A Finite-Element Flash Method for Measuring Thermal Conductivity of Liquid

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A laser beam is used to heat a portion of liquid closed in a vessel from the top of it to avoid convection. The temperature is measured at the bottom of the vessel by a laser waveguide sensor. The heating and measuring methods allow using a small amount of liquid of the order of a milliliter fraction. The heat flow through the liquid as well as the surrounding vessel and space is modeled by the finite-element method to calculate the temperature at the bottom vs. time. Thus obtained dependence is used then as a fitting function to get the liquid thermal conductivity among some other fitting parameters.

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1. Introduction

From the year of its invention [1], the laser flash method (LFM) for measuring thermal diffusivity has consisted in heating a sample from the front side and detecting the time-dependent temperature from the back side. The initial perfect conditions, i.e. homogeneous and one-dimensional material, homogeneous input energy distribution on the front, an infinitely short pulse and no heat losses, were soon enriched by radiation and convection losses on the front [2], side heat losses together with transient heat transfer in the sample and finite-pulse effects [3] (with a correction in [4], [5, 6]). The effects of nonuniform surface heating were taken into consideration in [7]. The actual dimensions of the heat pulse on the front face, and of the temperature sensing area on the back face of the sample were taken into account in [8]. Further improvements of the assumptions were made in [9], where additionally high-order solutions of the analytical description were applied together with the nonlinear regression routine allowing fitting experimental data to yield thermal diffusivity values with high accuracy. Other authors established analytical solutions by means of the Laplace transform [10] or the Green functions [11]. An application of the gray-body theory for thermal radiation inside a liquid sample at high temperatures [12] was another achievement. The same authors solved their model equations numerically using a finite-difference scheme.

In the presence of so many different factors that influence the heat-conduction process in a sample tested and that are difficult to describe analytically, the authors of this paper decided to take into account those factors by means of the finite-element method. The method has been oriented towards testing the thermal diffusivity and conductivity of a liquid material. The finite-element method allows to take into consideration, except many of the above-mentioned factors, also convection with varying temperature on the surface of the sample and surrounding vessel, convection in the liquid sample, heat losses by the temperature sensor as well as thermal expansion of it. The latter effect may be significant at a rapid temperature-variation stage since a waveguide temperature sensor, when widens non-uniformly, falsifies its temperature indication.

2. Method description

An infrared laser beam is used to heat a portion of liquid closed in a cylindrical vessel from the top of it, as it is shown in Fig. 1. The vessel has insulating properties and is made of foamed polystyrene. Heating the sample from the top helps avoiding a convection in the main phase of measurement when the heating is on. The liquid is covered by a metal plate with black oxidation. The plate floats on the liquid surface by means of the surface tension only to avoid errors connected with heat transfer by a plate mounting. The temperature is measured as a function of time at the bottom of the vessel by a laser waveguide sensor. The temperature sensor has its work area with a length that is equal to the sample diameter to avoid errors connected with temperature sensing area that does not cover the whole sample bottom [8]. The measuring device is placed in a temperature stabilized zone. The stabilization is ensured by thick outside walls made of steel; the wall-to-sample heat-capacity ratio is about $10^4$. The heating and measuring methods allow using a small amount of liquid of the order of a milliliter fraction.

The following heat losses have been taken into consideration:

- heat conduction through the insulating vessel,
- heat conduction through the surrounding air,
- free convection in air above the cover plate and vessel,
**the following parameters are known:**
- the constant ambient temperature \( T_0 \), that is also an initial temperature of the whole measuring system;
- the density, \( \rho \), and specific heat, \( c \), of the anisotropic liquid tested;
- the density, \( \rho_v \), and specific heat, \( c_v \), of the insulating vessel;
- the density, \( \rho_0 \), specific heat, \( c_0 \), thermal conductivity, \( \lambda_0 \), and kinetic viscosity, \( \nu_0 \), of air;
- the density, \( \rho_c \), specific heat, \( c_c \), thermal conductivity, \( \lambda_c \), and thermal emissivity, \( \varepsilon \), of the cover plate;
- the density, \( \rho_m \), specific heat, \( c_m \), and thermal conductivity, \( \lambda_m \), of the waveguide temperature meter;
- the time, \( t_0 \), of constant heating;

**the following parameters are initially assumed, though they are changed by the fitting method:**
- the heat flux, \( q \), of the laser radiation, in \( \text{W/m}^2 \);
- thermal conductivity, \( \lambda_v \), of the insulating vessel;
- the thermal conductivity, \( \lambda \), of liquid investigated (the main parameter sought);
- the following dimensions are known:
  - the vessel inside and outside diameter and height, \( D \) and \( D_0 \), \( L \) and \( L_0 \), respectively;
  - the thickness of the black metal cover, \( g_c \);
  - the inside and outside diameter, \( d_m \) and \( D_m \), of the waveguide temperature meter;
- the liquid region is divided into parallel cylindrical layers (assumption of constant temperature in a layer, justified by the outside insulation);
- the insulating vessel and the waveguide are divided into coaxial rings.

