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# DETERMINATION OF THE ERROR IN MEASUREMENTS OF THERMOPHYSICAL CHARACTERISTICS OF HEAT-INSULATING MATERIALS

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*A method of combined determination of thermophysical characteristics (TPC) of heat-insulating materials that is based on solving the inverse heat-conduction problem is discussed. The influence of the parameters of the calculation algorithm and the uncertainty in specifying the parameters of the measuring device on the TPC measurement error is analyzed. It is shown that the measurement error considerably exceeds the scatter of the results of repeated measurements of the same sample without disassembling the measuring device.*

In mathematical modeling of thermophysical processes a lack of reliable information on the thermophysical characteristics (TPC) of the materials is experienced in the majority of cases. Among those materials heat insulators form a special group. For them, on the one hand, it is difficult to determine the TPC measurement error due to the absence of reference samples while, on the other hand, it is often necessary to know this error in order that the calculated temperature not exceed the established limit. The majority of the handbooks on the TPC of materials [1-3] do not provide the errors in TPC determinations. In [4], various different reference data have been analyzed and the conclusion was made that "even for relatively well-studied hard refractory metals the results of investigations of different authors diverge considerably: for heat capacity these deviations amount to 15%, and for thermal conductivity 20–30%." Up to now the situation has remained almost unchanged.

Employing mathematical modeling, we have analyzed the influence of the parameters of the calculation algorithm and the uncertainty in specifying the parameters of the measuring device on the TPC measurement error for heat-insulating materials. It is shown that the error exceeds considerably the scatter in the measurement results obtained in repeated measurements of the same sample without disassembling the measuring device. In so doing, the inverse heat-conduction problem (IHCP) has been solved in a formulation that makes it possible to forego the use of a precision source of constant specific heat flux.

The measuring cell was a plane electric heater (with a thickness of $0.67 \cdot 10^{-3}$ m and a diameter of $5 \cdot 10^{-2}$ m) that provides a one-dimensional heat flux to which cylindrical $5 \cdot 10^{-2}$-m-diameter samples made of the investigated material were linked on both sides. Thermocouples were placed between the samples. The design of the cell was symmetric relative to the middle of the heating element, and therefore in Fig. 1 only half of it is shown. Thermocouples 4, 5, 6 were of informational value, and 7 served as a reference thermocouple by which it was determined that the thermal wave did not reach the opposite end of the sample. Fulfillment of this condition allowed the sample to be treated as a semiinfinite body. In the mathematical model of the measurement process it was assumed that a Nichrome plate is used instead of Nichrome wire. The thickness of this plate was set in such a way that mass and, consequently, the heat capacity of the wire and the plate were the same.

In heating a sample by a thin plane heating element, the heat capacity of the latter is commonly neglected and, as a consequence, it is assumed that the entire amount of heat released by the heating element enters the investigated sample. In measuring the TPC of highly efficient heat insulators with $\lambda \sim 10^{-2}$ W/(m·K) and $C \sim 10^5$ J/(m$^3$·K), the heat capacity of the heating element must not be neglected since it is commensurable with that of the heated layer of the investigated sample. Neglect of this fact leads to a methodological error.

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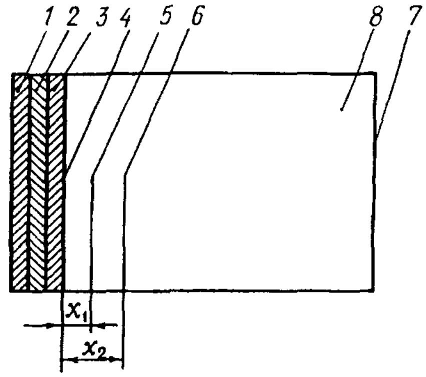


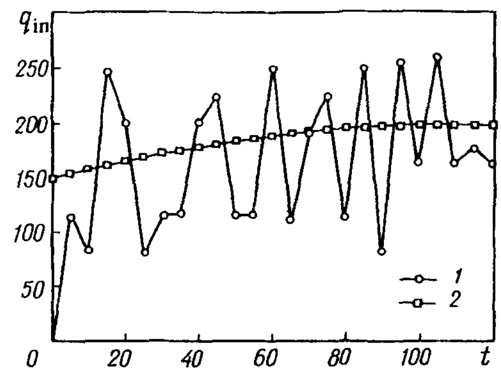


Fig. 1. Schematic of the measuring cell: 1) Nichrome, 2) mica (electrical insulator), 3) steel, 4, 5, 6, 7) thermocouples, 8) sample of the investigated material; $x_1$, $x_2$, coordinates of placement of the thermocouples.

