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THE IMPACT OF PLASMA-ELECTROLYTIC OXIDATION OF TITANIUM

Normurodov Aziz Normurodovich*Samarkand State Medical University, Uzbekistan***ABOUT ARTICLE****Key words:** low density, low modulus of elasticity, good formability, hardness similar to tooth enamel.**Received:** 14.04.2024**Accepted:** 19.04.2024**Published:** 24.04.2024**Abstract:** Titanium is one of the most promising structural materials used in the manufacture of implants in orthopedics and dentistry. This material is characterized by low density, low modulus of elasticity, good formability, hardness similar to tooth enamel, biocompatibility with living tissues, and corrosion resistance in biological media.**INTRODUCTION**

Titanium is one of the most promising structural materials used in the manufacture of implants in orthopedics and dentistry. This material is characterized by low density, low modulus of elasticity, good formability, hardness similar to tooth enamel, biocompatibility with living tissues, and corrosion resistance in biological media. The corrosion resistance of titanium in various environments is determined by the formation of a stable protective adhesive oxide film. Due to its high affinity for oxygen, the natural oxide film on the titanium surface forms almost instantaneously in any environment containing traces of surface oxide film integrity and disintegrates under mechanical action, resulting in severe corrosion of the unprotected titanium substrate, leading to premature implant failure and Leading to. One method of increasing the electrochemical stability of metals and their alloys is plasma electrolytic oxidation (PEO). It is based on anodic or alternating current polarization of the treated material with high voltage, creating plasma micro-discharges on the electrode surface. As a result of the localized high-energy exposure, a layer containing both metal oxides and electrolyte elements is formed on the product surface. The corrosion resistance of such layers depends on their chemical composition, structure, and thickness, and may differ significantly from the properties of conventional anodic oxide coatings. As a rule, the phase composition of PEO coatings is

represented by crystalline modifications of titanium dioxide (IV) such as anatase, rutile, and brookite. Rutile is the most stable and biologically active titanium dioxide; the presence of various calcium phosphate compounds in the structure of PEO coatings has a positive influence on the osseointegration process of titanium implants in vivo due to their similar chemical composition with natural bone tissue. The properties of the PEO coating can be modulated by changing the parameters of the oxidation process (electrolyte composition, frequency and current intensity, electrolysis time, etc.) and also as a result of subsequent treatment of the formed membrane (pore filling with bioactive and/or bioinert components, firing, etc.). This will allow to obtain composite materials based on titanium and its alloys with improved protective properties and biocompatibility, which may have practical applications in implantation surgery. The purpose of this study is to investigate the influence of the PEO process on the surface structure and corrosion protection properties of a PEO coating of VT1-0 titanium in a model solution simulating oral fluid.

METHODS

Plasma electrolytic oxidation was performed on VT1-0 titanium specimens 2x2 cm in size and 2 mm thick. The PEO process was carried out in an electrolyte containing g/dm³ NaH₂PO₄ - 12; Ca(OH)₂-10; (NH₂)₂CO (urea) - 12; Na₂SiO₃-8 at a pulse frequency of 1 Hz, pulse duration of 2, in electrolytic mode. The electrolysis time was 300 sec. The electrolysis time was 300 s, and the anode current density was 30 A/dm². Stainless steel X18N9T was used as the cathode. The composition and morphology of the titanium surface after PEO were examined using a scanning electron microscope JSM 5610 LV with EDX JED 2201 JEOL elemental analysis system. The phase composition of the coatings was examined using a Discover D8 diffractometer (Bruker). Table 1 shows the composition of model solutions simulating oral fluid. HC brand reagents were used for preparation and pH correction was performed with 1 M HCl. Electrochemical studies of the corrosion of VT1-0 titanium and the protective properties of PEO coatings in a model solution simulating oral fluid were performed on an Autolab PGSTAT potentiostat/galvanostat equipped with the impedance spectroscopy module FRA 32N in a three-electrode cell with side electrodes 302N. The following data were used for the study. The geometric area of the working electrode for electrochemical measurements was 1 cm². A saturated silver chloride electrode (Metrohm Autolab) served as reference electrode and a platinum grid as counter electrode. Impedance spectra were measured at steady-state potential values in the frequency range 10⁵-10⁻² Hz. The time to establish a steady-state potential was 30 minutes. Linear voltammetry was recorded over a potential range of -200 mV to +900 mV relative to the steady-state potential. The potential sweep rate

was 1 mV/s. Spectra were analyzed, equivalent circuits were selected, and the parameters of their elements were calculated using the ZView program.

