Development of bioceramic material for spinal surgery implants

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Abstract. Highly porous zirconia-based ceramics were prepared. The ceramic samples sintered at 1600°C had porosities from 40% to 43%, with pore size ranges as follows: "big pore" 100-220 μm and "small pore" 0.8-8 μm. This makes the ceramic structure to be very similar to the structure of the natural spinal bone. The level of mechanical properties of the synthesized zirconia-based ceramics is determined by the pore sizes. The values of the compressive strength and the effective Young's modulus are very similar to those characteristics of the natural spinal bone.

1. Introduction

Additive technologies are the most dynamically developing directions of bio-implants production [1]. Analysis of the publications as well as current trends of research in the field of spinal prosthesis showed that until recently, that the largest part of the intervertebral disc prosthesis made of metal, the main advantage is ease of manufacturing and processing [2-8].

An analysis of publications in the field of spinal prosthesis showed that until recently, the intervertebral disc prostheses have been made of metal [2-8]. However, an adverse effect of metal on the adjacent tissue and the whole organism forced searching new materials for spinal implants.

An alternative to metals in the manufacture of implants are ceramic materials. Ceramic materials are closest to the bone material in terms of biochemical compatibility.

Zirconia-based bioceramics produced from nanopowders is considered as one of the most promising materials for additive biotechnologies due to possessing a unique combination and range of properties including strength and fracture toughness, hardness and wear resistance, biocompatibility, bacteriostatic, and osteointegration [2].

The aim of this paper is to study the physical and mechanical properties of the sintered porous zirconia ceramics.

2. Experimental materials and investigation procedure

The Zr, Mg, and Al aqueous salt solutions served as the original materials for obtaining porous ceramic ceramics; the salts were converted into powders in the high-frequency plasma discharge. The powders had the composition as follows: ZrO_2 +9 mol % MgO. Samples for sintering were prepared by slip casting. The required porosity and configuration of the pores were provided by introducing in a ceramic mass a predetermined amount of organic binder and blowing agent such as granular UHMWPE powder with grain size in the range 90 to 200 μm. The sintering of the ceramic samples was performed in an air furnace at 1600°C, isothermal holding at the sintering temperature was 1, 5, 10 hours. Investigation of the structure of sintered ceramics was carried out by optical microscope "Altami M1" and scanning electron microscope "VEGA3 TESCAN". X-ray diffraction data were obtained using an X-ray diffractometer with CuK_α radiation with a step of 0.05° in the range of 2 theta angles from 20 to 80 degrees. Samples of porous ceramics were uniaxial compressed using a universal testing machine "GP DLC 30 kN". Effective Young's modulus (E) was determined as the angle of a tangent line slope to a straight-line segment of the "stress-strain" curve.

3. Results and discussion

Studying the structure of sintered ceramics showed that porosity reduced for increasing the sintering time from 1 up to 10 hours (Figure 1a). The ceramic samples had porosities with pore sizes in two categories: "big pores" around 100-220 μm and "small pores", around 0.8-8 μm (Figure 1).
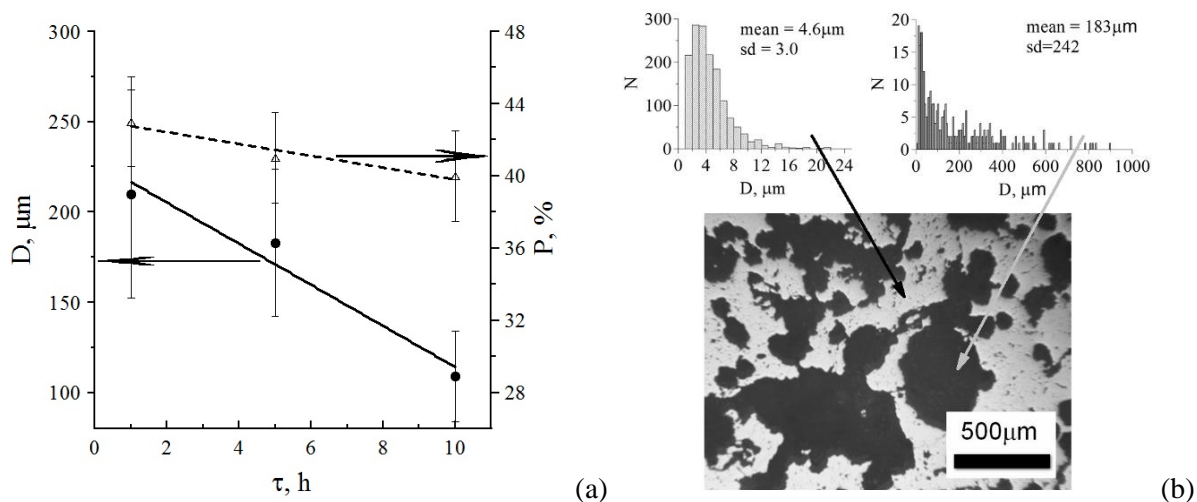

Figure 1. Dependence of porosity (P) and the average pore size (D) of the isothermal holding time (a), typical structure and distribution of pores in ceramics, sintered at a temperature of 1600°C, for 5 h (b).