Fig. 2. Time (sec) dependence of the input heat flux (W/m$^2$): 1) numerical differentiation, 2) calculation by (8)-(10).

The mathematical model of sample heating concerned a semiinfinite body at the boundary of which a variable heat flux was specified. In [5], a solution of this problem is given. After correction of a misprint made in [5] the final formula acquires the form

$$T(x, t) = \frac{\chi^{1/2}}{\lambda \pi} \int_0^t q_{in}(t - \tau) \exp\left(-\frac{x^2}{4\chi\tau}\right) \tau^{-1/2} d\tau . \tag{1}$$

The heat flux $q_{in}(t)$ entering the sample is determined from the equation of heat balance of the heating element

$$W\Delta t = C_h \Delta T + q_{in} \Delta t , \tag{2}$$

where $\Delta t$ is a small interval of time; $W$ is the heat flux released in the heating element, determined as the electric power supplied to the Nichrome wire divided by the heater area. In writing Eq. (2), it is assumed that all parts of the heating element have the same temperature. This assumption is based on the fact that the effective thermal conductivity of the heating element is two orders of magnitude larger than that of the investigated heat insulator. From (2) it is easy to derive the time dependence of the heat flux entering the sample

$$q_{in}(t) = W - C_h \frac{dT}{dt} . \tag{3}$$

The temperature measured by thermocouple 4 (Fig. 1) can be used, in conformity with (3), to determine the heat flux entering the sample.

To determine TPC of the sample, we formulate the extremum IHCP [6] as

$$J(\lambda, C) = \sum_{i=1}^{2} \int_0^{t_m} [T(\lambda, C, x_i, t) - f_i(t)]^2 dt \to \min_{\lambda, C} . \tag{4}$$

The uniqueness of its solution is shown in [7]. We will construct the solution using an iteration technique without calculating the derivatives of the goal functional [8]. The iteration formulas for determination of $\lambda$ and $C$ at the $(n+1)$-th iteration step have the form

$$\lambda^{n+1} = \lambda^n + \alpha_\lambda^n , \quad C^{n+1} = C^n + \alpha_C^n . \tag{5}$$

A value of the goal functional (4) at the $(n + 1)$-th iteration step can be represented as

$$J(\lambda^{n+1}, C^{n+1}) = \sum_{i=1}^{2} \int_0^{t_m} \left[ T(\lambda^n, C^n, x_i, t) + \alpha_\lambda^n \frac{\partial T}{\partial \lambda} + \alpha_C^n \frac{\partial T}{\partial C} - f_i(t) \right]^2 dt . \tag{6}$$

To find the minimum of this functional in $\alpha_\lambda^n$ and $\alpha_C^n$, we differentiate (6) with respect to these variables and equate the expressions obtained to zero. After some rearrangement we arrive at the following system

$$\alpha_\lambda^n A_{11} + \alpha_C^n A_{12} = B_1 , \quad \alpha_\lambda^n A_{21} + \alpha_C^n A_{22} = B_2 , \tag{7}$$

where

$$A_{11} = \sum_{i=1}^{2} \int_0^{t_m} \left( \frac{\partial T(x_i, t)}{\partial \lambda} \right)^2 dt ; \quad A_{12} = \sum_{i=1}^{2} \int_0^{t_m} \left( \frac{\partial T(x_i, t)}{\partial \lambda} \frac{\partial T(x_i, t)}{\partial C} \right) dt ;$$

$$A_{21} = A_{12} ; \quad A_{22} = \sum_{i=1}^{2} \int_0^{t_m} \left( \frac{\partial T(x_i, t)}{\partial C} \right)^2 dt ;$$

$$B_1 = \sum_{i=1}^{2} \int_0^{t_m} \left[ f_i(t) - T(\lambda^n, C^n, x_i, t) \right] \frac{\partial T(x_i, t)}{\partial \lambda} dt ;$$

$$B_2 = \sum_{i=1}^{2} \int_0^{t_m} \left[ f_i(t) - T(\lambda^n, C^n, x_i, t) \right] \frac{\partial T(x_i, t)}{\partial C} dt .$$

