Experimental Estimation of Thermophysical Properties of Materials

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Abstract

Analysis of potential of the guarded hot plate method and non-stationary temperature regime for experimental estimation of the thermal diffusivity and specific heat capacity of homogenous building materials is described in this paper. The advantages/disadvantages of different measurement schemes are analysed. Numerical estimation of accuracy influence of the process parameters is presented.

Introduction

Experimental device for measurement of heat conductivity of materials has been presented in work [1]. The device with a hot plate allowed to set up the constant power of the heater and to maintain constant temperature on surfaces of an investigated material. Such stationary temperature mode is used for standard measurement [2] of heat conductivity of a material. For the complete description of the thermophysical properties of a material in the non-stationary conditions it is necessary to know value of thermal diffusivity, therefore the electric scheme of the device and the software have been advanced in such away, that power of the heater could be changed harmonically (sine in time). Further, several methods for estimation of thermophysical parameters of materials employing sinusoidal mode of heating are considered.

1. Mathematical description of heat conductivity

Process of the heat transfer can be assumed being one-dimensional because of the design features of the device. Governing equation of heat conductivity is heat diffusion equation \( T_x = \alpha T_{xx} \), where \( \alpha = \lambda / \rho c \) is the thermal diffusivity, \( c \) is the specific heat capacity, \( \lambda \) is the heat conductivity and \( \rho \) is the density of substance. Material properties are assumed not dependent on temperatures, because the range of change of working temperature is relatively small (about 50° C). The solution of the heat diffusion equation in case of the heater power represented by a sine wave is searched in a following particular form: \( T = T_c(x) + T(x)e^{i\omega t} \), where \( i = \sqrt{-1} \) and \( \omega \) is the cyclic frequency. After substitution of temperature with the particular solution in the heat conductivity equation we get:

\[
(i \omega T - a T_{xx}) e^{i\omega t} = a T_{xx}.
\]

The equation splits up into two independent equations

\[
i \omega T - a \frac{\partial^2 T}{\partial x^2} = 0, \quad \frac{\partial^2 T_c}{\partial x^2} = 0.
\]  

(1.1), (1.2)

The equation (1.1) contains only amplitude and phase of the fluctuations of temperature, and the equation (1.2) describes the stationary mode of the heat transfer. Thus, the sine wave operating mode can be used also for measurement of heat conductivity of a material with the minor difference with the usual stationary mode that the temperature used in
calculations should be averaged over the time period. Let’s introduce $A^2 = \omega a / \sqrt{2a}$. Then the equation of the heat transfer (1.1) becomes:

$$\frac{\partial^2 T}{\partial x^2} - A^2 T = 0. \quad (2)$$

The solution of the equation (2) is the function

$$T(x) = C_1 e^{Ax} + C_2 e^{-Ax}. \quad (3)$$

Our purpose is to estimate value of $A$, therefore it is necessary to formulate three boundary conditions for definition of all unknown variables $C_1, C_2, A$. We shall consider the basic possible cases of the measurement of the parameter $A$

1) Lumped capacity case (Fig. 1a). The heater is switched on for a short period of time. At the same time temperature $T_1$ and heater power is measured.

2) Periodic process in case of one slab of the tested material. The measured values of temperature on boundaries of the testes material ($T_0, T_1$) and the density of heat flux (Fig. 1b) is used as initial data.

3) Periodic process in case of two slabs of investigated materials. In this case temperatures $T_0, T_1$ and $T_2$ (Fig. 1c) are measured.

It is assumed that the value of the heat conductivity is known. It can be measured by the stationary measurement mode [1].

2. **Lumped capacity case**

This is the simplest method based on the energy conservation law. Let’s assume that heat losses $Q_{out} = K(T_{min} - T)$ are proportional to the difference of the minimal temperature in the sample and the reference temperature. Then the heat capacity of a material $C_m = m \cdot c$, where $m$ is density of the material, is easy to estimate by using recursive procedure

$$\frac{E_h - E^{(i-1)}_{out}}{\Delta T} = C_m^{(i)}, \quad K^{(i+1)} = C_m^{(i)}(T_{max} - T_L)/\int_{t_{max}}^{t_{L}} (T_{min} - T) dt; \quad E^{(i)}_{out} = K^{(i+1)} \int_{0}^{t_{max}} (T_{min} - T) dt,$$

where $E_h$ is energy emitted by the heater, $E_{out}$ is the heat losses, index in the brackets point to iteration number.

