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METHODOLOGICAL PECULIARITIES OF SHORT MEASUREMENTS AT THE STAGE OF IRREGULAR THERMAL REGIME

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The article discusses the peculiarities of thermophysical measurements when low-inertia heaters are used; it also presents the results of measurements of thermal activity.

The present technical state and the technology of applying thin conducting films open up broad possibilities of using them in thermophysical experiments. Particularly promising is their use as low-inertia elements for short impulse measurements at the stage of irregular thermal regime. With their aid it is possible to study the thermophysical characteristics of such objects as liquids, gases, or solids that ensure adhesion to the layers that had been vaporized on. Calculations show [1] that when vaporized-on resistor elements (RE) are used, the influence of the proper heat capacity of the element on the results of measuring the thermal activity of a solid or liquid is practically insignificant already when impulse measurements last 10^{-5}-10^{-4} sec. When elements with extremely small thickness (70-80 Å) are used, this time can be reduced to 10^{-6} sec.

In short-term thermophysical experiments, thin metal filaments can be used together with vaporized-on layers. The technology of making such filaments has by now been well mastered. Evaluations of the influence of the proper heat capacity of a filament on the results of the measurement of thermal conductivity show [2] that for a filament with radius 10^{-6} m the influence of the heat capacity is small already when the impulses last so much as 10^{-3} sec.

Since the measurements are short, the method is bound to have a number of advantages. In particular, favorable conditions are created for using thermal measurements to diagnose dynamic processes. Some experience in using thermal diagnostics for studying chemical reactions, phase transformations, diffusion and other processes has already found expression in various works [3-8].

The smallness of the spatial region in which the temperature field is non-steady-state is another favorable feature of these measurements: heat transfer occurring under such specific conditions reflects the molecular heat transfer that is only slightly distorted by radiation [9, 10]. In consequence, short measurements have an advantage as a matter of principle as compared with a number of other methods, in particular, steady-state methods.

On the other hand, if methods of short measurement are to be applied correctly, we must examine the peculiarities of this application occasioned by the small length of diffusion of the temperature field into the investigated medium, by the considerable temperature gradient, etc.

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First of all we will examine the correctness of using Fourier's basic law $q = -\lambda \nabla T$ for describing heat transfer in the spatial region characteristic of short-term experiments. For this purpose we evaluate the values of the Knudsen numbers ($Kn = l/l_{ef}$). When the measurements last 10^{-5}–10^{-3} sec, for most dielectrics (liquids and solids), the length of diffusion of the temperature field into the investigated medium (l_{ef}) amounts to 10^{-6}–10^{-5} m, whereas the mean free path length of a phonon (l) of the parameter determining molecular heat transfer is characterized by values of 10^{-9} m. Thus, the Knudsen numbers are small (10^{-3}–10^{-4}), and it is therefore permissible to view the medium as a homogenous continuum.

It is known that the basic law of heat conductivity in its accurate form corresponds only to infinitely large speed of propagation of heat, whereas the real speed is finite. For highly intensive processes, the additional term [11] $\delta q = -\tau(\partial q/\partial t)$ has to be introduced into the expression for the thermal flux.

We will examine to what extent it is justified to neglect it under conditions of short-term experiments. We estimate the relative magnitude of the additional term on the assumption that it is small ($\delta q/q$) $\ll 1$. For this purpose we present the model thermal problem on which the measurements of thermal activity are based (we recall that these measurements are briefest):

$$\begin{gathered}\frac{\partial T_i}{\partial t} - a_i \frac{\partial^2 T_i}{\partial x^2} = 0 \quad (i = 1,2), \quad t \geqslant 0, \\ T_1 = T_2 = 0 \quad \text{for} \quad t = 0, \\ T_1 = 0, \; T_2 = 0 \quad \text{for} \quad |x| \to \infty, \\ T_1 = T_2, \; \lambda_1 \frac{\partial T_1}{\partial x} - \lambda_2 \frac{\partial T_2}{\partial x} = q_0 \quad \text{for} \quad x = 0.\end{gathered} \tag{1}$$

The subscript 1 corresponds to the substrate, 2 to the investigated medium. The solution of (1) has a simple form

$$T(x, t) = \frac{2q_0 t^{\frac{1}{2}}}{\varepsilon_1 + \varepsilon_2} \operatorname{ierfc} \frac{x}{2\sqrt{a_2 t}} \quad (x \geqslant 0). \tag{2}$$

