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EFFECT OF SINTERING TEMPERATURE ON MICROSTRUCTURE AND PROPERTIES OF ZIRCONIA CERAMICS FOR THE NEEDS OF NUCLEAR ENERGY

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The paper provides experimental results of obtaining high-density ZrO_2+3%Y_2O_3 ceramics, which is promising for use as a matrix for immobilization of HLW. The effect of sintering temperature in the range of 1100...1650 °C on the microstructure of the sintered tablets was studied. Phase composition and microstructure of experimental samples were characterized by XRD and SEM. Grain size distribution analysis was carried out using the "Thixomet" image analyzer. Microhardness was determined using a metallographic complex LECO (USA), an inverted microscope IM-3MET and a hardness tester UIT HVB-30. It was established that increase in the sintering temperature leads to a significant increase in the average grain size (from 85 nm to 1000 nm) and increase in the density of the sintered tablets. Sintering temperature should be at least 1550...1650ºC to produce high-dense ceramics (97...98 % of theoretical value). Obtained ceramics is characterized by high values of microhardness HV > 12 GPa and crack resistance of 5.5...6.3 MPa·$m^{1/2}$.
Keywords: *Zirconium oxide; Sintering; Microstructure; Microhardness; Crack resistance*
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INTRODUCTION

Zirconia-based materials (for example, ZrO_2+Y_2O_3, $Zr_2Gd_2O_7$) are widely used as matrices for high-level waste (HLW) due to their high radiation resistance and ability to bind in the crystal lattice (incorporation) of such elements as Pu, Am, Np and Ln in spent nuclear fuel (SNF) [1-3].

Recently, there is a considerable interest in obtaining and studying the characteristics of nanostructured ZrO_2-based ceramics with a grain size of less than 100 nm [4-6]. Thus, a number of works revealed that this material with a grain size of 25 nm and 38 nm has the ability to resist amorphization under Krypton ions irradiation (Kr^+, 400 keV) up to a fluence of $5.36 \cdot 10^{16}$ ion/cm^2 (129 dpa), and also has enhanced radiation resistance [7, 8].

In addition, nanosized zirconia-based powders are used for the manufacturing of oxide dispersion-strengthened (ODS) materials [9, 10] – a promising class of structural materials for the next-generation nuclear reactors [11]. Exactly, the presence of such nanooxide precipitates with a uniform distribution into the matrix improves the radiation resistance of materials, as well as their mechanical and corrosion properties, especially at elevated temperatures.

Another field of ZrO_2 materials application is the structural and refractory ceramics [12, 13]. Also, ZrO_2 is widely used in medicine due to its high biocompatibility [14]. In order to achieve the highest operational characteristics, methods for manufacturing of ZrO_2 nanopowders, as well as methods of their compaction and sintering to obtain products with a fine-grained structure, are actively developed [15-17].

The methods aimed on compacting of the raw powders and obtaining oxide ceramics in relation to its usage as both HLW matrices and structural ceramics are traditionally developed at NSC KIPT [18-20].

The goal of this work is to study the possibility of obtaining experimental samples from nanosized yttria-partially stabilized zirconia (Y-PSZ) powder by cold-pressing at different modes and subsequent sintering in air into dense ceramics with a fine-grained structure. Also, research of physical and mechanical characteristics of obtained ceramics is focused too.

MATERIALS AND METHODS

The powder of partially-stabilized zirconia PSZ-5.3YB manufactured by "Stanford Materials Corporation" (USA) was used as the raw material. According to the manufacturer, the powder was obtained the chemical co-precipitation of zirconium and yttrium salts solutions, followed by filtration, drying and calcination. PSZ-5.3YB powder is characterized by the good ability for pressing, as it contains an organic binder based on polyvinyl alcohol, which was introduced into the material at the stage of spray drying. This powder consists of micro-spherical granules size of 70...100 μm (Figure 1a), and the size of the particles of which the granules are composed is 30...40 nm (Figure 1b). Chemical composition of material according to the manufacturer: Zr(Hf)O_2 -94.5%; Y_2O_3 -5.2 ± 0.2%wt.

Compacting powders in the form of tablets (Ø10 mm and height 6-7 mm) was carried out by the method of uniaxial bilateral cold pressing (CP) in a steel mold at a pressure of 200 MPa. The density of samples after CP was 2.84±0.02 g/cm^3 (46 % of theoretical).

