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THE EFFECT OF PLASMA-ELECTROLYTIC OXIDATION OF TITANIUM VT1-0 ON ORAL FLUID

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ABOUT ARTICLE

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Abstract: Titanium is one of the most promising structural materials used for the manufacture of implants in orthopedics and dentistry. This material is characterized by low density, low modulus of elasticity, good formability, hardness close to that of tooth enamel, biocompatibility with biological tissues and corrosion resistance in biological media.

INTRODUCTION

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 can almost instantly form in any environment containing traces of the integrity of the surface oxide film and collapse under mechanical action, as a result of which the unprotected titanium substrate undergoes intense corrosion, leading to premature destruction of the implant, which leads to the failure of the implant. One of the methods of increasing the electrochemical stability of metals and their alloys is plasma electrolytic oxidation (PEO), which is based on anodic or alternating current polarization of the processed material at high voltage, causing plasma micro-discharges on the electrode surface. As a result of local high-energy exposure, a layer containing both metal oxide and electrolyte elements is formed on the surface of the product. 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 positively affects the process of osseointegration of titanium implants in vivo due to the similarity of their chemical composition with natural bone tissue.The properties of PEO coatings can also be modulated by changing the parameters of the oxidation process (electrolyte composition, frequency and current strength, duration of electrolysis, etc.), and also as a result of subsequent processing of the formed membrane (filling of pores with bioactive and/or bioinert components, calcination, etc.). This makes it possible to obtain composite materials based on titanium and its alloys with improved protective properties and biocompatibility, which are expected to find practical application in implantation surgery. The purpose of this study is to study the effect of the PEO process on the surface structure and anticorrosive properties of PEO coatings of VT1-0 titanium in a model solution simulating oral fluid.

METHODS

Plasma-electrolytic oxidation was performed on VT1-0 titanium samples with a size of 2x2 cm and a thickness of 2 mm. Previously, the surface of the samples was degreased in 96% ethanol solution for 3 minutes and dried in air. The PEO process was carried out in an electrolyte containing g/dm3 NaH2 PO4- 12; Ca(OH)2-10; (NH2) 2 CO (urea) - 12; Na2 SiO3-8, at a pulse frequency of 1 Hz, pulse duration 2, Electrolysis was carried out in the mode. The electrolysis time was 300 s, 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 was studied using a scanning electron microscope JSM 5610 LV equipped with an EDX JED 2201 JEOL elemental analysis system. The phase composition of the coatings was studied using a Discover D8 diffractometer (Bruker). Table 1 shows the composition of model solutions simulating oral fluid. Reagents of the HC brand were used for preparation, pH correction was carried out with 1 M HCl. Electrochemical studies of corrosion of VT1-0 titanium and the protective properties of PEO coatings in a model solution simulating oral fluid were carried out on an Autolab PGSTAT potentiostat/galvanostat with an impedance spectroscopy module FRA 32N in a three-electrode cell equipped with side electrodes 302N. The geometric area of the working electrode for electrochemical measurements was 1 cm2. The saturated silver chloride electrode (Metrohm Autolab) served as a reference electrode, and the platinum grid served as a counter electrode. The impedance spectra were measured at a steady-state potential value in the frequency range 105-10-2 Hz. The time to establish a stationary potential was 30 minutes. Linear voltammetry was recorded in the potential range from -200 mV to +900 mV relative to the steady-state potential. The potential sweep rate was 1 mV/s. The

