CERTAIN METHODS AND INSTRUMENTS TO STUDY THERMOPHYSICAL CHARACTERISTICS

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The study of the processes involved in the transfer of heat assumes particular importance and urgency in connection with the rapid growth of production, science, and engineering. The ever increasing flow of new synthetic materials is confronting researchers with the problems involved in seeking effective means and facilities to study their physicochemical properties. In addition to the purely experimental methods of research, the theoretical calculation of the heat conduction of porous materials, of complex multicomponent systems, and of mixtures of various substances is gaining increasingly greater acceptance and development. In this connection, the requirements for accuracy in the experimental investigations are increasing, because these must serve not only as a source of primary information, but also as an objective criterion to evaluate the accuracy of the theoretical calculations. Unable to cover the entire complex of research carried out in recent years at the thermophysics laboratory of the Institute of Heat- and Mass-Transfer of the Belorussian Academy of Sciences [IHMT, AS BSSR], we will dwell briefly only on certain of the results relating to the experimental methods of investigating the thermal properties of materials.

The experimental investigation of thermophysical characteristics proceeded along three fundamental lines:

1) the development of new, unique, and more complete methods of determining thermal conductivity, thermal diffusivity, and specific heat;

2) the theoretical analysis of optimum experimental conditions;

3) the design of experimental stands and instruments to study the thermophysical characteristics in a temperature range between 80 and 1500° K.

The common feature of the methods for determining thermophysical characteristics lies primarily in the fact that each is based on an analysis of the solutions of the differential equation of heat conduction for specific initial and boundary conditions, whereas their difference lies in the means and facilities to achieve a final result. Since the quantitative relationships governing the development of temperature fields are functions both of the nature of the specified boundary conditions and of their effective duration, it is possible, in principle, to use a single solution for the development of a number of methods. Below we will consider the methods and instruments whose analytical bases are the solutions of problems pertaining to a plate and a cylinder with an internal constant-power source and heat transfer at the boundary. In addition to the familiar methods of determining the thermophysical characteristics, we will also examine certain new developments by the authors of this article. Comparison of the described methods with the methods of other authors is presented primarily only when there is a common theoretical basis or if the methods for the calculation of the thermophysical characteristics are essentially close.

Most of the methods developed at the thermophysics laboratory of the IHMT were devised from analysis of the rigorous analytical solutions of the differential heat-conduction equation cited in the works of A. V. Luikov. The temperature fields at the initial stage in the development of the thermal process are most complex in nature. The yield of final results, just as the monitoring of proper experimental procedure, in these cases involves certain difficulties. In this connection, the method and instrument for the integrated determination of the thermophysical characteristics of solid and granular nonmetallic materials, proposed by Luikov and developed by Verzhinskaya will doubtlessly be of interest. The theoretical basis for this method is the solution of the problem for a semi-infinite body whose bounding surface is heated by a constant flow of heat. Since the same problem will be considered later on with somewhat different boundary conditions, we present its formulation and solution:

$$\frac{\partial t(x,\tau)}{\partial \tau} = a\frac{\partial^2 t(x,\tau)}{\partial x^2}; \tag{1}$$

$$t(x,0) = t_0 = \text{const}, \quad \lambda\frac{\partial t(0,\tau)}{\partial x} + q = 0; \tag{2}$$

$$\frac{\partial t(\infty, t)}{\partial x} = 0; \tag{3}$$

$$t(x,\tau) = \frac{2q}{\lambda}\sqrt{a\tau}\,\operatorname{ierfc}\frac{x}{2\sqrt{a\tau}}. \tag{4}$$

Solution (4) is the basis for the calculation of the thermophysical characteristics. The uniqueness of the instrument technique lies in the fact that the automatic recorder registers the temperature at a speed inversely proportional to the square root of the time, allowing monitoring of the correctness of the actual experiment (it should show a straight line), as well as simplifying the processing of the experimental data to the maximum.

