



Porous Ceramics Obtained with the Use of Aluminum Hydroxide Powder

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ABSTRACT

This work was aimed at studying the influence of structure and properties of powders of aluminum hydroxide and zirconium dioxide on the structure and properties of the obtained ceramics. The work studied Al_2O_3 ceramics obtained from aluminum hydroxide and composite materials ZrO_2 - Al_2O_3 obtained from mixtures of zirconium dioxide and aluminum hydroxide powders. The work shows the data about the structure and properties of the initial powders used for the production of ceramics, as well as data about the structure and properties of Al_2O_3 and ZrO_2 - Al_2O_3 ceramic materials. It has been found that increasing aluminum hydroxide content in the initial mixture with the zirconium dioxide powder leads to increasing porosity of ZrO_2 - Al_2O_3 composites sintered within the temperature range between 1,400 and 1,650 °C. It has been shown that increasing sintering temperature of the Al_2O_3 ceramics leads to increasing compressive strength from 6 MPa to 800 MPa at the temperature of sintering between 1,300 and 1,500 °C, respectively.

Key words: Alumina, hydroxide aluminum, zirconium dioxide, strength, porosity, composite, properties.

INTRODUCTION

Among the variety of modern materials, ceramics takes a special place due to its physical and chemical properties, such as high corrosion resistance, temperature resistance, resistance to

radiation treatment, and biological compatibility¹⁻⁵. Materials of aluminum oxide and composites based on it⁶⁻⁹ have the greatest ability to preserve structure and properties when exposed to aggressive environments without degradation of properties, corrosion and chemical resistance, high strength.

Al_2O_3 and $\text{ZrO}_2\text{-Al}_2\text{O}_3$ ceramics with predefined porosity, size and shape of the pores, properly complies with the requirements to catalyst carriers, filters and bioimplants⁹⁻¹². To date, there are many technological approaches that ensure necessary porosity in ceramics, among which the most common is the introduction of organic pore-forming additives. The main disadvantage of this method is the presence of the products of pore-forming agent blowing in the obtained porous material, namely, carbon and its compounds, which is unacceptable for medical supplies and some catalyst carriers. The method based on the use of hydroxides makes it possible to avoid the presence of impurities [13]. Analysis of literature shows that currently there is a lack of systematic studies of porous ceramics obtained with the use of hydroxides. This work was

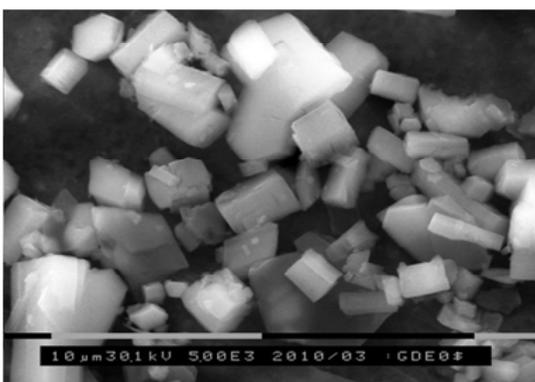


Fig. 1: SEM image of the aluminum hydroxide powder

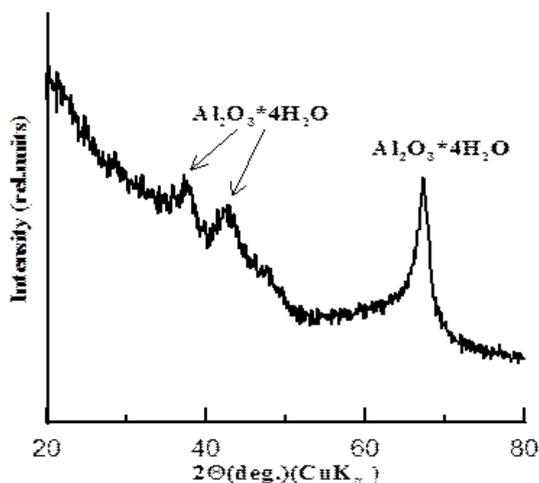


Fig. 2: X-Ray photograph of the aluminum hydroxide powder

aimed at studying the influence of structure and properties of powders of aluminum hydroxide and zirconium dioxide on the structure and properties of the obtained ceramics.

