

Methodology of the testing of model for contact pulse transient method and influence of the disturbance effects on evaluating thermophysical parameters of the PMMA

V. Boháč, Ľ. Kubičár, V. Vretenár

Department of Thermophysics, Institute of Physics, Slovak Academy of Sciences,
Dúbravská cesta 9, 845 11 Bratislava, Slovak Republic
Email: bohac@savba.sk

Abstract. *The transient methods for the testing of thermophysical properties of materials are suitable for the material characterization, regarding the stability of the structure or structure changes invoked by treatment after production within technology process. Contact pulse transient technique is sensitive enough to be able to record the change in material structure during any kind of material treatment that influences overall material property.*

The reliability of any method in practice depends on fact how much does fit the model with experimental conditions. A detailed study has to be performed to find experimental circumstances when disturbing effects influence experimental data.

In this paper we discuss the methodology of mathematical tests of physical model for pulse transient method. Additional tests were found for the case of real experiment when disturbance effects like heat loss from the specimen surface and non-ideality of the heat source influence the ideal model.

Keywords: *pulse transient method, thermal diffusivity, thermal conductivity, specific heat, model testing*

1. Introduction

The transient methods for the testing of thermophysical properties of materials offer the possibility to observe the changes in material structure through variations of the thermodynamical state. Recently, some papers were published on the new class of transient techniques [1, 2, 3, 4, 5, 6, 7].

The use of any technique is conditioned by good knowledge of the basic model and of the effects influencing the resulted data. Generally the reliability of any method depends on the agreement of the model with the experimental set-up.

In this paper the testing methodology for Contact Pulse Transient method is discussed. This method provides all three thermophysical parameters within a single measurement (thermal diffusivity, specific heat and thermal conductivity). Originally the ideal model for this method was developed for infinitive media and for the heat source having the same thermophysical properties like the measured material [1].

In the real experiment there are some principal problems that could not be avoided: The specimen geometry is not infinitive. The heat source is not ideal and generates not perfect planar temperature isotherm. The ideal model assumes the heat pulse in a form of Dirac pulse, but in experiment it is generated with certain duration. The influence of the radiative part of the heat transport through the media is not included in model. The described problems results in the fact that heat flux from the heat source is homogenized after some time (or when penetrates a certain specimen volume). Then the apparent dependency of measured

thermophysical parameters on the material thickness should appear [8, 9, 10].

Additional effects are changing the initial and boundary conditions in the model for a certain time periods of transient recording. The effects usually, are unknown and are not included in model. The disturbance effects influences real measurement and thus the measured data could not be described by model properly. Paper discusses additional tests that are required for reliable data evaluation.

The model and experimental data for the pulse transient method were analyzed in several ways. The previous analysis on the parameter estimation of the ideal model [8, 11] via theory of the sensitivity coefficients [12] shows that at the lower and the higher times of the transient record the linear dependency of sensitivity coefficients causes the scatter of fitted parameters. This was confirmed by additional test of experimental data – by the difference analysis. This method offers an additional test, especially in the case when disturbance effects are not included in model.

A methodology of the evaluation procedure is discussed how to avoid influence of above mentioned effects at the experimental data evaluation. Experimental data included in this test were obtained on polymethyl methacrylate (PMMA) that is often used as a standard reference material (SRM).

2. Theoretical model

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 (Figure 1).

