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Zirconia ceramics with additions of Alumina for advanced tribological and biomedical applications

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ABSTRACT

The results of an investigation on slip cast and sintered Y_2O_3 (3 wt%)- stabilized ZrO_2 with additions of 5, 10, 15 wt% Al_2O_3 are reported. The surface roughness, porosity and density of the samples were measured. The hardness HRc and Hv, fracture toughness K_{1C}, and friction coefficients were also measured using standard methods. The structural properties of the samples were observed by Scanning Electron Microscopy (SEM). The surface topography was evaluated by means of Chromatic White Light Interferometry using MicroSpy® Topo of FRT Rauheit Kontur before and after tribological tests. The phase and chemical composition were analyzed by X-Ray Diffractometry (XRD), Energy Dispersive X-ray (EDX) spectroscopy, and Raman spectroscopy. Results show that the addition of Al_2O_3 into YSZ ceramics in the range of 5–10% allows the mechanical and tribological characteristics of the material that can be applied in different mechanical machines for different metallurgical processes to be improved, as well as in chemical engineering or medicine.

Keywords: Ceramics, Yttria-stabilized zirconia, composites, Mechanical properties, tribological, performance

1. Introduction

The special properties of ceramic materials are becoming increasingly important for many applications in electrical, chemical and mechanical engineering. Oxide ceramic materials are used in a wide range of industries, including mining, aerospace, electronics, medicine, etc. These materials show a high strength and hardness, as well as a thermal, cracking, and corrosion resistance [1,2]. Alumina (Al_2O_3) and Zirconia (ZrO_2) ceramics have occupied leading places among studied oxide materials.

Alumina-based ceramics favorably combine high strength and good hardness. Ceramics have shown excellent wear resistance, but low fracture toughness parameters. Fracture toughness and the strength of alumina ceramics should be improved significantly by the addition of 10–20 wt% of tetragonal zirconia particles [3]. A fraction of ZrO2 added to Al_2O_3 reduced the incidence of fracture and resulted in a composite material of increased toughness [4].

Yttria-Stabilized Zirconia (YSZ) ceramics are considered as popular engineering materials due to their excellent mechanical properties: good fracture toughness, high strength, elastic modulus, and wear resistance, used in many engineering applications such as engine elements, valves, cutting tools, and moulds [5,6]. In recent years, YSZ with its superior combination of mechanical properties and chemical inertness has been employed in the biomedical field as an implant material [7–9].

Generally, ceramic materials have been presented and considered as the alternative to metal implants in medicine. Among of different solutions, the Zirconia ceramics have received special attentions in dentistry due to their high mechanical characteristics, bioacceptability, colours similarity to the natural teeth and good antibacterial response. The perspective areas of applications of advanced Zirconia ceramics are orthopaedic and trauma surgery (heads of the prosthesis, fragments of skeleton, bone defects elements), and dental medicine (crowns, implants). These applications require the improvement of mechanical strength and tribological performance of ceramic products.

Zirconia-based ceramics can be stabilized in tetragonal or cubic phases depending on dopant concentration (Y_2O_3, MgO, CaO) and temperature during the thermal treatments. The tetragonal-monoclinic (t-m) transformation of ZrO_2 is accompanied by considerable volumetric changes [10,11].

The advantages of the combined superior hardness of Alumina with the high fracture toughness of Yttria-Stabilized Zirconia (YSZ) make

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the Al_2O_3-YSZ system an alternative choice to Alumina and Zirconia monolithic ceramics for structural and functional applications. The effects of the Al_2O_3 content on the ZrO_2 phase composition in the Al_2O_3-ZrO_2 powders were analyzed, demonstrating the major influence of Al_2O_3 additions on the grain growth of Zirconia and the stability of the tetragonal phase of Zirconia [12].

The effects of additives for Zirconia ceramics have been aimed at improving the microstructure and properties. It has been reported [13] that Zirconia-based ceramics with optimized mechanical parameters could be obtained by the addition of more than one stabilizer to Zirconia. The role of MgO and CaO additions in the structural and compositional behavior of ZrO2-Al2O3 composites was analyzed, and has resulted in enhanced features of strength and toughness [14,15].

The increase of hardness with Alumina addition can be explained by some factors, such as the growth restriction of Zirconia grains on sintering and crack deflection mechanisms [16–18]. Previous results [19] confirmed that the Al_2O_3 additions were beneficial for improving the Vickers hardness and fracture toughness of Zirconia sintered at low temperatures.

The mechanical and tribological properties of modern ceramic materials can be significantly improved by the modification of structure parameters and the composition of ceramics. In the process of composite structure formation, the influence of additions on the structure and properties of matrix materials is of great interest.

