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# Joint experimental/computational techniques to measure thermal properties of solids

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**Abstract.** Experimental techniques to measure the thermal properties of solids are discussed. Experimental measurements and parameter estimation techniques are used to estimate the thermal properties. The importance of experimental design is emphasized, and criteria that give insight to the parameter estimation process and experiment are cited. A code-to-code communication technique is presented, which allows standard stand-alone direct solution programs to be linked with parameter estimation codes without modification. This method is called reusable interface technology (RIT). Aspects of this computational technique are discussed. The experimental/computational techniques are demonstrated with an experiment to estimate the thermal properties of an orthotropic carbon–carbon composite material. Two-dimensional, transient temperature and heat flux data are used to estimate two components of thermal conductivity (in the principal directions of the material) and the volumetric heat capacity.

## Nomenclature

| | |
|---|---|
| $\boldsymbol{b}$ | parameter vector |
| $\hat{\boldsymbol{b}}$ | estimated parameter vector |
| $C$ | specific heat (J kg$^{-1}$ °C$^{-1}$) |
| $k$ | thermal conductivity (W m$^{-1}$ °C$^{-1}$) |
| $L$ | length (m) |
| $q$ | heat flux (W m$^{-2}$) |
| $S$ | sum-of-squares function |
| $T$ | temperature (°C) |
| $\mathbf{W}$ | weighting matrix for sensors (°C$^{-2}$) |
| $X$, $\mathbf{X}$ | sensitivity coefficient, matrix |
| $\bar{X}$, $\bar{\mathbf{X}}$ | scaled sensitivity coefficient, matrix (°C) |
| $Y$, $\boldsymbol{Y}$ | measured temperature, vector (°C) |
| $\alpha$ | thermal diffusivity (m$^2$ s$^{-1}$) |
| $\Delta$ | optimality criteria |
| $\rho$ | density (kg m$^{-3}$) |
| $\Omega$ | electrical resistance ($\Omega$) |

### Subscripts

| | |
|---|---|
| $cc$ | carbon–carbon |
| $x$ | direction parallel to the fibre direction |
| $y$ | direction normal to the fibre direction |

## 1. Introduction

Frequently in thermal engineering the modelling of energy transfer in a solid requires coefficients or parameters in the describing equation(s). Examples of these parameters are thermal conductivity, emittance, convection coefficient, specific heat, and density. These parameters relate the microscopic nature of the solid to the observed macroscopic energy transfer. Because the relationship between microscopic and macroscopic energy transfer is quite complex, empirical results are typically used to determine the relationship. In this paper we restrict our focus to the thermal properties associated with solids, but the techniques and concepts are applicable to a diverse range of physics.

The techniques use an experiment which records an easily measured quantity, such as temperature and/or electrical power, to determine desired (unknown) parameters which cannot be easily measured. Measurements from the experiment are related to the parameters through a computational model. The model can be an algebraic equation, an ordinary differential equation, or a partial differential equation. Problems that use the interaction of measurements (experiments) and a describing model (analysis) to infer *other* aspects of the model are generally called inverse problems. Two main distinctions of inverse problems are parameter estimation and function estimation. In parameter estimation, a small number of unknowns (typically less than five) are estimated; the parameters are usually coefficients in the describing equations. Function estimation, however, determines functions represented by many unknowns, possibly several thousand, which may vary spatially and with time. Estimating a function tends to be ill-posed and very sensitive to measurement errors (Beck *et al* 1985). The functions that are estimated generally appear as boundary conditions in the describing equations. The function estimation and parameter estimation problems have similar attributes and are closely related. In this paper the focus is restricted to parameter estimation. Books by Kurpisz and Nowak (1995), Alifanov (1994), and Beck *et al* (1985) discuss several approaches for function estimation, or ill-posed problems.

Several experimental approaches exist for measuring the thermal properties of solids. However, this paper is not intended to be a comprehensive survey of experimental techniques to measure thermal properties—two techniques are selected to illustrate accepted experimental approaches to the problem and contrast the techniques with the method presented in this paper. An early method, called the guarded hot plate technique (ASTM 1997), imposes a measured heat flux across a sample of known thickness. The thermal conductivity is computed from a discrete approximation of Fourier's law

$$k = qL/\Delta T. \tag{1}$$

In equation (1) $q$ is the measured heat flux, $L$ is the sample thickness, and $\Delta T$ is the temperature difference across the sample. Another experimental approach is known as the flash diffusivity method (Parker *et al* 1961). In the flash diffusivity method a short duration burst of energy, typically from a laser, heats the surface of a thin specimen approximately 2 cm in diameter. Transient temperature is measured on the face of the specimen opposite the heating. Thermal diffusivity is computed as (Parker *et al* 1961)

$$\alpha = \frac{k}{\rho C} = \frac{1.38L^2}{\pi^2 t_{1/2}} \tag{2}$$

where $L$ is the specimen thickness and $t_{1/2}$ is the time required for the temperature at the back surface to reach one-half its maximum, the so called half-time (other time intervals may also be used, e.g. $t_{1/3}$, $t_{1/4}$).

A common aspect for the two experimental approaches discussed is the simple basis of the analysis. An algebraic expression is evaluated to compute the unknown parameter. In order to arrive at a simple algebraic data reduction equation, a number of assumptions must be made. The two experimental cases discussed assume that heat flow is one dimensional, heat losses to the environment are negligible, the material is isotropic, and thermal properties are independent of temperature. Often it is difficult to satisfy these assumptions. Furthermore, the estimated parameters are more accurate when these effects, which may be more influential than expected, are included in the analysis. The methodology presented here can relieve the necessity of making the above assumptions.

The main advantage of using the referenced analysis techniques is that they are simple. There are several disadvantages to using analysis techniques that simplify the process to a 'plug-and-chug' operation, such as that shown for the guarded hot plate and flash diffusivity methods, as follows.