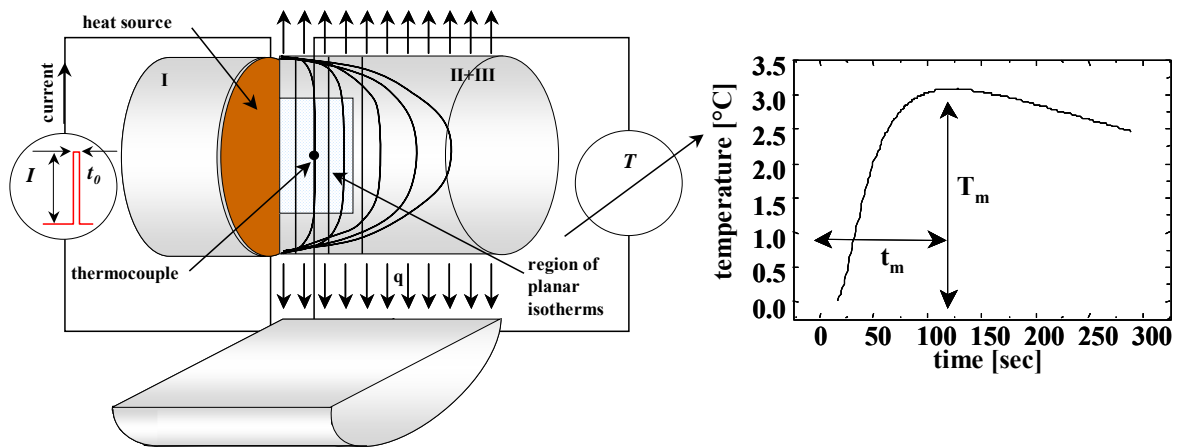


Fig. 1. The principle of the pulse transient method and the heat loss effect model with drawn isotherms (left). Example of temperature response (right).

Transient temperature response measured at the distance h from the heat source is calculated according temperature function $T(h,t)$ providing that ideal model is valid[1]

$$T(h,t) = \frac{Q}{c\rho\sqrt{\pi at}} \exp\left(-\frac{h^2}{4at}\right) \quad (1)$$

Here Q means total pulse heat energy, c is specific heat, a is thermal diffusivity and t is time. At the standard experiment we use simple relations for the thermal diffusivity, specific heat and thermal conductivity that were derived for the maximum of temperature response (one-point evaluation procedure). Then the thermal diffusivity is calculated according equation

$$a = h^2/2t_m \quad (2)$$

the specific heat

$$c = Q / \sqrt{2\pi e} \rho h T_m \quad (3)$$

and thermal conductivity is a product of the former two ones

$$\lambda = h^2 Q / 2t_m \sqrt{2\pi e} h T_m \quad (4)$$

where ρ is the density of material, T_m is maximum of transient response at time t_m . The situation is shown in figure 1.

In real experiment a pulse of lower power, but of longer duration replaces the Dirac pulse. This difference in pulse duration causes the deformation of the onset of temperature response. In a modified model the correction considering pulse width has to be applied to ideal model. Then the modified model is characterized by [1]

$$T(h,t) = \frac{2 \cdot Q}{c \rho \sqrt{a}} \left[\sqrt{t} \cdot i\Phi^* \left(\frac{h}{2\sqrt{at}} \right) - \sqrt{t-t_0} \cdot i\Phi^* \left(\frac{h}{2\sqrt{a(t-t_0)}} \right) \right] \quad (5)$$

where

$$i\Phi^* = \frac{e^{-x^2}}{\sqrt{\pi}} - x \cdot \operatorname{erfc}(x) \quad (6)$$

For the thermal diffusivity using maximum of the temperature response (one-point evaluation procedure) we have

$$a = h^2 / 2t_m \cdot f_a \quad (7)$$

and for specific heat

$$c = Q / \sqrt{2\pi e} \rho h T_m \cdot f_c \quad (8)$$

where f_a and f_c are correction factors

$$f_a = (t_m/t_0 - 1) \cdot \ln \left(\frac{t_m/t_0}{t_m/t_0 - 1} \right) \quad (9)$$

$$f_c = 2 \cdot \exp(1/2) \sqrt{\pi} f_a \cdot t_m/t_0 \left\{ 1/\sqrt{\pi} \left[\exp(-f_a/2) - \sqrt{(t_m/t_0 - 1)/t_m} \exp(t_m/t_0 f_a / 2(t_m/t_0 - 1)) \right] - \sqrt{f_a/2} \left[\Phi^* \left(\sqrt{f_a/2} \right) - \operatorname{erfc} \left(\sqrt{t_m/t_0 f_a / 2(t_m/t_0 - 1)} \right) \right] \right\} \quad (10)$$

Thermal conductivity is given by

$$\lambda = a \cdot c \cdot \rho \quad (11)$$

3. Evaluation procedures

Thermophysical parameters were measured on PMMA sample modified according to figure 1. This configuration allows measurements the same piece of sample for different distances h . The measured curves obtained for some distances are on figure 2.