The aim of this study is to investigate the effect of composition and structural properties of ZrO_2 (3% Y_2O_3) ceramic on the mechanical parameters of the obtained ceramic composites for further tribological tests of the performance of ceramic-bearing surfaces. The analysis was performed for the additions of 5%, 10% and 15% Al_2O_3.

2. Materials and methods

Various types of compaction of ceramics such as die casting of thermoplastic slips or slip casting into plaster moulds are used in practice [20]. An advantage of slip casting into plaster moulds is the possibility of producing products of complex shape and form for various industries.

In this study, Zirconia ZrO_2 (3% Y_2O_3) (Stanford Materials Corporation, USA), with 0.05 µm particles and Alumina Al_2O_3 (Almatis, Germany) with 0.5 µm particles were used as the initial materials. Ultradense ZrO_2 – 3% Y_2O_3 ceramic is produced by the slip casting method with subsequent sintering. Slips were prepared from the powders by adding distilled water and a deflocculating agent. As a deflocculating agent, DOLAPIX grade FF 7 (Germany) was used. The slips were prepared in volume flasks, filtered through a sieve, and kept in a vacuum chamber. Samples of different compositions of ZrO_2 stabilized by Y_2O_3 with additions of 5%, 10%, 15 wt% Alumina (further referred as Z3Y, Z3Y5A, Z3Y10A, Z3Y15A samples) were produced from the slips, using casting into plaster moulds. The samples were dried for 24 h at temperatures of 60–80 °C, then sintered at 1600 °C at the rate of heating and cooling of about 200 °C/h using a laboratory furnace produced by Nabertherm P310 (Germany) and equipped with molybdenum disilicide heaters. The applied method allows us to produce the dense, finely crystalline structure of Zirconia with uniformly arranged disperse particles of Alumina. The diffusion proceeding during the sintering process causes the pores to close up, resulting in the densification of the ceramic material. The matrix grains are grown during the sintering process. The other phases are uniformly distributed between the grains of the main ceramic phase. The sintering process determines the final ceramic grains sizes, as well as the physical and chemical homogeneity. In this way, ceramic discs of 32 mm diameter and 3 mm thickness were prepared for mechanical and tribological measurements. Ceramic discs were sequentially polished with diamond pastes. Each step of the polishing sequence was performed for the same time for all investigated samples.

Next, the structure and chemical composition of the ceramic materials were analyzed by Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray (EDX) spectroscopy (Carl Zeiss AG-Evo-40, Germany, Bruker AXS GmbH, Germany), X-ray micro tomography (Phoenix v/tome/x m, GE Inspection Technologies GmbH, Germany), Raman spectroscopy (Renishaw In Via, S & I GmbH, Germany) and X-ray diffraction (XRD) (Bourevestinik DRON-3, Russia) methods and instruments. Before structural analysis with SEM, the investigated samples were coated with a thin Au film to achieve higher resolution for surface scanning. The porosity and the apparent density were evaluated using standard methods. Determinations of the apparent density and open (closed) porosity of samples after heating at 1600 °C were realized by means of hydrostatic weighing. The roughness parameters were evaluated by means of the Chromatic White Light Interferometry method using MicroSpy® Topo of FRT Rauheit Kontur (Germany). The mechanical properties of the ceramic materials were analyzed by means of Rockwell and Vickers hardness testers (Frank Fischer, Germany). Toughness is a bulk mechanical property of any material, which correlates with its wear resistance. For the determination of the fracture toughness, the K_{1C} testing method was used. For this purpose, three point bending of samples in the form of beams (3.5×5×45 mm) with 0.2 mm edge notch width was applied. Finally, the tribological measurements were carried out by tribometer CH-2000 (CSEM, Switzerland). A diamond counterpart (R=200 µm) was slide against the ceramics. The tests were made under normal load of 50 N, with a sliding speed of 10 mm/min. Friction coefficient (µ) values were continuously monitored during the tests. Wear tracks were analyzed by means of the Chromatic White Light Interferometry method in order to determine the depth of the wear tracks and evaluate the tribological performance of ceramic samples.

3. Results and discussion

The mechanical and tribological characteristics of ceramic materials are determined by the combination of bulk microstructural parameters and surface quality.

The ceramic samples' characteristics after sintering and polishing treatment are shown in Table 1. The optimal values of density and porosity of Zirconia-based ceramic composites have been gained by adding 5–10% Al_2O_3. Further addition of Alumina content in the Zirconia matrix led to a significant decrease of bulk density and an increase of ceramic porosity values.

The results presented in Table 1 include the roughness parameters of Z3Y, Z3Y5A, Z3Y10A, Z3Y15A ceramic samples. The lowest roughness parameters Ra=0.326 µm were gained in the case of 10% Al_2O_3 addition; then, an increase of Alumina content up to 15% caused a significant increase in roughness parameters to values R_a=0.605 µm because of the degradation of mechanical properties and destruction of the sample surface during thermal treatment processing (Fig. 1).

