Journal of Engineering Physics and Thermophysics, Vol. 71, No. 5, 1998

FEATURES OF THE METHOD OF IRREGULAR THERMAL REGIME IN STUDIES OF THE THERMAL CONDUCTIVITY OF SOLIDS

O. V. Belyaev, G. G. Spirin,
V. F. Formalev, and N. Yu. Nenarokov

UDC 536.2.08

A method of short-duration measurements was used in the stage of irregular thermal regime to evaluate the effect of contact resistance on measurements of the thermal conductivity of solids.

The contemporary level of heat technology imposes serious demands on methods of measuring thermal properties as regards their rapidity and the accuracy of the obtained experimental data. In this connection, in recent years an unsteady-state short-duration research method has been actively developed that uses the initial (irregular) stage of heat exchange between the sensor and the material of the sample. Short periods of measurements induce diffusion of the temperature field into the studied material. This diffusion is much shorter than the mean free path of photons and allows obtaining experimental data that practically are not distorted by the radiant component of the heat transfer. The probe in the form of a thin filament is located on the interface between the specimen and an elastically deformable material (substrate) that improves the thermal contact when pressed to the surface of the specimen. The probe is a source of thermal power when heated by short rectangular current pulses and allows determination of the temperature of the specimen by measuring its resistance. The measurements are taken in an atmosphere of inert gas. The experimental procedure, the experimental setup, and the scheme of measurement are given in [1-3].

The idealized model of the method considers a source with an infinitesimal radius and an intrinsic heat capacity. The source is located at the interface between the studied and elastically deformed media. A solution of the model problem is the known logarithmic relation [4]

$$\Delta T(r_0, t_2) - \Delta T(r_0, t_1) = \frac{q_L}{2\pi(\lambda_1 + \lambda_2)} \ln\left(\frac{t_2}{t_1}\right). \tag{1}$$

We consider the actual location of the sensor filament relative to the studied material (Fig. 1). The filament is shifted toward the elastically deformable material by the magnitude of the radius. The filament is in contact simultaneously with three materials, namely, the elastically deformable material, the solid material, and the inert gas. The area of contact with the elastically deformable material is the largest. The goal of the present work is to study the effect of these factors, which are a consequence of the asymmetric location of the filament, on heat transfer between the filament and the materials as concerns deviation of the temperature increment from the idealized logarithmic relation (1). In summary, these factors can be represented in terms of the contact resistance between the sensor and the studied material. The mathematical model that is closest to the actual experimental conditions is as follows (see Fig. 2a): the heat-conduction equation

$$c_i \rho_i \frac{\partial T}{\partial t} = \frac{\lambda_i}{r} \frac{\partial}{\partial r}\left(r \frac{\partial T}{\partial r}\right) + \frac{\lambda_i}{r^2} \frac{\partial^2 T}{\partial \theta^2}, \quad r, \theta \subset Q_i, \quad i = 1, 2, 4;$$

Moscow Aircraft Institute, Moscow, Russia. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 71, No. 5, pp. 805-810, September-October, 1998. Original article submitted August 27, 1996; revision submitted March 31, 1998.

 1062-0125/98/7105-0788$20.00

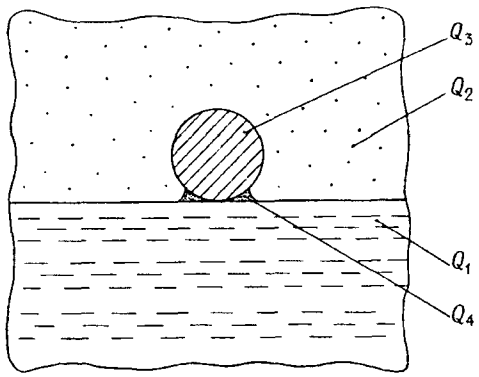

Fig. 1. Location of the sensor filament relative to the studied material.