The derivatives of the temperature with respect to the sought parameters $\partial T(x_i, t)/\partial \lambda$ and $\partial T(x_i, t)/\partial C$ are calculated analytically using formulas obtained by differentiating expression (1) with respect to $\lambda$ and $C$, respectively. The expressions for these derivatives are cumbersome and, therefore, are not given. Having solved system (7), we obtain $\alpha_\lambda^n$ and $\alpha_C^n$, needed to calculate $\lambda^{n+1}$ and $C^{n+1}$ using formulas (5).

To determine the heat flux entering the investigated sample, it is necessary to differentiate the recorded temperature of the heating element using formula (3). As is known, differentiation is an ill-defined operation, which leads to large errors in calculations of derivatives. To eliminate them, we adopted the following approach. Since the heating element possesses a considerably higher thermal conductivity than the investigated sample, we can use the analytical solution presented in [5] for the problem of the temperature distribution in a plate one of whose surfaces abuts a layer of an ideal inductor to which heat is supplied. This solution is an infinite descending series. If we restrict ourselves to its first term, the surface temperature can be represented in the form

$$T(t) = a(1 - \exp(-bt)) . \tag{8}$$

The parameters $a$ and $b$ in (8) are determined by the magnitude of the input heat flux and the TPC and can be found from the following condition:

$$J(a, b) = \int_0^{t_m} [a(1 - \exp(-bt)) - T_{\text{exp}}(t)]^2 dt \to \min_{a, b} . \tag{9}$$

We solved problem (9) using an iteration technique similar to the method used for solution of problem (4). To calculate $q_{\text{in}}(t)$ by formula (3), function (8) was differentiated with respect to time:

$$\frac{dT}{dt} = ab \exp(-bt) . \tag{10}$$

Figure 2 shows dependences $q_{\text{in}}(t)$ obtained by numerical differentiation using the central difference approximation and calculated by (8)-(10). As is seen, approximation of the experimental temperature by curve (8) completely eliminates the incorrectness inherent in numerical differentiation.

The calculation formulas for solving the IHCP contain various parameters that affect the error in TPC determination. Those are the parameters of the calculation algorithm and the parameters of the measuring device. The former concern errors of numerical integration, linear approximation of the goal functional by expression (6), and calculation of the heat flux entering the specimen using algorithm (8)-(10). The latter concern errors in the coordinates of placement of the thermocouples, in determination of the heat released by the heating element and the heat capacity of the heating element, and in temperature measurements. The accuracy of TPC determination also depends on the measurement time $t_m$.

The influence of all these parameters on the error in TPC determination was evaluated by the method of mathematical modeling. For this, using a finite-difference method and an implicit conservative scheme [9], we solved the direct problem for a specimen with $\lambda = 0.03$ W/(m·K) and $C = 3\cdot10^5$ J/(m$^3$·K), which corresponds to the TPC of a highly efficient heat insulator of the ATM type [2], for a heat flux entering the specimen of 500 W/m$^2$. This magnitude of the heat flux approaches that specified in the experiment. The temperature was calculated at the points $x_1 = 0.97\cdot10^{-3}$ m and $x_2 = 1.67\cdot10^{-3}$ m, which corresponded to the coordinates of placement of the thermocouples in the experiment. The temperatures obtained were used as $f_i(t)$, $i = 1, 2$ to solve IHCP (4); moreover $q_{in}(t)$ was not calculated by formula (3), we prescribed a constant value of it equal to 500 W/m$^2$.

