THE PROGRESS IN DEVELOPMENT OF NEW MODELS FOR PULSE TRANSIENT METHOD

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Abstract:
The ideal models that are describing measurement methods for the thermophysical properties of solids are usually simple. Their advantage is that they involve just one or two free parameters that could be estimated by simple mathematical procedures. Unfortunately in real experiment the situation is limited by specimen geometry and thus the principally simple model derived for usually infinitive media could not be valid. This gives rise to the additional effects that influence the accuracy of the measurements. The most important of them are the heat loss effect form the sample surface caused by final geometry of the specimen as well as the stabilized temperature of the specimen holder that fixes the temperature at the specimen surfaces. The result is that the heat flux that forms planar isotherm penetrates into the specimen is deformed in time and thus the ideal model is valid just in limited time window. Then the evaluation procedure is based on manual selection of the time window for evaluation. This is complicated and depends on the subjective selection of the user. Newly derived multi-parametric models are accounting mentioned parameters. This is on the cost of mathematical complexity of evaluation procedures but it is not dependent on user as the evaluation procedure uses not limited time window for parameters estimation.
This paper review the methodology used in several steps. It is shown how to develop procedures for testing and how to find criteria for particular cases. The improvements of the existing and new physical models that are taking into account the real experimental conditions are discussed.

Keywords:
pulse transient method, thermophysical properties, heat loss effect, real physical models

INTRODUCTION

A modern technology requires rapid development of materials and their testing methods. Thermal properties of materials are one of basic criteria how to recognize between good and bad in a new offer on a market. Thus the new testing procedures are required in this area also. The class of transient techniques has been developed that should satisfy all the testing requirements of new technology [1, 2, 3, 4, [5]].

The use of any technique is conditioned by good knowledge of physical model and the effects influencing the resulted data. Consequently the working methodology of measurements, the evaluation procedures and testing of physical models are prerequisite condition for perfect experiment. The basic question is the reliability of the method that depends on experimental conditions and adequate physical models that should account all experimental circumstances.

In the case of basic idel model for a pulse transient technique a specimen of infinitively large specimen was accounted. It was assumed a planar form of isotherms that penetrates into the specimen from the heat source that generates a heat in aform of dirac pulse. No additional effects were taken into account. The problem comes with real specimen size when disturibing effects are causing the decrease of measurement reliability. Thus research for a new physical approach to solve deficiency in the size (large amount) of testing material was started.

At the pulse transient method the problem was concentrated on a real problem with finite geometry of the specimen that invokes additional effects that harm the efficiency of standard way of the measurement evaluation. One of dominant effects found by data analysis is the heat loss effect from free specimen surface. This effect could be avoided by various ways using different physical models working with infinite and finite geometry and heat losses effect. The new physical aproach could solve a problem with the deficiency in a large amount of testing material.

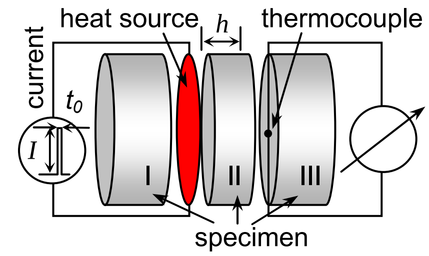
Three different models used for data evaluation and four evaluation methods are discussed in this paper. As a model material a PMMA specimens of different thickness were used. When the ideal model and standard one point evaluation procedure was used the data were strongly influenced by heat loss effect at different thickness of the specimen. There are discussed two approaches how to avoid this problem in real experiment. The first approach uses limited time of recorded data (time window) and ideal model with infinite geometry for parameters evaluation. Here it was prooved that the temperatures recorded at short times are not influenced by heat loss effect. The next approach at new models introduce next parameters - a heat transfer coefficient that represents heat loss effect from the free sample surface and real geometry, e.g. the real radius and length of the specimen. Results obtained within developed methodology agree with recommended data within 5% for all discussed models.

Methodology found show the way of parameters estimation when possible effects does not influence the reliability of the results.

