

## The Characteristic of Morphology Created at Different DC Voltage

### Review Article

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### Abstract

**Objective:** To investigate the physicochemical properties that titanium morphology was created with micro arc oxidation at different DC voltage.

**Methods:** The titanium was cut into  $10 \times 10 \times 1 \text{ mm}^3$  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  $40^\circ\text{C}$ , Electric current and other conditions were same.

**Results:** 1. The contact angle of the Ti group is the largest, which is  $(80.80 \pm 0.57)$ , and the MAO<sub>350V</sub> group is the smallest which is  $(35.00 \pm 0.79)$  degrees. 2. The critical load of morphology varied from 16N to 35N. When the voltage was 350V, the bonding strength of morphology is relatively good, and the critical load is  $(32.38 \pm 1.941)$  N. There is statistical significance among all groups. 3. The wear resistance of the ceramic film of the group MAO<sub>350V</sub> manifested the smallest friction coefficient, the value was approximately 0.4. 4. Its Self-corrosion voltage  $(-0.309\text{V})$  in MAO<sub>350V</sub> was way higher than the group Ti  $(-0.378\text{V})$ , and self-corrosion current  $[2.5 \times 10^{-5} (\text{A} \cdot \text{cm}^{-2})]$  in the MAO<sub>350V</sub> was much lower than the group Ti  $[5.2 \times 10^{-5} (\text{A} \cdot \text{cm}^{-2})]$ .

**Conclusion:** There is high quality of morphology created at 250-350V DC voltage.

### Keywords

Titanium; Micro-arc oxidation; Surface modification; DC voltage

### Abbreviations

MAO: Micro Arc Oxidation;  $\text{TiO}_2$ : Titanium Dioxide; SD: Standard Deviation; DC: Direct Current; EDTA: Ethylene Diamine Tetra Acetic acid

### Introduction

Micro arc oxidation (MAO) was a surface treatment method that could produce a well-characterized, biocompatible titanium dioxide ( $\text{TiO}_2$ ) morphology. Titanium and titanium alloys are widely used as dental implants because of their excellent physicochemical properties and biocompatibility. The clinical long-term

success of dental implants was related to their early Osseo integration, 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 Osseo integration.

**Materials**

**Materials and reagents**

**Pure titanium matrix material:** 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 content of impurity elements. The matrix material of medical pure titanium was TA2 in this experiment. The chemical composition was shown in Table (1) (GB/T13810-2007), which was provided by Hebei Xingtai Hengzhong metal material Co., Ltd. The line cutting samples would be processed into 10×10× 1mm<sup>3</sup>.

**Table 1:** The chemical compositions of TA2

Ti	Fe	C	N	H	O
Residual	0.1300	0.040	0.0300	0.0010	0.2000

**Table 2:** Contact angle of micro arc oxide film under different voltage(°,  $\bar{x} \pm s$ , n=5)

Group	contact angle	p
Ti	80.80±0.57	-
MAO <sub>200V</sub>	70.90±0.65	<0.01
MAO <sub>250V</sub>	61.70±0.67	<0.01
MAO <sub>300V</sub>	47.70±0.84	<0.01
MAO <sub>350V</sub>	35.00±0.79	<0.01
MAO <sub>400V</sub>	44.20±0.57	<0.01
MAO <sub>450V</sub>	47.00±0.79	<0.01

**Note:** the P value was the result that compared with the previous experimental group.

**Table 3:** Bonding strength of micro arc oxide film under different voltage (N, n=3,  $\bar{x} \pm s$ )

Group	Bonding strength	p
MAO <sub>200V</sub>	17.00±0.81	-
MAO <sub>250V</sub>	20.77±0.76	<0.05
MAO <sub>300V</sub>	25.38±2.62	<0.05
MAO <sub>350V</sub>	32.38±1.94	<0.01
MAO <sub>400V</sub>	28.36±0.92	<0.05
MAO <sub>450V</sub>	22.16±0.99	< 0.01

