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EXPERIMENTAL IDENTIFICATION OF THERMAL CONDUCTIVITY AND VOLUMETRIC HEAT CAPACITY

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Abstract. *This paper deals with the estimation of the thermal conductivity and of the volumetric heat capacity of solids. The experimental setup designed in our previous works consists of a heater symmetrically assembled between two pieces of the specimen with unknown properties. Transient temperature measurements taken in the heated surface of the specimen are used in two estimation procedures, namely: the Levenberg-Marquardt Method and the Sequential Parameter Estimation Technique. Results are presented for the estimation of the properties of Teflon.*

Keywords. *Thermal Conductivity, Volumetric Heat Capacity, Parameter Estimation, Levenberg-Marquardt, Sequential Parameter Estimation.*

1. Introduction

The accurate knowledge of thermophysical properties is of importance for the correct prediction of the thermal behavior of bodies. Several experimental techniques have been developed in the past for the estimation of thermal conductivity and volumetric heat capacity by using steady-state as well as transient experiments. Transient techniques have the advantage of involving faster experiments than steady-state techniques. More recently, the use of inverse analysis techniques of parameter estimation have been used for the identification of thermophysical properties, by utilizing minimization procedures involving transient measurements (Taktak et al, 1993, Dowding et al, 1995, 1996, Orlande and Ozisik, 1994, Orlande et al, 1995, Guimarães et al, 1997, Lima e Silva et al, 1999, Mejias et al, 1999, Oliveira et al, 1999, Rey Silva et al, 2000, Rey Silva and Orlande, 2001).

In a previous paper Oliveira et al (1999) discussed the design of optimum experiments for the simultaneous estimation of thermal conductivity and volumetric heat capacity of solids. Three possible arrangements for the experimental setup, involving a heater placed between two identical pieces of the specimen with unknown properties, were examined in such work. The arrangement resulting on smaller confidence regions for the parameters was that involving a constant temperature boundary condition for the non-heated surface. The experiment was also optimally designed with respect to the sensor location, heating time and duration of the experiment. The Levenberg-Marquardt Method (Beck and Arnold, 1977, Ozisik and Orlande, 2000) was used for the minimization of the least-squares norm. The accuracy of such a parameter estimation approach was verified by using transient simulated measurements containing random errors. Later, Rey Silva et al (2000) implemented and tested, by using transient simulated measurements, the Sequential Estimation Procedure advanced by Beck and Arnold (1977) for the identification of thermal conductivity and volumetric heat capacity. The experimental setup addressed was the same designed by Oliveira et al (1999).

The main objective of this paper is to use actual experimental data for the identification of thermal conductivity and volumetric heat capacity of solids, by using a recently built experimental setup based on the optimized experiment described by Oliveira el al (1999). For the estimation of such physical properties the Levenberg-Marquardt Method (Beck and Arnold, 1977, Oliveira et al, 1999, Ozisik and Orlande, 2000) and the Sequential Parameter Estimation Procedure (Beck and Arnold, 1977, Rey Silva et al 2000) are used. The experimental setup, as well as the estimation techniques utilized in this work, is described next.

2. Experimental setup

The experimental setup used in this work consists of a heater placed between two identical cylindrical specimens of the material with unknown thermal conductivity and volumetric heat capacity, as depicted in Fig. (1). With such an arrangement, half of the heat generated in the heater is expected to be conducted through each of the specimens. Temperature measurements are taken at the center of the heated surfaces of the two specimens, as well as at the center of their non-heated surfaces, with type E thermocouples. The thermocouples are connected to a Cole-Parmer MAC-14 data acquisition system, which permitted an acquisition rate of 1 reading per second, for each of the four channels used. Therefore, the symmetry of the problem can be monitored during the experiment by examining the temperature recordings taken in each specimen, which are expected to be identical, for the two heated surfaces, as well as for the two non-heated surfaces.

This work is basically concerned with the identification of thermal conductivity and volumetric heat capacity of insulating materials. Therefore, in order to approximate a constant temperature boundary condition for the non-heated surfaces, a material with high thermal conductivity is put in contact with the specimens. Two circular disks made of aluminum, 30.4 mm thick and with 100 mm of diameter, were used with such a purpose. The constant temperature boundary condition for the non-heated surfaces was chosen based on the optimum design of the experiment, aiming at the estimation of properties with high accuracy (Oliveira et al, 1999).

The heater, as well as the specimens, have a diameter of 100 mm. Such a diameter was chosen in such a way that, even if lateral heat losses are significant, the temperature at the center of the heated surfaces deviates by less than 4% from the one-dimensional case. For such a design, a strict case was taken into consideration, involving heat losses by convection through the lateral surfaces to the surrounding air with a heat transfer coefficient of 5 W/m^2 ^{o}C. However, the heater, specimens and aluminum disks have their lateral surface thermally insulated with styrofoam, with inner diameter of 100 mm and outer diameter of 200 mm. A photo of the disassembled experimental setup is presented in Fig. (2).

The specimens are 4 mm thick and the heater is 2.3 mm thick, with measured electrical resistance of 273 ± 3 Ω. The heater was connected to a DC font. The electrical voltage applied to the heater is measured with a Techmaster DM 8300 AW multimeter made by Sperry from Instruments Inc. The heater is turned on for a period $0 < t \leq t_h$. Transient temperature measurements taken at the heated surface of the solid in the period $0 < t \leq t_f$, where $t_h \leq t_f$, are used for the estimation of the properties, as described below. We note that the thickness of the specimens is different from that presented in Fig. (1). Such thickness was chosen in the optimum experimental design discussed by Oliveira et al (1999).

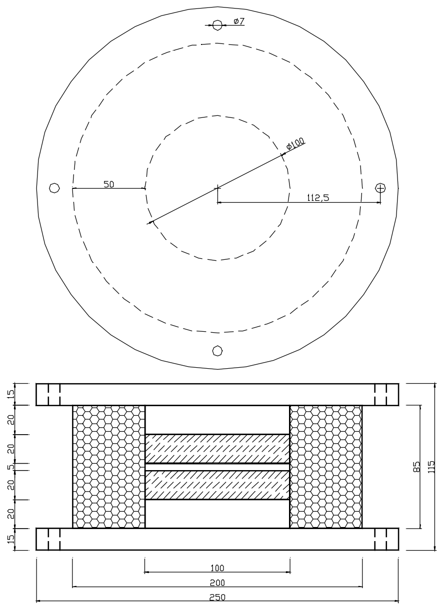

Figure 1. Geometry of the experimental setup.