Correspondingly, the temperature in the interface between the substrate and the medium (x = 0), i.e., the temperature of the resistor element (whose thickness is assumed to be infinitesimal) is equal to

$$T(x = 0, t) = 2q_0 t^{\frac{1}{2}} / \sqrt{\pi}(\varepsilon_1 + \varepsilon_2). \tag{3}$$

Using (2), we write the expression of the thermal flux in the investigated medium

$$q(x, t) = -\lambda_2 \frac{\partial T}{\partial x} = \frac{q_0 \varepsilon_2}{\sqrt{\pi}(\varepsilon_1 + \varepsilon_2)} \operatorname{erfc} \frac{x}{2\sqrt{a_2 t}}, \; x \geqslant 0. \tag{4}$$

Hence we have for the relative magnitude of the additional term:

$$\left| \frac{\delta q}{q} \right| = \tau \frac{\exp\left(-\frac{x^2}{4at}\right) \frac{x}{\sqrt{a_2 t}} \frac{1}{2t}}{\sqrt{\pi} \operatorname{erfc}\left(\frac{x}{2\sqrt{a_2 t}}\right)}. \tag{5}$$

For approximate estimates it suffices to have the value of the ratio (5) only for the coordinate that is equal to the length of diffusion of the temperature field into the investigated medium $l_{ef} = (a_2 t)^{1/2}$. In this case, (5) is simplified

$$\frac{\delta q}{q} \sim \tau \frac{a_2}{l_{ef}^2}. \tag{6}$$

It can be easily seen that the additional term in the generalized Fourier law is small. In fact, when $l_{ef} \sim 10^{-6}$ m (for $\tau \sim 10^{-10}$ sec), we have ($\delta q/q$) $\sim 10^{-5}$.

It is known that the surface of a solid acts on an adjacent liquid by the forces of intermolecular attraction or repulsion. Specifically, on the interface between a wetting liquid and a solid, a polymolecular, structurally inhomogeneous layer forms. In [12, 13] it was shown that these layers are 10^{-8}–10^{-7} m thick, and the macroscopic properties differ from the properties of the bulk of the liquid.

Thus, the boundary of the solid changes the thermophysical characteristics of the liquid, and this has to be taken into account in substantiating the method of short measurements. For quantitative evaluations we represent the absorption layer in the form of a film with thickness L which is homogeneous as to its proper-

ties. Since the time of measurement is chosen such that the influence of the proper heat capacity is small, we will assume, like in (1), that its thickness is infinitesimally small. In that case, the following model corresponds to the measurement:

$$\begin{gathered}\frac{\partial T_i}{\partial t}=a_i\frac{\partial^2 T_i}{\partial x^2}\ (i=1,\ 2,\ 3,),\\ T_1=T_2=T_3\ \text{ for }\ t=0,\\ T_1\to 0\ \text{ for }\ x\to-\infty,\ T_1=T_3\ \text{ for }\ x=0,\\ T_3=T_2\ \text{ for }\ x=L,\ T_2\to 0\ \text{ for }\ x\to\infty,\\ \lambda_1\frac{\partial T}{\partial x}-\lambda_3\frac{\partial T_3}{\partial x}=q_0\ \text{ for }\ x=0,\\ \lambda_3\frac{\partial T_3}{\partial x}-\lambda_2\frac{\partial T_2}{\partial x}=0\ \text{ for }\ x=L.\end{gathered}\tag{7}$$

This problem was examined in [14] in connection with the substantiation of the method of investigating the thermophysical characteristics of thin layers of a liquid.

