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МОРФОЛОГИЯ ПОВЕРХНОСТИ ПОКРЫТИЙ ОКСИДОВ И/ИЛИ ОКСИНИТРИДОВ ТИТАНА, ОСАЖДЕННЫХ МЕТОДОМ РЕАКТИВНОГО МАГНЕТРОННОГО РАСПЫЛЕНИЯ

Г.В. Арышева, Е.А. Мазурик, А.А. Пустовалова

Научный руководитель: профессор, д.ф.-м.н. В.Ф. Пичугин

Национальный исследовательский Томский политехнический университет,

Россия, г. Томск, пр. Ленина, 30, 634050

E-mail: mazurik_ea@mail.ru

THE SURFACE MORPHOLOGY OF TITANIUM OXIDE AND OXYNITRIDE COATINGS DEPOSITED BY REACTIVE MAGNETRON SPUTTERING

G.V. Arysheva, E.A. Mazurik, A.A. Pustovalova

Scientific Supervisor: Prof., Dr. V.F. Pichugin

Language Advisor: L.V. Maletina, Assistant Professor

Tomsk Polytechnic University, Russia, Tomsk, Lenin str., 30, 634050

E-mail: mazurik_ea@mail.ru

This article presents the results of a study of the surface morphology of biocompatible ca-Sydney and / or titanium oxynitride coatings on stainless steel substrate 12X18H10T, co-torye were synthesized by reactive magnetron sputtering

Cardiovascular diseases are one of the leading causes of death, so the problem of their treatment is topical. Coronary stenting (stent placement in the narrowed location of the vessel) is one of the effective treatments. The problems of *biocompatibility, hemocompatibility and thrombogenicity associated with the surface characteristics*: the roughness, electric potential and free energy [1] are *keys ones*. The control of these characteristics of the surface is important for biocompatibility of the stent with the vessel walls.

The surface roughness of the stent connected with its structure which is critical for interaction with the blood. Coatings based on titanium oxides and oxynitrides are optimal for stents with inorganic coatings on the modern stage. The aim of this work is the analysis the topography of the coatings surface depending on the modes of their deposition.

The titanium oxides and oxynitride coatings were produces by reactive magnetron sputtering deposition with the hand-made installation "UVN-200MI" in Tomsk Polytechnic University. Stainless steel 12X18H10T was used as a substrate. Stainless steel 12X18H10T refers to austenitic, corrosion-resistant steels has high strength characteristics, *resistance to adverse conditions* and many kinds of acids [2]. The mixture of oxygen (O₂) and/or nitrogen (N₂) with different ratios was used as working and reactive gases. Sputtering conditions were: cathode -

Ti of VT-1.0, the operating pressure in the chamber - 0.1 Pa, power - 1 kW, current - 3 A, leakage rate of the working gas - 5ml/min, deposition time - 90 min, bias voltage from 0 to -100 V, the distance between the substrate and the magnetron - 100 mm. Ratio of partial pressure of the gases N_2 and O_2 : $p(O_2)/p(N_2)=1/3$ и $p(O_2)/p(N_2)=1/1$. All *samples* can be divided into groups according to the mode used: group № 1 - samples coated with titanium dioxide, group № 2 – coated titanium oxynitride with the ratio of partial pressure $p(O_2)/p(N_2)=1/3$, group of samples №3 - the coatings of titanium oxynitride with a ratio of partial pressure $p(O_2)/p(N_2)=1/1$. The *samples* of Group №№ 1-3 were deposited *under conditions* of grounded substrate ($U_{bias}=0$ V). The group of samples №4 (coated with titanium dioxide), № 5 (coated with titanium oxynitride with the partial pressure ratio $p(O_2)/p(N_2)=1/3$), and №6 (the coatings of titanium oxynitride with a ratio of partial pressure $p(O_2)/p(N_2)= 1/1$) – were deposited *under electrical bias* $U_{bias} = -100$ V.

The surface morphology and elemental composition of the coatings was *studied* by scanning electron microscope (SEM) ESEM Quanta 400 FEG from FEI with integrated EDX-analyzer (EDS analysis system Genesis 4000, S-UTW-Si (Li) detector) in Germany at the University of Duisburg-Essen, operating under high vacuum (10⁻⁵ Pa). SEM-image analysis and calculation of the sizes of the fragments *in the form of islands* (“grains”) constituting the coating were carried out by *secant method using the* Adobe-Photoshop program. All 6 groups of samples were used as objects of research on SEM-microscope.

It was found that the surface of the samples for which coatings were deposited under the bias voltage $U_{bias}=-100$ V have blurred outline that doesn't allow a standard research.

SEM image of the surface shown in *Figure 1* demonstrates that the surface coating is homogeneous, has a complex topography, without visible defects (cracks, pores, craters, etc.). The elements of the coating surface are small clusters in the form of *islands* with the *size* of several nanometers and more – agglomerates representing fragments of dome-shaped form. It is seen from the *SEM image* in *Figure 1a* that the islands have two level of *size*: about several nanometers and about of several hundred of nanometers which have a fine structure. A histogram showing the particle size distribution is represented in *Figure 1b*. These results *confirm* the bimodal distribution of the *island sizes*. Bimodal distribution of sample №1 is characterized by sharp peak with the maximum at 14 nm and a small broad peak with the maximum at 312 nm. Average particle size on the entire section of distribution is 116 nm.

