

Research Article

Effect of Microarc Oxidation Treatment with Different Concentrations of MgSiF_6 on Titanium-Porcelain Bonding Strength

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This study aims to evaluate the effects of different electrolyte concentrations on titanium-porcelain bonding strength after microarc oxidation (MAO) treatment. Four MgSiF_6 electrolyte concentrations (10 g/L, 20 g/L, 30 g/L, and 40 g/L) were chosen for the MAO bath solutions. According to ISO 9693, the bonding strengths of titanium-porcelain restorations were detected by the three-point bending test. Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) were applied to evaluate the morphologies and elemental compositions of the MAO coating, titanium-porcelain fracture surfaces, titanium-porcelain interfaces, and oxygen diffusion. The bonding strength of the 20 g/L MgSiF_6 group was significantly higher than that of the control group. However, overly high MgSiF_6 concentrations had a negative influence on the bonding strength between titanium and porcelain. The results demonstrate that MAO treatment with only appropriate electrolyte concentration can improve the titanium-porcelain bonding strength.

1. Introduction

Porcelain-fused metal (PFM) prosthetic restoration is widely applied in clinic. The metal substrate used for PFM should have superior biocompatibility, excellent physicochemical properties, and relatively low cost. Titanium appears to be a perfect candidate because of its corrosion resistance, outstanding biocompatibility, and low cost [1]. However, when the sintering temperature exceeds 800°C during porcelain fusion, titanium strongly reacts with gaseous elements such as oxygen [2]. It has been repeatedly reported that titanium oxidation is one of the most important reasons for titanium-porcelain bonding failure [3].

In clinic, various treatments have been applied to improve the titanium-porcelain bonding strength, such as airborne-particle abrasion or acid etching. However, the effects of these

methods on the titanium-porcelain bonding strength are still controversial. For instance, airborne-particle abrasion could contaminate the titanium surface with alumina particles, which may weaken the interfacial bonding strength [4]. To improve the bonding strengths of titanium-porcelain restorations, various interlayers have been induced to prevent the formation of excessive titanium oxide layer, such as silica coating [5], nanotitanium coating [6], and gold coating [7]. For example, Guo found that nanotitanium coating formed by sol-gel process could prevent the oxidation of titanium substrate and subsequently improve bonding strength between titanium and porcelain [6]. It has been repeatedly proved that interlayer would be an effective strategy to improve bonding strength of titanium-porcelain restoration. Microarc oxidation (MAO) is an electrochemical technology that involves an arc discharge reaction that forms a ceramic coating on the titanium substrate

in seconds. A MAO coating includes a compact inner coating to serve as an oxygen-diffusion barrier and a porous outer coating to increase the mechanical interlocking of the porcelain and titanium. In our previous study, we found that MAO with appropriate electrolytes, such as MgSiF_6 , can significantly improve the titanium-porcelain bonding strength [8]. However, the effect of MgSiF_6 concentration on the titanium-porcelain bonding strength needs further investigation.

The physicochemical properties of MAO coatings are affected by the concentration of the MAO electrolyte. In MAO, electrolyte anions are driven by the high voltage, bombarding the titanium surface and fusing into the ceramic coating. On the one hand, the quantity of anions fused into the coating depends on the concentration of the MAO electrolyte and then determines the chemical proportion of the MAO coating [9]. In our previous study, the elemental proportion of silicon and fluorine in bonding porcelain were 21.2% and 2.6%, respectively [10]. Thus, altering the chemical proportion of the MAO coating would alter the chemical bonding which is a major bonding force in titanium-porcelain restorations [11]. On the other hand, the concentration of the electrolyte affects the conductivity of the MAO bath solution and the bombarding energy of the electrolyte anions. As a result, the alterations of electrolyte concentration impact the morphology and structure of the MAO coating on titanium [12]. Therefore, the concentration of the electrolyte influences the physicochemical properties of the MAO coating and changes the bonding strength between titanium and porcelain, while the appropriate electrolyte concentration for titanium-porcelain restorations needs further study.

This study aims to determine the effects of MgSiF_6 concentration on titanium-porcelain bonding strength and the appropriate MAO electrolyte concentration. In this study, four different concentrations of MgSiF_6 were selected for the MAO electrolyte, and the physicochemical properties of the MAO coatings were investigated. The failure mode at the interface, the effect of the MAO coating on reducing titanium oxidation, and the influence of intermediate coatings on the bond strength improvement were evaluated.