For our purposes it is advisable to write the temperature of the RE (x = 0) in the form of an expansion in powers of the small magnitude $1/\sqrt{\mathrm{Fo}}$ ($\mathrm{Fo}=a_3 t/L^2$):

$$T(x=0)=\frac{2q_0\sqrt{t}}{\sqrt{\pi}(\varepsilon_1+\varepsilon_2)}\left[1+\frac{\sqrt{\pi}}{2}\frac{(\varepsilon_2^2-\varepsilon_3^2)}{\varepsilon_3(\varepsilon_1+\varepsilon_2)}\frac{1}{\sqrt{\mathrm{Fo}}}+O\left(\frac{1}{\sqrt{\mathrm{Fo}}}\right)\right].\tag{8}$$

The influence of the adsorption layer on the temperature of the RE is viewed here as a small "distortion." We want to point out that the factor in front of the square bracket corresponds to the solution of (3). It follows from (8) that the test for neglecting the adsorption layer may be written in the form

$$\mathrm{Fo}\gg\frac{\pi}{4}\left[\frac{\varepsilon_2^2-\varepsilon_3^2}{\varepsilon_3(\varepsilon_1+\varepsilon_2)}\right]^2.\tag{9}$$

Correspondingly, the relative magnitude of the "distortion" is

$$\left|\frac{\delta T}{T}\right|=\frac{\sqrt{\pi}}{2}\frac{|\varepsilon_2^2-\varepsilon_3^2|}{\varepsilon_3(\varepsilon_1+\varepsilon_2)\sqrt{\mathrm{Fo}}}.\tag{10}$$

An idea of the extent of the influence of the adsorption layers with thickness $L_1 = 10^{-8}$ m and $L_2 = 10^{-7}$ m is provided by Fig. 1. The calculation was carried out for the following conditions: $\varepsilon_2 = 600$ J/m$^2 \cdot$ deg K $\cdot$ sec$^{1/2}$, $\varepsilon_3 = 1500$ J/m$^2 \cdot$ deg K $\cdot$ sec$^{1/2}$, $a_3 = 10^{-7}$ m^2/sec, the difference in thermal activity of the adsorption layer and the bulk of the liquid was taken equal to 20%. The figure also shows, according to the results of [1], the influence of the proper heat capacity of the RE on the results of the measurement of its temperature. The resistive layer (nickel) was 200 Å thick. The values of ε_1 and ε_2 were the same as in the preceding example.

A comparison of the dependences shows that when $L_1 = 10^{-8}$ m, the influence of the proper heat capacity predominates. In this case the selection of the minimum duration of the measurement is determined by the heat capacity of the RE. When $L_2 = 10^{-7}$ m, the influence of the adsorption layer is commensurate with the distortions of the results of measuring the proper heat capacity of the RE. Naturally, it is possible to imagine a case when the influence of the adsorption layer predominates. This will distort the results of the investigation because the averaged thermophysical properties of the layers near the wall are measured, and these differ from the properties in the bulk of the liquid. To eliminate this, it is necessary to increase the length of the measurement. It can be seen from Fig. 1 that the influence of the adsorption layer decreases noticeably with an increase in time.

Since the scale of the investigated inhomogeneity in each actual case is unclear, it is expedient in setting up metrological measurements with low-inertia elements to carry out control experiments. In the final analysis it is indispensable to compare the results of measurements carried out with different lengths of impulses, or in other words, in sounding the liquid to different depths. A similar approach was applied by Filippov [15], who measured the thermal activity of a number of organic liquids while sounding with temperature waves of different frequencies (maximum frequency of the current generating the thermal flux was 165 Hz). For the experimental detection of an inhomogeneity it is expedient to use a compensation circuit. The experience of its application for studying surface inhomogeneities of the bases of resistance sensors with thin films was examined in [16]. Thus it must be pointed out that the influence of the adsorption layers is slight and will be imperceptible in measurements lasting 10^{-4} sec or more. However, in more rapid measurements (lasting

10^{-5} or 10^{-6} sec), the influence of the adsorption layer in substantiating the accuracy of the measurements has to be taken into account.

When low-inertia elements are used, especially vaporized-on resistive layers, attention must be principally given to their structure. It must be borne in mind that the influence of the proper heat capacity of vaporized-on layers in [1] was based on the homogeneity of the conducting layer across its thickness and on the uniform distribution of the sources of Joulean heat throughout its bulk. In consequence of this, the presence of flaws in the structure of the vaporized-on element or its nonuniform thickness may lead to such distortions of the temperature field that are difficult to take into account in the theoretical model.