XRD patterns of Z3Y, Z3Y5A, Z3Y10A, Z3Y15A ceramic samples are presented in Fig. 2. As it is shown, there is some difficulty to distinguish the presence of two different phases in ceramic samples. Moreover, in reference [21,22] authors have expressed difficulty in

Table 1
Characteristics of ZrO_2 stabilized by Y_2O_3 ceramics with different contents of Al_2O_3.

Material composition	Roughness parameter of the samples after sintering process (average meanings)		Porosity, %	Apparent density, g/cm^3	Relative density, %
	Ra [µm]	Rz [µm]			
Z3Y	0.435	3.630	1.47	6.01	98,5
Z3Y5A$_3$	0.421	3.311	1.78	5.96	98.2
Z3Y10A	0.326	2.262	1.96	5.87	98,0
Z3Y15A	0.605	5.640	3.39	5.50	96.5

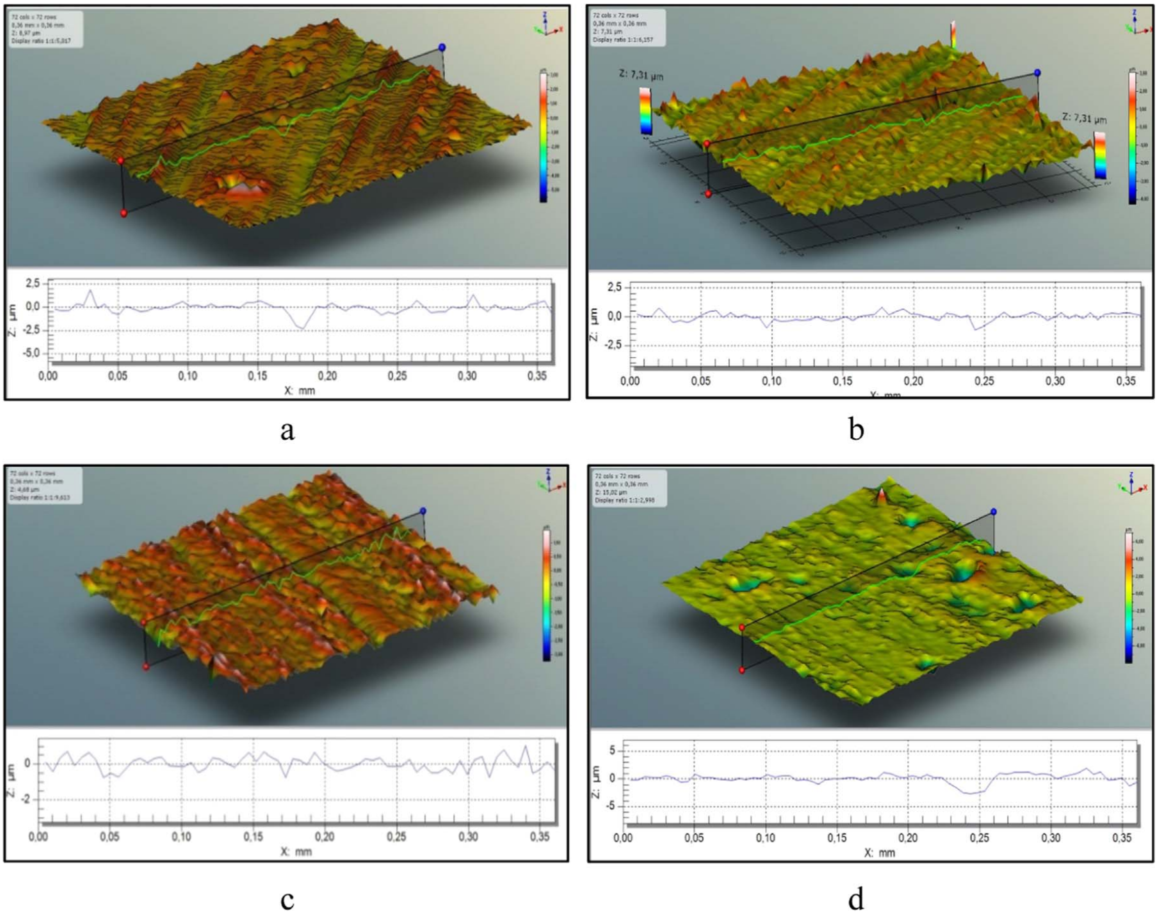

Fig. 1. Surface roughness of Zirconia composite ceramic samples: a. Z3Y, b. Z3Y5A, c. Z3Y10A, d. Z3Y15A.