1. The (heat conduction) model equation, in which the estimated parameters are ultimately used, is not necessarily satisfied or checked.
2. Typically few measurements are used, in some cases only one measurement.
3. The thermal properties $k$ and $\rho C$ cannot be obtained independently in all experimental configurations.
4. Generalizing these methods is difficult, if not impossible. For example, it is not apparent how to extend the methods for the multi-dimensional case, a more complex model including heat losses, or the nonlinear problem with temperature dependent thermal properties.
5. Using statistics to quantify the accuracy of the estimated parameters is not easily done.
6. The analysis does not provide insight to the adequacy of the experiment or insight to improve the experiment.

Parameter estimation techniques, which are applicable to the previously described experiments, do not possess the limitations of these other simplified analysis methods. Beck (1996) discusses applying parameter estimation techniques to the flash diffusivity experiment. Beck estimates parameter groups describing the heat input and heat losses, in addition to the thermal diffusivity, and demonstrates that the model can be refined using parameter estimation results to improve the accuracy of the estimated thermal diffusivity. In this process, not only is the analysis improved, but parameter estimation also helps to better understand and indicate improvements in the experiment. Simply using equation (2) to calculate the thermal diffusivity does not permit such an analysis or provide (engineering) insight. Hence, the more general method of applying parameter estimation techniques is a preferred approach.

The theory of the experimental techniques in this paper, using electric heaters for estimating the thermal properties, are detailed in a book by Beck and Arnold (1977, see ch 7). A consequence of using electric heaters (and heat flux boundary conditions) is that typically both thermal conductivity and volumetric heat capacity can be estimated simultaneously.

Other methods with known heat flux and transient temperature measurements are described by Beck *et al* (1991), Scott and Beck (1992a, b), and Beck and Osman (1991). The first of these papers uses an internal heat flux transducer. The papers by Scott and Beck (1992a, b) relate to composite materials during and after curing. A method to sequentially estimate thermal properties by mathematically connecting a series of discrete experiments, with various temperature ranges, is presented by Beck and Osman (1991). Loh and Beck (1991) give results for experimentally determining two components of the thermal conductivity for a carbon–carbon composite. Papers by Garnier *et al* (1992, 1993) describe a method for estimating thermal properties without requiring temperature sensors inside the specimen(s). Also relevant is the study of optimal experiments, which are discussed in chapter 8 of Beck and Arnold (1977) and in the paper by Taktak *et al* (1993).

One advantage of an analysis such as that shown by equations (1) and (2) is that the relationships to compute the parameters are simple. However, several disadvantages were previously noted. The incorporation of parameter estimation techniques requires solving the model (heat conduction) equation, which for realistic experimental configurations requires a numerical solution. Hence to implement a parameter estimation solution, a thermal analysis code (such as finite element, finite difference, finite control volume, or boundary element) for solving the direct problem is coupled with a parameter estimation code. (A closed form analytical solution such as a Fourier series can also be used.) A general purpose thermal analysis code allows for multiple materials, arbitrary geometries, and convective and radiative heat losses, and is needed to model complex processes. The code is usually coupled with the parameter estimation techniques in an invasive manner and significant effort is required to modify the analysis code to link it with parameter estimation techniques (see Osman and Beck 1989). Such an implementation requires detailed knowledge of the thermal analysis code. An alternative to this process for parameter estimation uses reusable interface technology (RIT) (Blackwell and Eldred 1997).

In RIT a complex thermal analysis code is externally coupled to a parameter estimation code. In the past, thermal analysis codes were modified, in most cases converted to subroutines, to link them with the parameter estimation code to form a new parameter estimation/thermal analysis code. In this previous linking process, the connection between the original developers of the thermal analysis code and the new parameter estimation/thermal analysis code may be severed. Consequently, future enhancements to the original thermal analysis code may be difficult and time consuming to implement in the combined code. Also, it is possible to link commercial thermal analysis software (for which a source code is not available) to the parameter estimation code by means of RIT.

The RIT approach is to isolate the interface mechanisms from the optimization and analysis codes such that both codes (thermal analysis and parameter estimation) are allowed to evolve independently. That is, rather than modifying the source code of the thermal analysis package to convert it into a subroutine, one should allow the optimization and analysis systems to coexist on their own terms and build reusable communication links between them. Moreover, if these isolated interface mechanisms are built in a general, reusable manner, then the amount of work required to update the interfaces for new code versions can be minimized and sometimes eliminated entirely. This approach is the cornerstone of RIT.

In this paper, the use of parameter estimation techniques are demonstrated to estimate the thermal properties of a carbon–carbon composite material. Emphasis on parameter estimation results to understand and provide insight into the estimation procedure and help improve the experiment are discussed. RIT is shown to reduce the effort required to analyse experiments that require a complex analysis and has the potential to change the parameter estimation computational procedure.

Parameter estimation techniques are discussed in the next section. Aspects of RIT are addressed in section 3. An experiment to estimate the thermal properties of a carbon–carbon composite demonstrates the application of parameter estimation and RIT in section 4. Results are discussed in section 5 and the final section provides a summary and conclusions.

## 2. Parameter estimation techniques

In this section parameter estimation techniques are discussed as they apply to the estimation of thermal properties in the heat conduction equation. The techniques are not restricted to estimating thermal properties. These ideas are applicable to any process for which measurements are available and the parameters are related to the experimental measurements through a model equation. In applying parameter estimation techniques it is important that errors associated with the measurements are understood (see Beck and Arnold (1977) for a discussion of measurement errors).