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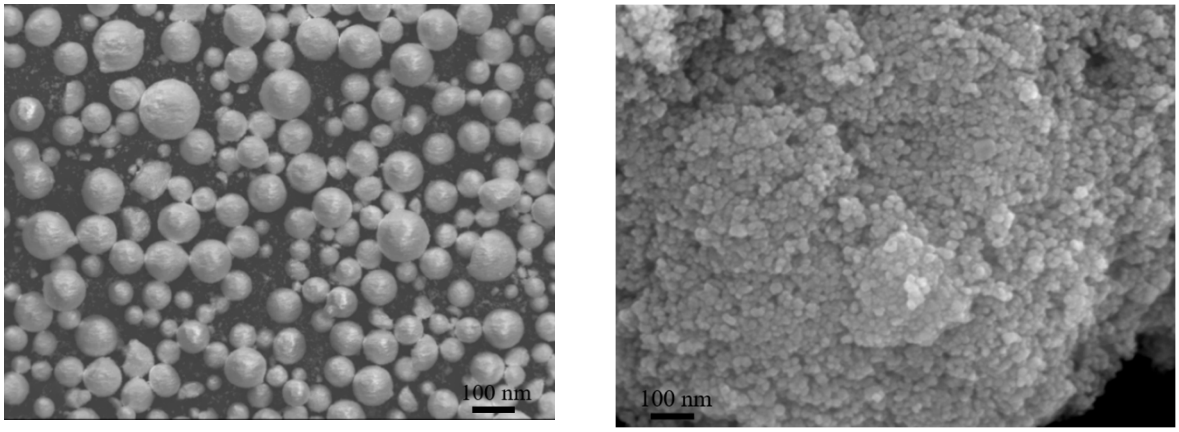


a b

Figure 1. SEM micrographs of the PSZ-5.3YB powder (ZrO_2+3mol%Y_2O_3)

Tablets were sintered in the temperature range of 1100...1650ºC for 3 hours in an air atmosphere. The rate of reaching the isothermal holding temperature was 5ºC/min.

Density and open porosity of sintered ceramics were determined by hydrostatic weighing in distilled water.

XRD measurements were conducted using DRON-4-07 diffractometer in a filtered Cu-Kα radiation (λ = 0.154187 nm). Phase composition and lattice parameters were determined by the Rietveld method (MAUD software). Microstructural characteristics of the samples (size of coherent scattering domains CSD) were estimated by the Williamson-Hall technique (integral breadth analysis).

Microhardness of the sintered samples was determined using a metallographic complex LECO (USA), an inverted microscope IM-3MET and a hardness tester UIT HVB-30. Final surface polishing was performed using diamond paste with a particles size of 1 μm.

Microstructure studies of the samples were carried out using JSM-7001F scanning electron microscope (JEOL, Japan). The average grain size was determined by the method of random secants; the number of analyzed chords for each microstructure image was 18 [21]. Calculation of the grains diameter, based on their area, as well as grain size distribution analysis was carried out using the "Thixomet" image analysis system.

The stress intensity factor K_{1C} (crack resistance) of ceramics was determined by the indentation method using Vickers diamond pyramid under the load of 10 kg and holding time of 15 s. Calculation of K_{1C} [MPa·m$^{1/2}$] was carried out according to the Niihara approach [22, 23]:

$$K_{1C} = 0.203(c/a)^{-3/2} \cdot H \cdot a^{1/2} \quad (1)$$

where, a – half value of the indentation diagonal, μm; c – crack length, μm; H – microhardness, MPa. For each sample 7 indentations were made and analyzed.

RESULTS AND DISCUSSION

XRD analysis revealed that the raw powder ZrO_2+3mol%Y_2O_3 consists of two zirconia polymorphs. The content of monoclinic zirconia ZrO_2-m is 54.9wt%, and content of tetragonal ZrO_2-t - 45.1wt%. Estimated CSD size for the ZrO_2-m and ZrO_2-t phases are 36.6 nm and 40.6 nm, respectively.

It was established that increase in the sintering temperature from 1100ºC to 1650ºC leads to an increase in the density of the sintered samples. Thus, the density of the samples at T = 1450ºC was 5.6...5.7 g/cm^3, and the open porosity was 1.0...1.5%. Increasing the sintering temperatures to T = 1550ºC and T = 1650ºC results in obtaining a denser ceramic (5.9...6.03 g/cm^3) with a linear shrinkage of 14...15%, which is characterized by the absence of open porosity. The characteristics of the sintered samples are given in Table 1.

Table 1. Characteristics of ZrO_2 +3mol%Y_2O_3 samples

Sample No	Sintering temperature, °C	Density, g/cm^3	Total porosity, %	Open porosity, %
T-11	1100	3.6	42	40
T-14	1450	5.7	6.6	1.5
T-15	1550	5.9	3.3	~0
T-16	1650	6	1.6	

Analysis of the grain size evaluation revealed that with the increase in the sintering temperature significant increase in the grain size occurs. Average grain size of samples sintered at 1450ºC, 1550ºC and 1650ºC was 309±23 nm, 563±52 nm and 1000±74 nm, respectively.

