АНАЛИЗ ТОПОГРАФИИ ПОВЕРХНОСТИ ОКСИНИТРИДНЫХ ПОКРЫТИЙ ТИТАНА МЕТОДОМ АТОМНОЙ СИЛОВОЙ МИКРОСКОПИИ

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ANALYSIS OF THE SURFACE TOPOGRAPHY OF TITANIUM OXYNITRIDE COATINGS BY THE METHOD OF ATOMIC FORCE MICROSCOPY

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In this paper we investigate the surface topography of coatings based on oxides and dioxins-titanium hydrides obtained by reactive sputtering, the analysis of roughness parameters, chemical composition and surface topography described.

Introduction. Today, the diseases of the cardiovascular system are reported to be the leading cause of human deaths. The most promising treatment for such diseases is coronary artery stenting. Frequently used metal for such stents is stainless steel (316L). Steel is well suited for mechanical properties, and shows sufficient corrosion resistance [1]. However, when using stainless steel the potential release of nickel ions, chromium, and molybdenum from the stent may cause allergic reactions, neointimal hyperplasia, and restenosis [2]. Therefore, the problem of biocompatibility of stainless steel remains relevant at present.

Surface modification of stents by coating formation is the most common way to overcome this problem. Much attention today is paid to the thin film coatings based on the titanium oxynitrides due to their ability to improve *the* thrombogenicity as well due to their high hemocompatibility [2,3].

The material of the coatings changes its properties depending on the N/O ratio in TiN_xO_y composition, that makes it promising for various applications. These coatings are used for coronary stents in vascular surgery. The coatings must be intact, with no cracks and defects, have good mechanical characteristics, and adhesion *strength* with a thickness of several hundred nanometers.

Among the methods of preparing such coatings, the dominants are: ion-plasma methods, sputtering with ion bombardment, arc ion plating, spraying, plasma immersion deposition [3]. Each method has its advantages and disadvantages. Reactive magnetron sputtering method is one of the most promising methods for the preparation of coatings based on titanium oxynitride. It delivers controlled chemical composition of the coating using inexpensive starting materials (metals and gases) of high purity.

Materials and methods The laboratory magnetron sputtering setup UVN - 200MI made at Tomsk Polytechnic University was used for deposition of Ti-O-N coatings [4]. Oxygen (O_2) or mixture of oxygen and nitrogen (N_2) were used as working gases. The material of the substrates is stainless steel 316L. The *spraying* *modes* are: the material of the cathode is Ti, the working pressure in the chamber is $P=10^{-1}$Pa , power - p=1kW , current I=3A, leakage rate of the working gas is 5ml/min, and the bias voltage is varied from 0V to *minus* 100V. The ratio of partial pressure of the gases is p (O_2)/p (N_2) = 1/1, deposition time t_1=90min and t_2=180 min. Centaur research complex is used to study the topography of the coatings by atomic force microscopy (AFM) and to define their surface chemical composition by Raman spectroscopy (LLC "Nano Scan Technology", Russia). The thickness of the coatings is measured by ellipsometry method using a "Ellipse - 1891 SAG" complex.

Experimental results and conclusions. This study focuses on the coating specimens with different chemical composition (oxides and oxynitrides of titanium) . The coating thickness is about 200 nm. The test specimens were divided into groups : Group number 1 is the specimens coated with TiO_2: bias voltage U_{bias} = -100 V, pure O_2, deposition time *t*=180 min, (Figure 1.a) , group number 2 is the specimens coated with titanium oxynitride TiON (*t* = 90min , U_{bias} = minus 100V , p(O_2)/p(N_2) = 1), and group number 3 is TiON specimens (*t* = 90min U_{bias}= minus 100V , p(O_2)/p(N_2) = 1/3). Determination of the topography and surface quality of the specimens was performed using atomic force microscopy (AFM). Surface roughness profile was built with an accuracy of 1nm in the “Origin” program (fig.1.b. and 2.a., b), showing that all coatings have developed a fragmented structure.

The analysis of the roughness parameters describing the morphology of the surface and allowing to quantitatively describe its geometry using GOST 2789-73, GOST 25142-82 (ISO 4287/1-1997), shows that the surface of the specimens number 1 and number 2 after the coating deposition is characterized by the parameters corresponding to the two scale levels (bimodal distribution).

It was found that the average roughness of the samples of the group number 1 on the first scale level (R_a^I) was 17 nm, and those of samples of group number 2 (R_a^{II}) was 6 nm, respectively.

