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EXP04

AN INVERSE ANALYSIS FOR POLYMERS THERMAL PROPERTIES ESTIMATION

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ABSTRACT

An approach combining the use of the hot-wire method with the solution of heat conduction inverse problems is proposed for the thermal characterization of new polymeric materials.

Due to the low thermal conductivity of the material, the cross technique is used instead of the parallel technique. The inverse heat conduction problem has been solved as a finite dimensional optimization problem with the Levenberg-Marquardt method.

Results are presented for phenolic foams with lignin thermal conductivity and diffusivity estimation from a real set of experimental data.

INTRODUCTION

A considerable amount of effort has been devoted for the fulfillment of the ever-growing demand for new materials with relevant applications in engineering and medicine. The recent trend has been the development of specific materials for new applications, with the latter coming first. It becomes mandatory, then, the use of the adequate techniques to characterize the new materials developed, through the determination of their properties.

Besides that, the accurate modeling (physical, mathematical and computational) of systems and components of engineering interest, relies heavily on the use of the proper values for the material properties.

For applications involving heat transfer by conduction, the thermal conductivity and diffusivity are the most relevant properties to be considered. Shai et al. (1993) measured the thermal diffusivity of solids, and Trevisan et al. (1993) measured the same property for saturated porous media. Carvalho et al. (1996) measured the thermal conductivity of polymers with the hot-wire method. This method was described in 1888 by Schiermacher, but its first practical application was reported in 1949 by Van der Held and Van Drunen, who used it to measure the thermal conductivity of liquids (Davis, 1984).

Experimentalists use a lot of engineering skills trying to control several degrees of freedom during the development and operation of experimental apparatus and techniques to determine the required material properties. Combining the experimental activities with the solution of heat conduction inverse problems, a larger number of degrees of freedom may be taken into account, even allowing the simultaneous estimation of new unknowns brought into the problem by the more accurate mathematical modeling.

Using the solution of inverse conduction problems Mikhalev and Reznik (1989) estimated the temperature dependence of the thermal conductivity of orthotropic materials, and Cheng and Zang (1994) estimated the spatially dependent thermal conductivity in optically opaque solids. Artyukhin (1976, 1982, 1982a), Tervola (1989) and Artyukhin et al. (1984, 1993) have used the expansion of the temperature dependence of unknown thermal conductivity, or thermal diffusivity, in known functions such as polynomials or splines, reducing the inverse problem of function estimation to that of determining the expansion coefficients.

One of the most relevant applications of the inverse analysis is related to the design of experimental apparatus and techniques as well as the optimal use of existing equipment or experimental data already acquired (Artyukhin and Okhapkin, 1984; Emery et al., 1993). The approach involving the solution of inverse problems is well fit to perform those tasks. In some of the techniques developed, sensitivity coefficients have to be calculated during the procedure used for the solution of the inverse problem (Beck et al., 1985), and the confidence bounds for the estimated values are related to these sensitivity coefficients. Going one step further, the criteria used for the design of optimal experiments may be related to the minimization of the confidence region (Taktak et al., 1993). Therefore, through this approach a significant gain in

experimental simplicity and productivity can be achieved (Goryachev and Yudin, 1983).

In this work we present the first results of our efforts towards the physical, mathematical and computational modeling of the hot-wire method for the estimation of new polymeric materials thermal properties, developing an improved use of the method through the solution of inverse heat conduction problems.

The hot-wire method has been successfully used for ceramic materials thermal conductivity determination, being now the worldwide standard for values up to 25 W/m^{o}C (Santos et al., 1995). Its application to polymers had not been demonstrated until 1989 (Thompson, 1989), and it seems to have been addressed for the first time by Carvalho et al. (1996). For polymeric materials the hot-wire parallel technique has been replaced by the cross technique, where a thermocouple junction is welded to the hot-wire that works as the heat source located in the middle of the material sample whose properties are being determined.

We proceed now with a brief description of the hot-wire method followed by the description of the inverse problem approach. Results for a test case with real experimental data are also presented.

THE TRADITIONAL EXPERIMENTAL APPROACH (HOT-WIRE TECHNIQUE)

In Fig.1 is shown an schematic representation of the experimental apparatus for the hot-wire cross technique used in polymeric materials thermal conductivity measurement. An electric resistance (hot-wire) is embedded in a sample of the material. As an electric current of fixed intensity flows through the wire, the electric resistance heats up and the sample of the polymeric material around it works as an insulator. From the transient measurements of the wire temperature, the thermal conductivity of the sample is determined.