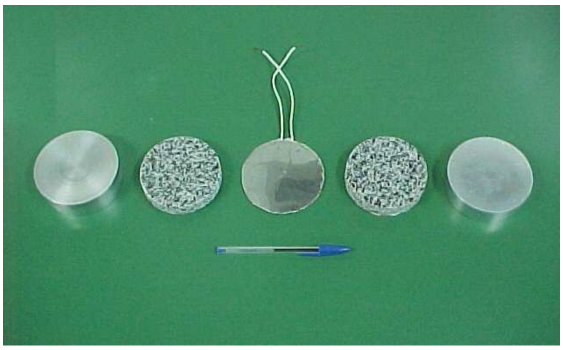

Figure 2. Disassembled experimental setup.

2. Mathematical Formulation

By taking into account the symmetry of the experimental apparatus, the mathematical formulation of the physical problem examined here is given in dimensionless form as:

$$C^* \frac{\partial \theta}{\partial \tau} = k^* \frac{\partial^2 \theta}{\partial X^2} \qquad \text{in } 0 < X < 1, \qquad \text{for } \tau > 0 \tag{1.a}$$

$$-k^* \frac{\partial \theta}{\partial X} = Q(\tau) \qquad \text{at } X = 0, \qquad \text{for } \tau > 0 \tag{1.b}$$

$$\theta = \theta_P \qquad \text{at } X = 1, \qquad \text{for } \tau > 0 \tag{1.c}$$

$$\theta = 0 \qquad \text{at } \tau = 0, \qquad \text{in } 0 < X < 1 \tag{1.d}$$

where the following dimensionless variables were defined:

$$X = \frac{x}{L},\ \tau = \frac{k_R}{C_R L^2} t,\ k^* = \frac{k}{k_R},\ C^* = \frac{C}{C_R},\ \theta = \frac{k_R}{q_0 L}(T - T_0),\ \theta_P = \frac{k_R}{q_0 L}(T_P - T_0),\ Q(\tau) = \frac{q(t)}{q_0} \tag{2.a-g}$$

In Eqs. (2), x is the dimensional space coordinate, t is the dimensional time coordinate, L is the thickness of the specimen, k and C are the thermal conductivity and the volumetric heat capacity of the specimen, respectively, k_R and C_R are reference values for thermal conductivity and volumetric heat capacity, respectively, q_0 is a reference heat flux, T is the dimensional temperature, T_0 is the initial temperature in the region, T_P is the constant temperature at the boundary x = L, and q(t) is the applied heat flux during the experiment.

The problem defined by Eqs. (1), with known thermophysical properties and known boundary and initial conditions, constitutes a *Direct Heat Conduction Problem*. The objective of the direct problem is to obtain the transient temperature field in the specimen. For the solution of the direct problem, we use the finite-volume method.

3. Inverse problem

For the *Inverse Problem* considered here, the thermal conductivity k* and the volumetric heat capacity C* are regarded as unknown quantities. For the estimation of such properties, we consider available for the inverse analysis the transient readings Y_i taken at times t_i, i = 1,…,I of one temperature sensor located at the heated surface of the solid with unknown properties. Since we have a symmetrical experimental assembly, the measurements used for the inverse analysis described below are taken as the average of the temperatures measured at the heated surfaces of each specimen. Similarly, the boundary condition at the non-heated surface (see Eq. (1.c)) is taken as the average temperature of the two sensors located at the non-heated surfaces.

In the present paper, two different estimation techniques are used: the *Levenberg-Marquardt Method* (Beck and Arnold, 1977, Oliveira et al, 1999, Ozisik and Orlande, 2000) and the *Sequential Estimation Procedure* (Beck and Arnold, 1977, Beck, 1999, Rey Silva et al, 2000). The main steps of both techniques are shown below.

3.1. The Levenberg-Marquardt Method

For the estimation of the unknown parameters with the Levenberg-Marquardt method, let us assume that the temperature measurement errors are additive, normally distributed with zero mean and known and constant standard-deviation. We also assume that all the other quantities appearing in the formulation of the problem are exactly known for the inverse analysis and that there are no errors in the independent variables. In this case, the least-squares norm becomes a minimum variance estimator (Beck and Arnold, 1977). Therefore, for the estimation of the unknown parameters we consider the minimization of the least-squares norm, written in matrix form as:

$$S(\mathbf{P}) = [\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T [\mathbf{Y} - \mathbf{T}(\mathbf{P})] \tag{3.a}$$

where

$$[\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T = [Y_1 - T_1(\mathbf{P}), Y_2 - T_2(\mathbf{P}), \ldots, Y_I - T_I(\mathbf{P})] \tag{3.b}$$

and

$$\mathbf{P} = [k^*, C^*] \tag{3.c}$$

The estimated temperatures $T_i(\mathbf{P})$ are obtained from the solution of the direct problem given by Eqs. (1) by using estimated values for the unknown parameters.

We use in this paper the Levenberg-Marquardt Method (Beck and Arnold, 1977, Ozisik and Orlande, 2000) for the minimization of the objective function given by Eq. (3.a). The iterative procedure of such method is given by:

$$\mathbf{P}^{k+1} = \mathbf{P}^{k} + (\mathbf{J}^{T}\mathbf{J} + \mu^{k}\Omega^{k})^{-1}\mathbf{J}^{T}[\mathbf{Y} - \mathbf{T}(\mathbf{P}^{k})] \tag{4}$$

where μ^k is the damping parameter and Ω^k is a diagonal matrix, which can be taken as the identity matrix or as the diagonal of $\mathbf{J}^T\mathbf{J}$. The *sensitivity matrix* **J** is defined as:

$$\mathbf{J}(\mathbf{P}) = \left[\frac{\partial \mathbf{T}^{T}(\mathbf{P})}{\partial \mathbf{P}}\right]^{T} = \begin{bmatrix} \frac{\partial T_1}{\partial k^*} & \frac{\partial T_1}{\partial C^*} \\ \frac{\partial T_2}{\partial k^*} & \frac{\partial T_2}{\partial C^*} \\ \vdots & \vdots \\ \frac{\partial T_I}{\partial k^*} & \frac{\partial T_I}{\partial C^*} \end{bmatrix} \tag{5}$$

The elements of the sensitivity matrix are denoted as the *sensitivity coefficients*. They provide a measure of the sensitivity of the estimated (or measured) temperatures with respect to changes in the unknown parameters. Clearly, the solution of inverse problems involving sensitivity coefficients with small magnitudes is extremely difficult, because the choice of very different values for the unknown parameters would result in basically the same value for the measured variables. Also, the columns of the sensitivity matrix are required to be linearly independent in order to have the matrix $\mathbf{J}^T\mathbf{J}$ invertible, that is, the determinant of $\mathbf{J}^T\mathbf{J}$ cannot be zero or even very small.