Techniques to estimate parameters in a model equation are also detailed in Beck and Arnold (1977, see ch 6 and 7). The basic process involves minimizing a sum-of-squares function

$$S = (\boldsymbol{Y} - \hat{\boldsymbol{T}})^{\mathrm{T}} \mathbf{W} (\boldsymbol{Y} - \hat{\boldsymbol{T}}) \tag{3}$$

where $\boldsymbol{Y}$ and $\hat{\boldsymbol{T}}$ are vectors of the measured and calculated temperatures and $\mathbf{W}$ is a weighting matrix (typically the identity matrix). To determine the thermal properties the function $S$ is minimized with respect to the thermal properties, i.e. $b_1 = (\rho C)_{cc}$, $b_2 = k_{y,cc}$ and $b_3 = k_{x,cc}$. This is accomplished by setting the first derivative of $S$ with respect to each parameter equal to zero, and solving for the estimated parameters ($\hat{\boldsymbol{b}}$). The derivatives of $S$ with respect to the parameters involves sensitivity coefficients, defined as

$$\mathbf{X} = [\boldsymbol{X}_{b_1}, \boldsymbol{X}_{b_2}, \ldots, \boldsymbol{X}_{b_p}] \tag{4}$$

$$\boldsymbol{X}_{b_i} = \partial \boldsymbol{T} / \partial b_i . \tag{5}$$

The sensitivity coefficients were computed using a finite difference approximation on the temperature output of the thermal analysis code COYOTE (Gartling and Hogan 1994). A relative finite difference step size of 0.005 was used. The minimization of $S$ was performed using the DAKOTA software (Eldred *et al* 1996a, b). DAKOTA in turn uses the commercially available optimization package DOT (Vanderplaats 1995), along with other optimization algorithms. The specific optimization method used for the work was the BFGS method (Broyden 1970, Fletcher 1970, Goldfarb 1970, Shanno 1970), which is gradient based.

The sensitivity coefficients can provide considerable insight into the estimation problem and aid in the design of the experiment to obtain estimates with optimum accuracy (Beck and Arnold 1977, ch 8). One criteria for an ‘optimal’ experiment that is valid for additive, zero mean normal errors in $\boldsymbol{Y}$, and errorless independent variables, is to maximize

$$\Delta \equiv |\mathbf{X}^{\mathrm{T}} \mathbf{X}|. \tag{6}$$

This criteria is appropriate because it corresponds to minimizing the volume of the confidence region for the estimated parameters (Beck and Arnold 1977). By studying the experimental design prior to conducting experiments, such that equation (6) is maximized, maximum information

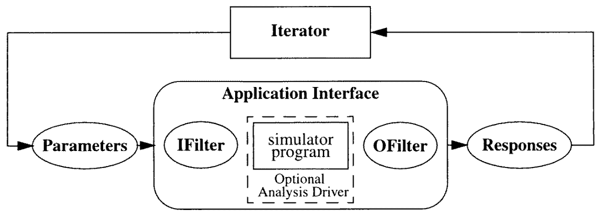


**Figure 1.** Reusable interface technology—the Application Interface.

is available from an experiment. In addition to the criteria proposed in equation (6), a constraint requiring a fixed number of observations and/or the same temperature rise, may be required to provide consistency in comparing experimental designs with multiple sensors. Additional investigations on experimental design are concerned with optimal placement of sensors (Fadale *et al* 1995a) and uncertainty information (Fadale *et al* 1995b, Emery and Fadale, 1996).

To implement the parameter estimation techniques a code-to-code communication technique is used; a complex thermal analysis code is linked with a parameter estimation code, completely external to both codes. The concept is called RIT and aspects of this method are now discussed.

## 3. Reusable interface technology (RIT)

### 3.1. Overview of RIT

In order for the thermal analysis code development to be independent of the optimization code development, it is desirable to employ a flexible, reusable communication mechanism which does not require modification of either the thermal analysis or the optimization packages. This approach will ensure that the two codes are always compatible and allows the codes to be developed independently. The DAKOTA iterator toolkit (Eldred *et al* 1996a, b, DAKOTA 1997) is the software system which implements the RIT concept.

In DAKOTA, interfaces are implemented in terms of communication protocols, such as CORBA, MPI, and file-based I/O, and specialized function evaluation interfaces, such as the Application Interface, the Test Function Interface, the Approximation Interface, and the Multidisciplinary Optimization Interface. The simplest example of a DAKOTA interface is the Application Interface, which utilizes system calls and file-based I/O for process spawning and data communication respectively. The Application Interface approach is sufficient for this parameter estimation application since the thermal analysis and optimization programs are executed on the same machine.

A schematic diagram of the Application Interface is given in figure 1. The Application Interface isolates application specifics from an iterator method by providing a generic interface for the mapping of a set of parameters (e.g. a vector of design variables) into a set of responses (e.g. an objective function, constraints, and/or sensitivities). Housed within the Application Interface are three pieces of software. The input filter program (IFilter) provides a communication link which transforms the set of input parameters into input files for the simulator program. The simulator program reads the input files and generates results in the form of output files or databases. Finally, the output filter program (OFilter) provides another communication link through the recovery of data from the output files and the computation of the desired response data set. Generally, the application developer (e.g. the thermal parameter estimation investigator) will develop these input and output filters for the particular analysis code of interest. If care is taken to develop quality filter programs, then libraries of input and output filters can be built up over time, thereby maximizing reuse and minimizing duplication effort. Moreover, the amount of work required to update the filter programs for new analysis and optimization package versions can be minimized and sometimes eliminated entirely.

This mapping of parameters to responses provides generic information to the iterator/estimator, and the application and implementation specifics are hidden. This encapsulation of application-related complexity is an essential part of providing a flexible and extensible capability for systems analysis in general, and thermal parameter estimation in particular.

### 3.2. Details of RIT

The schematic of the information flow process given in figure 1 is a simplification of the actual process. Some additional details are presented here. All the calculations presented are run on UNIX$^{TM}$ work stations. A shell script is used to run the various codes in sequence. The output filter is a FORTRAN code that reads data files containing the experimental temperatures and the computed temperatures, computes the mean square error $S$, and writes $S$ to a file to be read by the iterator.

The input filter is somewhat more complicated. The input to a large-scale finite element, finite difference, or finite volume code has a certain amount of structure to it. For each simulation, this input deck has to be rebuilt automatically without human intervention. This involves inserting the latest parameter values into the appropriate location in the input data file. While the UNIX$^{TM}$ utilities *sed* and *awk* could be used to perform some of these operations, we chose instead to use the algebraic preprocessor APREPRO (Sjaardema 1992) which allows one to build an input deck in symbolic form. For example, if the unknown thermal conductivity in the $x$-direction is given the symbolic name {cond_x} in the input file and a second file containing the line {cond_x = 59.46} is read by APREPRO, the output from APREPRO is a line containing the number 59.46. In effect, APREPRO replaces an alpha string by a numeric string. This code was originally developed to aid in the preparation of multiple input data files for parameter studies, and was ideally suited for the task at hand. The output from the iterator is a list of the latest parameters along with an alpha-numeric identifier; a simple FORTRAN code was written to translate the parameter list into a string that can be read by APREPRO.