2. Materials and Methods

2.1. Surface Treatment. A total of 50 samples ($25\text{ mm} \times 3\text{ mm} \times 0.5\text{ mm}$) of milling commercial pure titanium (CP-Ti) of ASTM Grade II (Bego, Bremen, Germany) were randomly divided into five groups. Four groups were treated with MAO (MAO-100, College of Materials Science and Engineering, Qingdao University, China). Four different concentrations of MgSiF_6 (Shanghai Chemical Reagent, Shanghai, China) were selected, including 10 g/L, 20 g/L, 30 g/L, and 40 g/L. The applied voltage, pulse frequency, and duty cycle were set to 300 V, 500 Hz, and 0.04, respectively. A stainless steel disc was used as the MAO cathode, and titanium specimens were used as the anode. The MAO treatment was performed under water-cooled conditions, keeping the temperature of the entire process below 30°C. The arc discharges occurred on the titanium surface at 280°C. Then, arc discharges spread all over the titanium

substrate when the MAO applied voltage arrived at 300°C. The duration of the MAO treatment at 300°C was 3 min. After MAO treatment, all the samples were cleaned continuously for 10 min with acetone, ethanol, and distilled water in an ultrasonic cleaner. Then, they were dried at room temperature. The control group was airborne-particle abraded with 150 μm aluminum oxide powder at a pressure of 0.2 MPa from a distance of 5 mm and at a 45° angle for 5 s.

2.2. Coating Morphology and Component Analysis. Five specimens were randomly selected from each group to evaluate the titanium surface morphology by scanning electron microscopy (SEM, JMS-6460, JEOL, Tokyo, Japan), and the elemental composition of the MAO coating was analyzed using energy-dispersive spectroscopy (EDS, INCA-sight, Oxford Instruments, London, England).

2.3. Porcelain Application. Porcelain was fused to dimensions of $8\text{ mm} \times 3\text{ mm} \times 1\text{ mm}$ according to ISO 9693. All the specimens were ultrasonically cleaned in ethanol, acetone, and deionized water for 15 min and then dried at room temperature. Low-fusing porcelain for titanium (Super porcelain Ti-22, Noritake, Tokyo, Japan) was used in this study. The bonding porcelain, opaque porcelain, and body porcelain were coated in the selected area with a short-bristled brush individually; the thickness of the porcelain was controlled by a self-made mold. The porcelain-firing parameters were controlled automatically in a dental porcelain furnace (Vita Vacumat 6000 M, Säckingen, Germany) according to the manufacturer's instructions. The thicknesses of the porcelain coatings and the manufacturer's instructions are listed in Table 1.

2.4. Bond Strength Testing Procedure. In accordance with ISO 9693, the titanium-porcelain bonding strengths were evaluated by three-point bending tests with a universal testing machine (Shimadzu AGS-J, Tokyo, Japan). Each specimen was placed on the supports (20 mm span distance). A round loading piston with a radius of 1 mm was pressed to the center of the titanium specimen, and the loading rate was set to 0.5 mm/min. The load was applied continuously until the load-deflection curve of the porcelain-titanium restoration was obtained. Bond failure was recorded digitally using the software provided by the manufacturer of the testing machine. The bending strength was calculated according to the following formula:

$$\Sigma = k \cdot F \left[\frac{N}{\text{mm}^2} \right], \quad (1)$$

where F is the maximum force, k is the constant calculated according to ISO 9693, and Σ is the bond strength in N/mm^2 . After the three-point bending test, the titanium fracture surfaces of each group were observed by SEM to evaluate the failure mode.

TABLE 1: Sintering condition of the porcelain.

| Porcelain | Starting temperature (°C) | Sintering temperature (°C) | Vacuum (MPa) | Sintering time (min) | Porcelain coating thickness (mm) |
|---------------|---------------------------|----------------------------|--------------|----------------------|----------------------------------|
| Bonding agent | 500 | 800 | 0.099 | 3 | 0.2 |
| Opaque | 500 | 780 | 0.099 | 3 | 0.2 |
| Dentin | 500 | 760 | 0.096 | 5 | 0.6 |

2.5. SEM and EDS Analysis of the Titanium-Porcelain Interface. A specimen was randomly selected from each group and embedded in auto polymerizing acrylic resin for the exposure of the titanium-porcelain interface. SiC papers with different grits from 600 to 2000 were used to polish the sectioned specimens. Then, the specimens were polished with 0.5 μm alumina on a flannelette and ultrasonically cleaned with ethanol and distilled water for 10 min. Finally, after coating the titanium-porcelain interface and elemental composition of the specimens with a layer of graphite, the specimens were evaluated using SEM coupled with EDS.