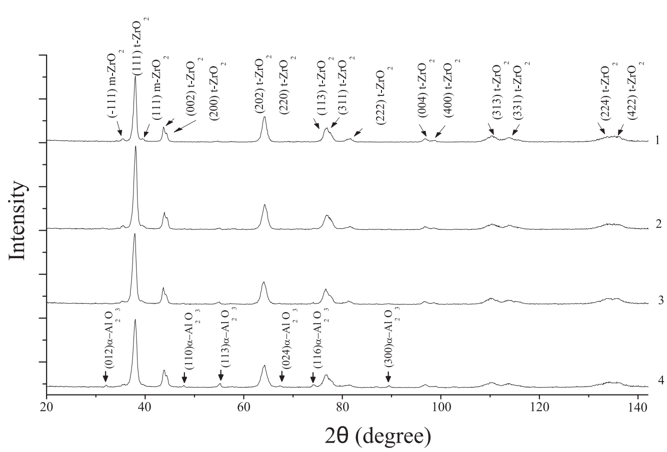

Fig. 2. XRD patterns of ZrO_2 (Y_2O_3)-Al_2O_3-doped ceramics: 1 – Z3Y, 2 – Z3Y5A; 3 – Z3Y10A; 4 – Z3Y15A.

determining the relative contribution of the monoclinic and the tetragonal phase of zirconia, mainly due to the line broadening and the overlapping of Bragg reflections. The intensities of monoclinic peak at 2θ=28.2 and 31.5° and the intensity of tetragonal peak at 2θ=31.5° have demonstrated no principal differences depending on Alumina additions concentration. XRD analysis revealed that some percentage of monoclinic phases (m-ZrO_2) was observed in the tetragonal Zirconia (t-ZrO_2) matrix (up to 5%), indicating good stability, as a result of 3% yttria stabilizing agent with respect to ZrO_2. The addition of Alumina phase clearly modified the relative intensity of the reflection lines. The reflection lines occurring from crystallographic planes related to α-corundum were clearly marked at 2θ=37.9; 43.4; 57.5; 61.3; 68.2; 76.9° for higher Alumina concentrations (Fig. 2). For the concentration range of 5–15 wt% Al_2O_3 the coherent dispersion area of t-ZrO_2 decreased to 14 nm.

Further on, the chemical compositions of ZrO_2 stabilized by Y_2O_3 ceramics with different contents of Al_2O_3 were investigated by the energy dispersive X-ray (EDX) spectroscopy method. The EDX spectra were observed and confirmed the presence of content of 5–15% Al_2O_3

Table 2
Element contents by EDS spectra of Zirconia ceramics.

Material composition	Elements content by X-ray (EDX) spectroscopy					
	Oxygen AN (8) **wt%** error %	Aluminium AN (13) **wt%** error %	Yttrium AN (39) **wt%** error %	Zirconium AN (40) **wt%v** error %	Gold AN (79) **wt%** error %	Total **wt%**
Z3Y	17.69 ± 2.98		7.05 ± 0.25	72.81 ± 2.24	2.45 ± 0.13	100
Z3Y5A$_3$	19.31 ± 3.23	1.93 ± 0.17	6.74 ± 0.24	69.73 ± 2.21	2.29 ± 0.12	100
Z3Y10A	25.44 ± 3.30	3.10 ± 0.29	5.73 ± 0.22	63.40 ± 2.17	2.33 ± 0.13	100
Z3Y15A	25.97 ± 3.37	10.60 ± 0.47	5.45 ± 0..20	55.26 ± 2.15	2.72 ± 0.14	100