THEORY

In previous works we observed various disturbance effects that were eliminated by searching the ideal geometry of the specimen. The modified model used in older works considered real pulse width instead of Dirac's pulse. Experimental arrangement is draw in Figure 1.

Ideal model with infinite specimen geometry is used to keep low number of unknown parameters but sometimes do not satisfy the real experiment. A detailed study has to be performed to find experimental circumstances when disturbing effects influence ideal model. Then, the modified model has to be used that take into account additional disturbing effects characterized by corresponding, and usually unknown parameters [6, 7, 8, 9]. In the following we introduce a difference in models based on ideal case when assuming infinite specimen geometry and the real pulse duration and a new model with real sample radius and heat loss effect from the free sample surface. The principle of the method is to record the temperature transient response to the heat pulse generated by plane heat source and to calculate the thermophysical parameters from the characteristic features of measured curve (Fig. 1). Transient temperature response measured at the distance *h* from the heat source is calculated according temperature function *T(h,t)* providing that ideal model (Eq. 1.) is valid [1]. In an ideal model we assume that a planar temperature wave is not deformed as it penetrates into the deep of the specimen bulk (white-dotted area in the Fig. 1). The problem is that the temperature isotherms are not planar over the cross section of the specimen and are deformed at the edges by the heat losses from the sample surface for large distances.



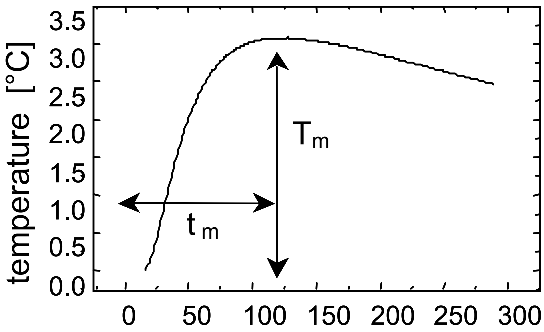


Figure 1. The principle of the pulse transient method (left). The example of the temperature response for PMMA is on the right.

Ideal model

In previous experiments a correction of model considering the real pulse width t_0 was applied to ideal model. Pulse of lower power, but of longer duration replaces the Dirac's pulse as the big instant power can damage specimen. Then, the modified ideal model is characterized by equation [1]

$$T(h,t) = \frac{2 \cdot q_0}{c\rho\sqrt{k}} \left[\sqrt{t} \cdot i\Phi^* \left(\frac{h}{2\sqrt{kt}} \right) - \sqrt{t - t_0} \cdot i\Phi^* \left(\frac{h}{2\sqrt{k(t - t_0)}} \right) \right] \quad (1)$$

where

$$i\Phi^* = \frac{e^{-x^2}}{\sqrt{\pi}} - x \cdot erfc(x) \quad (2)$$

Here q_0 means heat flux from the source, *c* is specific heat, *k* is thermal diffusivity and *t* is time. Equation 1 should be used for data evaluation by fitting procedure.

One point evaluation model

At the standard experiment due to fast calculations we use simple relations for the evaluation of the thermal diffusivity, specific heat and thermal conductivity. These relations were derived for the maximum of temperature response (one-point evaluation procedure). The thermal diffusivity is calculated according equation

$$k = h^2 / (2t_m \cdot f_a) \quad (3)$$

and specific heat

$$c = q_0 t_0 / (\sqrt{2\pi e}\rho h T_m) \cdot f_c \quad (4)$$

where f_a and f_c are correction factors and ρ is the density of material. T_m is maximum of transient temperature response at time t_m (Fig. 1.)

$$f_a = (t_m/t_0 - 1) \cdot \ln\left(\frac{t_m/t_0}{t_m/t_0 - 1}\right) \quad (5)$$

$$f_c = 2 \cdot \exp(1/2)\sqrt{\pi f_a} \cdot t_m/t_0 \left\{1/\sqrt{\pi}\left[\exp(-f_a/2) - \sqrt{1 - t_0/t_m}\exp\left(f_a(t_m/t_0)/2(t_m/t_0 - 1)\right)\right] - \right.$$
$$\left. - \sqrt{f_a/2}\left[erfc\left(\sqrt{f_a/2}\right) - erfc\left(\sqrt{f_a(t_m/t_0)/2(t_m/t_0 - 1)}\right)\right]\right\} \quad (6)$$