Measured temperature responses were evaluated by various procedures. Standard one point evaluation procedure uses Eq. 2, 3 and 4. This evaluation is very simple, but it works just in the conditions very close to ideal model when no disturbance effects are take place, e.g. for

optimized geometry of the specimen [8, 11]. The problem with duration of the heat pulse is then solved by using Eq. 7, 8 and 11 where the correction factors for the duration of the heat pulse are included. A more complicated evaluation using Eq. 5 for with the fitting the experimental data in time window when temperature response was not influenced by heat losses from the sample surface, e.g. low times of transient record was used too.

During the test the theoretical temperature responses were calculated using Eq. 5 with parameters obtained by one point evaluation (red dotted line) and by fitting procedure for lower times of temperature response (blue dashed curve). The problem of heat losses from the sample surface causes drop of temperature response at higher times.

There is evident the shift of the curve maximum for the cases of larger material thicknesses 9 and 20 mm. The drop is evident from figure 2 where the experimentally measured responses are compared with theoretically calculated one. One can see also the shift of the maxima that are depicted by circles and triangles.

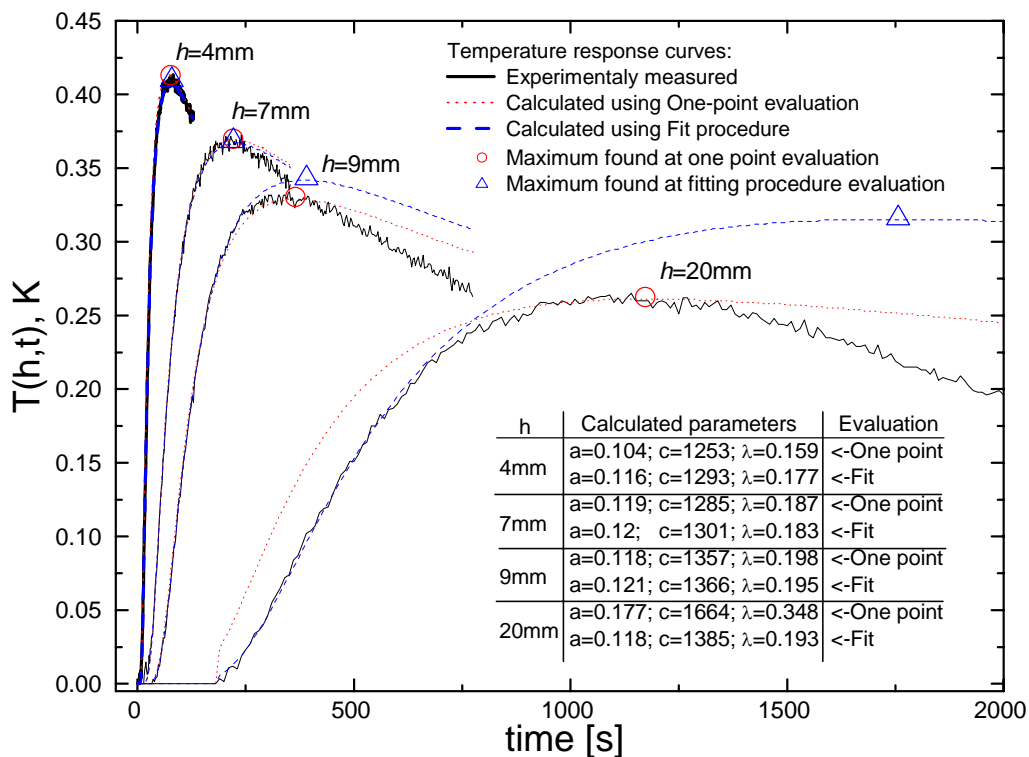


Fig. 2. Measured temperature responses measured for the sample thickness of 4, 7, 9 and 20 mm compared with theoretically calculated one.

