Porous refractory articles were prepared at the Magnesite Combine using fused periclase grains (uniform granules) of fractions of 1-0.5 mm and granules of the laminated structure with the applied layer consisting of 97% MgO and 3% ZrO_2. The pressing pressure was 10 MPa. The articles were fired in a tunnel kiln at 1080°C.

It is clear from Table 3 that the articles made from the laminated granules are significantly stronger than those made from the continuous granules. The thermal-shock resistance and the open porosity are also higher.

CONCLUSIONS

It is shown that it is possible to produce strong granules of a laminated structure based on dense periclase grains and active finely dispersed materials. The optimum production parameters have been determined. Some features of the structure and phase composition of the laminated granules obtained on the basis of periclase materials activated by additions of ZrO_2, Al_2O_3, La_2O_3, and mixtures of these are described.

It is established that a characteristic feature of the structure of laminated granules is the formation on the surface layer of minerals whose presence can intensify the sintering of the masses and consequently direct the structure-formation processes in the production of an industrial ceramic.

The accumulation of industrial experience has shown that on the basis of laminated granules it is possible to make periclase articles with an open porosity $\geq$ 60%. By comparison with articles made from granules of a uniform structure their ultimate compressive strength and thermal-shock resistance is double.

LITERATURE CITED

1. I. S. Kainarskii, Processes in Refractory Technology [in Russian], Moscow (1969).
2. V. N. Belousov, Izv. Tomsk. Politekh. Inst., No. 5, 198-207 (1959).
3. A. I. Gaodu, N. S. Pashchenko, and N. G. Subochev, Ogneupory, No. 11, 39-41 (1978).
4. N. I. Voronin and N. I. Krasotina, Ogneupory, No. 3, 129-184 (1959).
5. K. V. Simonov, L. Ya. Osipova, V. P. Chernyavskaya, et al., Ogneupory, No. 12, 2-8 (1971).
6. K. V. Simonov, E. P. Mezentsev, V. A. Sokolov, et al., Ogneupory, No. 11, 8-11 (1978).
7. S. M. Zubakov, P. N. Babin, and A. Kh. Akishev, Ogneupory, No. 5, 47-51 (1979).
8. P. N. Babin, A. Kh. Akishev, F. M. Menzhulina, and S. G. Utesheva, "Refractory materials based on MgO, ZrO_2, and rare-earth oxides," in: Express Information [in Russian], Kazakh Scientific-Research Institute of Scientific—and Technical Information, Ser. 9., No. 18, Alma-Ata (1979).

EFFECT OF HYDROSTATIC PRESSING ON SOME PROPERTIES OF A CERAMIC BASED ON Al_2O_3 AND Sc_2O_3

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High-pressure hydrostatic pressing of powders is widely used in the treatment of metals and their alloys [1, 2]. This method is used comparatively rarely in ceramic production [3].

It is well known that the density and strength of a material are increased under the action of high pressures. We have now studied the effect of the pressure in hydrostatic pressing on some of the physicomechanical properties of high-melting oxides.

As the experimental materials we used alumina powder modified with small additions of magnesium oxide (0.3%) and also scandium oxide powder, "exceptionally pure-99.9" grade. Blanks were made from the starting materials in metal molds under a pressure of 100 MPa. Some of the blanks were then hydrostatically

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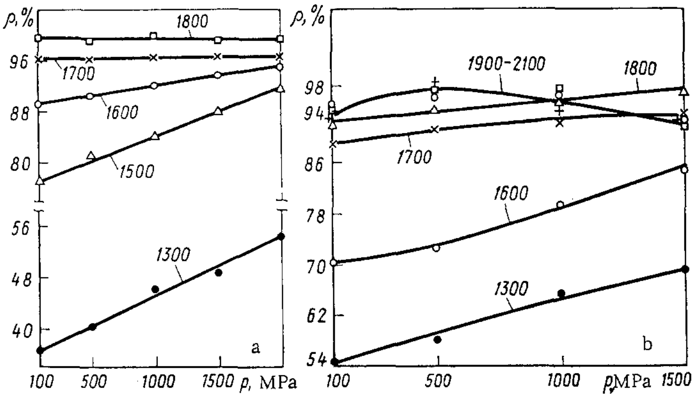

Fig. 1. Dependence of the relative density ρ of specimens of Al_2O_3 (a) and Sc_2O_3 (b) on the pressing pressure p; figures on curves indicate the heat-treatment temperature of the specimens, °C.

pressed at 100 up to 2000 MPa in a container with a working diameter of 20 mm and a height of 200 mm mounted in a PD-476 press. As the working liquid we used the industrial "20" lubricant.