Thermal conductivity is given by

$$\lambda = hq_0 t_0 / (2t_m \sqrt{2\pi e} T_m) \frac{f_c}{f_a} \quad (7)$$

Real model

In the next steps several models were derived that takes into account the real radius of the specimen and it‘s real lenght as well as the next parameter that represents the heat flow from the specimen surface into the surrounding. The case of real specimen with heat loss effect is drawn in figure 3. and the infrared picture from experiment in figure 4.

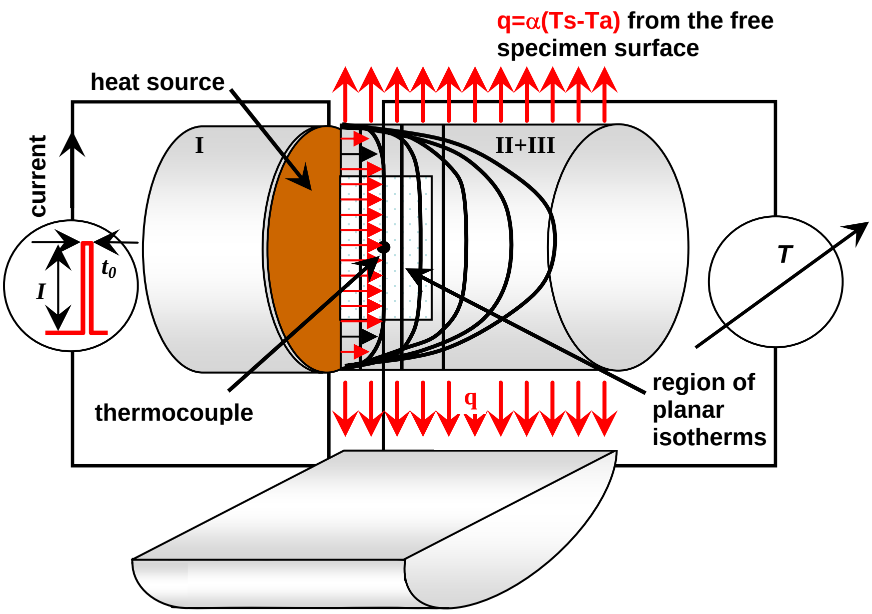


Figure 2. Drawing that represents development of the heat flow outside the specimen. Planar isotherms inside the specimen near the surface are deformed. q is the heat flow outside the specimen, Ts and Ta – surface and ambient temperatures, α - Heat transfer coefficient.