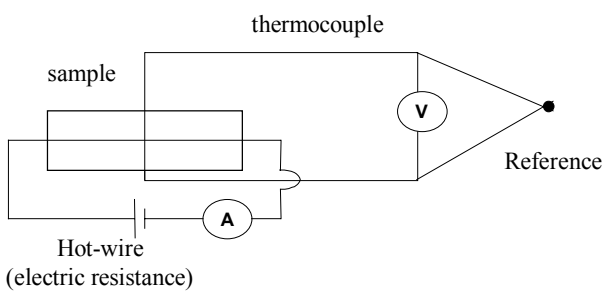

Figure 1 – Schematic representation of the experimental apparatus.

In the traditional experimental approach, the following simplified model is constructed: consider an infinite line source of constant intensity embedded in an infinite medium initially at the temperature T_{amb}, that at time t=0 starts to release its energy. At sufficiently long times and/or small radial distances from the wire, r, the excess temperature, θ, presents the following time dependence (Bejan, 1993)

$$\theta(t) = T(t) - T_{amb} \propto \frac{q'}{4\pi k} \ln(t) \qquad (1)$$

where T is the temperature, k is the thermal conductivity and q' is the source linear power density. Due to the linearity in Eq.(1), if we take two temperature measurements at t_1 and t_2, the slope of the line in a plot θ x ln t is

$$\text{slope} = \frac{\theta_2 - \theta_1}{\ln(t_2) - \ln(t_1)} = \frac{q'}{4\pi k} \qquad (2)$$

In a real experiment a set of noisy experimental data is collected, (t_i , θ_i), i = 1, 2, ..., N, with N >> 2. Therefore, the slope of the line that fits the data (ln t_i , θ_i), i = 1, 2, ..., N, is obtained using the least squares algorithm, yielding the sample thermal conductivity estimate k = q' / (4 π . slope) .

As the experimental samples are always of finite size, at a time t^* heat begins to be transferred to the ambient by natural convection. When that happens a deviation of the linearity predicted by Eq. (1) takes place. Therefore, experimental data has to be acquired only for $t < t^*$. For materials with high values for the thermal diffusivity ($\alpha = k / \rho c_p$ where ρ is the density and c_p is the heat capacity), t^* may become too small, consisting, on a severe limitation for the application of the hot-wire method.

As Eq.(1) is derived considering a constant thermal conductivity, the approach described here can not be used if this property presents a variation with temperature.

THE INVERSE PROBLEM APPROACH

Using a more accurate physical and mathematical modeling of the experimental apparatus, and stating the thermal properties estimation problem as an inverse problem, we are able to extract from the same experimental data given above, (t_i, θ_i), i = 1,2,..., N, not only the thermal conductivity, k, but also the heat capacity, c_p. An attempt may also be made in order to estimate other unknowns brought into the problem by the relatively more elaborate modeling. In this case one has to deal with further data quality and sufficiency concerns. Through a careful analysis of the model and/or a proper experiment design, an evaluation can be done on the adequacy of using the proposed approach for the estimation of additional unknowns. If temperature measurements are taken for $t > t^*$, we may even try to estimate the heat transfer coefficient from the sample to the ambient around it.

Consider a sample of finite size, i.e. radius R, with a line source at r=0, and long enough such that heat conduction can be considered only in the radial direction. The sample is initially at the same temperature of the ambient, $T = T_{amb}$, and the heat source, whose intensity may vary with time, g(t), starts to release its energy at t = 0.

The mathematical formulation of the physical situation described is given by

$$\frac{1}{r}\frac{\partial}{\partial r}\left(kr\frac{\partial T}{\partial r}\right)+g(t)=\rho c_p\frac{\partial T(r,t)}{\partial t} \quad \text{in } 0<r<R\text{, for } t>0 \tag{3a}$$

$$-k\frac{\partial T}{\partial r}=h(T-T_{amb}) \quad \text{in } r=R\text{, for } t>0 \tag{3b}$$

$$T(r,0)=T_{amb} \quad \text{in } 0\le r\le R\text{, for } t=0 \tag{3c}$$

where h is the heat transfer coefficient and the other symbols have already been defined.

When the geometry, the material properties, the intensity of the heat source, and the initial and boundary conditions are known, the temperature distribution, T(r, t), can be calculated. This is known as the direct problem. When any of these quantities, or a combination of them, are unknown, we have an inverse problem.

