Ceramics with a hierarchical porous structure based on aluminum oxide

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Abstract. It have been studied porous ceramic Al₂O₃ obtained via decomposition of aluminum hydroxide. This method allows obtaining ceramics without any impurities on the inner surfaces of porous material. Initial powders were subjected to mechanical treatment in a planetary ball mill for 48 hours and next cold molding samples were sintered in air at 1450-1650 °C for a one hour. It was shown that the porosity of the ceramic increases with increasing content of aluminum hydroxide in the powder mixture and was equal 64% for samples contains 100% Al(OH)₃ sintered at 1450 °C. It was found that the compressive strength increases with increasing sintering temperature and concentration of aluminum hydroxide affects on the compaction rate of ceramic samples. The maximum strength of these ceramics was 180 MPa for samples without of hydroxide. It has been estimate that activation energy of the sintering process was 40 ± 5 kJ / mol.

1. Introduction
It is well known, what ceramics has a special place among the materials. This is due to a wide range of applications and high characteristics of ceramics. Oxide ceramics due to its corrosion resistance, the ability to work at high temperatures, high physics-mechanical characteristics has found its application in many areas of industry Porous ceramics can be used in the manufacture of filter elements, in catalytic carriers, for fine gas purification. The main characteristics of these ceramics are porosity and its morphology [1-3].

There are many methods of pore formation in ceramic materials one of which is a method based on decomposition of aluminum hydroxide to alumina and H₂O accompanied by gassing and as a consequence, the formation of the required porous structure. In this case, a different concentration of hydroxide in the powder mixture will affect the porosity in the sample. This method allows forming a multi-level pore structure without blowing agent method [4-6].

The aim of this work was to study the influence of the concentration aluminum hydroxide in hydroxide-oxide mixture and sintering temperature on formation of the pore structure in the alumina and its mechanical properties of sintered ceramic.

2. Materials and Experimental Procedure
Gibbsite modification of aluminum hydroxide powder and aluminum oxide powder obtained by heat treatment aluminum hydroxide powder were used as initial components. The volume change of the pore space in the ceramic was provided by varying the sintering temperature and the concentration of aluminum hydroxide in a mixture.

Alumina powder was obtained by annealing the aluminum hydroxide powder at a temperature of 1100 °C 1h. The alumina powder and aluminum hydroxide powder were mechanically activated in a...
ball mill for 48 hours. After mechanical activation alumina powder was mixed with aluminum hydroxide powder (in concentrations of 0-100%) in a ball mill during 10 h. The mixture was cold pressed at 200 MPa. Sintering of compacts was carried out in air at temperatures 1450 - 1650 °C. Compression tests of sintered samples were carried out on a universal test machine at a strain rate 10^{-2} \text{c}^{-1}.

3. Results and Discussion

SEM images of powders and distribution of particle size are shown on Figure 1. As one can see the particle shape is nearly spherical. The average particle size was 50.3 µm; standard deviation was 22.7 µm.

![Figure 1.](image1.png)

Figure 1. (a) SEM-image of alumina powder after annealing, (b) SEM-image of aluminum hydroxide powder and (c) its particle size distribution before mechanical activation.

SEM image of the powder mixture from a mechanically activated aluminum hydroxide and aluminum oxide in Fig. 2. Mechanical activation made it possible to break up particles with $<D> = 50.3 \text{ µm}$ to $<D> = 2.2 \text{ µm}$ (powdered alumina and aluminum hydroxide).

![Figure 2.](image2.png)

Figure 2. SEM image of the powder mixture from a mechanically activated aluminum hydroxide and aluminum oxide.

SEM images of the fracture surface of ceramics synthesized at various sintering temperatures are shown on Fig. 3. One can see that the particles of ceramics sintered at 1450 °C have a fractured shape, while the particles of the ceramic sintered at 1650 °C have a rounded shape.
Figure 3. SEM images of fracture surface of porous ceramic samples obtained from alumina powder with addition of a hydroxide powder at different sintering temperatures: (a) 5% Al(OH)$_3$, 1450 °C, (b) 75% Al(OH)$_3$, 1650 °C.

Figure 4. Dependence of porosity from sintering temperature with different concentrations of Al(OH)$_3$.

Figure 5. Dependence of compressive strength from sintering temperature with different concentrations of Al(OH)$_3$.

The dependence of porosity vs. sintering temperature is shown in Fig.4. With increasing sintering temperature, the porosity of ceramic samples decreases. Aluminum hydroxide affects the porosity dependence on the sintering temperature. Decreasing the aluminum hydroxide amount in the powder mixture, the greater is an effect of sintering temperature on the porosity. One can see that the effect of aluminum hydroxide on porosity at a sintering temperature of 1450 °C is lower in too time of aluminum hydroxide on the porosity at a sintering temperature of 1650 °C.

When the sintering temperature increases, the pore volume in the ceramic samples decreases, and porosity in Al(OH)$_3$ ceramics decreases less than in case of ceramics made of pure alumina. The increase of Al(OH)$_3$ content leads to more noticeable decrease in pore volume with increasing sintering temperature from 1450 to 1650 °C. Density of ceramic after sintering at 1450 °C varies slightly and is equal to 2.2± 0.2 g/cm$^3$. After sintering at higher temperatures, an increase in density is observed. The highest density 3.3 g/cm$^3$ was obtained for samples sintered at 1650 °C. The dependence of the sample density on the sintering temperature allowed to estimate the activation energy of the compaction process during sintering, Q = 45 ± 5 kJ / mol, which corresponds to the literature data for highly disperse powders [7, 8] and corresponds to surface diffusion.
Figure 5 shows the dependence of compressive strength on sintering temperature for ceramics with different aluminum hydroxide content. One can see that the compressive strength increases with increasing sintering temperature, while the tensile strength differs for ceramics with different aluminum hydroxide content sintered at the same temperature. The ultimate strength of ceramics without aluminum hydroxide is maximal and increases almost 3 times with sintering temperature increasing.

Based on the data obtained, one can conclude that with increasing sintering temperature the porosity decreases. This is due to the fact that during the sintering, the formation and growth of contacts between particles, this means a decrease in the pore size, which is accompanied by an increase in their size.

Dependence of porosity of materials based on alumina and aluminum hydroxide powders at different sintering temperatures determine the possibility of synthesizing strength ceramic materials with a porosity of 30-50% changing the corresponding technological parameters.

4. Conclusion
It was found that the decomposition of aluminum hydroxide during sintering leads to the formation of porosity in the ceramic. It was shown that with increasing sintering temperature density and shrinkage also increase. It was shown that the porosity in ceramic samples increases with the content of aluminum hydroxide in the powder mixture. It has been found that the compressive strength increases with increasing sintering temperature and the concentration of aluminum hydroxide affects on the compaction rate of ceramic samples. It was shown that the porosity of the ceramic samples increases with increasing content of aluminum hydroxide in the powder mixture and was equal 64% for samples contains 100% Al(OH)₃ sintered at 1450 °C. The maximum strength of these ceramics was 180 MPa for samples without of hydroxide. The average activation energy of the sintering process is 40±5 kJ/mol.

Acknowledgement
The work presented was carried out in accordance with Tomsk State University’s competitiveness program and with partial support of project 14.584.21.0026 (RFMEFI58417X0026).

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