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Titanium Changes Generated at Different DC

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Abstract

Objective: To investigate the optimal DC voltage that titanium morphology was created with micro arc oxidation.

Methods: The titanium was cut into 10 mm, 10 mm, 1 mm and they were grind and polished respectively. DC voltage that treated titanium was used single variable control: 200V, 250V, 300V, 350V, 400V, 450V; treatment time: 5S; the treatment temperature was less than 40℃, Electric current and other conditions were same.

Results: The morphology on titanium surface was multipore structure, and the pore size was different after titanium treated with micro arc oxidation. The average pore size of MAO250V, MAO300V and MAO350V groups were bigger than 1 μm, MAO200V, MAO400V and MAO450V groups were less than 1 μm, the porosity was 17.4% at MAO200V group, 37% at MAO250V, MAO300V and MAO350V groups, 25.2%, 20.7% at MAO400V and MAO450V group; thickness was from 0.63 μm at MAO200V to 6.87 μm at MAO450V group. There was significant between the groups in the pore size and thickness.

Conclusion: Morphology could meet the needs of clinic at 250-350V DC voltage.

Keywords: Titanium • Micro arc oxidation • DC voltage • Morphology

Introduction

Titanium and titanium alloys may be considered viable substitutes in the fabrication of dental implants. The advantages given by these technologies that include the possibility to create porous complex structures to improve osseointegration and mechanical properties, to produce custom-made implants, to lower processing costs according to the data for the patient acquired via computed tomography and to reduce waste [1]. The development of titanium and titanium alloys to manufacture dental implants has emerged in recent years due to the increased failure of commercially pure titanium implants [2]. To overcome the material's inherent inertness, surface-coating modification is a crucial pathway to enhancing biocompatibility. Thus, before this study we must review existing information about the chemical, mechanical, electrochemical and biological properties of the main titanium and titanium alloys developed over the past few years to provide scientific evidence in favor of using titanium-based alloys as alternative to pure titanium [3]. Such evidence is given by the enhanced properties of titanium and titanium alloys, such as a low elastic modulus, high tensile strength, satisfactory biocompatibility, and good corrosion and wears resistances. In addition, titanium and titanium alloys can be modified at the chemical, structural and thermo mechanical levels, that allows the development of materials in accordance with the demands of several situations encountered in clinical practice.

Titanium and titanium alloys are widely used as dental implants because of their excellent physiochemical properties and biocompatibility [4]. The clinical long-term success of dental implants was related to their early osseointegration, thus morphology surface of implant plays an important role in the progression. Effects of titanium surface treatment determine the optimum surface on the behavior of osteoblast-like cells to promote early osseointegration. An oxide ceramic coating can be formed on the surface of titanium alloy by micro-arc oxidation. In this paper, a general overview of the surface treatment of micro-arc oxidation on the surface of titanium alloy is presented. Micro Arc Oxidation (MAO) was a surface treatment method that could produce a well-characterized,

biocompatible Titanium Dioxide (TiO_2) morphology, including calcium, phosphorus, and some well-distributed micropore [5].

Materials and Methods

Content of titanium should be no less than 98%, which contains a small amount of impurities such as oxygen, nitrogen, hydrogen, carbon, silicon and iron. Pure titanium in China was classified into several grades, such as TA1, TA2, TA3 and TA4 according to the

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content of impurity elements. The matrix material of medical pure titanium was TA2 in this experiment. The chemical composition was provided by Hebei Xingtai Hengzhong metal material Co., Ltd. The line cutting samples would be processed into 10 mm, 10 mm, 1 mm. In this study, according to previous experiments and domestic and foreign literature, titanium was treated with micro arc oxidation as single variable control method: 200V, 250V, 300V, 350V, 400V, 450V; treatment time: 5S; the treatment temperature was less than 40℃, the electrolyte parameter was calcium acetate (0.075 mol/L), sodium dihydrogen phosphate (0.03 mol/L) and Ethylene diamine tetra acetic acid EDTA-2Na (10 g/L).

Micro arc oxidation process

Surface treatment of titanium: The medical pure titanium has been cut into 10 mm, 10 mm, 1 mm. The titanium surface was ground with 600 grit, 800 grit, 1000 grit and 1200 grit SiC papers, and then ultrasonically cleaned with acetone, absolute ethanol and distilled water for 15 min in series. Then cleaned with the acid solution (hydrofluoric acid: hydrogen nitrate: distilled water was 1:4:5). The titanium was treated with MAO in an electrolyte for 5 seconds, ultrasonically rinsed with distilled water for 15 min.

