MECHANICAL AND TRIBOLOGICAL CHARACTERISTICS OF ZIRCONIUM BASED CERAMIC COATINGS FOR MICRO-BEARING APPLICATION

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The application of metal materials with ceramic coatings is the effective way of alternative bearing surfaces formation. The oxide ZrO_2, nitride ZrN and oxynitride ZrON coatings were deposited by magnetron sputtering method on stainless steel (AlSi 316) discs. The adhesion properties, hardness and elastic modulus were evaluated by standard methods. The surface parameters were observed by scanning electron microscopy (SEM). The chemical composition of the coatings was analyzed by energy dispersive X-ray (EDX) spectroscopy. Friction coefficients and wear resistance were measured in the tribological tests. Results show that the mechanical parameters increased in the case of oxynitride in comparison with oxide and nitride coatings.

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INTRODUCTION

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) [1-4]. It is evident that the oxide and multilayer ceramic coatings often exhibit relatively high friction coefficients as sliding against many mating materials. Composite coatings based on Al, Zr, Ti oxides demonstrate unique properties [5-7]: high inductivity, density, bio- and chemical inertness, which are very important for various tribological applications. The ceramic ZrN, ZrO_2 and oxynitride coatings are widely used as protective coatings against diffusion and corrosion. The enhancement of coating's mechanical properties such as hardness parameters, effective Young's modulus, and adhesion strength is very important for wear protection properties and final tribological performance.

The aim of the present study was investigating the effect of compositional, structural and morphological properties of ZrN, ZrO_2 and oxynitride ZrON films on mechanical and tribological characteristics of obtained ceramic coatings for next tribological performance of ceramic coatings on metal bearing devices.

1. MATERIALS AND METHODS

The stainless steel (AlSi 316) discs with diameter 32 mm and thickness 3 mm were used as substrates. The substrates were ultrasonically cleaned in acetone, ethanol and deionised water in sequence and next were dried in a dryer.

The ZrO_2, ZrN and oxynitride ZrON coatings were deposited by magnetron sputtering method in high vacuum pumping system with the base pressure about $1\cdot10^{-3}$ Pa.

The magnetron discharge power was 4...5 kW. Inductive capacitive plasma source for oxygen activation with RF power up to 1 kW was applied. The main parameters of deposition process were the following: Ar pressure $P_{ar}=2.3\cdot10^{-1}$ Pa, oxygen mass flow rate q=35 sccm, nitrogen mass flow rate q=27 sccm, magnetron voltage Um=500...520 V, magnetron current Im=7.0...7.6 A, total pressure $P=(2.8...3.0)\cdot10^{-1}$ Pa, coating deposition rate 6...8 μm/hour. The ion source (type Radical) was applied for cleaning the surface of samples before deposition.

There was the problem of target oxidation during the deposition. Under the excessive oxygen flow conditions the process shifts to the target passivation regime (lower part of Volt-Ampere characteristics (VAC) curves Fig. 1) The sputtering was carried out in the regimes far from the target passivation areas for next oxide coatings deposition with highly stoichiometric composition. Such deposition conditions allowed also increasing the wear resistance properties and avoiding the micro-arcs and micro-drops formation. The optimum conditions were realized for the upper part of VAC curves of magnetron discharge in argon for zirconium target materials (see Fig. 1). In the case of nitrogen gas using, such hysteresis in the form of S-shaped curve similar to the oxygen case was not observed. The optimum conditions were realized for the upper part of curves of magnetron discharge in argon for both oxygen and nitrogen gases.

The coatings thickness was measured by Calotest. The adhesion properties, hardness and elastic modulus were evaluated by standard methods with the use of the Revetest (CSM Instrumets) and the Rockwell test with a diamond type C Rockwell indenter [8]. To determine the micro hardness the Fischerscope® HM2000 tester was applied. The surface roughness parameters were measured by profilometer Hommel Werke T8000.

The surface morphology and topography were observed by optical microscopy and electron scanning

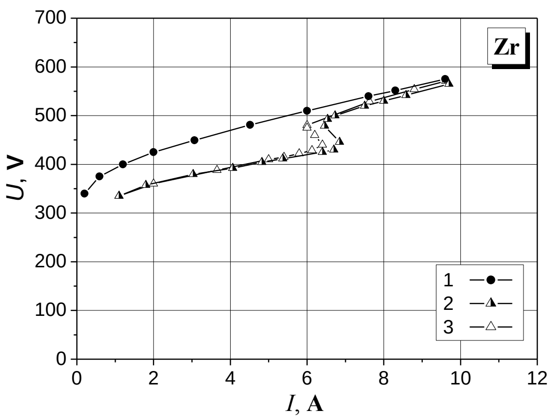

Fig. 1. Volt-ampere characteristics of magnetron discharge in argon with oxygen. The pressure Ar was $P_{ar} = 6 \cdot 10^{-2}$ Pa, oxygen flow: 1 – q=0 sccm; 2, 3 – q=35 sccm for zirconium target material

microscope JSM 5500 LV. The chemical composition of the coatings was analyzed by energy dispersive X-ray (EDX) spectroscopy (Oxford Link ISIS 300). For wear rate and friction coefficient in air measurements the ball-on-disc tests were carried out on a T-10 Tester.