The average "small pore" sizes decreased from 8.5 to 0.8 μm, while sintering time increased from 1 to 10 hours.

The average "big pores" sizes decreased from 210 to 110 μm when sintering time increased from 1 to 10 hours (Figure 1a). The formation of "Big pores" is associated with blowing agent removal during sintering of ceramics.

The fact that D the average pore sizes of the two types decreased with increasing the holding time of sintering may be associated with the densification and recrystallization ceramics during sintering.

Figure 2 shows the scanning electron microscope images of porous ceramics fracture surfaces. The fracture of porous ceramics is mainly by intercrystalline mechanism with microcracks shown by arrows in Figure 2b. The analysis of the human bone porous structure showed that it is inhomogeneous by the bone depth [5]. A spinal bone consists of cortical (compact) and trabecular (spongy) substances. They both form a lamellar bone tissue. The pore size is depending on the area of bone and ranges from

0.5 to 500 μm. Comparative studies of the structure of the porous ceramic materials and human trabecular bone [5] showed the similarity between structural components of porous ceramics and with the bone tissue.

Figure 3 shows the XRD patterns of porous ceramics after sintering at 1600° C for 1 and 10 h. As can be seen from this Figure the ceramics is composed of two phases: monoclinic (M) ZrO2 phase and cubic (K) ZrO_2 phase. Quantitative phase analysis showed that cubic/monoclinic phase ratio is 6/4 and corresponds to that of known ZrO_2 - MgO state diagram [9]. Increasing the sintering time from 1 to 10 hours leads to reducing the cubic phase content.

Figure 4 shows stress–strain curves of the porous ceramic sample and human trabecular bone [10] after uniaxial compression tests. This kind of deformation curve is typical for the fracture of porous zirconia-base ceramics [11]. However, a distinguishing feature of the deformation curves obtained for porous zirconia-base ceramics is that there is a deviation from linearity at high stress as well as downward oscillating branch with a gradual decrease in the stress due to the accumulation of microcracks (Figure 2b).

Ceramic mechanical properties studies have shown that both compressive strength and effective elasticity modulus increased when decreasing the pore sizes. The compressive strength for ~40% porosity materials is 22 MPa. Effective elasticity modulus determined from the slope of the deformation curves for the studied materials is in the range 1.0-1.8 GPa. Quasi-static compression tests on porous ceramics showed that the material had satisfactory strength for the use as osteogenesis material.
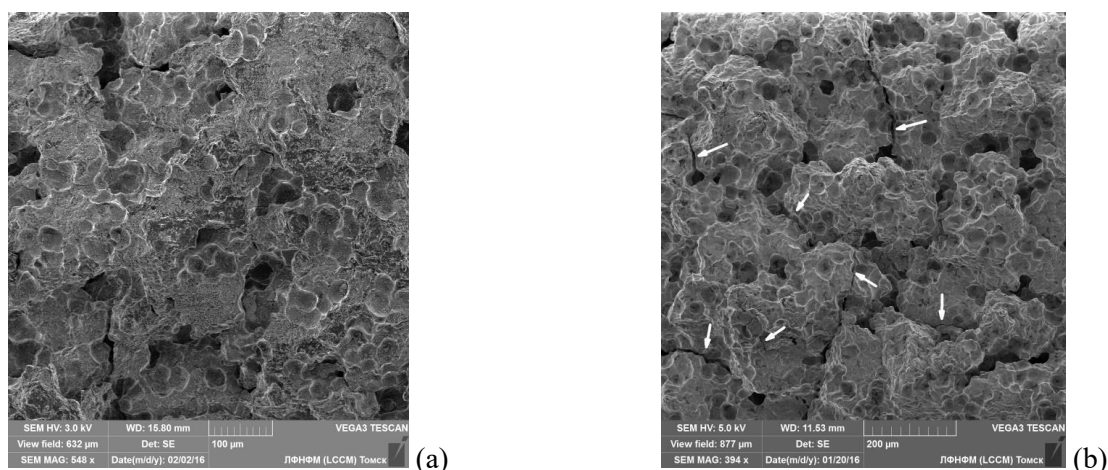

Figure 2. The SEM image of the fracture surface of porous ceramics (after the quasi-static compression tests). Arrows in the Figure 2b, indicate of microcracks.
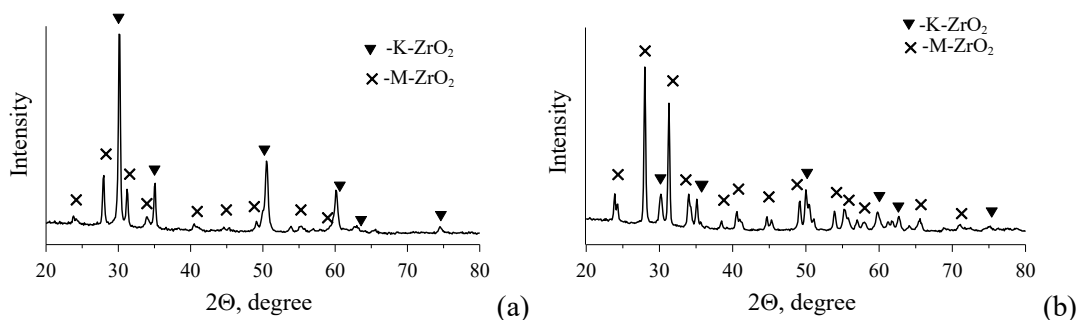