The heat equation in cylindrical coordinates

\[
\lambda \left( \frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial r^2} + \frac{\partial^2 T}{\partial z^2} \right) = \rho c \frac{\partial T}{\partial t} + q,
\]

(1)
is transformed by the finite-element method to the following equation for an inner thin cylindrical element of liquid sample with radius \( r = D/2 \):

\[
T(z, t + \Delta t) = T(z, t) + \frac{2\lambda_{sv}}{\rho c r \Delta r} [T_v(r + \Delta r, z, t) - T(z, t)]
\]

\[
- T(z, t) + \frac{\lambda_0}{\rho c r^2 \Delta r} [T(z + \Delta z, t) - 2T(z, t)]
\]

\[
+ T(z - \Delta z, t),
\]

(2)

where \( z \) is the down-sense vertical coordinate of the thin liquid element, \( T \) is its temperature and \( \Delta z \) is its thickness, \( T_v \) is the temperature of the insulating-vessel thin ring with the radius, axial and radial thickness, \( r = D/2, \Delta z \) and \( \Delta r \), respectively, \( \lambda_{sv} \) is the effective thermal conductivity for the sample-vessel boundary and is given by

\[
\lambda_{sv} = \frac{2\lambda_v}{\lambda + \lambda_v}.
\]

(3)

Equation (1), for the thin cover plate with the radius \( r = D/2 \), is transformed by the finite element method to

\[
T_c(z, t + \Delta t) = T_c(z, t)
\]

\[
+ \frac{2\lambda_{cv}}{\rho c_0 c_r \Delta r} [T_v(r + \Delta r, z, t) - T_c(z, t)]
\]

\[
+ \frac{\lambda_{cs}}{\rho c_0 c_s \Delta z^2} [T(z + \Delta z, t) - T_c(z, t)]
\]

\[
+ \frac{\lambda_{co}}{\rho c_0 c_o \Delta z^2} [T_0(z - \Delta z, t) - T_c(z, t)]
\]

\[
+ \frac{\varepsilon q(t) \Delta r}{\rho c_0 c_0 \Delta z} + \frac{h \Delta r}{\rho c_0 c_0 \Delta z} [T_0 - T_c(z, t)]
\]

\[
+ \frac{\sigma \Delta r}{\rho c_0 c_0 \Delta z} [T^4(z, t) - T_c(z, t)],
\]

(4)

where \( T_c \) and \( T_0 \) are the temperatures of the plate-cover and the ambient air, respectively, \( z = 0 \) for the cover, \( \lambda_{cs}, \lambda_{co} \) and \( \lambda_{cv} \), are the effective thermal conductivities for the cover-sample, cover-air and cover-vessel boundary, respectively, that are determined by the similar-to-Eq. (3) formulae, \( q(t) \) is the time-dependent heat flux, \( q(t) = \text{const} > 0 \) for \( 0 \leq t \leq t_0 \), \( \sigma \) is the Stefan–Boltzmann constant and \( h \) is the laminar-convection conductivity for a horizontal plate given by [13]:

Fig. 1. Diagram of the measuring system.
\[ h = 0.54 \lambda_0 \text{Ra}^{1/4}, \quad \text{Ra} = \frac{g \beta}{\nu_0 \alpha_0} (T_b - T_0) \delta^3 \]  

(5)

where \( \delta \) is the characteristic length, \( \delta = \pi r/2 \), \( \text{Ra} \) is the Rayleigh number, \( g \) is acceleration of gravity, \( \beta = 1/T_0 \) for an ideal gas and \( \nu_0 \) and \( \alpha_0 \) are the kinetic viscosity and thermal diffusivity of air, respectively.

Finite-element equations for other elements, i.e. coaxial vessel rings, coaxial waveguide rings and concentric semispherical air layers, have been created similarly to Eq. (2) and (4). The numerically obtained dependence of the temperature at the bottom, \( T_b(t) \equiv T(L, t) \), upon the time has been used as a fitting function to get the liquid thermal conductivity, \( \lambda \), among other fitting parameters, \( q \) and \( \lambda_v \). Since the insulating-vessel thermal conductivity, \( \lambda_v \), is difficult to measure and varies along with different kinds of foamed polystyrene, it is convenient to make it a fitting parameter. Therefore, time range for temperature measurement must be wide enough to include the long time of self-cooling so that the measuring curve features more characteristic, as it is shown in Fig. 2. The partial derivatives, \( \partial T_b/\partial \lambda, \partial T_b/\partial q \) and \( \partial T_b/\partial \lambda_v \), needed during curve-fitting has been calculated numerically, which requires double computing the whole finite-element procedure for each derivative for two different values of \( \lambda, q \) or \( \lambda_v \), and of course for each time.

The method has been tested and calibrated on water and diethylene glycol. The thermal conductivity of the investigated liquid, 10% MgAl_2O_4-diethylene-glycol nanofluid, has been estimated by the fitting procedure at \( \lambda = (0.28547 \pm 0.00024) \text{ W/mK} \). The uncertainty in the above result has been calculated by the fitting procedure from the measuring-points scattering, which is visually negligible compared to the next factor, and from the numerical model ability to adapt to measuring points. This uncertainty, denoted by \( u_0(\lambda) \), must be enlarged to the following expression for the total uncertainty \( u(\lambda) \):

\[
\sqrt{u_0^2(\lambda) + \sum_i \left( \frac{\partial \lambda}{\partial a_i} u(a_i) \right)^2} = \lambda \sqrt{k_0^2 + \sum_i \left( k_i \frac{u(a_i)}{a_i} \right)^2},
\]

(6)

where \( a_i \) and \( u(a_i) \) are any input parameter and its uncertainty, adequately, \( k_i \) is the coefficient of influence of the parameter uncertainty on the thermal conductivity uncertainty. The derivatives in Eq. (6) have been calculated numerically. The \( k_i \) coefficients are shown in Table.

3. Conclusions

The thermal-conductivity relative uncertainty, \( k_0 \), achieved during fitting the numerical heat-transfer model to experimental data, shows the quality of the model itself, though such accuracy is accomplished when every parameter is measured with relative uncertainty less than \( k_0/k_i \). Such accuracy has been achieved for the sample with a quarter-milliliter volume.

**References**