DETERMINING THE COMPLEX OF THERMOPHYSICAL PROPERTIES IN CERAMICS AT HIGH TEMPERATURES. CONSIDERATION OF HEAT LOSS BY THE SPECIMEN THROUGH THE SIDES

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We present the results obtained in the measurement of the volumetric heat capacity of a corundum ceramic at temperatures of 900-1800 K, obtained by the linear temperature gradient method. We have demonstrated the need for the introduction of correction factors for the loss of heat through the sides and we provide a method for its calculation.

The heat losses through the side surface of a specimen represent one of the main sources of error in the measurement of thermophysical coefficients. Such error is usually minimized through the selection of a specimen geometry in the form, for example, of a disk in which the thickness-to-diameter ratio H/D < 0.25-0.33 [1], and through the introduction of appropriate correction factors in the calculation formulas, these factors determined from precise or approximate solution of the heat-conduction equation (HE).

In the present study we examine the correction factors introduced for the loss of heat through the sides insofar as these pertain to the computational formulas for heat conduction, thermal diffusivity, and the volumetric heat capacity for one of the methods of monotonic heating, i.e., the time-linear temperature gradient (LTG) method [2], realized in the plane variant for heat insulators with an effective thermal conductivity of $\lambda_{eff} = 0.1-1.0$ W/(m·K). This method is based on the quantitative relationships governing the heating of a specimen comprised of two disks in a medium with a constant temperature T_0 by a time-linear flow of heat evolved from an internal source of heat in the symmetry plane of the specimen. The exchange of heat between the specimen and the ambient medium is accomplished by radiation.

The expressions for the correction factors have been derived from the solution of the one-dimensional HE for a specimen with a temperature and flow of heat averaged through its cross section. In the measurement of the temperature and its difference across the specimen by means of thermocouples whose electrodes are positioned in channels through the width of the specimen and parallel to the end surfaces of the cylindrical specimen, we can accomplish the transition to temperatures averaged over the cross section without additional simplification [3].

Let us examine a cylindrical specimen of radius R and height 2H, within whose plane of symmetry a local heat source $q_0(t)$ is active, so that the temperature in the x = 0 plane varies linearly over time. The side and end surfaces exchange heat with the ambient medium having a temperature T_0 by means of radiation. The field of averaged excess temperatures $[\theta(x, t) \ll T_0]$ is described by a linearized HE:

$$\frac{\partial\theta(x, t)}{\partial t} = a\frac{\partial^2\theta(x, t)}{\partial x^2} - a\frac{y_H^2}{H^2}\theta(x, t), \qquad t = 0, \quad \theta(x, 0) = 0, \tag{1}$$

$$x = 0, \quad \theta(0, t) = b_1 t \quad \text{or} \quad \frac{\partial\theta(x, t)}{\partial x} = -\frac{q_0(t)}{\lambda}, \quad x = H, \quad \frac{\partial\theta(x, t)}{\partial x} = -\frac{\mathrm{Bi}_H}{H}\theta(H, t), \tag{2}$$

where $\theta(x, t) \equiv \bar{\theta}(x, r, t) = \frac{2}{R^2}\int_0^R \theta(x, r, t)r\,dr$ is the excess temperature that is the average over the cross section.

Experimental studies [2] show that the solution of the HE for the regular stage of heating is described by the function

$$\theta(x, t) = f(x)t + \varphi(x). \tag{3}$$

After substitution of (3) into (1) and separation of the variables, Eq. (1) is integrated by elements, but the resulting solutions θ(x, t) and q(x, t) are of complex form, thus making their analysis more difficult.

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The structure of the correction factors for the computational formulas can be determined more simply from the expressions for the temperature $\theta(x, t)$ and the heat flow $q(x, t)$, found from the solution of the HE by the method of successive approximations.

Separating the variables and integrating the HE with boundary conditions (2), neglecting the heat losses, we obtain the zeroth and first approximations:

$$\theta^{(0)}(x, t) = \left(b_1 - b_2 \frac{x}{H}\right) t + b_1 \frac{x^2}{2a} - b_2 \frac{x^3}{6a} - \frac{Hx}{3ab_1} (b_1^2 + b_1 b_H + b_H^2), \tag{4}$$

$$\theta^{(1)}(x, t) = \theta^{(0)}(x, t) - \frac{y_H^2}{2}\left[\left(b_1 - \frac{b_2}{3}\right)\frac{x}{H} - \left(b_1 - \frac{b_2}{3}\frac{x}{H}\right)\frac{x^2}{H^2}\right] t -$$

$$- \frac{y_H^2 x^3}{12aH}\left[b_1\left(1 - \frac{x}{H}\right) - \frac{b_2}{3}\left(1 - \frac{3x^2}{5H^2}\right) + \frac{2}{3b_1} B_1\right] + \frac{y_H^2 Hx}{15a} \frac{B_2}{B_y}, \tag{5}$$

where for determination of the constant we have also used the heating rate b_H for the outside surface of the specimen and the change in the temperature difference b_2. The heat flow q in the x = 0 plane, its rate of change b_{q0}, and the temperature difference $\Delta\theta$ across the specimen are correspondingly determined from the expressions

