THICKNESS DEPENDENCE IN THERMOPHYSICAL PARAMETERS MEASUREMENT

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Abstract:
The paper reports on measurements of thermophysical properties of PC (polycarbonate). The theory of the dynamic plane source method and the experimental apparatus are described. The thickness dependency of the thermophysical parameters is measured and processed using the least squares procedure and difference analysis. The results are statistically evaluated and the relative standard uncertainties are estimated as 2 % for thermal conductivity and diffusivity and 1 % for specific heat capacity.

Keywords:
transient method, thermal diffusivity, thermal conductivity, specific heat capacity, difference analysis, polycarbonate

INTRODUCTION

Progress in electronics and computer technologies in the last decades has resulted in a transition from stationary to dynamic methods of measuring thermophysical parameters of solids. The advantages of the dynamic methods consist in reduction of measurement duration and simplification of the experimental apparatus. The dynamic methods can be divided into contact (transient) and non-contact (flash) methods.

Transient methods [1] are based on the generation of the dynamic heat field inside the specimen. This experimental arrangement suppresses the sample surface influence on the measuring process. The thermal conductivity λ and diffusivity *a* of the specimen can be calculated from the temperature response.

EXTENDED DYNAMIC PLANE SOURCE METHOD

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 thermal conductivity $\lambda < 2\ \mathrm{Wm^{-1}K^{-1}}$. The principle of the method is outlined in Fig. 1. The plane source, which simultaneously serves as the heat source and thermometer, is placed between two identical specimens. Heat sink, made of very good heat conducting material, provides isothermal boundary conditions of the experiment.

The theoretical model of the experiment is described by the partial differential equation for the heat transport. The temperature function is a solution to this equation with boundary and initial conditions corresponding with the experimental arrangement. The temperature function [3] is given by

$$T(t,\lambda,a,\tau) = \frac{ql}{\lambda\sqrt{\pi}} F(t,a) + \tau\,, \tag{1}$$

where

$$F(t,a)=\sqrt{\frac{at}{l^2}}\left(1+2\sqrt{\pi}\sum_{\mathrm{n=1}}^{\infty}\beta^{\mathrm{n}}\,\mathrm{ierfc}\left(\mathrm{n}\sqrt{\frac{l^2}{at}}\right)\right). \tag{2}$$

q is the heat current density and l is the thickness of the specimen. τ is an additional (nuisance) parameter which represents the temperature offset of the plane source due to its imperfections, heat capacity and thermal contact with the specimen, termed the source effect. β describes the heat sink imperfection [3] and ierfc is the error function integral [4]. The characteristic time of the specimen is given by

$$\Theta = l^2 / a\,. \tag{3}$$

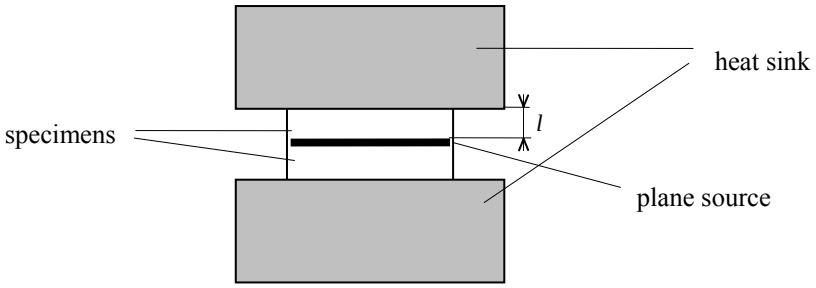

Figure 1 The setup of the experiment.

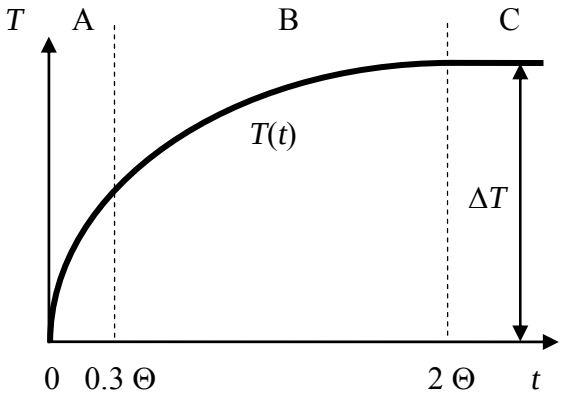

Figure 2 Temperature function and characteristic intervals definition

Figure 2 shows the temperature function and characteristic intervals. In the interval A the temperature function becomes