Micro arc oxidation treatment: Electrolyte contented 0.03 mol/L calcium acetate, 0.075 mol/L sodium dihydrogen phosphate, EDTA-2Na 10g/L. Electrolyte was mixed by electromagnetic centrifugal. Then it was poured into the electrolytic tank, and the temperature of the electrolyte was ensured less than 40°C before the test. The pure titanium was the anode and the platinum was the cathode. The sample was soaked in the electrolytic, and the sample could not contact with tank. It was cleaned with ultrasonic wave and distilled water for 15 min after the test. Then it was dried, sealed, and stored.

Characterization and analysis of morphology

Analysis with field emission scanning electron microscopy: The field emission scanning electron microscope (ZEISS IGMA) and its Energy Spectrum System (EDS) were used to detect the surface morphology, the distribution characteristics and content of the elements of the morphology was to be analyzed.

Analysis on size of micropores: The morphology porosity was calculated with field emission scanning electron microscopy and Image-Pro Plus 6 software, Each picture was analyzed for three times.

Morphology thickness test: The thickness of the morphology was measured with DT-156 thickness gauge, the accuracy was 0.1 μm, and the measurement range was 0~1250 μm.

Results

The experimental data were expressed as mean ± Standard Deviation (SD). Statistical analysis was performed with SPSS 13.0 software (SPSS Inc., Chicago, USA). Paired T test was used to assess the effects of the different voltage treatments. $P<0.05$ was considered statistically significant. The micropore size, average size and porosity at different voltage are seen in Table 1 and effect of micro arc oxidation voltage on the thickness of morphology is seen in Table 2.

Table 1. Micropore size, average size and porosity at different voltage.

Group	Pore size (μm)	Average (μm)	Porosity (%)
MAO200V	<1	0.38	17.40%
MAO250V	0.5~1.5	1.03	37.90%
MAO300V	0.7~1.8	1.11	37.80%
MAO350V	0.8~2	1.34	37.60%
MAO400V	<0.5; 1.5~2.5	0.81	25.20%
MAO450V	<0.5 2~3	0.66	20.70%

Table 2. Effect of micro arc oxidation voltage on the thickness of morphology.

Group	Thickness (μm)	P
MAO200V	0.63 ± 0.2406	-
MAO250V	1.23 ± 0.3335	<0.05
MAO300V	2.32 ± 0.6179	<0.01
MAO350V	3.00 ± 0.6815	<0.01
MAO400V	5.05 ± 0.5968	<0.01
MAO450V	6.87 ± 0.5272	<0.01

Discussion

The Titanium Dioxide (TiO_2) layer provided by the Micro Arc Oxidation (MAO) has been shown to improve cellular response in vitro and promote new bone formation around the implant in vivo. Although several in vitro studies have established the superiority of titanium alloys over pure titanium, mainly in terms of their mechanical properties, there is no scientific evidence that supports the total replacement of this material in vivo. However, it is evident that in vivo studies are encouraged to test new alloys to consolidate their use as substitutes for pure titanium. These improvements were attributed to Ca and P micropores morphology, which could increase mechanical interlocking between bone tissue and implant. This interaction was termed “osteoconduction”, which was defined as appositional bone growth and permitting bone formation on a surface or into micropore. According to Tables 1 and 2, it was known that titanium dioxide layer was very thin, and a large number of micropores were formed, the thickness of titanium dioxide layer in this experiment increased gradually from 0.63, 1.23, 2.32, 3.00, 5.05, 6.87 μm. The surface had formed a large number of micropores morphology. The size also increased gradually, from 0.5 ~ 1.5 μm, 0.7 ~ 1.8 μm to 0.8 ~ 2 μm, the porosity was 17.4%, 37.9%, 37.8%, 37.6%, 25.2% and 20.7% at 250V, 300V, 350V, 400V and 450V respectively.

Conclusion

The micropores size was less than 1μm and the porosity was 17.4% at 200V. The size was 2 ~ 3 μm, it was not only showed obvious cracks, but also a large number of circular deposits at 450V. The number of micropores began to increase and show two poles distribution and visible cracks at 400V. It could be seen that increasing of the DC voltage, a large number of crater shapes were formed, the size of micropores and thickness of membrane became larger gradually. The higher voltage, the thicker thickness was. This was initial formation of titanium dioxide when the treatment voltage reached the critical breakdown point, titanium surface was broken down, and the instantaneous higher temperature caused the titanium surface to melt and extrude [6,7]. The discharge channel also formed at the same time. A pores structure was left due to the cool electrolyte. The surface, when the size of the micropore is 1 ~ 4 μm, is beneficial to the adhesion and growth of cells. The porosity of membrane also affected the expression of osteoblast. It should be higher than 20% ~ 25%, some thought 30% ~ 40%. Normally thickness of membrane less than 10 μm is beneficial morphology; otherwise the quality is not good. Above of all, it showed that morphology treated at 250-350V DC voltage could meet the needs of clinic.

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