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Estimation of Thermal Properties of Composite Materials Without Instrumentation Inside the Samples

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Recent contributions of parameter estimation in the measurement of thermal properties are of great importance. In comparison with other techniques such as steady state (hot guarded plate, etc.) or transient (line source method, flash method, etc.), the use of parameter estimation provides more information and, in most cases, produces faster results. With this technique the thermal conductivity and the volumetric specific heat are estimated simultaneously and as a function of time, temperature, or position. This method requires experimental data, such as transient temperature and heat flux measurements. Previously, the temperature measurements came from thermocouples embedded in the sample. These thermocouples are introduced in the sample either by drilling holes or by molding the material around a series of thermocouples. Both operations are time-consuming and costly and are needed for each sample. In this study, temperature measurements are made only on the two sides of the samples with thin resistance thermometers. Since the sensors are not inside the material, the effect of the thermal contact conductance between sensor and sample was first investigated. The value of this thermal contact conductance was estimated by using samples of high-conductivity material. Using these values, the estimated thermal properties obtained with surface temperature measurements are compared with values provided by other methods for several low-thermal conductivity materials; agreement has been very good.

KEY WORDS: composite materials; low-conductivity materials; nonlinear least squares; nonlinear regression; parameter estimation; thermal property; thermal conductivity; volumetric specific heat.

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1. INTRODUCTION AND LITERATURE REVIEW

Many different techniques are available for measurement of thermal properties of solids. Calorimetry and immersion provide the volumetric heat capacity. Thermal conductivity is usually determined from steady-state (hot guarded plates) or transient line source methods; it is also determined from transient techniques, such as the quenching or flash method, from which the thermal diffusivity is found [1]. Each of these methods characteristically provides only one parameter at a time and this for one temperature. The recently developed technique called parameter estimation is unique in that thermal conductivity and volumetric heat capacity can be estimated simultaneously as a function of temperature from only one experiment. The computer program PROP1D developed at Michigan State University, primarily with support from the Oak Ridge National Laboratory, has all these features [2].

The objective of this study is to develop and test an experimental setup which provides fast and easy measurements of the thermal properties of materials of low thermal conductivity. Parameter estimation technique is used to analyze heat flux and temperature measurements. In previous works [3, 4], temperature measurements were performed with thermocouples embedded inside the samples and this involved a time-consuming and costly instrumentation of each sample. In the proposed experimental setup, temperatures are measured on the two sides of the samples using resistance thermometers and thermocouples.

An outline of the paper is as follows. First, a description of the experimental setup and of the parameter estimation technique is presented. Second, the effects of variations of thermal properties and thicknesses of silicone grease layers are investigated. Third, an example of a complete analysis showing measurements and information given during the estimation procedure is presented. Finally, estimated thermal properties for different materials are compared with literature values.

2. EXPERIMENTAL SETUP

2.1. Description of the Experimental Setup

The experimental setup was evolved by taking into account the concepts of optimal design of experiments, at various stages in the development. Parameter estimation problems can be sensitive to measurement errors, with the accuracy of the results very adversely affected by small measurement errors. There are many possible "optimal" experiments. The optimal experiment depends upon what boundary conditions are considered and what variability is permitted. This paper considers the measurement of the thermal conductivity, k, and volumetric specific heat, ρc_p, from transient temperature and heat flux measurements. The focus in this paper is upon experiments for low-conductivity materials (such as epoxy-matrix composites) and for simple on–off control of a heater, not an arbitrary time variation. For this restricted class of possible designs, the "best" design (which would provide the most accurate results) suggested by Beck and Arnold [5] and Taktak et al. [6] consists of a finite body with a step increase in heat flux at one boundary and an isothermal condition at the other boundary.

