

FORMATION OF MODERN BEARING SURFACES BY CERAMIC COATING DEPOSITION FOR MICRO-BEARING AND BIOMEDICAL APPLICATIONS*

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Abstract. The application of ceramic coatings on metallic materials represents a promising alternative to ball bearing manufacture. The compositional, structural and morphological properties of Al_2O_3 , AlN and nitride/oxide AlN/ Al_2O_3 ceramic coatings deposited on titanium and stainless steel alloys were examined by XRD, XPS, SEM, AFM methods. Abrasion tests were also conducted by a micro-scale abrasion tester. A correlation between the mechanical properties and coating structure was observed. The bilayer AlN/ Al_2O_3 coatings demonstrate the minimal values of the total surface abrasive wear and increased wear resistance in comparison with uncoated substrates and monolayer Al_2O_3 , AlN coatings. The enhancement of the mechanical properties of the coatings is very important for the tribological performance of ceramic coated ball bearing devices.

Key words: magnetron sputtering, bilayer coating deposition, mechanical properties, tribological performance, bearing surfaces.

1. INTRODUCTION

The efforts directed to improving the tribological parameters of human joints have recently emphasized the advancement of the joint's sliding coupling characteristics (metal-metal, metal-ceramic, ceramic-ceramic couples) and the search for alternative materials (metal, ceramic, coatings) [1-3]. At the main cases of metal-on-metal couples applications the mixed lubrication of friction surfaces

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takes place. Ceramics, such as alumina parts, are used in ceramic ball bearings. In some applications, heat generated due to friction during rolling can cause problems for metal bearings; these problems can be reduced by using ceramics. Ceramics are also more chemically resistant and can be used in wet environments where steel bearings would rust. In many cases electrically insulating properties of ceramic materials may also be valuable in bearings. Furthermore, the use of ceramic materials allows one to improve the joint wettability characteristics and fluid friction conditions [4]. The ceramic materials possess high hardness, wear resistance parameters and biocompatibility in comparison with metal elements; however, there is the risk of brittle failure of ceramic heads [5, 6]. Other negative results of metal and ceramic materials application are the accumulation of toxic wear debris in the surrounded implant tissues and the following dissemination of wear particles to the internal organs: liver, spleen, kidneys [7–10]. In this respect, nano structured coatings based on Al, Zr, Ti oxides exhibit unique properties [11–13], such as high inductivity, density, bio- and chemically inertness, which are very important for the future biomedical applications. Ceramic AlN, Al₂O₃ and oxynitride coatings have been widely used as protective coatings against diffusion and corrosion. Most of our previous investigations have dealt with the corrosion resistance characteristics of oxide, nitride and multilayer coatings [14, 15]; however, few data have been reported on the wear resistance properties [16]. The application of metal materials with ceramic coatings is an effective way of alternative bearing surfaces formation. The mechanical properties enhancement, such as hardness parameters and toughness characteristics of the coatings, is very important in view of the wear protection properties and the final tribological performance.

2. MATERIALS AND METHODS

The aim of the present study was the investigation of the effect of compositional, structural and morphological properties of Al₂O₃, AlN and nitride/oxide AlN/Al₂O₃ films on the mechanical and tribological characteristics of the prepared ceramic coatings.

The titanium-based alloy Ti (Ti4Al6V) and SS stainless steel (1H18N9) were used as substrates. These alloys were selected because of their being the most popular load-bearing materials. The process of Al₂O₃, AlN and nitride/oxide AlN/Al₂O₃ coating deposition by magnetron sputtering method was performed in a high vacuum pumping system at a base pressure of about 10⁻³ Pa.

The magnetron discharge power was 4–5 kW, the power of the activated oxygen source was up to 1 kW, the Ar pressure $p_{Ar} = 1.8 \cdot 10^{-1}$ Pa, the oxygen mass flow rate was $q = 30$ sccm, the nitrogen mass flow rate was $q = 23.5$ sccm. The magnetron voltage was $U_m = 570$ V, the magnetron current was $I_m = 8$ A, the total

pressure was $p = 2.2 \cdot 10^{-1}$ Pa, with the coating deposition rate being $8 \mu\text{m}/\text{hour}$. An ion source was used for cleaning the samples' surface before deposition.

