The errors when determining the thermophysical characteristics of liquids by the laser flash method

Mark D. Katz[1,*], and *Ilija* M. Katz[1]

[1]National Research Tomsk Polytechnic University, 634050 Tomsk, Russia

Abstract. The analysis of errors in determining the thermophysical characteristics of typical organic liquids, specified by conductive-radiative heat transfer of ultrathin heated up to high temperature sample layer, under conditions consistent with the implementation of the laser pulse method, when exposed to the surface of the collimated laser pulse of finite duration was made. The influence to the error of the radiation absorption process was discovered.

1 Introduction

Organic liquids are increasingly used in various fields of science and technology. It used as solvents, coolant-cutting fluids and high temperature fluids [1]. Every year, new organic liquids are developing for special purpose of machinery and technology.

A thermal process in the technical fluids and its thermal characteristics plays an important part in reliability control and normal operation of technical systems.

For these reasons, determination of the thermal characteristics with high accuracy becomes more actual task, especially in the development of new products and technical systems.

To investigate the thermal properties of such materials, it is best to use highly efficient procedures, for example, the laser pulse – laser flash (LFA) method [2, 3].

It is based on the absorption of a pulse of radiant energy in a thin layer by the frontal "hot" surface of the sample and the approximate solution of the one-dimensional heat conduction problem for an infinite plate.

From the time-temperature dependence of the reverse ("cold") surface of the sample, one can determine the maximum heating temperature $T_{\max}$ and the time τ_{05} for half the maximum heating temperature of the cold surface of the sample to be attained.

The heat capacity c , thermal diffusivity a, thermal conductivity λ of the material being investigated when using LFA method, is calculated as:

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[*] Corresponding author: riki@tpu.ru

$$c = Q / (T_{\max} \cdot \rho \cdot L);$$
$$a = 0.1388 \cdot L^2 / \tau_{0.5};, \tag{1}$$
$$\lambda = a \cdot c \cdot \rho.$$

where Q – is the energy, absorbed by the sample, J/m^2 ; L is the thickness of the sample, m; ρ is the material density, kg/m^3; τ_{05} is the time for half the maximum heating temperature of the cold surface of the sample to be attained, s.

The LFA method is applying for determination of the thermal characteristics of organic liquids and its compositions [4–6]. For example, the laser flash analyzers Netzch are using for determination of liquids thermal diffusivity in a wide range of the thermal characteristics and measuring temperatures.

There is a practical interest for determination of the partially-transparent liquids thermal characteristics. The heating of these liquids is attended with heat energy accumulation. The sources of this heat energy are thermal diffusivity and bulk absorption in certain spectral range, which depend from the heat emission wavelength.

The condition of LFA method applying for determination of the thermal characteristics of partially –transparent solid materials are discussed in [7–8]. For solving of this task the one-dimensional Thermal Conductivitymethod was used. Also, it was discovered that systematic error of applying LFA method for such materials is increasing. The reason for this is that radiative heat transfer is increasing heating process more than the heating process of opaque materials [9].

The solved problem of determination of the thermal characteristics for solid materials[2] is complicated for liquids, because it's necessary to form thin layer of it. Front surface of this sample is heating, and temperature of the back surface is measuring.

The other problem of applying LFA for liquids thermal characteristics determination is providing following conditions: small duration and high heating intensity without boiling of heating surface.

The purpose of this work is analysis of the conditions determining the thermal characteristics of typical organic liquid (Ethanol) by a laser pulse method [2–3], using conductive-radiative heat transfer mathematical model.

2 Statement of the problem

In case of a problem definition it was supposed that the color of the sample is gray. Sample self thermal radiation is far less then external radiative flux. This supposition is not apply restrictions to the problem statement and to the real conditions of the heat-transfer processes of translucent material.

Also, it was supposed, that thermal characteristics of the materials of the measuring cell and organic liquid (Ethanol) doesn't depend on temperature. This assumption is justified for many materials in real-rated range of possible changes in temperature during the experiments even at high values of the heat flux to the on-heated surface of the sample due to the short pulse duration.

In case of a problem definition it was supposed that the transverse size of the measuring cell is many times more liquid layer thickness cannot be taken into account a possible two-dimensional heat-transfer process and heating due to the heat on the side walls of the cylindrical body. The problem is solved in a Cartesian coordinate system. Solutions represent a three layer plate total thickness L3 (fig. 1).