2.6. Statistical Analysis. All the data were reported as the mean \pm standard deviation (SD). The statistically significant differences were analyzed by one-way analysis of variance (ANOVA) and Tukey's multiple range tests using SPSS software. The statistical significance was defined as $p < 0.05$.

3. Results

3.1. Coating Morphology and Component Analysis. During the MAO treatment, numerous wandering sparkles were observed on the anode surfaces. The reaction became more intense with increasing concentration of MgSiF_6 . The morphology of each group is shown in Figure 1. The specimen surface of the control group was rough and irregular (Figure 1(a)), which was caused by airborne-particle abrasion. The MAO coatings of the 10 g/L MgSiF_6 group (Figure 1(b)) and 20 g/L MgSiF_6 group (Figure 1(c)) were both porous and homogeneous. However, comparing with the pore size of the 10 g/L MgSiF_6 group (0.5–1 μm diameter), larger pores (approximately 1–2 μm diameter) were found on the MAO coating of the 20 g/L MgSiF_6 group. The MAO coating morphologies of the 30 g/L MgSiF_6 group (Figure 1(c)) and 40 g/L MgSiF_6 group (Figure 1(d)) were different from those of other MAO groups. For example, the coating of the 40 g/L MgSiF_6 group was uneven with numerous distributed cracks and plaques.

To further investigate the effect of electrolyte concentration on the MAO coating, EDS was used to analyze the chemical proportion of the MAO coatings. As listed in Table 2, the electrolyte concentration can significantly affect the chemical proportion of the MAO coating. With increasing electrolyte concentration, a higher weight percentage of fluorine was found in the MAO coating. For example, the weight percent of fluorine in the 40 g/L MgSiF_6 group (71.43 ± 0.65 wt%) was significantly higher than that in the 10 g/L MgSiF_6 group (1.86 ± 0.21 wt%). However, the weight percentage of silicon in the MAO coating decreased with increasing concentration of MAO electrolyte.

3.2. Bonding Strength of Titanium-Porcelain Restorations. The bonding strengths of each group are presented in Table 3. We found that, compared with the control group, MAO treatment with appropriate electrolyte concentration can significantly improve the bonding strengths. For instance, compared with the control group, the 10 g/L MgSiF_6 group and 20 g/L MgSiF_6 group showed 1.41-fold and 1.65-fold increases in the bonding strength, respectively. However, no significant difference in bonding strength was found between the control and 40 g/L MgSiF_6 groups ($p > 0.05$).

3.3. SEM Analysis of the Titanium-Porcelain Fracture Surface. The titanium fracture surface of each group after the three-point bending test is shown in Figure 2. As shown, there was no residual porcelain on the specimens of the control (Figure 2(a)) and 40 g/L MgSiF_6 (Figure 2(e)) groups, indicating that the titanium-porcelain bonding fracture occurred between the oxide layer and titanium substrate. In contrast, we found a significant amount of residual porcelain at the titanium surface of the 10 g/L MgSiF_6 (Figure 2(b)) and 20 g/L MgSiF_6 (Figure 2(c)) groups, indicating that crack propagation occurred within the porcelain. Meanwhile, only a small amount of residual porcelain was found at the fracture surface of 30 g/L MgSiF_6 (Figure 2(d)).

3.4. SEM and EDS Analysis of the Titanium-Porcelain Interface. The morphologies of the titanium-porcelain interface of each group are shown in Figure 3. Noticeable cracks and pores can be found at the titanium-porcelain interfaces of the control (Figure 3(a)) and 40 g/L MgSiF_6 groups (Figure 3(e)). Meanwhile, several tiny pores occurred at the interfaces of the 10 g/L MgSiF_6 (Figure 3(b)) and 30 g/L MgSiF_6 groups (Figure 3(d)). However, the interface of the 20 g/L MgSiF_6 group (Figure 3(c)) was uniformly compact and free of cracks and pores.