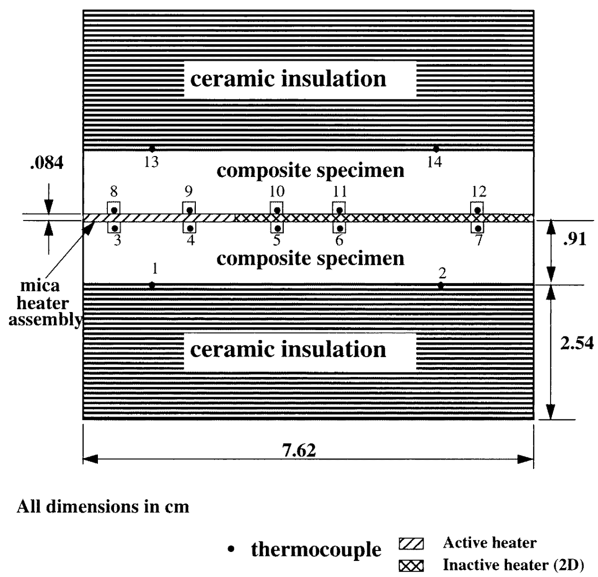


**Figure 2.** Schematic of experimental set-up used for estimating thermal properties of a carbon–carbon composite. For one-dimensional experiments all heaters are activated. Two-dimensional experiments activate only one heater.

## 4. Experimental aspects

To demonstrate the application of parameter estimation techniques and RIT, an experiment to estimate the thermal properties of a carbon–carbon composite is presented. A sketch of the experimental set-up is shown in figure 2. It consist of two nominally identical carbon–carbon specimens (7.62 cm × 7.62 cm × 0.914 cm) and ceramic insulations (7.62 cm × 7.62 cm × 2.54 cm, Zircar Products Inc., Florida, NY) with a mica heater assembly (Thermal Circuits, Inc., Salem, MA, $\Omega(T_{room}) = 33\ \Omega$) located between the identical halves. Five thermocouples (Type E, 0.254 mm nominal wire diameter) are embedded on the surface of each carbon–carbon specimen at the heater/specimen interface. The thermocouples (insulation removed) are attached with electrically insulating high-temperature cement into grooves (0.38 mm by 0.46 mm) that extend the length of the specimen. Two thermocouples are located at each interface of the carbon–carbon specimen and the ceramic insulation. The entire set-up is mounted between two 3.18 mm thick aluminum plates that are connected with threaded rods and hold the numerous layers firmly in place; the apparatus is placed in a furnace which allows variation of the initial temperature. Further details of the experimental procedure are discussed in Ulbrich *et al* (1994).

The experiments are conducted and processed using a 12-bit data acquisition system (National Instruments) with a 486 PC. The system provides accurate data acquisition with minimum sampling intervals in the microsecond range. Two eight-channel data acquisition boards are linked providing sixteen channels of data acquisition. The system controls and acquires the power (voltage and current) delivered to the heaters and acquires the thermocouple voltages. The current is acquired by measuring the voltage across a known resistance. The heat flux is calculated from the power measurements assuming the heating is uniform over the heating area (7.62 cm × 7.62 cm) and divides equally to the symmetric experimental halves.

**Table 1.** Sensor locations in experimental apparatus.

| Sensor number | Location $x$ (cm) | Location $y$ (cm) |
|---|---|---|
| 3, 8 | 0.89 | 0 |
| 4, 9 | 1.91 | 0 |
| 5, 10 | 3.18 | 0 |
| 6, 11 | 4.45 | 0 |
| 7, 12 | 6.73 | 0 |
| 1, 13 | 1.27 | 0.91($L_y$) |
| 2, 14 | 6.35 | 0.91($L_y$) |

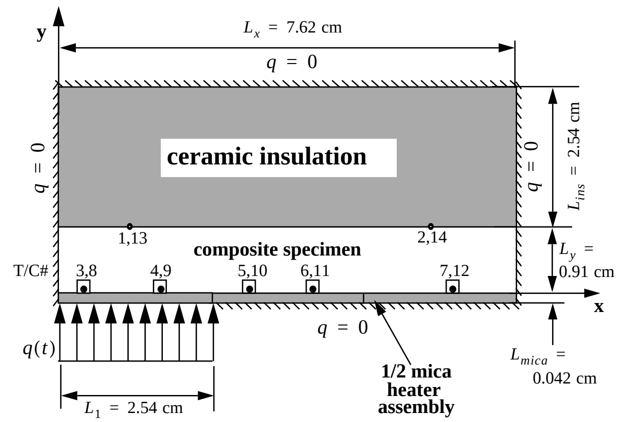


**Figure 3.** Heat transfer model for two-dimensional experiments.

The measured temperatures are averaged on opposite sides of the heater assembly to determine the temperature at each location. The locations of the thermocouples are shown in figure 2 and given in table 1. The sensors that are embedded in the specimen are assumed to measure the temperature at the surface of the specimen. Since thermal conductivity normal to the fibre is much greater than the conductivity of the insulation ($k_{y,cc} \gg k_{ins}$), small temperature gradients exist in the specimen near the specimen/insulation interface. The non-embedded sensors are assumed to measure the temperature at the rear of the carbon–carbon specimen.