![Fig. 1. Schemes of experiments for measurement of coefficient $A$](image)

![Fig. 2. The schematically representation of process temperature in time](image)
3. One layer case

Periodic process in the case of one layer of the material is described by following system of equations:

\[ T(x) = C_1 e^{\lambda x} + C_2 e^{-\lambda x}, \quad T(0) = T_0, \quad T(L) = T_1, \quad \lambda \frac{\partial T(x)}{\partial x} \bigg|_{x=0} = -Q. \]  

\[ (4) \]

From here follows the equation for determination of \( A \):

\[ T_0 \cosh(AL) - \frac{Q}{\lambda A} \sinh(AL) - T_1 = 0 \]

\[ (5) \]

It is necessary to note, that in real conditions the equation (5) is not exact. The left hand size part of the equation should be understood as the function which should be minimized. Implicitly differentiating expression (5) we can find sensitivity of \( A \) to the change of the parameters. All values of sensitivity are shown in Table 1.

Table 1. Sensitivity of \( A \) to the change of the parameters \( T_0, T_1, Q \) and \( L \)

<table>
<thead>
<tr>
<th>( \frac{\partial A}{\partial T_0} )</th>
<th>( \frac{\partial A}{\partial T_1} )</th>
<th>( \frac{\partial A}{\partial Q} )</th>
<th>( \frac{\partial A}{\partial L} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( -A^2 \cosh(AL)/Z ) *</td>
<td>( A^2/Z )</td>
<td>( A \sinh(AL)/Z )</td>
<td>( -A^2 (T_0 A \sinh(AL) - Q \cosh(AL))/Z )</td>
</tr>
</tbody>
</table>

* \( Z = T_0 LA^2 \sinh(AL) + Q \sinh(AL) - QLA \cosh(AL) \)

4. Two layers case

In the case of several layers of material thermal contact resistance \( R = \Delta l / \lambda \) can influence distribution of temperature. For the estimation of the contact resistance characteristic properties of the tested materials and air are used (see Table 2). For a layer of air between materials \( \Delta l \approx 1 \) mm resistance is \( R \approx 0.04 \) [(m\(^2\)×K)/W], for a layer of foam concrete \( \Delta l \approx 5 \) cm resistance is \( R \approx 0.4 \) [(m\(^2\)×K)/W]. Thus, the jump of temperatures on contact of investigated materials can reach 10% of the common temperatures difference in the stationary mode. The contact resistance between ceramic surfaces given in literature (for example [3]) varies within the interval 3.3·10\(^{-4}\)-2·10\(^{-3}\) [(m\(^2\)×K)/W]. Such values of the parameters give smaller jump of temperature. The periodic mode is characterized by the following ratio of thermal resistances:

\[ \frac{A_a \Delta l_a}{A_m \Delta l_m} = \left( \frac{\lambda_m \rho_a c_a}{\lambda_a \rho_m c_m} \right) \frac{\Delta l_a}{\Delta l_m} \approx 8 \cdot 10^{-5}, \]

where index \( a \) corresponds to air and \( m \) to tested material. Thus, the influence of the contact resistance to the periodic mode is negligible comparing to the stationary case resistance. If it is possible to use different contact materials, then material should be considered satisfying: \( \rho_a c_a < \rho_m c_m, \lambda_a > \lambda_m \).

Let's introduce the system of

Table 2. Properties of tested materials

<table>
<thead>
<tr>
<th>material</th>
<th>( \lambda ) W/(m-K)</th>
<th>( \rho ) kg/m(^3)</th>
<th>( c ) J/(kg-K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>foam concrete</td>
<td>0.120</td>
<td>400</td>
<td>1000</td>
</tr>
<tr>
<td>concrete</td>
<td>0.120</td>
<td>400</td>
<td>1000</td>
</tr>
<tr>
<td>air</td>
<td>0.026</td>
<td>1.293</td>
<td>1006</td>
</tr>
</tbody>
</table>
coordinates with the origin on the interface of materials. Temperature in the left material is \( T^{(1)}(x) \), and temperature in the material to the right is \( T^{(2)}(x) \) (see Fig. 3). Temperatures on the boundaries of slabs are known, and spatial distribution of the temperature is in the form \( T^{(1)}(x) = C_1 e^{A_1 x} + C_2 e^{-A_1 x} \), \( T^{(2)}(x) = C_3 e^{A_2 x} + C_4 e^{-A_2 x} \), therefore periodic process is described by the following system of equations:

\[
\begin{cases}
T^{(1)}(0) = T_0, & T^{(2)}(0) = T_3 \\
\lambda_1 \frac{\partial T^{(1)}}{\partial x} \bigg|_{x=0} = \lambda_2 \frac{\partial T^{(2)}}{\partial x} \bigg|_{x=0}, & R\lambda_1 \frac{\partial T^{(1)}}{\partial x} \bigg|_{x=0} = T^{(1)}(0) - T^{(2)}(0)
\end{cases}
\]

Neglecting contact resistance \( R=0 \) and requiring that the average temperature between materials is equal to the measured temperature \( T^{(1)}(0) + T^{(2)}(0) = 2T_2 \), we have the equation for the determination of \( A_2 \):

\[
Q_1 = -\frac{\lambda_2 A_2 (T_3 - T_2 \cosh(A_2 L_2))}{\sinh(A_2 L_2)}
\]

where the heat flux through the interface between layers is \( Q_1 = -\lambda_2 A_2 (T_2 \cosh(A_2 L_2) - T_3)/\sinh(A_2 L_2) \). Assuming, that parameters of the material 1 are known, sensitivity to the change of the parameters is defined in Table 3, but accuracy of definition of the heat flux can be found by differentiating \( Q_1(A_1, L_1, \lambda_1, T_0, T_2) \) by all of the parameters. In case of the identical slabs \( A_1 L_1 = A_2 L_2 = AL \) the equation (7) essentially simplifies:

\[
2T_2 \cosh(AL) = T_0 + T_3.
\]

Table 3. Sensitivity of \( A \) to the change of the parameters \( L, T_0, T_1 \) and \( T_3 \) in the case of two equal layers

<table>
<thead>
<tr>
<th>( \partial A / \partial L )</th>
<th>( \partial A / \partial T_0 )</th>
<th>( \partial A / \partial T_1 )</th>
<th>( \partial A / \partial T_2 )</th>
<th>( \partial A / \partial T_3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 1/(2T_2 L \cosh(AL)) )</td>
<td>( 1/(2T_2 L \cosh(AL)) )</td>
<td>( -\sinh(AL)/(T_2 L \cosh(AL)) )</td>
<td>( -A/L )</td>
<td></td>
</tr>
</tbody>
</table>

5. Estimation of the properties of the measuring system

We have employed the lumped capacity model for determination of effective characteristics of the heater (Fig. 1a). Applying the recursive procedure for the received data, we obtain \( C_m=1047 \) [J/kg] and the effective specific heat capacity of the heater \( c_h=421\) [J/(kg·K)].

Complexity of the estimation of the heat conductivity is in fact that the maximal heater temperature is unknown. To overcome the problem we employ solution for semi-infinite region [3] of the heat conductivity equation. Initial temperature of material is equal to \( T_{\min} \) and the heat flux from the heater is equal to \( Q \):

\[
T(x,t) - T_{\min} = \frac{Q}{\lambda} \left( 2 \frac{at}{\pi} e^{-\frac{x^2}{4at}} - x \cdot \text{erfc} \left( \frac{x}{2\sqrt{at}} \right) \right)
\]

The given assumption will hold only in the case of small deviations of temperature from \( T_{\min} \). Using data of experiment (Fig. 4a) and the least squares method, we find the coefficient of heat conductivity.

Calculated effective heater parameters are: weight of the heater \( m_h=5.6 \) [kg], its density \( \rho_h=5185 \) [kg/m³], specific heat capacity \( c_h=421 \) [J/(kg·K)], heat conductivity...
\( \lambda_h = 0.32 \text{[W/(m·K)]} \) and heat diffusion coefficient \( a_h = 1.45 \cdot 10^{-7} \text{[m}^2/\text{s}] \). The approximate calculation for the layered heater (width of copper is 6mm, ceramics - 3mm and air - 3mm) gives the following values: \( \rho_h = 5217 \text{[kg/m}^3]\), \( c_h = 415 \text{[J/(kg·K)]}\), \( \lambda_h = 0.1 \text{[W/(m·K)]}\). These results are in good agreement with the experimental data.