In the problem presented it may be of interest to estimate k and c_p , as well as h and g(t), being the latter a real concern during the period of data acquisition because in the traditional approach previously described, it must be kept constant. As already mentioned in this section, extreme care and proper analysis has to be used in order to perform such estimations. Furthermore, as a rule of thumb, we may say that it is best to estimate as few parameters as necessary.

As the real experimental data collected with the experiments conducted so far, have considered only measurements taken for $t < t^*$, we will focus in this work only on the estimation of the vector of unknowns

$$\underline{Z}=\{k,c_p\}^T \tag{4}$$

As the density is easily determined using other techniques, it is considered given. Therefore, with the estimation of k and c_p, the thermal diffusivity, α is also estimated. If any of these properties presents temperature dependence, say k (T), it can be approximated by a polynomial

$$k(T)=\sum_{l=0}^{L} a_l T^l \tag{5}$$

or any other set of known functions, reducing the problem of estimation in an infinite dimensional space to that of determining the set of coefficients a_l , l = 0, 1,..., L, in a finite dimensional space. The vector of unknowns would then be enlarged to take into account the new unknowns to be estimated

$$\underline{Z}^* = \{a_0, a_1, \ldots, a_L, c_p\}^T \tag{6}$$

A comment is in order. Other methods can be used for the function estimation directly, without requiring the parametrization given by Eq. (5). Silva Neto and Özisik (1994, 1994 a) used the Conjugate Gradient Method with an adjoint equation for the solution of inverse problems involving function estimation.

As previously said we will concentrate here on the estimation of the unknown vector given by Eq. (4), i.e. in this work no temperature dependence of the thermal properties will be taken into account.

THE SOLUTION OF THE INVERSE PROBLEM

To solve the inverse problem of estimating the vector of unknowns $\underline{Z}$ we have used the Levenberg–Marquardt method. Silva Neto and Özisik (1993,1995) have used this method for the solution of inverse problems involving radiative heat transfer as well as the combined mode conduction–radiation. The method will now be briefly described.

As the number of measured data, M, is larger than the number of parameters to be estimated, the problem is overdetermined. It can be solved, then, as a finite dimensional optimization problem in which we want to minimize the norm of squared residues

$$Q=\sum_{i=1}^{M}\left[T_i(k,c_p)-W_i\right]^2=\underline{F}^T\underline{F} \tag{7}$$

with the elements of vector $\underline{F}$ given by

$$F_i=T_i(k,c_p)-W_i,\ i=1,2,\ldots,M \tag{8}$$

where W_i , i = 1, 2, ..., M are the measured temperatures, $W_i = \theta_i + T_{amb}$, and T_i are the calculated temperatures. The index i represents the discretization of the time interval in which the temperature measurements are taken.

To minimize Q we differentiate Eq. (7) with respect to each of the unknown parameters yielding

$$\frac{\partial Q}{\partial \underline{Z}}=\frac{\partial}{\partial \underline{Z}}\left(\underline{F}^T\underline{F}\right)=0 \tag{9}$$

Using a Taylor's expansion, keeping only the first order terms,

$$\begin{aligned} F_i(\underline{Z}+\Delta\underline{Z}) &= F_i\big|_{\underline{Z}}+\sum_{j=1}^{L}\frac{\partial F_i}{\partial Z_j}\Big|_{\underline{Z}}\Delta Z_j \\ &= F_i\big|_{\underline{Z}}+\sum_{j=1}^{L}\frac{\partial T_i}{\partial Z_j}\Big|_{\underline{Z}}\Delta Z_j \quad ,\ i=1,2,\ldots,M \end{aligned} \tag{10}$$

and plugging it into Eqs. (9), we get

$$J^T J\,\Delta\underline{Z}=-J^T\underline{F} \tag{11}$$

where the Jacobian elements correspond to the sensitivity coefficients

$$J_{ij}=\frac{\partial T_i}{\partial Z_j},\ i=1,2,\ldots,M\ ,\ j=1,2,\ldots,L \tag{12}$$

Observe that in our case we have just two unknowns, i.e. L = 2. Therefore, $J^T J$ is a 2×2 matrix.

To help with convergence, a damping parameter λ is added to the diagonal of the matrix $J^T J$, leading to the Levenberg–Marquardt method (Marquardt, 1963),

$$\left(J^T J + I\lambda\right)\Delta \underline{Z} = -J^T \underline{F} \tag{13}$$

where I represents the diagonal matrix.