MATERIALS AND METHODS

For obtaining porous Al_2O_3 ceramics, powder of hydrated aluminum oxide obtained by decomposing aluminate solution was used as the original component. For obtaining $\text{ZrO}_2\text{-Al}_2\text{O}_3$ porous composite materials, a mixture of powder of zirconium dioxide obtained by chemical deposition of salts and hydrated aluminum oxide was used. The volume fraction of aluminum hydroxide mixed with zirconium dioxide was 1, 5, 10, 25, 50%. The powders were pressed in a steel mold at pressure up to 200 MPa. The change in porosity of ceramics was ensured by varying the sintering temperature of the samples. The press-works of aluminum hydroxide were sintered in the open air at temperatures of 1,300, 1,400, 1,500 °C with isothermal exposure for 1 hour, press-works from a mixture of powders of zirconium dioxide and aluminum hydroxide were sintered at temperatures of 1,400, 1,500, 1,550, 1,600, 1,650 °C, in the same conditions.

X-ray studies were performed with the use of diffractometer (CuK α radiation, $\lambda = 1.54060 \text{ \AA}$, room temperature) in step mode ($\Delta\theta = 0.1$, exposure 5 seconds), in the angle range 2θ from 20 to 80. The size of the areas of x-rays coherent scattering (CSA) was measured at small diffraction angles. The phase was identified by comparing the peaks of the x-ray photographs with the catalog of the American Society

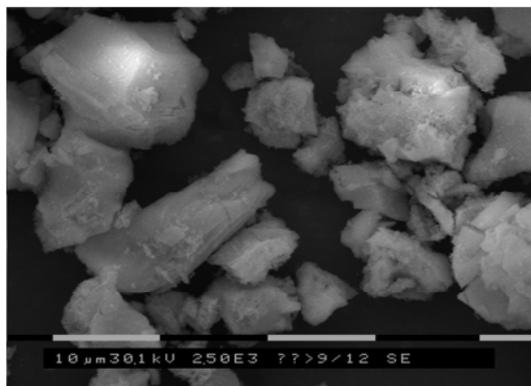


Fig. 3: SEM image of $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ powder

of Testing Materials (ASTM). The content zirconium phases was assessed by the ratio of the integrated intensity of lines I (111) of the tetragonal (T) phase and I (111), I (111) of the monoclinic (M) phase.

Powder morphology and the structure of the sintered ceramics were studied with the use of a scanning electron microscope Philips SEM 515. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed with the use of a thermoanalyzer in a platinum crucible in an inert atmosphere at temperatures between 20 and 1,300 °C. Ceramics samples were compression-tested at the “INSTRON – 1185” test installation with the loading rate of 0.2 mm/s.

RESULTS

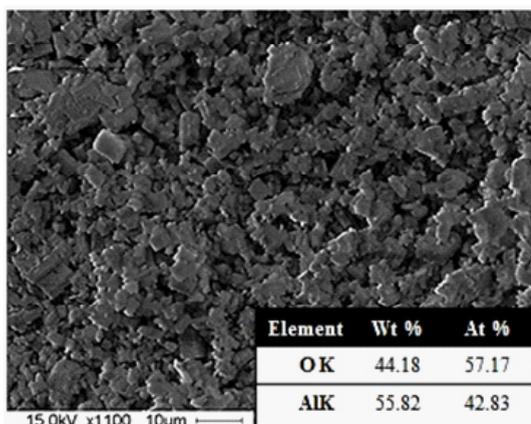
Powders

Aluminum hydroxide powder consisted of isolated polycrystalline particles with the average size of 2.6 μm with the rms (root-mean-square)