Simultaneous estimation of thermal properties requires heat flux and temperature measurements [7]. Generating the heat flux electrically permits its simple measurement. There is no need for a heat flux transducer, which requires careful calibration and provides less accurate measurements. The experimental setup (see Fig. 1) is symmetric in relation to the electric heater and requires two similar cylindrical samples. On both sides of the heater there are successively a resistance thermometer, a sample, and an aluminum block with a thermocouple in a groove at its face in contact with the sample. Temperature measurements are provided by thermocouples and resistance thermometers. The use of a resistance thermometer between the heater and the sample has a number of advantages. The first relates to the nonuniform heater temperature; the heater is made of an etched copper foil and provides nonuniform heating. A thermocouple would give a different measurement according to its location over the interface heater/sensor. In contrast, a resistance thermometer provides the average

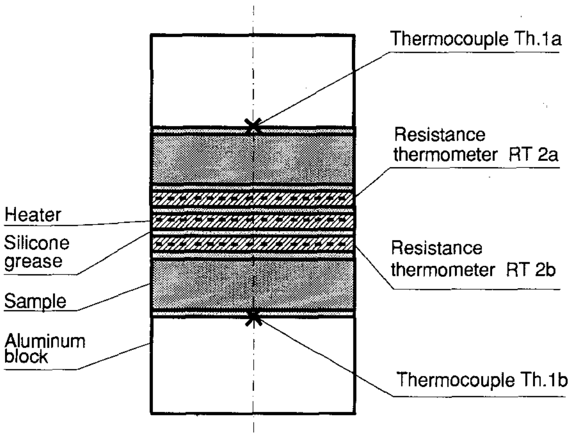

Fig. 1. Experimental setup A for thermal properties estimation of composite materials.

temperature all over this interface. Second, the location of the temperature sensor is accurately known. Third, the temperature is measured near the heated surface and the heat capacity of the heater is small. As is known, such a combination uses optimal experiment concepts because the heated specimen surface is the "best" location and the minimum heat capacity of the heater/sensor is desired. Fourth and finally, the measurements have been found to be extremely accurate compared to using thermocouples.

The purpose of the aluminum block is to obtain a quasi-isothermal condition at the nonheated face of the sample. Each element of the experiment is separated with a layer of silicone grease to promote good thermal contact. The experimental components have a diameter of 76.2 mm (3 in.), the samples are about 6 mm (0.25 in.) thick, and the aluminum block is 38.1 mm (1.5 in.) thick. Heat losses on the edge of each sample are small. Using the basic laws for natural convection and radiation, heat losses between the edge of each sample and the surrounding atmosphere does not exceed 0.3% of the heat flux electrically generated by the heater when the temperature difference is about 20°C.

2.2. Description of the Heating and Temperature Measurement Devices

In this study, two setups are presented according to the device used for the electric heating and temperature measurement with resistance thermometers. For the first one (setup A), the electric heater and resistance thermometers are three similar Minco heaters separated by silicone grease layers. The central heater provides the heating, the two others with their etched foil oriented perpendicularly to the etched copper foil of the heater provide temperature measurements. Each heater is made of an etched copper foil glued between two thin layers of Kapton and has a total thickness of 0.162 mm.

In the second setup (setup B), the heater and resistance thermometers are inside a unique device called thermofoil heater/sensors designed by Minco Corp. especially for this experiment. It consists of four layers of Kapton, two platinum foils, and one etched copper foil. The platinum foils used as resistance thermometers are located between the first and the second and between the third and the fourth layer of Kapton. The heater element (etched copper foil) is glued between the two central Kapton layers. Setup B is expected to give more accurate results for two reasons. First, the overall thickness, about 0.32 mm, of the thermofoil heater/sensors for setup B is smaller than the total thickness of three heaters with two silicone grease layers (setup A). Second, the temperature dependence of the electrical resistance of platinum is about 22 times larger than that of copper.

In both setups (A and B), the variation of the electrical resistance of the resistance thermometers versus temperature is detected using Wheatstone bridges. The relationship between the output voltage of the Wheatstone bridge and the temperature was found by calibration. In this process, the temperature T of the wire of each resistance thermometer was interpolated using the two temperatures measured by thermocouples located on each side of the resistance thermometer. This temperature T was collected versus the output voltage of the Wheatstone bridge during a transient experiment with a small cooling rate. Wheatstone bridges are supplied by a precision voltage source (Digitec 310). The temperatures and voltages are recorded using accurate amplifiers (Ectron Corp. Model 687 DC) equipped with electronic reference junctions for the thermocouples. A remote DC power supply (Hewlett-Packard Model 6024A) provides electric heat flux in the heater of the experiment. A Microvax II with digital/analog (D/A) and analog/digital (A/D) boards controls the power supply of the heater and measures and records temperatures and heat fluxes.

