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Hydrophilic properties of surface of nanostructured tantalum films and its oxynitride compounds

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Abstract. The structure and hydrophilic properties of tantalum coatings on stainless steel and glass slide were investigated using atomic force microscopy (AFM) and sessile drop method. Coatings are characterized by a cellular structure. As a result of the modification, the hydrophobicity of the sample surfaces is increased.

1. Introduction

The various types of materials are used for the production of metal stents: stainless steel, platinum-iridium alloys, tantalum, cobalt-chrome alloys, etc. [1, 2]. Implants used in cardiovascular surgery should not cause destruction of surrounding tissues, electrolyte imbalance, toxic, allergic, carcinogenic and immune reactions, as well as thrombosis and thromboembolism, disturbances of cellular blood elements, denaturation of plasma proteins, destruction of blood enzymes [3]. Therefore, they are subject to sufficiently high requirements and, in order to improve their performance, the surface of the stents is covered with various types of coatings [1, 2, 4]. Tantalum is one of the most promising coatings for stainless steel stents due to the combination of the properties – mechanical strength, good biocompatibility, corrosion resistance and X-ray contrast.

The biological response of the body to the use of biomaterial is mainly determined by its chemical composition and structure. Today, methods for regulating biological properties are relevant to significantly change of the physicochemical, structural and functional properties of surface, modify surface relief, surface energy, hydrophilicity (hydrophobicity) in a wide range, to form various functional groups, to ensure protein immobilization and adsorption, adhesion cells.

The important characteristic of the material are the adhesion properties, since the risk of blood clots on the implantable material is quite high. Modern research methods, such as the AFM and the sessile drop method allows to obtain a series of data of the surface properties of materials. The AFM determine the local properties at the nanoscale level in the region of contact between the cantilever and the sample, and the sessile drop method analyze provide a number of data of the surface properties of material.

The aim of the research is to estimate the surface energy of nanostructured tantalum films and its compounds on glass and stainless steel substrates of type 316 LSS by the methods static force spectroscopy of AFM and the sessile drop.



2. Materials and methods

Samples of tantalum coatings on glass and steel were obtained at the Kharkov Institute of Physics and Technology of the National Academy of Sciences of Ukraine. The Ta, TaN, Ta₂O₅ and TaON coatings were deposited on glass and stainless steel substrates in a high-vacuum system with a turbopump at a base pressure of about 10⁻³ Pa using DC planar magnetron sputtering (Ta, Ta₂O₅) and. The tantalum target with a diameter of 170 mm was used. The maximum magnetron power was 6 kW. The samples were cleaned by standard methods in the ultrasonic bath before deposition process. Further cleaning and activation of the samples was carried out using an ion. The parameters of ion source were: ion acceleration voltage of 2.5 kV, and ion current source of 30 mA. For the reactive deposition, oxygen was delivered through the inductively coupled plasma source, q = 60 sccm, the magnetron voltage was Um = 700 V, and the magnetron current was about Im = 6 A. The sputtering process was conducted in modes far from the target passivation one for further oxide coatings deposition with highly stoichiometric composition. Also, such deposition conditions allowed us to avoid micro-arcs and micro-drops formation. The deposition process was carried out by a simultaneous bombardment of the growing film by argon ions using an ion source [4].

The investigation of the microstructure and determination of the values of adhesion force was performed by the AFM method on a Dimension FastScan device (Bruker, USA) in PeakForce QNM mode. Standard silicon cantilevers CSG10_SS (Micromasch, Estonia) with a radius of tip curvature of 1.1 nm and a console stiffness of 0.365 N/m and MPP-21100 (Bruker, USA) with a radius of tip curvature of 11.6 nm and a console stiffness of 2.63 N/m were used. The adhesion force was determined as the force value at the probe tip detachment from the sample surface. The surface free energy at the interface between the probe and the sample was calculated by the formula for elastic adhesive contacts of the parabolic profile probe [5]. The surface energy determined by the sessile drop method with using DSA 100 E device (KRUSS, Germany). Three test liquids were used - water, glycerol and diiodomethane. The surface free energy was calculated using the Owens, Wendt, Rabel and Kaelble (OWRK) method [6].

3. Results and discussion

The results of the surface free energy of nanostructured tantalum films presented in table 1.

Table 1. The values of the surface free energy of nanostructured films of tantalum compounds on glass and stainless steel 316 LSS substrates using AFM (γ_1) and the sessile drop methods (γ_2).