The solution of Eq. (1) for condition (2) and a boundary condition of the form

$$-\lambda\frac{\partial t(R,\tau)}{\partial x} + \alpha\,[t_w - t(R,\tau)] = 0, \tag{5}$$

being the more general, served as a basis for the development of a series of other methods to determine the thermophysical characteristics. Let us examine some of these. In the absence of a source within the plate (q = 0) the problem reduces to the heating of an unbounded plate. This solution, as well as the analogous solution for an infinite cylinder, was used to determine the coefficient of thermal-diffusivity under conditions in which $\alpha \to \infty$ (a boundary condition of the first kind). In the experimental procedures developed by us, the boundary condition of the first kind is maintained only at the bases (in the case of a plate) and at the side surface (case of a cylinder), which is technically more convenient than providing for these conditions at all of the surfaces of the specimen (see, for example, the diagram of a calorimeter). The necessary theoretical relationships can be found easily from the corresponding solutions. In addition to the method of calculating the coefficient of thermal diffusivity—generally employed in the theory of a regular regime—we also employed another method which eliminated the need of plotting the $\ln t = f(\tau)$ curve. The essence of this method lies in the recording of the time interval during which the temperature difference between the cooling unit and the specimen during the cooling process varies by a specific fraction of its initial value.

Let us examine certain variants of the integrated determination of the thermophysical characteristics in the presence of a constant-power source within the plate (cylinder) and with heat transfer at the bounding surfaces. The solution of the differential equation (1) with the initial and boundary condition (2) and the simplifying condition (5) ($t_W = t_W \alpha \to \infty$) can be presented in the form

$$t(x, \tau) - t_0 = \frac{q(R-x)}{\lambda} - \frac{2qR}{\lambda} \sum_{n=1}^{\infty} \frac{1}{\mu_n^2} (-1)^{n+1} \sin \mu_n \frac{R-x}{R} \exp(-\mu_n^2 \mathrm{Fo}). \tag{6}$$

The final effect of the development of the temperature field in this case is the steady thermal state for which the coefficient of heat conduction is usually found. It is obvious that the analysis of the temperature field during the course of its development and in the steady state makes it possible to achieve a method for the integrated determination of the thermophysical characteristics. We noted such an approach in the method developed by Fogel and Alekseev. The essence of this method reduces to the calculation of the function

$$\frac{\theta}{\theta_{st}} = \frac{t(x, \tau) - t_0}{t_{st}(x, \tau) - t_0} = f\left(\frac{x}{R}, \mathrm{Fo}\right) \tag{7}$$

and the experimental determination of $t(x, \tau)$ and $t_{st}(x, t)$. The drawbacks of this version are that the ratio (7) is a function of the coordinates, and that it is necessary to use tables prepared in advance. We can avoid this shortcoming by introducing the rate of heating for a given system. Indeed, it follows from relationship (6) that during the course of regularization, a characteristic feature of the development of the temperature field is the constancy of the quantities

$$m = \frac{\ln b_1 - \ln b_2}{\tau_2 - \tau_1} = \frac{\ln(t_{st} - t_1) - \ln(t_{st} - t_2)}{\tau_2 - \tau_1} = \frac{\pi^2}{4R^2} a, \tag{8}$$

where b is the rate of heating and t_{st} is the temperature in the steady state. Analogous relationships are also found from corresponding solutions for bodies of other geometrical shapes. Thus, the nonsteady thermal process preceding the steady state may be uniquely employed to determine the coefficient of thermal diffusivity, based on a single method which follows from (8). With small values for the Fourier number the thermophysical characteristics may be calculated by using solution (4). These considerations served as the basis for the simple instrument developed by Volokhov to study the thermophysical characteristics of solid and granular materials at room temperature. The basic elements of the instrument are: a galvanometer, a power source for the heater, a timer, a vise with two hollow plane-parallel copper blocks and a cylindrical form to study granular materials. In addition, it contains a simple relay system permitting the automatic switching on and off of the timer. The power source is a transformer whose winding is fashioned so that the voltage that is taken can be varied according to the law: U_0, $(2)^{1/2}U_0$, $(3)^{1/2}U_0$, ... (a total of ten ranges). The heat-flow and temperature sensors are of the usual type: a low-lag flat or cylindrical heater and a differential thermocouple, one of whose junctions is caulked into the plane of the refrigerating unit, while the other is housed in the specimen. In addition to the theoretical methods, provision is also made in the instrument for semiautomatic determination of the thermophysical characteristics. We will demonstrate this with an example of the determination of the coefficient of thermal diffusivity. First we record the temperature difference between the specimen at room temperature and the "refrigeration unit" through which water is passed. Before the introduction of the specimen into the space between the "refrigeration units", photoresistances capable of moving freely along the scale of the galvanometer "mark off" some portion of the steady temperature difference. The specimen is then brought into the space between the "refrigeration units" and compressed by their sides. A process of natural temperature leveling begins through the volume of the specimen, during which the time in which the temperature difference changes by the specified magnitude is automatically recorded. It is obvious that the readings of the timer will be proportional to R^2/a, regardless of the initial temperature difference.

