

Development of modern technology, including aviation, requires the creation of high-temperature ceramics that possess a range of specified properties that are frequently incompatible.

The aim of the present work was to develop a ceramic material and technology for making sensors for measuring high temperatures in aviation gas-turbine engines. These sensors should be capable of operating in combustion products from jet fuels at working-media temperatures of 1600-2000°C, and satisfy severe operating demands in relation to vibration loads. The sensors are very complex in shape and should have high-precision dimensions. Thus, the ceramic articles should have geometrical tolerances of  $\pm 0.3$  mm, and a surface finish equal to class 0.8-0.63. This applies to the internal surfaces, including small-diameter apertures.

The task was approached by selecting materials with a melting temperature above 2000°C in the form of magnesia and alumina, and also magnesium spinel,  $MgAl_2O_4$ , synthesized from a stoichiometric mixture of these oxides. Calcined magnesia fired at high temperatures has a good resistance to organic acids and acidic gases. In terms of resistance to carbon, pure magnesia exceeds many high-temperature oxides.

The authors of [1] show that in the 1580-2080°C range in vacuum, the reaction of  $Al_2O_3$ ,  $BeO$ , and  $ThO_2$  with carbon is limited by the stages of diffusion and is subject to the parabolic law. The rate of reaction between carbon and  $MgO$  or  $MgAl_2O_4$  is determined by the reactions of the phase boundaries, and obeys a linear function. The relative reaction capacity increases in the series  $BeO$ - $MgO$ - $ThO_2$ - $Al_2O_3$ - $TiO_2$ .

Moreover, many properties of ceramics can be controlled by alloying with other oxides, which influence the technical properties, and also the formation of their structure and phase composition [2]. We tried a range of rare-earth oxides (REO) in amounts of 4% in the magnesia-spinel composition: yttrium, ytterbium, holmium, dysprosium, and cerium. The quantity of additive used was determined by the solubility limit, approximately equal to 4% for the  $MgO$ -REO system.

The microstructure of the spinel ceramic fired at 1750°C without and with additives of 4% (added to the batch on 100%) of rare earths was studied on polished slides. To obtain a clearer picture the slides were etched for 15 min in  $KOH$ .

Investigations showed that spinel ceramic containing 4% REO has a finely crystalline structure with very dense grain packing (see Fig. 1). The average grain size of the spinel without additives is 15-20  $\mu$ , with ytterbium and dysprosium oxides up to 10-15  $\mu$ , and with yttria additions- 20-25  $\mu$ .

The intracrystalline porosity is only 1-4%. This indicates the low rate of grain growth in the spinel (in contrast with periclase), owing to which the pores are not entrapped by their boundaries. The structure of spinel ceramics contains direct bonds between the weakly profiled octahedral crystals. The addition of yttria contributes to the formation of a clearer grain configuration, inside which pores are almost absent. The finer crystallized spinel with clearly contoured grains is characteristic of ceramics containing dysprosium oxide, and especially ytterbium oxide. Cerium oxide does not have a marked influence on the structure of the spinel, and is present in the form of fine inclusions, orientated at the grain boundaries.