4. Results

The complete results depending on type of evaluation procedure for two specimen diameters 30 and 50 mm and for the thicknesses from 4 to 20 mm are in figure 3. The shift of data at lower times as well as at higher times is evident. Data stability region is larger for the specimen diameter of 50 mm because of the effect of heat loss from the sample surface is suppressed. Model accounting real pulse width was applied on temperature response and then the resulting parameters are practically on straight line. Measured temperature responses for the sample thickness of 4, 7, 9 and 20 mm were compared with the theoretically calculated one. The theoretical responses were calculated using input parameters coming from standard one point procedure as well as fitting procedure.

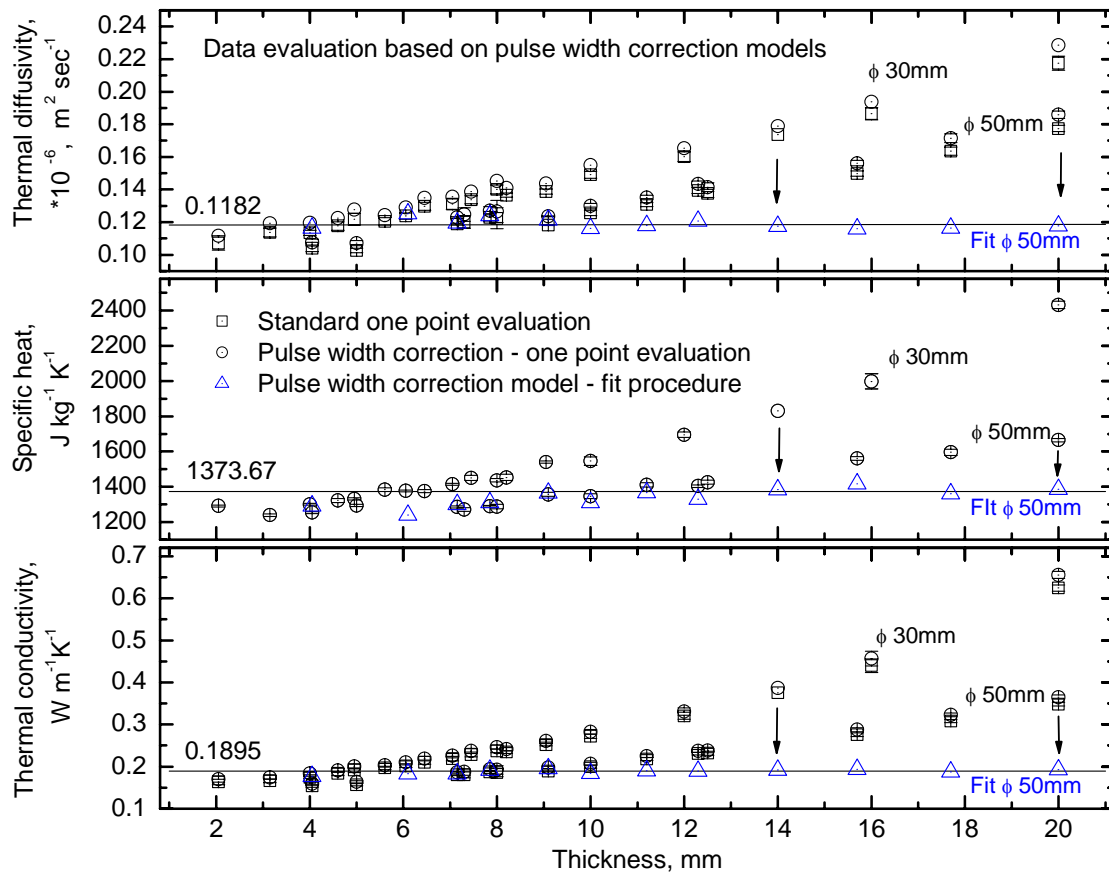


Fig. 3. The thermophysical parameters measured for two diameters of the specimen at different thicknesses of material. The straight line denotes average values of data calculated by fitting procedure.