In the first stage, we determined the influence of the integration step $\Delta t$ on the error in solving the IHCP. Use of the integration steps of 1, 2.5, 5, and 10 sec showed that the TPC obtained by solving these IHCPs differed from the exact values $\lambda = 0.03$ W/(m·K) and $C = 3\cdot10^5$ J/(m$^3$·K) by less than 0.1%. Therefore, we can conclude that numerical integration and linear approximation of the goal functional do not bring about an error. To determine the error in calculating $q_{in}(t)$ from the heating-element temperature, we adopted the following procedure. We calculated the temperature in the construction shown in Fig. 1. In this calculation the heat flux entering layer 1 was equal to 500 W/m$^2$. The thicknesses of layers 1, 2, and 3 corresponded to those of the Nichrome, mica, and steel of the heating element. The temperature was determined at point 4 as well as at points 5 and 6, which had the coordinates $x_1 = 0.97\cdot10^{-3}$ m and $x_2 = 1.67\cdot10^{-3}$ m, respectively. The values obtained were used to model the true (measured without error) temperatures at points 4, 5, and 6, which were specified as $T_{exp}(t)$ and $f_i(t)$, $i = 1$, 2, respectively. Then IHCP (4) in which $q_{in}(t)$ was determined by algorithm (8)-(10) was solved. The differences of the thermophysical characteristics obtained from the IHCP from the exact values were $\delta\lambda = 1.0\%$ and $\delta C = 3.5\%$. They corresponded to the errors in calculations of $q_{in}(t)$ from the heating-element temperature using algorithm (8)-(10).

Next, we determined the influence of the measurement time $t_m$ on an error in TPC determinations. As in the case of $q_{in}(t)$ error determination, we solved the direct problem and calculated the temperatures at points 4, 5, and 6. Then these temperatures were adopted as the initial data in solving the IHCP for various $t_m$ values. The calculations revealed the existence of some critical $t_m$. At $t_m < 40$ sec iteration process (4)-(7) did not converge. At $t_m > 40$ sec the results were practically the same and the error was determined by the error of heat-flux calculation by algorithm (8)-(10). The nonconvergence of iteration process (4)-(7) at $t_m < 40$ sec is apparently attributable to the fact that at a small $t_m$ the influence of the thermophysical characteristics of the specimen on the temperatures at points 5 and 6 is insignificant.

In practice, it is impossible to measure the parameters of the measuring device exactly. To elucidate the influence of the error in their measurement on the error in TPC determination, we solved the same direct problem as in the investigation of the computational-algorithm parameters. In so doing, the temperatures obtained served as the initial data for solving eight IHCPs, which corresponded to the number of parameters of the measuring device. In each IHCP solution one of the parameters was specified with an error, which modeled the error in measurement of this parameter in the experiment. This led to an error in TPC determination (see Table 1). The error values for the parameters corresponded to the estimates of the maximum errors with which the parameters of the measuring device were specified. The comparatively large error in the coordinates of placement of the thermocouples was attributable to the fact that the thermojunctions were nonuniformly imbedded in the specimens of the investigated material between which the thermocouple was placed. The multiplicative error in temperature measurements (lines 6, 7, and 8 in the table) was caused by drift of the measuring channels.

TABLE 1. Influence of the Parameters of the Measuring Device on the Accuracy of TPC Measurement

| Line No. | Parameters | | Relative error in $\lambda$ and $C$ determination, respectively, % |
|---|---|---|---|
| | name | error | |
| 1 | Coordinate of thermocouple 5 | $\Delta x_1 = 0.1 \cdot 10^{-3}$ m | 11.8; 3.0 |
| 2 | Coordinate of thermocouple 6 | $\Delta x_2 = 0.1 \cdot 10^{-3}$ m | 11.9; 7.9 |
| 3 | Heat output in the heating element | $\delta W = 5\%$ | 6.6; 5.7 |
| 4 | Heat capacity of the heating element | $\delta C = 5\%$ | 10.5; 10.5 |
| 5 | Additive random noise of the temperatures at points 4, 5, and 6 | $\Delta T = 0.5$ K | 0.01; 0.01 |
| 6 | Multiplicative error of the temperature at point 4 | $K_T = 2.5\%$ | 5.4; 5.6 |
| 7 | The same | $K_T = 2.5\%$ | 12.1; 2.9 |
| 8 | The same | $K_T = 2.5\%$ | 8.8; 5.4 |

