

Properties of oxide-hydroxide sintered ceramics

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Abstract. In this paper the study of porous ceramics obtained from aluminum hydroxide with gibbsite modification is presented. It was shown that aluminum hydroxide may be used for pore formation and pore volume in the sintered ceramics can be controlled by varying the aluminum hydroxide concentration and sintering temperature. It was shown that compressive strength of alumina ceramics increases by 40 times with decreasing the pore volume from 65 to 15%. Based on these results one can conclude that the obtained structure is very close to inorganic bone matrix and can be used as promising material for bone implants production.

1. Introduction

The development of material based on porous oxides with high strength is a fundamental challenge to modern materials [1-5]. Ceramics with a porous structure can be used to produce filters, catalyst supports, membranes etc. [6]. Porous ceramics is also interesting for medical applications due to its properties are close to bone tissue.

The traditional method of creating highly porous materials is based on “burn-pore-forming” additives [7]. On the one hand this method is effective in terms of management of the pore volume, but on the other hand the possible formation of carbon on the inner surfaces of pore is unacceptable for the manufacture of bio-implants and can lead to rejection of the implant [8]. To form a multi-level pore structure without addition of pore-former one can use the method of component decomposition, for example, the hydroxides to oxides, accompanied by the release of gas and, as a consequence, the formation of porosity [9]. This method based on the decomposition of aluminum hydroxide to the oxide has been known for a long time [10-12], but there is small amount data on the regulation of the pore space volume and the dependence of the pore space formation sintered at different temperatures, particle size of initial powders, as well as no data about dependences of the strength of the ceramic with changing of porosity [13].

The aim of this work is to study formation of porous structures in ceramic synthesized from aluminium hydroxide due to decomposition of hydroxide to the oxide.

2. Materials and experimental procedure

Aluminum hydroxide with gibbsite modification $\text{Al}_2\text{O}_3 \cdot 4\text{H}_2\text{O}$ obtained by the decomposition of the aluminates solution was used as a starting component. The average particle size of the powder was 2.6 μm . Cylindrical form samples were pressed at 20 kN load, sintered in air at 1300, 1400 and 1500°C, with isothermal one hour holding at these temperatures. The structure, morphology, pore space, grain size and phase composition were investigated. The volume of the pore space was estimated by the



formula $\theta = (\rho_{th} - \rho_e) / \rho_k * 100 \%$, where ρ_{th} the theoretical density material, ρ_e experimental density of the material. The pore structure of ceramics and morphology of the powder was investigated using scanning electron microscope (SEM) Philips-SEM 515 and optical microscope. The mean pore size of ceramics and the powder particles were calculated by the secant method [10].

X-ray diffraction studies were carried out on X-ray diffractometer with filtered $CuK\alpha$ radiation. Diffraction spectra were obtained in the range of angles $2\theta = 20 - 80$ degrees with a step $\Delta(2\theta)=0.05$, the exposure time was selected to achieve accuracy better when 0.5%. Compression testing of ceramics samples was carried out on universal test machine “Devotrans” with strain rate $2*10^{-4} s^{-1}$ [9].

3. Results and Discussion

On the XRD pattern of aluminum oxide samples peaks have a large width and only three peaks are clearly visible. Their angular positions correspond to the strongest reflections of the hydroxide of aluminum oxide of the $Al_2O_3*4H_2O$ composition [14-16]. X-ray peaks broadening could be caused by a small size of the coherent diffracting domain (CDD), or concentration in homogeneity by hydration degree of Al_2O_3 powder. These factors affect the specific surface - the specific surface of the powder is $173 m^2/g$, the similar data were obtained in [17, 18].

According to the EDAX analysis the samples obtained contain $59,1 \pm 2,3\%$ (at.) of oxygen and $40,9 \pm 1,3\%$ (at.) aluminum, which corresponds to the chemical formula of aluminum oxide. No impurities in ceramics were detected.

The structure of sintered ceramics is presented in Fig. 1. As one can see grains of aluminum oxide have irregular shape and all samples are characterized by the presence of interparticle porosity regardless the sintering temperature. It was also observed that the increasing sintering temperature leads to a decrease in the interparticle pore size. The pore sizes for samples with increasing sintering temperature are decreasing (Figure 2(a,b,c)).