3. PARAMETER ESTIMATION PROCEDURE AND MATHEMATICAL MODEL

The parameter estimation technique involves minimizing a least-squares function S with respect to the parameters to be estimated. In our experiment, errors in heat flux measurement are assumed to be negligible compared to the temperature measurement errors (with setup B the temperature measurements are so accurate that this assumption may not be valid). The least-squares function S can be expressed mathematically as

$$S = [\mathbf{Y} - \mathbf{T}(\boldsymbol{\beta})]^T [\mathbf{Y} - \mathbf{T}(\boldsymbol{\beta})] \tag{1}$$

where $\mathbf{Y}$ represents the vector containing measured temperatures and $\mathbf{T}$ is the vector containing calculated temperature. The vector $\boldsymbol{\beta}$ contains the "true" value of the parameters. Estimated values (vector $\mathbf{b}$) of the parameters are found by minimizing S using a modified Gauss method. An iterative scheme is used to calculate $\mathbf{b}$ from the recurrence expression [5]:

$$\mathbf{b}^{(i+1)} = \mathbf{b}^{(i)} + (\mathbf{X}^{T(i)}\mathbf{X}^{(i)})^{-1} [\mathbf{X}^{T(i)}(\mathbf{Y} - \mathbf{T}^{(i)})] \tag{2}$$

where i is the iteration number and $\mathbf{T}^{(i)}$ is the temperature vector calculated knowing $\mathbf{b}^{(i)}$, the vector containing the estimated parameters at the ith iteration. The quantity $\mathbf{X}$ is the sensitivity coefficient matrix and can be written as [5]:

$$\mathbf{X}(\mathbf{b}) = [\nabla_{\mathrm{b}} \mathbf{T}^T(\mathbf{b})]^T \tag{3}$$

At each iteration, the calculated vector temperature $\mathbf{T}^{(i)}$ is computed using the numerical solution of the governing equation (one-dimensional heat transfer equation):

$$\frac{\partial}{\partial x}\left(k_j \frac{\partial T_j}{\partial x}\right) = (\rho c_{\rm p})_{\rm j} \frac{\partial T_j}{\partial t}, \qquad j \text{ is the index for materials}, \qquad j = 1, 2, \ldots, N \tag{4}$$

where N is the number of "regions," which may or may not be different materials. At the silicone grease–aluminum interface ($x = L$), the temperature is measured and prescribed,

$$T_N(L, t) = T_N(t) \tag{5}$$

The parameter estimation program PROP1D developed by Beck [2] uses finite difference approximations with the Crank–Nicolson method. For the calculation of the calculated temperature vector $\mathbf{T}$, the system of equations also contains the boundary conditions obtained during the experiment. PROP1D permits consideration of more than one material, so all the components of the experiment from heater to sample can be taken into account. The physical heat transfer model for setup A is shown in Fig. 2; it involves seven regions on only one side of the electrical wire of the heater because of the symmetry of the experimental setup. Due to their small thicknesses (less than 0.03 mm) and high thermal conductivities, the etched

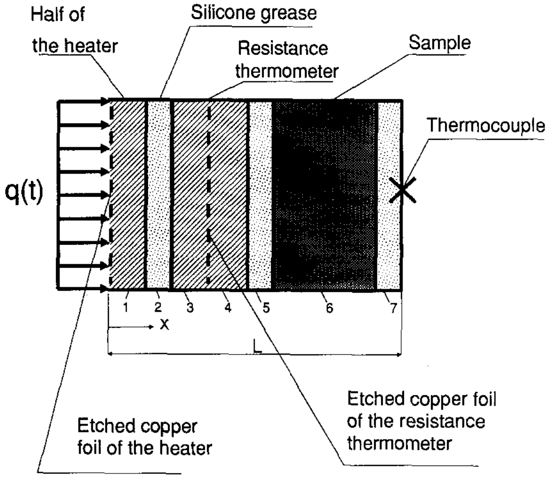

Fig. 2. Heat transfer model and temperature measurements locations (setup A).