The blanks were fired at 1300°C for 1 h in an air medium and sintered in vacuum with a residual gas pressure of 0.133-1.33 MPa in the 1500-1800°C interval (Al_2O_3 specimens) and 1500-2100°C (specimens of Sc_2O_3) in steps of 100°C and a dwell of 1 h at the maximum temperature. For these specimens we studied the effect of high pressures on the density, microstructure, microhardness, and ultimate bend strength.

The density of the specimens was determined by hydrostatic weighing in distilled water. The microstructure of polished slices was determined using an MBI-11 microscope. The microhardness was determined in a PMT-3 microhardness meter using the usual method; the load on the Al_2O_3 was 100 g and on the Sc_2O_3, 50 g. The ultimate bend strength, σ_{bend}, was measured with a PT250M in accordance with a three-point bend system on specimens measuring 7 × 7 × 60 mm.

Figure 1 shows the dependence of the density of the Al_2O_3 and Sc_2O_3 specimens on the pressure of the hydrostatic pressing at various sintering temperatures. At relatively low temperatures the density of the specimens of both compositions increases with the hydrostatic pressing pressure at pressures of 1500-2000 MPa by 50% on average for the Al_2O_3 specimens and by 25% for the Sc_2O_3. When the Al_2O_3 is sintered in the 1500-1600°C interval and Sc_2O_3 in the 1600-1900°C interval, the effect of pressure on the density of the specimens is quite significant but with an increase in temperature the effect becomes markedly weaker. The pressing pressure has little effect on the average grain size of the Sc_2O_3 ceramic (Table 1). An increase in the grain size in the Al_2O_3 specimens from 10 up to 15-20 μm is observed at 1700°C and from 40 to 50-60 μm at 1800°C.

A preliminary analysis of the experimental dependences of σ_{bend} on the sintering temperature and pressure under hydrostatic pressing showed that in the experimental interval of these parameters, the strength

TABLE 1. Characteristics of Specimens of Al_2O_3 and Sc_2O_3 Ceramics

Characteristic	Value of characteristic for specimen of			
	Al_2O_3		Sc_2O_3	
	obtained by pressing in			
	semidry method	hydrostatic method*	semidry method	hydrostatic method†
Sintering temperature, °C	1800	1700/1700	2000	1900/1900
Relative density, %	99,0	98,9/99,0	94,5	94,6/98,0
Average size of crystals, μm	40	13/19	11	11/11
Microhardness, MPa	22,0	22,3/25,8	8,5	8,6/9,5
Ultimate bend strength, MPa	180	150/310	113	102/155

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*Numerator, under pressure of 100 MPa; denominator, 2000 MPa.

†Numerator, under 100 MPa; denominator, 800 MPa.

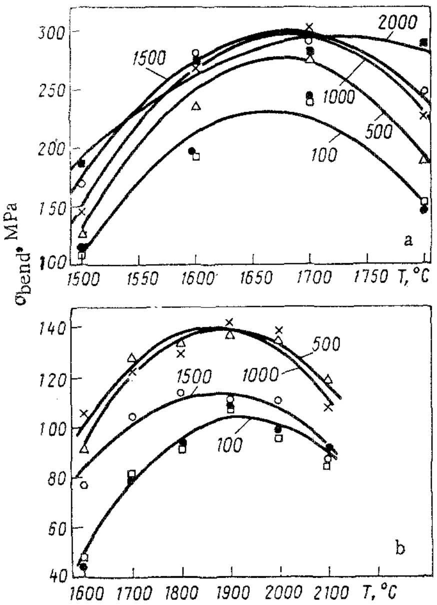

Fig. 2. The calculated dependence of the ultimate bend strength σ_{bend} on the sintering temperature T of a ceramic based on Al_2O_3 (a) and Sc_2O_3 (b): ●, Δ, x, o, ■) extremal points corresponding to the hydrostatic pressure; □) points corresponding to semidry pressing; figures on curves indicate the pressing pressure, MPa.

reaches extremal values (each experimental point was calculated as the average of the results from 5-10 measurements). In order to determine the optimal parameters for the production process of obtaining an oxide ceramic with improved strength, the results were processed using the AS OSD [4] system in an M-222 computer according to the method involving a multifactor regression analysis [5].