It should be noted that after sintering at temperature of 1100ºC the material is still nanocrystalline, since the average grain size is 85±10 nm (Figure 2).

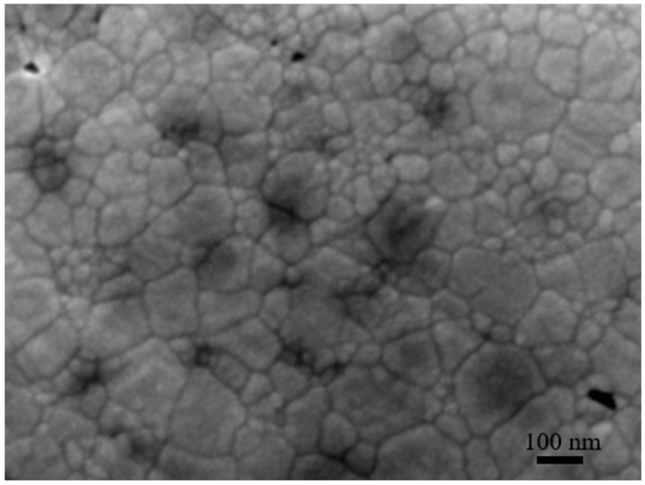


Figure 2. Microstructure of the sample sintered at 1100ºC, 3 hours

Ceramics obtained under these conditions is low-density (3.6 g/cm^3) and remains two-phase: it still consists of both monoclinic ZrO_2-m and tetragonal ZrO_2-t modifications of zirconia, although the content of ZrO_2-t phase increases up to 94.2wt%. Lattice parameters of the ZrO_2-t modification are: a = 0.3607 nm; c = 0.5173 nm and the crystallite size reaches the value of D = 121.7 nm.

In works [5, 6] comparative studies of sintered nanoceramics from partially stabilized zirconia with a similar composition of brands TZ3Y-SE, TZ-3YB (Tosoh, Japan) and Z3Y (BUT, Czech Republic) were carried out. The authors established that low-temperature sintering of ceramics up to 99 % of the theoretical density can be realized for the Z3Y powder at a temperature of 1100ºC/4 hours. At the same time, obtained ceramics remains in nanocrystalline state (grain size is less than 80 nm). However, the sintering of zirconium dioxide nanopowder of the Japanese manufacturer TZ3Y-SE or TZ-3YB at this temperature is insignificant; and increase in temperature to 1460ºC is required to obtain a density of 99 % of the theoretical one. However, the average grain size was already 200 nm [5].

In our case, the difference in the average grain size obtained by the secant method (85 nm) and according to XRD data (121.7 nm) can be explained by the fact that in the secant method the image is analyzed in plane (2D size of grains), while XRD analyzes data in a certain volume of the sample (3D grain size). In [25] similar aspects regarding the average grain size in UO_2 tablets were discussed, and the authors suggested introducing a correction factor of 1.22 to reconcile the measurement results. In addition, the ASTM E112-96 standard introduces a scaling factor of 1.5 for calculating the spatial diameter (3D grain size) [21].

The grain size values, obtained in present study at different sintering temperatures of ZrO_2+3mol%Y_2O_3 ceramics, are shown in Figure 3.

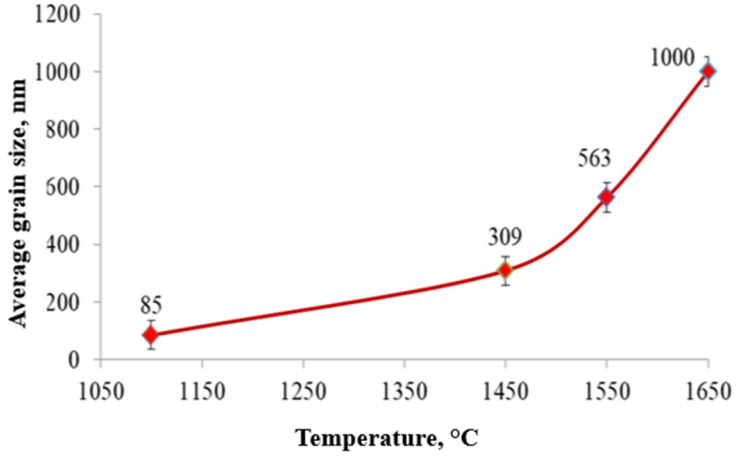
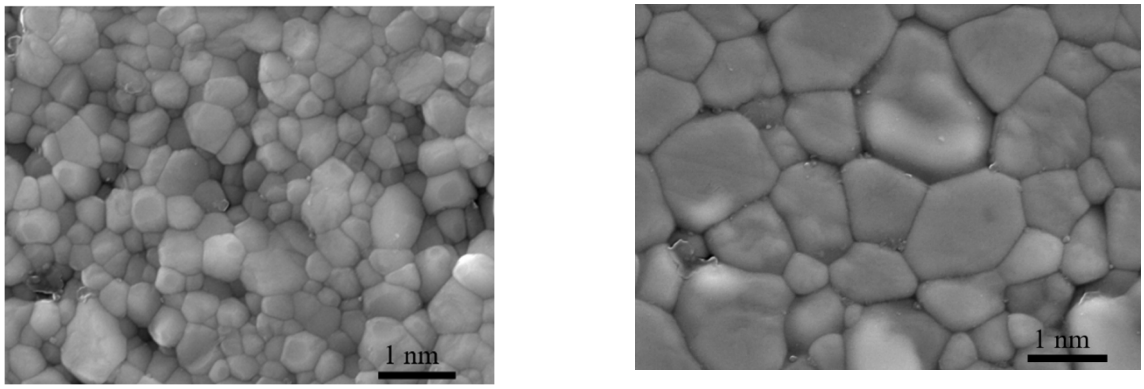