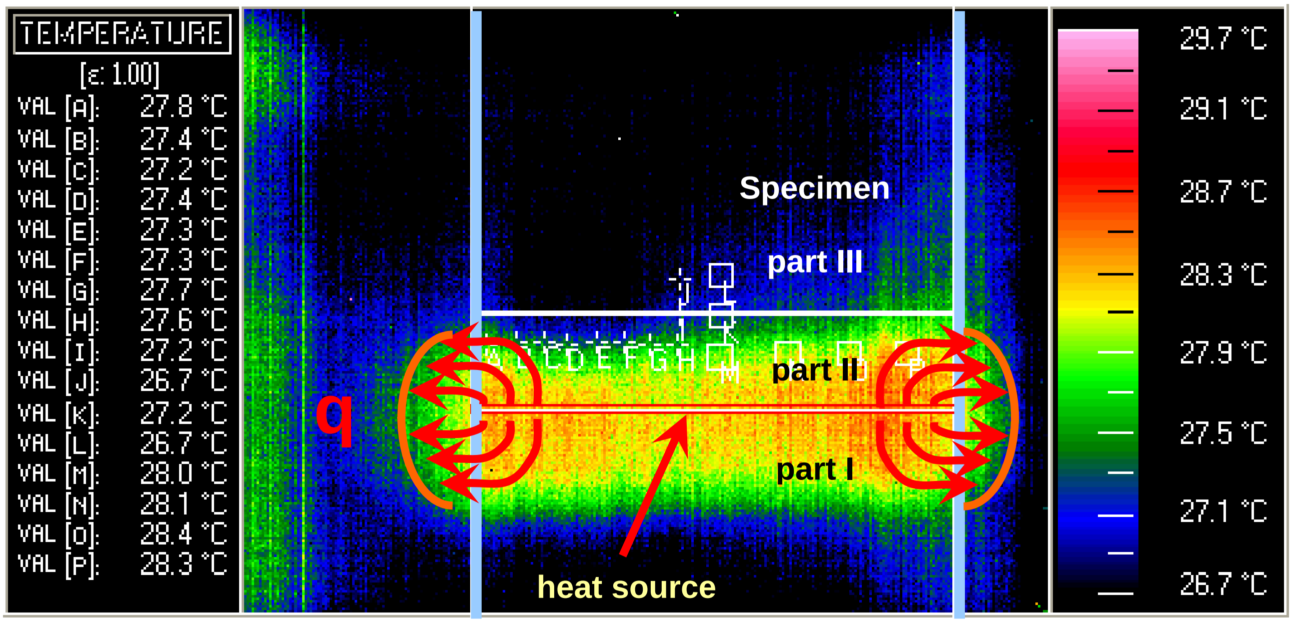


Figure 3. Infrared picture of specimen representing the spreaded heat pulse taken in time when maximum of the temperature response was ritched. The heat flow from the sample surface is denoted by red arrows.

The previous problem with heat loss effect shown in drawing and in picture of a heat loss effect was solved in a two steps. The new models were derived at defined initial and boundary conditions for basic heat transport equation. The heat losses were taken into account at real radius of the specimen and heat source *R* for the first model and also infinite length *L* for the second new model.

Real model for finit radius and infinite length (model 3). This model considering heat losses from the specimen surface, the specimen radius, and infinite length (figure 4). In this case the heat loss from the specimen surface is considered by heat transfer coefficient α. The temperature function of the heat equation according to [10] has a form

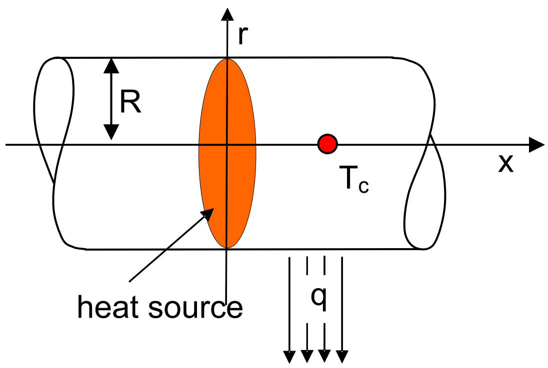


Figure 4. Model of specimen having finite radius R, infinite length and heat losses form the sample surface. The model parameters are of the same meaning like in a case of ideal model.

$$T_c(t,x,r)=\beta\frac{Q}{\lambda}R\sum_{i=1}^{\infty}\frac{J_0\left(\xi_i\frac{r}{R}\right)}{\xi_i\left(\xi_i^2+\beta^2\right)J_0(\xi_i)}\left[e^{-\xi_i\frac{x}{R}}\Phi^*\left(\frac{x}{2\sqrt{kt}}-\xi_i\frac{\sqrt{kt}}{R}\right)-e^{\xi_i\frac{x}{R}}\Phi^*\left(\frac{x}{2\sqrt{kt}}+\xi_i\frac{\sqrt{kt}}{R}\right)\right] \quad (8)$$

where $\beta = \frac{\alpha R}{\lambda}$; $\{\xi_i\}$ are the roots of the equation $\beta J_o(\xi) - \xi J_1(\xi) = 0$, Q – is the heat output power per unit area of the heat source and $\Phi^*(x) = 1 - \Phi(x)$ is complementary error function,. $\Phi(x) = \frac{2}{\sqrt{\pi}}\int_0^x e^{-t^2}dt$.