copper foils of the heater and of the resistance thermometers are not taking into account as regions in the one-dimensional heat transfer equation (4). Temperature measurements noted **Y** come from the etched copper foil of the resistance thermometer located between region 3 and region 4. Boundary conditions are at $x=0$, a time-variable (on or off) heat flux prescribed by the etched copper foil of the heater, and at $x=L$, a variable temperature given by a thermocouple. Setup B, containing the thermofoil heater/sensors, has a physical heat transfer model similar to setup A except region 2 (i.e., one silicone grease layer) and region 3 (i.e., one Kapton layer), which are not present. Therefore in setup B, between the etched copper foil of the heater and the platinum foil of the resistance thermometer, there is only one region which is a layer of Kapton material (region 1).

The parameter estimation program PROP1D can also find thermal properties as a function of temperature and various kinds of boundary condition can be considered. To run PROP1D, an input control parameter file and an input datafile are required. The first one contains the description of the physical heat transfer model (number of materials, their thicknesses, and their thermal properties except for the one to be estimated). It also includes the control parameters for the numerical resolution of the heat transfer equation and the parameter estimation procedure (time step, time domain, number of iterations allowed, initial guess for the parameters, etc.). The second file contains the values of the boundary conditions (heat flux or temperature) and the temperatures measured by the internal sensors for each experimental time step. The program as currently configured runs on a IBM Personal Computer (286/386/486) or compatibles equipped with a math coprocessor. In our case (thermal properties constant with temperature, 250 time steps, 4 iterations), the run time is between 2 and 3 min using the Zenith 386 with 16 MHz.

4. EFFECT OF SILICONE GREASE LAYERS

4.1. Introduction

In the heat transfer model, three different kinds of materials are used. Only the thermal properties of the samples are estimated. The thermal properties ($k=0.98\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$, $\rho c_p = 1.871 \times 10^6\ \mathrm{J \cdot m^{-3} \cdot K^{-1}}$) and thickness of the Kapton layers were provided by the heater manufacturer. The thermal properties, and especially the thicknesses of silicone grease layers, were not accurately known. Consequently, the heat capacity was measured with a calorimeter, the density by the immersion technique, and the thickness with a micrometer. The thermal conductivity was estimated using the actual experimental setup and the parameter estimation technique. Thermal property measurement errors can occur in the specimen because of errors in thicknesses and thermal properties of these three materials (silicone grease, Kapton and specimen). Variation of 10% of the thermal properties and variation of 5% in the thickness of Kapton layers modified the estimated values by less than 0.5%. Effects of the variation in the estimation of the thermal properties of silicone grease are investigated. Since thicknesses are variable from one experiment to the other when new layers are applied, it is also necessary to study the reproducibility of the estimated values when the silicone grease layers are renewed.

4.2. Thickness and Thermal Property of Silicone Grease Layers

With the classical technique noted above, the volumetric specific heat of the silicone grease was found to equal $2.005 \times 10^6\ \mathrm{J \cdot m^{-3} \cdot K^{-1}}$. The thickness of different silicone grease layers applied carefully with a "comb" between two disks was measured using an outside micrometer and was found to be equal to $(0.070 \pm 0.014)\ 10^{-3}$ m. Estimation of the thermal properties of the silicone grease with the experimental setup was difficult because, first, the very small thickness of the layers involves small temperature differences and, second, the thermal properties of composite materials are also unknown. Therefore, Armco iron samples, which have higher and well-known thermal properties, were substituted for the composite samples and short-time experiments were performed. In contrast to the composite materials, they provided nearly an isothermal condition and involved higher temperature gradients and differences inside the silicone grease layers during the heating. (Higher heat fluxes are now applied, for a brief time interval concentrating the temperature variations near the silicone grease interface, rather than in the specimen.) These fluxes induced higher sensitivity coefficients, which produce smaller and acceptable confidence bands for the estimation of the thermal conductivity of the silicone grease.