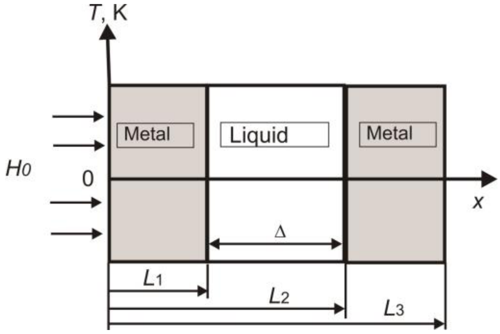

Fig. 1. Scheme of the area of solution.

Surface segment was heated by the collimated heat flux. The heat flux direction is vertical to heating surface. The radiant flux density is H_0 pulse duration is τ_{imp}.

3 Mathematical model and solution method

The mathematical model consists of the three nonstationary heat equation

$$c_1\rho_1\frac{\partial T_1}{\partial t}=\lambda_1\left(\frac{\partial^2 T_1}{\partial x^2}\right),\quad 0<x<L_1 \tag{2}$$

$$c_2\rho_2\frac{\partial T_2}{\partial t}=\lambda_2\left(\frac{\partial^2 T_2}{\partial x^2}\right),\quad L_1<x<L_2 \tag{3}$$

$$c_3\rho_3\frac{\partial T_3}{\partial t}=\lambda_3\left(\frac{\partial^2 T_3}{\partial x^2}\right),\quad L_2<x<L_3 \tag{4}$$

with boundary conditions:

$$x=0:\quad -\lambda_1\frac{\partial T_1}{\partial x}=q,\quad \tau_{\text{imp}}>t>0; \tag{5}$$

$$x=0:\quad -\lambda_1\frac{\partial T_1}{\partial x}=0,\quad t>\tau_{\text{imp}}; \tag{6}$$

$$x=L_1:\quad T_1=T_2;\ -\lambda_1\left(\frac{\partial T_1}{\partial x}\right)=-\lambda_2\left(\frac{\partial T_2}{\partial x}\right) \tag{7}$$

$$x=L_2:\quad T_2=T_3;\ -\lambda_2\left(\frac{\partial T_2}{\partial x}\right)=-\lambda_3\left(\frac{\partial T_3}{\partial x}\right) \tag{8}$$

$$x=L_3:\quad \lambda_3\frac{\partial T_3}{\partial x}=0,\quad t>0; \tag{9}$$

$$t=0:\quad T_1=T_2=T_3=T_0, \tag{10}$$

where T and T_0 are the actual and initial temperature. Subscripts 1, 3 are referred relate to the material of the measuring cell and 2 the liquid, K; t is the time; t is time, s; τ_{pulse} is laser pulse duration, s.

The radiant flux density of the material was calculated by Bouguer–Lambert–Beer Law[10]:

$$H(x) = H_0 \cdot \exp(-k_\lambda \cdot x), \tag{11}$$

where H(x) – the radiant flux density of the material, J/m^2; kλ - coefficient of power adsorption of collimated laser flux, m^{-1}.

The boundary value problem (2)–(11) was solved by the method of finite differences [11].

Problem (2)–(11) belongs to the category of singularly perturbed problems [12].

This problem is more complicated those problems in [7-8]. The main difficulty of this task is the necessity of solving a system of three heat equations with a significant (hundreds of times) difference of materials heat diffusion of each layer.

Therefore, the numerical solution of heat diffusion problem for three-layer infinite plate with significantly different coefficients of thermal conductivity in a high-intensity pulsed heating is independent problem. Its solution is largely due to the choice of grid parameters. The procedure of the grid parameters choosing is the same as for thermal diffusivity phase [13] and chemical conversions [14].

However, the procedures [13, 14] are used for values of the heat flux less then 10^5 W/ m2 and large heating time.

Described above thermal diffusivity was solved for more complicated [13-14] conditions of thermal action. Therefore, the parameters of the grid to solve the problem (1) - (11) were chosen such that further their solution had no effect on results of numerical modeling of temperature pattern.

The values of heat transfer rate and pulse duration taken in accordance with the actual possible characteristics of the laser source of identification of thermal properties of materials research apparatus.

For further simulation following parameters of laser heating source were used: laser pulse energy is less then 25 J/pulse; pulse duration is τimp=0.3…1 ms.

4 Results and discussion

The errors in determining the thermal characteristics of material can be defined [7–9] by comparison of the actual values of thermal diffusivity (a) and heat capacity (c) with the material obtained by numerical simulation of the implementation conditions of experiments on determination of thermal characteristics materials by this method. The errors of liquids thermal conductivity are determined by the method specified in [2–3].