The equation (8) characterizes the step-wise measuring regime. For the duration of the heat pulse t_0, the temperature for $t > t_0$ is expressed by the equation

$$T^*(t,x,r) = T(t,x,r) - T(t - t_o, x, r) \qquad (9)$$

where $T(t,x,r)$ and $T(t - t_0, x, r)$ are given by the equation (8). The equation (9) characterizes the pulse transient regime.

$$c = \frac{\lambda}{k \cdot \rho}$$

Real model for finit radius, finite length and stabilized temperature of the specimen holder at the specimen end (model 9). This model considering the heat loss from specimen surface (finit specimen radius), finite length of the specimen and T_0 – the heat sink temperature is on ther Figure 5.

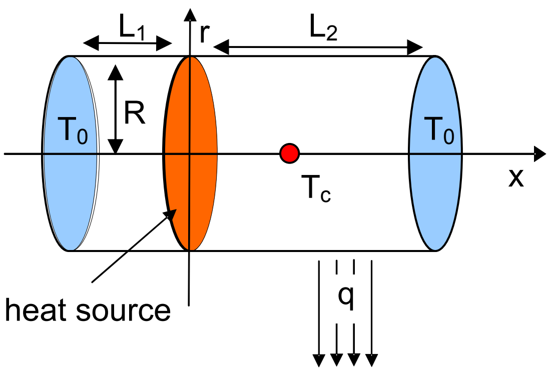


Figure 5. Model using the specimen of finite radius R, finite length L and L_1 and heat losses form the sample surface represented by.

The temperature function of the heat equation in this case has the form

$$T_c(t,x,r) = T_0 \frac{8R^2}{(L_1 + L_2)x} \sum_{\zeta} \frac{\beta}{\zeta^2 + \beta^2} \frac{J_0\left(\zeta \frac{r}{R}\right)}{J_0(\zeta)} \times g(t,x,\zeta) \qquad (10)$$

where $\beta = \frac{R\alpha}{\lambda}$, $T_0 = \frac{qx}{\lambda}$ and

$$g(t,x,\zeta) = \sum_{n=1}^{\infty} (-1)^{n-1} \frac{1 - \exp\left\{\frac{-kt}{R^2}\left[\zeta^2 + \left(n\pi \frac{R}{L_1 + L_2}\right)^2\right]\right\}}{\zeta^2 + \left(n\pi \frac{R}{L_1 + L_2}\right)^2} \sin\left(n\pi \frac{L_1}{L_1 + L_2}\right) \sin\left(n\pi \frac{L_2 - x}{L_1 + L_2}\right)$$

Meaning of another parameters are denoted in figure 6 as well as in previous model. Again, for the duration of the heat pulse t_0, the temperature for $t > t_0$ is expressed by the equation 9.

MEASUREMENTS

All experimental measurements were performed in RTB1.02 chamber (IP SAS) at the temperature 25°C with the temperature stability 0.01÷0.02 K. As a model material a PMMA specimens of different thickness were used. For higher data reliability the statistics at least 5 subsequent measurements were done for each sample thickness.

For the data evaluation there were used various methods (procedures) that uses one point procedure evaluation (ideal model) as well as fitting procedures. Subsequently there were calculated a theoretical responses using corresponding models and were compared with experimentally measured one. The models used assumed experimental conditions with model that uses real pulse width and infinite geometry of the specimen (uses one point evaluation procedure and time window fit procedure), real pulse width, heat loss, finite radius and infinite length of the specimen (fit procedure), real pulse width, heat loss, finite radius and finite length of the specimen (fit procedure).