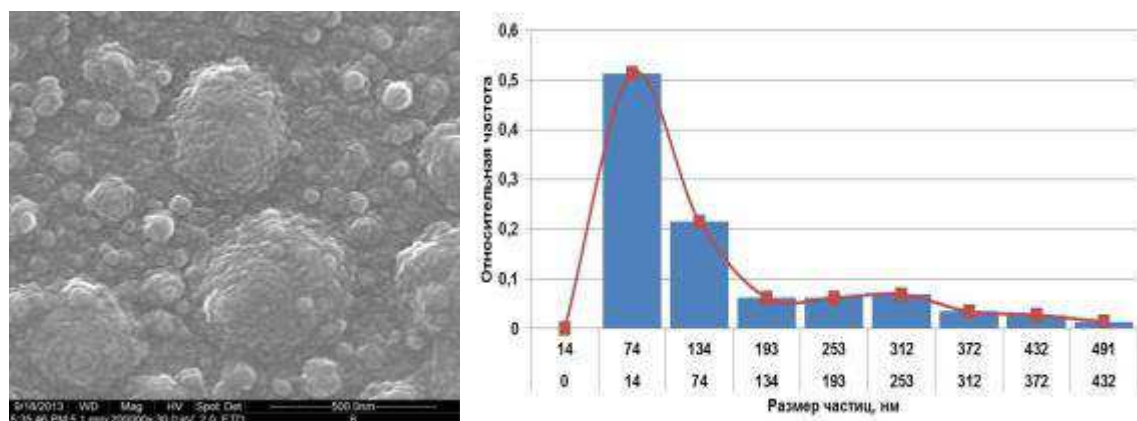


Fig. 1. SEM-image of the surface of the sample №1 (a) and the histogram of the distribution of particle sizes (b)

The surface morphology of the samples №2 and №3 is homogeneous, has developed relief and dispersion-distributed on the surface particles. A more detailed analysis of the surface morphology made by scanning

electron microscopy shows that the size of the structural elements of the coating depends on the deposition parameters

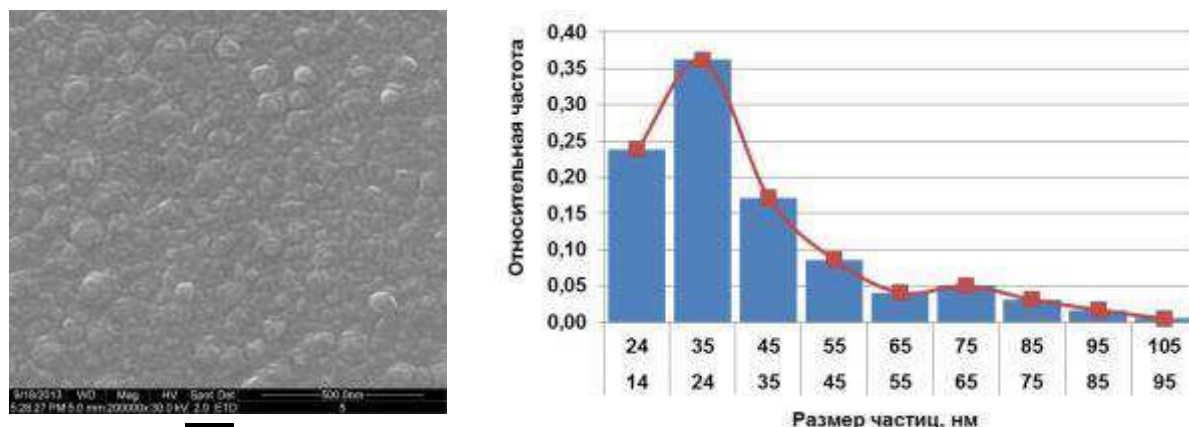


Fig. 2. SEM-image of the surface of the sample №2 (a) and the histogram of the distribution of particle sizes (b)

At the histogram in the Figure 2b it can be seen a distinct peak of particle size at 35 nm, and wide and less one at 75 nm. From this it can be assumed that in this case there is also a bimodal distribution of the fragments. Average particle size on the entire section of distribution is 31 nm

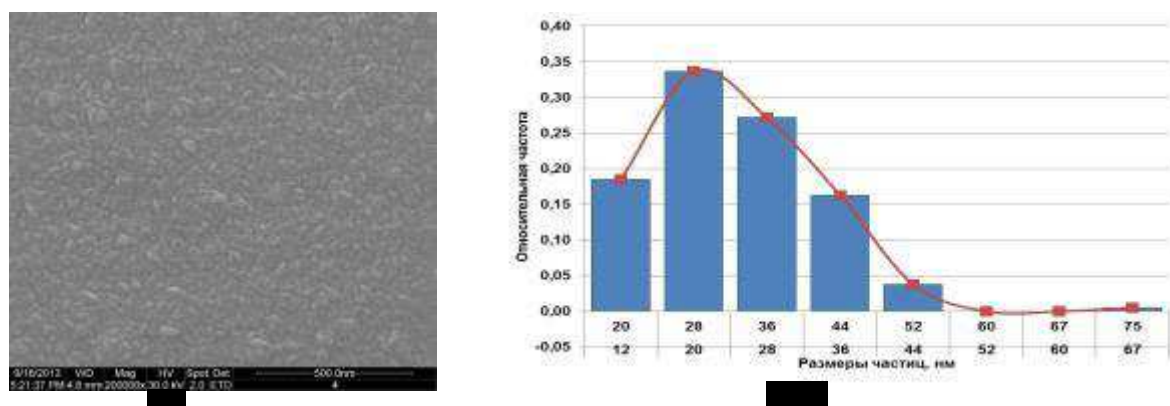


Fig. 3. SEM-image of the surface of the sample №1 (a) and the histogram of the distribution of particle sizes (b)

However, in the case of samples of group №3 (Fig. 3, b) a single broad peak in the range 10 ÷ 50 nm is observed. So we can conclude that this distribution is close to the unimodal [3]. Average particle size is 28 nm.

Thus, the demonstrated data show that the grain size distribution and the particle structure are strongly depends on the modes of the coating formation.

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