DETERMINATION OF THERMAL STABILITY AND THERMOPHYSICAL CHARACTERISTICS OF CORUNDUM MATERIALS

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In order to determine the capacity of brittle materials to resist the effect of thermal loading, it is necessary to determine both the thermal stability of these materials and the temperature dependence of their thermophysical properties, which are responsible for peculiarities of temperature distribution leading to disruptive thermal loading. It is appropriate that experiments be conducted for this purpose [1, 2], since in this way we not only diminish the amount of necessary work, but we increase our confidence in the reliability of the results. This excludes the possible effect of technological variations in preparing the samples, properties of which are not generally reproduced with absolute fidelity.

In the present work we have developed a method of composite investigations of thermal stability and thermophysical characteristics of materials in a wide range of temperatures (up to the melting points of the materials). This information was previously used only for determining the thermal stability of test samples.

The experimental part of this work was carried out on devices ([3] and others) designed for studying the thermal stability of materials in hollow cylindrical samples (1 in Fig. 1). In the samples, which were arranged in a packet of height $H \geq 8R$ to obtain a uniform temperature field in the middle part of the packet, a temperature drop was produced by means of a resistance heater (2), causing failure, and this was registered by an automatic recorder (3). During the thermal loading, automatically recording ÉPP-09M3

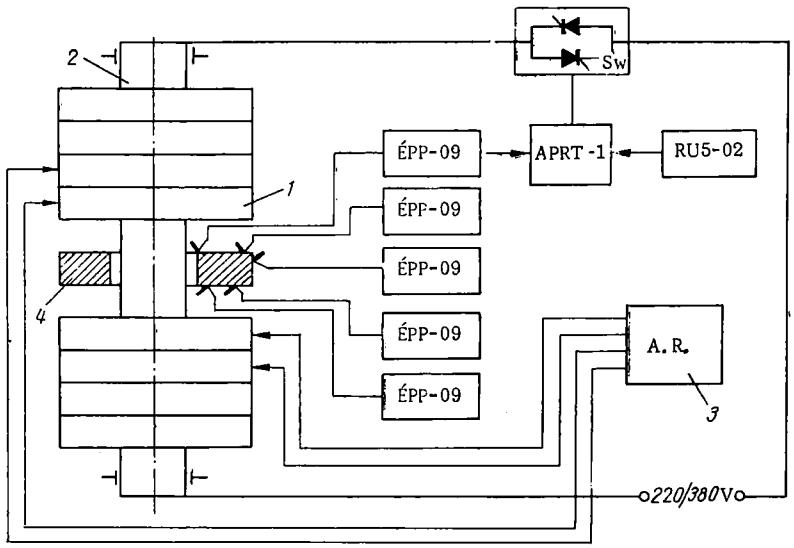

Fig. 1. Experimental setup.

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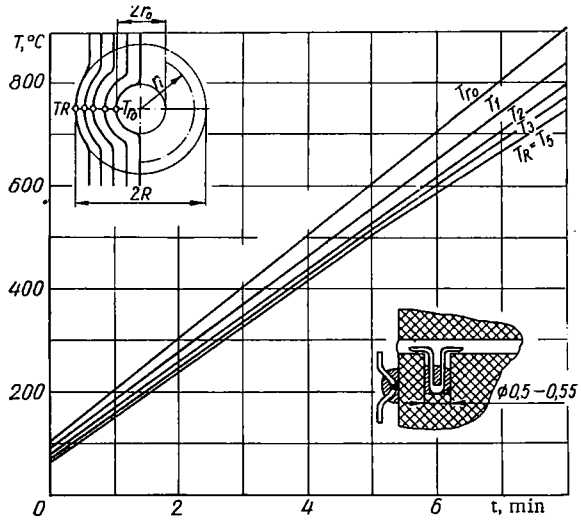

Fig. 2

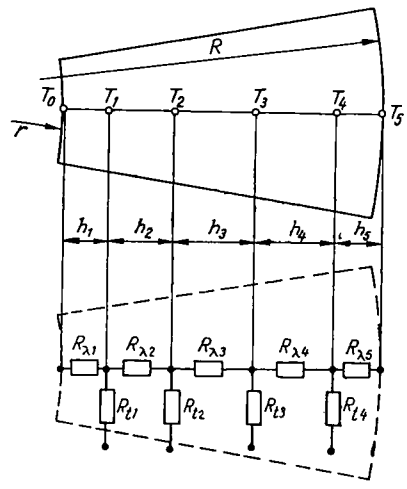

Fig. 3

Fig. 2. Time dependence of experimental temperatures at different points in a sample of Al_2O_3 (composition 1).