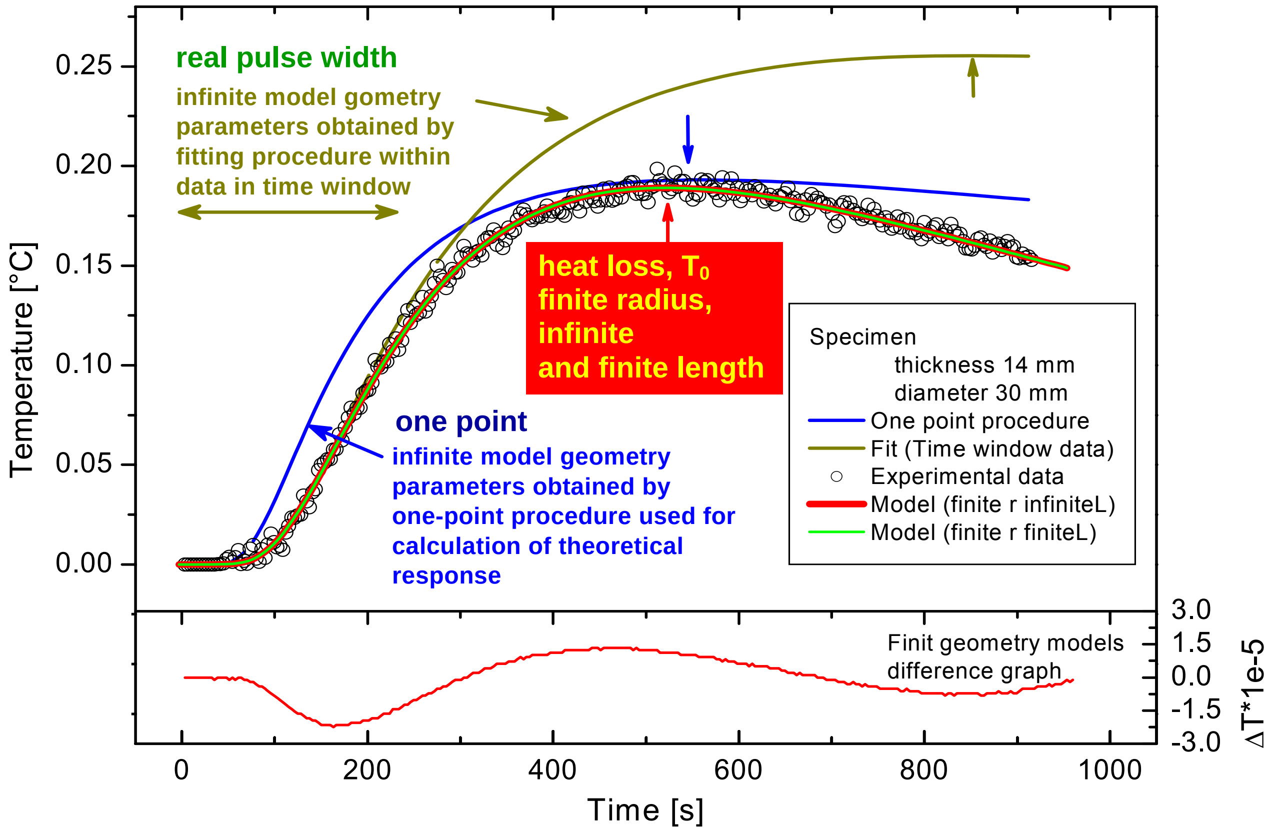


Figure 6. Comparison of experimentaly measured data of temperature response (circles) with the theoretically calculated temperature responses using 4 different evaluation procedures based on 3 models. The difference graph is at the bottom of drawing to illustrate small difference in fit quality between the new models.

The comparison betweeen the real data and theoretically calculated temperature responses based on three models we illustrate in Figure 6 on the data measured on specimen with diameter of

50 mm and the specimen thickness of 20 mm. For the calculation of theoretical responses the parameters obtained by 4 evaluation procedures were used for the calculation of theoretical responses. All the theoretical temperature responses in figure 6 were calculated using thermophysical parameters evaluated by four different procedures – one point evaluation (Eq. 2 and 3.), fit of the data for small times up to 200 s. using Eq. 1 and the fit of all data using combinations of Eq. 9 with equation 8 for first model and equation 10 for second model with real geometry.