At excessive oxygen or nitrogen flow conditions, the process shifts to the so-called target-poisoning mode. Thus, we conducted the sputtering process in regimes far from the target-poisoning mode in view of nitride/oxide coatings deposition with a highly stoichiometric composition. Also, such deposition conditions allow one to avoid micro-arcs and micro-drops formation that would worsen the corrosion-resistance properties.

Figure 1 presents the current-voltage characteristic of the magnetron with target of aluminum in a mixture of argon with oxygen or nitrogen at various reactive gas mass flow rates. All current-voltage curves are S-shaped and consist of a transition region and two saturation regions: the higher one for pure argon, the lower one being related to the target-poisoning mode at sufficiently high mass flow rates of reactive gases (Fig. 1a, b).

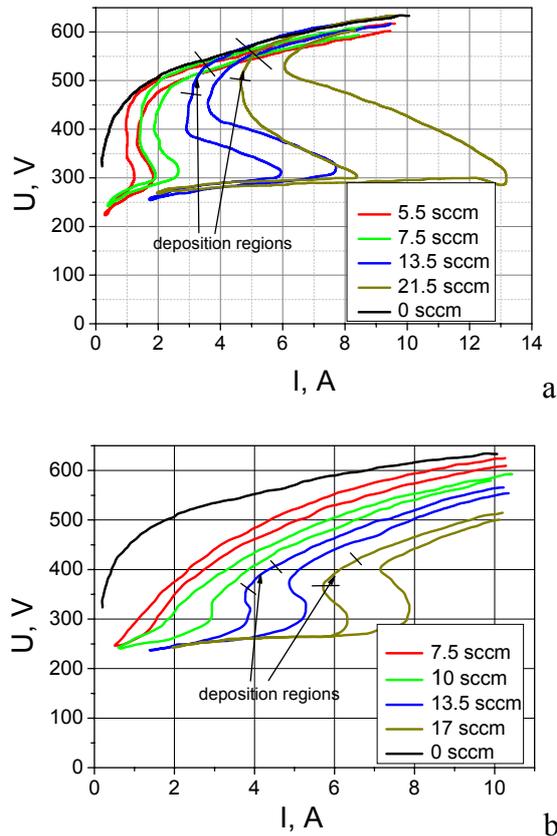


Fig.1 – Current-voltage characteristics of magnetron discharge for different mass flow rates of oxygen (a) and nitrogen (b), argon pressure $p = 1.8 \cdot 10^{-1}$ Pa.

In the transition region, a hysteresis effect is observed. As the reactive gas flow is increased, the width of the hysteretic loop increases, too. The optimum conditions were realized in the upper part of the magnetron-discharge current-voltage curves in argon for both oxygen and nitrogen gasses.

The coatings' adhesion properties, hardness and elastic moduli were evaluated by standard methods with the use of the Revetest (CSM Instruments) and the Rockwell indenter with a tip radius 200 μm , within the load range of 200N. The surface roughness parameters were measured by a profilometer (Hommel T-2000). The roughness values were in the range R_a/R_z 0.037/0.240 μm for Al_2O_3 , 0.053/0.350 μm for AlN and 0.048/0.310 μm for nitride/oxide AlN/ Al_2O_3 coatings. The coating surface structure and morphology were estimated by means of scanning electron microscopy (SEM, JEM 2100) and atomic force microscopy (AFM, Quesant Instrument Corporation, USA) methods.

The mechanical properties of the coatings are the hardness H and the effective Young's modulus E^* . The mechanical behavior of a film is characterized by the ratio H/E^* [17, 18]. This ratio is proportional to the fracture toughness of the film and the material's resistance 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 coatings' structure. The bilayer coatings' formation increases the values of H , H/E^* and the mechanical properties of the nitride/oxide films.

The mechanical parameters of the Al_2O_3 , AlN and the nitride/oxide AlN/ Al_2O_3 coatings deposited on the SS and Ti alloy substrates are presented in the Table 1.