**Note:** the P value was the result that compared with the previous experimental group.

**Table 4:** Corrosion parameters on the surface of each sample material

Group	E <sub>corr</sub> /V	i <sub>corr</sub> /(A·cm <sup>-2</sup> )
Ti	-0.378	5.2×10 <sup>-5</sup>
MAO <sub>200V</sub>	-0.358	5.0×10 <sup>-5</sup>
MAO <sub>250V</sub>	-0.348	4.6×10 <sup>-5</sup>
MAO <sub>300V</sub>	-0.339	3.9×10 <sup>-5</sup>
MAO <sub>350V</sub>	-0.309	2.5×10 <sup>-5</sup>
MAO <sub>400V</sub>	-0.320	2.9×10 <sup>-5</sup>
MAO <sub>450V</sub>	-0.330	3.0×10 <sup>-5</sup>

**Process parameters**

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°C, the electrolyte parameter was calcium acetate (0.075mol/L), sodium dihydrogen phosphate (0.03mol/L) and Ethylene diamine tetra acetic acid EDTA-2Na (10g/L).

**Groups**

Group Ti; Group MAO<sub>200V</sub>; Group MAO<sub>250V</sub>; Group MAO<sub>300V</sub>; Group MAO<sub>350V</sub>; Group MAO<sub>400V</sub>; Group MAO<sub>450V</sub> 5Pices respectively

**Micro arc oxidation process**

**Surface treatment of titanium:** The medical pure titanium has been cut into 10×10×1mm<sup>3</sup>. 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). In accordance with previous work [1], 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 0.03mol/L calcium acetate, 0.075mol/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 15min after the test. Then it was dried, sealed, and stored.

### Hydrophilic test

A static contact angle measuring instrument (German Kruss Company) was used to test the static contact angle of the pure titanium. 5 specimens were tested in each group.

### Bonding strength test

The interface bonding strength between the ceramic membrane and the matrix material was measured by the WS-2005 coating adhesion automatic scratch test instrument (Lanzhou ZhongKeKaiHua Science and Technology Development Co., Ltd.). The loading of this test was 30N/min. The same sample was measured three times in the adjacent place of the same area. The two stable data at last were adopted.

### Test of wear resistance

The friction and wear test was carried out by HSR-2M high speed reciprocating friction and wear test machine (Lanzhou ZhongKeKaiHua Science and Technology Development Co., Ltd.). The computer records data every 3 seconds. The obtained data were processed by Origin 8 software, and the friction coefficient and friction time curve was obtained.

### Corrosion resistance test

The experimental instrument was the electrochemical workstation (Princeton PARSTAT 3000A, USA). Polarization curve method was used to evaluate the corrosion resistance of titanium ceramic membrane. The self corrosion voltage and the magnitude of self corrosion current of each sample were calculated by polarization diagram.

### Statistical analysis

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.

### Results

5 Detection results of static water contact angles of micro arc oxidation ceramic coatings with different voltages

### Discussions

Nowadays, patients' desire to have beautiful teeth and using tooth-colored restorations has increased; Implant is one of the ideal repair methods. However, the surface of implants should have good friction resistance, corrosion resistance, good bonding strength and good cell adsorption in order to keep implant to use for long time.

Generally, the surface of materials has good hydrophilicity, which is conducive to cell and protein adhesion. The better hydrophilicity is, the easier it is to promote bone formation. The results in this experiment show when the voltage was 350V, 300V, 250V, 200V, the contact angle was  $(35.00 \pm 0.79)$ ,  $(47.70 \pm 0.84)$ ,  $(61.70 \pm 0.67)$  and  $(70.90 \pm 0.65)^\circ$  respectively, so it can quickly promote the penetration of fluid between implants, stabilize blood clots, and it is benefit for cell adsorption on surface of titanium, then form bone (Figure 1 and Table 2).

