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Sintering mode optimization of ceramics based on synthesized powders of ZrO_2 - Y_2O_3 - CeO_2 system with different contents of Al_2O_3 for development of bioinert ceramic materials.

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ABSTRACT

The production of new bioinert ceramic materials based on the synthesized nanopowder with a higher resistance against the low-temperature degradation in water, which may be used to produce full-ceramic constructions for dental and orthopedic prosthesis, is a vital task. The paper presents results of experimental studies aimed at the optimization of ceramic sintering based on synthesized powders in the system ZrO_2 - Y_2O_3 - CeO_2 - Al_2O_3 . It was found that the maximum density and the minimum average grain size was obtained in the ceramic after its sintering at 1400 °C. Further increase of the sintering temperature up to 1500 °C leads to the increase in ceramic porosity. The study examined the impact of the sintering temperature and the content of aluminum oxide on the microstructure and the average grain size of the ceramic. Introduction of aluminum oxide leads to the formation of new phases of different shape, which discharge rise above the surface and stay at the edges of the agglomerate grains, which was shown in the microstructural analysis of ceramics. The experiments clarified the impact of ultrafine additives on the sintering process and properties of ceramics. The introduction of aluminum oxide leads to a decrease in the average grain size from 112 ± 12 nm to 90 ± 8 nm.

Keywords: nanoparticles, nanocrystalline powders, the system ZrO_2 - Y_2O_3 - CeO_2 - Al_2O_3 , bioceramics, sintering, microstructure, grain size, agglomerates.

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INTRODUCTION

Current research activities are largely associated with the need to process the smallest details after sintering to strictly specified dimensions for their future use in microtechnology medical equipment (particularly in dentistry) [1, 2], bio-implants [3, 4], structural and cutting ceramics [5], solid electrolytes for fuel cells and their operation at relatively low temperatures [6].

It is established that the properties of sintered-hard alloys are determined by the composition, microstructure and characteristics of the intragranular structure of hard metal components (sub-microstructure) [7, 8]. Properties of sintered alloys are largely determined by the characteristics of feedstock and hard metal production intermediates, which vary depending on various process parameters. In view of the huge difference between the initial sintering temperatures of micron and nanoparticles, the use of ultrafine powders as sintering activators is of great interest [9-12].

Specific objective of nanoceramics sintering - preservation of small grain size and prevention of their coarsening during sintering of compacted samples. This is possible provided high-density compacts (not less than 0.7 of the theoretical density of the material), when sintering proceeds quite fast; at a relatively low sintering temperature [13].

Oxide materials are based on various multilayer composites of the system ZrO_2 - Y_2O_3 - CeO_2 - Al_2O_3 , belonging to subsolidus compounds and in most cases present solid solution composites based on ZrO_2 (T- ZrO_2 and M- ZrO_2) and dispersed particles α - Al_2O_3 . Properties of composites in this system will be determined by a combination of material properties in binary and ternary systems, which limit it [14]. Materials developed on the basis of the studied systems can be divided into two classes: coarse, designed for operation at temperatures up to 2200°C and fine-grained, with improved physical and mechanical properties.

Thus, this system can be used to develop composites different by structure and hardening mechanisms.

Traditionally, ceramics based on ZrO_2 was used in the metallurgy to produce melting crucibles. Today zirconia ceramics is one of the most promising construction and instrumental ceramic materials, which is used in manufacturing parts of gas turbine and diesel engines, friction units, pumps, sealing rings, valve elements, nozzles of spray chambers, dies used for wiredrawing, cutting tools. In addition, ceramics based on ZrO_2 is used in medicine for the manufacture of bone tissue level implants [3].

Whereas the production of bio-implants is based on using T- ZrO_2 ceramics, the main criterion when choosing a method of synthesis implied maximum content of metastable T- ZrO_2 , stabilized by yttrium and cerium additives. It is difficult to produce ceramics in which the particle size of zirconia would be less critical, needed to maintain the metastable T- ZrO_2 after sintering, using the powders obtained by conventional physical and chemical methods. These powders have various size of particles and contain many structural defects. Highly pure homogeneous nanocrystalline powders with a narrow particle size distribution synthesized by chemical methods are characterized by high activity during sintering. Their use contributes to maintaining high content of metastable T- ZrO_2 after cooling [15].

Phase transition kinetics depends on the chemical composition of the ceramic, characteristics of primary powders, heat treatment conditions, porosity and grain size. One can assume that the system ZrO_2 - Y_2O_3 - CeO_2 - Al_2O_3 is promising in terms of creating different classes of ceramic materials, which will have the required properties.

“Composition → structure → dispersion → properties” is the basic concept in materials science, which establishes a direct link between the characteristics of the primary powders and properties of the obtained materials. In recent years, studies conducted in different countries, found that nanodisperse powders have qualitatively new physical and chemical properties that are far from thermodynamic equilibrium. This served as the basis for the creation and application of nanocrystalline powders in the production of new materials: under the constant chemical composition of the system, variation of the powder processing conditions provides materials with different phase composition, microstructure and properties [16, 17, 18].