Thus, the method applied here can serve also as the alternative method for calculation of the heat diffusion coefficient.

4. Results of thermal diffusivity calculations

Results of calculation of parameters of plasterboard (which density is \( \rho_m = 773 \text{[kg/m}^3]\)) are presented in Table 4. The experiment setup corresponds to Fig. 1c. Two layers were made with two equal 12.5 mm width plasterboard slabs. First slab was separated from heater by soft rubber substrate, in which the temperature sensors have been installed. Considering design features of the device we shall note some important features of calculations. The phase of the heat flux entering the test material does not coincide with the phase of the heater power. It is possible to compensate for phase deviation using equation (5) as well as using effective heater parameters. Phase deviation should be equal to \( \Delta \phi = 25.4^\circ \) accordingly to the calculation in chapter 3.

Fig. 5a corresponds to the case of one layer when the phase of heat flux was assumed equal to heater power phase \( \Delta \phi = 0^\circ \) (an advancing of temperature by phase to heater power phase is 69.2°). The Fig. 5b displays a case when the phase is compensated by the inertia of the heater (phase delay \( \Delta \phi = 25.4^\circ \)). In Fig. 5c the phase is compensated so that the equation (5) holds precisely (phase delay \( \Delta \phi = 36.1^\circ \)). The scheme with two layers does not have drawback because the heat flux is defined by temperatures.

<table>
<thead>
<tr>
<th>Method</th>
<th>( c ) [\text{J/(kg·K)}]</th>
<th>( \lambda ) [\text{W/(m·K)}]</th>
<th>( a ) [\text{m}^2/\text{s}]</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Lumped capacity case</td>
<td>1621</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>One layer case (one slab)</td>
<td>1326</td>
<td>0.25</td>
<td>2.438 \cdot 10^{-7}</td>
<td></td>
</tr>
<tr>
<td>One layer case (two slabs together)</td>
<td>1211</td>
<td>0.25</td>
<td>2.672 \cdot 10^{-7}</td>
<td></td>
</tr>
<tr>
<td>Two layer case</td>
<td>1229</td>
<td>0.25</td>
<td>2.632 \cdot 10^{-7}</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 5. Phase (curve crossing the abscissa axis) and amplitude of the left hand size part of the equation (5) at different phase delay $\Delta \phi$. On abscissa axis $A = \log(a)$

Table 5. Numerical values of sensitivity of $A$ to the change of parameters

<table>
<thead>
<tr>
<th>Method</th>
<th>$\partial A / \partial T_0$</th>
<th>$\partial A / \partial T_1$</th>
<th>$\partial A / \partial T_2$</th>
<th>$\partial A / \partial Q$</th>
<th>$\partial A / \partial L$</th>
</tr>
</thead>
<tbody>
<tr>
<td>One layer case (one slab)</td>
<td>$10.00 e^{-2.73}$</td>
<td>$9.70 e^{0.11}$</td>
<td>-</td>
<td>$0.49 e^{0.21}$</td>
<td>$25936 e^{1.01}$</td>
</tr>
<tr>
<td>One layer case (two slabs together)</td>
<td>$2.21 e^{-1.40}$</td>
<td>$1.60 e^{0.76}$</td>
<td>-</td>
<td>$0.16 e^{1.14}$</td>
<td>$4727 e^{2.39}$</td>
</tr>
<tr>
<td>Two layer case</td>
<td>$8.06 e^{-0.33}$</td>
<td>$25.22 e^{-2.30}$</td>
<td>$8.06 e^{-0.33}$</td>
<td>-</td>
<td>$2432 e^{-2.36}$</td>
</tr>
</tbody>
</table>

Conclusions

Comparison of different experimental schemes of thermal diffusivity measurements using guarded hot plate shows that:

- it is possible to accurately estimate the thermal inertia of the heater, as well as the impact of the contact resistance using simplified calculation models;
- functional minimisation method can be successfully used for the estimation of the thermal diffusivity of materials on the basis of the collected measurement data.

References


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