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DETERMINATION OF THERMOPHYSICAL PROPERTIES

METHOD OF NONDESTRUCTIVE DETERMINATION OF THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS

N. P. Zhukov

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A method of nondestructive determination of the thermal conductivity and thermal diffusivity of solid materials by a section of the thermogram, which is characterized by regularization of heat fluxes, with account for registration of the time interval during which the studied object can be thought of as semi-bounded is considered.

In solution of the problem of operative control of thermophysical properties of materials, of certain interest are nondestructive methods using a linear pulse heat source on the surface of a thermally semi-bounded object. Pulse methods where a heat source acts on a comparatively small part of the specimen, thus producing a directed heat flux and, consequently, nonstationary temperature disturbance, offer wide technical possibilities [1, 2] of independent determination of two thermophysical characteristics of the specimen. The advantages of these methods are the comparative simplicity of technical realization of them and the small experimental time [3–5]. At present, studies in the field are still urgent. This is dictated, first of all, by the fact that the thermophysical properties (TPP) of a material are determined from indirect experiments and are calculated by certain mathematical models. As a result, the accuracy and reliability of determination of TPP is in many respects stipulated by how adequately the mathematical model describes the thermal processes occurring in measurement.

Analysis of measurement processes, their models, and error sources shows that within the time interval of measurements, substantial changes can take place in the thermal system that do not allow description of the whole process by one analytical model with constant restrictions and conditions [1–5].

We consider the model of nonstationary heat transfer from a linear pulse heat source acting on the heat-insulated surface of a semi-bounded body [4, 5]. In this case, the power of the heater q is a periodic function of time τ and can be written in the form

$$q(\tau) = \begin{cases} q_0, & 0 \le \tau \le \tau_0; \\ 0, & \tau_0 < \tau < \Delta\tau; \end{cases} \quad q(\tau + n\Delta\tau) = q(\tau), \quad n = 1, 2, \dots . \tag{1}$$

According to the method of sources, the formula that determines the temperature field in the half-space from the linear heat source with an arbitrary law of power supply on the heater is the following:

$$T(r, \tau) = \frac{1}{2\pi\lambda} \int_0^\tau \frac{q(u) \exp\left[-\dfrac{r^2}{4a(\tau - u)}\right]}{(\tau - u)} du . \tag{2}$$

Having substituted (1) into (2), with (1) being preliminarily expanded into Fourier series, we have the following expression for describing of the temperature field of the system with a linear heater acting on the surface of a semi-bounded body:

Tambov State Technical University (TSTU), 106 Sovetskaya Str., Tambov, 392000, Russia. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 77, No. 5, pp. 139–145, September–October, 2004. Original article submitted April 1, 2003; revision submitted October 15, 2003.

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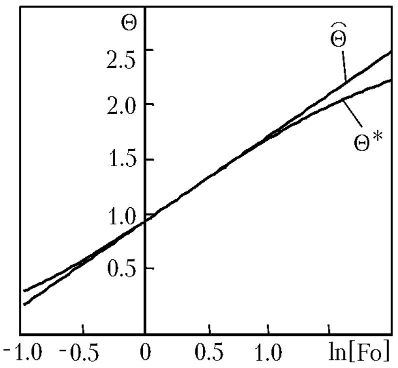

Fig. 1. Dependences $\Theta^* = f(\ln\,[\mathrm{Fo}])$ and $\overset{\frown}{\Theta} = f(\ln\,[\mathrm{Fo}])$.

$$T(r,\tau) = \frac{q_0\tau_0}{2\pi\lambda\Delta\tau} E_1\left[\frac{r^2}{4a\tau}\right] + \frac{q_0}{2\pi^2\lambda}\sum_{k=1}^{\infty}\frac{1}{k}\left\{\int_0^{\tau}\frac{\left(\sin\left[\frac{2k\pi}{\Delta\tau}(\tau_0-u)\right]+\sin\left[\frac{2k\pi u}{\Delta\tau}\right]\right)\exp\left[-\frac{r^2}{4a(\tau-u)}\right]}{(\tau-u)}du\right\}, \tag{3}$$

where $E_1[x] = \int_x^{\infty}\frac{\exp[-u]}{u}du$ is the exponential integral [3].