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 the VT1-0 titanium surface before and after PEO treatment showed that electrochemical treatment leads to the formation of a highly porous structural coating. Data from the pore distribution histogram for the PEO coating showed that the pore size was mainly in the range of 1.0-2.5 microns, which indicates that the pore size is mainly in the range of 1.0 to 2.5 microns. Moreover, at higher current densities in the PEO, the porosity of the coating decreases due to the higher ion current velocity and the sealing effect of micro-sized pores . Therefore, to ensure the formation of a homogeneous coating with isotropically evenly distributed pores, an anode current density of 30 A/dm2 was used in FEO. It should be noted that during the electrochemical process, overheating of the electrolyte is not allowed. To determine the thickness of the formed coating and study its structure, SEM images of the transverse sliding of titanium VT1-0 after applying PEO were obtained. The results showed that the thickness of the PEO layer is about 10 microns. The titanium substrate after PEO also has a characteristic structure of relief grooves. Analysis of EDX maps of the distribution of chemical elements on the surface of the resulting coating showed that Ca and P are present on the surface of titanium after PEO, distributed evenly over the thickness of the coating along with Ti, and their content does not exceed 15 wt.% The molar ratio of Ca/P is on average 2:3. Therefore, it is believed that phosphorus and calcium enter the structure of the oxide layer in the form of hydrated calcium hydrophosphate and dihydrophosphate To study the phase composition of the surface of titanium VT1-0 after PEO, X-ray diffraction methods were used. According to the diffraction diagrams obtained, the phase composition of the surface of titanium after PEO is represented by anatase and rutile. It is known that the phase transition from anatase to rutile begins at temperatures above 600 °C [14]. During the FEO process, the temperature in the discharge channel can reach more than 3000 °C, therefore, the formation of a mixture of titanium (IV) oxides is due to thermal effects during the growth of the oxide film. It should be noted that rutile and anatase (101) crystals are biocompatible and have similar lattice constants to hydroxyapatite (0001) crystals. As a result, epitaxial growth of apatite crystals is possible on the surface of the formed porous coating. Therefore, the resulting coating on the surface of titanium VT1-0 has a 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 presented data, the corrosion potential of the initial sample of titanium VT1-0 is -0.32 V. The anode branch of the PC at potentials more negative than -0.15 V has a passive region with a Taffel gradient in

the E-lgi coordinate of 0.17 V. The rate of electrochemical processes in this area is determined by the density of the passivating current (i pass), which is about 3,10-6 -5,10-6 A/cm2. PEO on titanium VT1-0 (Fig. 5, curve 2) shifted the corrosion potential of the sample to the plus region of 0.26 V, reducing the i pass by almost 10 times. In the PEO coating on the anode branch of the PC, a passivation zone can be distinguished, characterized by a Taffel gradient of 0.39 V and 0.10 V. observed at more positive potentials. An increase in anode polarization leads to a progressive increase in current density, which indicates an intensification of the oxidation process. The current density during corrosion of titanium VT1-0 in the oral fluid was 2.47× 10-7 A/cm2; PEO treatment significantly reduced the corrosion rate of titanium VT1-0 by 4.6 times, which corresponds to a protective effect of 78.3%. The impedance spectra according to the Nyquist linear diagram equation of the studied samples in a model solution simulating oral fluid are shown in Fig. 6. The impedance hodograph of the initial VT1-0 titanium sample is characterized by the presence of only one time constant in the form of a capacitive semicircle. This indicates that the corrosion process proceeds with limited charge transfer [16, 17]; two time constants are visible on the Nyquist diagram of the sample after PEO; the presence of the second time constant is associated with the presence of an external porous layer. The analysis of the obtained data shows that PEO contributes to an increase in the resistance of the VT1-0 titanium surface, limiting the contact between the corrosive medium and the titanium substrate, which leads to a decrease in the corrosion rate. At the same time, the inner layer of the formed coating is more stable than the outer one. The value of the exponent n2 indicates the diffusion limit of the corrosion process of the PEO-coated sample. Conclusions 1. PEO promotes the formation of highly porous structural coatings with pore sizes of 1.0-2.5 microns on the surface of titanium VT1-0. An increase in the current density of PEO leads to a decrease in the overall porosity of the coating, accompanied by an increase in the ion current velocity and the sequestering effect of micro-sized pores. At a current density of 30 A/dm2, the formation of homogeneous coatings up to 10 microns thick with isotropically evenly distributed pores was observed.2. The phase composition of the PEO coating is a mixture of anatase and rutile titanium (IV) oxides, which is due to the thermal effect on the surface during the growth of the oxide film. Calcium and phosphorus are evenly distributed over the thickness of the coating, their content does not exceed 15 wt. %, the average molar ratio of Ca/P is 2:3. This suggests that phosphorus and calcium are more or less included in the structure of the oxide layer in the form of X-ray amorphous calcium hydrophosphate and dihydrophosphate hydrates.3. Data from electrochemical studies in model solutions simulating oral fluid indicate that titanium VT1-0 PEO shifts the corrosion potential of the sample by 0.26 V to the positive region, increasing the surface resistance of titanium VT1-0, limiting the

contact of the corrosive medium with the titanium substrate and thereby reducing the corrosion rate by 4.6 times.

CONCLUSION

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

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