Figure 3. Average grain size values at different sintering temperatures of ZrO_2+3mol%Y_2O_3

As the sintering temperature increases, an active recrystallization process and growth of individual grains occurs, the number of which is small. Thus, sample T-14 is characterized by a fine-grained structure (d_{90} < 500 nm) and the presence of closed pores up to 5 μm in size. The microstructure of sample T-16 is characterized by the predominant grain size d_{90} < 1800 nm, while the maximum grain size is 3.4 μm. The microstructure of these samples is shown in Figure 4.



(a) *(b)*

Figure 4. Microstructure of samples sintered at 1450ºC (a) and 1650ºC (b)

Digital image processing of the T-15 sample microstructure by the graphic analyzer "Thixomet" is presented in Figure 5, and the grain size distribution is shown in Figure 6. The average grain size in this sample is 563 nm, and the maximum grain size is 1.8 μm.

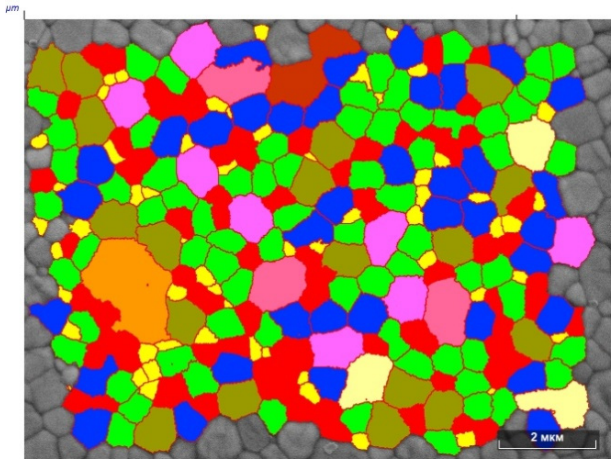


Figure 5. Microstructure of the sample T-15, processed by the Thixomet image analyzer

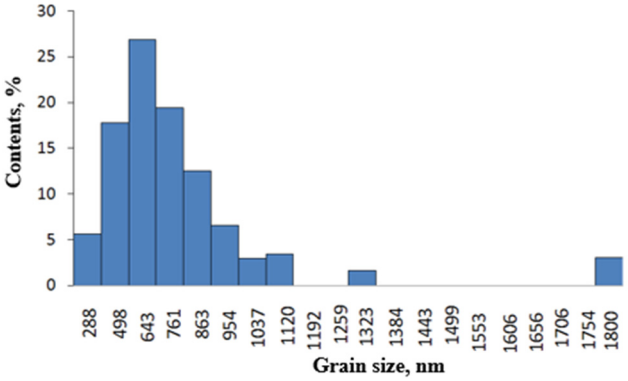


Figure 6. Grain size distribution in the sample T-15 sintered at 1550ºC (calculated by the Thixomet image analyzer)

XRD analysis revealed that samples sintered in the temperature range of 1450...1650°C have an equilibrium structural-phase state: they mostly consist of tetragonal ZrO_2-t modification (85...88)wt%, which is typical for Y-PSZ ceramics [26]. Representative diffraction pattern of samples sintered at temperatures of 1450-1650°C is shown in Figure 7.