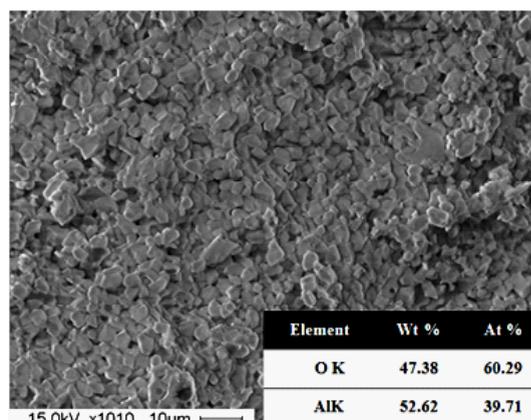
deviation of 1.5 μm, Figure 1. In distribution of particle by size, only one maximum was present, whereby the predominant number of particles had the size between 0.5 and 4 μm, also single particles up to 8 μm were present. The value of specific surface area of the aluminum hydroxide powder was 173 m²/g.

X-ray diffraction study showed that the aluminum hydroxide powder was represented by structural modification of Al₂O₃·4H₂O in the amorphous state, as evidenced by the high background at low angles of diffraction, Figure 2.

Thermal analysis showed that powder heating was accompanied by a mass loss occurring in two stages at different temperatures: 25 – 550 °C, 800 – 1,150 °C. Mass loss was computed by the method of thermogravimetry, and amounted to Δm = 17.6 %. Two endothermic reflexes are present on powder DSC curve: one in the temperature range between 20 and 380 °C, the second in



SEM image, elemental analysis of ceramic sintered at 1,300 °C.



SEM image, elemental analysis of ceramic sintered at 1,500 °C.

Fig. 4: A SEM image of Al₂O₃ ceramic sintered a - at 1,300 °C, b - at 1,500 °C

Table 1 :

	1,300 °C	1,400 °C	1,500 °C
The average particle size (<d>)	<d> = 3.1	<d> = 4.4	<d> = 5.1
and the standard rms deviation (σ), μm	σ = 1.3	σ = 1.6	σ = 2.5
The average pores size (<d>)	<d> = 2.4	<d> = 2.2	<d> = 1.6
and the standard rms deviation (σ), μm	σ = 3.4	σ = 0.9	σ = 1.1
Porosity, %	60	45	20

the temperature range between 800 and 1,100 °C, and the exothermic reflex in the temperature range between 1,100 and 1,300 °C. It should be noted that the temperature intervals of the sections that correspond to decreasing powder mass are consistent with the position of the endothermic reflexes on the DSC curve.

ZrO₂(Y₂O₃) powder was a solid solution of composition 97 mole % of ZrO₂ - 3 mole % of Y₂O₃ obtained by chemical vapor deposition. The powder consisted of dense polycrystalline agglomerates, Figure 3. According to the data obtained, type of agglomerates distribution by size is unimodal, the prevailing amount of agglomerates had a size between 1 and 12 μm, but agglomerates were also present with the size that reached 80 μm. The average size of the agglomerates was 12 μm; the average size of particles in the agglomerates was 0.2 μm. The specific surface of the powder amounted to 7.6 m²/g. X-ray phase analysis showed the presence of the tetragonal and monoclinic phases of zirconium dioxide powder. The content of phases was 65 and 35%, respectively. The average size of CSA of the tetragonal phase was 20 nm, and that of the monoclinic phase – 40 nm. The value of crystalline lattice micro distortion was $2.374 \cdot 10^{-3}$, lattice parameter $c = 5.1849 \text{ \AA}$, $a = 5.0908 \text{ \AA}$.