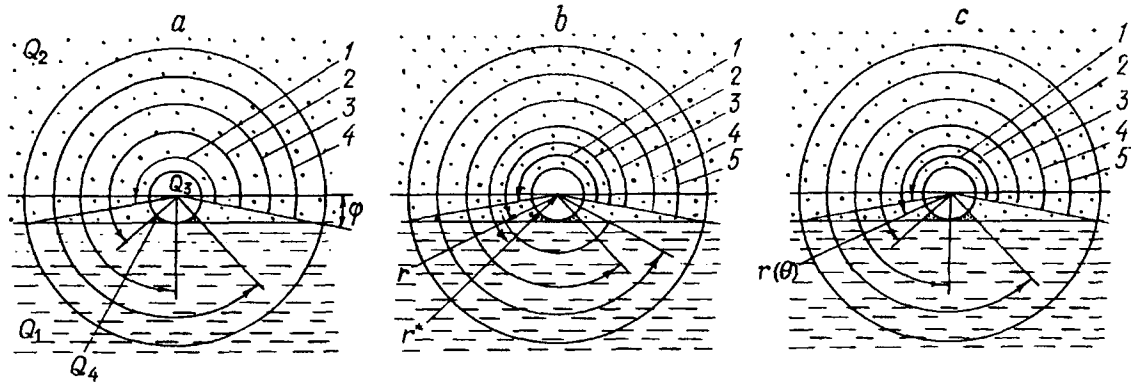

Fig. 2. Domains of definition of the equations of the initial two-dimensional problem (a) [1) θ_0, 2) θ_2, 3) θ_1, 4) θ_3], of the longitudinal one-dimensional subproblem (b) [1) θ_0, 2) $\theta(r)$, 3) θ_2, 4) θ_3, 5) θ_4], and of the radial one-dimensional subproblem (c) [1) θ_0, 2) θ, 3) θ_2, 4) θ_1, 5) θ_3].

$$c_3\rho_3\frac{\partial T}{\partial t}=\frac{\lambda_3}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T}{\partial r}\right)+\frac{\lambda_3}{r^2}\frac{\partial^2 T}{\partial\theta^2}+q_L/\pi r_0^2,\quad r,\theta\subset Q_3;$$

boundary conditions of the fourth kind:

a) between the studied material and the substrate and between the studied material and the inert gas:

$$\lambda_1\frac{\partial T}{\partial n}\bigg|_{n-0}=\lambda_i\frac{\partial T}{\partial n}\bigg|_{n+0},\quad i=2,4;\quad T|_{n-0}=T|_{n+0};$$

b) between the probe and the substrate, the probe and the inert gas, and the probe and the studied material:

$$\lambda_3\frac{\partial T}{\partial r}\bigg|_{r=r_0-0}=\lambda_i\frac{\partial T}{\partial r}\bigg|_{r=r_0+0},\quad i=1,2,4;\quad T|_{r=r_0-0}=T|_{r=r_0+0};$$

c) the condition of constancy of the temperature at infinity:

$$\lambda_i\frac{\partial T}{\partial r}\bigg|_{r\to R}=0,\quad i=1,2;$$

the initial condition

$$T(r;\theta;0)=T_{\text{in}}.$$

In these equations the subscript i is chosen from the condition of occurrence of Q_i in the corresponding region (see Fig. 2a). This system of equations is to be solved by the method of fractional steps [5]. By fixing the

angle or the radius in the division grid, the solution of the two-dimensional problem is reduced to successive solution (alternation in the time lattice) of two one-dimensional subproblems: longitudinal one-dimensional and radial one-dimensional ones.

The longitudinal one-dimensional problem (see Fig. 2b). The heat-conduction equation

$$c_1 \rho_1 \frac{\partial T}{\partial t} = \frac{\lambda_1}{r^2} \frac{\partial^2 T}{\partial \theta^2}, \quad r_0 < r < R, \quad \theta(r) < \theta < \theta_4;$$

$$c_2 \rho_2 \frac{\partial T}{\partial t} = \frac{\lambda_2}{r^2} \frac{\partial^2 T}{\partial \theta^2}, \quad r_0 < r < R, \quad \{0 < \theta < \theta(r) \cup \theta_4 < \theta \leq 2\pi\} \setminus \{\theta_2 \leq \theta \leq \theta_3\};$$

$$c_3 \rho_3 \frac{\partial T}{\partial t} = \frac{\lambda_3}{r^2} \frac{\partial^2 T}{\partial \theta^2} + q_L/\pi r_0^2, \quad \varepsilon < r < r_0, \quad 0 < \theta \leq 2\pi;$$

$$c_4 \rho_4 \frac{\partial T}{\partial t} = \frac{\lambda_4}{r^2} \frac{\partial^2 T}{\partial \theta^2}, \quad r_0 < r < r^*, \quad \theta_2 \leq \theta < \theta(r) \cup \theta_4 < \theta \leq \theta_3;$$

boundary conditions of the fourth kind: between the studied material and the substrate, the studied material and the inert gas, and the substrate and the inert gas:

$$\lambda_2 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)-0} = \lambda_1 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)+0}, \quad r^* < r < R,$$

$$\lambda_4 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)-0} = \lambda_1 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)+0}, \quad r_0 < r < r^*,$$

$$\lambda_4 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)-0} = \lambda_2 \frac{1}{r} \frac{\partial T}{\partial \theta}\bigg|_{\theta(r)+0}, \quad r = r^*,$$

$$T|_{\theta(r)-0} = T|_{\theta(r)+0}, \quad r_0 < r < R;$$

the initial condition

$$T(r, \theta, 0) = T_{\text{in}}, \quad \varepsilon < r \leq R, \quad 0 < \theta \leq 2\pi.$$

The radial one-dimensional problem (see Fig. 2c). The heat-conduction equation where $r(\theta) = r_0/\sin(\theta - \pi - \varphi)$;

$$c_2 \rho_2 \frac{\partial T}{\partial t} = \frac{\lambda_2}{r} \frac{\partial}{\partial r}\left(r \frac{\partial T}{\partial r}\right), \quad r_0 < r < r(\theta), \quad 0 < \theta < \theta_2 \cup \theta_3 < \theta < 2\pi;$$

$$c_3 \rho_3 \frac{\partial T}{\partial t} = \frac{\lambda_3}{r} \frac{\partial}{\partial r}\left(r \frac{\partial T}{\partial r}\right) + q_L/\pi r_0^2, \quad \varepsilon < r < r_0, \quad 0 < \theta \leq 2\pi;$$

$$c_4 \rho_4 \frac{\partial T}{\partial t} = \frac{\lambda_4}{r} \frac{\partial}{\partial r}\left(r \frac{\partial T}{\partial r}\right), \quad r_0 < r < r(\theta), \quad \theta_2 \leq \theta < 1.5\pi + \varphi \cup 1.5\pi + \varphi < \theta \leq \theta_3;$$

boundary conditions of the fourth kind:

a) between the studied material and the substrate, the studied material and the inert gas:

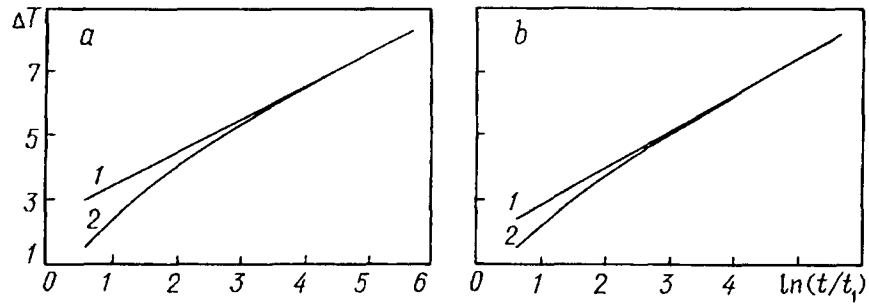

Fig. 3. Results of a calculation of the time dependence of the temperature increment of the filament for the system of platinum–quartz glass–HPMQ–argon ($r_0 = 1 \cdot 10^{-5}$ m): a) for $T_{in} = 300$ K, b) 1300 K; 1) logarithmic curve corresponding to the idealized mathematical model, 2) calculated curve of the temperature increment versus time, $t_1 = 0.01$ sec is the reference time.

$$\lambda_2 \frac{\partial T}{\partial r}\bigg|_{r=r(\theta)-0} = \lambda_1 \frac{\partial T}{\partial r}\bigg|_{r=r(\theta)+0}, \quad r > r_0, \quad \theta_0 < \theta < \theta_2 \cup \theta_3 < \theta \leq 2\pi;$$

$$\lambda_4 \frac{\partial T}{\partial r}\bigg|_{r=r(\theta)-0} = \lambda_1 \frac{\partial T}{\partial r}\bigg|_{r=r(\theta)+0}, \quad r > r_0, \quad \theta_2 \leq \theta < 1.5\pi + \varphi \cup 1.5\pi + \varphi < \theta \leq \theta_3;$$

$$T|_{r=r(\theta)-0} = T|_{r=r(\theta)+0}, \quad r > r_0, \quad \theta_0 < \theta \leq 2\pi;$$

b) between the probe and the substrate, the probe and the inert gas, and the probe and the studied material:

$$\lambda_3 \frac{\partial T}{\partial r}\bigg|_{r=r_0-0} = \lambda_2 \frac{\partial T}{\partial r}\bigg|_{r=r_0+0}, \quad 0 < \theta < \theta_2 \cup \theta_3 < \theta \leq 2\pi;$$

$$\lambda_3 \frac{\partial T}{\partial r}\bigg|_{r=r_0-0} = \lambda_4 \frac{\partial T}{\partial r}\bigg|_{r=r_0+0}, \quad \theta_2 \leq \theta < 1.5\pi + \varphi \cup 1.5\pi + \varphi < \theta \leq \theta_3;$$

$$\lambda_3 \frac{\partial T}{\partial r}\bigg|_{r=r_0-0} = \lambda_1 \frac{\partial T}{\partial r}\bigg|_{r=r_0+0}, \quad \theta = 1.5\pi + \varphi;$$

$$T|_{r=r_0-0} = T|_{r=r_0+0}, \quad 0 < \theta \leq 2\pi;$$

c) the condition of constancy of the temperature at infinity:

$$\lambda_1 \frac{\partial T}{\partial r}\bigg|_{r\to R} = 0, \quad \theta_0 < \theta \leq 2\pi; \quad \lambda_2 \frac{\partial T}{\partial r}\bigg|_{r\to R} = 0, \quad 0 < \theta \leq \theta_0;$$

the initial condition

$$T_{in} = T(r; \theta; t)$$

from the solution of the longitudinal problem, $\varepsilon < r \leq R$, $0 < \theta \leq 2\pi$.

Numerical solution. Approximation of the differential equations of the one-dimensional subproblems (for the radial one over the radius and for the longitudinal one over the angle) at each inner point of the division grid and of the boundary conditions at the boundary points gives a closed system of linear algebraic equations. Its solution is an approximate solution of the initial problem. A solution for the radial problem is sought by the factorization method and that for the longitudinal problem, by the cyclic-factorization method [6].

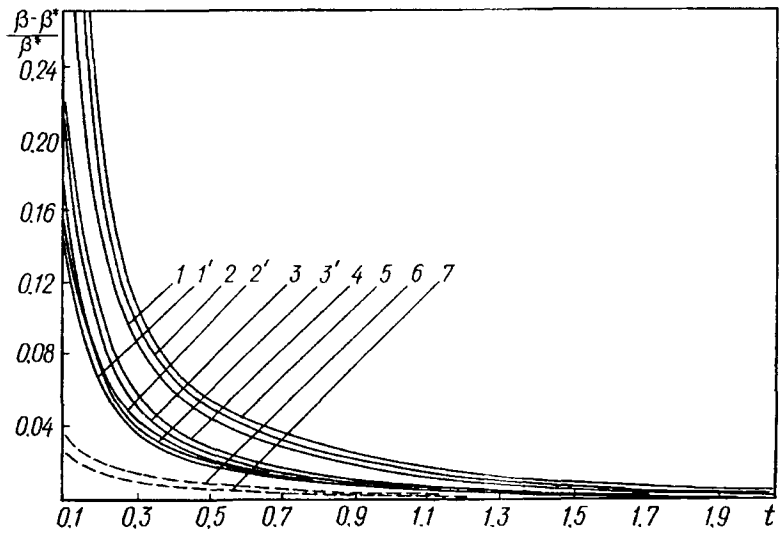

Fig. 4. Deviation of the slope of the curve of the excess temperature of the sensor filament to the logarithmic time axis (β) relative to the steady-state value (β^*) ($r_0 = 1 \cdot 10^{-5}$ m): 1, 1') for the system of platinum–quartz glass–HPMQ–argon (300 and 1300 K, respectively), 2, 2') platinum–single crystal LiF–HPMQ–argon (300 and 1000 K), 3, 3') platinum–organic glass–HPMQ–argon (300 and 380 K), 4) platinum–quartz glass–HPMQ –helium (300 K), 5) platinum–quartz glass–foam plastic–argon (300 K), 6, 7) maximum effect of the intrinsic thermal conductivity of the sensor filament for systems 1-4 and 5, respectively (300 K).

The angle θ_2 does not remain constant from one experiment to another and depends, in particular, on the force of pressing of the substrate to the surface of the specimen. Therefore, calculation was carried out in the whole range of this angle 220–240° (in view of the property of elastic deformability of the substrate). Later in this work we give results of a calculation for the most unfavorable case ($\theta_2 = \theta_{2\min}$).