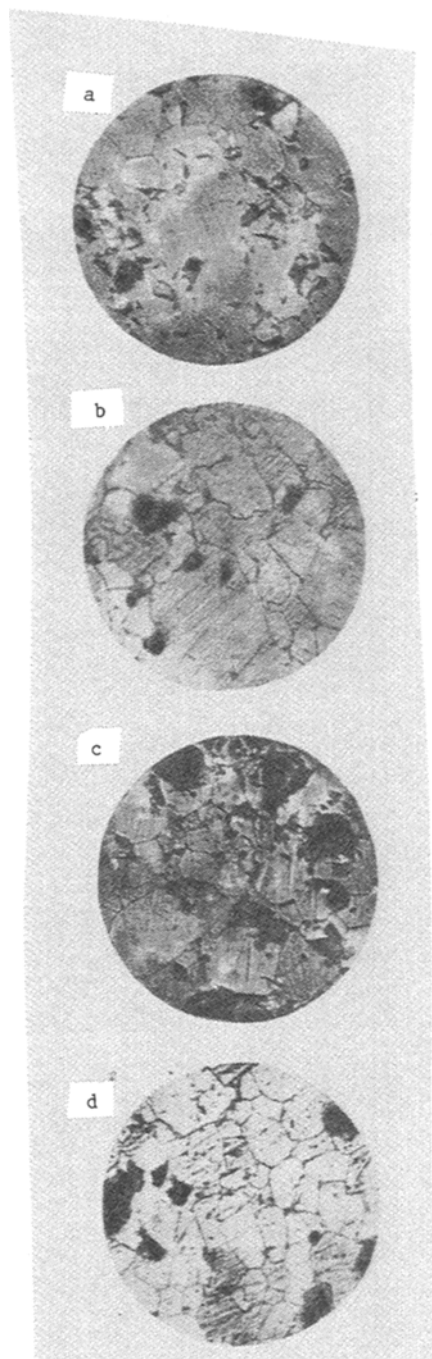


Fig. 1. Microstructure of magnesium spinel ceramic ( $\times 500$ ): a) no additives; b) with 4%  $Y_2O_3$ ; c) 4%  $Yb_2O_3$ ; d) 4%  $Dy_2O_3$ .

The efficacy of the various rare earths on sintering and the formation of the microstructure of magnesia spinel is explained by the crystallochemical features of the cations which cause an increase in the activity of rare-earth oxides in forming cationic vacancies. The properties of the fired specimens of this spinel are shown in Table 1.

As we see, alloying spinel ceramic with rare earths generally improves their physical properties. However, their action on sintering, the structure formation, and the properties of the ceramics is irregular. Among the compositions studied here the  $Y_2O_3$  addition increases only the tensile strength, but reduces the thermal-shock resistance, and spinel containing  $Ho_2O_3$  has low compressive and bending strengths. Ceramics containing ytterbium and dysprosium have better and more stable properties.

TABLE 1

Property	Properties of specimen based on $MgAl_2O_4$					
	without additive	with additive				
		$Y_2O_3$	$Yb_2O_3$	$Dy_2O_3$	$Ho_2O_3$	$CeO_2$
Linear shrinkage, %	19.6	16.5	19.5	19.7	17.8	19.4
Apparent density, $g/cm^3$	3.39	3.42	3.45	3.44	3.38	3.40
Strength, MPa						
compressive	251.0	263.5	276.8	286.0	167.8	267.5
bending						
in air	145.5	145.7	153.5	169.7	64.8	128.4
in vacuum $5 \times 10^5$ mm Hg						
stretching	149.8	145.8	154.5	171.2	67.1	141.7
Thermal coefficient of expansion $10^{-6} \text{ } ^\circ C^{-1}$	19.8	26.7	22.5	24.2	25.4	26.5
Thermal-shock resistance (1250°C-air), heat cycles to cracking	8.21	8.22	8.19	8.30	8.44	8.25
Thermal-shock resistance (1250°C-air), heat cycles to cracking	14	10	12	17	15	17

To obtain useful articles with a completed structure we used spinel with 4%  $Dy_2O_3$ , and hot casting under pressure. The magnesium spinel presynthesized with 4%  $Dy_2O_3$  was milled in air for 24 h to obtain particles of  $< 45 \mu$ . The slip was dried, and calcined at 1200°C, since even the residual moisture of 0.1-0.2% in the powder sharply impairs the wettability of the temporary bond, and as a result the casting properties of the system.

The thermoplasticizing components consisted of activated paraffin with a density of 0.9 and 0.8  $g/cm^3$  in the melted state.

To improve the wetting of mineral powder and obtain stable, fluid casting slips with relatively small contents of organic bond, we used oleic acid as the surfactant. The amount of plasticizing bond was chosen empirically; it varied in the range 15-23% of the mass of the mineral powder.

The working temperature of the slip for casting articles with a wall thickness of less than 3-4 mm was 55-60 °C, and for thicknesses of more than 5 mm at 53-55°C.

The articles were cast in metal molds at 0.4-0.6 MPa. In developing these molds we considered the rigid size tolerance for the goods, the complex shape with large ratios of length to diameter (more than 12), the staged internal apertures of low diameter, the configuration, the site and dimensions of the casting heads, and also the problem of splitting the mold and the arrangement for removing the casting and ejecting it from the mold.