Table 1

Mechanical characteristics of ceramic coatings deposited on the SS and Ti alloy substrates

Material/Coating type	Mechanical parameters (average results 10 tests)		H/E^*	Adhesion [N]
	Hardness H [GPa]	Young Modulus [GPa]		
SS/ Al_2O_3	9.7	174.7	0.057	43.1
SS/ AlN	14.3	184.4	0.078	50.3
SS/ AlN/ Al_2O_3	12.9	178.6	0.071	45.9
Ti/ Al_2O_3	9.2	170.3	0.052	37.1
Ti/ AlN	13.8	183.1	0.075	47.2
Ti/ AlN/ Al_2O_3	12.5	177.9	0.069	40.7

To measure the wear resistance of the coatings, abrasion tests was performed on a CAT-S-AE (CSM Instruments) micro-scale abrasion tester. The ball was a micro-blasted 25 mm diameter hardened steel sphere (SAE 52100, 61 ± 2 HRC, $R_a = 2.5 \pm 0.3 \mu\text{m}$), while the abrasive slurry was a suspension of diamond particles

(mean size 2-4 μm). The ball rotational speed was set to provide a linear velocity of 0.1 m/s in all tests. The total abrasive wear of the ceramic coatings at abrasion action conditions was estimated according to the methods presented in [19, 20].

3. RESULTS AND DISCUSSION

The structure of the oxide, nitride and bilayer ceramic coatings was investigated by means of X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) methods. The XRD profiles of magnetron sputtered Al_2O_3 coatings demonstrated an amorphous structure, any peaks were not observed. The AlN coatings shown a lower crystallinity with two main peaks at $2\theta = 38.5$ and $2\theta = 44.7$ degrees, which were associated with reflections from the (111) and (200), correspondently (Fig. 2). The texture of AlN coatings was formed mainly by c-AlN (JPCDS 25-1133) crystallites oriented in $\{111\}$ direction. The compositional analysis of coatings by means of XPS method was made. The high-resolution photoelectron spectra of Al2p, O1s, N1s were observed (Fig. 3). The chemical composition was found to be close to stoichiometric composition.

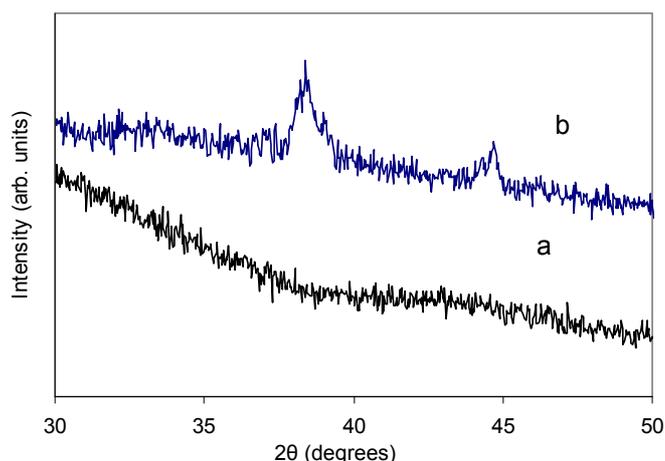


Fig. 2 – X-ray diffraction profiles of Al_2O_3 (a) and AlN (b) films.

The Al2p peak with binding energy position $E = 73.9$ eV, which is corresponded to the AlN composition was observed. For the AlN/ Al_2O_3 structure there was the shift of the Al2p peak to position $E = 75.1$ eV. The N1s peak was observed at binding energy $E = 396.2$ eV, associated with Al-N chemical bond. The O1s high-resolution spectra demonstrate the peak at binding energy position $E = 531.2$ eV, associated with Al-O chemical bond.