Fig. 3. Schematic view of the element of a test specimen and its electrical analog.

potentiometers noted the temperatures measured by five platinum–platinorhodium thermocouples with thermoelectrodes 0.2 mm in diameter set in a thermometric sample (4). These thermocouples were calibrated in the testing laboratory of the Committee of Standards of the USSR. The accuracy of readings of the potentiometers connected with the thermocouples was checked before the beginning of the experiment and after its completion by a PP-63 control potentiometer. Necessary corrections were employed in plotting the function T = f(t), one curve of this function being shown in Fig. 2.* To reduce the error in measurements, the junctions of the thermocouples were soldered ultrasonically into depressions 0.5 mm in diameter, and the electrodes of the thermocouple were placed on the samples along isotherms. The cold joints of the thermocouples were thermostatically controlled. In order to avoid induction, a compensating lead was attached to the ground shield. In addition, each electrode of the thermocouples was grounded through paper capacitors, and special filters were used to suppress noise. The test samples of corundum were prepared at a pilot plant and in the laboratory of the Ukrainian Scientific Research Institute for the Organization of Production and Management of Industry (UNIIO). The principal characteristics of the materials are shown in Table 1.

Investigations were made in linear heating intervals [4] during which the change of temperature with time at all points in the samples followed the linear law with a rather high degree of accuracy. Heating of the specimens was controlled by means of an APRT-1 automatic regulator connected to the switch (Sw). For all tests, the rate of temperature change on the internal surfaces of the specimens was constant, amounting to 100 deg/min.

The criterion for failure of a sample was the formation of radial fractures in it, and the thermal stability of the sample was determined from the value of temperature drop ΔT between the inner and outer

* In this figure T_4 is not shown; $T_4 - T_5 \approx 5$–15°C.

TABLE 1

Material	Chemical composition, %			Porosity, %	Specific weight, g/cm³
	Al_2O_3	introduced additive	other		
Composition 1	99,73	—	0,27	1	3,67
Composition 2	99,37	—	0,63	29,8	2,66
Composition 3	99,80	0,02% MgO	0,18	0	3,80
Composition 4	85	15% Z_2SO_4	—	23	3,45

TABLE 2

Material	T_{max}, °C	ΔT_{av}, deg	R, deg	R', kcal/m·h
Composition 1*	730	121	292	2220
Composition 2	775	215	59	85,5
Composition 3	680	112	478	3394
Composition 4	740	247	372	1414

* For other sample groups of this composition, ΔT = 110 °C.

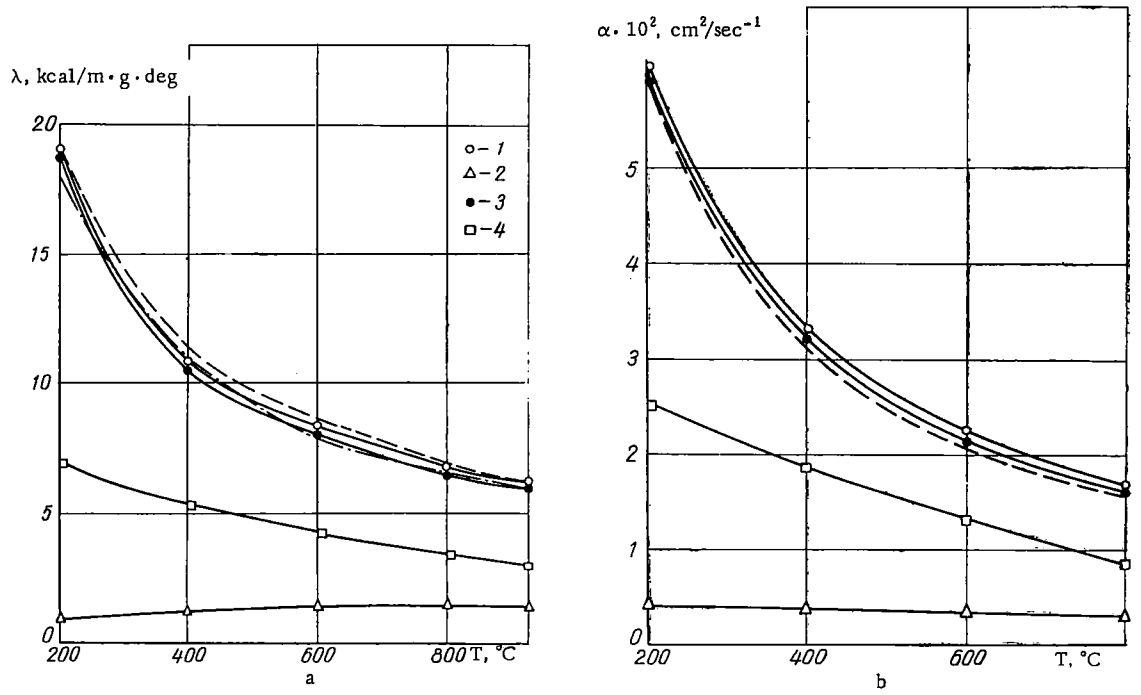

Fig. 4. Temperature dependence of the coefficients of thermal conductivity (a) and thermal diffusivity (b): 1) composition 1; 2) composition 2; 3) composition 3; 4) composition 4.