The second term of the right-hand side of Eq. (3) is a bounded periodic function of time [6]; when the number of pulses $n > 5$–7, this term can be neglected by referring the temperature measurements to a random error, i.e., starting from some instant of time $\tau > (5$–$7)\Delta\tau$, for the point of the surface of a semi-bounded body the dependence

$$T(r,\tau) = \frac{q_0\tau_0}{2\pi\lambda\Delta\tau} E_1\left[\frac{r^2}{4a\tau}\right] \tag{4}$$

holds.

Since real objects of measurement have finite dimensions, formula (4) will hold only during a limited time of testing. In order to fix the time in which the studied object can be thought of as semi-bounded, in the method suggested the temperature difference between two points on the object surface is measured, with the remote point being at a distance from the heater that is not larger than the thickness of the tested object. For this temperature difference we can write the equation

$$T^*(s, r_1, \tau) = \frac{q_0\tau_0}{2\pi\lambda\Delta\tau}\left(E_1\left[\frac{r_1^2}{4a\tau}\right] - E_1\left[\frac{s^2 r_1^2}{4a\tau}\right]\right). \tag{5}$$

Expression (5) can be rewritten in the dimensionless form

$$\Theta^*(s, \mathrm{Fo}) = E_1\left[\frac{1}{4\,\mathrm{Fo}}\right] - E_1\left[\frac{s^2}{4\,\mathrm{Fo}}\right], \tag{6}$$

where $\mathrm{Fo} = \frac{a\tau}{r_1^2}$ is the Fourier number (dimensionless time) and $\Theta^* = \frac{T^*}{q_0\tau_0/2\pi\lambda\Delta\tau}$ is the dimensionless temperature.

The characteristic feature of the function $\Theta^*(s, \mathrm{Fo})$ is the fact that for any specified value $s > 1$ the inflection point will be observed on the graph $\Theta^* = \Theta^*(s, \mathrm{Fo})$ (e.g., in Fig. 1 at $s = 4$, $\mathrm{Fo}^* = 1.35$, $\ln(\mathrm{Fo}^*) = 0.3$). This inflection point corresponds to the value

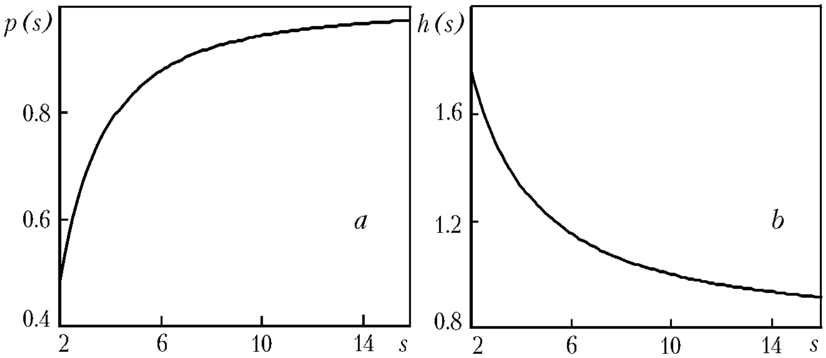

Fig. 2. Dependences $p = f(s)$ (a) and $h = f(s)$ (b).

$$\mathrm{Fo}^* = \frac{1}{8}\frac{s^2 - 1}{\ln[s]}\,, \tag{7}$$

which is obtained from the solution of the equation

$$\frac{d^2\Theta^*(s, \ln[\mathrm{Fo}^*])}{d(\ln[\mathrm{Fo}])^2} = \frac{1}{4}\exp\left[-\ln[\mathrm{Fo}^*] - \frac{1}{4\,\mathrm{Fo}^*}\right] - \frac{1}{4}s^2\exp\left[-\ln[\mathrm{Fo}^*] - \frac{s^2}{4\,\mathrm{Fo}^*}\right] = 0\,. \tag{8}$$

The tangent to the point $\Theta^*(s, \mathrm{Fo}^*)$ of the curve $\Theta^*(s, \mathrm{Fo})$ has the form (Fig. 1)

$$\overset{\frown}{\Theta}(s, \mathrm{Fo}) = p(s)\,(\ln[\mathrm{Fo}] + h(s))\,, \tag{9}$$

where $p(s)$ and $h(s)$ are found by the formulas

$$p(s) = \left(s^{-\frac{2}{s^2-1}} - s^{-\frac{2s^2}{s^2-1}}\right), \quad h(s) = 3\ln[2] - \ln\left[\frac{s^2-1}{\ln[s]}\right] + \frac{E_1\left[\frac{2\ln[s]}{s^2-1}\right] - E_1\left[\frac{2s^2\ln[s]}{s^2-1}\right]}{s^{-\frac{2}{s^2-1}} - s^{-\frac{2s^2}{s^2-1}}}$$

and are presented in Fig. 2 graphically. The inflection point will correspond to the maximum of the curve $\frac{d\Theta^*(s, \ln[\mathrm{Fo}])}{d(\ln[\mathrm{Fo}])}$ and the value

$$\frac{d\Theta^*(s, \ln \mathrm{Fo}^*])}{d(\ln[\mathrm{Fo}])} = \mathrm{Fo}^*\,\frac{d\Theta^*(s, \mathrm{Fo}^*)}{d\,\mathrm{Fo}} = p(s)\,. \tag{10}$$

