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Effect of zirconia stabilized by ittria additions on the structure and mechanical properties of alumina based ceramics

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The ceramic samples of pure alumina and alumina with additions of 10, 15, 20 wt. % ZrO_2 stabilized by 5.3 % wt. % Y_2O_3 were produced by slip casting method with the subsequent sintering. The enhancement of ceramic mechanical parameters such as Vickers hardness, bending strength, fracture toughness, thermal resistance, wear resistance due to additions of ZrO_2 (5.3 % Y_2O_3) was observed. The effect of additions of ZrO_2 on the formation of dense, tough and fine-grained structure of alumina ceramics was determined. The best complex of mechanical parameters was obtained on the alumina samples with 15 wt. % ZrO_2.

Керамические образцы алюмооксидной керамики без добавок и с добавками 10, 15, 20 массовых процентов оксида циркония стабилизированного оксидом иттрия, получены методом шликерного литья с последующим спеканием. Показано, что добавки способствовали повышению твердости керамики по Виккерсу, прочности на изгиб, трещиностойкости, термостойкости, износостойкости. Установлено, что добавки ZrO_2 привели к формированию плотной мелкодисперсной структуры. Наилучший комплекс механических свойств демонстрировали образцы керамики, с содержанием 15 масс. % ZrO_2.

Вплив добавок оксиду цирконію стабілізованого оксидом ітрію, на структуру та механічні властивості алюмооксидної кераміки. *Б.Вархолінський, А.Гилевіч, О.Лупіцька, Я.Роховіч, С.Саєнко, Є.Світличний, А.Зикова.*

Керамічні зразки алюмооксидної кераміки без добавок та з добавками 10, 15, 20 процентів оксиду цирконію стабілізованого оксидом ітрію, отримано методом шлікерного лиття з наступним спіканням. Показано, що добавки призвели до підвищення механічних властивостей керамічних зразків, таких як твердість за Віккерсом, міцність на згин, тріщиностійкість, термостійкість, зносостійкість. Доведено, що добавки ZrO_2 призвели до формування щільної, міцної та дрібно дисперсної структури. Найліпшій комплекс механічних властивостей продемонстрували керамічні зразки з вміщенням 15 мас. % ZrO_2.

1. Introduction

The special character of ceramic materials gives rise to many applications in materials engineering, electrical engineering, chemical engineering and mechanical engineering. Ceramic materials are used in a wide range of industries, including mining, aerospace, electronics, medicine, etc. These materials show high strength and hardness, as well as thermal, cracking, and corrosion resistance [1, 2].

Ceramics such as alumina, boron carbide, silicon carbide parts are used in ceramic ball bearings. In very high speed applications, heat from friction during rolling can cause problems for metal bearings; problems which are reduced by the use of ceramics. Various types of compaction, the die casting of thermoplastic slips, the slip casting into plaster moulds are used [3]. An advantage of the slip casting into plaster moulds is the possibility of producing thin wall and complex shape products for various industries. Matrix materials based on alumina composites, reinforced by discrete high-strength filling compounds are challenging for application as sliding friction units of tribo technical systems. Such materials possess high wear and corrosion resistance and antifriction properties. The stability of matrix materials to abrasion and corrosion failure is the key factor for future tribological and mechanical applications. A generally accepted method for composite producing is an introduction of finely disperse ZrO_2 into alumina ceramics and its uniform distribution all over the volume of corundum matrix [4–6]. The tetragonal-monoclinic (t–m) transformation of ZrO_2 is accompanied with considerable volume changes [7]. Owing to the phase transformations of ZrO_2 during the manufacture of ceramics shearing strains resulting in increasing compressive stresses and microcracking in a material take place that hampers development of cracks appearing as a result of mechanical and thermal loadings [8]. On condition that ZrO_2 is retained in metastable tetragonal phase some mechanism becomes apparent based on the transformation of metastable modification of t–ZrO_2 into m–ZrO_2 in front of the top of cracks extending during the service [9]. The modification of structure parameters and composition of novel ceramic composite materials has the significant effect on their mechanical properties and further tribological performance.

Recently the main directions of friction pairs tribological parameters improvement are the advancing of existed sliding coupling characteristics (metal-metal, metal-ceramic, ceramic-ceramic couples) and search for alternative materials (metal, ceramic, coatings) [10, 11]. The aim of the present study was the investigation of the effect of compositional and structural properties of alumina ceramic with 10 %, 15 % and 20 % ZrO_2 (5.3% Y_2O_3) on mechanical parameters of obtained ceramic composites for the further tribological performance of ceramic bearing surfaces.