The existing unavoidable self-organization processes and their multivariance in nonequilibrium disperse systems identified the complexity of technologies used for the production of new materials based on zirconium dioxide. However, at the same time, low density, high surface area and sintering properties of nanocrystalline powders hinders the application of traditional molding and heat treatment operations for their processing, which is the scientific basis for optimizing the entire fabrication cycle [19, 20, 21].

The purpose of this research is to study the impact of small aluminum oxide additives on the phase composition and compaction of nanopowders processes as well as on sintering properties of ceramics in the system ZrO_2 - Y_2O_3 - CeO_2 - Al_2O_3 .

STARTING MATERIALS

The experiment implied synthesis of powders containing yttrium and cerium oxides, as stabilizing additives, as well as different amounts of alumina additives. Compositions of powders and the content of salts are given in Table 1.

Table 1 - Compositions of powders and the content of zirconium, yttrium, cerium and aluminum salts in solutions

	Stabilized content, mol.%			Salt content, g			
	Y_2O_3	CeO_2	Al_2O_3	$ZrOCl_2 \times 8H_2O$	$Y(NO_3)_3 \times 6H_2O$	$Ce(NO_3)_3 \times 6H_2O$	$Al(NO_3)_3 \times 9H_2O$
1	2	4	0	47,62	2,41	2,73	0
2	2	4	1	47,14	2,38	2,703	1,49
3	2	4	3	46,19	2,35	2,27	4,42

Synthesis of powders was performed by reverse chemical subsidence using the sol-gel technology. The sol-gel method implies preparation of sol, followed by its transfer into gel, i.e. into the colloidal system consisting of a liquid dispersion medium contained in the three-dimensional grid formed by connected particles of the dispersed phase.

RESEARCH METHODS

Samples were formed by uniaxial cold pressing in a steel mold at a compaction pressure of 200 MPa on a manual hydraulic press «Karl Zeiss Jena». The pressing process was improved by adding (as a temporary binder) 4% aqueous solution of polyvinyl alcohol PVA 7/2 GOST 10779-69 in amounts of 10% by weight of the powder sample. Sintering was carried out in an electric furnace EHF- 0.6.

Stability of ceramic material containing zirconia to hydrothermal aging was carried out by the change in phase composition. Before testing, samples were examined for the monoclinic phase content. Afterwards, they were placed in a suitable autoclave and subjected to steam at a temperature of $(134 \pm 2) ^\circ C$ and a pressure of 0.2 MPa for 5 hours. After cooling, the samples were dried. Then the samples were re-tested for the monoclinic phase content; according to ISO 13356 requirements, after testing for accelerated "aging" the content of monoclinic phase in the samples should not exceed 25% [22].

Microstructural analysis of the samples after sintering on the etched metallographic specimens was performed using scanning probe microscopy through the multifunctional microscope "FemtoScan".

Chemical resistance tests were carried out by DIN EN ISO 6872-2008 in 4% solution of acetic acid, which was evaluated by the change in sample mass after soaking in this solution at a temperature of 80 °C for 16 hours.

Tests for hardness and crack resistance were carried out by polished surface indentation by the Vickers pyramid by using a hardness tester TP-7R-1 with a load 98,1-196,1 N. The length of diagonal indentation and cracks forming in the corners was measured by using the optical microscope NEOPHOT-32.

RESULTS AND DISCUSSION

The experimental data related to density, porosity and shrinkage after sintering of $ZrO_2-2Y_2O_3-4CeO_2$ powder samples with different aluminium oxide content are given in Table 2.

Figure 1 contains graphs showing the impact of sintering temperature on the linear shrinkage, apparent density and porosity of $ZrO_2-2Y_2O_3-4CeO_2$ powder samples with different aluminium oxide content. Isothermal time made 3 hours.

It may be noted that maximum density at all sintering temperatures was observed in the samples without aluminium oxide additive. After sintering at a temperature of 1400 °C minimum residual porosity of the samples made 1.3%. The introduction of aluminium oxide increased ceramic porosity, which was especially noticeable at 1350 °C. With further increase in the sintering temperature, the samples containing 1 and 3 wt.% Al_2O_3 had almost similar porosity (Figure 1. a). The ceramics with aluminium oxide additives reached maximum apparent density after sintering at 1400 °C. The residual porosity made 4.5% in samples with 1% Al_2O_3 and 6.2% in samples containing 3% Al_2O_3 . Raising the sintering temperature up to 1500 °C leads to an increase in the ceramic porosity irrespective of aluminium oxide content. (Figure 1. b). This leads to grain growth and transition of zirconia from tetragonal to monoclinic phase, which starts after 1400 °C. Reduction in the density of ceramic samples is affected by the isothermal time increase at an optimum temperature.