$$q(t)_{x=0} = b_{q0} t + c_p \rho H \frac{B_1}{3b_1} - c_p \rho H \frac{y_H^2}{15} \frac{B_2}{B_y}, \tag{6}$$

$$b_{q0} = \lambda \frac{b_2}{H} + \lambda \frac{y_H^2}{2H}\left(b_1 - \frac{b_2}{3}\right), \tag{7}$$

$$\Delta\theta = b_2 t + \frac{H^2}{6ab_1} b_H (b_1 + 2b_H) - \frac{y_H^2 H^2}{15a} \frac{b_H (b_1 - b_H)}{B_y} -$$

$$- \frac{y_H^4 H^2}{90ab_1} \frac{(b_1 + 2b_H)(4b_1^2 + 6b_1 b_H + 5b_H^2)}{B_y}, \tag{8}$$

where $B_1 = b_1^2 + b_1 b_H + b_H^2$, $B_2 = 4b_1^2 + 7b_1 b_H + 4b_H^2$, $B_y = 6b_1 - y_H^2(b_1 + 2b_H)$.

It follows from (7), representing the sum of the rates of change in the axial flow and in the flow of losses, that

$$\lambda = \frac{b_{q0}}{b_2} H \left(1 + \frac{y_H^2}{6} \frac{2b_1 + b_H}{b_2}\right)^{-1} = \frac{b_{q0}}{b_2} H \frac{1}{1 + \Psi_\lambda}. \tag{9}$$

The correction $(1 + \Psi_\lambda)^{-1}$, obtained by the method of successive approximations, for the case in which $y_H^2 < 0.9$ with accuracy to terms of the third order of smallness, coincides with the expression for the thermal conductivity from the exact solution of the HE (1).

For the Biot criterion we will write

$$\mathrm{Bi}_H = \frac{b_2}{b_H}\left(1 - \frac{y_H^2}{6} \frac{b_1 + 2b_H}{b_2}\right). \tag{10}$$

Equation (6) for the density of the heat flux in the x = 0 plane, in addition to the terms describing the transit heat flow and the absorption flow (the first and second terms of the expression) contains a term that is associated with the heat-loss flow q_ℓ. This term is determined by the delay in the heat flow within the specimen in its relation to the heat flow q_0 generated by the heat source, in terms of the quantity $2q_\ell$ that is related to the regularization of the heating regime in terms of temperature, and to conserve the balance of heat on transition in Eq. (6) to the heat flow q_0 generated by the heat source, the quantity $2\,|q_\ell|$ should be added. In this regard, the flow of heat introduced into the specimen is determined by the relationship

$$q_0 = b_{q0} t + c_p \rho H \frac{B_1}{3b_1} (1 + \Psi_c), \tag{11}$$

$$\Psi_c = y_H^2 B_2/(5B_1 B_y),$$

from which

$$c_p \rho = \frac{q_0 - b_{q0}\theta_1/b_1}{b_1^2 + b_1 b_H + b_H^2} \frac{3b_1}{H} \frac{1}{1 + \Psi_c}. \tag{12}$$

In accordance with (8) the thermal diffusivity is determined from the expression

$$a = \frac{b_H(b_1 + 2b_H)}{\Delta\theta - b_2\theta_1/b_1} \frac{H^2}{6b_1}(1 - \Psi_a), \tag{13}$$

where

$$\Psi_a = \frac{2y_H^2}{5B_y}\left[\frac{b_1(b_1 - b_H)}{b_1 + 2b_H} + \frac{y_H^2}{6}\frac{4b_1^2 + 6b_1 b_H + 5b_H^2}{b_H}\right].$$

As follows out of the cited relationships, a unique feature of the LTG method is the fact that it is possible to calculate the magnitudes of the correction factors for the loss of heat through the sides directly from experimental data, since information about the integral emissivity of the specimen surface can be obtained through direct measurements of the Biot criterion (10).

An estimate of the magnitude of the correction factors for thermal insulators with effective parameters of λ = 0.1-1.0 W/(m·K) and a = (0.1-1.0)·10^{-6} m^2/sec, a reference surface emissivity of ε = 0.2 at temperatures of 1700-2000 K, shows that when we take into consideration correction factors with an error of 20% the nonexcluded portion of the correction factors for the heat losses through the sides amounts to approximately 5-6% for thermal conductivity, 2-3% for the volumetric heat capacity, and 4-6% for the thermal diffusivity.

Figure 1 shows the results obtained in the measurement of the relationship to temperature on the part of the volumetric heat capacity for chemically pure (not less than 99% Al_2O_3) corundum ceramics based on hollow sintered microspheres with a diameter of approximately 40-50 μm (foam corundum).