Sample	γ_1 , mJ/m ²	γ_2 , mJ/m ²	θ	γ^d , mJ/m ²	γ^p , mJ/m ²
316 LSS	0.18	34.5	75.0	31.7	2.8
Ta / (316 LSS)	0.11	29.1	84.8	26.4	2.7
Ta₂O₅ / (316 LSS)	0.04	31.1	89.0	30.0	1.1
TaN/ (316 LSS)	0.10	37.5	85.4	34.8	2.7
TaON/ (316 LSS)	0.21	37.8	94.1	37.7	0.1
Glass slide	2.56	77.3	5.0	7.8	69.5
Ta/ (glass)	0.08	36.3	84.7	36.0	0.3
Ta₂O₅ / (glass)	0.13	34.3	93.0	32.3	2.0
TaON / (glass)	0.19	36.3	34.9	36.0	0.3

AFM images of the tantalum films deposited on the glass slide and the initial substrates are shown in figure 1.

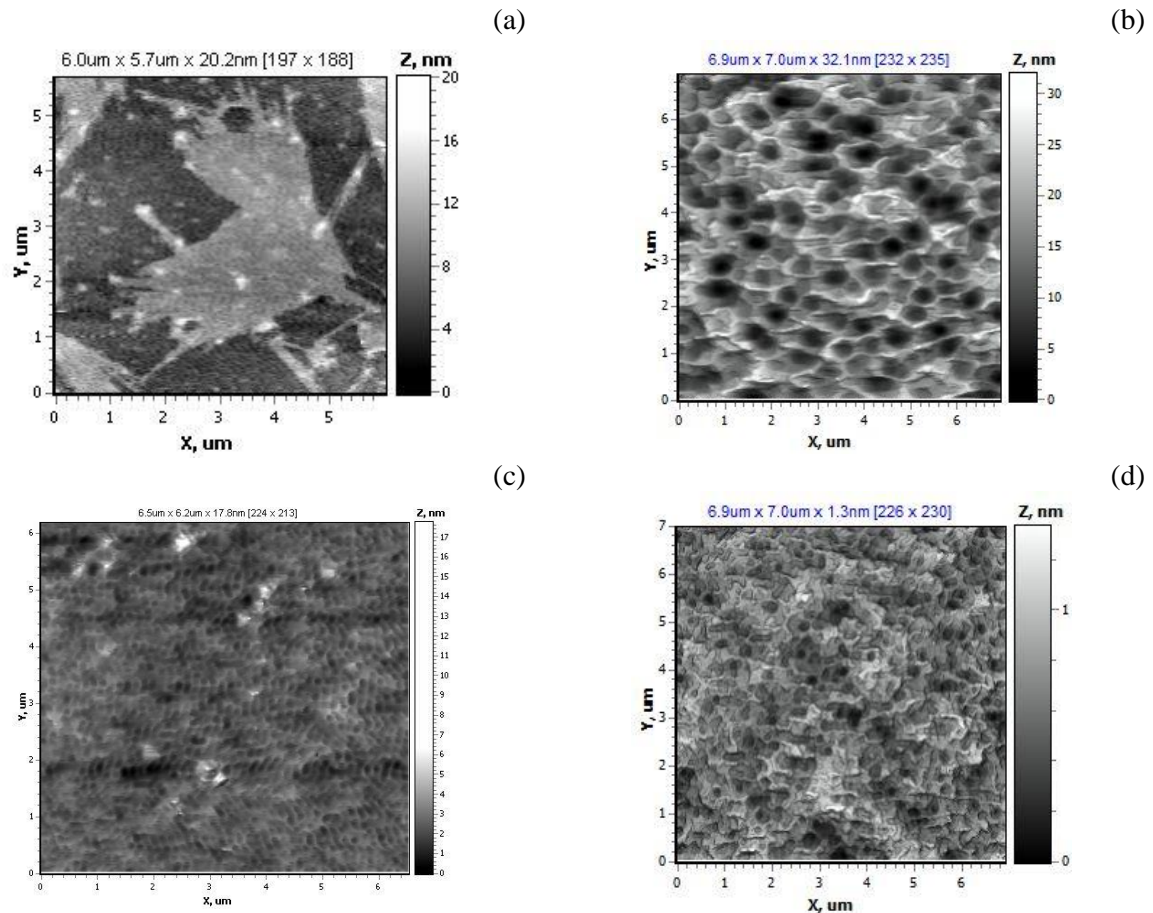


Figure 1. AFM images of the glass structure (a), Ta /glass (b), Ta₂O₅ /glass (c) and TaON /glass (d).

The tantalum compounds coating replicate the structure of stainless steel surface (figure 2) and reduce the cell diameter from 109 to 95 nm.