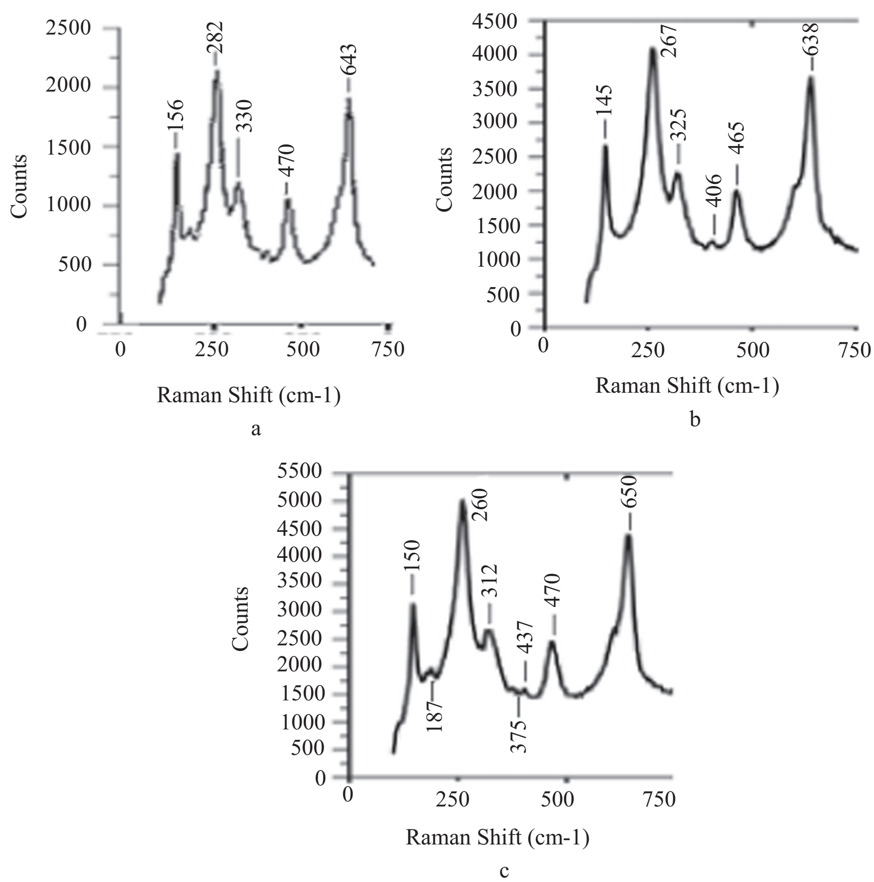

Fig. 3. Raman spectra of Zirconia composite ceramic samples: a. Z3Y, b. Z3Y10A, c. Z3Y15A.

in the ZrO_2 (3% Y_2O_3) matrix (Table 2).

The Raman spectroscopy analysis was performed. In Fig. 3 the ceramics ZrO_2 (3% Y_2O_3) are presented by tetragonal Zirconia (t-ZrO_2) phases. Other modified peaks are not shown in the spectrum of pure Zirconia stabilized by yttria. The ratio of the peak intensity of tetragonal phases (I_{262}/I_{330}) is characterized by the order of the crystal lattice [23], and is about 1.5. In addition, for further tetragonal phase identification, the ratio of peak intensity (I_{262}/I_{643}) is used, about 1.3.

Raman spectra of ZrO_2 (3% Y_2O_3) ceramic samples with the addition of 5–15% Al_2O_3 were analyzed, and are presented as a mixture of monoclinic and tetragonal phases of ZrO_2 [23]. The ratio of peak intensity of tetragonal phases (I_{260}/I_{312}) is 1.6, when I_{260}/I_{650} is about 0.9 in the case of 15 wt% Al_2O_3. Also, in the case of Z3Y15A ceramic new peaks were appeared at 187 cm^{-1} and 375 cm^{-1}. These peaks are usually attributed to representative of the monoclinic spectra [24]. This fact causes the possibility of the t-m phase transformation and monoclinic phase detection on Raman spectra. The main peaks were significantly shifted compared to those of pure Z3Y ceramic samples. The analysis shows that the addition of Alumina to Zirconia causes the formation of crystal lattice distortions in the Zirconia matrix, in particular in O–O bonds. The distortions result in a change in the Zr–O bond length and bond angle in Z3Y as compared to Z3Y15A ceramics with consequent changing in the Raman spectra.

Residual stresses greatly affect the structural properties of ceramic materials. Many parameters can influence on the residual stresses, but the most common approach is associated with differences in thermal expansion coefficients [25,26]. There is not significant mismatch between the coefficients of thermal expansion of Alumina and Zirconia. Other reason of the residual stresses formation, excepting of the thermal mismatch, is the volumetric expansion caused by the martensitic transformation in Zirconia. There is a linear relationship between the degree of Zirconia phase transformation and the residual stresses [27]. During the Zirconia phase transformation process, the volume expansion of transformed zirconia grains is resisted by the incorporated alumina. Possibly, a phase transformation process took place in the Z3Y15A ceramic. The presence of monoclinic phase effects on the residual stress in the Zirconia ceramics and further shifts in the Raman spectra.

The evolution of fractured surface microstructure of ZrO_2 stabilized by Y_2O_3 ceramics with different contents of Al_2O_3 was qualitatively investigated by electron scanning microscopy. Fig. 4 illustrates the microstructure of Z3Y sample after sintering at 1600 ºC and holding time of 1 h. From this figure, it is visible that the Zirconia after sintering is very dense (> 95% of theoretical density) having a granular structure. The grains are well crystallized and have a distinct faceting (Fig. 4). Such a structural formation is typical for 5% and 10% additions of Alumina in Zirconia-based ceramic stabilized by yttria. In the case of Z3Y15A sample, a non-uniform structural formation was observed. The structure changing was correlated with decreasing of density parameters lower than 95% of theoretical density and higher open porosity about 3.39%, measured by standard method according to the values given in Table 1.

The dense and well-grained structure ensures better mechanical properties of materials [28]. A fine-grained structure increases the toughness and improves the wear resistance of ceramics. The size of the grains is also an important factor, which influences the surface finish quality. A fine-grained structure decreases the size of the surface micro asperities after the surface finishing operation. This decreases the friction coefficient.