The thermal model for the two-dimensional experiment is shown in figure 3. All outer surfaces are assumed to be adiabatic, except for the surface where the energy is introduced by the heater. The energy to the heater is assumed to divide equally between the two halves and emanate from the middle of the heater assembly ($y = -0.042$ cm). The adequacy of the assumed adiabatic outer surfaces for the model can be verified by a comparison of heat losses by natural convection with the anticipated applied heat flux from the heater assembly. Since a temperature rise of 20 to 25 °C above ambient is expected for a typical experiment, the heat loss is mainly due to natural convection ($h \approx 4$ W m$^{-2}$ °C). These losses

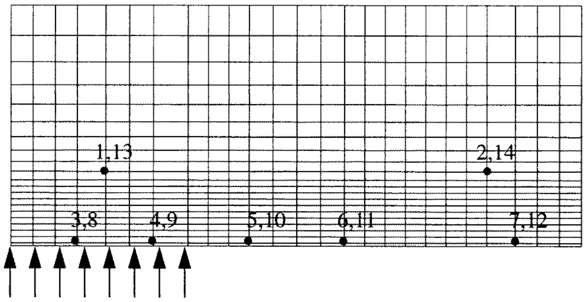


**Figure 4.** Mesh 1, the coarsest mesh (525 elements), showing sensor placement.

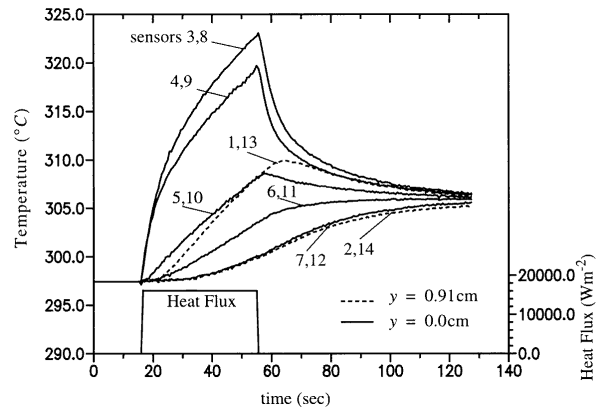


**Figure 5.** Experimental data for the two-dimensional case, see table 1 for sensor locations.

are negligible ($q_{conv} \approx 100$ W m$^{-2}$) in comparison to the applied heat flux, which was 17 000 W m$^{-2}$ for the experiment analysed. Hence, adiabatic boundary conditions on the outer surfaces are a good approximation.

Numerical issues are more important for the inverse (parameter estimation) analysis than they may be for a standard direct solution. Because inverse problems are more computationally intensive, numerical issues such as the mesh size and time step selected for the numerical method can greatly influence the amount of time required to obtain a solution and the accuracy of this solution. The coarsest mesh used for this analysis contains 525 (quadrilateral) elements and is shown in figure 4. Twenty-five elements are along the 7.62 cm surface ($x$-direction) for all materials. There is one element across the mica heater assembly and ten elements across the carbon–carbon specimen and ceramic insulation ($y$-direction). The computational time step chosen was 0.32 s, which is half the experimental time step. Some results from other refined meshes are also presented.

## 5. Results and discussion

### 5.1. Introduction

In the thermal model (figures 3 and 4), in addition to the carbon–carbon material, the mica heater and ceramic insulation are present. To determine the properties of the carbon–carbon specimen, the mica heater and ceramic insulation are included in the model so their thermal properties must be known or estimated. Neglecting the mica heater in the model is not appropriate because the contact resistance results in a large temperature drop between the heater and carbon–carbon specimen. If the heater is neglected, the carbon–carbon properties will incorrectly reflect this effect. Also, including the heat loss to the insulation, instead of assuming a perfect insulated condition (at the specimen/insulation interface), increases the accuracy of the properties estimated for the carbon–carbon. If known (tabulated or published) properties are used for these materials (mica and insulation), several problems arise. First, thermal properties are typically not known very accurately. Second, contact resistance between adjacent layers is typically not negligible and must be considered. Third, the ceramic insulation was sprayed with a rigidizing material, possibly changing its thermal properties. By experimentally estimating the effective properties of these materials, these problems are less influential in the estimated properties of the carbon–carbon.

This approach requires additional experimental work, however. The experimental conditions (length of experiment and heating duration) to determine the properties of the mica and insulation are quite different from the conditions necessary to estimate the properties of the carbon–carbon. Separate series of tests are performed, one set with the carbon–carbon specimen removed, to determine the properties of the insulation and mica heater assembly. These are effective properties, which will account for any contact resistance due to imperfect contacts between the different material layers. The contact resistance may vary, however, when the set-up is reconfigured to test the carbon–carbon composite. Therefore, to determine the properties of the carbon–carbon composite, a short duration experiment is conducted to characterize the effective thermal properties of the mica heater and contact resistance. The short duration experiment is conducted such that the thermal properties of the mica heater, including the contact resistance, are important in the thermal model, but the properties of the carbon–carbon are not important. The importance of the materials in the model is quantified by the sensitivity coefficients (discussed in section 2). A duration is selected that produces results that are sensitive to the properties of the mica, but relatively insensitive to the properties of the carbon–carbon. A discussion of the design and sensitivity coefficients for an experiment to estimate effective properties of the mica heater are given in Dowding *et al* (1998).

The data analysed in this paper are part of a larger study to determine the temperature dependent thermal properties of a carbon–carbon composite. Several experiments over a temperature range of 500 °C with one- and two-dimensional heat flows were analysed to estimate the thermal properties. Two-dimensional results are supported by independent one-dimensional results (see Dowding *et al* (1995, 1996) for the complete analysis of one- and two-dimensional experiments respectively). Only one of the two-dimensional experiments is presented in this paper.

### 5.2. Experimental data

Experimental data for a two-dimensional test that started at an initial temperature of 297 °C are shown in figure 5. A sample interval of 0.64 s is used to acquire data for this experiment. The heating begins at approximately 16 s and ends at approximately 56 s. The complexity of this experiment is that volumetric heat capacity and two components of thermal conductivity are simultaneously estimated. The experimental conditions must be selected to provide information about all three parameters. An alternative is to conduct a series of experiments, each experiment providing information on one (or more) particular parameter. Then the different experiments are analysed in a sequential manner. Beck and Osman (1991) used such a procedure to estimate temperature dependent thermal properties. For this model a single experiment provides adequate information on all three thermal properties and a sequential procedure is not required.