3.2. The Sequential Parameter Estimation Technique

We also examine in this paper a second estimation procedure, where the parameters are estimated by using the transient measurements Y_i, for t_i, i = 1,…,I, sequentially in time (Beck and Arnold, 1977, Beck, 1999).

The starting point for the *Sequential Parameter Estimation Technique* advanced by Beck and Arnold (1977) and by Beck (1999) is the minimization of the *Maximum a Posteriori* objective function. Such objective function, for the estimation of the vector of unknown parameters **P** = [k*,C*], is defined as (Beck and Arnold, 1977):

$$S(\mathbf{P}) = [\mathbf{Y} - \mathbf{T}(\mathbf{P})]^{T}\mathbf{W}[\mathbf{Y} - \mathbf{T}(\mathbf{P})] + (\boldsymbol{\mu} - \mathbf{P})^{T}\mathbf{V}^{-1}(\boldsymbol{\mu} - \mathbf{P}) \tag{6.a}$$

where **W** is a weighting matrix and

$$[\mathbf{Y} - \mathbf{T}(\mathbf{P})]^{T} = [Y_1 - T_1(\mathbf{P}), Y_2 - T_2(\mathbf{P}), \ldots, Y_I - T_I(\mathbf{P})] \tag{6.b}$$

is the vector containing the differences between measured (Y_i) and estimated (T_i) temperatures.

The use of the *maximum a posteriori* objective function involves the following statistical assumptions (Beck and Arnold, 1977):

- the errors are additive and normally distributed with zero mean;
- the statistical parameters describing the errors are known;
- there are no errors in the independent variables, such as time;
- **P** is a random vector with known mean $\boldsymbol{\mu}$ and known covariance matrix **V**. **P** is distributed normally and **P** and **V** are uncorrelated.

We note that the above hypotheses do not involve any assumptions regarding the errors being uncorrelated or not, and the covariance matrix of the errors being constant or not. Also, note that prior information available for the parameters can be taken into account on the inverse analysis, through the vector $\boldsymbol{\mu}$ and the covariance matrix **V**.

The minimization of S(**P**) requires that its gradient be null. Thus,

$$\nabla S(\mathbf{P}) = -2\mathbf{J}^{T}\mathbf{W}[\mathbf{Y} - \mathbf{T}] - 2\mathbf{V}^{-1}(\boldsymbol{\mu} - \mathbf{P}) = 0 \tag{7}$$

By linearizing the vector of estimated temperatures with a Taylor series expansion around the estimated parameters at iteration k, that is,

$$\mathbf{T}(\mathbf{P}) = \mathbf{T}(\mathbf{P}^{k}) + \mathbf{J}^{k}(\mathbf{P} - \mathbf{P}^{k}) \tag{8}$$

we can write an iterative procedure for the estimation of the parameters **P** in the form (Beck and Arnold, 1977):

$$\mathbf{P}^{k+1} = \mathbf{P}^{k} + [\mathbf{J}^{T}\mathbf{W}\mathbf{J} + \mathbf{V}^{-1}]^{-1}\{\mathbf{J}^{T}\mathbf{W}[\mathbf{Y} - \mathbf{T}(\mathbf{P}^{k})] + \mathbf{V}^{-1}(\boldsymbol{\mu} - \mathbf{P}^{k})\} \tag{9}$$

where **J** is the *sensitivity matrix* defined by Eq. (5) above. For convenience in the analysis, we rewrite the sensitivity matrix here as

$$\mathbf{J}(\mathbf{P}) = \begin{bmatrix} \mathbf{J}_1 \\ \mathbf{J}_2 \\ \vdots \\ \mathbf{J}_I \end{bmatrix} \tag{10.a}$$

where

$$\mathbf{J}_i = \left[\frac{\partial T_i}{\partial k^*}, \frac{\partial T_i}{\partial C^*} \right] \tag{10.b}$$

If we make the additional assumption that the measurement errors are uncorrelated, the weighting matrix is given by

$$\mathbf{W} = \begin{bmatrix} W_1 & 0 & \cdots & 0 \\ 0 & W_2 & & \vdots \\ \vdots & & \ddots & 0 \\ 0 & \cdots & 0 & W_I \end{bmatrix} \tag{11.a}$$

where

$$W_i = 1/\sigma_i^2 \quad \text{for} \quad i = 1,\ldots,I \tag{11.b}$$

and σ_i is the standard-deviation of the measurement Y_i.

For the sequential nonlinear estimation, such as the one under picture in this paper, Beck and Arnold (1977) recommend that the parameters be initially estimated by using all measurements simultaneously. Afterwards, the problem is solved once more, this time sequentially, by using the parameters estimated simultaneously and its covariance matrix in the place of $\boldsymbol{\mu}$ and **V**, respectively.

In order to apply the sequential estimation approach, the linearization is performed around $\mathbf{P}^k$, which is taken as

$$\begin{aligned} \mathbf{P}^{0} &= \boldsymbol{\mu} \quad \text{for } k = 0 \\ \mathbf{P}^{k} &= \mathbf{P}_I^{k} \quad \text{for } k = 1,2,... \end{aligned} \tag{12}$$

where $\mathbf{P}_I^k$ is the vector with the values estimated sequentially for the parameters at iteration k, obtained by using all I measurements.

The main steps for the computational algorithm of the sequential estimation approach can be organized as follows:

Step 1. Initialize the procedure with k = 0 and

$$\mathbf{P}^{0} = \boldsymbol{\mu} \tag{13.a}$$

$$\mathbf{C}^{0} = \mathbf{V}^{-1} \tag{13.b}$$

$$\mathbf{D}^{0} = \mathbf{V}^{-1}(\boldsymbol{\mu} - \mathbf{P}^{k}) \tag{13.c}$$

Step 2. Compute the estimate for the vector of unknown parameters sequentially, for i = 1,…,I with

$$\mathbf{P}_i^{k+1} = \mathbf{P}^{k} + \mathbf{C}_i^{-1}\mathbf{D}_i \tag{14.a}$$

where

$$\mathbf{C}_i = \mathbf{C}_{i-1} + \mathbf{J}_i^{T}\mathbf{W}_i\,\mathbf{J}_i \tag{14.b}$$

$$\mathbf{D}_i = \mathbf{D}_{i-1} + \mathbf{J}_i^T \mathbf{W}_i [\mathbf{Y}_i - \mathbf{T}_i(\mathbf{P}^k)] \tag{14.c}$$

Step 3. Check convergence with the values estimated sequentially with all I measurements, that is,

$$\left\| \mathbf{P}_I^{k+1} - \mathbf{P}_I^k \right\| < \varepsilon \tag{15.a}$$

If the criterion given by Eq. (15.a) is not satisfied, increment k, make

$$\mathbf{P}^k = \mathbf{P}_I^k \tag{15.b}$$

and return to **Step 2**.