The investigation of thermophysical characteristics over a wide range of temperatures is an incomparably more difficult problem than the determination of these at temperatures near room temperature. The methods considered above may be employed fundamentally for broad-range temperature studies in which the subject specimens are heated to the required temperature level. Thus, for example, the Verzhinskaya method was used by Vishnevskii and Dzyubenko to measure the heat-conduction coefficients and the coefficients of thermal diffusivity for refractory materials, where no less than 6–7 hr were required to carry out measurements every 50° in an interval of 300–1000° K. In this connection, the methods of investigation carried out during a process of continuous heating are more effective. Among these methods we find, first of all, the quasi-steady methods whose theory was developed by Luikov, and the methods of monotonic heating, developed by Dul'nev and his co-workers. One way of achieving a quasi-steady thermal state is the symmetric heating of a specimen by a time-constant heat flow. As an example of a way in which time-constant boundary conditions of the second kind can be achieved, the literature usually describes the Semenov method. In evaluating the symmetry of the heat flow, i.e., the conditions of its constancy, the author of the method considered a system of bodies, including a plate with ideal thermal insulation, a source of constant power, and a semi-infinite body. We also examined a similar system, and found that the ratio of the heat flowing into the region of the semi-infinite body and into the plate, is determined by the relationship

$$\frac{q_2}{q_1} = \left(\operatorname{erfc}\frac{R}{2\sqrt{a\tau}} + 1\right)\left(\operatorname{erfc}\frac{R}{\sqrt{a\tau}} - 1\right)^{-1}. \quad (9)$$

The calculations carried out on the basis of (9) have demonstrated that in such a system there is continuous redistribution of the heat flows, and in the general energy balance the specific weight of the flow entering the region of the semi-infinite body increases gradually. Although these conclusions do not agree with those of Semenov who found that the symmetry of the heat flows is accurate to within 2%, it is nevertheless possible to demonstrate that the approximation of the temperature field for the plate by the laws of a quasi-steady regime is possible within a narrow time interval. The constancy of the heat flow at the surface of the central plate is achieved in the Krisher method by using four heaters. However, this is a particularly inconvenient system, since it requires a large number of plate-like specimens and identical heaters. The various procedures which we examined above for the testing of materials in the quasi-steady regime are essentially "room" variants and cannot be employed effectively in broad-range temperature studies, since the application of substantial electrical power on the probes inserted into the specimens will result in corresponding increased temperature differences, which necessarily will lead to a disruption in the unidimensionality of the temperature field in the measurement area. Thus, attainment of constancy conditions for the heat flow at the surface of the specimen over a wide range of temperatures cannot be achieved in principle without a special system to regulate and maintain this flow or corresponding temperatures. One of the possible and most frequently employed means of obtaining a constant heat flow is heating the specimen under adiabatic conditions. Vasil'ev developed a method and equipment to determine the thermophysical characteristics of poor heat conductors in the 80–400° K range. The precision amplification equipment developed by him makes it possible to maintain the adiabatic conditions of heating in the indicated temperature range, with a high degree of accuracy.

In addition to the methods which provide for specification of a constant heat flow, great acceptance has been gained by quasi-steady methods involving linear heating for boundary conditions of the first and third kinds. The idea of determining the thermal diffusivity in a quasi-steady regime was first proposed by Luikov. The quasi-steady methods were subsequently developed and refined by many authors, with the methods of determining thermal diffusivity being developed most extensively. The theoretical basis for the absolute method of integrated determination of thermophysical characteristics, proposed by Fraiman, was the quasi-steady solution of the problem involving the heating of an infinite hollow cylinder with a constant-power source, in a medium whose temperature varies linearly. This method and the installation makes it possible to undertake an integrated study of the thermophysical properties of nonmetallic materials in a temperature range of 300–1500° without using any standard reference materials. The earlier proposed method provided for heating the specimen twice, at the same rate, with the internal heater connected and disconnected. In the Lazarev method, employing analogous theoretical solutions, the simultaneous heating of two identical specimens is proposed (with and without a heater) under identical conditions. Both versions exhibit certain shortcomings which stem, respectively, from the time and space dissociation in the experiment. Before we indicate how to remove these drawbacks, let us dwell briefly on a method for the integrated determination of the thermophysical characteristics of specimens in the form of plates and disks, when these are heated linearly.