To obtain a model of the process we used passive observations. As the variables which affect the process we chose the sintering temperature T and pressure p. On the basis of accumulated statistics, we obtained the equations of a pairwise regression of the output parameter, σ_{bend}, as a function of the variable T with fixed values of p. Second-order polynomials of the form

$$\sigma_{bend} = a_0 + a_1 T + a_2 T^2, \tag{1}$$

where a_0, a_1, and a_2 are coefficients, were found to be adequate dependences in all cases.

Consequently, for all fixed values of the pressure there is a temperature at which σ_{bend} has a maximum value (Fig. 2).

In the second stage of the study we carried out a statistical treatment of the results using multifactor regression analysis. Power regression equations were obtained for the experimental materials which were considered as the first approximation of the process model.

In the case of the Al_2O_3 ceramic, the power regression equation in a standardized scale has the following form:

$$\sigma_{bend} = -36.46 + 80.09T + 0.68Tp - 0.32p^2 - 43.1T^2. \tag{2}$$

Equation (2) made it possible to calculate the optimal values of p and T in order to obtain maximum values of σ_{bend}: T_{opt} = 1686°C and p_{opt} = 2000 MPa.

It is clear from Eq. (2) that σ_{bend} of the ceramic depends significantly on the sintering temperature, the interaction factor of the temperature and pressure, and the square of the pressure. There is no term in the equation which takes into account the effect of the pressure in the experimental range on σ_{bend} since the coefficient in this case is insignificant.

It is clear from the curves shown in Fig. 2a that the greatest value of σ_{bend} is found when T'_{opt} = 1683°C and p'_{opt} = 1500 MPa; this is in good agreement with the conclusions reached from the power regression equation. The difference in the value of the optimum pressure leads to a 2% change in σ_{bend}, confirming the very small effect due to pressure in this particular range.

In the case of Sc_2O_3, the multifactor regression equation has the following form in a standardized scale:

$$\sigma_{\text{bend}} = -6{,}93 + 1{,}95p + 15{,}92T - 0{,}95pT - 1.33p^2 - 8.59T^2. \tag{3}$$

Hence p_{opt} = 822 MPa and T_{opt} = 1900°C.

From the family of curves in Fig. 2b, we have the following: p'_{opt} = 1000 MPa and T'_{opt} = 1878°C (the difference between the values of σ_{bend} for p_{opt}, T_{opt} and p'_{opt}, T'_{opt} is $\leq 2\%$).

An analysis of Eq. (3) suggested that in the case of Sc_2O_3 as in the case of Al_2O_3, σ_{bend} depends significantly on the sintering temperature and to a lesser extent on the pressing pressure.

In processing the experimental material, we considered a linear model which would take into account both the linear terms and also the interaction coefficients. In both cases the equations representing a complete second-order polynomial as a function of two variables were adequate. The quality (correctness) of the obtained regression equations were checked using remainder analysis [4].

The mathematical treatment of the experimental data made it possible to evaluate the character of the effect of the production parameters (p and T) on the mechanical properties of the experimental materials and to calculate their optimal values.

The data in Table 1 show that at the same pressures for the semidry and hydrostatic pressing methods (100 MPa), the characteristics of the ceramic differ very little. The use of high pressure in hydrostatic pressing makes it possible to obtain high-density ceramic materials at a sintering temperature 100-150°C below the temperature of specimens pressed under a pressure of 100 MPa and also to increase the strength of the materials.

CONCLUSIONS

The effect of the hydrostatic-pressing pressure on some of the physicomechanical properties of ceramics based on Al_2O_3 and Sc_2O_3 is shown. Mathematical models describing the dependences of the strength of the Al_2O_3 and Sc_2O_3 materials on the sintering conditions and the pressures of the hydrostatic pressing have been constructed.

The regimes for producing a ceramic based on Al_2O_3 and Sc_2O_3 with improved properties have been optimized.

LITERATURE CITED

1. H. L. Puch, Mechanical Properties of Materials Under a High Pressure [Russian translation], No. I, Moscow (1973).
2. G. P. Mikhailenko and Yu. F. Chernyi, Poroshk. Metall., No. 9, 21-27 (1977).
3. K. K. Artemova, S. P. Lalykin, L. A. Rudenko, and G. Ya. Maslova, in: All-Union Conference, "Hydrostatic Treatment of Materials": Discussion Themes [in Russian], Donets (1979), p. 73.
4. V. A. Vasil'ev, I. V. Gorynin, S. K. Mikhailov, et al., An Automatic System for Processing Statistical Data [in Russian], Leningrad (1977).
5. N. V. Smirnov and N. V. Dunin-Barkovskii, Course in Probability Theory and Mathematical Statistics for Technical Applications [in Russian], Moscow (1969).