Figure 3. The XRD pattern of polished porous ceramics surface after sintering 1 h (a) and 10 h (b) at 1600°C.
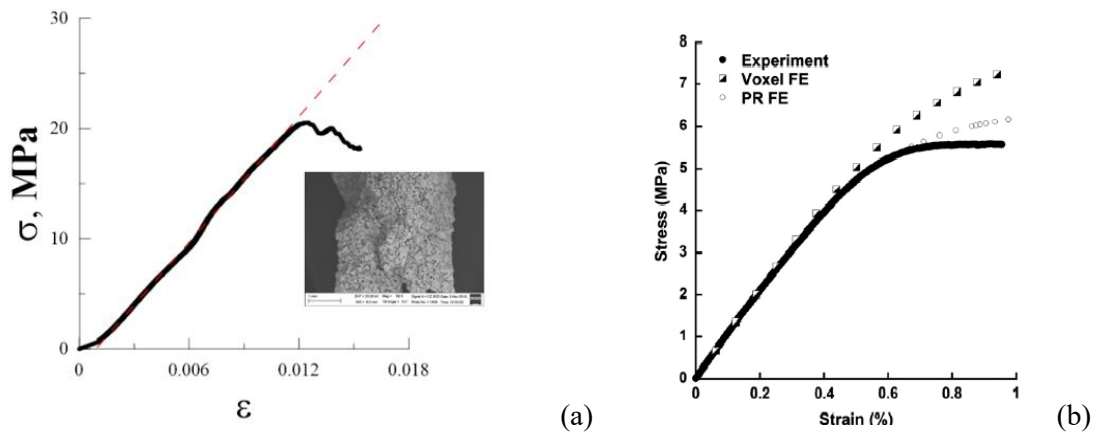
Figure 4. Stress–strain curve and macrograph of ceramic with a porosity of 40% subjected to compression (a); the stress–strain curve of human trabecular spinal bone subjected to compression (b) [10].

4. Conclusion

It was shown that after sintering at 1600°C that zirconia ceramic structure was characterized by the presence of pores with pore sizes in two categories such as "big pore" 100-220 μm and "small pore" 0.8-8 μm. This makes the ceramic structure very similar to the structure of the natural spinal bone.

The level of mechanical properties of the synthesized zirconia-based ceramics is determined by the pore size. The values of the compressive strength and the effective Young's modulus are very similar to those of the natural spinal bone.

References

[1]   Bartolo P, Kruth J P, Silva J, Levy G, Malshe A, et al. 2012 *CIRP Annals - Manufacturing Technology* **61** 635–655

[2]   McEntire B J, Bal B S., Rahaman M N., Chevalier, Pezzotti *G* 2015 *Journal of the European Ceramic Society* **35** 4327–4369

[3]   Davis W, Allouni A K, Mankad K, Prezzi D, Elias T, Rankine J, Davagnanam I 2013 *Clinical Radiology* **68** 64-74

[4]   Staiger M P, Pietak A M, Huadmai J, Dias G 2006 *Biomaterials* **27** 1728–1734

[5]   Wua S, Liu X, Yeung K W K, Liu C, Yang X 2014 *Materials Science and Engineering* **R 80** 1–36

[6]   Savchenko N L, Sevostyanova I N, Sablina T Y, Molnar L, Geber R, Gomze L A, Kulkov S N, Gomze L N 2014 *Építôanyag - Journal of Silicate Based and Composite Materials* **66** 44-47

[7]   Savchenko N L, Sevostyanova I N, Sablina T Yu, Gomze L, Kulkov S N   2014 *Physical Mesomechanics of Multilevel Systems 2014: AIP Conf. Proc* **1623** 547-550

[8]   Grigoriev M V, Kulkov S N 2011 *Russian Physics Journal*   **1** 1305–1311

[9]   Odnik H M, McMurdie H F Phase Diagrams for Zirconium and Zirconium system. 1999 *Maryland: Phase Diagram Data Center*

[10]   Ji W, Bin Z, Sherry L, Aaron J. F, Arnav S, Xiutao S, Adams M, Keaveny T M, Guo X E 2015 *Bone* **72** 71–80.

[11]   Savchenko N L, Sablina T Yu, Sevostyanova I N, Buyakova S P, Kulkov S N 2016 *Russian Physics Journal* **58**/11 (*Russian Original* 2015 11 56-60).