The load and sliding speeds were chosen to extract as much as possible information about the coating before its destruction. The tribological tests were made. Total distance was 1500 m, load 5 N, velocity 0.2 m/s, track radius 10 mm, rotational speed 191 rpm. Alumina ceramic balls of 8 mm in diameter were used.

The data were collected continuously by computer acquisition system. The measurements for selected track were done.

2. RESULT AND DISCUSSION

The surface topography and morphology of ZrO_2, ZrN and oxynitride ZrON were investigated by SEM (Fig. 2). The surfaces of oxide ZrO_2, ZrN and oxynitride ZrON coatings had smooth relief with uniform cross-section structure. The coating's thickness and roughness parameters are presented in Table 1.

The structure and composition of oxide, nitride and oxynitride magnetron sputtering coatings were investigated by energy dispersive X-ray (EDX) spectroscopy method. The EDX spectra were observed. They confirmed the stoichiometric composition of obtained coatings (Fig. 3).

The mechanical properties of the coatings are the hardness H and effective Young's modulus E*.

Table 1. Characteristics of of ZrO_2, ZrN and oxynitride ZrON coatings

Material/Coating type	Roughness parameters		
	Ra [μm]	Rz [μm]	Thickness [μm]
(AlSi 316) / ZrO_2	0.145	0.849	1.0
(AlSi 316) / ZrN	0.088	0.566	0.6
(AlSi 316) / ZrON	0.129	0.807	0.8

Mechanical behavior of the films is characterized by the ratio H/E*[9-11]. The ratio is proportional to the fracture toughness of the film and to the resistance of the material to plastic deformation. It means that the films with enhanced resistance to cracking and plastic deformation should have lower values of effective Young's modulus. There is the correlation between the mechanical properties and coating structure. The formation of oxynitride coatings improves the mechanical properties of coated materials.

The hardness parameters increase in comparison with oxide films and elastic parameters rise in comparison with nitride coatings. The mechanical parameters of ZrO_2, ZrN and oxynitride ZrON deposited on the stainless steel samples (AlSi 316) substrates are presented in the Table 2.

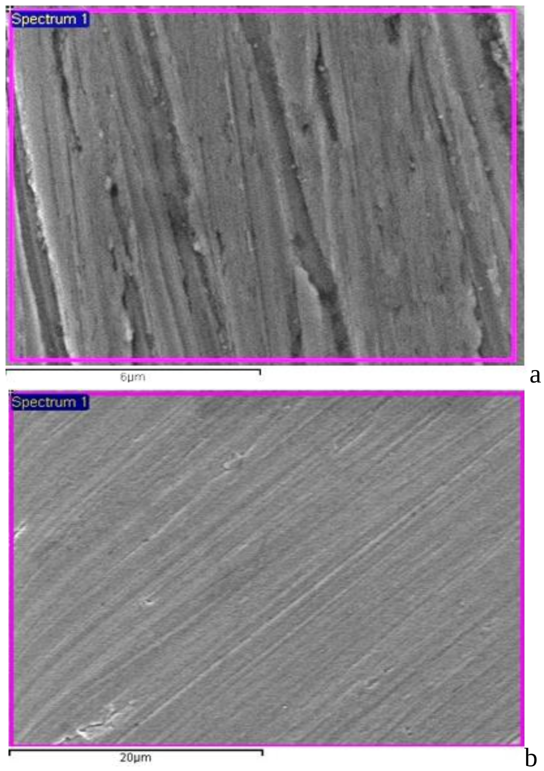

Fig. 2. The SEM micrographs of ZrO_2 (a) and oxynitride ZrON (b)