$$T(t)=\frac{q}{\lambda}\sqrt{\frac{at}{\pi}}\,, \tag{4}$$

which corresponds with the one-dimensional heat flow into an infinite medium (Hot plate transient method [1]). The slope of the graph of $T(t)$ against $\sqrt{t}$ will give the effusivity $\lambda/\sqrt{a}$ of the specimen. Unfortunately, the experimental data in this interval can be distorted by the source effect. In the interval C the temperature function approaches the value

$$\Delta T=\frac{ql}{2\lambda}, \tag{5}$$

which is expected for the steady-state condition and λ is readily obtained. The experimental data can be distorted by surface effect caused by heat losses from the lateral sides of the specimen and by the heat sink imperfection. The third thermophysical parameter - specific heat capacity c can by determined by the formula

$$c = \frac{\lambda}{a\rho}, \tag{6}$$

where ρ is the specimen density.

LEAST SQUARES PROCEDURE

The principle of the method is based on fitting the theoretical temperature function over the experimental points. As the temperature function is nonlinear only in one parameter we can expand it using Taylor series as follows

$$T(t,\lambda,a,\tau) = T(t,\lambda,a_0,\tau) + \left(\frac{\partial T(t,\lambda,a,\tau)}{\partial a}\right)_{a=a_0} \cdot (a - a_0), \tag{7}$$

where a_0 represents a good guess for parameter a. Then we can linearize the temperature function in the following manner

$$T(t,\lambda,a,\tau) = \frac{ql}{\lambda\sqrt{\pi}} \cdot F(t,a_0) + \frac{ql}{\lambda\sqrt{\pi}}(a - a_0) \cdot \left(\frac{\partial F(t,a)}{\partial a}\right)_{a=a_0} + \tau = b_1 f_1 + b_2 f_2 + b_3 f_3 \tag{8}$$

The linear least squares procedure [5-7] in matrix notation is given by the form

$$\vec{y} = \mathbf{X} \cdot \vec{b} + \vec{\varepsilon} \tag{9}$$

where $\vec{y}$ is the observation vector of temperature measured at k points t_i, $\vec{b}$ is the vector of unknown parameters, $\vec{\varepsilon}$ is the vector of errors and **X** is sensitivity matrix defined by

$$\{\mathbf{X}\}_{ij} = f_j(t_i) \tag{10}$$

The least squares estimate of the parameter vector is given

$$\vec{b}_{\mathrm{LS}} = \left(\mathbf{X}^T \cdot \mathbf{X}\right)^{-1} \cdot \mathbf{X}^T \cdot \vec{y} \tag{11}$$

and thermophysical parameters can be computed using the following relations

$$\lambda = \frac{ql}{b_{\mathrm{LS1}}\sqrt{\pi}}, \qquad a = a_0 + \frac{b_{\mathrm{LS2}}}{b_{\mathrm{LS1}}} \tag{12}$$

EXPERIMENT

The plane source in the form of a meander is made from a 20 μm thick nickel foil covered on both sides with 25 μm kapton layer. The diameter of the plane source is 20 mm and the electrical resistance about 1 Ω. The electrical current in the plane source was set to 540 mA. The measurements were performed on polycarbonat (PC) plates of thickness d = 1.13 mm. Eight circles 20 mm in diameter were cut so that specimens of four different thicknesses l = nd (n = 1, 2, 3 and 4) could be assembled. The thermal contact between the individual parts of the specimen set was

improved by silicon oil. The experimental conditions are listed in Table 1. Each temperature response was recorded in 300 points. All measurements were made at the temperature of 20°C.