2. Materials and methods

The base material was alumina oxide powder CT 3000 SG (Almatis, Germany) with a prevailing grain size of 0.5 μm. Additions were zirconia stabilized by 5.3 % Y_2O_3 Stanford Materials Corp (United States) with a prevailing grain size of 0.05 μm. As a deflocculating agent DOLAPIX grade FF 7 (Germany) was used. Slips were prepared from the powders by adding distilled water and deflocculating agent. The slips were prepared in volume flasks, filtered through a sieve, and kept in a vacuum chamber. The samples of different compositions of alumina with additions of ZrO_2 stabilized by Y_2O_3 in the proportion 10, 15, 20 wt. % were produced from the slips, using casting into plaster moulds. The samples were dried for 24 h at temperatures of $T = 60–80°C$, then sintered at a temperature of $T = 1550°C$ with the rate of heating and cooling about 200°C/h using a laboratory furnace equipped with molybdenum disilicide heaters. Diffusion proceeding during sintering process causes the pores to close up resulting in densification of the alumina 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 alumina grains sizes and further the physical and the chemical homogeneity.

The ceramic discs with diameter 32 mm and thickness 3 mm were prepared for tribological and mechanical measurements. The samples of composition Al_2O_3 and Al_2O_3 with 15 % ZrO_2 (5.3 % Y_2O_3) after sintering process are shown in Fig. 1.

The surface roughness parameters were measured by profilometer Hommel Werke T8000. The open porosity and the apparent density were evaluated using standard methods. Determinations of apparent density and open porosity of samples after firing at 1550°C were realized by means of hydrostatic weighing as well as three point bending strength was determined on samples in form of beams 3×4×45 mm^3. Toughness is a bulk material mechanical property, which correlates with its wear resistance. For the determination of the fracture toughness K_{1c} testing method in three point bending of samples in a form of beams 3.5×5×45 mm^3 with edge notch 0.2 mm wide was used. Thermal shock resistance was determined in accordance with ENV 820-3, EN 843-1.

The surface morphology and topography were observed by optical microscopy and electron scanning microscope JSM 5500 LV. The samples were coated by thin Cr film for the best surface scan resolution. The chemical composition of the ceramics was analyzed by energy dispersive X-ray (EDX) spectroscopy (Oxford Link ISIS 300).

To determine the micro hardness the Vickers hardness tester FV-700 was applied. Wear rate and friction coefficient in was assessed using the ball-on-disc system (T-10 Tester) in air conditions and humidity about 50 %. Alumina ceramic balls of 8 mm in diameter were used. The load and sliding speeds were chosen to extract as much as possible information about the materials before its destruction. Total distance was 1500 m, load 20 N, velocity 0.2 m/s, track radius 10 mm, rotational speed 191 rpm. The data were collected continuously by computer acquisition system. The measurements for selected track were done.

3. Results and discussion

Characteristics of friction and wear of ceramic materials are determined by the combination of its bulk microstructure parameters and surface quality. The main characteristics of ceramics are presented in Table 1.

The cleaved surface topography and morphology of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3 were investigated by electron scanning microscope. Figure 2 demonstrates the microstructure of the pure alumina and Al_2O_3 ceramic with 15 % ZrO_2 (5.3 % Y_2O_3), which has a very dense granular structure. Such structure formation was typical for all additions of zirconia stabilized by yttria in alumina-based ceramic.

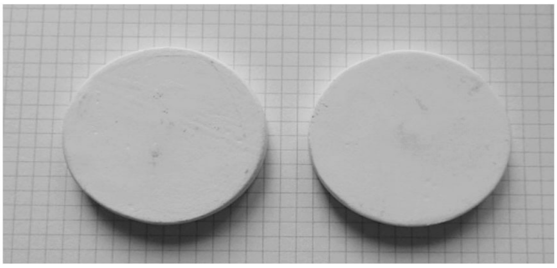


Fig. 1. The samples of composition Al_2O_3 and Al_2O_3 with 15 % ZrO_2 (5.3 % Y_2O_3) after sintering process.

The composition of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3 were investigated by energy dispersive X-ray (EDX) spectroscopy method. The EDX spectra were observed and confirmed the presence of content of 15 % ZrO_2 (5.3 % Y_2O_3) (Fig. 3). Such spectrum was typical for other contents of zirconia stabilized by yttria in alumina-based ceramic.

The average α–Al_2O_3 grain size is about 2–5 μm for pure alumina and fine grains in the range 1–3 μm are presented in the case of alumina ceramics with 15 % ZrO_2 (5.3 % Y_2O_3). The α–Al_2O_3 grains are well crystallized and have a distinct faceting. Crystalline inclusions of ZrO_2 (5.3 % Y_2O_3) are uniformly distributed over the volume of the ceramics (Fig. 2). The dense and well-grained structure is responsible for the best mechanical properties of materials [12]. Fine grained structure results in increased toughness and better wear resistance of ceramics. Grain size also determines the surface finish quality. Fine grained structure allows to decrease the size of the surface micro asperities after the surface finish operation resulting in lower coefficient of friction.

