TRANSIENT TECHNIQUES FOR MEASUREMENTS OF THERMAL PROPERTIES OF SOLIDS: DATA EVALUATION WITHIN OPTIMIZED TIME INTERVALS

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Abstract:- The Dynamic Plane Source (DPS) and the Transient Plane Source (TPS) are transient techniques used for simultaneous measurements of thermal transport properties of solids that are related to direct heat conduction namely; thermal conductivity, thermal diffusivity and specific heat capacity. The theories and the experimental apparatus for these techniques are highlighted. These techniques have been investigated in more details in terms of the view point of the computational methods which must be considered to reveal the deviations from the ideal experimental conditions. The possibility of extending the theoretical analysis to improve the data evaluation procedures using sensitivity coefficients has been implemented. Then the optimum time interval (window) has been selected to obtain the thermal conductivity and diffusivity from the temperature response. Measurements have been done on polymethylmetacrylate (PMMA), and on polycarbonate samples.

Keywords:- transient method, thermal diffusivity, thermal conductivity, specific heat capacity, sensitivity analysis, polycarbonate, polymethylmetacrylate

1 Introduction

New technology essentially requires new composite materials such as ceramics, rocks, different building materials, etc. The knowledge of the thermal properties and the physical processes that enhance or restrict the transport of thermal energy within such materials is also essential. Therefore it is necessary to develop the measurement techniques that can explore such possibilities. This can be achieved either by introducing new techniques or extending and modifying the existing ones, so that they would be more reliable and easy to operate, even under extreme conditions. The reliability of a specific method to measure thermal properties is given by several factors, such as the speed of operation, the required accuracy and performance under various environmental conditions, the physical nature of material, and the geometry of the available sample. However, in most methods the main concern is to obtain a controlled heat flow in a prescribed direction, such that the actual boundary conditions in the experiment agree with those assumed in the theory. Realizing the fact that there is no one technique which is suitable for all conditions and over all temperature ranges however, there are some transient techniques which can be used over a quite broad range of temperatures. The advantages of the transient (dynamic) techniques are short measuring time and simple experimental setups [1]. The dynamic methods can be divided into contact (transient) and noncontact (flash) methods. The contact (transient) dynamic methods are a class of methods for measurements of thermal properties of materials that are directly related to thermal conduction. These properties are obtained from the experimental temperature response.

In this work, we will focus on two types of contact (transient) techniques, namely; the Extended Dynamic Plane source (EDPS) [2-3] and the Transient Plane Source technique (TPS) known as the Hot Disk[4-6]. These techniques have the potential to measure simultaneously the three main parameters: the thermal conductivityλ, diffusivity *k* and the specific heat c of the specimen. The principle of these methods is simple. The sample is initially kept at thermal equilibrium, and then a small disturbance is applied to the sample in a form of a short heating pulse. The change in temperature is monitored during the time of measurement. The thermal properties are then evaluated by correlating (fitting) the experimental temperature measurements with the theoretical relationship obtained from the solution of the differential heat equation. Carslaw and Jaeger [7] give solutions for different experimental arrangements at various initial and boundary conditions. To improve the accuracy and certainty of the measurements using the fitting procedures of the temperature response we use the sensitivity coefficients analysis based on parameter estimation theory [8]. This theory deals with measurements and model errors in a statistical context that provides useful information to optimize the experiment and to estimate thermal properties from dynamic experiments.

2 Theory

The parameter estimation algorithm utilizes a method of least squares to minimize the sum (σ) with respect to the desired parameters:

$$\sigma = \sum_{i=1}^{N} [\, T_{\exp}(t)_i - T_{th}(t)_i \,] \qquad (1)$$

Where, $T_{exp}(t)_i$ are the experimental data values and $T_{th}(t)_i$ are calculated theoretical values using the solution of the differential heat equation. Where the thermal properties coefficients in the corresponding theoretical formula are replaced by unknown parameters such as $p_1 = \lambda$, $p_2 = k$, etc. The reliability of the estimated parameters obtained depends on the sensitivity coefficient of each parameter [9]. The sensitivity coefficient (β_j) for each parameter is obtained from the first derivative for the expression of temperature evolution $T(x,t)$ with respect to the parameter p_j when normalized. According to refs [8-9], it can be expressed as follows:

$$\beta_j = p_j \frac{\partial T(x,t)}{\partial p_j} \qquad (2)$$

These coefficients will illustrate how the estimated parameters could be affected by the magnitude of the temperature response. According to the theory, the simultaneous determination of two parameters, within a certain time range (window), will not be possible if their corresponding sensitivity coefficients are small or linearly correlated/dependant. In the discussion section we will investigate the possibilities of such correlation among the sensitivity coefficients of λ and κ using measured data obtained from the EDPS techniques. Based on the values of the sensitivity coefficients the valid time windows (the data stability intervals) have been chosen within which the thermal conductivityλ, diffusivity κ, and the specific heat c of the specimen can be determined. The values of the sensitivity coefficients for measurements done using the TPS has been already calculated by Boháč et al. [10]. However, due to the fact that the TPS uses exactly the same type of sensor/heater and a very similar EDPS arrangement to collect the experimental data we though it worthwhile to first highlight the TPS basic principle and then look at the similarities/ dissimilarities to compare the results. Furthermore, both techniques emerged from the same laboratory and one of us worked in the development of both, as a matter of fact, the idea of implementing the DPS and its extension EDPS became much easer by using the TPS sensor/heater.

3 Experiment

3.1 The TPS technique

The experiment is simply performed by recording the voltage variations over the TPS-sensor while its temperature is slightly raised by a constant electrical current pulse. The sensor is clamped between two identical cylinders or square-shaped pieces to insure a good thermal contact between the sensor and the sample pieces as shown in Fig. 1. The duration of the current pulse (time of measurements) can ranges from several seconds to several minutes. During this short period, care is taken not to increase the temperature in the samples more than few degrees, since a small temperature increase (small gradient) will reduce errors that may mask some phase transition which occur within narrow temperature regions during measurements[5].

The theory of the method is based on a three-dimensional heat flow inside the sample, which can be regarded as an infinite medium, if the time of the transient recording is ended before the thermal wave reaches the boundaries of the sample. The time dependent resistance of the TPS-sensor during the transient recording can be expressed as

$$R(t) = R_0\left[1 + \alpha\Delta T(t)\right] \qquad (1)$$

where R_0 (≈ 4 Ω at room temperature) is the resistance of the TPS element before the transient recording has been initiated, α is the temperature coefficient of resistance (TCR), for the TPS sensor (α ≈ 4.0 x 10^{-3} K^{-1} at room temperature) and $\Delta T(t)$ is the time dependent temperature increase of the TPS sensor.

The assessment of $\Delta T(t)$ in the heater depends on the power output in the TPS sensor, the design parameters of the sensor and the thermal transport properties of the surrounding sample. For the disk-shaped sensor $\Delta T(t)$ is given by the following equation, from which the thermal conductivity and diffusivity can be obtained

$$\Delta T(\tau) = P_0(\pi^{2/3}a\lambda)^{-1}D(\tau) \qquad (2)$$

Where, P_0 is the total output power, λ is the thermal conductivity of the sample, and a is the radius of the sensor. $D(\tau)$ is the theoretical expression of the time dependent temperature increase, which describes the conducting pattern of the disk shaped sensor, assuming that the disk consists of a number m of concentric ring sources[4], see Fig. 1. For convenience the mean temperature change of the sensor is defined in terms of the non-dimensional variable τ, where $\tau = \left[\kappa t/a^2\right]^{1/2}$ or $\tau = (t/\Theta)^{1/2}$, t is the time measured from the start of the transient heating, $\Theta = a^2/\kappa$ is the characteristic time, and κ is the thermal diffusivity of the sample. The specific heat is related to these parameters through the density ρ via the relation $\rho C_p = \lambda/\kappa$

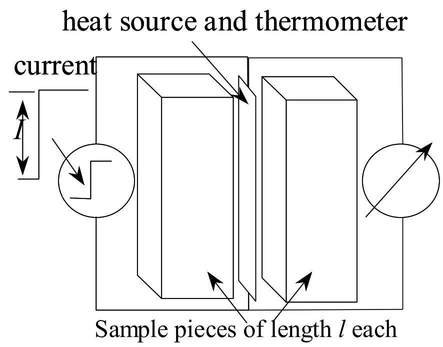

Fig. 1: Schematic diagram shows the experimental set-up for EDPS & TPS Techniques; and The TPS sensor.

3.2 The EDPS Technique

The Dynamic Plane Source (DPS) method [2] is arranged for one-dimensional heat flow into a finite sample. The Extended Dynamic Plane Source (EDPS) method [3] is a modification of the DPS method for materials with relatively low thermal conductivity $\lambda < 2\ \mathrm{Wm^{-1}K^{-1}}$.

The main features distinguishing DPS/EDPS from the TPS can be summarized as:

(i) DPS is arranged for a one-dimensional heat flow into a finite sample, which is in a contact with very good heat conducting material (heat sink) such as copper [3].