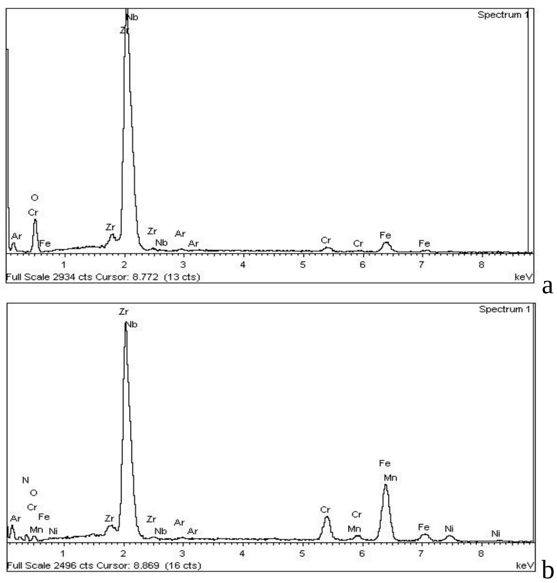

Fig. 3. The EDS spectra of ZrO_2(a) and oxynitride ZrON (b)

Table 2. Mechanical characteristics of oxide, nitride and oxynitride coatings deposited on the stainless steel (AlSi 316) substrates

Material/ Coating type	Mechanical parameters (average results of 10 tests)			
	Hardness H_V [GPa]	Young Modulus [GPa]	H/E*	Adhesion [N]
AlSi 316 / ZrO_2	14.5	133.7	0.11	17.1
AlSi 316 / ZrN	17.6	119.4	0.15	12.7
(AlSi 316) / ZrON	15.7	132.8	0.12	19.6

Friction coefficient was in the range from 0.1 for oxynitride coatings to 0.15 for pure nitride films. Under the conditions of the experiment, the correlation was found between the nitrogen content in the oxynitride coatings and their behaviour in friction.

The wear resistance properties of obtained coatings are very important for bearing applications [12-14]. The abrasive wear was in the range from $0.8 \cdot 10^{-4}$ mm^3/Nm for nitride films to $1.2 \cdot 10^{-4}$ mm^3/Nm in the case of oxynitride coatings. The contact loading in the joints conditions is approximately 10 MPa and average value of volume wear up to 10^{-3} mm^3/Nm [15]. Therefore, the best tribological characteristics for biomedical applications demonstrate oxide and oxynitride films with lower values of friction coefficient and abrasive wear in comparison with nitride coatings.

CONCLUSIONS

The results demonstrate the improvement of tribological characteristics of metal surfaces coated by ceramic coatings. The hardness parameters, toughness and wear resistance were increased in the case of oxynitride coatings. Friction coefficient had minimal values in the case of oxynitride coatings in comparison with nitride films. The formation of innovative bearing surfaces by ceramic coating deposition on metal substrates (stainless steel AlSi 316) allows combining the advantages of ceramic and metal materials for further advanced biomedical and micro bearing applications.

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МЕХАНИЧЕСКИЕ И ТРИБОЛОГИЧЕСКИЕ ХАРАКТЕРИСТИКИ КЕРАМИЧЕСКИХ ПОКРЫТИЙ НА ОСНОВЕ ЦИРКОНИЯ ДЛЯ МИКРОСКОЛЬЖЕНИЯ

Б. Вархолинский, А. Гилевич, О. Лупицка, Я. Рохович, А. Зыкова, В. Сафонов, С. Яковин

Использование металлических материалов с керамическими покрытиями является эффективным способом формирования альтернативных поверхностей скольжения. Покрытия оксидов ZrO_2, нитридов ZrN и оксинитридов ZrON были нанесены на стальные диски (AlSi 316) методом магнетронного напыления. Адгезия, твердость и упругие модули покрытий измерялись стандартными методами. Параметры поверхности оценивались методом сканирующей электронной микроскопии. Химический состав покрытий анализировался методом энергетически дисперсной спектроскопии. Коэффициент трения и износ оценивали в трибологических тестах. Результаты демонстрируют, что механические параметры возрастают в случае оксинитридных покрытий по сравнению с нитридами и оксидами.

МЕХАНІЧНІ ТА ТРИБОЛОГІЧНІ ВЛАСТИВОСТІ КЕРАМІЧНИХ ПОКРИТТІВ НА ОСНОВІ ЦИРКОНІЮ ДЛЯ МІКРОКОВЗАННЯ

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Використання металевих матеріалів із керамічними покриттями є ефективним засобом формування альтернативних поверхонь ковзання. Покриття оксидів ZrO_2, нітридів ZrN та оксинітридів ZrON були нанесені на сталеві диски (AlSi 316) методом магнетронного розпилювання. Адгезія, твердість та пружні модулі покриттів вимірювались стандартними методами. Параметри поверхні оцінювались методом скануючої електронної мікроскопії. Хімічний склад покриттів було проаналізовано методом енергетично дисперсної спектроскопії. Коефіцієнт тертя та знос оцінювали в трибологічних тестах. Результати продемонстрували, що механічні параметри зростають у випадку оксинітридних покриттів порівняно з нітридами та оксидами.