The density of PC specimen was determined by measuring the mass and dimensions of each circle. The mass density was stated as 1172 kg/m^3 with a combined standard uncertainty of 5 kg/m^3

Table 1 Experimental conditions and the results of the difference analysis

Measurement number	1	2	3	4	5	
Number of plates - n	1	2	3	4	4	
Sampling period [s]	0.2	0.2	0.2	0.2	0.5	
Evaluation	1	2	3	4	5a	5b
Size of the interval t_S [s]	30	30	30	30	30	75
λ [W m^{-1} K^{-1}]	0.209	0.225	0.237	0.249	0.245	0.244
$a \cdot 10^6$ [m^2 s^{-1}]	0.149	0.155	0.160	0.178	0.169	0.167
$c \cdot 10^3$ [J kg^{-1} K^1]	1.20	1.24	1.26	1.19	1.24	1.25

RESULTS AND DISCUSSION

Figure 3 shows five temperature responses measured at the conditions defined in Tab. 1. The temperature is plotted against the square root of time so that the applicability of the equation (4) can be investigated. The first step of the evaluation consists in finding a close guess a_0 for thermal diffusivity a. We can see that the curve 1 is appropriate for the determination of thermal conductivity λ using equation (5) and curve 4 for detemination of effusivity $\lambda/\sqrt{a}$ using (4). Combining both methods guess value $a_0 = 0.169 \cdot 10^{-6}$ m^2 s^{-1} was obtained.

The second step lies in practical application of the difference analysis [8], which is based on fitting the theoretical temperature function over the experimental points within the time interval $(t_B, t_B + t_S)$, where t_B is the beginning and t_S is the size of the interval. When t_B is successively increased and t_S is kept constant, the results of fitting can be plotted against t_B. If the time interval is not suitable for parameter estimation, the results are erroneous and the plot is scattered. The sizes of the time interval t_S for each evaluation are presented in Tab. 1. The results of fitting are plotted against t_B as shown in Fig. 4 and 5.

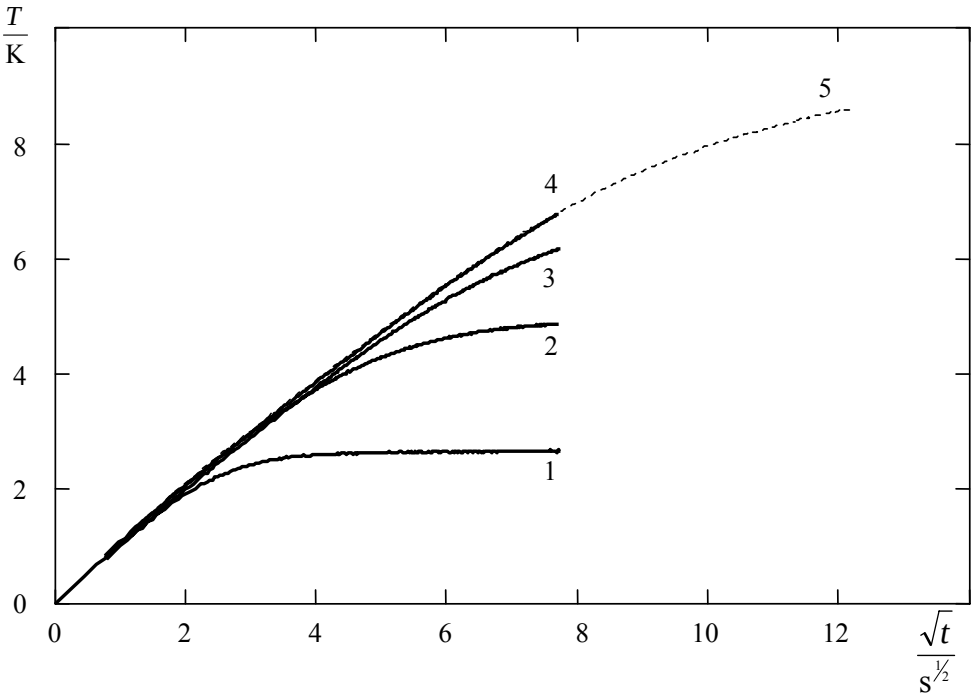

Figure 3 Temperature responses measured at conditions defined in Tab. 1.