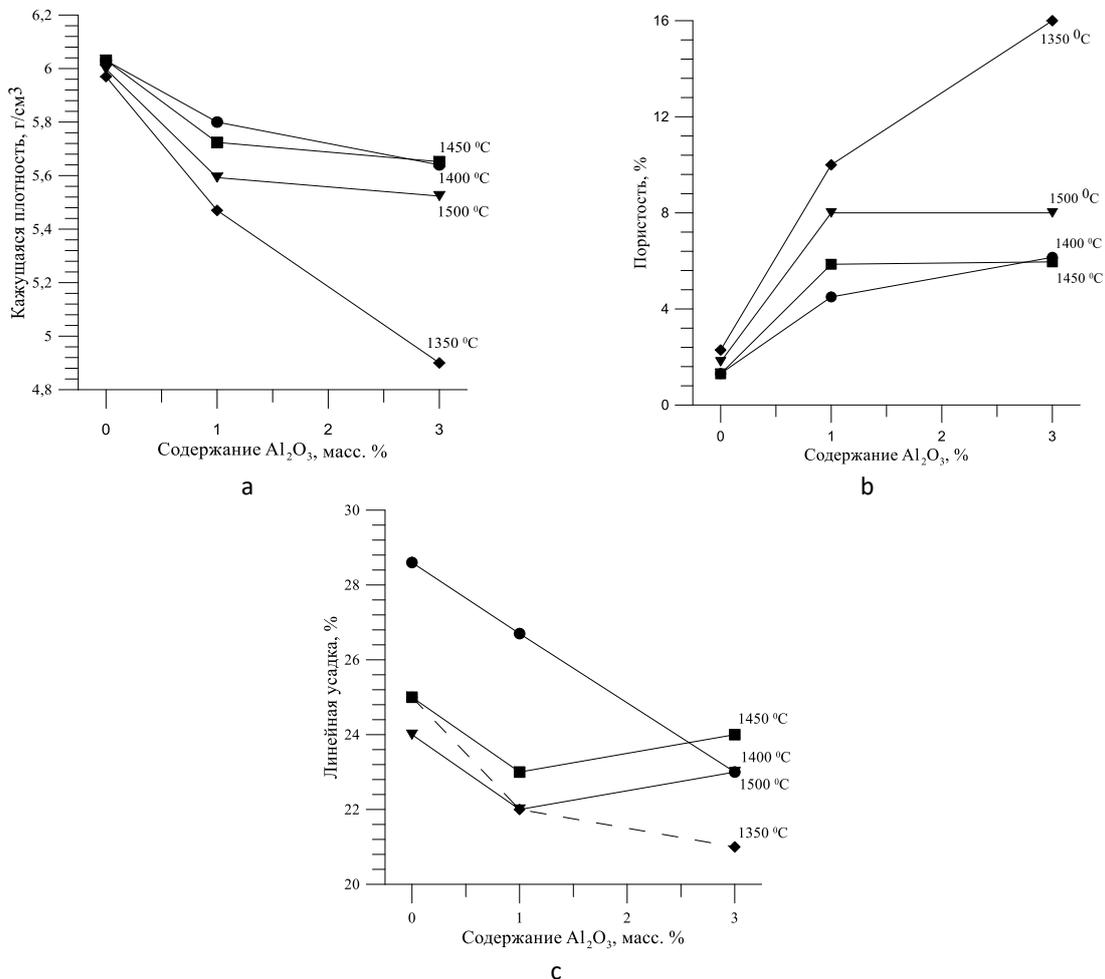


Figure 1 - graphs showing the impact of sintering temperature on the linear shrinkage of powder samples $ZrO_2-2Y_2O_3-4CeO_2$ with different content of aluminium oxide. Isothermal time - 3 hours: a–apparent density of samples; b–porosity of samples; c–linear shrinkage of samples.

Translation of elements Figure 1: Содержание Al_2O_3 , масс.% - Content of Al_2O_3 , wt.%, кажущаяся плотность - apparent density, пористость – porosity, линейная усадка - linear shrinkage

The obtained data show that samples without aluminium additives have the most substantial shrinkage (26-29%, Figure 1. c). After the introduction of alumina additives linear shrinkage reduced to 22-24%. Heat treatment at 1400 °C caused maximum linear shrinkage of all samples.

After sintering the samples (isothermal time made 2 hours), maximum density and minimum porosity was observed in samples sintered at temperatures of 1400 and 1450 °C (Figure 2 a, b). Isothermal time reduction did not lead to significant changes. The curves showing change in the apparent density and porosity depending on the alumina content is similar to the above description.

Table 2. Characteristics of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2-Al_2O_3$. Sintering temperature - 1350-1500 °C, isothermal time - 2 and 3 hours.