Table I contains the different thermal conductivities of silicone grease

Table I. Effect of the Thickness (e) of Silicone Grease on the Estimation of its Thermal Conductivity (k) (Setup A, Armco Iron Samples)

e (mm)	RMS (°C)	$k \pm \Delta k\ (\mathrm{W \cdot m^{-1} \cdot K^{-1}})$
0.05	0.445	0.1240 ± 0.0085
0.07	0.445	0.1736 ± 0.0012
0.09	0.445	0.2232 ± 0.0015

estimated versus its thickness introduced in the heat transfer model. The root mean square (RMS) and the thermal conductance k/e, where k is the estimated thermal conductivity and e the thickness of the silicone grease layer, are nearly the same for every case. This is an effect of the linearity of the governing equation used in the parameter estimation technique. For the range of thickness e measured previously, the estimated thermal conductivities show that the value given by the manufacturer in a technical note ($k = 0.418\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$) is too large. The difference between the estimated values shown in Table I are quite significant but the thickness of the silicone grease layers is small, resulting in minimal effects on the accuracy of k and ρc_p of low-thermal conductivity materials.

4.3. Effect of Thickness and Thermal Properties of Silicone Grease on the Estimated Thermal Properties of Composite Materials

In Table II, it is shown that the estimated thermal properties, k and ρc_p, of composite materials are little affected by the different values attributed to the thermal conductivity, k_g, and thickness, e, of silicone grease (0.2% variation for k and 2.3% variation for ρc_p). Also, an increase of 10% in the volumetric capacity of silicone grease results in only a 0.06% increase in k and 0.7% decrease in ρc_p.

4.4. Reproducibility of the Estimated Thermal Properties of Composite Materials

From one experimental setup to the other, the thicknesses of the silicone grease layers are variable. To study the effect on the estimated thermal properties, new silicone grease layers were applied three times involving three experimental setups. For each experimental setup, experiments (heat flux and temperature measurements) were performed twice. Results are presented in Table III. Within one experimental setup, estimated values are reproducible (the variation is less than 0.3% for the

Table II. Effect of the Thickness (e) and Thermal Conductivity (k_g) of Silicone Grease on the Estimated Thermal Properties of Plexiglas (Setup A)

e (mm)	k_g ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)	RMS (°C)	$k \pm \Delta k$ ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)	$\rho c_p \pm \Delta \rho c_p$ ($\mathrm{J \cdot m^{-3} \cdot K^{-1}}$)
0.05	0.1240	0.065	0.1899 ± 0.0005	$(1.560 \pm 0.014) \cdot 10^6$
0.07	0.1736	0.072	0.1901 ± 0.0007	$(1.527 \pm 0.017) \cdot 10^6$
0.09	0.2232	0.079	0.1903 ± 0.0010	$(1.492 \pm 0.022) \cdot 10^6$

Table III. Reproducibility of the Estimated Thermal Properties with Experimental Setup B (Epoxy B)

Setup	RMS (°C)	$k \pm \Delta k$ ($W \cdot m^{-1} \cdot K^{-1}$)	$\rho c_p \pm \Delta \rho c_p$ ($J \cdot m^{-3} \cdot K^{-1}$)
1	0.087	0.2264 ± 0.0031	$(1.423 \pm 0.035) \cdot 10^6$
1	0.081	0.2257 ± 0.0029	$(1.435 \pm 0.033) \cdot 10^6$
2	0.080	0.2236 ± 0.0029	$(1.446 \pm 0.033) \cdot 10^6$
2	0.079	0.2243 ± 0.0029	$(1.454 \pm 0.033) \cdot 10^6$
3	0.057	0.2252 ± 0.0019	$(1.515 \pm 0.023) \cdot 10^6$
3	0.042	0.2245 ± 0.0012	$(1.520 \pm 0.017) \cdot 10^6$

thermal conductivity and less than 0.85% for the volumetric heat capacity). However, from one experimental setup to the other one, the differences are larger. Thermal conductivity varies within a range of 1.7%, and volumetric heat capacity within a range of 6.8%. The variations for the thermal conductivity are still small; the variability of the volumetric heat capacity values can be reduced if the experiments are performed with shorter heating times. With the same temperature rise, the sensitivity coefficients for the volumetric heat capacity would be larger and would involve smaller confidence bands for this parameter and better reproducibility.