The above computational algorithm is not in a suitable form for computational implementation. A more convenient form can be obtained by writing the sequential estimation explicitly, that is, the estimate for the vector of parameters $\mathbf{P}_i^{k+1}$, obtained with measurements up to time t_i at iteration k+1, is obtained directly from the estimate obtained with measurements up to time t_{i-1} at the same iteration, $\mathbf{P}_{i-1}^{k+1}$, instead of $\mathbf{P}^k$ as in Eq. (14.a).

In order to derive such alternative form for the sequential estimation procedure we rewrite Eq. (14.a) for the $(k+1)^{th}$ iteration, with measurements up to time i+1, as:

$$\mathbf{P}_{i+1}^{k+1} = \mathbf{P}^k + [\mathbf{J}_{i+1}^T \mathbf{W}_{i+1} \mathbf{J}_{i+1} + \mathbf{C}_i]^{-1} \{\mathbf{J}_{i+1}^T \mathbf{W}_{i+1} [\mathbf{Y}_{i+1} - \mathbf{T}_{i+1}(\mathbf{P}^k)] + \mathbf{D}_i\} \tag{16.a}$$

or, alternatively,

$$[\mathbf{J}_{i+1}^T \mathbf{W}_{i+1} \mathbf{J}_{i+1} + \mathbf{C}_i][\mathbf{P}_{i+1}^{k+1} - \mathbf{P}^k] = \mathbf{J}_{i+1}^T \mathbf{W}_{i+1} [\mathbf{Y}_{i+1} - \mathbf{T}_{i+1}(\mathbf{P}^k)] + \mathbf{D}_i \tag{16.b}$$

By subtracting $[\mathbf{J}_{i+1}^T \mathbf{W}_{i+1} \mathbf{J}_{i+1} + \mathbf{C}_i]\mathbf{P}_i^{k+1}$ from both sides of Eq. (16.b) and after performing some algebraic manipulations we obtain:

$$\mathbf{P}_{i+1}^{k+1} = \mathbf{P}_i^k + \mathbf{V}_{i+1} \mathbf{J}_{i+1}^T \mathbf{W}_{i+1} \{[\mathbf{Y}_{i+1} - \mathbf{T}_{i+1}(\mathbf{P}^k)] - \mathbf{J}_{i+1} [\mathbf{P}_i^{k+1} - \mathbf{P}^k]\} \tag{17.a}$$

where

$$\mathbf{V}_{i+1} = [\mathbf{J}_{i+1}^T \mathbf{W}_{i+1} \mathbf{J}_{i+1} + \mathbf{C}_i]^{-1} \tag{17.b}$$

$\mathbf{V}_{i+1}$ is the covariance matrix for the linear *maximum a posteriori* estimator using i+1 measurements, which is used as an approximation for the nonlinear estimator (Beck and Arnold, 1977).

By using the following matrix identities (Beck and Arnold, 1977):

$$\begin{aligned} \mathbf{V}_{i+1} &= \mathbf{V}_i - \mathbf{V}_i \mathbf{J}_{i+1}^T (\mathbf{J}_{i+1} \mathbf{V}_i \mathbf{J}_{i+1}^T + \mathbf{W}_{i+1}^{-1})^{-1} \mathbf{J}_{i+1} \mathbf{V}_i \\ \mathbf{V}_{i+1} \mathbf{J}_{i+1}^T \mathbf{W}_{i+1} &= \mathbf{V}_i \mathbf{J}_{i+1}^T (\mathbf{J}_{i+1} \mathbf{V}_i \mathbf{J}_{i+1}^T + \mathbf{W}_{i+1}^{-1})^{-1} \end{aligned} \tag{18.a,b}$$

where Eq. (18.a) is referred to as the *Matrix Inversion Lemma* (Beck and Arnold, 1977, Beck, 1999), we can write the following computational algorithm for the sequential estimation approach:

Step 1. Initialize the procedure with k = 0 and

$$\mathbf{P}^0 = \boldsymbol{\mu} \tag{19}$$

Step 2. Compute the estimate for the vector of unknown parameters sequentially, for i = 0,…,I-1 with

$$\mathbf{A} = \mathbf{V}_i \mathbf{J}_{i+1}^T \tag{20.a}$$

$$\boldsymbol{\Delta} = \mathbf{J}_{i+1} \mathbf{A} + \mathbf{W}_{i+1}^{-1} \tag{20.b}$$

$$\mathbf{K} = \mathbf{A} \boldsymbol{\Delta}^{-1} \tag{20.c}$$

$$\mathbf{E}_{i+1} = \mathbf{Y}_{i+1} - \mathbf{T}_{i+1}(\mathbf{P}^k) \tag{20.d}$$

$$\mathbf{P}_{i+1}^{k+1} = \mathbf{P}_i^{k+1} + \mathbf{K}[\mathbf{E}_{i+1} - \mathbf{J}_{i+1}(\mathbf{P}_i^{k+1} - \mathbf{P}^k)] \tag{20.e}$$

$$\mathbf{V}_{i+1} = \mathbf{V}_i - \mathbf{K}\,\mathbf{J}_{i+1}\mathbf{V}_i \quad (20.f)$$

where

$$\mathbf{V}_0 = \mathbf{V} \quad (20.g)$$

$$\mathbf{P}_0^k = \boldsymbol{\mu} \quad (20.h)$$

Step 3. Check convergence with the values estimated sequentially with all I measurements, that is,

$$\left\| \mathbf{P}_I^{k+1} - \mathbf{P}_I^k \right\| < \varepsilon \quad (21.a)$$

If the criterion given by Eq. (21.a) is not satisfied, increment k, make

$$\mathbf{P}^k = \mathbf{P}_I^k \quad (21.b)$$

and return to step 2.