Content of Al_2O_3 , wt.%	T_{sin} , °C	τ , hours	ρ , g/cc	P, %	Shrinkage,%	
0	1350 °C	3	5,97	2,3	25	
		2	5,96	2,5	24	
	1400 °C	3	6,03	1,3	29	
		2	6,03	1,2	24	
	1450 °C	3	6,03	1,3	25	
		2	6,07	0,6	25	
	1500 °C	3	6,00	1,8	24	
		2	6,02	1,5	24	
	1	1350 °C	3	5,47	10	22
			2	5,7024	6,2	22
		1400 °C	3	5,805	4,5	27
			2	5,8203	4,3	23
1450 °C		3	5,724	5,86	23	
		2	5,8044	4,5	23	
1500 °C		3	5,593	8	22	
		2	5,619	7,6	22	
3		1350 °C	3	4,92	16	21
			2	5,4911	16	23
		1400 °C	3	5,64	6,15	23
			2	5,672	5,6	24
	1450 °C	3	5,652	5,96	24	
		2	5,6561	5,88	23	
	1500 °C	3	5,524	8	23	
		2	5,5473	7,7	23	

The experiments gave the possibility to study the impact of sintering temperature on the ceramic microstructure and its grain size. The microstructure of the samples without alumina additives after sintering at 1350 °C is shown in Figure 3. a, b. Their structure is homogeneous, without inclusions and pores, consists of rounded grains with a size ranging from 80 to 230 nm. The average grain size makes 144 nm (Table 3). The picture (Figure 3. c) shows the simplest way to analyze the height and width of objects - by making cross-sections. The average cross section is made in order to determine the average grain size.

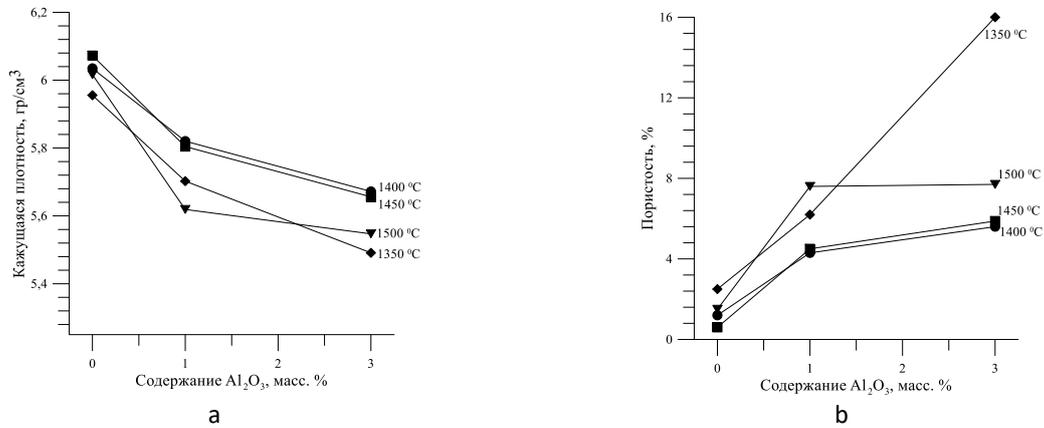


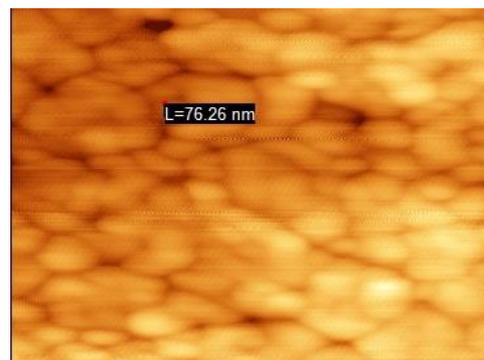
Figure 2 – The impact of sintering temperature on the linear shrinkage of powder samples $ZrO_2-2Y_2O_3-4CeO_2$ with different content of aluminum oxide. Isothermal time - 2 hours: a - apparent density of samples; b - porosity of samples;

The introduction of 1 and 3 wt.% Al_2O_3 led to the reduction in average ceramic grain size to 98 and 92 nm, respectively (Figure 4, 5). The microstructure of ceramics with 3 wt.% Al_2O_3 is heterogeneous, contains pores and ceramic grains present aggregates composed of separate grains.

The ceramic microstructure with different content of alumina after sintering at 1400 °C is shown in Figures 6-8. The average grain size of the ceramic without alumina additives made 112 nm. Introduction of a 1% aluminum oxide leads to a decrease in the average grain size to 90 nm, and the introduction of 3% Al_2O_3 up to 83 nm. In addition, the introduction of aluminum oxide to the microstructure of ceramics leads to the formation of new phases of different shape, which discharge rise above the surface and stay at the edges of the agglomerate grains.