We investigated foam corundum specimens with an apparent density of 1.12·10^3 kg/m^3, 30 mm in diameter, and a thickness of 4.82 mm at temperatures of 900-1800 K in a vacuum of about 10^{-3} Pa. The temperature of the specimen and the temperature differences were measured by means of VR 5/20 thermocouples with electrode diameters of 100 μm, mounted in the channels across the width of the cylinder, parallel to the surfaces of the specimen to a depth of 0.2-0.3 mm from the surface. The temperature of the inside surface of the specimen was raised by no more than 5-7 K within 200 sec of heating. The volumetric heat capacity at each point was measured 5-7 times, and the results were subsequently averaged. The limits of random measurement error amount approximately to 8%, and without consideration of the loss of heat through the sides the system error amounts approximately to 15% for any heating rate.

Comparison of the experimental results obtained without consideration of the lateral heat losses against the values of the volumetric heat capacity calculated on the basis of the data recommended for corundum [4] shows that the difference between the averaged curve and the recommended values amounts approximately to 25% and exceeds the error in the experimental data.

On introduction of correction factors for the lateral heat losses, we come up against two possibilities: the utilization of the recommended data with respect to the radiative characteristics of corundum [5] and the utilization of the radiation characteristics of the specimen, determined from experimental data.

For purposes of introducing correction factors for the lateral heat losses, the utilization of a hemispherical integral emissivity (Fig. 2), calculated by means of the recommended values for normal emissivity [5] under the assumption that these values exhibit a cosine distribution, such as is valid for a diffusion surface, demonstrated that such an approach leads to a lowering of the heat-capacity values in the high-temperature regions and to a change in the temperature coefficient $c_p\rho$ to the negative which, apparently, is associated with exaggeration of the correction factors in the case of high temperatures.

The values of the hemispherical integral emissivity determined from (10) in the low-temperature region coincides with the recommended data, while in the high-temperature region they differ from the former by 35-40% (Fig. 2), which is a consequence of the state of the specimen surface. Research results with respect to the radiative properties of corundum surfaces, such as those obtained by various authors, demonstrate a broad range of variations with respect to both magnitude and the temperature coefficient [5] (Fig. 2). The error in the calculation of ε_{the} in conjunction with consideration of the error in the radiative characteristics of the shielding is estimated at 20-25%.

Introduction of correction factors which take into consideration the radiative properties of the specimen produced no change in the temperature curve of the volumetric heat capacity (see Fig. 1). Divergence from the recommended data on the part of the experimental results amounts approximately to 10-12% and does not exceed experimental error.

These results give evidence to the effect that the proposed mathematical model satisfactorily describes a regular regime of monotonic heating of a bounded cylinder by means of an internal heat source with time-linear release of heat in the presence of heat exchange between the side surface and the ambient medium, and it can be used to find the correction factors for the lateral losses of heat in the calculation of the thermophysical coefficients when $y_H^2 < 0.9$.

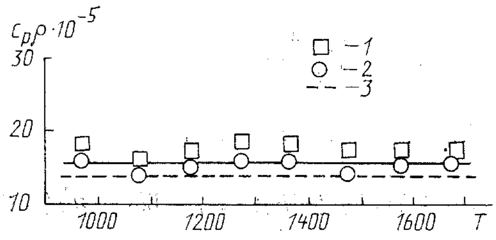

Fig. 1

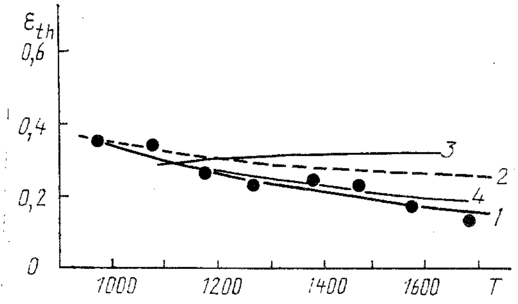

Fig. 2

Fig. 1. Volumetric heat capacity of the foam corundum as a function of temperature: 1) prior to introduction of correction factor; 2) subsequent to introduction of correction factors with respect to the loss of heat through the sides; 3) volumetric heat capacity, calculated on the basis of the data recommended in [4]. $c_p\rho$, $J/(m^3 \cdot K)$; T, K.

Fig. 2. Hemispherical integral emissivity: 1) experimental results; theoretical results; 2) according to the data for normal emissivity, as recommended in [5]; 3, 4) on the basis of data presented in [5].

NOTATION

λ, $c_p\rho$, a, coefficients of thermal conductivity, volumetric heat capacity at constant pressure, coefficient of thermal diffusivity for the specimen; T_0, temperature of the ambient medium; x, r, cylindrical coordinates; $\theta(x, r, t) = T(x, r, t) - T_0$, excess specimen temperature; $y_{H^2} = 8\varepsilon_R\varphi_R\sigma T_0^3H^2/(\lambda R)$, parameter characterizing lateral heat losses; b_2, rates of change in temperature at inside and outside surfaces of the specimens; b_1, b_H, rate of change in pressure difference across the specimen; $q_0(t)$, b_{q0}, heat flow in the x = 0 plane and the rate of its change; $Bi_H = 4\varepsilon_H\varphi_H\sigma T_0^3H/\lambda$, Biot criterion for the end surface; H, R, thickness and radius of specimen; ε_H, ε_R, reduced emissivities; φ_H, φ_R, angular coefficients for end and side surfaces, respectively; ε_{the}, hemispherical integral emissivity.

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