Long-term high bond strength is of great importance for effective restoration. The more bonding strength of

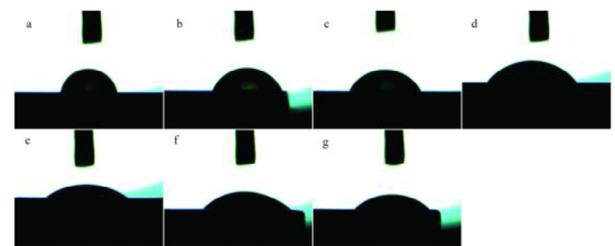


Figure 1: Contact angle of micro arc oxide film under different voltage (a)Ti ,(b)MAO<sub>200V</sub> ,(c)MAO<sub>250V</sub> ,(d)MAO<sub>300V</sub> ,(e)MAO<sub>350V</sub> ,(f)MAO<sub>400V</sub> ,(g)MAO<sub>450V</sub>

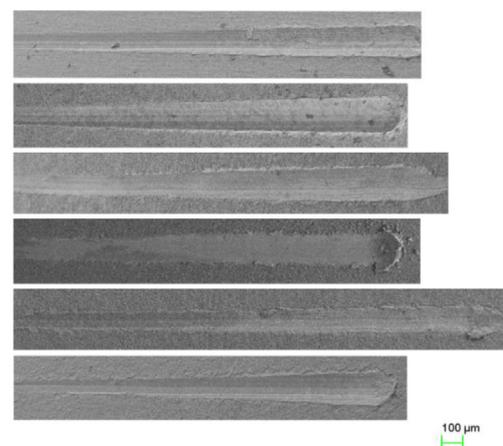
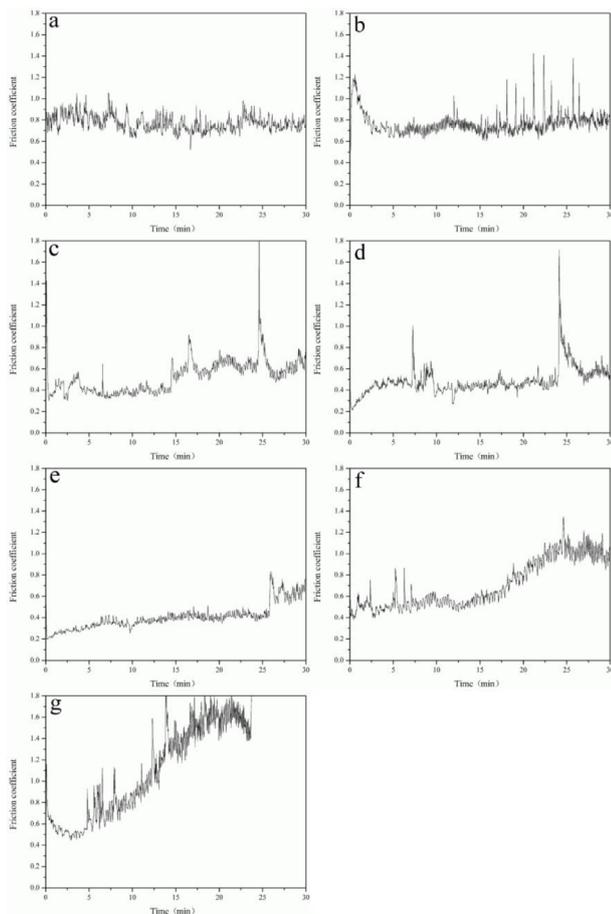
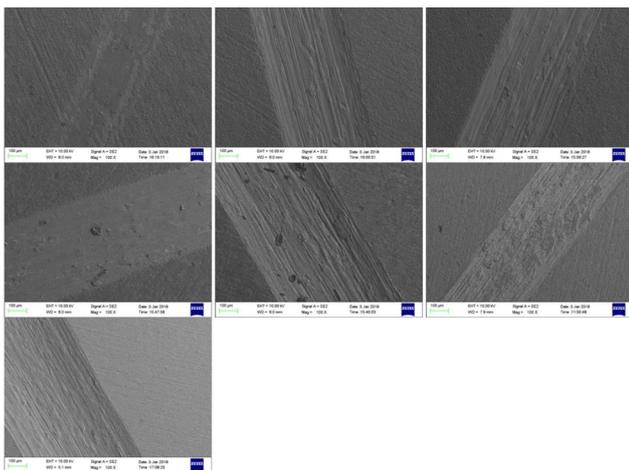