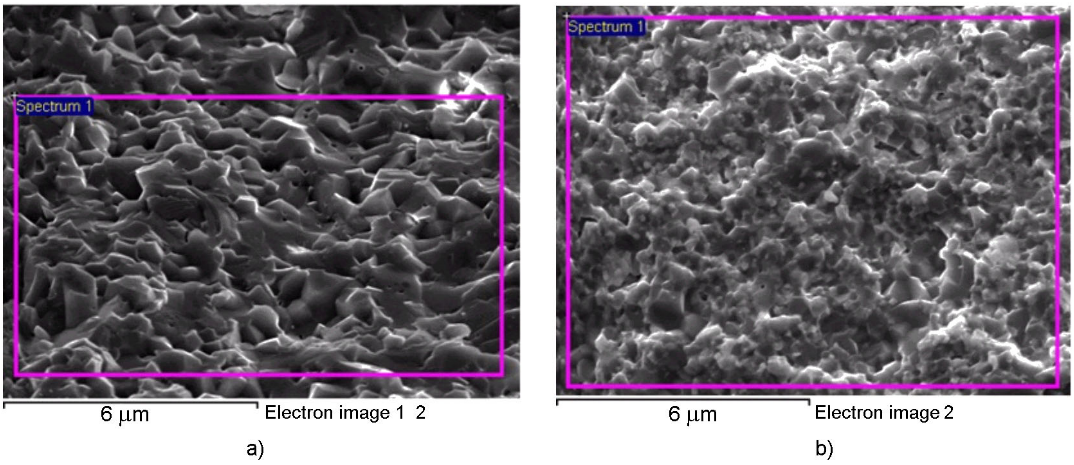
An effect of crack size on the fracture strength σ_c of a ceramic material is expressed by the Griffith equation:

$$\sigma_c = K_{1c}/(Y(\pi \cdot a)^{1/2}),$$

where: K_{1c} — stress-intensity factor; a — the crack size; Y — geometry factor. Ac-

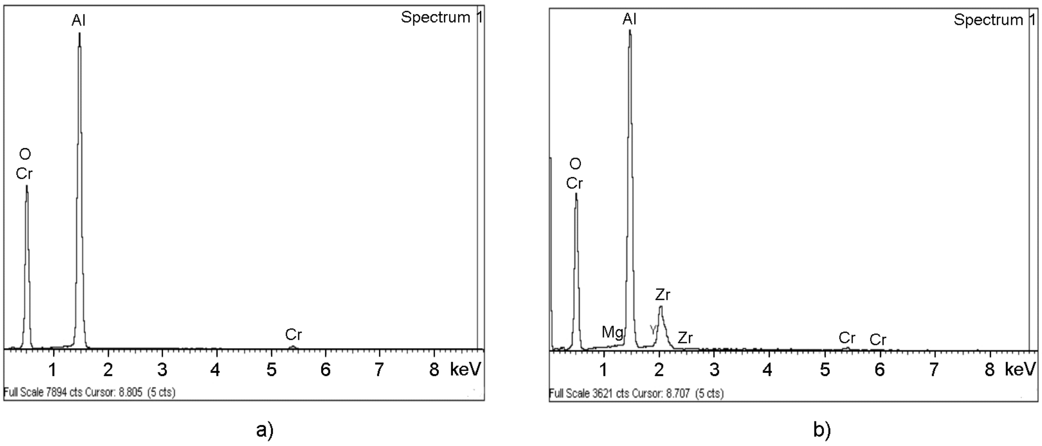
Table 1. Characteristics of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3

Material composition	Roughness parameter of the samples after sintering process (average meanings)		Open porosity, %	Apparent density, g/cm^3	Relative density, %
	Ra [μm]	Rz [μm]			
Al_2O_3	1.067	5.051	0.5–1.5	3.90–3.95	97
Al_2O_3 + 10 % ZrO_2	0.679	3.860	0.2–1.0	4.08–4.12	97
Al_2O_3 + 15 % ZrO_2	0.423	2.542	0	4.20–4.25	98
Al_2O_3 + 20 % ZrO_2	0.509	3.517	0	4.30–4.32	98



a) b)

Fig. 2. The SEM micrographs of alumina ceramics: (a) pure Al_2O_3 and (b) Al_2O_3 with 15 % ZrO_2 (5.3 % Y_2O_3).



a) b)

Fig. 3. The EDS spectra of alumina ceramics: (a) pure Al_2O_3 and (b) Al_2O_3 with 15 % ZrO_2 (5.3 % Y_2O_3).