The nonsteady solution of Eq. (1), which we found for conditions (2) and (5) under the assumption that $t_w = t_0 + b\tau$, has the following form:

$$t(x,\tau) - t_0 = b\tau - \frac{b}{2a}\left[R^2\left(1 + \frac{2}{\mathrm{Bi}}\right) - x^2\right] + \frac{q}{\lambda}(R - x) + \frac{q}{\alpha} + \frac{bR^2}{a}\sum_{n=1}^{\infty}\frac{A_n}{\mu_n^2}\cos\mu_n\frac{x}{R} \times \exp(-\mu_n^2\,\mathrm{Fo}) - \frac{q}{\alpha}\sum_{n=1}^{\infty}A_n\cos\mu_n\cos\mu_n\frac{x}{R} \times \exp(-\mu_n^2\,\mathrm{Fo}) - \frac{qR}{\lambda}\sum_{n=1}^{\infty}\frac{A_n\sin\mu_n\cos\mu_n\frac{x}{R}}{\mu_n} \times \exp(-\mu_n^2\,\mathrm{Fo}), \quad (10)$$

where μ_n are the roots of the characteristic equation

$$\operatorname{ctg}\mu_n = \frac{1}{\mathrm{Bi}}\mu; \qquad A_n = (-1)^{n+1}\frac{2\,\mathrm{Bi}\sqrt{\mathrm{Bi}^2+\mu_n^2}}{\mu_n(\mathrm{Bi}^2+\mathrm{Bi}+\mu_n^2)}.$$

Detailed tables of the values of μ_n and A_n are given in the literature.

In the quasi-steady state when Bi = ∞ expression (10) assumes the simpler form

$$t(x,\ \tau)-t_0 = b\tau + \frac{bx^2}{2a} - \frac{qx}{\lambda} - \frac{bR^2}{2a} + \frac{qR}{\lambda}. \qquad (11)$$

The temperature field in this case is a parabola shifted relative to the coordinate origin (nonsymmetric heating) and in general (the exception is adiabatic heating) to eliminate the time or space dissocation of the experiment, the temperature must be established at three points. An analogous method was used by Shakhtin and Vishnevskii in determining the coefficient of thermal diffusivity under conditions of nonsymmetric heating. The necessary theoretical relationships can be found easily from relationship (11) and we therefore do not present them here. The experimental installation includes a low-lag furnace with a power of no more than 2.5 kW and the equipment for the specified linear heating and temperature measurement. The furnace is designed to test specimens in the form of plates and disks in a temperature range of 300–1300° K. The methods of achieving linear heating may vary greatly: manual, semiautomatic, and automatic temperature regulation.

The methods of monotonic heating, whose common nature with purely quasi-steady methods lies in the fact that the necessary information for both is derived from an experiment in continuous progress, are being developed and are gaining recognition on an ever greater scale. The technical achievement of monotonic heating methods is simpler than that of quasi-steady methods. However, the processing of the experimental data in the former case seems more complex to us than for the latter. The comparative evaluation of accuracy in these methods becomes difficult because of a lack of comparable experimental data. The methods considered above for the determination of thermophysical characteristics are based on an analysis of solutions for the one-dimensional equation of heat conduction with constant coefficients. In this connection, research into the selection of optimum experimentation regimes assumes particular importance. With broad-range temperature tests on materials experiencing no phase conversions, the method of zonal calculation of the thermophysical characteristics is completely justified. The validity of this approach has been demonstrated by many authors and it has been given a theoretical foundation by E. S. Platunov. It should be stressed here that rigorous adherence to the boundary conditions will considerably facilitate the problem of finding the required relationships, since the only factor responsible in this case for a change in the temperature field and in the corresponding gradient is the change in the thermophysical properties of the material under investigation. No less important a factor—determining the quality of the investigation to a great extent—is adherence to the conditions of one-dimensionality for the heat flows during the course of the experiment. One-dimensionality for the heat flow is generally achieved either by the installation of special protective devices or by selection of appropriate relationships between the linear dimensions of the specimen. Another testing procedure is preferable; however, its rigorous theoretical foundation is possible only through an analysis of corresponding two-dimensional temperature fields. This work provided foundations for most of the methods of determining thermophysical characteristics, developed at the thermophysics laboratory of the Institute of Heat- and Mass-Transfer of the Belorussian Academy of Sciences.

Raising the level and efficiency of thermophysics research requires continued theoretical generalization, unification, and standardization of existing methods, improvement in experimentation techniques as a whole, and the development and series production of reliable instruments that are convenient to use for the determination of thermophysical charcteristics. There is no doubt that these and similar problems facing Soviet thermophysicists will be resolved successfully, and this will be due to the continued expansion and blossoming of all branches of our production, science, and engineering.

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