It should be noted that at the forming and firing stages it was necessary, without special processing, to obtain a high-purity level on the external and internal surfaces. This was achieved by careful treatment of the mold metal, chrome plating, and obtaining a perfectly clean surface during firing.

The cast articles were heated in a filling of alumina. Calcined at 1300-1350°C (10-60  $\mu$ ) for 45-50 h in order to remove the paraffin.

After the first firing, without additional treatment, the articles were fired in a gas chamber furnace in 145 h with a 6-h soak at a maximum temperature of 1750°C.

Besides the tabulated properties of the spinel ceramic with the dysprosium oxide we also determined a range of specific properties.

Tests for vacuum tightness were made on the PTI-6 leak detector using the blowing method and several specimens. They were found to be vacuum sealed with a sensitivity of 0.3.

Tests in lithium and potassium chloride fusions at 350-650°C showed satisfactory resistance for the ceramic spinel coatings in 6 h.

Testing spinel ceramics alloyed with dysprosium oxide in a current of cesium ions for 5 h, with a particle energy of 500 eV, and a beam power of 500 W established that these can be used as a design material that is resistant to cesium vapor.

Investigations of the resistance of the ceramic in a vacuum of  $(8-5) \cdot 10^{-5}$  mm Hg showed that at 1700°C the mass loss of magnesia spinel without additives was 59.7%, with 4% dysprosium, 10.1%.

Thus, alloying magnesia spinel with rare-earth oxides will ensure the formation of a finely crystalline structure with very dense grain packing and with a range of outstanding physical properties. This led to the development of a technology for making sensors for measuring high temperatures in aviation gas turbine engines.

#### LITERATURE CITED

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#### SPALLING-RESISTANT KILN FURNITURE BASED ON MECHANICALLY ACTIVATED CERAMIC BONDS

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UDC 666.762:666.768

Reliable operation of supportive kiln furniture used in firing various ceramic materials is provided both by improving the design and by creating special structures of refractory materials used in their manufacture [1, 2].

Refractory concretes containing various bonds [3, 4] are now important in manufacturing kiln furniture for lining furnaces producing building materials. However, the use of these refractory concretes is limited because of the reduction in strength in certain temperature ranges due to the chemical destruction of the bonds.

In recent years some success has been achieved in ceramics and refractories technology owing to the use of highly concentrated ceramic bonds obtained with a special method and whose setting and strengthening are mainly due to polycondensation effects [5]. The strength of the material is determined by the composition of the dispersion medium, the state of the surface, and the fineness of the particles of solid phase, their packing density in the materials, and the strengthening conditions and cycles.

The aim of the present investigation was to develop (using the main findings of the authors of [6]) compositions and procedures for mechanically activated ceramic bonds (MACB) for unfired refractory bodies - the basis for effective linings of tunnel kiln cars used in the building materials industry.

MACB were prepared by using chamotte scrap grades ShB and ShV of the general purpose type (GOST 380-83) and orthophosphoric acid (GOST 10678-63). The scrap had the following chemical composition (% here and subsequently mass contents are shown): 67.0 SiO<sub>2</sub>, 28.0 Al<sub>2</sub>O<sub>3</sub>, 1.9 TiO<sub>2</sub>, 1.1 Fe<sub>2</sub>O<sub>3</sub>, 0.75 RO, 0.8 R<sub>2</sub>O, and the grain size corresponded to fractions 2-0.5 mm.

MACB were obtained by wet milling in a ballmill (8-liter capacity) with a porcelain frame and grinding bodies of uralit. The dispersion medium consisted of a solution of orthophosphoric acid of density 1.11 g/cm<sup>3</sup>. The ratio (mass) of materials: balls: dispersion medium in all experiments was 4:6:1. Specimens of ceramic bonds were formed by slip casting in plastic molds. After completion of the structure forming, the molds were opened, and the specimens heated in a drying cupboard at 300°C for 2 h.

The parameters for the mechanical-chemical treatment of chamotte scrap are shown in Table 1. The optimum process time is 5 h. The suspensions are thixotropic and have a minimum viscosity (74 Pa·sec) and the castings have the maximum density (1.96 g/cm<sup>3</sup>) and strengths (20 N/mm<sup>2</sup>), with a minimum open porosity equal to 19%.

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Belorussian Institute of Building Materials. Translated from Steklo i Keramika, No. 4, pp. 20-21, April, 1993.