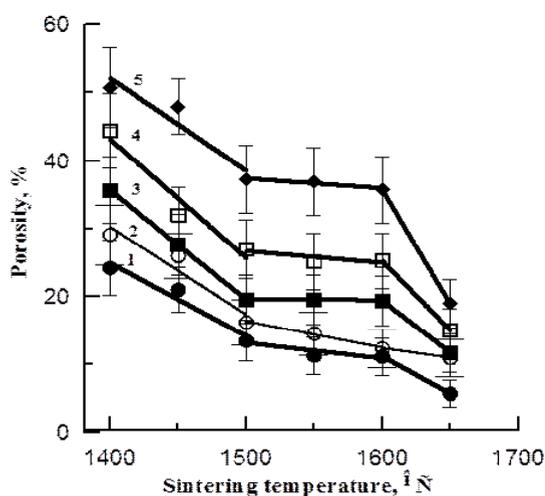


Fig. 5: Dependency of porosity on the sintering temperature of ZrO₂-Al₂O₃ composites. The dependencies correspond to the content of aluminum hydroxide in the initial material 1 - 1%, 2- 5%, 3- 10%, 4- 25 %, 5 -50%

Al₂O₃ ceramics from aluminum hydroxide

Phase analysis of alumina ceramics sintered at 1,300 °C and above is represented by the high-temperature a modification - Al₂O₃. Ceramics sintered at 1,300°C is represented by grains of two types: those of irregular shape and those close to spherical shape. The average size of grains was to 3.1 μm with the average squared deviation of 1.3 μm. As the sintering temperature increased, the size of aluminum oxide grains increased, too. In ceramics sintered at 1,400 and 1,500 °C, the grains of aluminum oxide mostly had spherical shape. Besides, the structure of the ceramics had interparticle porosity, which decreased with increasing sintering temperature. In the ceramics studied, total porosity (\bar{E}) was 60% at the temperature of sintering of 1,300 °C, $\bar{E} \approx 45\%$ at 1,400 °C, and exceeded 20% for samples sintered at 1,500 °C. The shrink ratio increased from 7% to 30% with the sintering temperature increasing from 1,300 to 1,500 °C, respectively. The generalized data about the average size of pores and grains in Al₂O₃ ceramics are shown in Table 1. A SEM image of polished surface and elementary analysis of the obtained ceramics are shown in Figure 4.

Studying the mechanical properties of the obtained ceramic samples showed that with increasing the temperature of sintering, there is a significant increase in compressive strength from 6 MPa at 1,300 °C to 800 MPa at 1,500 °C, respectively. Increasing mechanical properties is obviously related

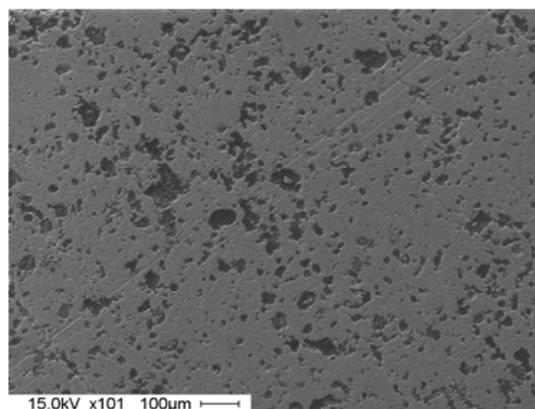


Fig. 6: A SEM image of polished surface of ZrO₂ - Al₂O₃ composites. The composites were obtained from a mixture of 50 % of ZrO₂(Y₂O₃) and 50% of Al₂O₃*4H₂O, the temperature of sintering was 1,500 °C

to reduction of porosity of sintered ceramics, and, consequently to density increase.

ZrO₂-Al₂O₃ composite materials

Figure 5 shows the dependence of porosity in ZrO₂ - Al₂O₃ composite materials on the temperature of their sintering. The figure shows that regardless of the initial powder mixture composition, increasing sintering temperature was accompanied by a decrease in the pore volume in the composites. The increase in the volume fraction of aluminum hydroxide in powder mixtures was accompanied by an increase in porosity in the resulting composites. Whereby, in the composites sintered in the temperature range between 1,500 and 1,600 °C, the volume fraction of pores was almost unchanged. Analysis of pores' average size in ZrO₂ - Al₂O₃ composites sintered at 1,400 °C, depending on the volume fraction of aluminum hydroxide in the initial powder mixture, showed that with increasing the volume fraction of aluminum hydroxide, the average pore size increased from 7 to 18 μm, with increasing the volume fraction of aluminum hydroxide in the initial material from 1% to 50%, respectively. A SEM image of the polished surface of ZrO₂ - Al₂O₃ ceramics is shown in Figure 6.