cording to the equation, cracks of lower size result in increased material toughness and higher wear resistance. The size of crack is generally proportional to the grain size. Homogeneous distribution of zirconia toughening particles incorporated between the matrix particles results in lowering the crack size and consequently in increase of the fracture strength, according to the Griffith equation. Higher fracture strength causes higher wear resistance [13–16]. The homogeneity of the microstructure allows to create fine and homogeneous surface finish with low content of surface cracks [17]. There is the correlation between the mechanical properties and ceramic structure. The additives of zirconia phase in the matrix of alumina improve the mechanical properties of ceramic materials [18–20]. The ceramic with the most perfect fine grained structure and a greater density demonstrates the best tribological characteristics. The mechanical parameters of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3 are presented in the Table 2. The main mechanical parameters such as hardness, bending strength, thermal shock resistance and fracture toughness were increased in comparison with pure alumina ceramics. The enhancement of ceramic mechanical parameters for samples with additions of 10 and 15 wt.% ZrO_2 (5.3 % Y_2O_3) was observed. The further increasing of zirconia content up to 20 wt.% ZrO_2 was resulted in some decreasing of mechanical properties to values some higher than for pure alumina ceramics.

Friction coefficient determined from tribotests was in the range from 0.3 for Al_2O_3 matrix with 20 % and 15 % ZrO_2 (5.3 % Y_2O_3) to 0.5 for pure alumina. Rough ceramic surface with relatively large micro

Table 2. Mechanical characteristics of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3

Material composition	Mechanical parameters (average results 10 tests)			
	Hardness HV [GPa]	Bending strength, MPa	Thermal shock resistance, ΔT, °C	Fracture toughness, K_{1c} [$MPa \cdot m^{1/2}$]
Al_2O_3	15.3	300	300	4.5
Al_2O_3 + 10 % ZrO_2	16.5	430	400	6.5
Al_2O_3 + 15 % ZrO_2	16.8	490	400	6.9
Al_2O_3 + 20 % ZrO_2	15.7	480	400	6.0

asperities causes direct contact between the rubbing surfaces and results in increasing of friction and wear. High quality surface possess low coefficient of friction. Relatively large values of friction coefficients were correlated with high initial roughness of samples after sintering process and presence of surface non- uniformity even after polishing process. The surface finish quality allows to improve the tribological characteristics of alumina. In conditions of the experiment, the correlation was found between the zirconia content in the alumina ceramics and further ceramic behaviour in friction. The wear of brittle ceramics results in roughening the surface. The effect of roughening during friction is lower in toughened ceramics. The abrasive wear was in the range from $0.2 \cdot 10^{-4}$ mm^3/Nm for Al_2O_3 ceramics with 10 % and 15 % ZrO_2 (5.3 % Y_2O_3) to $0.5 \cdot 10^{-4}$ mm^3/Nm in the case of pure alumina. The further increasing of zirconia content up to 20 wt.% ZrO_2 had not significant effect on the tribological parameters of ceramic samples. The contact loading in the joints conditions is approximately 10 MPa and average value of volume wear up to 10^{-3} mm^3/Nm [21]. Therefore, the best mechanical and tribological characteristics for biomedical applications demonstrate Al_2O_3 ceramics with 10 % and 15 % ZrO_2 (5.3 % Y_2O_3) additions which possess higher values of hardness, fracture toughness and lower parameters of friction coefficient, abrasive wear in comparison with pure alumina ceramic.

4. Conclusions

The results demonstrate the advancing of mechanical characteristics of ceramic materials by formation the optimal compound composition of ceramic matrix material with fixed nano structural additions for principal improvement of tribological performance of sliding couples for further micro-bearing applications. Tribotests of ceramics of various compositions were carried out under dry friction conditions. It has been shown that all samples have a high wear resistance, while the Al_2O_3 ceramic with addition of 10 % and 15 % ZrO_2 (5.3 % Y_2O_3) possess the lowest wear rate and the highest hardness parameters. The bending strength and fracture toughness were increased in the case of Al_2O_3 ceramics with different content ZrO_2 stabilized by Y_2O_3. This is due to the fine grained structure of the Al_2O_3 ceramic with 10 % and 15 % ZrO_2 (5.3 % Y_2O_3) and their higher density compared to the others ceramic compositions. Also, the friction coefficient had minimal value in the case of Al_2O_3 ceramic with 15 % ZrO_2 (5.3 % Y_2O_3) additions when compared with other compositions and pure alumina matrix. The formation of innovative bearing surfaces by advancing of ceramic mechanical properties allows to propose novel multifunctional materials for biomedical and micro bearing applications.

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