5. EXAMPLE OF THERMAL PROPERTIES ESTIMATION

The following is an example of thermal properties estimation with epoxy samples and experimental setup B, which utilizes the combined

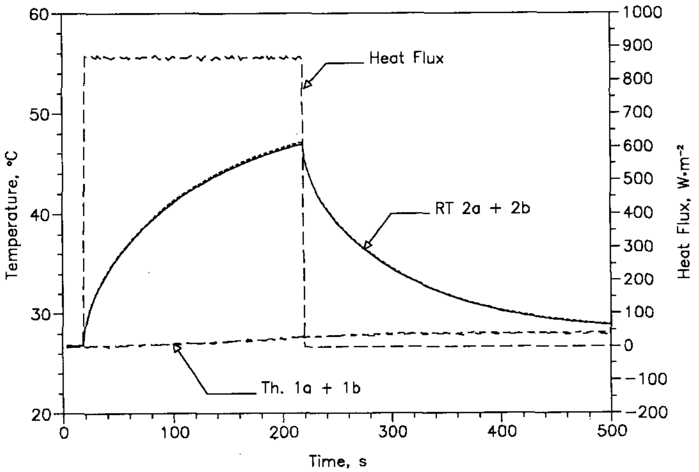

Fig. 3. Heat flux and temperature measurements with epoxy B samples (setup B; experimental time step equal to 2 s).

heater/sensor elements. Measurements from the experiment and information from the parameter estimation procedure are presented.

Heat flux and temperature measurements recorded during a transient experiment are shown in Fig. 3. The difference between the temperatures measured by the two resistance thermometers is less than 0.22°C, which is extremely small. The fact that the resistance thermometer RT 2b compared to RT 2a shows a temperature slightly higher can be explained by a difference in the thicknesses of the silicone grease layers applied on both sides of the thermofoil heater/sensors. Another explanation is a small bias in the temperature calibration of the resistance thermometers.

The parameter estimation program PROP1D uses the concept of sequential estimation [5]. The parameters are updated as new observations are added. Therefore, estimated thermal properties can be represented as functions of time; they are then commonly called sequential values. Observation of the sequential values is a way to check the heat conduction model used. Sequential values which continue to change over the time domain indicate imperfection in the model or bias in the measurements. On the other hand, sequential values that are nearly constant indicate both accurate parameter values and a good mathematical model. In Fig. 4, the sequential estimates of the parameters are nearly constant after the end of the heating, where variations do not exceed 0.4% for the thermal conductivity and 0.9% for the volumetric heat capacity. This indicates a good mathematical model and accurate results. The model could be imperfect due to heat losses, large variation in the thermal properties, and inaccurate temperature measurements.

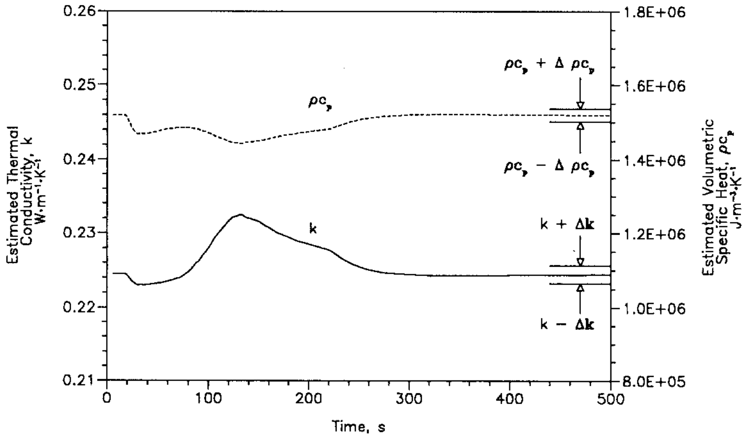

Fig. 4. Sequential estimation of thermal conductivity k and volumetric specific heat ρc_p (experimental data from the experiment shown in Fig. 3).