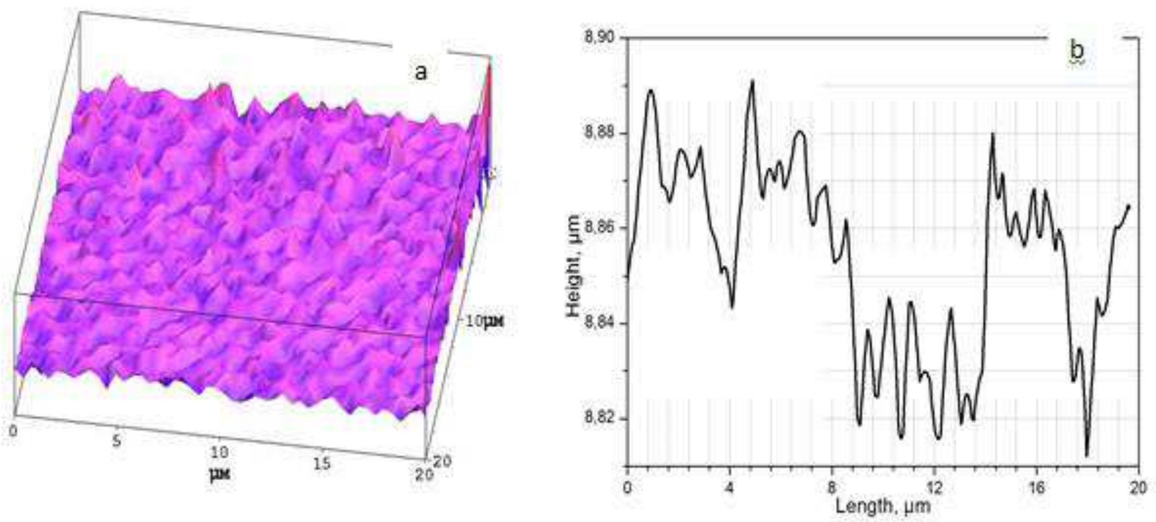

Fig.1. 3D AFM image of the sample group number 1 (a),
the roughness profile of the sample group number 2 (b)

Particular attention should be given to the S_m parameter which is an average period of the surface profile at different levels of scale that make up the first large-scale level S_m^I = 14 microns(μm), and the second - S_m^{II} = 1,4 μm, respectively. Roughness class is 13.

In accordance with the obtained data, the same calculation of roughness parameters can be performed for the group of specimens’ number 2 and 3. Average roughness , which on the first scale level for the specimens of the group number 2 (R_a^I) was 23 nm , and on the second scale level (R_a^{II}) - 5 nm , the average period of the surface profile is S_m^I = 5,8 µm, and S_m^{II} = 2,5 µm, on the first and second scale level respectively. For the specimens of the group number 3 (R_a^I) was 35 nm , and for the second (R_a^{II}) - 15 nm , the average period of the surface profile is S_m^I = 11,16 µm. Roughness class is 13. In addition, the calculated asymmetry parameter (R_{sk}), which describes the symmetry of the scatter profile relative to the midline. For samples № 1, № 2, and number № 3 on the first large-scale levels received R_{sk}^I = 0,019, R_{sk}^I = 1,46, R_{sk}^I = 1,45 respectively. In all cases obtained R_{sk} <| 1.5 |, that uniquely describes the topography of the specimen surface.

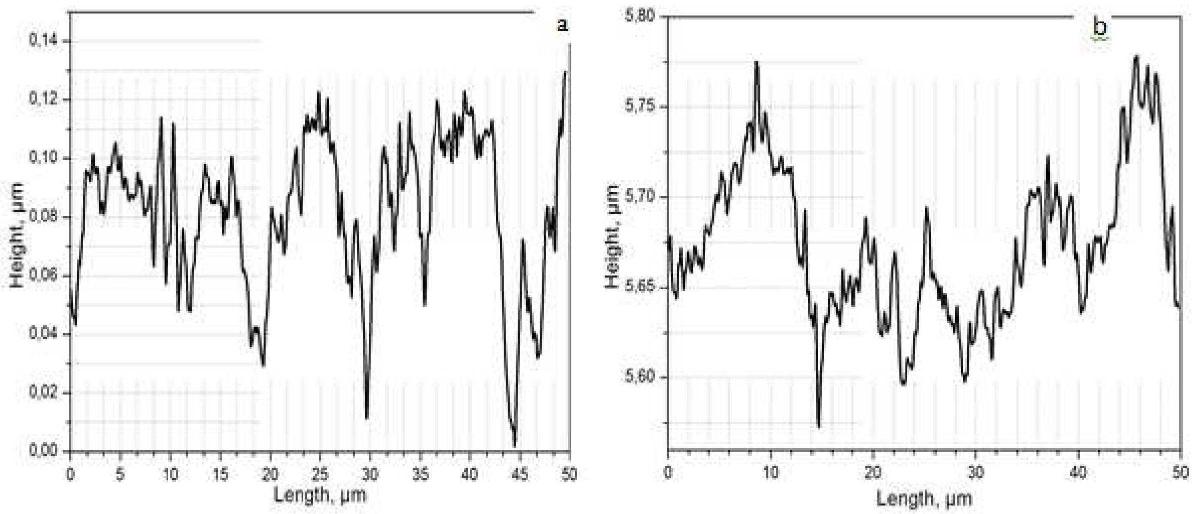

Fig. 2. Profile of surface roughness samples number 2 (a) and number 3 (b)

Thus, according to the analysis results of the is specimens № 1, № 2, and number 3, surface topography is characterized by a bimodal distribution of roughness parameters, which suggests a complex heterogeneous surface structure. In addition, the elements of the large scale level are formed by the smaller fragments.

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