a



b

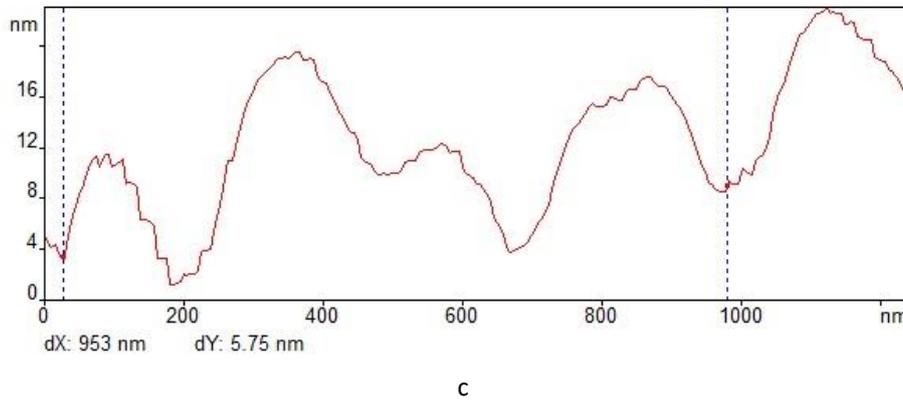


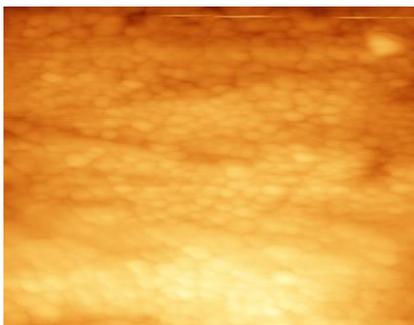
Figure 3-AFM-picture showing microstructure of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2$ without Al_2O_3 . Sintering temperature 1350 °C, isothermal time – 3 hours:
 a-scanning field 10×10um; b-scanning field 2,5×2,5 um;
 c-surface contour

Further increase in the sintering temperature up to 1450 °C leads to coarsening of the ceramic structure. Spherical discharges are observed on the surface of the ceramic samples without aluminum oxide; agglomerate clusters of grains take polyhedral shape. The average grain size of the ceramic without alumina makes 127 nm. The introduction of a 1% aluminum oxide leads to a decrease in the average grain size to 98 nm, and the introduction of 3% Al_2O_3 - up to 94 nm.

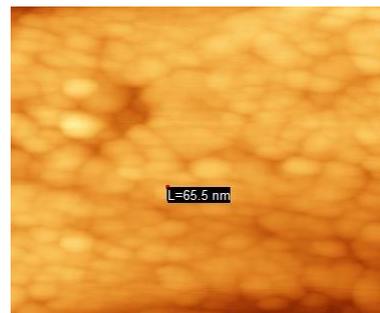
Figure 9 shows dependence of the average size of ceramic grains with $ZrO_2-2Y_2O_3-4CeO_2$ composition after sintering on alumina content. When the sintering temperature reaches 1400 °C ceramic grain size is minimal. The introduction of aluminum oxide reduces the average grain size irrespective of the sintering temperature.

Table 3 –The impact of sintering temperature on average ceramic grain size

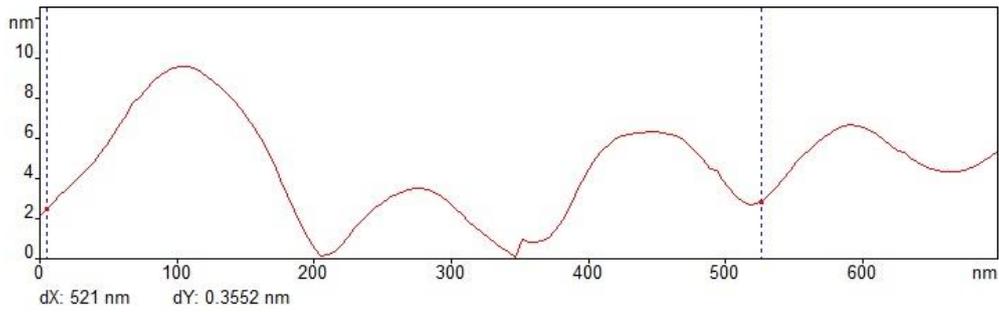
Sintering temperature, °C	Alumina content, %	Spread of grain size values, nm	Average grain size, nm
1350 °C	0	80-231	144±18
	1	55-173	98±10
	3	40-153	92±10
1400 °C	0	50-229	112±12
	1	52-170	90±8
	3	40-128	83±8
1450 °C	0	73-192	127±11
	1	40-175	99±12
	3	36-207	94±16



a

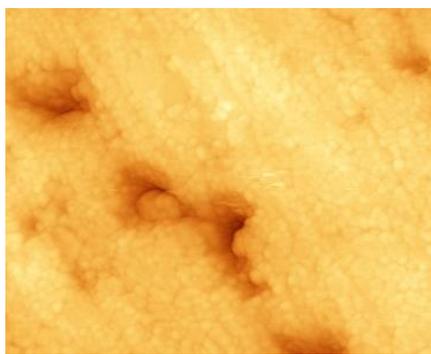


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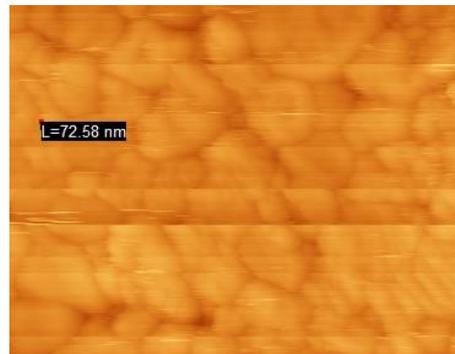