The effect of the orthotropic thermal conductivity can be seen by comparing (in figure 5) the temperature rise for sensors (5, 10) at $x = 3.81$ cm on the heated surface and sensors (1, 13) at $x = 1.27$ cm on the insulated surface. The larger thermal conductivity in the $x$-direction results in a nearly instantaneous response at sensors (5, 10) on the heated surface, while sensors (1, 13) on the insulated surface have approximately a four second time delay before responding. This delay exists even though the sensors are approximately the same distance from the active heater (~10% difference, 0.835 and 0.914 cm from the sensor on the heated surface and insulated surface respectively).

The temperature data are acquired after the heating has ended. Continuing to acquire data after stopping the heat flux results in better estimates because the sensitivity coefficients change character after heating stops. These effects result in a more accurate estimation of multiple thermal properties based on the criteria 'D-optimality with constraints' (Beck and Arnold 1977, p 459). Possible heat losses in the experimental set-up can also be monitored with these data, although significant losses do not appear in this experiment, since all temperature sensors converge to a constant.

### 5.3. Parameter estimation

Using the experimental temperatures given in figure 5 and Mesh 1 given in figure 4, the thermal parameters of the carbon–carbon composite were estimated using the DAKOTA/COYOTE (Eldred *et al* 1996a, b) code combination. The sample rate for the experimental data was 0.64 s; the time step for the Mesh 1 simulations was 0.32 s. A fully implicit time integrator with a lumped capacitance matrix was utilized in COYOTE (Gartling and Hogan 1994), a Galerkin finite element thermal analysis code. The resulting parameter estimates are given in table 2. Five iterations with 30 function evaluations were required for convergence of the iterative process (for Mesh 1). The convergence criterion used was that the relative change in the sum-of-squares function must be less than $10^{-5}$ for two successive iterations. A relative finite difference step

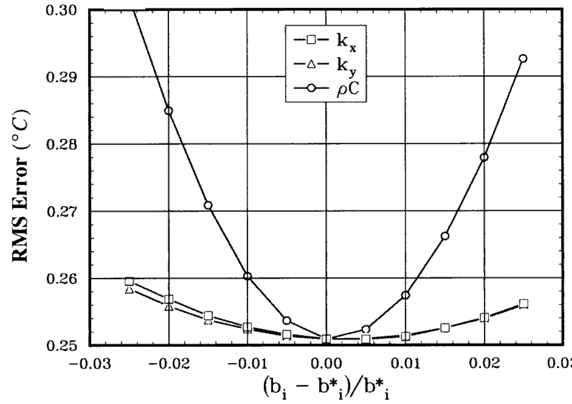


**Figure 6.** Root mean square (RMS) error in the vicinity of the minimum; $b_i^*$ is the converged value of parameter $b_i$. The relative finite difference step size was 0.005.

size of 0.005 was used for the calculation of the sensitivity coefficients.

Ideally, parameter estimation should be performed on grid converged numerical results. In practice, there will always be some numerical errors present. In order to investigate the effect of grid convergence errors, the mesh was refined both spatially and temporally. Since a rectangular mesh was used, it was easy to double the number of elements in each coordinate direction; this gives a factor of four increase in the total number of elements. Since a first-order time integration scheme was used, the time step for Mesh 2 (0.08 s) was a factor of four smaller than that for Mesh 1 (0.32 s). The parameter estimation results for Mesh 2 are also given in table 2, along with the per cent difference between the two results. The Mesh 1 parameter estimation results are different by approximately 1%. The variation in the estimated parameters between Mesh 1 and Mesh 2 is not significant. The difference between the two meshes is of the order of the computed confidence intervals, modelling measurement errors only in temperature, from a past analysis of this experiment (Dowding 1997). The estimated properties and confidence intervals from this previous investigation are shown in the final row of table 2. A more accurate indication of the uncertainty accounts for errors in all measured experimental quantities. A quantification of the experimental uncertainty is also given near the bottom of table 2.

It is advisable to look at the shape of the objective function in the vicinity of the minimum. The DAKOTA software has a convenient option to perform such a parameter study. Figure 6 presents the root mean square (RMS) error as a function of normalized parameter values; at the converged value, the normalized parameter values are zero. All three parameters have the classical bowl shape in the vicinity of the minimum. Both $k_x$ and $k_y$ have similar shapes, indicating that they both have approximately the same sensitivity in the experiment. The $\rho C$ curve is much steeper, indicating that the estimated volumetric heat capacity is known with greater certainty than is $k_x$ or $k_y$. Additional insight into the sensitivity of the experiment can be gained by looking at the sensitivity coefficients; this is done later.

**Table 2.** Summary of estimated parameters.

| | $k_{x,cc}$ (W m$^{-1}$ °C$^{-1}$) | $k_{y,cc}$ (W m$^{-1}$ °C$^{-1}$) | $(\rho C)_{cc}$ (J m$^{-3}$ °C$^{-1}$) |
|---|---|---|---|
| Mesh 1, 525 elements[a] | 59.46 | 5.011 | $2.356 \times 10^6$ |
| Mesh 2, 2100 elements[a] | 60.13 | 4.933 | $2.371 \times 10^6$ |
| Per cent difference mesh 1 and 2 | 1.11 | −1.57 | 0.65 |
| Mesh 1, 525 elements[b] | 58.8 | 4.97 | $2.36 \times 10^6$ |
| Confidence intervals[b] | ± 0.5 | ± 0.05 | $\pm 0.01 \times 10^6$ |
| Experimental uncertainty[b] | ± 2.5 | ± 0.3 | $\pm 0.09 \times 10^6$ |

[a] Present investigation.
[b] Dowding (1997).

On the scale of the results presented in figure 6 (relative finite difference step size of 0.005), the RMS errors appear to be smooth functions of the parameter values. Theoretically, one would expect this to be the case. However, additional computational experiments indicate that if a relative finite difference step size of 0.001 is used, there is enough numerical noise for the solution space to not appear smooth. If the solution space is non-smooth, then gradient-based methods may have significant difficulty in locating the minimum. All the parameter estimation computations reported here were run on Unix workstations using single precision arithmetic; the impact of using double precision arithmetic on the numerical noise present in the objective function is presently under investigation. For any new experimental configuration for which parameter estimation techniques are to be used, it is recommended that computational experiments be performed to develop some understanding of the numerical noise in the solution.