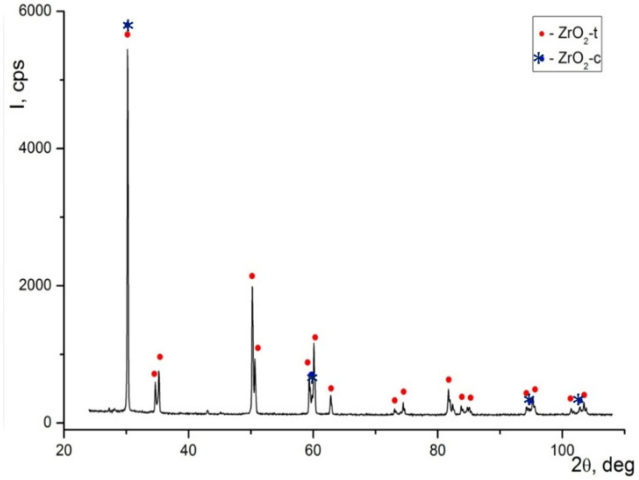


Figure 7. Representative diffraction pattern of samples sintered at 1450...1650 °C

The study of the effect of density and microstructure on some mechanical characteristics of the obtained ceramics consists in determining the critical intensity stress factor (crack resistance K_{1C}) using a technique based on the study of

the characteristics of indentations and cracks, formed by the Vickers diamond pyramid. This technique is characterized by the simplicity of sample preparation and experiment [24-26, 29, 30]. It is known that while loading the surface of ceramic materials with a Vickers indenter, only two types of cracks can be realized: Palmqvist cracks and half-disc (median) cracks [22, 27, 28]. In this regard, a number of researchers propose to use the *c/a* ratio (*c* is the crack length from the center of the indent to the crack tip, and *a* is the half value of the indentation diagonal) to establish the crack type and empirical dependence of processing results. If $c/a < 2.5$, then the cracks are the Palmqvist type, otherwise - median cracks [22]. Another well-known technique of determining the cracks type is to study the cracks after re-polishing the surface of the sample [28, 30]. Interruption of the crack near the indentation corner confirms the presence of Palmqvist-type cracks. In present study, exactly this type of cracks was observed after applied indentation load of 10 kg. The characteristics of the diamond pyramid indentations and cracks for the high-density samples are shown in Table 2.

Table 2. Characteristics of ZrO_2+3mol%Y_2O_3 tablets and cracks

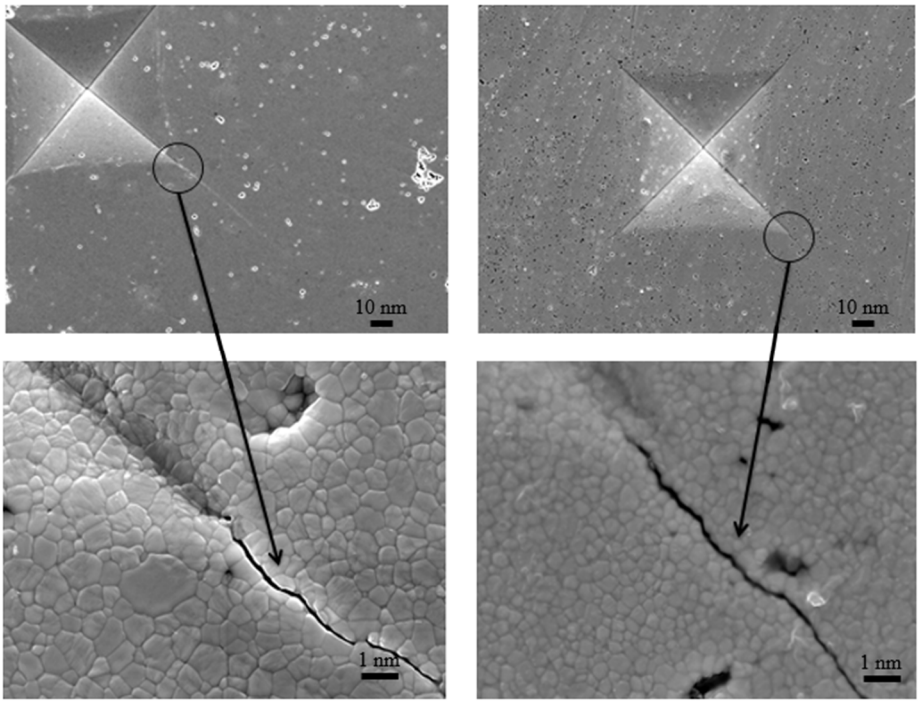
Sample No	Density, g/cm^3	a, μm	c, μm	c/a	HV, GPa	K_{1C}, MPa·m$^{1/2}$
T-14	5.7	65.9 (1.2)	141.6 (2.6)	2.15	11.6 (1.3)	5.52 (0.11)
T-15	5.9	62.0 (0.9)	140.0 (2.9)	2.26	12.8 (0.6)	5.56 (0.13)
T-16	6	62.1 (0.7)	128.9(1.5)	2.08	12.2 (0.6)	6.29 (0.11)

in brackets () the mean square deviation is given

It can be seen that with an increase in the density of the samples, there is an increase in microhardness and crack resistance, which is in good agreement with the results in [30]. Obtained ratio $c/a < 2.5$ also indicates the presence of Palmqvist-type cracks and the correctness of the chosen calculating model for K_{1C}. In a number of works [23, 28] it is shown that the calculated value of K_{1C} using various empirical dependencies (Antists, Casellas, Palmqvist, Niihara models) can be very different. In addition, the value of the applied load on the indenter also significantly affects the resulting value of K_{1C}.