We are now in a position to describe the iterative procedure used for the inverse problem solution. Starting with an initial guess for the unknowns, $\underline{Z}^o$, new estimates are obtained

$$\underline{Z}^{n+1} = \underline{Z}^n + \Delta \underline{Z}^n \ , \ n = 0,1,2,\ldots \tag{14}$$

with the corrections ΔZ^n computed from Eq. (13) as

$$\Delta \underline{Z}^n = -[\left(J^n\right)^T J^n + I\lambda^n]\left(J^n\right)^T \underline{F}^n \tag{15}$$

The iterative procedure is interrupted when a stopping criterion established a priori is satisfied, e.g. $\|\Delta \underline{Z}^n\| < \varepsilon$ with ε being a small number.

The damping parameter is varied along the iterative procedure according to the algorithm proposed by Marquardt (1963).

At every step of the iterative procedure we have to calculate the temperatures at the positions where the temperature sensors are located (see Eq. (7)). This computation is done using Eqs. (3a–c) with the estimates obtained for the unknowns at each iteration. To solve the direct problem, Eqs. (3a–c), we have used the finite difference method with an explicit formulation

CONFIDENCE BOUNDS

Following a procedure discussed by Huang and Özisik(1990), the 99% confidence bounds for the estimates are $\underline{Z} \pm 2.576\ \sigma_{\underline{z}}$, with $\sigma_{\underline{z}}$ determined as

$$\sigma_{\underline{z}} = \sigma\left\{\mathrm{diag}\left[\left(\frac{\partial T^T}{\partial \underline{Z}}\right)\left(\frac{\partial T}{\partial \underline{Z}^T}\right)\right]^{-1}\right\}^{1/2} \tag{16}$$

RESULTS

We have already collected a large amount of experimental data for new polymeric materials, mainly for phenolic resins and foams with and without lignin. Lignin is obtained from sugar cane bagasse and may be used as a co-monomer in polymeric materials production (Carvalho,1997). All experimental data has been taken for times $t < t^*$, because so far the main focus had been the use of the hot-wire method solely for the thermal conductivity determination.

In Fig. 2 is shown one set of such experimental data, on a plot W_i x $\ln t_i$, for a phenolic foam with lignin. In this case a total of 26 temperature measurements were taken from t=60 s up to t=110 s with a regular 2 s interval between every two consecutive readings.

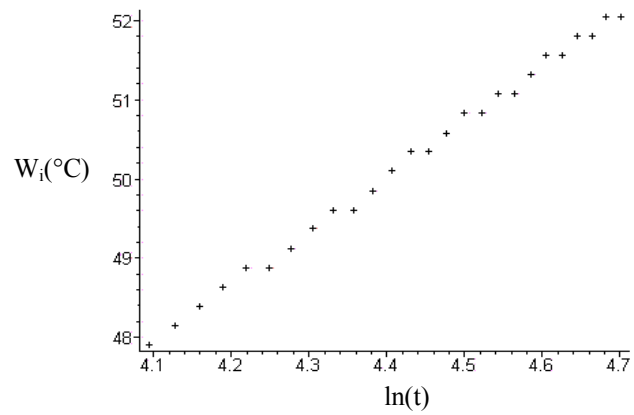

Figure 2 – Experimental data for a phenolic foam with lignin.

In the process of obtaining such experimental data the linear power density was 6.46 W/m obtained with an electric current of 0.975 A going through an electrical resistance of R/L =6.8 Ω/m.

In the traditional experimental approach, fitting a line to the experimental data with a least squares approach, we obtain the slope of the line and from Eq. (2) we calculate k = 0.072 ± 0.002 W/m °C.

Using the inverse problem approach as described before, we were able to estimate from the same set of experimental data not only the thermal conductivity but the heat capacity as well, with their respective confidence bounds as shown in Table 1.

Table 1 – Estimates and 99% confidence bounds obtained with the solution of the inverse problem for a phenolic foam with lignin.

Property	Unit	Estimate	Upper limit	Lower limit
k	W/m °C	0.07319	0.07325	0.07313
c_p	J/kg °C	1563.0	1566.4	1559.6

Note : No analysis of significant digits has been performed for the numerical results.

In Fig. 3 is presented a plot of temperature x time, with the temperatures being calculated with the estimated properties shown in Table 1. Superimposed, and represented by crosses, are the experimental data used as input for the inverse problem. These are the same data shown in Fig. 2.