The structure of vaporized-on metallic layers may change from fine-grained, which are detected only with magnifications attained in electron optics, to coarse-grained, observed with ordinary optical means. The process of vaporizing on the film in vacuum depends on a large number of factors such as the cleanliness of the substrate, its temperature, the presence of an adsorbed gas, the entrance angle of the metal ions, the pressure in the system, etc. To a considerable extent they are not reproducible, and therefore the film depends on the conditions of obtaining it. Consequently, it is fairly complicated to work out the technology of applying metal coatings that reproduces, e.g., with small deviations such indicators as resistance or the resistance temperature coefficient. However, the problem of producing fine-grained structures that have mechanical strength is simpler and feasible. Such films in particular are suitable for short measurements. With the characteristic grain size of 100 Å and their uniform distribution over the surface of the base, the pattern of the temperature field is fairly complicated only at the initial stage of heating the element. As the temperature field diffuses into the medium, the temperature inhomogeneities are statistically averaged, and when $l_{ef} \sim 10^{-6}$ m, we may obviously speak fairly strictly of plane symmetry of such a field.

Short measurements are characterized by small length of diffusion of the temperature field into the medium; this length is substantially smaller than the dimensions of the element (length and width) and of the investigated system. Since the distribution of the temperature field and its peculiarities associated with the fulfillment of the boundary conditions are a function of the dimensionless time $Fo = at/L^2$ (L is the characteristic dimension), we may say that with the same accuracy of specifying the boundary conditions, the characteristic dimension of the element and of the investigated system decrease with decreasing length of measurement by a factor $\sim t^{1/2}$. Thus, we find yet another feature of short measurements: they can be effected with small sensors and with small amounts of the substance. It is therefore of interest to evaluate the error of the measurements associated with the fulfillment of the boundary conditions. In connection with linear heat sources, this problem was examined in [2], and we will therefore confine ourselves to the examination of plane REs.

We will evaluate the error caused by the deviation of a real physical model of measurement from the idealized unidimensional model, i.e., we take into account the finiteness of the width of the resistive base. Since in real elements the length substantially (5-10 times) exceeds the width, this evaluation is decisive. We use the results of Drozdov and Salokhin [17], who examined heating with a constant heat flux of an infinitely long strip of width l, situated at the interface of two semibounded media. For the case of equal media, e.g., media identical with the base (this is admissible for evaluations), the mean temperature of the element is determined by the expression

$$T = \frac{1}{l}\int_{-l/2}^{l/2} T(x=0,\ y)\,dy = \frac{q_0 t^{1/2}}{\sqrt{\pi}\,\varepsilon}\left[1 - \frac{Fo^{1/2}}{\sqrt{\pi}} + \frac{8\,Fo^{5/2}}{\sqrt{\pi}}\exp\left(-\frac{1}{4Fo}\right) + \ldots\right] (Fo = at/l^2). \qquad (11)$$

We want to point out that the factor in front of the bracket in (11) is merely the solution of (3) (the case of $\varepsilon_1 = \varepsilon_2 = \varepsilon$), and the variable terms of the series express the influence of the edge effects. Now it is easy to write the expression for the relative error caused by heat exchange at the edges of the resistive path:

$$|\delta T/T| \approx Fo^{1/2}/\sqrt{\pi}. \qquad (12)$$

For instance, for the case of a glass base and duration of measurement 10^{-4} sec, with the element 10^{-3} m wide, this error amounts to about 0.3%. If the measurement lasted 1 sec and the partial error were specified, the width of the element would have to be increased to 10^{-1} m.

Using the results of [14], we can easily write the condition of semiboundedness of the investigated medium. In the model problem (1) the medium is assumed to be semibounded, and in fact it has finite thickness (L) bounded, e.g., by the wall of the vessel. The corresponding test has the form

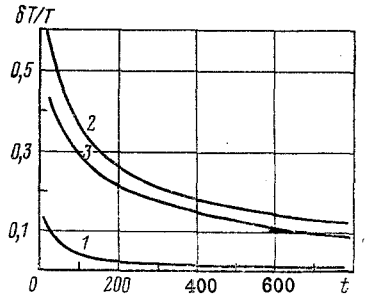

Fig. 1

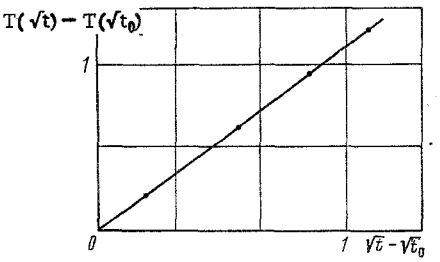

Fig. 2

Fig. 1. Distortion of the temperature of the resistor element by the adsorption layers $L_1 = 10^{-8}$ m (1), and $L_2 = 10^{-7}$ m (2), and by the proper heat capacity of the element (3). $\delta T/T$, %; t, μsec.