Procedure one. In the previous works it was used ideal model (Eq. 1), and one point evaluation procedure for parameters estimation (Equations 3, 4 and 7). Parameters calculated by this procedure were influenced by heat loss effect and data were shifted towards higher values as it is cler from Figure 7 – the plot of thermophysical data vs. specimen thickness. Due to problem with the heat loss effect just data measured at the specinen thickness that ranges from 6 to 8 mm are supposed for reliable. This thickness range was declared as optimized geometry. Within this model, when using standard one point evaluation procedure (Equations 3, 4 and 7) it was found that the heat losses from the sample surface aparently increases the measured values of all thermophysical parameters (Figure 7). The data shift dependens on specimen geometry, (e.g. the thickness and radius) and depends on time during which the experiment was influenced by this factor. Next it was found that the influence of heat source effects for low thicknesses causes lowering of values of thermophysical parameters. This is caused by heat capacifty of the heat source as well as by contact resistance.

Procedure two. The next evaluation procedure uses data in limited time region for data evaluation by fitting procedure based on Equation 1 [9]. In practice this limited time region was named as a “time window for data evaluation” and was already taken from a region of temperatures recorded at lower times, e.g. the times when temperatures did not reach the maximum of the temperature response. With an increasing time the increase of temperature difference between the theoretically calculated temperature response and really measured one is clear (Figure 6). These differences satisfy to effect of the heat loss from the sample surface when the temperature inside the specinmen is lowered (Figure 2 and 6). Figure 6 shows an illustration of this effect measured on PMMA specimen. All the experimental details for this procedure were described in [9]. Figure 6 shows that the shape of experimental response is different as the theoreticaly calculated one for this model due to known effect.

Compared results obtained with the previous procedure are in Figure 7. Situation is improved and all data were pushed down towards the values on the line with recommended data.

Procedure three. Experimental data of temperature response was fitted by equation 9 assuming equation 8 for given geometry of this model. The fit on Figure 6 match the experimental data. The resulted parameters are in figure 7 and are again dissipated around recommended values of thermophysical parameters.

Procedure four. Experimental data of temperature response was fitted by equation 9 assuming equation 10 for this last geometry arrangement. The fit Figure 6 match the experimental data in whole time range also. The resulted parametwers are in figure 7 and are closer to recommended values of thermophysical parameters than previous model mainly for higher specimen thicknesses.

The difference in shape of a theoretically calculated temperature response using parameters calculated by one point procedure from equations 1 and 2 is evident. The better situation is in the case of fitting procedure of data from initial time window (in the range from 1 up to 200 s depicted by line with arrows) by the equation 1. Here we can see clear effect of the decrease of the temperature response in comparison with ideal case when no heat loss is assumed. In the case of two models assuming real sample radius, finit length and heat loosses from the sample surface the values of the temperature response theoretically calculated fits the experimentaly measured one. There is just small difference between the fit of the new models. A difference graph in figure 7 at the bottom is given for illustration.