### 5.4. Parameter estimation results

An advantage of parameter estimation is that the accuracy of the estimated properties can be evaluated using residuals, sensitivity coefficients, and sequential estimates of the properties. These quantities provide insight into the estimation as well as insight into the experiment. Observing them can help improve the understanding of the experiment and support the accuracy of the estimated properties. The typical procedure uses these results in an iterative manner to develop insight into the experiment and analysis. As experiments and analyses are repeated, observation of these quantities helps to develop a deeper understanding of the parameter estimation process. The residuals, sequential estimates, and sensitivity coefficients provide a link between the analysis and experiment.

Currently the analysis code used in RIT does not return the sequential estimates or the sensitivity coefficients. Sequential estimates have been presented and discussed in Dowding *et al* (1995, 1996). A separate analysis code (Dowding *et al* 1996) was used to compute the sensitivity coefficients shown in this investigation. Temperature residuals and sensitivity coefficients are discussed next.

**5.4.1. Residuals.** It is important to look at the temperature residuals in order to ascertain the quality of the parameter estimation results. The residuals represent the difference between the measured and calculated temperatures. Figure 7 presents these temperature residuals for a representative run. Sensors 8 and 9, which are the closest to the active portion of the heater, have the greatest residuals. There is a pronounced spike in these residuals, both when the heater is turned on and when it is turned off. Some of this is due to the difficulty in numerically simulating a step change in the heat flux while using a finite size time step. In general, the residuals are smaller during the cool down phase than during the heating phase.

**5.4.2. Sensitivity coefficients.** As previously noted, observation of the sensitivity coefficients can provide insight into the estimation problem. Observation of the sensitivity coefficients at this stage may be too late, since the experiment is essentially designed and moving sensors or changing the heated area is not easily done. However, some minor modifications may improve the accuracy, such as changing the heating duration or magnitude. When possible, an analysis of the sensitivity coefficients should be conducted prior to running the experiments. The quality of the parameter estimates is a strong function of the time spent on experiment design.

Sensitivity coefficients provide information that may be used to design and understand experiments. In general, the scaled sensitivity coefficients are desired to be large for each parameter and uncorrelated (linearly independent) for different parameters. A sense of the magnitude of the sensitivity coefficients is gained through normalizing the sensitivity coefficients. Normalization is performed by multiplying by the parameters, resulting in units of temperature for all the scaled sensitivity coefficients. The scaled sensitivity coefficient for parameter $\eta$ is

$$\bar{X}_\eta = \eta \partial T/\partial \eta. \quad (7)$$

A comparison is then permitted with the temperature rise of the experiment. Using a separate finite element code the scaled sensitivity coefficients are computed for the current experimental design and are shown in figures 8 to 10. Figure 8 shows the sensitivity to $\rho C_{cc}$, $\bar{X}_{\rho C}$, and figures 9 and 10 show the sensitivity to the thermal conductivities $k_{y,cc}$ and $k_{x,cc}$, $\bar{X}_{k_y}$ and $\bar{X}_{k_x}$. Because the sensitivity coefficients are scaled, a direct comparison of their magnitudes is possible. Some observations are drawn from the sensitivity coefficient plots.

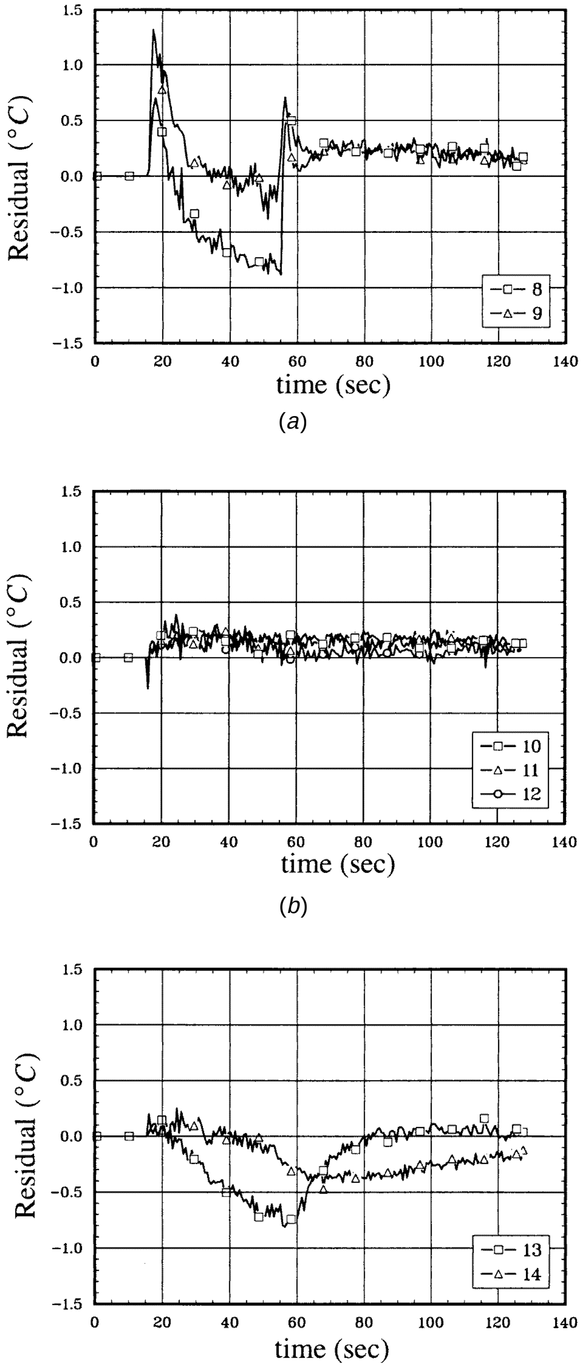


**Figure 7.** Temperature residuals for experiment begun at $T = 297\,^{\circ}\mathrm{C}$ and Mesh 1: (*a*) on the surface below the active heater; (*b*) on the heated surface, but not on the area of the active heater; and (*c*) at the carbon–carbon/insulation interface.