Figure 2: Scratch morphology of micro arc oxide film under different voltage



**Figure 3:** Friction coefficient of micro arc oxidation film with different voltage under SBF condition (a)Ti, (b)MAO<sub>200V</sub>, (c) MAO<sub>250V</sub>, (d)MAO<sub>300V</sub>, (e)MAO<sub>350V</sub>, (f)MAO<sub>400V</sub>, (g)MAO<sub>450V</sub>

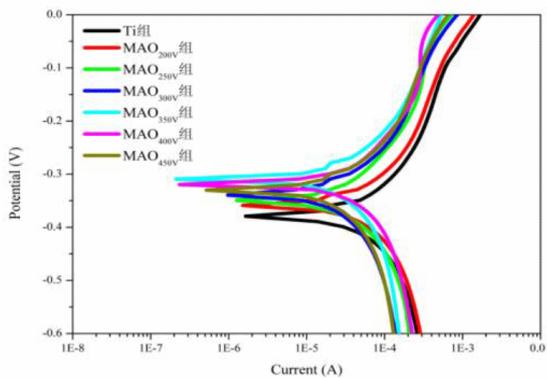


**Figure 4:** Friction and wear SEM morphology of micro arc oxidation film under different voltage conditions under SBF condition (a)Ti, (b)MAO<sub>200V</sub>, (c)MAO<sub>250V</sub>, (d)MAO<sub>300V</sub>, (e)MAO<sub>350V</sub>, (f) MAO<sub>400V</sub>, (g)MAO<sub>450V</sub>

titanium morphology, the longer live the implant. The bonding strength between the morphology and the titanium material at different voltage has been obviously changed (Figure 2, Table 3). The critical load of morphology varies from 16N to 35N. When the voltage was 350V, 300V, 250V, 200V, the bonding strength of the material was  $(25.38 \pm 2.62)$  N,  $(20.77 \pm 0.76)$  N  $(32.38 \pm 1.941)$  N and  $(17 \pm 0.81)$  N respectively. This long-term high bond strength is attributed to the combined effect of physical and chemical bonding. Bony penetration results in a significant increase in strength, judged to be sufficient for support of dentures.

The friction coefficient has a certain influence on the stress of the interface between bone and tissue. If the lower friction coefficient, the large interface stress, the poorer wear resistance of the implant material. The fine frictional fibers can be used to form very small knots for microsurgical vessel and organ ligature in medicine or embryology. The results show that the friction coefficient of group Ti was basically stable in the whole course of test, about 0.8, but the friction coefficient of group MAO<sub>250V</sub>, MAO<sub>300V</sub> and MAO<sub>350V</sub> was low in the initial period of time, and has been fluctuating around 0.4, while the initial friction coefficient of MAO<sub>350V</sub> group was relatively stable, and the fluctuation was relatively small, the MAO<sub>400V</sub> group and the MAO<sub>450V</sub> group, the friction coefficient was unstable, the friction coefficient was obviously rising, and > 0.8. Figure 3,4. Because the morphology of all the specimens is thin, the friction coefficient is less than 0.8, when the voltage is 350 V, its bio-tribological performance as an implant is relatively optimal.

The corrosion resistance of the morphology of each group is determined by the thickness, density, roughness and phase structure. Because the corrosion resistance of the superficial layer of the morphology directly determines the safety of the titanium implants and the length of the service life. The results show that the MAO<sub>350V</sub> group had the lowest self-corrosion current density,  $2.5 \times 10^{-5}$  (A·cm<sup>-2</sup>) and the highest self-corrosion voltage - 0.309V. The self-corrosion voltage of Ti group was - 0.378V, and the self-corrosion current was  $5.2 \times 10^{-5}$  (A·cm<sup>-2</sup>). Morphology with cracks is not conducive to the corrosion resistance of the material and that with complete structure and thickness can effectively resist the corrosion of medical pure titanium matrix by corrosive solution shown in (Figure 5 and Table 4). The greater the corrosion resistance, the less likely the implant will be damaged.



**Figure 5:** Tafel curves of each specimen material

The titanium morphology prepared by this method that used as dental implant material have better hydrophilic, bonding strength, wear resistance, corrosion resistance.

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### Availability of data and materials

Data sharing is applicable to this article, datasets were generated or analyzed during the current study.

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