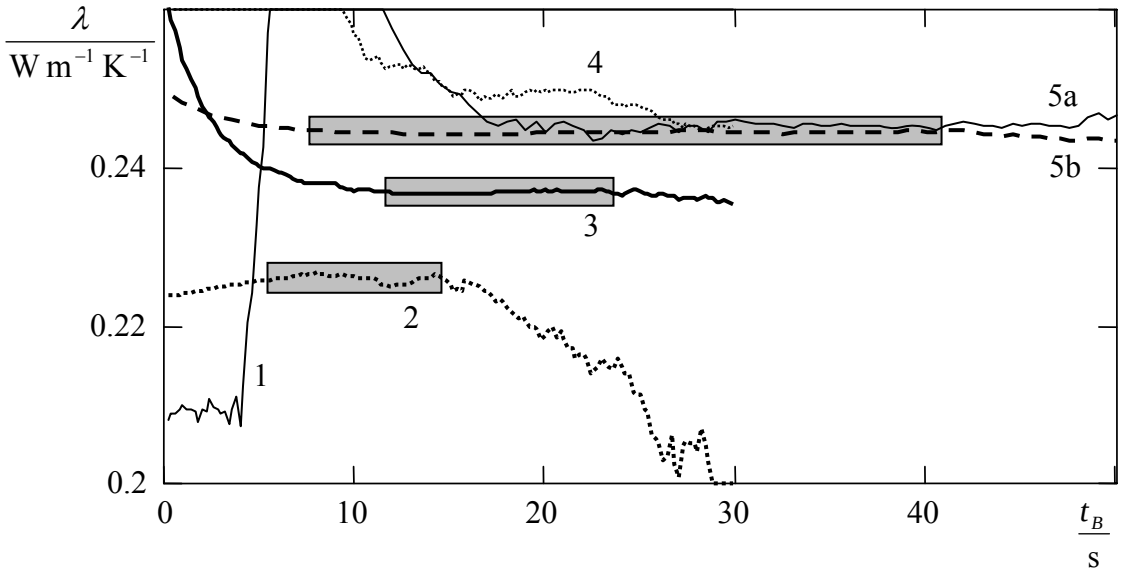

Figure 4 Thermal conductivity as obtained by the difference analysis for conditions defined in Tab. 1. Boxes indicate the data stability intervals.

The aim of the difference analysis is to find the data stability interval [9] within which the results of fitting are considered to be reliable. The time intervals are estimated for every specimen thickness and indicated in figures by boxes. The data stability interval is limited by the source effect at the beginning and by surface effect at the end. In addition, the data stability is influenced by sensitivity coefficients [10], temperature measurement uncertainty [11], sampling rate and size of the interval t_S. Low value of t_S makes the curve noisy, high value causes the curve shortening and

thus also the plateau shortening. The specific heat capacity was determined using equation (6). The results of thermophysical parameters estimation are listed in Tab. 1.