To quantify the ceramics properties, the effect of crack size on the fracture strength, σ_c, of a ceramic material is expressed by the Griffith equation:

$$\sigma_c = K_{1c}/(Y(\pi\ a)^{½}) \quad (1)$$

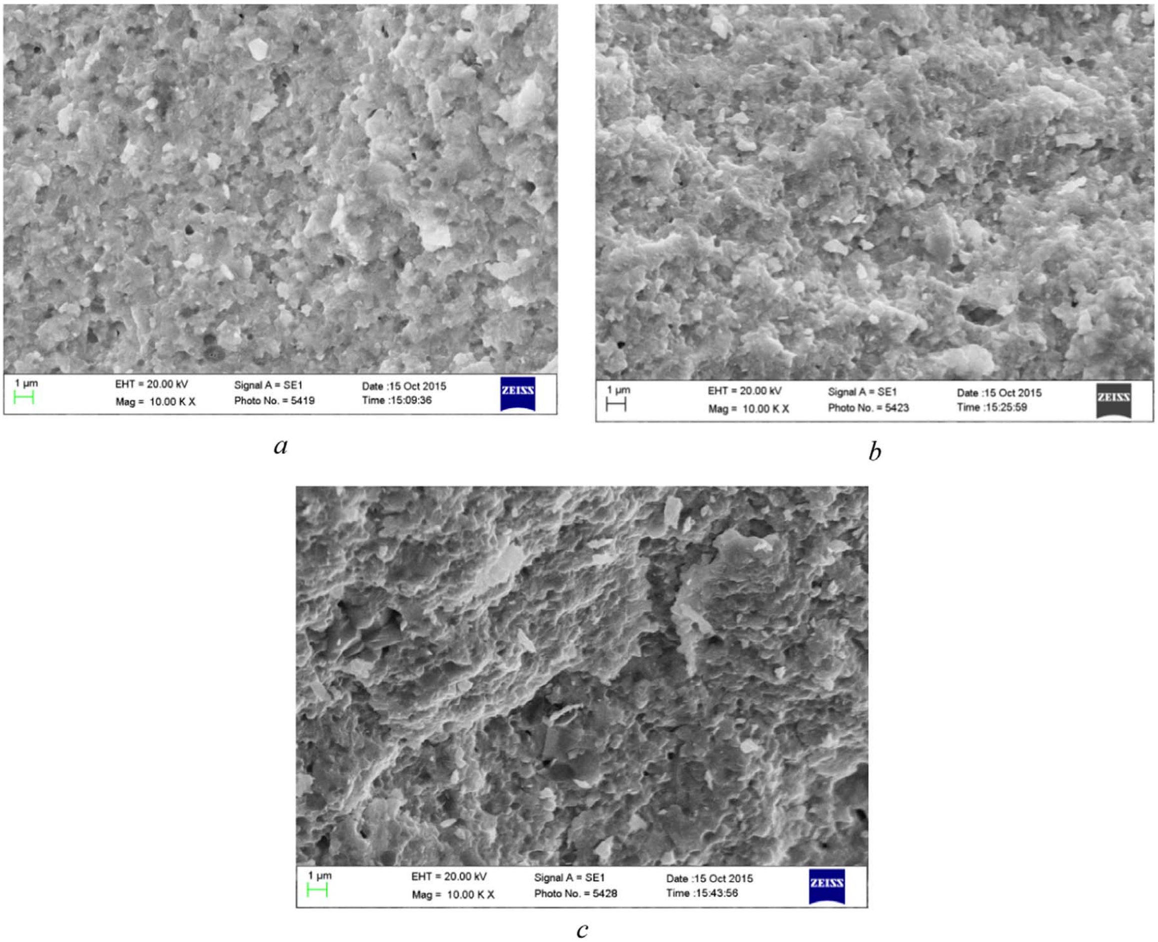

Fig. 4. SEM micrographs of Zirconia ceramics: a. Z3Y, b. Z3Y10A, c. Z3Y15A.

in which K_{1c} is the stress-intensity factor, a is the crack size and Y is the geometry factor. According to the Griffith equation, smaller cracks lead to increasing fracture strength of ceramic materials. The size of the crack is generally proportional to the grain size. A homogeneous distribution of Alumina particles incorporated between the Zirconia matrix causes a decrease in the crack size, consequently increasing the fracture strength [29]. Generally, higher fracture strength causes higher wear resistance [30–33]. The homogeneity of the microstructure allows a fine and uniform surface finish to be created with a low content of surface cracks [34,35].

Moreover, there is a correlation between the mechanical properties and the structure of ceramics. The ceramics with greater density and a fine-grained structure demonstrate better tribological characteristics. The main mechanical and tribological parameters such as hardness, fracture toughness and friction coefficient of the ceramics were also tested, and are presented in Table 3.