The most information is available on the active heater surface (sensor locations $x = 0.89$ cm and 1.91 cm for $y = 0$). At these locations the sensitivity coefficients have the largest magnitudes and therefore have the most influence on the values of the estimated properties. Notice in figure 9 that $X_{k_y}$ undergoes a sign change across the specimen (in the $y$-direction) and in figure 10 $\bar{X}_{k_x}$

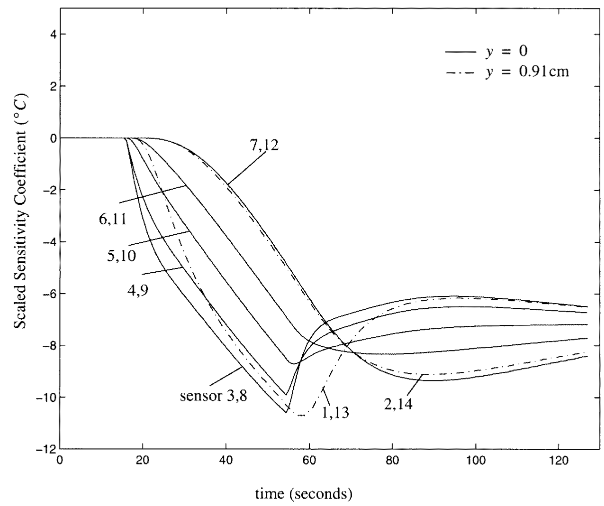


**Figure 8.** Scaled sensitivity coefficient for volumetric heat capacity, $\bar{X}_{\rho C}$.

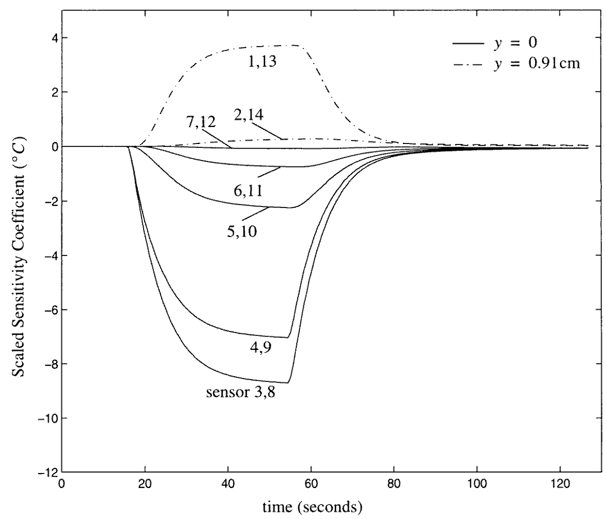


**Figure 9.** Scaled sensitivity coefficient for thermal conductivity in $y$-direction, $\bar{X}_{k_y}$.

undergoes a sign change along the specimen (in the $x$-direction). The implications of these sign changes are that there exist (1) a $y$-location within the body where the temperature is insensitive to $k_{y,cc}$ and, more importantly, (2) a $x$-location where the temperature is insensitive to $k_{x,cc}$. The latter result is more important for this case where surfaces temperatures are measured, because seemingly logical locations along the measurement surfaces $y = 0$, $L_y$ may be insensitive to $k_{x,cc}$. Although it is desirable to avoid locations where the sensitivity coefficients change sign because the sensitivity is quite small, sensor locations that have sensitivity coefficients with opposite signs are beneficial. This situation produces contrasting effects which improves the accuracy of the estimates. Hence, the locations near the edges of the specimen ($x = 0$, 7.62 cm), which have oppositely signed sensitivity coefficients, are chosen for the measurement locations. Fortunately, the surfaces of the specimen ($y = 0$, $L_y$) are the most sensitive

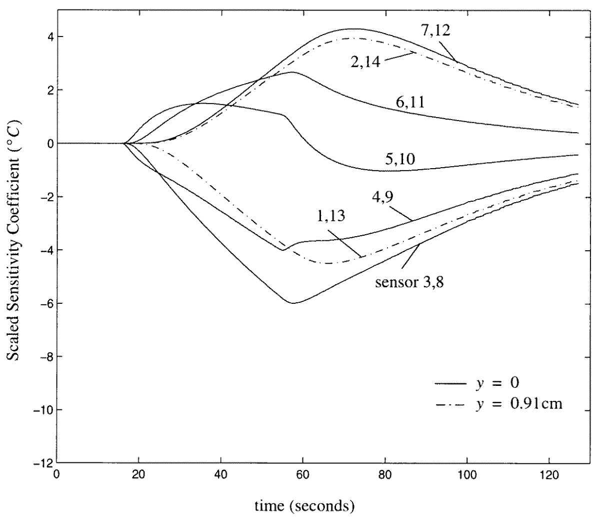


**Figure 10.** Scaled sensitivity coefficient for thermal conductivity in $x$-direction, $\bar{X}_{k_x}$.

in that direction. Finally, the experiment was halted after heating for approximately 40 s in order to make the magnitudes of the sensitivity coefficients for all thermal properties comparable. In figure 9 $\bar{X}_{k_y}$ approaches a steady state value, while the sensitivities to $\bar{X}_{k_x}$, figure 10, and $\bar{X}_{\rho C}$, figure 8, do not. The sensitivity coefficient $\bar{X}_{\rho C}$ does not have a steady state solution and will continue to increase linearly with time. If heating was continued for a longer duration $\bar{X}_{k_y}$ and $\bar{X}_{\rho C}$ would be much larger than $\bar{X}_{k_x}$. Consequently, the estimates for the two former properties would be considerably more accurate than that for the latter.

## 6. Summary

Techniques for estimating thermal properties from experimental measurements have been presented. Parameter estimation techniques permit analysis for general thermal models and are applicable to other problems involving different physics and parameters. The benefits of using parameter estimation and methods for aiding the experimental design were discussed. The use of parameter estimation provides a link between the experiment and analysis providing insight into both aspects. The quantities used in this process are residuals, sensitivity coefficients, and sequential estimates.

An approach for linking complex (thermal) analysis codes with parameter estimation techniques was discussed. It is a powerful alternative to previous approaches. Called RIT, the procedure allows analysis codes and parameter estimation codes to be developed separately and independently.

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