(ii) DPS also works in the time region where the sample is treated as a finite medium and is not restricted only to the time region where the sample is regarded as infinite medium.

(iii) DPS has the potential to give λ,κ and ρC_p from a single measurement even if the experimental arrangement resembles a one-dimensional heat flow.

The temperature increase in the plane x = 0, (0< x < *l*), as a function of time will be given by

$$\Delta T(t) = \frac{q\ell}{\lambda\sqrt{\pi}} F(\Theta, t) \quad (3)$$

Where *q* is the total output power per unit area dissipated by the heater, 2ℓ is the length of the sample (see Fig. 1) and $\Theta = \ell^2/\kappa$ is the characteristic time of the sample. $F(\Theta, t)$ is a theoretical expression of the time dependent temperature increase [3] given by

$$F(\Theta, t) = \sqrt{\frac{t}{\Theta}}\left(1 + 2\sqrt{\pi}\sum_{n=1}^{\infty}\xi^n ierfc\left(n\sqrt{\frac{t}{\Theta}}\right)\right) \quad (4)$$

and whose value depends on the measuring time and the sample characteristics as follows:

For times $0 < t < 0.3\Theta$, $F(\Theta, t) = (t/\Theta)^{1/2}$ from which the value of effusivity $\lambda / \sqrt{\kappa} = \sqrt{(\lambda c)}$ is obtained, then for times $t \geq 2\Theta$, $\Delta T(t)$ will approach the value $q\ell/2\lambda$ and λ can be readily obtained.

For times $t \geq 0.5\Theta$, the natural logarithm of the derivative of *ΔT(t)* can be expressed as:

$$\ln\frac{d\Delta T(t)}{dt} = \ln\frac{q}{\rho C_p \ell} - \frac{\pi^2 t}{4\Theta} \quad (5)$$

Then ρC_p can be obtained from the intercept of the graph between $\ln[d\Delta(t)/dt]$ and *t*.

Finally, a further check for the obtained value of ρC_p can be verified via the data consistency relation $\rho C_p = \lambda/\kappa$, where ρ is the mass density.

4. Results and Discussion

As it has been already mentioned in the theory section, the fitting procedure does not work properly when sensitivity coefficients are small or linearly dependent on each other. Therefore, the analysis of the sensitivity coefficients (β_j) determines the time window in which the evaluation procedures can be applied to the temperature response measured using both techniques.

4.1 EDPS technique

Figure 2 shows the values of β_κ , β_λ and the temperature response given by equation (3). The behavior of these values indicates, with an exception at the very beginning, up to Θ the coefficients are not correlated. The actual valid time window was during the period [0.07Θ,Θ] within which the simultaneous determination of the λ and κ is possible. It should be noted that the maximum value of β_κ is around 0.3Θ to which the value of the effusivity $\lambda/\sqrt{\kappa}$ can be obtained as previously mentioned and according to expression given by equation (4).

The distorting time in the beginning of the transient event is characterized by means of the characteristic time $\Theta_\delta=\delta^2/\kappa_{ins}$, where δ and κ_{ins} are respectively the thickness and the mean thermal diffusivity of the layer/layers existing between the metallic heating pattern in the heater/sensor and the sample. We thus include into δ and κ_i not only the insulating layer supporting the heating element but also any other layer between the heating element and the sample as for example air pockets, etc. which might disturb the thermal contact. In principle all distortions resulting from the imperfections (defects) in the heater design, such as heat capacity of the metallic pattern in the sensor, the spacing between the successive strips in the pattern, and so on can be included and each defect will be designated by its own characteristic time. However, in the case of plane source sensors Θ_δ represents the most dominant contribution and it is the most representative to describe the heater influence. Thus, the initial part of the curve (for times below Θ_δ) deviating from linearity reveals the time duration of the heater influence.

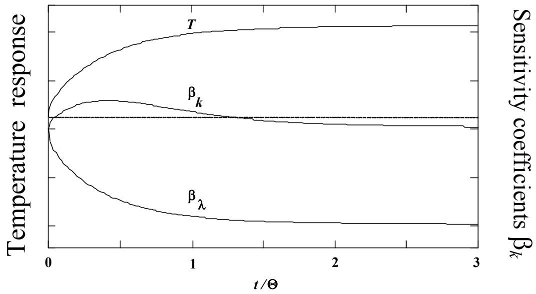

Fig. 2. Temperature response function and sensitivity coefficients β_λ and β_κ vs. nondimensional time scale *t/Θ*.