According to x-ray studies, the phase composition of the obtained ZrO₂ - Al₂O₃ composites is represented by tetragonal and monoclinic modifications of ZrO₂ and high-temperature modification α - Al₂O₃. With that, there was a difference in the shares of the high-temperature tetragonal and the low-temperature monoclinic modifications of zirconium dioxide, depending on the ratio of zirconium oxide and aluminum hydroxide in the powder mix and the composites sintering temperature. According to the obtained data, regardless of the amount of aluminum hydroxide in the initial mix, increasing sintering temperature was accompanied by the reduction in the share of tetragonal phase of zirconium dioxide in ZrO₂ - Al₂O₃ porous composites. With that, in the temperature range of composites sintering between 1,400 – 1,550 °C, the share of tetragonal phase of zirconium dioxide decreased on the average from 70 to 25% and remained unchanged with increasing the temperature of sintering to 1,650 °C.

Analysis of the influence of CSA size of the tetragonal phase of zirconium dioxide on the porosity of the composites showed that with decreasing the size of CSA of the tetragonal phase, the porosity of the composites decreases. With extrapolation of the approximating function on the non-porous state, the CSA size of the tetragonal phase of zirconium dioxide in the composite is 65 nm. The composites with 7 % porosity had the greatest tensile strength of 1,100 MPa.

DISCUSSION

The presence of the first endothermic reflex on the calorimetric curve of aluminum hydroxide powder is associated with removing moisture adsorbed on the particles surface and structured γ groups, the second endothermic reflex corresponds to transformation of hydrated aluminum oxide into the low-temperature γ - Al₂O₃. The presence of the exothermic reflex on the DSC curve is associated with structural transformation of the low-temperature modification of aluminum oxide into the high-temperature modification α of Al₂O₃. The x-ray photograph of aluminum hydroxide powder subjected to annealing at 1,300 °C had only the reflexes of the high-temperature α modification - Al₂O₃.

Work¹⁴ describes the nature of zirconium dioxide and its solid solutions. The authors of this work report that from the practical point of view interesting are only the solid solutions of various oxides based on ZrO₂, or mechanical mixture with a rigid matrix that are capable of stabilizing the high-temperature phases of ZrO₂ at low temperature. At room temperature, ZrO₂ is in the monoclinic phase, and when heated, it experiences phase transformations: m (monoclinic modification) - ZrO₂ → (1,197 °C) → t (tetragonal) ZrO₂ → (2,300 °C) → c (cubic) ZrO₂ → (2,600 °C) → melting. Transition t - ZrO₂ → c - ZrO₂ has diffusive nature and plays a very important role in manufacturing the so-called partially stabilized zirconium dioxide. Transformation m - ZrO₂ → t - ZrO₂ proceeds by a martensitic mechanism, and is accompanied by volumetric change by 5 – 9 %. Such a significant material expansion in course of cooling is accompanied by cracking, and makes it impossible to obtain compact products of pure ZrO₂. It has been found that ZrO₂ can form solid solutions of substitution type with several two-,