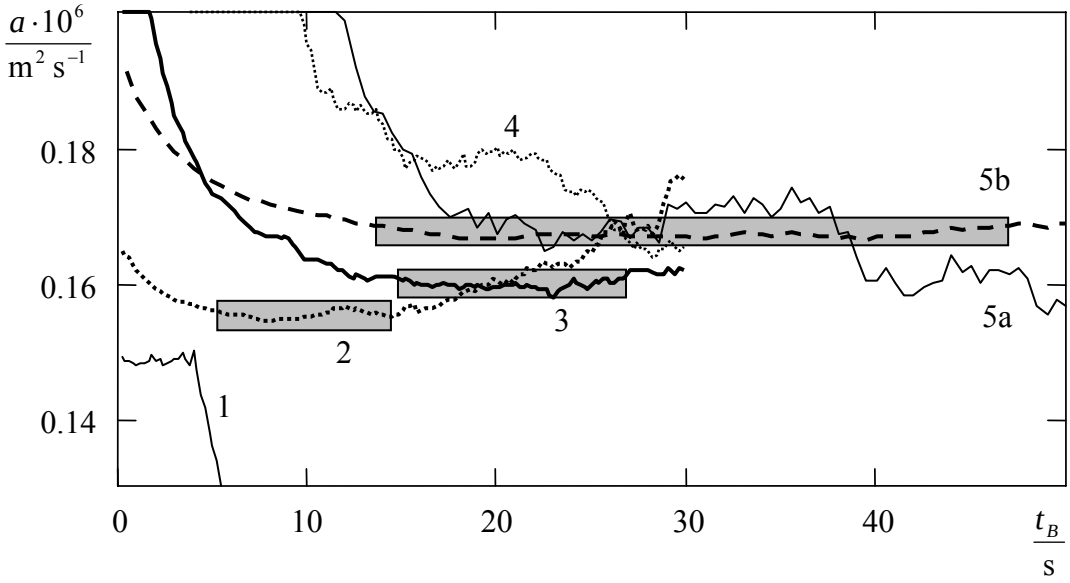

Figure 5 Thermal diffusivity as obtained by the difference analysis for conditions defined in Tab. 1. Boxes indicate the data stability intervals.

Table 2 Statistical evaluation of two sets of the results presented in Tab. 1

Quantity	Evaluation of 1, 2, 3 and 4			Evaluation of 2, 3 and 5b		
	$\bar{x}$	$s(\bar{x})$	$s(\bar{x})/\bar{x}$	$\bar{x}$	$s(\bar{x})$	$s(\bar{x})/\bar{x}$
$\lambda\ \left[\mathrm{W\,m^{-1}\,K^{-1}}\right]$	0.230	0.009	4 %	0.235	0.006	2 %
$a \cdot 10^6\ \left[\mathrm{m^2\,s^{-1}}\right]$	0.161	0.006	4 %	0.160	0.004	2 %
$c \cdot 10^3\ \left[\mathrm{J\,kg^{-1}\,K^{-1}}\right]$	1.22	0.017	1 %	1.25	0.007	1 %

Figures 4 and 5 deserve some more discussion. The distorting time at the beginning of the transient event is described by the characteristic time of the plane source, which was estimated in [12] as $\Theta_D \approx 5-10\,\mathrm{s}$. This was confirmed by the shape of curves 3 and 5b. Although the curve 1 shows the short plateau for $t_B < 3\,\mathrm{s}$, these data are distorted by source effect and the results are unreliable. As the curve 4 has a short noisy plateau at about $t_B \approx 20\,\mathrm{s}$, the measurement with thickness $l = 4d$ was repeated with the sampling period of 0.5s and sweep time of 150s (meas. no 5). When $t_S = 30\,\mathrm{s}$ was used in difference analysis, the curve (5a) was as noisy as curve 4 but the plateau changed. Far more stabile results were obtained with $t_S = 75\,\mathrm{s}$ (curve 5b). This analysis showed that the reliable results can be expected evaluating curves 2, 3 and 5b.

Table 2 presents the statistical evaluation of the thermophysical parameters measurement on PC, where $s(\bar{x})$ is the experimental standard deviation of the mean $\bar{x}$. On the left side there are the results of 4 measurements evaluation without any selection. The results on the right side were obtained using selected curves as described above.

CONCLUSIONS

The EDPS method provided excellent results of thermophysical parameters measurement on PC. This is the consequence of thorough knowledge of the method and effects that can cause the deviation of the experimental set-up from the ideal model. Good guess for parameter *a* enabled to determine the thermophysical parameters using least squares procedure in one step. The procedure was repeated with new value of a_0 but the results changed negligibly. Thorough data processing by difference analysis enabled to determine the data stability intervals and obtain reliable results. The curves 1, 4 and 5a, where the data stability was in doubt, were excluded, which caused a substantial fall in variance of the results.

The main sources of uncertainty in EDPS method were the measurement repeatability and the measurement of specimen thickness [12]. In this work the repeatability conditions were also fulfilled, because the instrument and specimens were disassembled and reassembled before each measurement. Therefore, the standard deviations in Tab. 2 can be regarded as the measurement uncertainties. The measurement repeatability contribution is probably caused by inaccurate positioning of the plane source and the specimens in the holder. Irregularities of the specimens can influence not only the thickness measurement but also the measurement repeatability. This problem was solved by cutting the circles from ideally smooth and planparallel plates (computer CD). Perhaps it is the reason why smaller values of uncertainties were obtained than in [12]. The measurement results are in good agreement with published [3] values for PC, $\lambda = 0.245$ W m^{-1} K^{-1} and $a = 0.171 \cdot 10^{-6}$ m^2 s^{-1}, though the materials are not exactly identical.

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