c

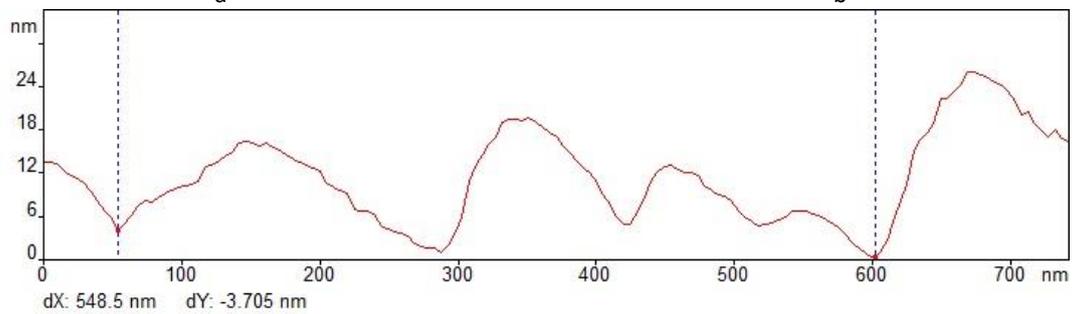
Figure 4 - AFM-picture of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2+1\%Al_2O_3$. Sintering temperature 1350 °C, isothermal time – 3 hours: a - scanning field 10×10 μm; b-scanning field 2,5×2,5 μm; c-surface contour



a

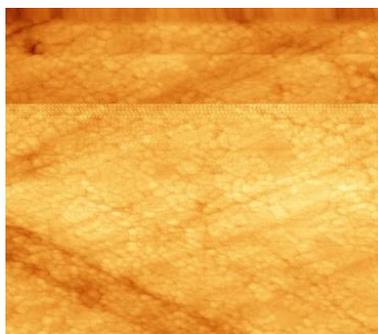


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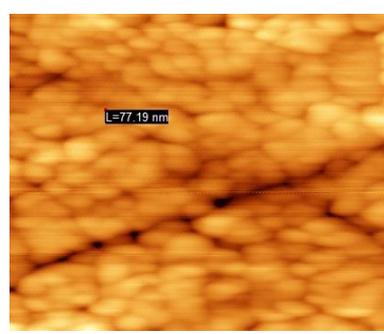


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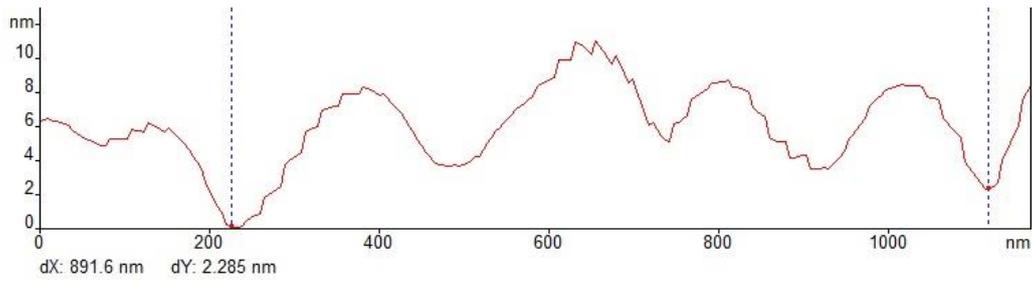
Figure5-AFM-picture of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2+3\%Al_2O_3$. Sintering temperature - 1350 °C, isothermal time – 3 hours: a - scanning field 10×10 μm; b - scanning field 2,5×2,5 μm; c - surface contour