surfaces of the cylinder causing the cracking. Results of the tests are shown in Table 2, in which we have given average values obtained from no fewer than eight to ten samples.

To determine the temperature dependence of the thermophysical characteristics of the test materials, a method was developed for solving the nonlinear inversion problems of transient thermal conductivity [5]. This method involved mathematical modeling based on the analogy between differential equations defining the processes of heat convection and those for the distribution of electrical potentials in an electrically conducting medium. In the investigated example, solution of the problem was carried out on an EINP grid integrator.* In this connection, the analogy between the finite-difference equation of thermal conductivity

$$2\frac{\dfrac{\lambda_{r_1}\dfrac{T_{1,n}-T_{0,n}}{h_1}-\lambda_{r_2}\dfrac{T_{0,n}-T_{2,n}}{h_2}}{h_1+h_2}+\frac{1}{r}}{\times\dfrac{\lambda_{r_1}(T_{1,n}-T_{0,n})-\lambda_{r_2}(T_{2,n}-T_{0,n})}{h_1+h_2}}+(c\varrho)_{0,n-1}\frac{T_{0,n-1}-T_{0,n}}{\delta t}=0, \quad (1)$$

approximating a differential equation in partial derivates defined in a cylindrical system of coordinates and the equation of electrical currents (Kirchhoff's law) in resistances convergent at a junction

$$\frac{V_{1,n}-V_{0,n}}{R_1}+\frac{V_{2,n}-V_{0,n}}{R_2}+\frac{V_{0,n-1}-V_{0,n}}{R_t}=0 \quad (2)$$

was used for computing the parameters of the model.

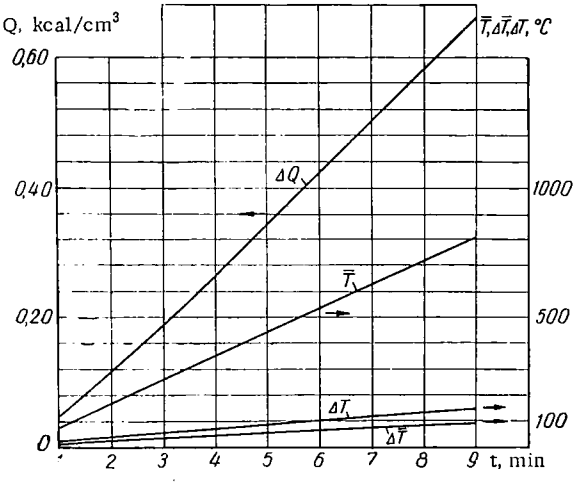

Fig. 5. Curves of mean integral temperature T, mean integral temperature drop $\Delta\overline{T}$, greatest temperature drop ΔT, and the quantity of heat accumulating in the sample.

* It is also possible to use the measuring schemes of series integrators of the EGDA, EI, USM, MSM, SEI, and other types for solving the proposed problem.

Figure 3 represents an electrical analog, an R grid, modeling the transient process of heat convection in a sample. The wall of the specimen is marked off in radial segments h corresponding to points of temperature measurement (positions of the thermocouples).

The total time of the test was broken down into n time intervals: steps in time ($t = n\delta t$). The thermal resistance of thermal convection and the thermal resistance accounting for the heat capacity of the sample were modeled by the ohmic resistances R_λ and R_t. These resistances were determined from relations [6] deriving from the analogy between expressions (1) and (2):

$$R_{\lambda_{1(2)}} = \frac{h_{1(2)} R_N}{2\lambda_{r_{1(2)}} \left(1 \pm \frac{h_{1(2)}}{2r}\right)}; \tag{3}$$

$$R_t = \frac{\delta t R_N}{(cQ)_{n-1} (h_1 + h_2) r}. \tag{4}$$

In the first step of the solution, the model parameters R_λ and R_t were computed from the given values of λ and c_v adopted as initial values. These values were determined at 150°C for compositions 1, 3, and 4 at the Ukrainian Scientific-Research Institute for the Organization of Production and Management of Industry (UNIIO) and for composition 2 at the Institute of Strength of Materials of the Academy of Sciences, Ukrainian SSR (IMP AN USSR).

