionalized the same way as the sensitivity coefficients of Chapter 3 given by Equations (3.8a) and (3.9a). The resulting nondimensional sensitivity coefficients are  $(k/q_0L)\times(k(\partial T/\partial k))$  and  $(k/q_0L)\times(\rho c(\partial T/\partial \rho c))$ . The minimum of curve "k1" of Figure 6.2 is

$$X_{k1,min}^{*} = \frac{1}{q_0 L/k} k \left( \frac{\partial T}{\partial k} \right) = \frac{1}{337.5 \times 5.62 \times 10^{-3} / 0.217} \times (-8) = -0.915$$
(6.1)

which occurs at about

$$t_{k1,min}^{+} = \frac{\alpha t}{L^2} = \frac{0.217}{1.35 \times 10^6} \frac{550}{(5.62 \times 10^{-3})^2} = 2.8$$
(6.2)

These two values  $X_{k1,min}^{+}$  and  $t_{k1,min}^{+}$  are compared respectively with -1 and 3 obtained from Figure 3.3. Curve "c1" of Figure 6.2 shows a decrease up to a minimum, then goes to zero. The minimum value is

$$X_{c1,min}^{+} = \frac{1}{q_0 L/k} \rho c \left(\frac{\partial T}{\partial \rho c}\right) = \frac{1}{337.5 \times 5.62 \times 10^{-3}/0.217} \times (-2) = -0.229$$
(6.3)

which occurs at about

$$t_{c1,min}^{+} = \frac{\alpha t}{L^2} = \frac{0.217}{1.35 \times 10^6} \frac{150}{(5.62 \times 10^{-3})^2} = 0.763$$
(6.4)

these two values  $X_{c1,min}^{+}$  and  $t_{c1,min}^{+}$  are compared respectively with -0.3 and 0.5 obtained from Figure 3.4.

The same is done for curves "k3" and "c3" of Figure 6.3 during the heating period (which is the condition for which Figures 3.3 and 3.4 were generated). The minimum value  $X_{k3,min}^{+}$  was -0.921, and occurs at the non-dimensional time  $t_{k3,min}^{+}=2.82$ . The two values  $X_{k3,min}^{+}$  and  $t_{k3,min}^{+}$  are compared with -1 and 3 respectively. The minimum value  $X_{c3,min}^{+}$  was -0.276, and occurs at the non-dimensional time  $t_{c3,min}^{+}=0.719$ . These two values are compared respectively with -0.3 and 0.5 obtained from Figure 3.4. The above similarities indicate that the experimental results were consistent with the theory of Chapter 3. This consistency was not achieved at an earlier stage of the research with the setup described in Chapter 3.

The residuals of the thermocouple at the heated surface of the specimen were examined next. They are shown in Figure 6.4 for all four experiments. The maximum value of each curve was compared with the maximum temperature rise. The largest value of all four residual curves was about 0.15°C; the corresponding maximum temperature rise was 17.13°C. Comparing these two quantities, the residuals are less than 1% of the maximum temperature rise, as opposed to 2.9% using the setup of Chapter 2.

The similarity between the theoretical and experimental sensitivity coefficient curves, as well as the low values of the residuals meant improved thermal properties estimates. These estimates are shown as functions of time in Figures 6.5 and 6.6. Figure 6.5 shows the thermal conductivities corresponding to the four experiments. The conductivity estimate was 0.218 W/m°C for the optimal experiments of Figure 6.5. For the non-optimal experiments, the estimation process does not show any leveling off for some time to a constant value. Figure 6.6 shows the transient volumetric heat capacities



Figure 6.4 Residuals of the Four Experiments Performed to Investigate Optimal One-Dimensional Designs

Thermal Conductivity (W/mK)

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corresponding to the four experiments, and the same convergence/non-convergence behavior noted in Figure 6.5 was true for Figure 6.6.

The estimated thermal properties corresponding to the four experiments of Section 6.2 are summarized in Table 6.2 along with their confidence intervals. The confidence regions of the four experiments are shown in Figure 6.7. The small squares formed by the open circles (labeled 5) and triangles (labeled 3) (there are in fact four triangles: two on two of two) correspond to the optimal experiments while the large squares formed by the closed rectangles (labeled 6) and diamonds (labeled 7) correspond to the non-optimal experiments. 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 (exp 1) was 16.32 (W/m°C)(J/m<sup>3</sup>°C) as opposed to the non-optimal one (exp 2) of 2883.58. The area of the confidence region corresponding to the second optimal experiment (exp 3) was 11.43 as opposed to the non-optimal one (exp 4) 2291.53. Much better experiments (reflected by smaller confidence region areas) were therefore achieved when the dimensionless heating time was 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.

In conclusion, the setup developed by Garnier, Delaunay, and Beck (1991) improved the quality of the experimental results to the point where these results and the theory of Chapter 3 on the design of optimal experiments are the same. Furthermore, the transient experiments demonstrated that optimal experiments produce substantially more accurate parameter estimates than non-optimal experiments. This is exactly what the theoretical investigation of Chapter 3 showed.