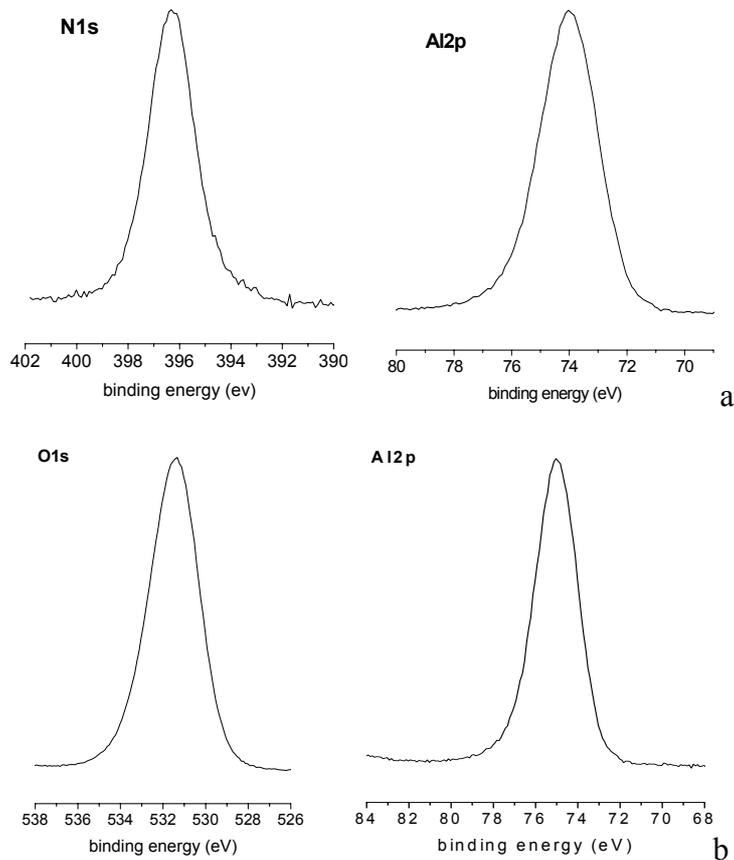


Fig. 3 – High-resolution photoelectron spectra of AlN (a) and AlN/Al₂O₃ (b) films.

The ceramic coatings with different morphological features were formed by the deposition conditions varying. The surface of the oxide Al₂O₃ coatings had a smooth relief with a dense and uniform cross-section structure. The AlN coatings had a crystalline structure with a crystalline-phase cross-section (Fig. 4). The surface morphology of the deposited oxide Al₂O₃ and bilayer AlN/ Al₂O₃ coatings was characterized by AFM method (Fig. 5). The observed changes of the coatings' structure result in some mechanical properties changes.

The total abrasive wear of ceramic coatings at abrasion action conditions is presented (Fig. 6). The bilayer coatings deposited on Ti alloy substrates exhibit an increased wear resistance in comparison with both uncoated substrates and monolayer oxide and nitride coatings.

The character of the abrasive effect on the coatings' structure is illustrated by the micrographs (Fig. 7).

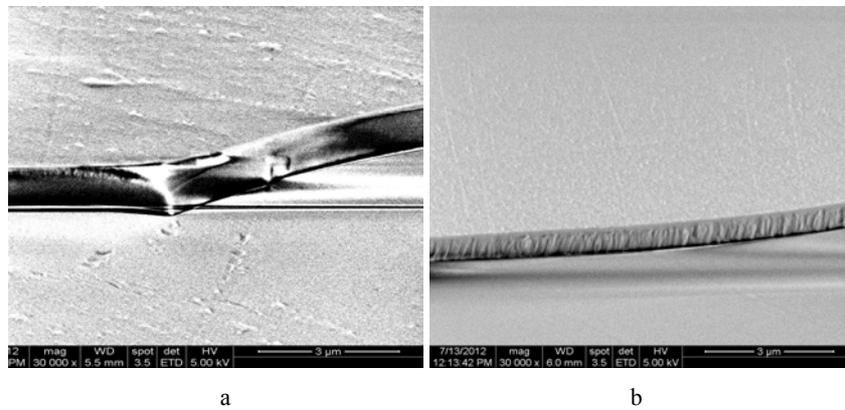


Fig. 4 – SEM micrographs of cross section of Al₂O₃ (a) and AlN (b) films.

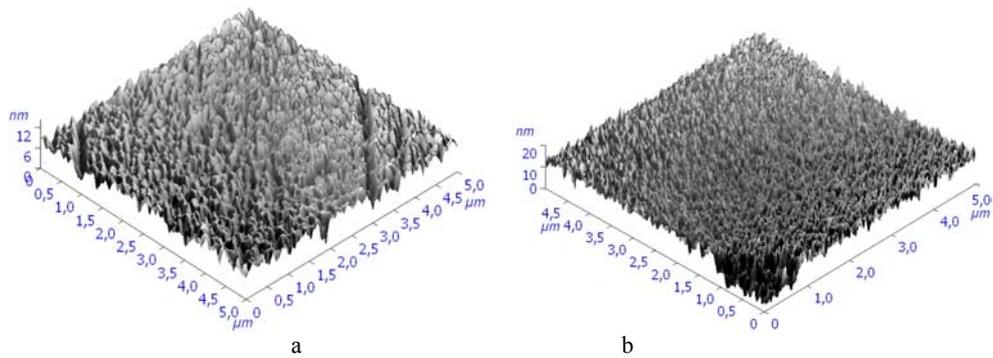


Fig. 5 – AFM images of the surface structure of the deposited oxide Al₂O₃ (a) and bilayer AlN/ Al₂O₃ (b) coatings.