According to the data obtained by the sessile drop method, the deposition of tantalum and tantalum nitride coatings on the surface of stainless steel does not lead to changes in hydrophilic properties, while the AFM method has established a decrease of the surface free energy values to 0.11 mJ/m². This difference is due to the methodological features of the methods - the values of adhesion force, measured by the AFM method; estimate the local characteristics of the material, at the point. When measuring adhesion forces in the air by AFM, the tip and the sample interact through a thin film of water condensing from the air.

The condensing film can be localized inhomogeneously, to a greater extent, along the grain boundaries. In the case of determination the contact angle, we obtain the integral characteristic of the surface, averaging the properties of the material in the area of the investigated surface. The hydrophilic properties of the tantalum and tantalum nitride coatings are similar – the contact angle of the tantalum film is 84.8°, and the tantalum nitride is 85.4°. The hydrophobic properties of the surface of steel modified with tantalum oxide films are due to a decrease in the polar component of the surface energy, while the dispersion component remains unchanged. These changes were recorded by the AFM method – the surface energy values decreased 4.5 times.

4. Conclusion

It is established the increase of hydrophobic properties of stainless steel and glass surface after the deposition of nanostructured films of tantalum compounds, that further will allow to reduce platelet

aggregation on the implants surface and increase their service life and also to expand area of application.

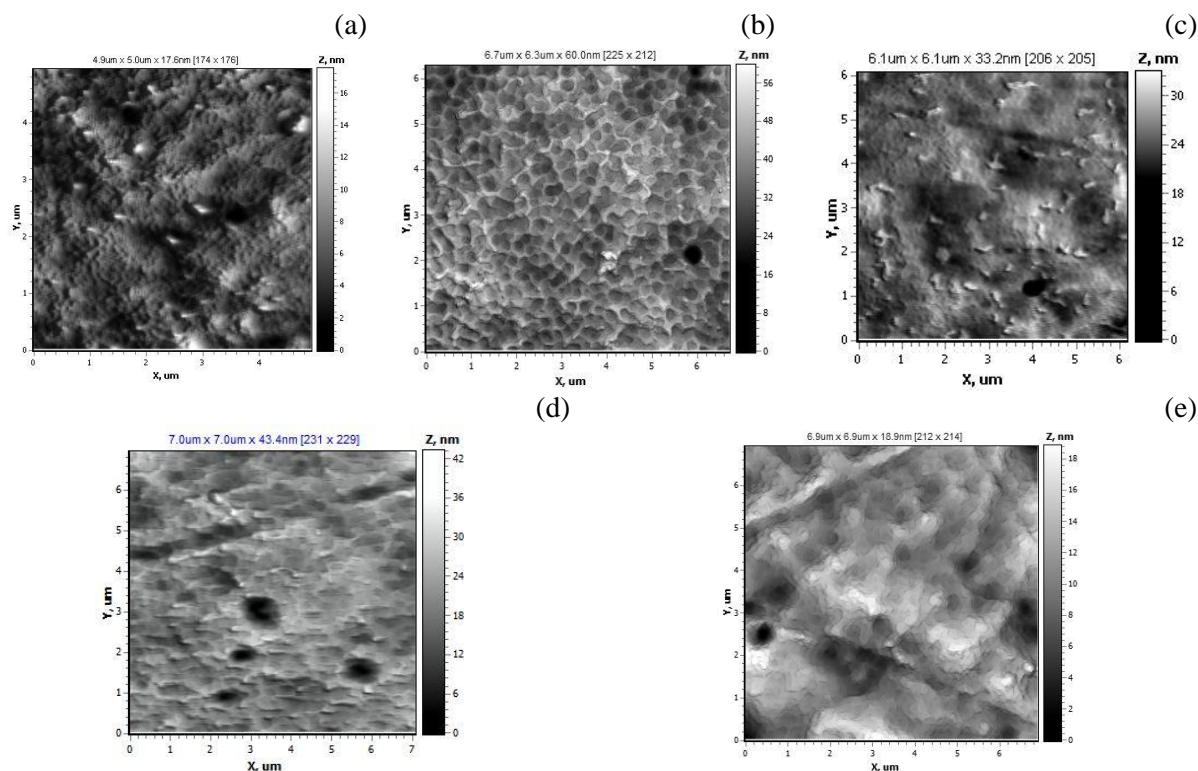


Figure 2. AFM-images of the structure of 316L SS (a), Ta/316L SS (b), Ta₂O₅/316LSS (c), TaN/316L SS (d) and TaON/316L SS (e).

Acknowledgments

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