A quite important computational feature of the above algorithm is that, if one measurement is added at a time, such as for the case involving transient measurements of a single sensor of this work, no matrix inversion is performed because $\boldsymbol{\Delta}$ and $\mathbf{W}_{i+1}$ are scalars. In fact, even if transient measurements of multiple sensors are used in the analysis, they can be arranged so that one single measurement is added to the sequential estimation procedure at a time, so that no matrix inversions need to be performed.

The above computational algorithm was derived for a case where previous estimates were available for the vector of parameters and for their covariance matrix, obtained by using all measurements simultaneously, i.e., not sequentially. However, it can also be used for cases where no previous estimations are available, or if available, they have large uncertainty. For such cases, we take $\boldsymbol{\mu}$ as any vector, say, with null components. Also, we take **V** as a diagonal matrix with large values on the diagonal as compared to the square of the expected values for the parameters.

3.3. Statistical analysis

After the minimization of the objective function, given either by Eq. (3.a) or Eq. (6.a), a statistical analysis can be performed in order to obtain confidence intervals for the estimated parameters. Confidence intervals at the 99% confidence level are obtained as (Beck and Arnold, 1977, Ozisik and Orlande, 2000):

$$\hat{P}_j - 2.576\sigma_{\hat{P}_j} \le P_j \le \hat{P}_j + 2.576\sigma_{\hat{P}_j} \quad (22.a)$$

where $\hat{P}$ are the values estimated for the unknown parameters, and $\sigma_{\hat{P}}$ are the standard deviations for the unknown parameters, given by

$$\sigma_{\hat{P}_j} = \sqrt{V_{jj}} \quad (22.b)$$

V_{jj} is the j^{th} element in the diagonal of the covariance matrix for the estimated parameters, **V**. Expressions can be obtained for such matrix in linear estimation problems, by using either the ordinary least-squares norm or the *maximum a posteriori* objective function (Beck and Arnold, 1977). These linear expressions can be approximately used for non-linear cases, such as the one of this work. The covariance matrix of the estimated parameters for the ordinary least-squares norm and for the *maximum a posteriori* objective function are given, respectively, by:

$$\mathbf{V} = (\mathbf{J}^T\mathbf{J})^{-1}\sigma^2, \quad \text{for the ordinary least-squares norm} \quad (22.c)$$

$$\mathbf{V} = (\mathbf{J}^T\mathbf{W}\mathbf{J} + \mathbf{V}^{-1})^{-1}, \quad \text{for the } \textit{maximum a posteriori} \text{ objective function} \quad (22.d)$$

4. Results and discussions

We present below results obtained for the simultaneous estimation of the thermal conductivity and volumetric heat capacity of Teflon, by using the experimental setup and the estimation techniques discussed above. This material was chosen because of its applications in the space industry (Rey Silva and Orlande, 2001) and because there were tabulated values for the physical properties of interest here. We examined three different levels of heat flux applied to the specimens, as shown in Tab. (1). The voltage applied to the heater in each experiment is also shown in Tab. (1). For all three cases considered, the heating time was taken as t_h = 232 s and the final experimental time as t_f = 323 s (Oliveira et al, 1999).

Table 1. Applied voltage and generated heat flux in the experiments.

Experiment	Applied voltage (volts)	Theoretical heat flux (W/m^2)
#1	100 ± 1.5%	4664 ± 147
#2	90 ± 1.5%	3778 ± 131
#3	70 ± 1.5%	2285 ± 100

Preliminary results, obtained for the estimated parameters with the experimental data of the three experimental runs shown in Tab. (1), were completely inaccurate. It was found that this behavior was caused by large uncertainties on the actual functional form of the applied heat flux, which was assumed as a step function in the mathematical model described above. It happens that, because of the large mass of the heater, the applied heat flux was actually very different from such step function. As a result, the heat generated in the electrical resistance was not instantaneously conducted into the specimen. Such behavior was also observed by Lima e Silva et al (1999) and Lima (2001).

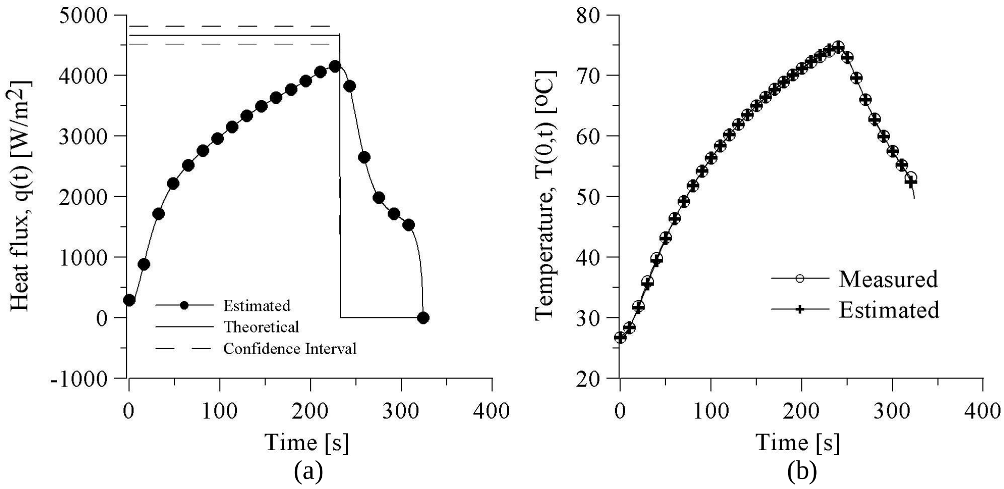

Figure 3. Experiment #1. (a) Estimated heat flux. (b) Measured and estimated temperatures.

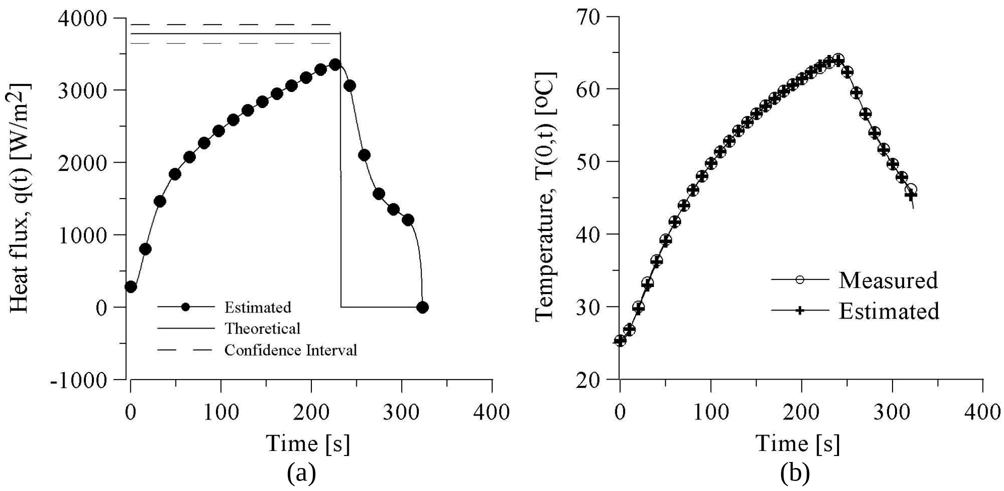

Figure 4. Experiment #2. (a) Estimated heat flux. (b) Measured and estimated temperatures.