It was found that for samples with a close value of density (5.9...6.0 g/cm^3) and an average grain size that differs by a factor 2 (563 nm and 1000 nm, sintered at T = 1550 °C and 1650 °C, respectively), K_{1C} factor is higher for material with a larger grain size. Obtained results are in good agreement with the published data [24], according to which ceramics with the maximum grain size have higher crack resistance. In above work, a sample of ceramics sintered at 1650 °C/10 hours from the partially stabilized zirconium dioxide powder of brand TZ-3YB (Tosoh, Japan) with a close value of density and grain size (6.08 g/cm^3 and 990 nm, respectively) had the value of K_{1C} = 6.4 MPa·m$^{1/2}$ at applied load of 10 kg; with the increase in the indenter load the value of K_{1C} decreased [24].

Study of the nature of the crack's propagation, coming from the indentation corners, revealed the following. For the sample with an average grain size of 562 nm (sintered at 1550 °C) crack propagation has both intercrystalline and transcrystalline nature (Figure 9a).



(a) (b)

Figure 9. Character of crack propagation in the indentation area of samples with an average grain size of 563 nm (a) and 309 nm (b).

The crack has no significant deviation from its direction. When a large grain is encountered, the latter is destroyed. For the sample with an average grain size of 309 nm (sintering temperature 1450 °C, density 5.7 g/cm^3) the nature of the crack propagation is intercrystalline, since we did not find destroyed grains, which is typical for fine-grained materials in general (Figure 9b).

Thus, the conducted studies showed the possibility of obtaining fine-grained ceramics based on nanosized Y-PSZ powder (ZrO_2+3mol%Y_2O_3) with high physical and mechanical characteristics. Obtained ceramics meet the requirements for the manufacturing of matrices for the incorporation of HLW, as well as for medical and structural purposes.

CONCLUSIONS

Research results showed the possibility of obtaining fine-grained zirconia-based ceramics by cold-pressing and sintering of nanosized Y-PSZ powder (ZrO_2+3mol%Y_2O_3).

The influence of the sintering temperature in the range of 1100...1650 °C on the microstructure of ceramics (ZrO_2+3mol%Y_2O_3) was studied. An increase in the sintering temperature leads to significant increase in the average grain size (from 85 nm to 1000 nm), increase in the density and decrease in the porosity of the samples.

Sintering temperature should be at least 1550...1650ºC to produce dense ceramics (97...98 % of theoretical value). Obtained ceramics is characterized by rather high values of microhardness HV > 12 GPa and crack resistance of 5.5...6.3 MPa·m$^{1/2}$.

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REFERENCES

[1] R. Heimann, T. Vandergraaf, “Cubic Zirconia as a Candidate Waste Form for Actinides Dispositions Studies,” J. Mater. Sci. Lett. **7**, 583-586 (1988). https://doi.org/10.1007/BF01730301

[2] S.S. Khin, *Immobilization of actinide wastes in ceramics for long-term disposal*, (Publikations server der RWTH Aachen University, 2006). http://publications.rwth-aachen.de/record/51927/files/Soe_Khin.pdf

[3] S. Saienko, G. Kholomieiev, Zh. Azhazha, L. Ledovskaia, and A. Pilipenko, “Assessing Efficiency of Radioactive Waste Isolation in Containers of Different Materials,” Nuclear and Radiation Safety, **3**, 36-42 (2015). https://doi.org/10.32918/nrs.2015.3(67).07 (in Russian)

[4] S.Yu. Sayenko, M.M. Belash, E.S. Gevorkyan, T.E. Konstantinova, O.E. Surkov, V.A. Chishkala, I.A. Danylenko, and F.V. Belkin, “Production of nanoceramics based on zirconium dioxide by hot vacuum pressing,” Fizika I tehnika vysokih davleniy, **18**(1), 47-52 (2008). (in Russian)

[5] K. Maca, M. Trunec, P. Dobsak, and J. Svejcar, “Sintering of Bulk Zirconia Nanoceramics,” Rev. Adv. Sci. **5**, 183-186 (2003). https://www.ipme.ru/e-journals/RAMS/no_3503/maca/maca.pdf

[6] K. Maca, M. Trunec, and P. Dobsak, “Bulk Zirconia Nanoceramics Prepared by Cold Isostatic Pressing and Presureless Sintering,” Rev. Adv. Sci. **10**, 84-88 (2005). https://www.ipme.ru/e-journals/RAMS/no_11005/maca.pdf