5. Conclusions

The analysis of disturbances caused by effects at the interface between the heat source and the specimen and the effect of heat losses from the free sample surface was performed. These effects are evident for low and for high thicknesses of material. The methodology of the optimization of specimen geometry was suggested. The optimal thickness for the data stability region between 6 and 8 mm and the specimen diameter 50 mm for PMMA material was found.

The result of analysis is the optimized time window suitable for the data evaluation by fitting procedure and the region of thicknesses with stable data, where the fitted parameters are not influenced by any disturbance and ideal model is still valid.

The optimised geometry in combination with proper time window increases data reliability providing that fitting procedure and pulse width correction model are used.

Acknowledgements

Authors are thankful to Dr. Hammerschmidt, PTB for providing specimens of PMMA. The work was supported by Slovak grant agency under a project numbers No. 2/2036/22 - Study of the heat transport in porous materials by dynamic methods and No. 2/5100/25 - Study and modeling of the thermophysical properties of composites.

References

- [1] Kubičár L. (1990): Pulse method of measuring basic thermophysical parameters. *Comprehensive Analytical Chemistry*, ed. G. Svehla, Elsevier, Amsterdam, Tokyo, Oxford, New York
- [2] Kubičár L. & Boháč, V. (1999): Review of several dynamic methods of measuring thermophysical parameters. in "Proc. of 24th Int. Conf. on Thermal Conductivity / 12th Int. Thermal Expansion Symposium", ed. P.S. Gaal, D.E. Apostolescu, Lancaster: Technomic Publishing Company, pp. 135–149.
- [3] Gustafsson S E, Karawacki E, Khan M N, 1979 *J. Phys. D: Appl. Phys.* 12 1411 – 1421
- [4] Gustafsson S.E. 1991. "Transient plane source technique for thermal conductivity and thermal diffusivity measurements of solid materials," *Rev. Sci. Instrum.* 62:797-804
- [5] Stosch R, Hammerschmidt U., "Finite element analysis of heat conduction in a transient hot-strip setup; Inner and outer boundary effects". Proc. Of 26th International Thermal Conductivity Conference, Cambridge, 4-6, August, 2001, USAXin - Gang Liang, 1995 *Meas. Sci. Technol.* 6 467 – 471
- [6] Lockmuller N., Redgrove J., Kubičár L', Measurement of thermal conductivity with the needle probe, *High Temp.- High Press.*, 2003/2004 Volume 35/36 issue number 2, pages 127 - 138
- [7] Blackwell J H, 1953 34 *Can. J. Phys.* 34 412 - 417
- [8] Boháč V., Kubičár L., Vretenár V., Methodology of parameter estimation of pulse transient method and the use of PMMA as standard reference material. CD ROM Proceedings of TEMPMEKO 2004, 22 - 25 June 2004 Cavtat – Dubrovnik, Croatia (CD ROM 2004)
- [9] Koniarczyk P., Zmywaczyk J., Effect of thermal conductivity reduction in determination of thermal conductivity of semitransparent media in the aspect of analytical analysis and radiation heat transfer equation. *Archives of Thermodynamics*, Vol. 24 (2003), No.4, pp. 35-46
- [10] Koniarczyk P., Some problems of "thickness effect curve" measurements in the determination of heat conductivity of semitransparent media using thin plate samples, *Archives of Thermodynamics*, Vol. 25 (2004), No.1, pp. 73-82
- [11] Kubičár L., Boháč V., Vretenár V., Transient methods for the measurement of thermophysical properties: theory and application. Proc. of the 8th International Symposium on Temperature and Thermal Measurements in Industry and Science, TEMPMEKO 2001, 19-21 June 2001, Berlin, Germany, Ed. B.Fellmuth, J.Seidel, G.Scholz, VDE VERLAG GMBH, Berlin, ISBN 3-8007-2676-9, (2002), pp.295-300
- [12] Beck J.V., Arnold K.J.: Parameter estimation in engineering and science, John Wiley & Sons, Inc., New York, London, Sydney, Toronto, 1977, ISBN 0-471-06118-2