To check the feasibility of using the suggested approach to finding the error in TPC determination, we compared the scatter of the results of repeated measurements of the same sample without disassembling the measuring device and the scatter obtained in mathematical modeling. We made six runs of measurements of the thermophysical characteristics of foam polyurethane heat-insulating material. The results were averaged and root-mean-square deviations were calculated:

$$\lambda = 0.066 \pm 4.5\% \ (S)\,, \quad \text{W/(m}\cdot\text{K)}\,; \quad C = 5.5 \cdot 10^5 \pm 5.5\% \ (S)\,, \quad \text{J/(m}^3\cdot\text{K)}\,. \tag{11}$$

Since the cell with the sample was not disassembled, this scatter was due to the scatter in the values of the heat released in the heating element, drift of the measuring channels determining the temperatures at points 4, 5, and 6, and the accuracy of $q_m(t)$ calculation. The root-mean-square deviation caused by these factors can be calculated as

$$S_\lambda = \frac{1}{\bar{\lambda}} \sqrt{\left( \sum_{i=1}^{5} \left( \frac{\Delta \lambda_i}{3} \right)^2 \right)} \cdot 100\%\,. \tag{12}$$

In (12), $\bar{\lambda}$ corresponds to the mean value obtained from six measurements and given in (11). The absolute maximum error in thermal-conductivity determination $\Delta\lambda_i$ was determined from its relative value given in Table 1 and the mean value $\lambda = 0.066$ W/(m·K). The threefold decrease in $\Delta\lambda_i$ in (12) is needed to pass from the absolute error, the value of which can be equated to $3S$, to $1S$. The root-mean-square deviation obtained using (12) was ±5.7% for $\lambda$ and ±3.7% for $C$. A comparison of these values with the data obtained on the measuring device shows their satisfactory agreement.

The total error in the TPC measurements for foam polyurethane was determined in a similar manner, only with regard for all the parameters given in Table 1. The measurement errors obtained were as follows:

$$\lambda = 0.066 \pm 26\% \ (S)\,, \quad \text{W/(m}\cdot\text{K)}\,; \quad C = 5.5 \cdot 10^5 \pm 17\% \ (S)\,, \quad \text{J/(m}^3\cdot\text{K)}\,. \tag{13}$$

A comparison of (13) and (11) reveals that the measurement error is severalfold greater than the scatter in the results of repeated measurements obtained for the same sample without disassembling the measuring cell.

An analysis of the accuracy of the IHCP solution is usually performed by imposing additive random noise on the initial temperatures [7, 8]. The error of this noise, as seen from Table 1 (lines 5-8), is more than two orders of magnitude smaller than the multiplicative error in the temperature measurements. Therefore we can conclude that it is insufficient to use only additive noise to analyze the accuracy of the IHCP solution.

Thus, the investigation of the influence of the parameters of both the computational algorithm and the measuring cell on the accuracy of TPC determination for highly efficient heat insulators has demonstrated that the use of mathematical modeling to solve this problem makes it possible to avoid repeated measurements on the same material at different parameters of the measuring cell. The scatter in the measurement results obtained without disassembling the measurement cell is severalfold smaller than the measurement error. Additive random noise imposed on the measured temperatures affects the measurement error only slightly as compared to the other parameters of the problem. The suggested approach can be used to evaluate the accuracy of other measurements.

## NOTATION

$T$, temperature; $x$, $t$, space and time coordinates; $\chi$, thermal diffusivity; $\lambda$, thermal conductivity; $x_i$, $i = 1$, 2, coordinates of placement of thermocouples 5 and 6 inside the sample; $C_h$, heat capacity of the heating element; $\Delta t$, time step; $t_m$, measurement time; $C$, heat capacity; $f_i(t)$, temperature measured at points 5 and 6; $T_{exp}$, measured temperature of the heating element; $\Delta$, absolute error; $\delta$, relative error; $S$, estimate of the relative root-mean-square deviation expressed in percent of the mean value; $K_T$, relative multiplicative temperature error; $n$, iteration number.

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