Another feature of the parameter estimation technique is to provide the sensitivity coefficients. As a general rule, the sensitivity coefficients should be uncorrelated (have different basic shapes) and have "large" magnitudes, when they are plotted in the form, $\beta\,\partial T/\partial\beta$, where β is a parameter. In such a form the units are those of temperature and are independent of whether k or ρc_p (which have much different numerical values) is used as the parameter. Figure 5 presents the sensitivity coefficients as functions of time for the experiment shown in Fig. 3. The sensitivity coefficients for the volumetric heat capacity start negative and then become positive, whereas those for the thermal conductivity are larger in magnitude and are always negative. Hence, sensitivity coefficients are not linearly dependent, and thus no difficulty is expected for the simultaneous estimation of the thermal properties of the epoxy samples with this experiment. In addition, the magnitudes of the sensitivity coefficients are large. The absolute value of the sum, S_c, of the two sensitivity coefficients at $t = 218$ s is close to the maximum temperature rise $\Delta T_r = 19.45$°C, which is the maximum possible for this case. Hence, the uncorrelated nature of the sensitivity coefficients and their relative large magnitudes testify to the effectiveness of the design of the experiment.

Residuals, defined as the difference between measured and calculated temperatures, are useful to check the validity of the heat transfer model. When designing the heat transfer experiment, heat losses occur very often and they are detected with the parameter estimation technique when the

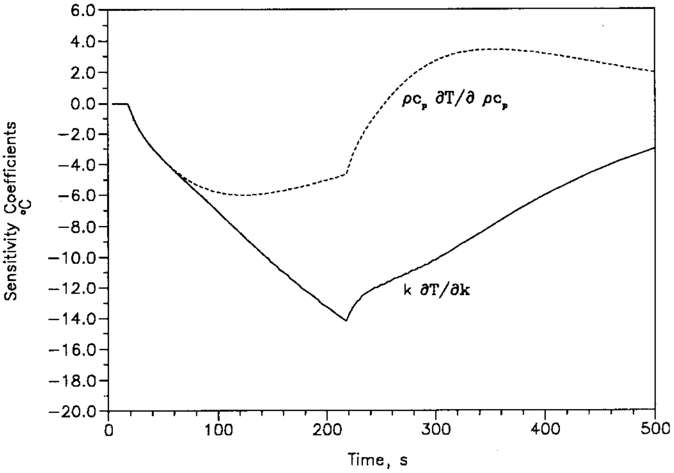

Fig. 5. Sensitivity coefficients for thermal conductivity k and volumetric heat capacity ρc_p at the location of the resistance thermometers (experimental data from the experiment presented in Fig. 3).

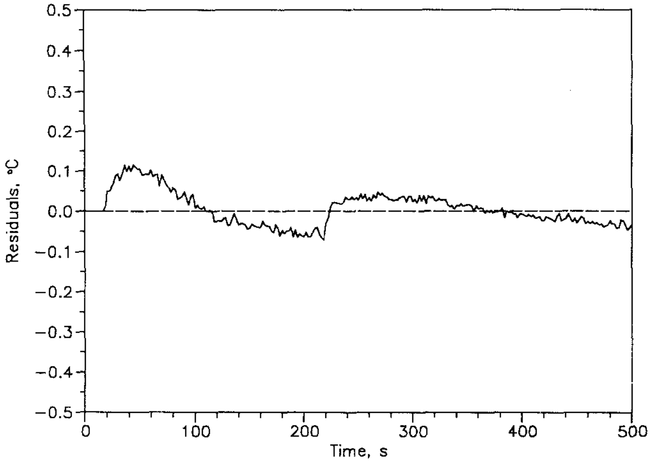

Fig. 6. Residuals (experimental data from the experiment shown in Fig. 3).