a

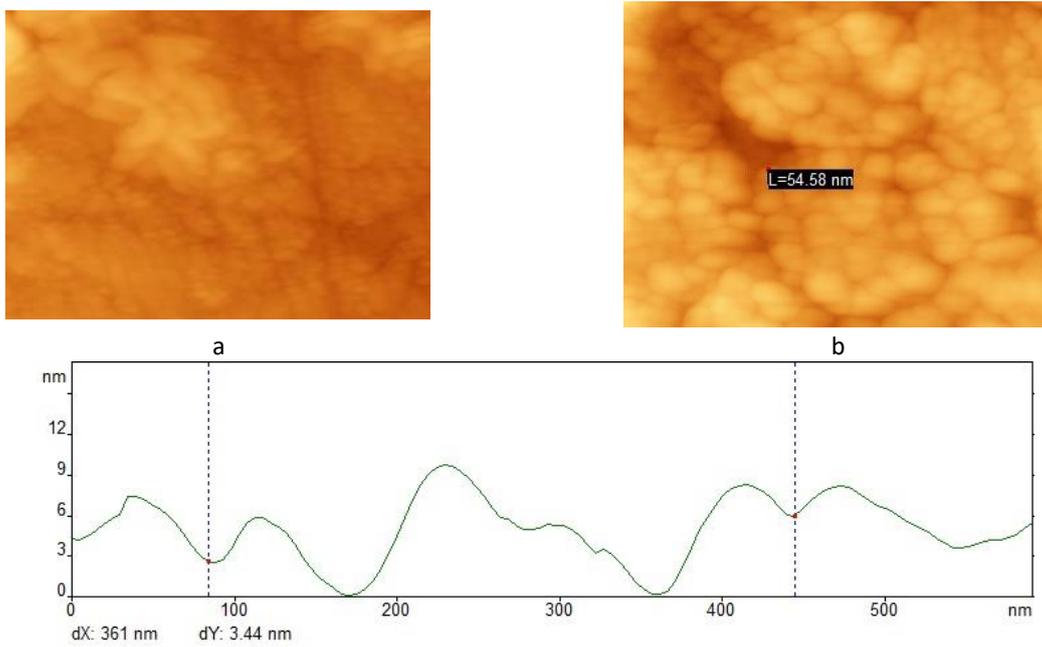


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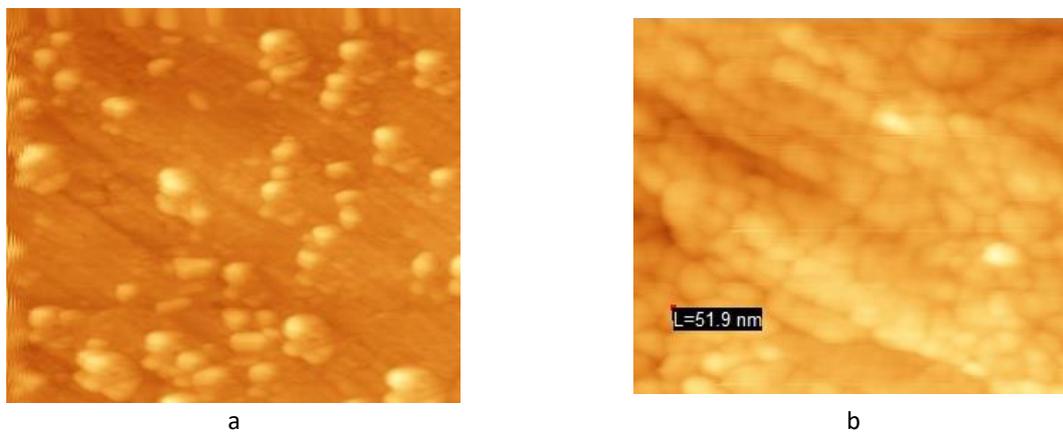
c

Figure 6 - AFM-picture of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2$ without alumina. Sintering temperature 1400 °C, isothermal time – 3 hours: a - scanning field 10×10 um; b - scanning field 2,5×2,5 um; c - surface contour



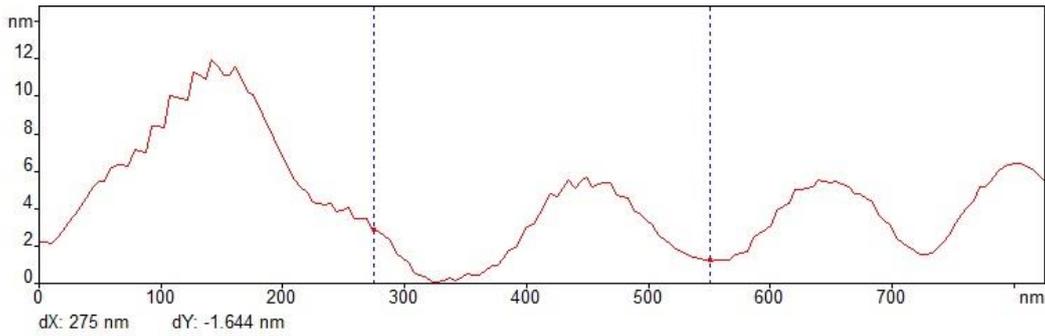
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Figure 7 - AFM-picture of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2+1\%Al_2O_3$. Sintering temperature 1400 °C, isothermal time – 3 hours: a - scanning field 10×10 um; b - scanning field 2,5×2,5 um; c - surface contour



a

b



c

Figure 8 - AFM-picture of ceramics based on $ZrO_2-2Y_2O_3-4CeO_2+3\%Al_2O_3$. Sintering temperature 1400 °C, isothermal time – 3 hours: a - scanning field 10×10 μm; b - scanning field 2,5×2,5 μm; c - surface contour