three - and tetravalent oxides. The common and very important for the ceramic technology feature of these solid solutions is the absence of reversible polymorphic transformations of type $m - \text{ZrO}_2 \rightarrow t - \text{ZrO}_2$ transition in pure ZrO_2 . Although the formed solid solutions are not thermodynamically balanced at low temperatures, in practice they can exist at these temperatures without disintegrating. Therefore, ZrO_2 exists only at very high temperatures, due to significant thermal vibrations of the crystalline lattice. When the temperatures decreases, the diffusion transition $c - \text{ZrO}_2 \rightarrow t - \text{ZrO}_2$ occurs. Tetragonal ZrO_2 , like cubic ZrO_2 has the fluorite type structure. With further decrease in temperature, when the diffusion is practically finished, and the tightness of oxygen ions increases, martensitic transformation of $t - \text{ZrO}_2 \rightarrow m - \text{ZrO}_2$ occurs. Expansion of the oxygen position that promotes creation of stable fluorite structure in a wide range of temperatures can be achieved either by partial substitution of ZrO_2^{4+} ions with the ions of larger radius, or by creating vacancies in the anion sublattice by replacing ZrO_2^{4+} with ions of lower valence. Small size of the ion radius is less favorable for stability of these solid solutions. In addition to formation of solid solutions based on ZrO_2 , another type of stabilization of high temperature modification of $t - \text{ZrO}_2$ is used in the production of high-strength ceramics. After sintering the mechanical mixture of $\text{Al}_2\text{O}_3 - \text{ZrO}_2$ with volumetric content of ZrO_2 of less than 20%, a rigid corundum matrix is formed that holds the dispersed inclusions of $t - \text{ZrO}_2$. This is explained by higher elasticity modulus of corundum and lower thermal expansion, as compared to ZrO_2 . Therefore, $t - \text{ZrO}_2$ particles are in the field compressive stress, and remain stable after the sintered material is cooled. The $t - \text{ZrO}_2$ particles stability also depends on their size: unalloyed grains $t - \text{ZrO}_2$ in the corundum matrix are transformed into $m - \text{ZrO}_2$ at 20 °C if their size exceeds 0.6 μm . Coarser grains are transformed at higher temperatures, as compared to finer ones. The possibility of obtaining high-strength zirconium ceramic is related to the discovery of the so-called transformational hardening due to controlled phase transition $t - \text{ZrO}_2 \rightarrow m - \text{ZrO}_2$. The essence of this effect stems from the phenomenon of ZrO_2 polymorphism, high-temperature $t -$ and $c -$ phases' ability to stabilize, and the martensitic $t \rightarrow m$ transition. Practically, the effect of transformation hardening is implemented in the case where the sintered material is composed of $t - \text{ZrO}_2$ particles that can transform into $m - \text{ZrO}_2$. The

cracks occurring under loading spread until $t - \text{ZrO}_2$ occur in their front. Such a particle in a compressed state (corundum matrix), or coherently bound with the matrix (with the predominance of $c - \text{ZrO}_2$ in the material) is resistive to the $t \rightarrow m$ transition even at low temperatures. After getting into the stress field in the top of a propagating crack, the particle gains energy sufficient for transformation. Thus, the energy of the propagating cracks is transferred to the energy of the $t \rightarrow m$ transition, and the catastrophic growth of the crack stops.

From the data in work¹⁴ it follows that the important property of the $\text{ZrO}_2 - \text{Al}_2\text{O}_3$ composites is the amount of tetragonal phase of zirconium dioxide. One of the factors that influenced the proportion of tetragonal and monoclinic modification of zirconium dioxide in obtained $\text{ZrO}_2 - \text{Al}_2\text{O}_3$ composites could be the individual grains of the tetragonal phase of zirconium dioxide that reached the "critical" size. It is known that for the tetragonal phase of ZrO_2 there is the concept of the "critical" grain size, upon reaching of which, a spontaneous tetragonal-monoclinic transformation occurs. The value of the "critical" grain size, according to the data¹⁵⁻¹⁷ depends on chemical composition of the solid solution, and is 1.2 μm for the $\text{ZrO}_2 - \text{Y}_2\text{O}_3$ system. However, work¹⁷ shows that the value of the "critical" grain size of the tetragonal modification in ceramics decreases with increasing porosity, and is only 0.2 microns at 30%. This is due to the decreasing level of elastic stresses generated by adjacent grains and constraining tetragonal-monoclinic transformation that occurs with the unit cell volume increase. For grains with a limited number of neighbors in case of large porosity, the probability of transition from the high temperature tetragonal modification of zirconium dioxide into the monoclinic low-temperature modification during cooling from the sintering temperature is high. Apparently, increasing the sintering temperature of the $\text{ZrO}_2 - \text{Al}_2\text{O}_3$ composites obtained in the work was accompanied by grain growth, and achieving the "critical" size of ZrO_2 tetragonal phase - by individual grains.