RESULTS AND DISCUSSION

Analysis of SEM images of VT1-0 titanium surfaces before and after PEO treatment indicated that electrochemical treatment results in the formation of highly porous structural coatings; pore distribution histogram data for the PEO coating indicated that pore sizes were primarily in the 1.0 to 2.5 micron range. Furthermore, as the current density of PEO increases, the ion current velocity increases and the porosity of the coating decreases due to the sealing effect of the micro-sized pores. Therefore, an anodic current density of 30 A/dm² was used in FEO to ensure the formation of a homogeneous coating with isotropically uniformly distributed pores. It should be noted that during the electrochemical process, the electrolyte is not allowed to overheat. To determine the thickness of the formed film and to examine its structure, SEM images of a transverse slide of titanium VT1-0 were obtained after the application of PEO. The results showed that the thickness of the PEO layer was approximately 10 microns. In addition, a characteristic relief groove structure was observed on the titanium substrate after PEO application. Analysis of EDX maps of the chemical elemental distribution on the surface of the obtained coatings indicated that Ca and P were present on the titanium surface after PEO and were uniformly distributed over the thickness of the coatings along with Ti, and their content did not exceed 15 wt.%. The molar ratio of Ca/P is on average 2:3. Thus, phosphorus and calcium are likely to have entered the structure of the oxide layer in the form of hydrated calcium phosphate and dihydrogen phosphate. To investigate the phase composition of the titanium VT1-0 surface after PEO, X-ray diffraction was used. According to the diffraction diagrams obtained, the phase composition of the titanium surface after PEO is represented by anatase and rutile. The phase transition from anatase to rutile is known to start at temperatures above 600°C [14]; during the FEO process, the temperature in the discharge channel can reach over 3000°C, so the formation of the titanium (IV) oxide mixture is due to thermal effects during oxide film growth. It should be noted that rutile and anatase (101) crystals are biocompatible and have lattice constants similar to those of hydroxyapatite (0001) crystals. As a result, epitaxial growth of apatite crystals is possible on the surface of the formed porous film. Therefore, the coating formed on the surface of titanium VT1-0 has sufficiently high biocompatibility. The absence of reflections on the diffraction lines, characteristic of calcium phosphate, may indicate its amorphous structure. As can be seen from the data presented, the corrosion potential of the initial sample of titanium VT1-0 is -0.32 V. The anodic branch of PC at potentials more negative than -0.15 V has a passive region with a Tafel gradient of 0.17 V in the E-*l*g*i* coordinate. The rate of electrochemical

processes in this region is determined by the density of passivation current (i_{pass}), which is about $3,10^{-6}$ - $5,10^{-6}$ A/cm². PEO on titanium VT1-0 (Figure 5, curve 2) shifted the corrosion potential of the sample to the positive region of 0.26 V and reduced i_{pass} by almost a factor of 10. In the PEO coating on the anode branch of the PC, a zone of passivation characterized by a Tafel gradient of 0.39 V and 0.10 V distinguishable and was observed at more positive potentials. The increase in anodic polarization leads to a progressive increase in current density, which indicates an intensification of the oxidation process. The current density during the corrosion of titanium VT1-0 in oral solution was 2.47×10^{-7} A/cm². PEO treatment significantly reduced the corrosion rate of titanium VT1-0 by a factor of 4.6, which corresponds to a 78.3% protection effect. The impedance spectra of the studied samples in model solutions simulating oral fluid according to the Nyquist diagram equation are shown in Fig. 6. The impedance hodograph of the initial VT1-0 titanium sample is characterized by the presence of only one capacitive semicircular time constant. This indicates that the corrosion process proceeds with limited charge transfer [16, 17]; the Nyquist diagram of the post-PEO sample shows two time constants, and the presence of the second time constant is associated with the presence of an external porous layer. Analysis of the obtained data shows that PEO contributes to the increase in resistance of the VT1-0 titanium surface, limiting the contact between the corrosion medium and the titanium substrate, leading to a decrease in the corrosion rate. At the same time, the inner layer of the formed coating is more stable than the outer layer. The value of the exponent n_2 indicates the diffusion limit of the corrosion process for PEO-coated samples. Conclusions 1. PEO promotes the formation of highly porous structural coatings with pore sizes of 1.0-2.5 microns on titanium VT1-0 surfaces. increasing the current density of PEO decreases the overall porosity of the coating, accompanied by an increase in ion current velocity and the sequestering effect of micro-sized pores. at a current density of 30 A/dm² density, the formation of homogeneous coatings up to 10 microns thick with isotropically evenly distributed pores was observed. Calcium and phosphorus are uniformly distributed across the thickness of the coating, with their content not exceeding 15 wt% and an average Ca/P molar ratio of 2:3. This suggests that phosphorus and calcium are more or less included in the structure of the oxide film in the form of x-ray amorphous calcium phosphate hydrate and dihydrogen phosphate hydrate³. Data from electrochemical studies in model solutions simulating oral fluid indicate that titanium VT1-0 PEO has a corrosion potential of the specimen of 0.26 V plus region, increasing the surface resistance of titanium VT1-0 and limiting the contact between the corrosion medium and the titanium substrate, thereby reducing the corrosion rate by a factor of 4.6.

CONCLUSION

In this case, the inner layer of the formed coating has a higher resistance than the outer layer. The corrosion process of the specimens is limited to the diffusion phase and the protective effect of the PEO coating is 78.3%⁴. PEO-titanium in electrolyte solutions containing calcium cations and phosphate anions helps to enhance surface corrosion resistance, biocompatibility, and osseointegration, and the resulting materials are promising for dental and orthopedic surgery as implants.

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