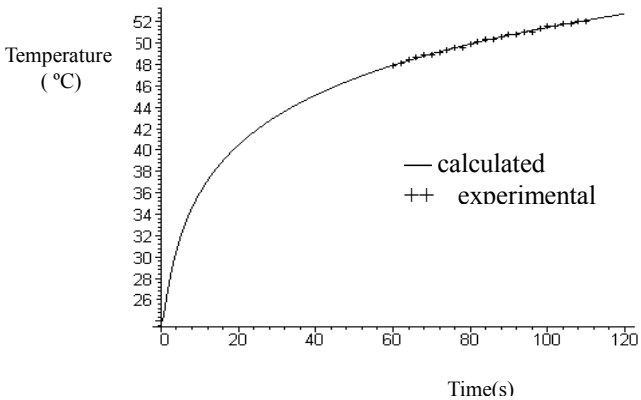

Figure 3 – Temperature x Time

With respect to the heat capacity, c_p , the reference by Vega (1984) indicates an expected value of 1590 J/kg °C for phenolic resins. Therefore, the estimated value is in very good agreement with the published value.

Convergence to the same solution was achieved using several pairs of initial guesses, $\underline{Z}^o = (k^o , c_p^o)$ = (0.05,1000.0); (0.1,2000.0); (0.1,1000.0); (0.05,2500.0) and (0.01,200.0).

In Table 2 are shown the estimates at each iteration, as well as the value for the norm of squared residues, Q, for a couple of values for the initial guesses.

Regarding the quality of the estimated values for the thermal properties, two remarks are in order : (*i*) Fig. 3 shows a very good agreement between calculated and measured temperatures, implying on small values of residuals between them; and (*ii*) As one can infer from Eq.(16) better estimates are associated with smaller confidence bounds, which is directly related to larger sensitivity coefficients $\partial T / \partial \underline{Z}$.

In Table 1 is shown that the estimates obtained with the inverse problem approach considered here are within reasonably small confidence bounds.

As the material under investigation has a density of 450 kg/m^3, the thermal diffusivity calculated considering the values for the estimated properties presented in Table 1 corresponds to 1.04 x 10 $^{-7}$ m^2/s.

FUTURE WORK – EXPERIMENT DESIGN

So far we have used the inverse problem approach to make a better use of the experimental data already collected. These data are limited to the time interval where linearity is observed between the temperature and time logarithm.

As part of an ongoing broader project, we are initiating the use of a full transient run, from the time the wire starts to heat up until steady-state is reached.

In the work presented here no thermal resistance was considered between the heating probe and specimen. The quality of results obtained seems to corroborate the assumption made. Nonetheless, to improve the model to be used in future applications, we will take into account the effects of this phenomenon (Blackwell, 1954).

In all cases considered in this work there is not a real concern with the design of the experiment. We are only looking for an optimal use, or at least a better use, of the already existing equipment, or already acquired experimental data.

Next step consists on an effective experiment design for thermal properties estimation (Beck and Arnold, 1977). Using the mathematical and computational modeling already developed, we will determine, a priori, the best location of temperature sensors , as well as the best time interval in which experimental data has to be taken in order to satisfy a design criteria previously established, such as the minimization of the confidence region (Taktak et al., 1993).

Table 2 - Estimates for k, c_p and norm Q at each iteration

Case 1 - $\underline{Z}^o$ = (0.05,1000.0)

Iteration	k (W/m °C)	c_p (J/kg °C)	Q (°C^2)
0	0.05	1000.0	4285.75
1	0.06584	1361.6	262.53
2	0.07245	1537.7	2.63
3	0.07319	1563.0	0.12
4	0.07319	1563.0	0.12

Case 2 – $\underline{Z}^o$ = (0.1, 2000.0)

Iteration	k (W/m °C)	c_p (J/kg °C).	Q (°C^2)
0	0.1	2000.0	1169.01
1	0.06337	1446.3	324.97
2	0.07187	1557.0	3.87
3	0.07319	1563.2	0.12
4	0.07319	1563.0	0.12
5	0.07319	1563.0	0.12

CONCLUSIONS

The accuracy of the results presented demonstrate that a step forward has been given by using an inverse heat conduction problem solution to extract as much information as possible from a set of experimental data that had already been collected. More important, though, is the potential that we devise for the development of knowledge intensive equipment for thermal characterization of new materials.

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