Fig. 2. Change in the temperature of the plane element in dependence on the duration of heating. $T(\sqrt{t}) - T(\sqrt{t_0})$, °C; $\sqrt{t} - \sqrt{t_0}$, $\sec^{1/2}$.

$$\frac{2\sqrt{\pi}\,\varepsilon_2}{\varepsilon_1 - \varepsilon_2} \sum_{n=1}^{\infty} \mu^n \operatorname{ierfc} \frac{n}{\sqrt{\mathrm{Fo}}} \ll 1, \tag{13}$$

where

$$\mu = (\varepsilon_1 - \varepsilon_2)(\varepsilon_3 - \varepsilon_2)/(\varepsilon_1 + \varepsilon_2)(\varepsilon_3 + \varepsilon_2).$$

When the layer is more than 10^{-4} m thick and the impulse lasts 10^{-3} sec, the corresponding error is extremely small (less than 10^{-2}%), regardless of the properties of the material of the vessel or the medium bounding the investigated substance.

It is an advantage to verify experimentally how the real physical process of measurement corresponds to its mathematical idealization. When plane elements are used, the result of such correspondence is the increase in temperature of the element according to the regularity $\sim t^{1/2}$. Naturally, the boundary conditions (1) have to be satisfied, too, in particular, a constant heat output has to be effected on the element in the course of the selected time.

In accordance with the method of [7] of determining the "instantaneous" values of the resistance (temperature), we investigated the increase in temperature of the heat sensors vaporized onto glass. The temperature measurements of the element were carried out for the following instants of time (this time is measured from the beginning of the heating): $t_0 = 5 \cdot 10^{-5}$ sec and $t = 10^{-4}$, $2 \cdot 10^{-4}$, $3 \cdot 10^{-4}$, $4 \cdot 10^{-4}$ sec. The results of one of the experiments, carried out in the form $[T(\sqrt{t}) - T(\sqrt{t_0})] - (\sqrt{t} - \sqrt{t_0})$, are presented in Fig. 2. It can be seen from the figure that the results of the experiments well reproduce the linear dependence (maximum deviation of the points from the averaging straight line does not exceed 0.3%); this indicates that the law (3) of the change of temperature of the element in dependence on the heating time can be fulfilled. This confirms that the real process of measurement corresponds well to its idealization.

The slope of the straight line in the graph ($\tan\varphi = [T(\sqrt{t}) - T(\sqrt{t_0})]/(\sqrt{t} - \sqrt{t_0})$) determines the thermal activity of the substrate. A calculation procedure analogous to [7] yielded that the thermal activity of the investigated base amounted to 1450 $J/m^2 \cdot \deg K \cdot \sec^{1/2}$ (at 20°C). The error of measurements included the evaluation of the following factors: proper heat capacity of the element, finite width of the resistive path, and also the instrument error of determining the area of the path, the stress acting on the element, the resistance temperature coefficient, and the magnitude of $\tan\varphi$. For the investigated heat sensors with the following parameters of the resistive path: thickness 200 Å, width 2 mm, and length 20 mm, the resulting error of measurement amounted to no more than 2%. Heating of the element at the end of the impulse did not exceed 2°C.

Analogous measurements, carried out with the heat sensors immersed in the liquid, made it possible to determine its thermal activity. Specifically, we carried out measurements in toluene and obtained the value of its thermal activity equal to 438 $J/m^2 \cdot \deg K \cdot \sec^{1/2}$ (at 20°C). The corresponding analysis of the error of the given measurement shows that with the error of the thermal activity of the base taken into account, it does not amount to more than 3%.

NOTATION

T, temperature, °C; q, specific heat flux, W/m^2; λ, thermal conductivity, W/deg K · m; a, thermal diffusivity, m^2/sec; ε, coefficient of thermal activity, $J/m^2 \cdot deg\,K \cdot sec^{1/2}$; t, time, sec; τ, relaxation time, sec; x, y, running coordinates, m. Subscripts: 1, substrate; 2, investigated medium and liquid; 3, adsorption layer and material of the vessel wall or other bounding medium.

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