Verification of mathematical models and numerical simulation of the reliability of the results of their study included a comparison with known experimental results as well as verification of conservativeness of difference scheme used. The numerical simulation of thermal diffusivity of the organic liquid Ethanol (thermal characteristics: $\lambda = 0.172$ W/(m·K), $c = 1909$ J/(kg·K), $\rho = 812.1$ кg/m^3) [16] was made in this paper. It was supposed, that the measuring cell was made from iron with wall thickness 0.5 mm ($\lambda = 20$ W/(m·K), $c = 551$ J/(kg·K), $\rho = 7894$ kg/m^3) [17], and flux density is $H_0 = 2.4 \cdot 10^7$ W/m^2, pulse duration 1 ms, T_0 = 273 K. Grid time steps was not above

$1 \cdot 10^{-6}$ s. The initial step in the coordinate $x = 5 \cdot 10^{-7}$ m, and in the areas near the borders of the materials chosen irregular (the algorithm of geometric series) [18 $x_{i+1} = zx_i$, where $z -$ coefficient of variation. At the boundary surface the step of simulation was decreased, and after crossing boundary surface it was increased. The coefficient of variation has been assumed constant and equal 0.02.The wavelength characteristic of Ethanol has four absorption bands in frequency bands 850–870 cm^{-1}, 940–1050 cm^{-1}, 2010–2900 cm^{-1}, 3070-3300 cm^{-1}, where absorption coefficient changes from 5 to 120 m^{-1}.

The bounds range of pulsed heat flux to the heating surface was discovered in [16]. These bounds is conform to conditions of significant determination of thermal characteristics of typical liquids: water, methylbenzene, Ethanol without changing of its physical state during conductive heating of the sample. The maximum value of laser pulse energy density is 18 J/pulse.

At the fig. 2 is shown the errors calculation of heat capacity and thermal diffusivity of Etalon distribution depending on the absorption coefficient k_λ .

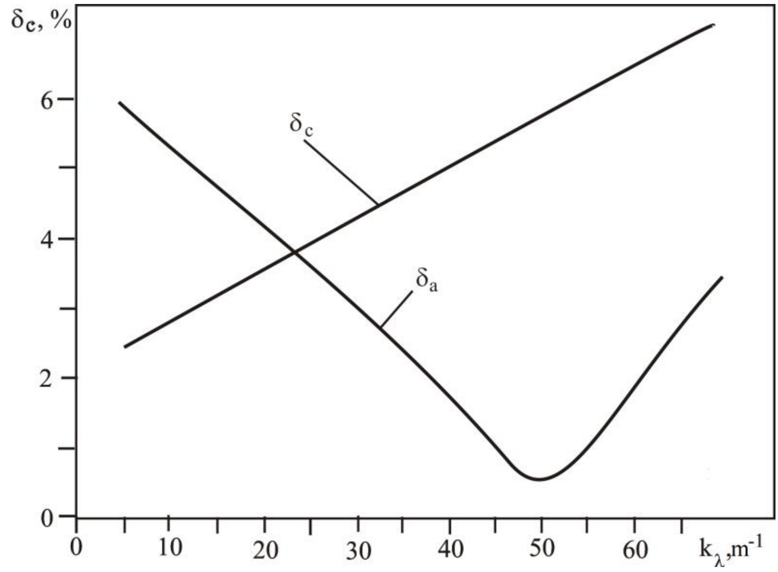

Fig. 2. The errors calculation of heat capacity and thermal diffusivity of Ethanol distribution depending on the absorption coefficient.

As can be seen from fig.2, the values δ_c and δ_a greatly depend of k_λ . With increasing values increase (Fig. 2) and reach 7%, when $k_\lambda = 70$ m^{-1}. The values change monotonically with increasing and reaches a minimum value of about 0.4% at kλ = 50 m^{-1}.

Behavior of changing can be explained by the nature of radiative heat transfer in conditions of low conductivity of the sample liquid.

Accumulated during the pulse duration energy of ultrathin heated up to high temperature sample layer energy is redistributed by thermal conductivity and radiation on the *x* coordinate direction. The heat conduction by this direction is used for calculation of liquid specific heat capacitance and thermal diffusivity by the procedure from [2].

For small values of k_λ the heat energy caused by radiative heat transfer is consumed on increasing liquid temperature. When k_λ you increase the radiation component of the heat flow in the area, close to the near-surface layer of the sample is small, and the heat transfer is carried out largely by conduction.

4 Conclusion

Analysis of the results (Figure 2) allows you to draw a conclusion about the existence of the conditions of the experiment providing the minimum methodological error in determining thermal characteristic partially -transparent liquids at other appropriate conditions.

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