From general considerations it is clear that the process of heating of the filament should be affected by the properties of the studied solid material, the elastically deformable material, the substance filling the space between the specimen, the substrate, and the probe, and the diameter of the probe. On the other hand, as is known from studies of the thermal properties of liquids and gases [11, 12], for short heating times, the deviation of the temperature of the probe from the idealized model can largely be explained by the effect of the intrinsic heat capacity of the filament. Therefore, it is necessary to understand the role of the contact resistance in simultaneous action of these negative factors.

The obtained time dependence of the temperature increment of the probe filament as applied to studies of the thermal conductivity of quartz glass KV at experimental temperatures of 300 and 1300 K [1] is shown in Fig. 3a and b, respectively (curve 2). For these studies the probe was made of Extra platinum (the radius of the filament is 10 μm). Measurerements were taken in an argon atmosphere. A heatproof material based on ultrathin quartz filaments (HPMQ) ($\rho = 120$ kg/m^3, the diameter of the filament is < 1 μm) was used as the elastically deformable material. The character of the deviation of the excess temperature of the filament from the idealized logarithmic curve for $T_{in} = 300$ K and 1300 K is shown in Fig. 4 (curves 1 and 1', respectively) in the form of the variation of the slope to the time axis relative to the steady-state value. It should be noted that a higher corresponding error is found at lower temperatures of the experiment. A similar calculation was conducted for substances with properties different from those of quartz glass: a single crystal of LiF ($\lambda_1/\lambda_2 \sim 10$) for $T_{in} = 300$ and 1000 K (Fig. 4, curves 2 and 2') and organic glass ($\lambda_1/\lambda_2 \sim 0.1$) for $T_{in} = 300$ and 380 K (Fig. 4, curves 3 and 3'). Next, the properties of the gas filling the space between the probe, the substrate, and the specimen (a case with helium was studied, curve 4) and the properties of the elastically deformable material (artificial asbestos amphibole, the curve almost coincides with curve 1, and foam plastic, curve 5) were varied. The data on the thermal conductivity of quartz glass

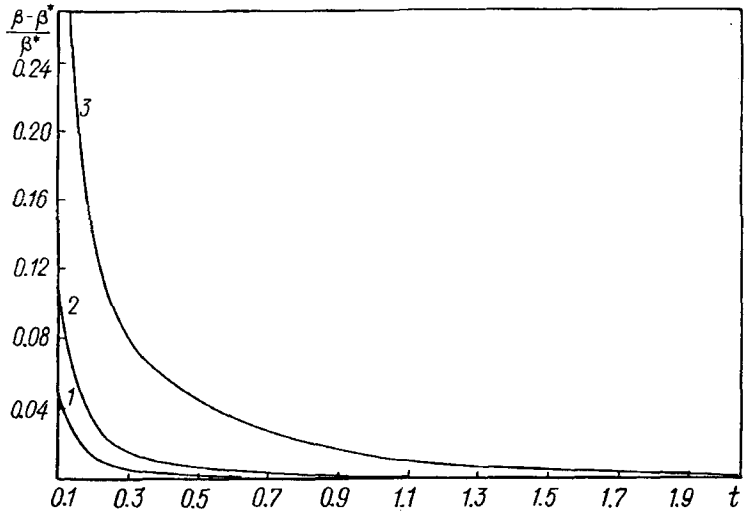

Fig. 5. Results of a study of the curve of contact resistance versus the radius of the probe filament for the system of platinum–quartz glass–HPMQ–argon (T_{in} = 300 K): 1) $r_0 = 0.25 \cdot 10^{-5}$ m, 2) $0.5 \cdot 10^{-5}$, 3) $1.0 \cdot 10^{-5}$.

and LiF correspond to those of [1] and [2], and the data on the heat capacity and density, to those of [7]. The thermal properties of HPMQ were chosen in accordance with [8], and those of the other compounds, with [9].

Use of liquids as an intermediate filling material seems unsuitable for two reasons. First, it can penetrate into pores and microroughnesses of the specimen and the substrate and change their thermal properties. Second, the temperature range of the experiment becomes substantially narrower with them.

The dashed lines in Fig. 4 show the errors caused by the intrinsic heat capacity of the filament (the evaluation was done in accordance with [10] with the assumption that the entire space surrounding the probe is filled with the elastically deformable material).

Figure 5 shows results of a study of the dependence of the distorting effect of the contact resistance on the experimental process on the radius of the probe filament for the system of platinum–quartz glass–HPMQ–argon.