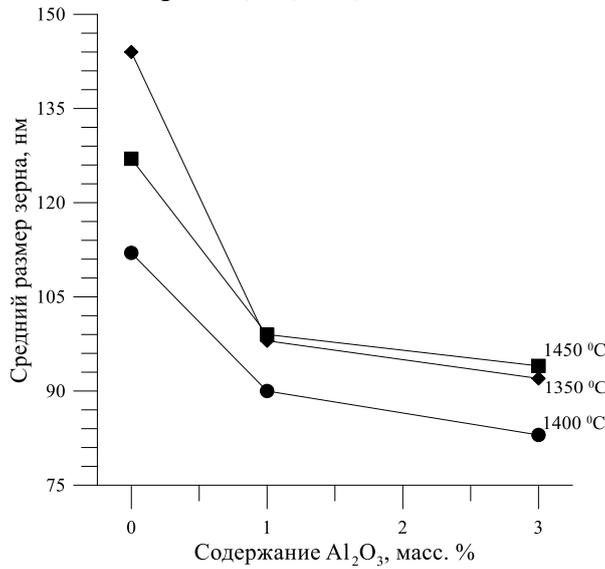


Figure 9 – The impact of sintering temperature and alumina additive on grain size. Translation of elements

Figure 9: средний размер зерна – average grain size, содержание Al₂O₃, масс. % - content of Al₂O₃, wt. %

Ceramic materials composed of $ZrO_2-2Y_2O_3-4CeO_2-Al_2O_3$, used as bio-implants, should be non-toxic, chemically inert, force resistant, mechanically strong, etc.

Table 4 shows chemical resistance of the synthesized powder samples. According to DIN EN ISO 6872, chemical solubility of the ceramic material should be less than or equal to 100 g · cm⁻². Tests showed that chemical solubility of samples without alumina is less than 60 g · cm⁻², introduction of alumina additive reduces this indicator up to 27 g · cm⁻². After the test, the sample without alumina lost its weight, and weight increase was observed in samples containing 1 and 3 wt. % of aluminum. Thus, it can be concluded that small addition of alumina had positive effect on the chemical resistance of the samples. All samples are chemically stable and can be used as ceramic materials for prosthetics.

Table 4. – Results of chemical resistance tests

Powder composition	Full area, cm	Average change in mass, μgr · cm ⁻²
$ZrO_2-2Y_2O_3-4CeO_2$	1,7182	-58,2
$ZrO_2-2Y_2O_3-4CeO_2+1\text{масс.}\% Al_2O_3$	1,8254	+27,5
$ZrO_2-2Y_2O_3-4CeO_2+3\text{масс.}\% Al_2O_3$	1,8738	+26,5

The analysis of hardness and crack resistance of the ceramic material based on $ZrO_2-2Y_2O_3-4CeO_2-Al_2O_3$ powder with different alumina content showed that no cracks were formed at a given load at the corners of the indenter prints. Cracks were observed in the sample without alumina. This indicates relatively high crack resistance of the obtained ceramic material. Ceramics without alumina additives has maximum hardness of 11.3 GPa. The introduction of alumina decreased the hardness of the material, due to greater porosity as compared to ceramic without alumina (Table. 5).

Table 5 - The influence of additives on alumina samples' hardness

Powder composition	HV, GPa	K_{Ic}^* , $MPa \times m^{1/2}$
$ZrO_2-2Y_2O_3-4CeO_2$	11,3±0,2	10,3
$ZrO_2-2Y_2O_3-4CeO_2+1\text{macc.}\% Al_2O_3$	9,7±0,1	-
$ZrO_2-2Y_2O_3-4CeO_2+3\text{macc.}\% Al_2O_3$	9,5±0,2	-

CONCLUSIONS

- Optimization of sintering mode for ceramics based on synthesized powders found that its maximum density was obtained at 1400 ° C, regardless of alumina content. Further increase of the sintering temperature up to 1500 ° C leads to an increase in ceramic porosity. Residual porosity of ceramic without alumina makes less than 2% and 4.0-6.0% - for ceramic with Al_2O_3 .
- The experiments revealed the impact of the sintering temperature and the alumina content on the microstructure and average grain size of ceramic. Microstructural analysis showed that the introduction of aluminum oxide leads to the formation of new phases of different shape, which discharge rise above the surface and stay at the edges of the agglomerate grains. After sintering at 1400 ° C, ceramics has minimum average grain size. The introduction of alumina leads to a decrease in average grain size from 112 ± 12 nm to $90 \text{ nm} \pm 8$ for ceramics with 1% Al_2O_3 and up to 83 ± 8 nm for ceramics with 3% Al_2O_3 .
- Chemical solubility of samples without alumina makes less than $60 \text{ g} \cdot \text{cm}^{-2}$, the introduction of alumina additive reduces this indicator to $27 \text{ g} \cdot \text{cm}^{-2}$.
- Relevant research showed that ceramics without alumina additives had maximum hardness of 11.3 GPa. This was achieved thanks to the fact that the development of materials based on $ZrO_2-Y_2O_3-CeO_2$ implied an integrated approach to the entire production process - from the initial receipt of nanocrystalline powders having complex composition to preforms and heat treatment products. This provides the possibility to achieve a high level of physical and mechanical properties of materials due to the formation of high-density and homogeneous microstructure at lower sintering temperatures. In addition, this provides the possibility to create bioinert implants resistant to the aging process in the human body.

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