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Table 6.2 Comparison of the Results of the Four Optimal One-Dimensional Experiments

exp.	time step (sec)	time heat on (sec)	time heat off (sec)	time exp. off (sec)	thermal cond. (W/m°C)	volumetric heat capacity (J/m <sup>30</sup> C)	rms
1	2.2	22	550	550	0.217 ± 0.0004	(1.35±0.01) ×10 <sup>6</sup>	0.021
2	4.4	4.4	110	110	0.2388 ± 0.0101	(1.19±0.07) ×10 <sup>6</sup>	0.041
3	2.7	27	558.9	675	0.223 ± 0.0003	(1.35±0.01) ×10 <sup>6</sup>	0.028
4	5.3	5.3	109.7	132.5	0.2365 ± 0.0089	(1.19±0.06) ×10 <sup>6</sup>	0.061

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Figure 6.7 Confidence Regions Corresponding to the Experimental Investigation of Optimal One-Dimensional Experiments

## **CHAPTER 7**

## **RESULTS AND CONCLUSIONS**

The goal of this research was to find and investigate new ways of improving the estimation of the thermal properties of composite materials. To reach this goal, analytical and experimental approaches were followed.

The first way of improving the estimation of the thermal properties related to a new experimental paradigm. This new paradigm consisted of two series of thermal properties estimation experiments. Each series consisted of four independent experiments. In the first series, the imposed heat flux was made large to cover the whole temperature range of interest (20°C to 100°C) in each experiment. In the second series, the same temperature range was covered but each of the four experiments covered a small portion of it. It was found that the temperature residuals were large, and led to large confidence regions. The large size of the confidence regions masked the outcome of the paradigm, and it was necessary to look for other ways of improving the transient experiments. One way was to design the experiments so that they are optimal; the other way was to quantify the thermocouple measurement error that led to the large residuals.

The second approach of improving the estimation of thermal properties was to

design the experiments so that they are optimal. This design of optimal experiments was done analytically, then verified experimentally. It was based on maximizing the determinant of the product of the sensitivity matrix and its transpose, which is the well known D optimality criterion. Different geometries were examined, and different parameters were of interest. First, a one-dimensional geometry was considered. It was found to have an optimal dimensionless heating time of 2.25, and a dimensionless "cooling" time of 0.73, or a total dimensionless optimal experimental duration of about 3. The choice of the optimal dimensionless heating time was found to be flexible (between 1.5 and 3.5). The optimal location of a sensor for this one-dimensional geometry was found to be at the heated surface. The temperature boundary condition used with this geometry proved to be superior to an insulated one. Next, a semi-infinite geometry was considered. The optimal dimensionless heating time was 1.5, the "cooling" time was 0.22, and the total optimal experimental duration was about 1.7. This geometry, unlike the one-dimensional one, showed less flexibility in the choice of the heating duration. The finite duration of the heat flux (with measurements lasting a dimensionless time 0.22 after the end of the heating) for this semi-infinite geometry was found better than a heat flux lasting through the whole experiment.

A two-dimensional geometry was also analyzed in the same context of designing optimal experiments. For this configuration, the optimal experiment was found to occur at steady state instead of a finite time. The optimal placement of two thermocouples was found to be at the two edges of the heat flux surface. Finally, some global design curves were obtained for different ratios of the area of the heated potion of the heat flux surface to that of the whole heat flux surface. The recommended curve corresponded to  $a_1/a=0.75$  for (a/b)sqrt $(k_y/k_x)=2.25$ .

The third and last technique used to improve the estimation of thermal properties was to characterize the error due to the embedded thermocouples. This error was quantified using the USE method, which was compared to the two popular numerical methods: the FE method and the FD method. The USE method gave more insight into the problem, required considerably less computational time for the estimation of the small quantity thermocouple disturbance, and gave accurate results. An experiment was implemented to verify the results of the USE method. The experiment showed higher dependence of the temperature disturbance on errors in the thermocouple location than errors in the thermal properties.

Finally, four experiments were run using a technique developed by Garnier, Delaunay, and Beck in 1991; the setup uses only surface thermocouples and resistance thermometers. The goal of the experiments was to verify the one-dimensional finite geometry optimal experiments' results. The experimental results were found similar to the analytical ones.

The following results and conclusions are drawn from this research:

1. The use of embedded thermocouples to investigate a new experimental paradigm led to large residuals and masked any possible improvements expected by the paradigm.

2. For the finite one-dimensional X21B50T0 case, the optimal heating time was 2.25, and the optimal total experimental time was 3.

3. The choice of the optimal heating time for the X21B50T0 case was flexible, between

1.5 and 3.5.

4. The heated surface was the optimal location to place a sensor at for the X21B50T0 case.

5. For the finite one-dimensional case, a constant temperature on one boundary with a finite duration constant heat flux on the opposite boundary led to better experiments than an insulated boundary with a the same finite duration constant heat flux on the opposite boundary.

6. For the semi-infinite X20B5T0 case, the optimal heating time was 1.5, and the optimal total experimental time was 1.7.

7. The choice of the optimal heating time for the X20B5T0 case was less flexible than for the X21B50T0 case.

8. For the two-dimensional X22B00Y12B0x5T0 case, the optimal experiment was attained at steady state.

9. The optimal placement of two thermocouples for the two-dimensional X22B00Y12B0x5T0 case was at the two edges of the heat flux surface.

10. The optimal experiment for the two-dimensional X22B00Y12B0x5T0 case occurred for  $a_1/a=0.75$  and  $(a/b)sqrt(k_x/k_x)=2.25$ .

11. The USE method permitted more insight than the FE or FD method in calculating the thermocouple-induced errors.

12. The USE method used less computational time than the FE or FD methods in calculating the thermocouple-induced errors.

13. The USE method gave more accurate results than the FE or FD methods.

14. The thermocouple location was the experimental parameter with most effect on the thermocouple-induced disturbance.

15. An alternative technique avoiding embedded thermocouples made it possible to check the analytical results of the design of optimal experiments.

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