The eventual thermal resistance between the rear surface of the sample and the sink will start to disturb the temperature development in the heater as soon as the heat pulse reaches the rear side of the sample. Theoretically, the penetration depth of the heat pulse can be given by $\sqrt{2\pi\kappa\, t}$, which indicates that the heat pulse arrives to the rear side of the sample at the time $t = \Theta/2\pi$. However, because of the limited resolution of the detecting devices, the influence of the rear side of the sample on the time history of the temperature development in the heater starts to be detectable at ≈ 0.3Θ. This comes from the fact that according to equation (4) at $t = 0.3\Theta$ the difference in $\Delta T(t)$ for a finite and infinite sample will still be less than 1%. However, for times $t > 0.3\Theta$ the thermal resistance at the rear side of the sample will start to influence considerably the expected temperature development in the heater. Our experiences show that the thermal resistance at the rear side of the sample, which is due to the air pockets between the sample and the sink, can be significantly reduced by making the rear surface of the sample sufficiently flat and by using a very thin layer of silicon based grease between the sample and the sink. Two samples were measured at room temperature (20°C) using the EDPS and data were handled by determining the best time windows from the evaluations of sensitivity coefficients. The results from temperature response over 300 points using current pulse of 540 mA and sampling period of 0.2 s, are depicted in table I below.

Table I Samples characteristics and thermal properties at 20 °C

Thermal properties	Samples characteristics	
	polymethylmetacrylate (PMMA) l = 3mm & ρ =1200 kg/m^3	Polycarbonate (PC) l = 1.13mm & ρ =1172 kg/m^3
$\lambda\ [\mathrm{W\,m^{-1}\,K^{-1}}]$	0.19	0.245
$\kappa \cdot 10^6\ [m^2\ s^{-1}]$	0.12	0.171
$c \cdot 10^3\ [\mathrm{J kg^{-1} K^{1}}]$	1.30	1.20

4.2 TPS technique

The calculated temperature response and sensitivity coefficients of λ and κ as function of the *t/Θ*, where the characteristic time $\Theta = a^2/\kappa$, has been determined by Bohac et al. [10] to measure λ and κ for polymethylmethacrylate and stainless steel. Similarly to our analysis β_κ has a maximum value at $t/\Theta \approx 0.33$ Since the TPS technique is a three dimensional one (the sample is considered as infinite medium) it would be increasingly difficult to evaluate the thermal diffusivity from measurements extending over much longer times. In other words, the time of the transient recording (t_{max}) must be ended before the thermal wave reaches the boundaries of the sample. In fact, according to their analysis a linear dependence of the sensitivity coefficients has been found for $t/\Theta > 1.0$. Their optimal time window within the region $[0, t_{max}]$ where $0.3 < t_{max}/\Theta < 1.1$, the sensitivity coefficients are linearly independent. Furthermore, their minimum time t_{min} must be greater than $\Theta_\delta=\delta^2/\kappa_{ins}$, and it corresponds experimentally to the setting time of the temperature gradient within the sensor. Therefore, the optimal time interval for the TPS would be [t_{min} , t_{max}] where $0.3 < t_{max}/\Theta < 1.1$ and $t_{min} \geq \delta^2/\kappa_{ins}$. It is worth to mention that the same type of sensor has been used for EDPS technique and this is might be the reason for the close agreements on the maximum value of β_k The thermal diffusivity is a physical parameter that expresses the response of the medium to a thermal perturbation. Therefore, as expected measurements of this parameter are associated with the dynamics behind the various conduction mechanisms and the effect of heater components. More studies are planed in vacuum, at different temperatures using different currents and to implement the difference analysis theory [11-12] which is another method for the time interval optimization.

5 Conclusions

The sensitivity coefficients analysis based on parameter estimation theory has been used to define the optimal time windows for the simultanious determination of λ and κ from a single recording. The optimal time window for the EDPS was found to be within [0.07Θ,Θ]. The minimum time corresponding to the value 0.07Θ is associated with the distorting time in the beginning of the transient event. It is characterized by means of the characteristic time $\Theta_\delta=\delta^2/\kappa_{ins}$, where δ and κ_{ins} are respectively the thickness and the mean thermal diffusivity of the layer/layers existing between the metallic heating pattern in the heater/sensor and the sample. The EDPS technique has been used for measurement on polymethylmetacrylate (PMMA), and on polycarbonate samples. The relative standard uncertainties are estimated as 2 % for thermal conductivity and diffusivity and 1 % for specific heat capacity.

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