Thus, when the hot-wire method and its variants (in particular, the method of irregular thermal regime) are used to study the thermal conductivity of solids, the duration of a measuring pulse is limited from below by the negative effect of the shift of the probe toward the pressing material on the accurracy of the measurements. As was shown by calculations for a wide class of materials ($\lambda_1 \sim 0.1-15$ W/(m·K)), the time in which the heating thermogram attains the idealized logarithmic dependence (τ^*) with an accuracy of 1% is equal to ~0.2–0.3 sec for a radius of the filament of 2.5 μm, 0.3–0.45 sec for a 5-μm radius, and 0.8–1.3 sec for a 10-μm radius (its should be noted that shorter times correspond to smaller ratios λ_1/λ_2). This, in turn, almost precludes obtaining reliable experimental data for specimens with a thickness less than $(a\tau^*)^{1/2}$. Apart from the aforesaid, in practice attention should be paid to the quality of the surfaces in contact with the probe to eliminate a possible effect of their roughness on the process.

NOTATION

T, temperature, K; ΔT, excess temperature of the probe; T_{in}, initial temperature; t, time, sec; t_1, t_2, moments of measurement, $t_2 > t_1$; τ^*, time in which the calculated temperature of the probe attains the idealized logarithmic curve with a specified error; r, θ, longitudinal and radial coordinates, m, rad; r_0, radius of the probe; R, distance exceeding the length of diffusion of the temperature field in the process of heating of the probe ($\sim 5 \cdot 10^{-3}$ m); φ, θ_i, $i = 1-4$, characteristic angles: $\varphi = \arcsin(r_0/R)$, $\theta_0 = \pi + 2\varphi$, $\theta_1 = 1.5\pi + \varphi$, $\theta_2 = (1.2222-1.3333)\pi + \varphi$, $\theta_{2min} = 1.2222\pi + \varphi$, $\theta_3 = 2\pi - (\theta_2 - \theta_0)$, $\theta_4 = 2\pi - \theta(r) + \theta_0$; r^*, $r(\theta)$, characteristic distances: $r^* = r_0/\sin(\theta_2 - \pi - \varphi)$, $r(\theta) = r_0/\sin(\theta - \pi - \varphi)$; $\varepsilon \ll r_0$, small quantity; n, direction of the normal to the interface between the studied and elastically deformable materials; Q_i, $i = 1-4$, domains of definition of the

heat conduction equations for the studied material, the substrate, the probe, and the filling gas, respectively; λ_i (W/(m·K)), c_i (J/(kg·K)), ρ_i (kg/m^3), $i = 1-4$, thermal conductivity, specific isobaric heat capacity, and density of the studied material, the substrate, the probe, and the filling gas, respectively; a, thermal diffusivity, m^2/sec; q_L, thermal power released in a unit of the length L of the probe, W/m.

REFERENCES

1. G. G. Spirin, Yu. K. Vinogradov, and O. V. Belyaev, Teplofiz. Vysok. Temperatur, 34, No. 1, 29-34 (1996).
2. O. V. Belyaev, G. G. Spirin, and Yu. K. Vinogradov, Teplofiz. Vysok. Temperatur, 34, No. 5, 29-34 (1996).
3. G. G. Spirin, Inzh.-Fiz. Zh., 38, No. 4, 656-661 (1986).
4. G. G. Spirin, E. A. Strelkova, and G. D. Ivanov, Teplofiz. Vysok. Temperatur, 24, No. 6, 111-1165 (1986).
5. N. N. Yanenko, The Method of Fractional Steps for Solution of Multidimensional Problems of Mathematical Physics [in Russian], Novosibirsk (1967).
6. A. A. Samarskii, Introduction to the Theory of Difference Schemes [in Russian], Moscow (1989).
7. O. A. Sergeev and A. A. Men', Thermal Properties of Semiconducting Materials [in Russian], Moscow (1977).
8. E. Ya. Litovskii and I. A. Puchkelevich, Thermal Properties of Refractories [in Russian], Moscow (1982).
9. I. S. Grigor'ev and E. Z. Meilikhov (eds.), Physical Quantities, Handbook [in Russian], Moscow (1991).
10. H. S. Carslaw and J. C. Jaeger, Conduction of Heat in Solids, Clarendon Press, Oxford (1959).
11. J. J. Healy, J. J. de Groot, and J. Kestin, Physica, 820, 392-408 (1976).
12. J. J. de Groot, J. Kestin, and H. Sookiazian, Physica, 75, 454-482 (1974).