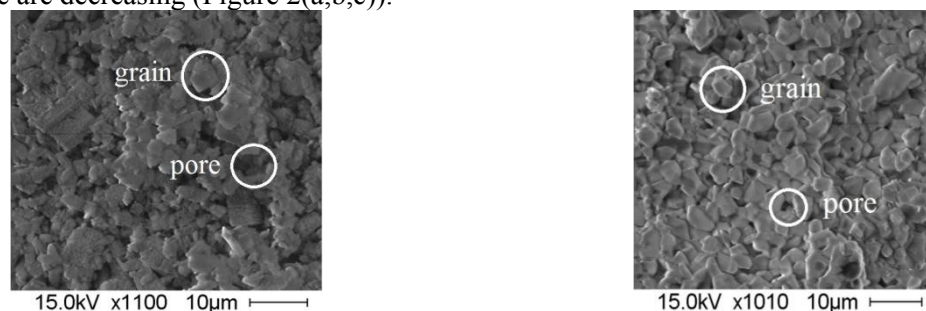


Figure 1. Ceramic structure after sintering at 1300°C and 1500°C

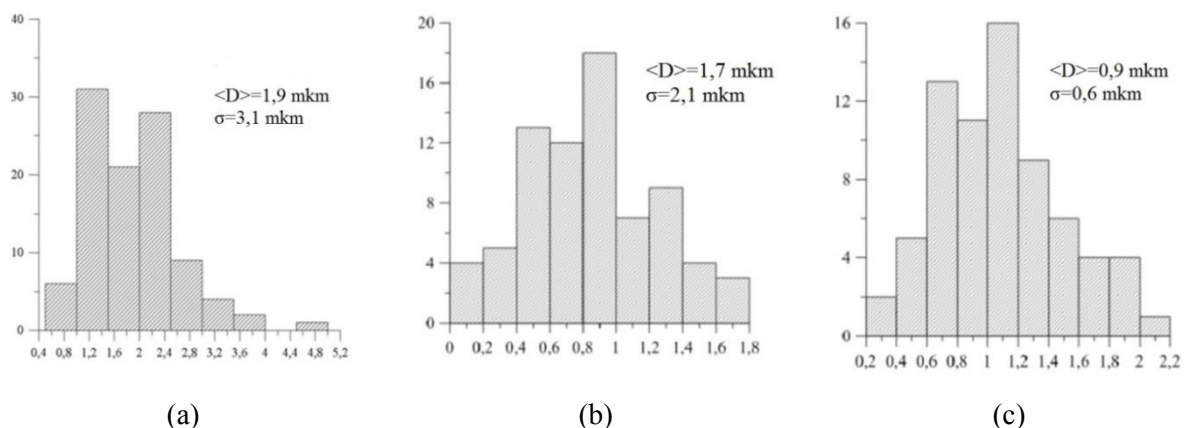


Figure 2. Pore size distribution, the sintered ceramics at the temperatures: (a) 1300°C, (b) 1400°C, (c) 1500°C

In Figure 3(a) the results of shrinkage and porosity of the samples are shown. Shrinkage has a linear dependence on sintering temperature. Ceramics sintered at 1300 °C demonstrates open porosity and at 1500 °C porosity is isolated.

The results of mechanical testing are shown in Figure 3(b). One can observe a significant increase in the compressive strength with increasing sintering temperature associated with the decrease in the pore space volume.

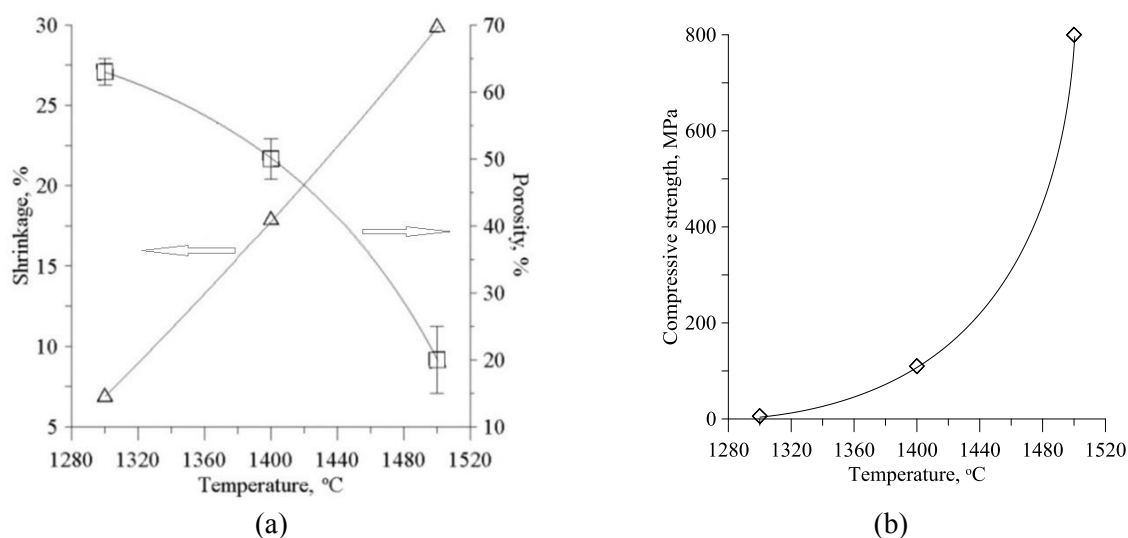


Figure 3. (a) Dependency of shrinkage porosity on sintering temperature, (b) the dependence of the strength of the sintering temperature.

4. Conclusions

In the work presented it was shown that average pore size of aluminum oxide ceramics decreases from 1,9 to 0,9 μ m with increasing sintering temperature. Shrinkage decreases from 28 to 8 % with increasing sintering temperature. It was shown that the compressive strength increases with increasing sintering temperature, minimum compression strength is 20 MPa for sintering temperature 1300°C and the maximum 800 MPa for 1500°C.

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