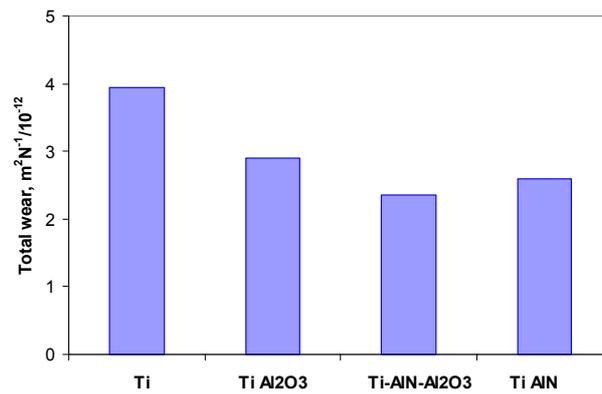


Fig. 6 – Total surface abrasive wear of Al₂O₃, AlN and AlN/Al₂O₃ coatings deposited on Ti alloy substrates.

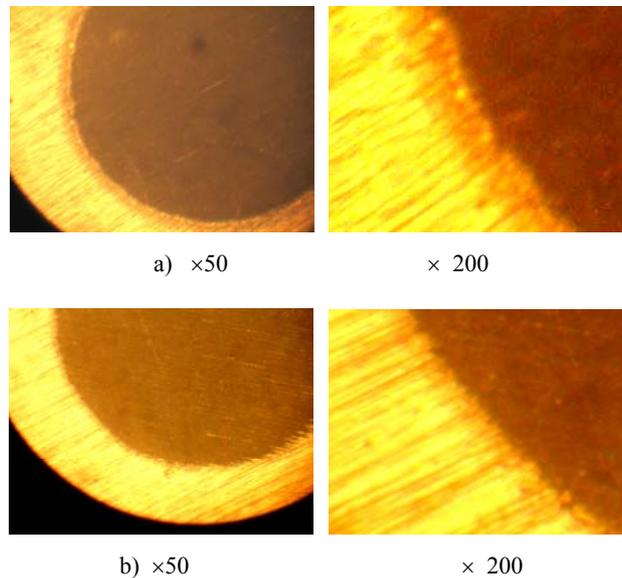


Fig. 7 – Micrographs (optical microscope, magnification $\times 50$, $\times 200$) of abrasive wear damage (top view of the wear crater) of $\text{AlN}/\text{Al}_2\text{O}_3$ (a) and Al_2O_3 (b) ceramic coatings deposited on Ti alloy substrates.

The results for stainless steel samples confirm the main results obtained in the case of Ti alloy substrates. The $\text{AlN}/\text{Al}_2\text{O}_3$ ceramic coatings deposited on stainless steel substrates demonstrate minimal total surface abrasive wear and increased wear resistance in comparison with uncoated substrates and Al_2O_3 , AlN coatings. The mechanical properties' enhancement, such as hardness, toughness and wear resistance of the nitride/oxide coatings, may prove to be very important for many tribological applications.

4. CONCLUSIONS

The development of novel low-cost innovative bearing surfaces poses challenges in view of the future micro-bearing, joints replacement and other biomedical applications. The formation of advanced bearing surfaces by ceramic coating deposition on metal substrates (stainless steel 1H18N9, titanium Ti6Al4V alloys) allows one to combine the ceramic materials' inertness with the hardness and failure strength of metals. Our results demonstrate the improvement of the tribological characteristics of metal surfaces coated by ceramic coatings. The deposition of bilayer coatings strongly increased the hardness of films (from 9 GPa to 14 GPa) and the ratio H/E^* (from 0.05 to 0.08). The wear resistance properties were improved in the case of nitride/oxide $\text{AlN}/\text{Al}_2\text{O}_3$ coatings in comparison with uncoated substrates and both mono-layer oxide and nitride coatings.

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