By using tabulated values for the thermal properties of Teflon, $k = 0.35$ W/(m ^{o}C), $\rho = 2200$ kg/m^3 and $c_P = 1050$ J/(kg ^{o}C) (Çengel, 1998), we used a procedure based on the conjugate gradient method of function estimation with adjoint problem (Ozisik and Orlande, 2000), in order to estimate the actual applied heat flux at the surface of the specimen. Figures (3), (4) and (5) present the estimated heat fluxes, as well as a comparison between the estimated and measured temperatures, for each of the experimental runs, respectively. Note in Figs. (3a), (4a) and (5a) that the actual applied heat flux is quite different from the theoretical step function, for all three cases considered. Note the small rate of increase in the heat flux when the heater is turned on, as well as the small rate of decrease of the heat flux when the heater is turned off, because of the large thermal capacity of the heater, which is too thick for this application. It is interesting to note that, generally, the functional form of the actual applied heat flux is not affected by the applied voltage to the heater. Also, note that the peak-value for the actual heat flux was smaller than the theoretical value for each of the experimental runs. Estimated and measured temperatures are in excellent agreement, as shown by Figs. (3b), (4b) and (5b).

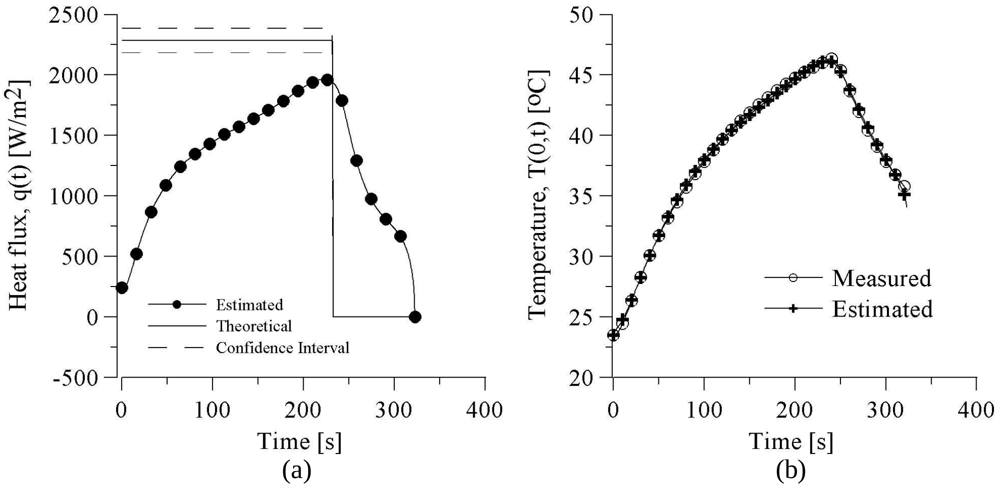

Figure 5. Experiment #3. (a) Estimated heat flux. (b) Measured and estimated temperatures.

We now use the temperature measurements, together with the estimated heat fluxes shown in Figs. (3)-(5), in order to estimate the tabulated values of thermal conductivity and volumetric heat capacity of Teflon. Table (2) presents the values obtained with the two estimation techniques examined in this work, for the three experimental runs. Confidence intervals for the estimated parameters, at the 99% confidence level, are also shown in this table. The standard-deviation of the measurement errors, obtained during the calibration of the measurement system, was 0.31 ^{o}C. Such standard-deviation was necessary to compute the confidence intervals for the estimated parameters, as given by Eqs. (22.a-d). For the sequential estimation technique, the vector $\boldsymbol{\mu}$ was taken as a vector with small components, and the covariance matrix **V** was taken as a diagonal matrix with large values on the diagonal as compared to the square of the expected values for the parameters. Note that no improvement was obtained for the estimated parameters with the sequential estimation technique.

Table 2. Estimated parameters.

Experiment	Parameter	Levenberg-Marquardt		Sequential Estimation	
		Estimated Parameters	Confidence Intervals	Estimated Parameters	Confidence Intervals
#1	k* [W/(m ^{o}C)] x 10^{-1}	3.497	(3.491 ; 3.503)	3.497	(3.491 ; 3.503)
	C* [J/(m^{3} ^{o}C)] x 10^{6}	2.32	(2.30 ; 2.34)	2.32	(2.30 ; 2.34)
#2	k* [W/(m ^{o}C)] x 10^{-1}	3.497	(3.489 ; 3.504)	3.497	(3.489 ; 3.504)
	C* [J/(m^{3} ^{o}C)] x 10^{6}	2.32	(2.30 ; 2.34)	2.32	(2.30 ; 2.34)
#3	k* [W/(m ^{o}C)] x 10^{-1}	3.50	(3.48 ; 3.51)	3.50	(3.48 ; 3.51)
	C* [J/(m^{3} ^{o}C)] x 10^{6}	2.34	(2.30 ; 2.37)	2.34	(2.30 ; 2.37)

The values estimated for each parameter, with the corresponding 99% confidence level, are shown in Figs. (6a,b), for each of the experimental runs. Note in these figures the larger confidence interval for experiment #3. Also, note that the values estimated for the parameters differ mostly from the tabulated values for this experimental run. This is probably because of the smaller magnitude of the applied heat flux for this case, which result on smaller increases on the measured temperatures (see Figs. (3b), (4b) and (5b)). Therefore, the measurement errors are more significant for experimental run #3 than for the other runs.