Since the distance r_2 from the heater to the remote point of temperature control, relative to which the temperature of the near point on the surface of the tested object is measured, is related to its thickness, the boundary conditions begin to have an effect on the points of the experimental curve that lie more to the right of Fo^* ($\mathrm{Fo} > \mathrm{Fo}^*$). Therefore, into Eq. (6) we must introduce an additional term the value of which is the larger the more to the right relative to Fo^* is the point of the experimental thermogram (the larger the value ($\mathrm{Fo} - \mathrm{Fo}^*$)). Since the method presupposes nondestructive determination of thermophysical properties, the account for these boundary conditions in an explicit form is difficult.

With account for that formulated above and allowing for the fact that the temperature difference is measured with a random error that also involves the discarded sum of the series (expression (3)), on the experimental thermo-

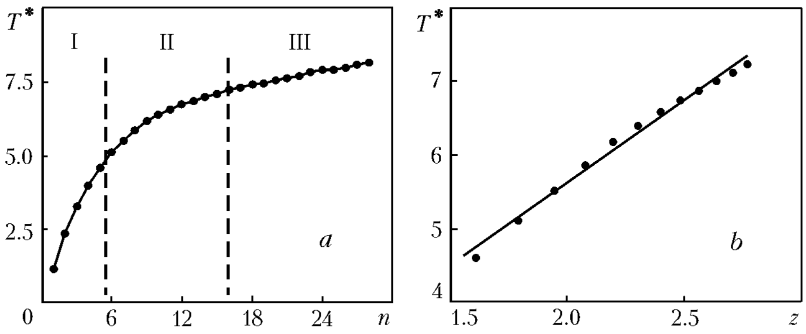

Fig. 3. Section of the experimental thermogram for ripor material (a) and the second (working) section in the coordinates T^* and z (b).

gram (Fig. 3a) in the coordinates $T^* = T^*(\ln [\tau])$ we can distinguish a rectilinear (working) section (Fig. 3b) that corresponds to the equation

$$T^* = \frac{p\,(s)\,q_0\tau_0}{2\pi\lambda\Delta\tau}\,(\ln [\tau] + \ln [a] - 2\ln [r_1] + h\,(s))\,, \tag{11}$$

which can be written as

$$T^* = \frac{\alpha}{\lambda}\,z + \frac{\alpha}{\lambda}\,(\ln [a] + \beta) \quad \text{or} \quad T^* = b_1 z + b_0\,, \tag{12}$$

where $z = \ln [\tau]$ is a new variable and α and β are the constants determined by operating and designed special features of the device that realizes the method:

$$\alpha = \frac{p\,(s)\,q_0\tau_0}{2\pi\Delta\tau}\,, \quad \beta = h\,(s) - 2\ln [r_1]\,; \tag{13}$$

b_1 and b_0 are the quantities that are determined directly from the experimental thermograms

$$b_1 = \frac{\alpha}{\lambda}\,, \quad b_0 = \frac{\alpha}{\lambda}\,(\ln [a] + \beta)\,. \tag{14}$$

As a result, the expressions for determination of the thermophysical properties of the materials studied will have the form

$$\lambda = \frac{\alpha}{b_1}\,, \quad a = \exp\left[\frac{b_0}{b_1} - \beta\right]. \tag{15}$$

This structure of the model of nonstationary heat transfer from the linear heat source acting on the thermally insulated surface of the semi-bounded body presupposes three sections to be distinguished on the thermograms (Fig. 3a).

Section I is characterized by the fact that the heat flux passing through the measurement point is time-variable and corresponds to the initial stage of the thermal process.

On section II of the thermogram, regularization of heat fluxes is observed (the heat flux passing through the measurement point becomes virtually constant).

Section III of the thermogram is characterized by the fact that the condition of nonboundedness of the studied object is violated and the heat flux passing through the measurement point becomes variable.