[7] D. Sanchita, J.W. Drazin, W. Yongqiang, *et al*., “Radiation Tolerance of Nanocrystalline Ceramics: Insight from Yttria Stabilized Zirconia,” Scientific Reports, **5**, 7746 (2015). https://doi.org/10.1038/srep07746

[8] D**.** Sanchita, J. Mardinly, W**.** Yongqiang, *et al*., “Irradiation-induced grain growth and defect evolution in nonocrystalline zirconia with doped grain boundaries,” Phys. Chem, Chem. Phys. **18**, 16921-16929 (2016). https://doi.org/10.1039/C6CP01763K

[9] A.N. Velikodnyi, V.N. Voyevodin, M.A. Tikhonovsky, V.V. Bryk, A.S. Kalchenko, S.V. Starostenko, I.V. Kolodiy, *et al*., “Structure and properties of austenitic ODS steel 08Cr18Ni10Ti,” PAST, 4, 94-102, (2014). https://vant.kipt.kharkov.ua/TABFRAME1.html

[10] I.V. Kolodiy, O.M. Velikodnyi, M.A. Tikhonovsky, V.N. Voyevodin, O.S. Kalchenko, R.L. Vasilenko, and V.S. Okovit, “Microstructure and mechanical properties of oxide dispersion strengthened high-entropy alloys CoCrFeMnNi and $CrFe_2MnNi$,” PAST, 2(132), 87-94 (2021), https://doi.org/10.46813/2021-132-087

[11] S. Ukai, and M. Fujiwara, “Perspective of ODS alloys application in nuclear environments,” J. Nucl. Mater. **307-311**, 749-757 (2002). https://doi.org/10.1016/S0022-3115(02)01043-7

[12] M. Lubszczyk, J. Wyrwa, K. Wojteczko, *et al*., “Electrical and Mechanical Properties of ZrO2-Y2O3-Al2O3 Composite Solid Electrolytes,” J. Electron. Mater. **50**, 5933–5945 (2021). https://doi.org/10.1007/s11664-021-09125-x

[13] R. Parveen, P. Kalita, R. Shukla, *et al*., “Investigation of radiation tolerance of yttria stabilized zirconia in the ballistic collision regime: Effect of grain size and environmental temperature,” **551**, 165344 (2024). https://doi.org/10.1016/j.nimb.2024.165344

[14] M.H. Ghaemi, S. Reichert, A. Krupa, M. Sawczak, A. Zykova, S. Sayenko, K. Lobach, and Y. Svitlychnyi, “Zirconia ceramics with additions of Alumina for advanced tribological and biomedical applications,” Ceramic International, **43**(13), 9746–9752 (2017). https://doi.org/10.1016/j.ceramint.2017.04.150

[15] A.V. Shevchenko, V.V. Lashneva, E.V. Dudnyk, A.K. Ruban, L.I. Podzorova, "Synthesis and physicochemical properties of ceramics from nanocrystalline zirconium dioxide powder,” Nanosystems, Nanomaterials, Nanotechnologies, **9**(4), 881-893 (2011). https://www.imp.kiev.ua/nanosys/media/pdf/2011/4/nano_vol9_iss4_p0881p0893_2011.pdf (in Russian)

[16] A.V. Shevchenko, V.V. Lashneva, E.V. Dudnyk, A.K. Ruban, V.P. Redko, V.V. Tsukrenko, D.G. Verbylo, N.N. Brychevsky, "Manufacturing technology and physicochemical properties of ceramics based on nanocrystalline powder of zirconium dioxide composite,” Nanosystems, Nanomaterials, Nanotechnologies, **12**(2), 333-345 (2014). https://www.imp.kiev.ua/nanosys/media/pdf/2014/2/nano_vol12_iss2_p0333p0345_2014.pdf (in Russian)