The hardness HRc and microhardness Hv parameters demonstrate that the addition of 15% Al_2O_3 to ZrO_2 (3% Y_2O_3) ceramics essentially degrades hardness parameters. The values of hardness HRc decreased from 77.1 to 46.6, and microhardness Hv from 13.74 to 7.80 GPa. Zirconia ceramics doped by 5–10% Al_2O_3 possess hardness parameters close to pure Zirconia samples. These will be favored for material surface treatment during the polishing process and surface finish quality advancing.

The fracture toughness values of the Z3Y, Z3Y5A, Z3Y10A, Z3Y15A samples changed in the range of 4.5–7.0 MPa $m^{1/2}$. The fact that the fracture toughness K_{1c} did not change significantly for 5–10% Al_2O_3 -doped Zirconia ceramics compared to pure ZrO_2 (3% Y_2O_3) was caused by the tetragonal phase stability of the Zirconia matrix [36,37]. On the contrary, the addition of 15% Al_2O_3 leads to significant degradation of the mechanical properties such as hardness and fracture toughness parameters.

Table 3
Mechanical and tribological characteristics of ZrO_2 stabilized by Y_2O_3 ceramics with different contents of Al_2O_3.

Material composition	Mechanical and tribological parameters (average results from 10 tests)			
	Hardness HV [GPa]	Hardness HRc	Fracture toughness, K_{1c} [MPa m½]	Friction coefficient Load 50 N
Z3Y	13.74	77.1	6.9	0.099
Z3Y5A₃	13.43	76.9	6.9	0.091
Z3Y10A	13.39	76.8	7.0	0.0840
Z3Y15A	7.83	46.6	4.5	0.110

Generally, rough ceramic surfaces with relatively large micro asperities cause direct contact between the rubbing surfaces, increasing friction and wear. High-quality surfaces possess low coefficients of friction. Relatively, large values of friction coefficients were correlated with higher roughnesses of samples after sintering and treatment processes, and to the presence of surface non-uniformities, even after polishing. Improving the surface finish quality allows one to gain better tribological characteristics of the ceramics. The samples Z3Y, Z3Y5A, Z3Y10A, and Z3Y15A demonstrate different tribological behavior. For the tested materials, the mean values of the friction coefficient were in the range of 0.099 for Z3Y sample to 0.091 and 0.084 for Z3Y5A and Z3Y10A samples, respectively. The surface profiles of the wear tracks were analyzed by means of the Chromatic White Light Interferometry