In addition to the sintering temperature and ZrO_2 grain size, the ratio of high-temperature and low-temperature modifications of ZrO_2 in the $\text{ZrO}_2 - \text{Al}_2\text{O}_3$ composites is, apparently, could be influenced by the presence of Al_2O_3 . As shown above, for ZrO_2 composites with the content of aluminum

hydroxide in the initial mixture equal to 25 and 50 %, which corresponds to Al_2O_3 content in sintered ceramics of 21 % and 42%, respectively, the amount of tetragonal phase of zirconium dioxide decreased sharply at the sintering temperatures of over 1,500 °C. This phenomenon could also be caused by formation of the $\text{Y}_3\text{Al}_5\text{O}_{12}$ chemical compound, named yttrium-aluminum garnet. Work ¹⁸ shows that, regardless of the composition of the mixture, the interaction of yttrium oxide and aluminum oxide starts at the temperature of 900 – 950 °C, with formation of the $\text{Y}_4\text{Al}_2\text{O}_9$ compound, at 1,100 °C, formation of YAlO_3 is observed, and starting with the temperature of 1,300 °C, yttrium-aluminum garnet $\text{Y}_3\text{Al}_5\text{O}_{12}$ is crystallized. Thus, in a Y_2O_3 - Al_2O_3 system, at temperatures above 1,300 °C, regardless of the mixture composition, it is possible to form the stable $\text{Y}_3\text{Al}_5\text{O}_{12}$ compound [19]. Since the stabilized zirconium dioxides are solid substitution solutions, the process of destabilization occurs by disintegration of solid solutions into oxide systems that are components thereof. The products of disintegration are a monoclinic phase of zirconium oxide, the oxide of the stabilizing element, and the intermediate oxide compound of zirconium dioxide and the stabilizing element. Formation of yttrium-aluminum garnet in the ZrO_2 - Al_2O_3 composite materials could, apparently, lead to destabilization of the high temperature crystalline modification of zirconium dioxide. However, in the x-ray photographs of ZrO_2 - Al_2O_3 composites, reflexes belonging to composites could not be detected due to low content of yttrium oxide in the system.

In course of extrapolation onto a dense state, the maximum compression strength of the composites is 1,300 - 2,400 MPa; these values are consistent with the data shown in^{20,21}.

CONCLUSION

Thus, in course of the study, the structure and properties of porous Al_2O_3 and ZrO_2 - Al_2O_3 ceramics obtained with the use of aluminum hydroxide have been studied. The required porosity value in Al_2O_3 ceramics was achieved by varying the temperature of samples sintering. It has been found that increasing aluminum hydroxide content in the initial mixture with the zirconium dioxide powder leads to increasing porosity of ZrO_2 - Al_2O_3 composites sintered within the temperature range between 1,400 and 1,650 °C. It has been shown that the structure of Al_2O_3 ceramics had interparticle porosity, which decreased with increasing the temperature of sintering, wherewith the limit of the compressive strength increased with increasing the temperature of sintering. It was found that non-porous state of composite ZrO_2 - Al_2O_3 materials is achieved when the size of crystallite is 65 nm. In subsequent works, it is planned to continue studying porous ceramics obtained by the use of aluminum hydroxide powders of various modifications. It is planned to study properties like thermal conductivity and thermal-expansion coefficient in the obtained porous ceramics.

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