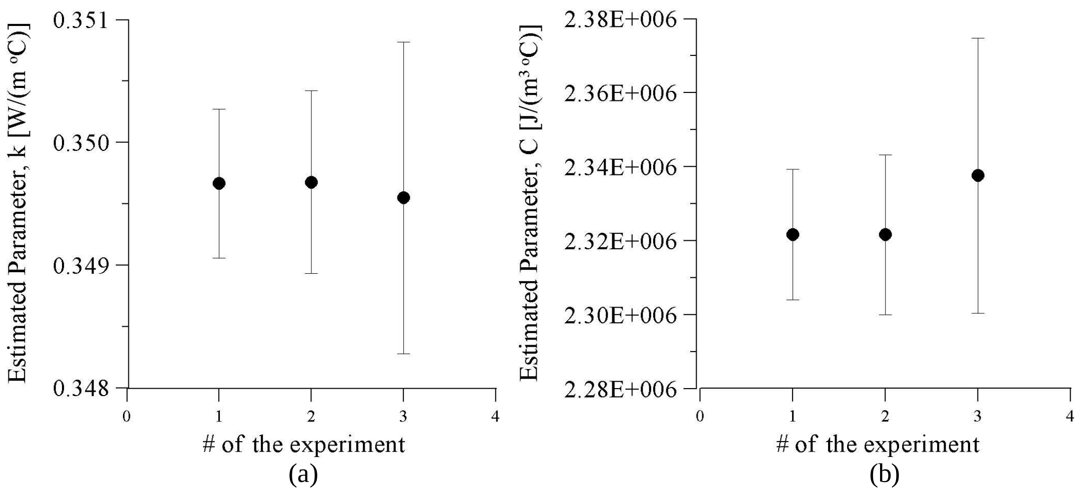

Figure 6. Estimated parameters with 99% confidence intervals when using Levenberg-Marquardt.
(a) Thermal conductivity. (b) Volumetric heat capacity.

Figures (7a-c) present the residuals between measured and estimated temperatures, for each of the experimental runs, respectively. Note the small values of the residuals, as compared to the magnitude of the measured temperatures shown in Figs. (3b), (4b) and (5b). On the other hand, note in Figs. (7a-c) that the residuals are highly correlated. This can be due to inconsistencies between the mathematical model and the actual physical problem, such as the constant and uniform temperature boundary condition for the non-heated surface, or the uniform heating. This can also be due to the correlation of the measurements, which was not taken into account for the estimation procedures examined in this work.

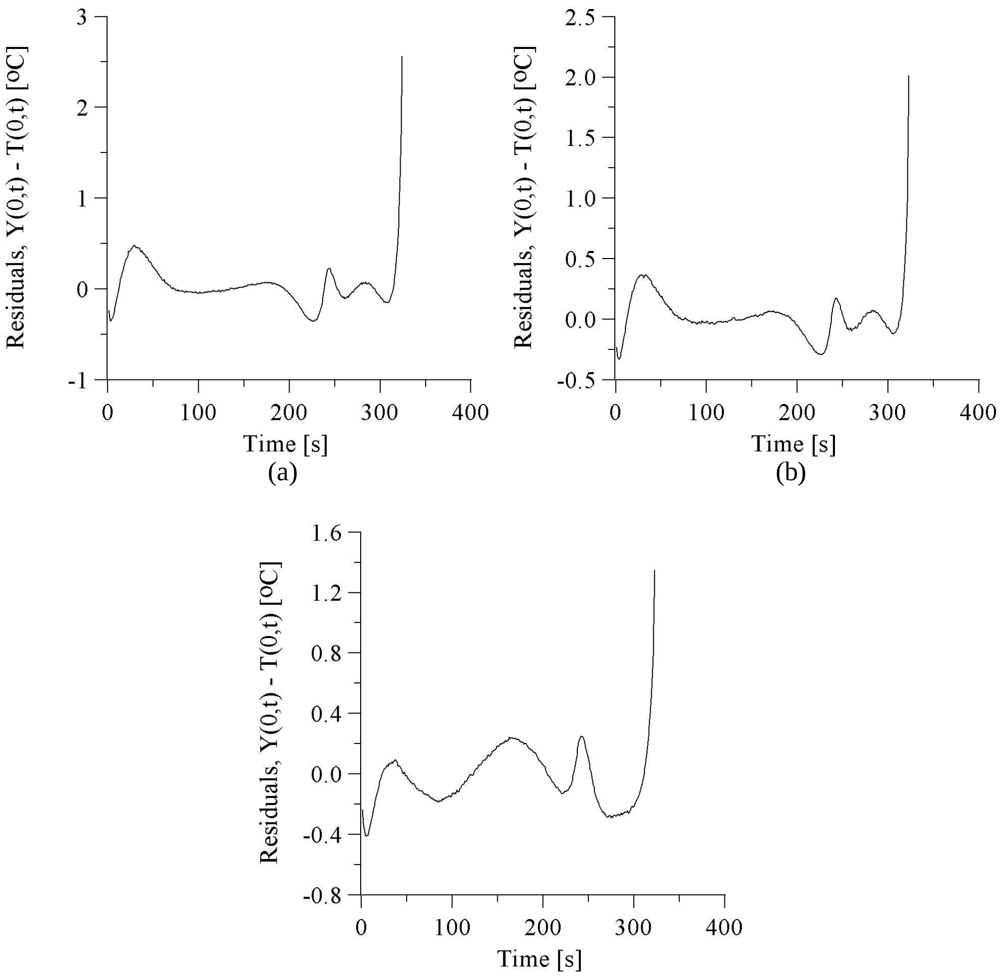

Figure 7. Residuals for the estimation of the unknown properties with the Levenberg-Marquardt method.
(a) Experiment #1. (b) Experiment #2. (c) Experiment #3.

The sequential estimation of the unknown parameters is illustrated in Figs. (8a,b), for experimental run #1. Note in these figures that, after some oscillations for small times, the parameters converge fast to the tabulated values. Therefore, the duration of the experiment, as well as the number of transient measurements used in the inverse analysis, is appropriate to obtain accurate estimations for the parameters.