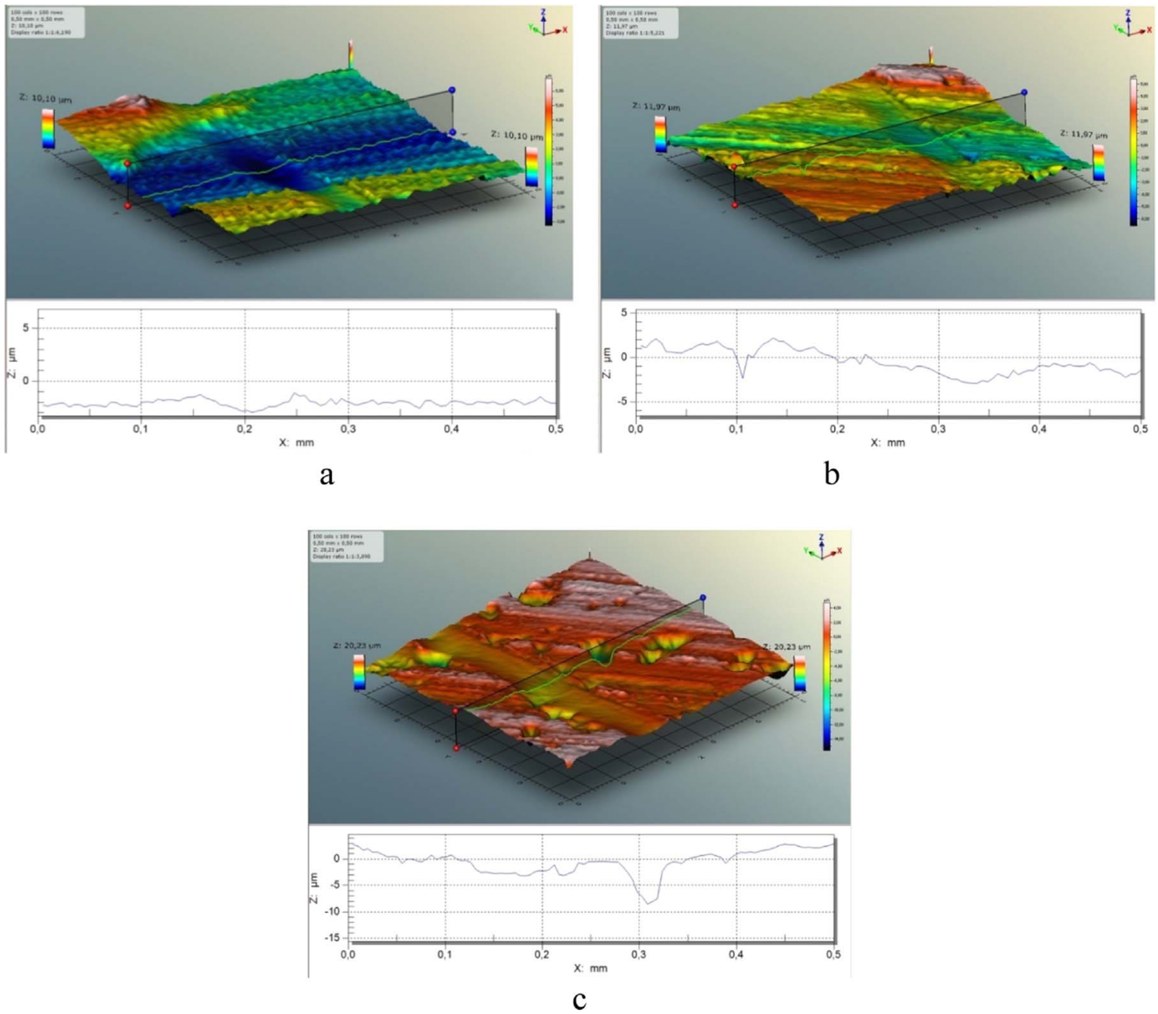

Fig. 5. Surface profiles of the wear tracks of Zirconia composite ceramic samples: a. Z3Y, b. Z3Y10A, c. Z3Y15A.

method after tribological tests. Surface profiles shown in Fig. 5 present the depth of the wear track for ceramics. Sample of Z3Y15A ceramic shows the deepest wear track up to 3.2 µm, compared to the Z3Y and Z3Y10A samples which possess the wear depth about 1,8 µm and 2.0 µm, respectively. The best wear performance was demonstrated in the case Z3Y and Z3Y10A samples in comparison to Z3Y15A ceramic.

For the aforementioned conditions of the experiment, a correlation between the Alumina content in the Zirconia ceramics and further frictional behavior of the ceramic was observed. The wear of brittle ceramics results in a roughening the surface. However, the effect of roughening during friction is lower in toughened ceramics. Better mechanical and tribological characteristics of ZrO_2 (3% Y_2O_3) ceramics with 10% Al_2O_3 additions were gained. After polishing, it has similar values of hardness and fracture toughness, and also lower friction coefficient with decreased roughness compared to pure Zirconia ceramics.

These results show that the addition of 5–10% Al_2O_3 in Zirconium oxide ceramic leads to an improvement in the ceramic treatment conditions with improving structural parameters. The Al_2O_3-doped Zirconia ceramics demonstrate a slight decrease of micro hardness with the reduction of surface roughness parameters from 0.44 µm to 0.33 µm. Further additions of Al_2O_3 (up to 15%) cause a significant decrease of micro hardness parameters from 14 GPa to 8 GPa with the increase of roughness parameters up to 0.65 µm. The addition of Al_2O_3 up to 15% results in a significant decrease of mechanical parameters of the composite Zirconia ceramics. The values of hardness HRc decreased from 77.1 to 46.6, and fracture toughness K_{1c} from 7.0 to 4.5 MPa $m^{1/2}$.

4. Conclusions

The results demonstrate how the mechanical and tribological characteristics of ceramic materials can be improved by choosing the optimal composition of added Al_2O_3 to the ceramic matrix material. The proposed method allows us to produce composite Zirconia ceramics with an ultra-dense structure. The beneficial effect of Al_2O_3 in enhancing of the structural parameters of ZrO_2 (3% Y_2O_3) ceramic has been revealed. The proposed Al_2O_3 -doped YSZ ceramic achieved high density (> 98% of theoretical density) and low porosity parameters. 5–10% Al_2O_3 -doped composite Zirconia ceramics show the improvement of treatment conditions due to the changing of the structural parameters. The ceramics demonstrate lower surface roughness parameters after treatment and polishing processes. Ceramics with 10% Al_2O_3 addition demonstrate optimal mechanical and tribological characteristics, having high values of hardness, fracture toughness, and lower friction coefficient and roughness parameters after polishing. On a contrary, 15% Al_2O_3 -doped composite ceramics demonstrate a significant decrease of hardness with increasing roughness parameters.

Advanced YSZ ceramics with the addition of 5–10% Al_2O_3 are a perspective material for a wide range of industrial applications. The development of new composite ceramics with enhanced tribological characteristics is the main challenge for future micro-bearing and biomedical applications; the results of this study could be helpful in this regard.

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