[17] W. Li, A. Armani, M.C. Leu, *et al*., "Properties of partially stabilized zirconia components fabricated by the ceramic on-demand extrusion process," in: *Proceedings of the 27th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference Reviewed Paper*, (2016). https://www.researchgate.net/publication/313158956
[18] S.Yu. Sayenko, "Isolation of radioactive waste using hot isostatic pressing," Nuclear and Radiation Safety, **1**(65), 41-48 (2015). https://doi.org/10.32918/nrs.2015.1(65).10
[19] K.V. Lobach, S.Yu. Sayenko, E.O. Svitlichnyi, and O.E. Surkov, "Application of the electroconsolidation method for obtaining ceramics on the basis ZrO_2-3%Y_2O_3," PAST, 76(6), 99-102, (2011). https://vant.kipt.kharkov.ua/ARTICLE/VANT_2011_6/article_2011_6_99.pdf (in Russian)
[20] E.O. Svitlychnyi, "Preparation of ZrO_2-3%Y_2O_3 ceramics from nanoscale powder and study of its properties," Zbirnyk naukovyh prats PAT "UkrNDIvognetryviv im. A.S. Berezhnogo," **113**, 53-57 (2013). http://nbuv.gov.ua/UJRN/vognetryv_2013_113_9 (in Russian)
[21] ASTM E 112-96 Standard Test Method for Determination Average Grain Size.
[22] K. Niihara, R. Morena, and D.P.H. Hasselman, "Evaluation of K_{1C} of brittle solids by the indentation method with low crack-to-indent ratios," J. Mater. Sci. Lett. **1**, 13-16 (1982). https://doi.org/10.1007/BF00724706.
[23] K. Harada, A. Shinya, D. Yokokama, and A. Shinya, "Effect of loading conditions on the fracture toughness of zirconia," Journal of Prosthodomic Research, **57**, 82-87 (2013). https://doi.org/10.1016/j.jpor.2013.01.005
[24] M. Trurnec, "Effect of grain size on mechanical properties of 3Y-TZP ceramics," Ceramics – Silicaty, **52**(3), 165-171 (2008). https://www.ceramics-silikaty.cz/2008/pdf/2008_03_165.pdf
[25] M. Yinbin, M. Kun, M. Laura, *et al*., *Experimental studies of Micro- and Nano-grained UO_2: Grain Growth Behavior, Surface Morphology, and Fracture Toughness ANL/NE-16/12/*, (Nuclear Engineering Division, Argonne National Laboratory, 2016). https://publications.anl.gov/anlpubs/2016/10/130669.pdf
[26] R. Ruh, K.S. Mazdiyasni, P.G. Valentine, and H.O. Bielstein, "Phase relations in the system ZrO_2-Y_2O_3 at low Y_2O_3 contents," J. Am. Ceram. Soc. **67**, 190-192 (1984). https://doi.org/10.1111/j.1151-2916.1984.tb19618.x
[27] G.A. Gogotsy, and A.V. Bashta, "Investigation of ceramics by Vickers diamond pyramid," Problemy prochnosti, **9**, 49-54 (1990). https://dspace.nuft.edu.ua/handle/123456789/9764 (in Russian)
[28] D. Coric, R.M. Manic, L. Curkovic, and I. Zmak, "Indentation fracture toughness of Y-TZP dental ceramics," in: *16th International Conference on New Trends in Fatigue and Fracture (NT2F16)*, (Dubrovnic, Croatia, 2016). https://core.ac.uk/download/pdf/79433762.pdf
[29] A.G. Evans, and E.A. Charles, "Fracture Toughness Determination by Indentation," J. Am. Ceram. **59**, 371-372 (1976). https://doi.org/10.1111/j.1151-2916.1976.tb10991.x
[30] J.-D. Lin, and J.-G. Duh, "Fracture toughness and hardness of ceria and yttria-doped tetragonal zirconia ceramics," Mater. Chem. Phys. **78**, 253–261 (2002). https://doi.org/10.1016/S0254-0584(02)00327-9

ВПЛИВ ТЕМПЕРАТУРИ СПІКАННЯ НА МІКРОСТРУКТУРУ ТА ВЛАСТИВОСТІ КЕРАМІКИ З ОКСИДУ ЦИРКОНІЮ ДЛЯ ПОТРЕБ ЯДЕРНОЇ ЕНЕРГЕТИКИ

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У статті наведено експериментальні результати отримання високощільної кераміки на основі (ZrO_2+3%Y_2O_3), яка є перспективною для використання як матриця зберігання високоактивних відходів. Досліджено вплив температури спікання в діапазоні 1100…1650 °С на мікроструктуру спечених таблеток. Фазовий склад і мікроструктуру дослідних зразків було охарактеризовано за допомогою рентгенівської дифрактометрії (РСА) та сканувальної електронної мікроскопії (SEM). Аналіз розподілу зерен за розмірами проводили з використанням аналізатора зображень "Thixomet". Мікротвердість зразків визначали з використанням металографічного комплексу LECO (США), інвертованого мікроскопа IM-3MET і стаціонарного твердоміра UIT HVB-30. Встановлено, що підвищення температури спікання призводить до суттєвого збільшення середнього розміру зерен (від 85 нм до 1000 нм) і підвищення щільності спечених таблеток. Для отримання щільної кераміки (щільність 97...98% від теор.) температури спікання становили 1550…1650ºС, отримані значеннями мікротвердості склали > 12 ГПа та тріщиностійкості 5,5…6,3 МПа·м$^{1/2}$.

Ключові слова: *оксид цирконію; спікання; мікроструктура; мікротвердість; тріщиностійкість*