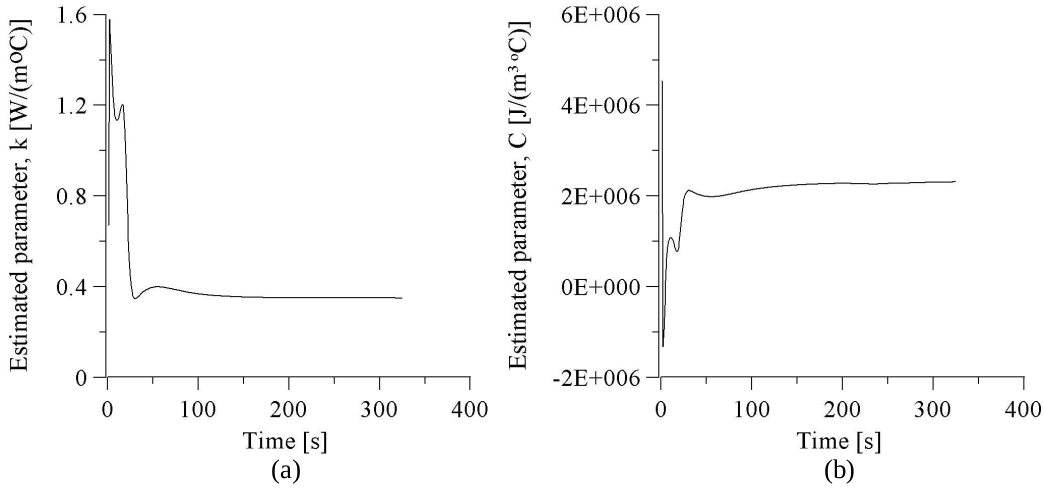

Figure 8. Sequential Estimation of the parameters for experiment #1.

5. Conclusions

In this paper, we presented preliminary results obtained for the simultaneous estimation of thermal conductivity and volumetric heat capacity of solids, with an experimental apparatus developed in LTTC/COPPE/UFRJ. Such apparatus consisted of two identical specimens of the material with unknown properties, with a heater sandwiched between them. The preliminary results were obtained for Teflon, because of known tabulated values for the thermophysical properties of interest here. The properties were identified by using two different estimation procedures, namely: the Levenberg-Marquardt Method and the Sequential Parameter Estimation Technique.

The results shown above reveal that the thermal capacity of the heater used was very large. As a result, the applied heat flux was quite different from the theoretical one. By using the estimated heat flux instead of the theoretical step-function, we were able to obtain accurate estimations for the unknown properties, which were compared to the tabulated values encountered in the literature.

We are currently changing the heater to one with smaller mass, which can provide heat fluxes closer to the theoretical step-function. We are also using other experimental techniques for the identification of thermal conductivity and volumetric heat capacity, in order to compare to the one now under development.

6. Dedication

This work is dedicated to the memory of Professor Roberto de Souza, for his enthusiasm, dedication and substantial achievements in the development of experimental apparatus and techniques at the Laboratory of Heat Transmission and Technology of PEM/COPPE. Professor Roberto de Souza was responsible for establishing the first steps towards the development of the experimental apparatus presented in this work.

7. References

Beck, J.V. and Arnold, K.J., 1977, "Parameter estimation in engineering and science", Wiley, New York.

Beck, J.V., 1999, "Sequential methods in parameter estimation" (Tutorial Session), Proceedings of the 3rd International Conference on Inverse Problems in Engineering, Port Ludlow, WA, USA.

Çengel, Y.A., 1998, "Heat transfer: a practical approach", McGraw-Hill, USA.

Dowding, K., Beck, J., Ulbrich, A., Blackwell, B. and Hayes, J., 1995, "Estimation of thermal properties and surface heat flux in carbon-carbon composite", Journal of Thermophysics and Heat Transfer, Vol. 9, No. 2, pp. 345-351.

Dowding, K.J., Beck, J.V. and Blackwell, B.F., 1996, "Estimation of directional-dependent thermal properties in a carbon-carbon composite", International Journal of Heat and Mass Transfer, Vol. 39, No. 15, pp. 3157-3164.

Guimarães, G., Teodoro, E.B. and Philipi, P.C., 1997, "On optimizing the estimation of thermophysical properties", Journal of the Brazilian Society of Mechanical Sciences, Vol. XIX, No. 1, pp. 15-29.

Lima, F.R.S., 2001, "Modelagem tridimensional de problemas inversos em condução de calor: aplicação em processos de usinagem, Tese de Doutorado, Faculdade de Engenharia Mecânica, Universidade Federal de Uberlândia.

Lima e Silva, S.M.M., Guimarães, G., Guths, S., Laurent, M., Raynaud, M., 1999, "Determinação da difusividade térmica e da condutividade térmica de materiais metálicos utilizando técnicas de otimização", Proceedings of the 15th Brazilian Congress of Mechanical Engineering, November 22-26, Águas de Lindóia, SP, Brazil.

Mejias, M.M., Orlande, H.R.B. and Ozisik M.N., 1999, "Design of optimum experiments for the estimation of thermal conductivity components of orthotropic solids", Hybrid Methods in Engineering – Modeling, Programming, Analysis, Animation, Vol. 1, No. 1, pp. 37-53.

Oliveira, A.P., Rey Silva, D.V.F.M., Orlande, H.R.B. and Souza, R., 1999, "Estimation of thermal conductivity and volumetric heat capacity in transient experiments", Proceedings of the 15th Brazilian Congress of Mechanical Engineering, November 22-26, Águas de Lindóia, SP, Brazil.

Orlande, H.R.B. and Ozisik, M.N., 1994, "Simultaneous estimation of thermal diffusivity and relaxation time with a hyperbolic heat conduction model", Proceedings of the 10th International Heat Transfer Conference, Paper 15-CI-20, Vol. 7, pp. 403-408.

Orlande, H.R.B., Ozisik, M.N. and Tzou, D.Y., 1995, "Inverse analysis for estimating the electron-phonon coupling factor in thin metal films", Journal of Applied Physics, Vol. 78, No. 3, pp. 1843-1849.

Ozisik, M.N. and Orlande, H.R.B., 2000, "Inverse heat transfer: fundamentals and applications", Taylor and Francis, Pennsylvania.

Ozisik, M.N., 1993, "Heat Conduction", 2nd ed., Wiley, New York.

Rey Silva, D.V.F.M., Oliveira, A.P., Orlande, H.R.B. and Souza, R., 2000, "Sequential estimation of thermal conductivity and volumetric heat capacity", Proceedings of the 8th Brazilian Congress of Thermal Engineering and Sciences, ENCIT 2000, Porto Alegre, RS, Brazil.

Rey Silva, D.V.F.M. and Orlande, H.R.B., 2001, "Estimation of thermal properties of ablating materials", Proceedings of the International Symposium on Inverse Problems in Engineering 2001, Nagano, Japan.

Taktak, R., Beck, J.V. and Scott, E.P., 1993, "Optimal experimental design for estimating thermal properties of composite materials", International Journal of Heat and Mass Transfer, Vol. 36, No. 12, pp. 2977-2986.