residuals increase with time at the end of the experiment and have a characteristic shape from experiment to experiment. Inspection of the residuals is a way to check the standard statistical assumptions (Beck and Arnold [5]) about the measurements errors used in the parameter estimation procedure. The residuals obtained with the previous experiment

Table IV. Estimated Thermal Conductivity (k) and Volumetric Heat Capacity (ρc_p) for Different Materials and Comparison with Literature Values (Setups A and B)

Material (setup A/B)	RMS (°C)	$k \pm \Delta k$ ($W \cdot m^{-1} \cdot K^{-1}$)	$\rho c_p \pm \Delta \rho c_p$ ($10^6 J \cdot m^{-3} \cdot K^{-1}$)	Literature value
Carbon fiber comp. (A)	0.185	0.4949 ± 0.0024	1.437 ± 0.021	—
Comp. 1 (A)	0.117	0.1713 ± 0.0025	1.171 ± 0.042	—
Glass-fiber comp (A)	0.374	0.4225 ± 0.0225	1.666 ± 0.230	—
MPDA epoxy Epon 828 (A)	0.163	0.1905 ± 0.0051	1.200 ± 0.085	$k = 0.183\ W \cdot m^{-1} \cdot K^{-1}$[a]
Plexiglas (A)	0.072	0.1901 ± 0.007	1.527 ± 0.017	$k = 0.195\ W \cdot m^{-1} \cdot K^{-1}$[b] $k = 0.196\ W \cdot m^{-1} \cdot K^{-1}$[c]
Comp. 2 (A)	0.124	0.5888 ± 0.0071	1.664 ± 0.056	$k = 0.579 \pm 0.019\ W \cdot m^{-1} \cdot K^{-1}$[a] $\rho c_p = (1.77 \pm 0.18) \cdot 10^6\ J \cdot m^{-3} \cdot K^{-1}$[a]
Epoxy B (B)	0.042	0.2245 ± 0.0012	1.520 ± 0.017	—

[a] From Ref. 3.
[b] From Refs. 8 and 9.
[c] From Ref. 10.

(Fig. 3) show a small correlation between the measurement errors (Fig. 6). The magnitude of the residuals is quite remarkable, since they are smaller than 0.12°C, which corresponds to only 0.06% of the maximum temperature rise. This amount is the contribution of all the errors in temperature measurements (calibration, etc.) and in the mathematical model and its numerical solution. Therefore, small confidence bands for the estimates are expected, which is shown in Table IV (epoxy B samples).

6. ESTIMATION OF THERMAL PROPERTIES OF DIFFERENT MATERIALS: COMPARISON WITH LITERATURE VALUES (SETUPS A and B)

For various materials, Table IV presents the estimated thermal properties with their confidence bands. It is of interest to compare them with literature values. The discrepancy is at most 4% for the thermal conductivity and 7% for the volumetric heat capacity. These results were obtained with the experimental setup noted A, better estimation is achieved with setup B as shown by the RMS values obtained with epoxy B samples.

7. CONCLUSION

The technique using resistance thermometers to measure the temperature on both sides of the heater gives excellent results. With the experimental setup presented, estimation of thermal properties is very reproducible. Moreover, the results obtained are in good agreement with literature values. The confidence bands are very small, especially when using resistance thermometers made of platinum (setup B). With this device, the magnitude of the residuals is even smaller than those obtained when the temperature is measured with thermocouples.

The other advantage of the experimental setup developed in this study is that no instrumentation (thermocouples, heat flux transducer, etc.) is needed inside the samples. Therefore, with the parameter estimation technique, thermal properties of composite materials can be measured very quickly; measurement of the thermal conductivity and volumetric heat capacity can be performed in 45 min. This includes the installation of the samples in the experimental setup (application of silicone grease, etc.), the experimental run, and the analysis of the transient measurement with the computer program PROP1D, which provides the estimation of the thermal properties and other information such as confidence bands, sensitivity coefficients, sequential values, and residuals.

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