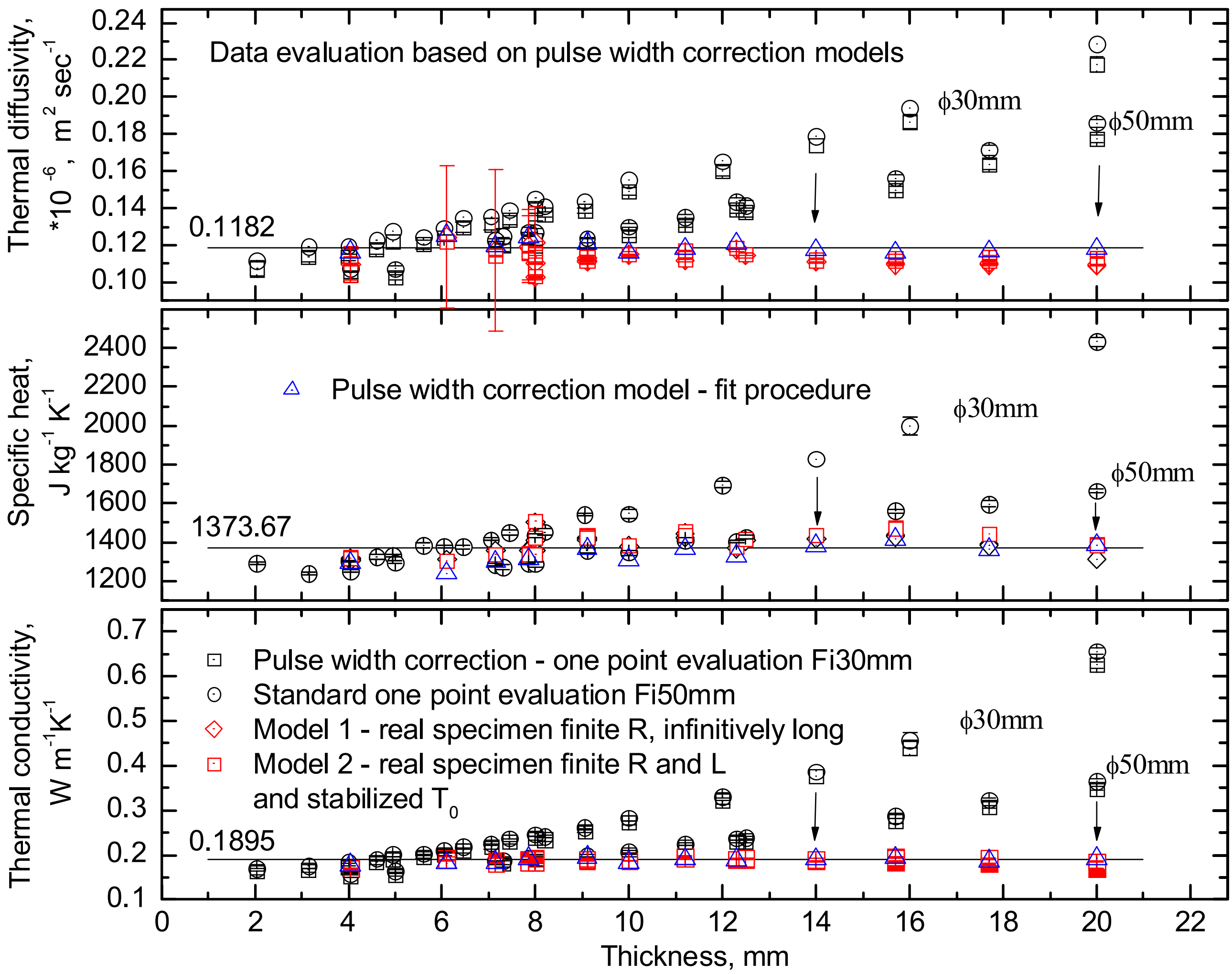


Figure 7. thermophysical data measured on PMMA obtained for different specimen thicknesses.

CONCLUSIONS

The PMMA was used for the measurement of thermophysical properties as model material for the testing of new and existing thermophysical models for pulse transient technique. It was shown methodology how to optimize the experiment geometry and shown methodology how to test and improve the model reliability.

It was discussed heat loss effect and it's influence on the reliability of measured parameters. It was given a methodology reviewed in several steps using different ways of parameters estimation, e.g. the model assuming real pulse width using one point procedure that is available only for the specimens having optimal geometry, the same model using fitting procedure of onset of temperature response (subjective for the choice of the fitting time interval), and models assuming heat loss from the sample surface as well as finite geometry of the specimen.

The optimal geometry as well as proper time window criteria was found when no influences of mentioned effects take place during experiment. Here the one point evaluation model was used for the case of data measured at optimal geometry and the fitting procedure evaluation for the data taken from the onset of a temperature response (time window) for larger specimen thicknesses.

It was shown that the fitting procedure based on model assuming infinite sample geometry should be used just in a time window of the initial onset of the temperature response. This procedure requires to sets manually the time window for evaluation by fitting procedure and thus it seems to be not very objective as it depends on user. The advantage of one point evaluation procedure that uses ideal model is, that is simple and independend on user, but it is available only under the condition when optimized specimen geometry is used [9].

The presented new models are assuming real sample radius and heat loss effect from the sample surface. The fit of the real experiment is excellent and operates automatically.

In a given methodology the error of the measurements were suppressed. Resulted estimated parameters are within 5 percents of the recommended values.

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