To find the coefficients of thermal capacity and the volume specific heat c_v at each step in time, the values of R_λ and R_t were selected on the basis of the best description of experimental curves of $T(r, t)$ by Eq. (1); in practice they were selected from the condition of coincidence of temperatures at the investigated points. Thus, determination of the temperature dependence of $\lambda(T)$ and $c_v(T)$ reduced to minimization of the value

$$\Delta = \sum_{k=1}^{m} \sum_{i=1}^{n} [T(r_k, t_i)_M - T(r_k, t_i)_e]^2, \tag{5}$$

where $k = 1, 2, \ldots, m$ represents the number of experimental points except the boundary points; $i = 1, \ldots, n$ represent fixed moments of time; T_M is the solution obtained on the electrical model; and T_e the value from experimental data.

The values of the coefficients λ and a computed from (3) and (4) are shown in Fig. 4. The coefficient of thermal conductivity was determined from the well-known formula $a = \lambda / c_v$.

The value Δ is the criterion for determining the quality of the solution obtained, which should be carefully analyzed at each step. We should take into consideration the possibility of secondary peculiarities of the investigated phenomenon (particularly structural transformations, observed in many brittle materials). It must be noted that in solving the inverse problem by means of the electrical analog method it is easy to recognize breakdown of the law $\lambda(T)$. This leads us to conclude that inhomogeneities in structure of the sample in the radial direction are possible (such as irregular porosity, phase transformation of part of the sample, etc.), which commonly cannot be established by other methods, and without consideration of which one may obtain erroneous data concerning the thermal stability of the material.

The results of each iteration are used for obtaining the value of condition (5), from which the parameters of the model are changed. To facilitate the computation, the iteration process for determining values of λ and c_v may be made automatic.

In order to reduce to a minimum the error in determining the thermophysical characteristics, the experimental temperature fields were measured with minimal error. The accuracy assumed for the electrical analog method is about 1%.

A comparison of the values of λ and c_v determined by the indicated method with results obtained by other methods (data of Skarbinskii [7] for a composition similar to composition 1 (dashed lines) and of the Ukrainian Scientific Research Institute for the Organization of Production and Management of Industry (UNIIO) for composition 1 (dash-dot line)) is shown in Fig. 4. As seen from this figure, the values of thermophysical characteristics obtained in the present work differ from those used in the comparison by $\pm 10\%$, which may be considered completely satisfactory. Such precision in determining the values of λ and c_v for the method here proposed is not ultimate, but follows from the fact that the comparison was made with results having substantial scatter. According to experimental data in determining the values of λ and

c_v for materials for which these characteristics are rather well known (for example, carbon steel 08) the error does not exceed ±5%.

The described method of the attendant thermophysical investigations permits us, with a small number of experimental points of temperature measurement, to restore the temperature field throughout the entire thickness of the sample. The interpolation made here is linear and valid when there are no phase or other structural transformations in the investigated material, i.e., when $\lambda(T)$ and $c_v(T)$ vary smoothly and uniformly.

Thus, because of the use of the electrical analog method along with determination of the values of λ and c_v, we may simultaneously and automatically solve the problem of interpolation of the temperature field and may replace the plotting of interpolation polynomials and graphic interpolation previously used in such investigations of thermal stability. The error in our interpolation does not exceed 1% of the maximum temperature value. Use of the indicated method when it is necessary to know the temperature at many points in the sample in order to compute thermal stresses and potential energy accumulating in the sample, and other properties, proves to be less laborious and more reliable than, for example, the graphical method of interpolation.

In order to obtain more complete information concerning the limiting state of thermally loaded samples tested for thermal stability, it is important to determine the mean integral temperature

$$\overline{T} = \frac{2}{R^2 - r_0^2} \int_{r_0}^{R} rT(r, t)\, dr, \tag{6}$$

the mean integral temperature drop $\Delta\overline{T} = T_{max} - \overline{T}$, and also the corresponding specific quantity of heat per unit volume accumulating in the sample during the time of loading,

$$\Delta Q_i = \sum_{1}^{i} c_{i-0.5}\varrho\,(\overline{T}_i - \overline{T}_{i-1}), \tag{7}$$

where $c_{i-0.5} = c_i + c_{i-1}/2$ (i is the time).

As an example, in Fig. 5 we have shown the results of numerical calculations of ΔQ, $\overline{T}$, $\Delta\overline{T}$, and ΔT for composition 1.

The values of λ and a (see Fig. 4) were used for computing the well-known criteria of thermal stability R and R', which define, according to Kingery's data [8], the formation of cracks in the samples (see Table 2).

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