This structure of the model allows one to realize measurement of the thermophysical properties of the studied material on the basis of the analytical relation for section II.

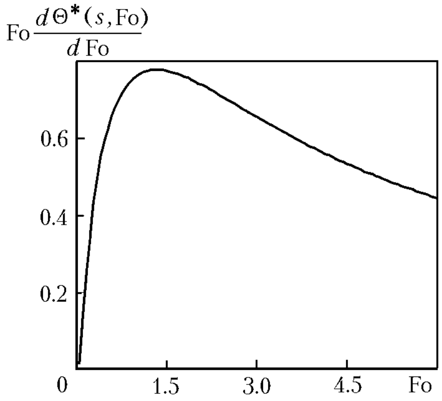

Fig. 4. Dependence $\text{Fo} = \frac{d\Theta^*(s,\text{Fo})}{d(\text{Fo})} = f(\text{Fo})$.

To determine the working sections of the experimental thermograms in the method suggested we use:

(a) properties of function (6) according to which the rectilinear sections correspond to the working sections on the thermograms in the coordinates T^* and ln [τ];

(b) qualitative information obtained in analysis of expression (6) and the derived computation relation (11) according to which the apex of the curve $dT^*(z)/dz = f(z)$ will correspond to the working section of the thermogram;

(c) statistical processing of the experimental results because the temperature difference in the experiment is recorded at discrete points with a certain random error.

Assuming that not less than k points belong to the working section of the thermogram and the total number of points is $n > k$, we consider successively the sections of the thermograms with point numbers 1, ..., k; 2, ..., $k + 1$; ...; $n - k$, ..., n. We denote each section by the subscript i ($i = k$, ..., n). For each of these sections the linear dependences

$$T^* = \alpha_{1i} z + \alpha_{0i}\,, \quad i = k, k+1, ..., n \tag{16}$$

are constructed on the basis of the following formulas:

$$b_{1i} = \frac{\sum\limits_{j=i-k+1}^{i} T_j^* (\ln(j\Delta\tau) - \bar{z}_i)}{\sum\limits_{j=i-k+1}^{i} (\ln(j\Delta\tau) - \bar{z}_i)^2}\,, \quad b_{0i} = \bar{T}_i^* - b_{1i}\bar{z}_i\,, \tag{17}$$

$$\bar{T}_i^* = \frac{1}{k} \sum_{j=i-k+1}^{i} T_j^*\,, \quad \bar{z}_i = \frac{1}{k} \sum_{j=i-k+1}^{i} \ln(j\Delta\tau)\,,$$

where T_j^* is the temperature difference obtained from the experiment at the point numbered by j for the ith section; b_{0i} and b_{1i} are the estimates of the coefficients α_{0i} and α_{1i} in Eq. (16); T^* is the temperature calculated by Eq. (16), and z = ln [τ].

On the graph that presents the dependence of b_{1i} on $\bar{z}_i$, the apex corresponding to the working section of the thermogram is observed. In order to decrease the time of the experiment, in the method suggested, b_{1i}, which is compared to $b_{1,i-1}$, is calculated at each step of measurement. The tests are completed when $b_{1i} < b_{1,i-1}$, i.e., when b_{1i} begins to decrease. In order to guarantee determination of the thermophysical properties with a specified accuracy, in the method suggested the period of supply of pulses is specified proceeding from the following. For determination of the thermal conductivity the coefficient b_1, which is found directly from the thermogram, is used. This coefficient is di-

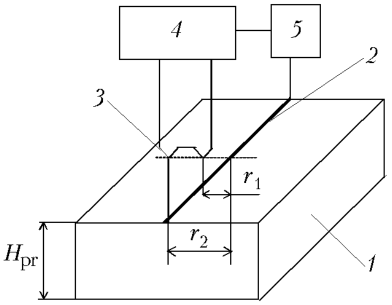

Fig. 5. Schematic of realization of the method of determination of thermophysical properties.

TABLE 1. Results of Determination of the Thermophysical Properties of Different Materials

Material	Thermophysical properties				
	by the developed method		reference data		measured by the steady-state method
	λ, W/(m·K)	$a\cdot10^7$, m^2/sec	λ, W/(m·K)	$a\cdot10^7$, m^2/sec	λ, W/(m·K)
PMMA	0.194	1.05	0.195	1.02	—
Ripor	0.027	4.45	0.028	4.61	—
PTFE	0.300	0.47	—	—	0.29
Caprolone-V	0.420	0.71	—	—	0.36

rectly proportional to the value of $p(s)$, which, in turn, is the maximum of the curve $\frac{d\Theta^*(s, \ln[\text{Fo}])}{d(\ln[\text{Fo}])} = \text{Fo}\frac{d\Theta^*(s, \text{Fo})}{d(\text{Fo})} = \exp\left[-\frac{1}{4\text{Fo}}\right] - \exp\left[-\frac{s^2}{4\text{Fo}}\right]$ (Fig. 4) corresponding to a value of Fo*. Therefore, for the points lying more to the right and more to the left of Fo*, the error δ is calculated as

$$\delta = 1 - \frac{\exp\left[-\frac{1}{4\,\text{Fo}}\right] - \exp\left[-\frac{s^2}{4\,\text{Fo}}\right]}{s^{-\frac{2}{s^2-1}} - s^{-\frac{2s^2}{s^2-1}}}\,. \tag{18}$$

Having solved this equation relative to Fo, we obtain two values: Fo$_1$, which lies more to the left of Fo*, and Fo$_2$, which lies more to the right of Fo*. Based on Fo$_1$ and Fo$_2$ the frequency of supply of pulses is specified:

$$\Delta\tau = \frac{\tau_2 - \tau_1}{k}\,, \quad \tau_1 = \frac{\text{Fo}_1 r_1^2}{a_{\max}}\,, \quad \tau_2 = \frac{\text{Fo}_2 r_1^2}{a_{\max}}\,, \tag{19}$$

where $a_{\max}$ is the maximum value from the range of determination of thermal diffusivity and k is a positive integer number larger than five.

Since for calculation of the coefficient b_1 the points that lie between τ_1 and τ_2 are used, the real error of determination of the thermophysical properties will be smaller than δ.

Implementation of this method is illustrated by the scheme given in Fig. 5. The studied specimen 1 under real conditions can represent the ready product on the free surface of which are positioned linear heater 2 and sensor 3, which measures the temperature difference between two points on the specimen surface lying at a distance r_1 and r_2 from the heater such that the following condition could be met:

$$H_{\text{pr}} < r_2 \,.$$

On preparing for the tests, thermal contacts between the heater and the specimen and between the sensor and the specimen are built up. The process of thermostating is controlled by a measurement-computation device 4. When the value of the temperature difference T^* becomes smaller than an *a priori* specified value determined by the accuracy of temperature measurement, the measurement-computation device supplies to the heater, with the aid of the stabilized power source 5, electric current by pulses (of constant power and duration $\Delta\tau$) with a frequency determined from expression (19). Simultaneously with electric current supply the temperature difference T^* is measured after each pulse.

At each ith step, the value of the coefficient b_{1i} is determined according to (17) and is compared with the value of $b_{1,i-1}$ at the $(i-1)$th step of measurement. The tests terminate when the condition $b_{1i} < b_{1,i-1}$ is met. According to the above-formulated technique, the thermophysical properties are determined by the formulas

$$\lambda = \frac{\alpha}{b_{1,i-1}}\,, \quad a = \exp\left[\frac{b_{0,i-1}}{b_{1,i-1}} - \beta\right]. \tag{20}$$

In order to verify the technique [7, 8], measurements were made on polymethyl-methacrylate (PMMA) and ripor specimens with the known thermophysical properties and on specimens made of polytetrafluoroethylene (PTFE) and caprolone-V whose thermal conductivity was determined by the steady-state method using an IT-3 thermophysical device (see Table 1).

The tests showed that the obtained values of thermal conductivity and thermal diffusivity agree with the known values of these quantities and those determined by the steady-state method.

NOTATION

a, thermal diffusivity; $\Delta\tau$, pulse-repetition interval; Θ^*, dimensionless temperature; b_1 and b_0, quantities determined directly from the experimental thermograms; q, heater power; q_0, power of one pulse per unit length of the heater; λ, thermal conductivity; Fo, Fourier number; Fo^*, value of the Fourier number to which the inflection point of the function $\Theta^*(s, \text{Fo}^*)$ corresponds; r, distance between the line of heat-source action and the control point; r_1, distance between the line of heat-source action and the near point of control; r_2, distance between the line of heat-source action and the remote control point; $s = r_2/r_1$; T, temperature; T^*, difference of temperature between two points on the surface of the tested object; τ, time; τ_0, duration of one pulse; u, integration parameter; H_{pr}, specimen (product) thickness. Indices: max, maximum value; pr, product.

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