To further evaluate the effect of interface morphology on oxygen diffusion, linear elemental scanning was employed (Figure 4). It was found that the oxygen curves of the 20 g/L MgSiF_6 group declined sharply over a narrow range after crossing the titanium-porcelain interface (Figure 4(c)), which indicates that the MAO coating can act as a barrier for oxygen diffusion of titanium-porcelain restorations. In contrast, the oxygen curves of other groups declined slowly after crossing the interface of the titanium-porcelain restoration.

4. Discussion

To improve titanium-porcelain bonding strength, the ideal coating should (1) shield against oxygen diffusion into titanium, (2) increase the titanium-porcelain bonding

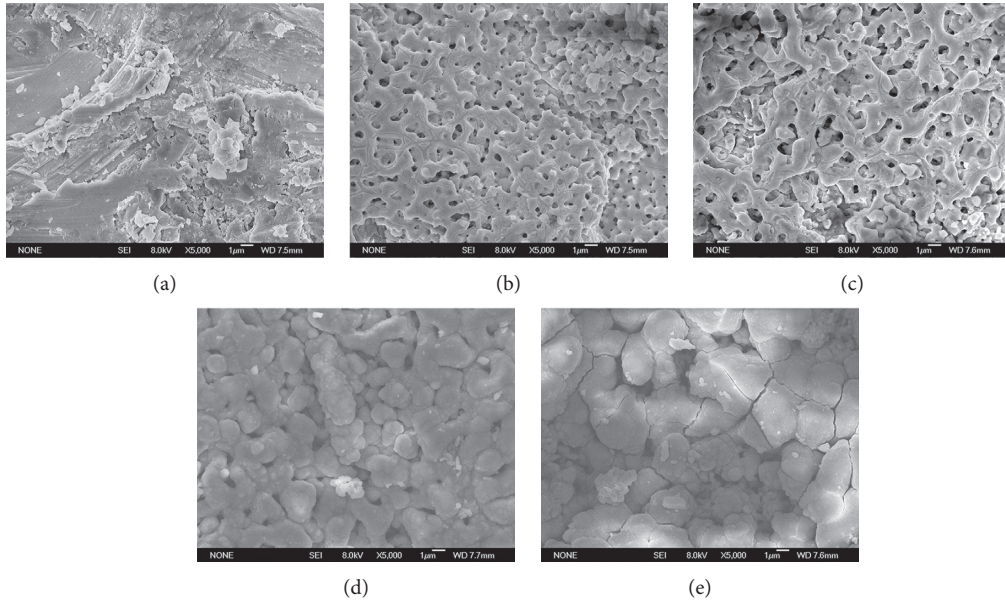


FIGURE 1: SEM microphotographs of MAO coating: (a) control group, (b) 10 g/L MgSiF₆ group, (c) 20 g/L MgSiF₆ group, (d) 30 g/L MgSiF₆ group, and (e) 40 g/L MgSiF₆ group. Bar = 1 μm.

TABLE 2: Effect of electrolyte concentrations on the Mao coating (wt%, $n = 6$).

| Groups | T_i | S_i | O | F |
|---------------------------|--------------|-------------|--------------|--------------|
| 10 g/L MgSiF ₆ | 40.65 ± 0.60 | 8.91 ± 0.51 | 48.59 ± 0.27 | 1.86 ± 0.21 |
| 20 g/L MgSiF ₆ | 40.59 ± 0.92 | 3.16 ± 0.24 | 39.30 ± 1.19 | 16.95 ± 2.03 |
| 30 g/L MgSiF ₆ | 28.83 ± 0.94 | 1.87 ± 0.10 | 24.23 ± 0.74 | 45.07 ± 1.68 |
| 40 g/L MgSiF ₆ | 13.92 ± 0.44 | 0.76 ± 0.13 | 13.89 ± 0.32 | 71.43 ± 2.65 |

* $p < 0.05$, compared to the control group.

TABLE 3: Bonding strength of different titanium-porcelain restorations (mean ± SD, $n = 10$).

| Group | K | Bond strength (MPa) |
|---------------------------|-----|---------------------|
| 10 g/L MgSiF ₆ | 4.7 | 38.18 ± 2.65* |
| 20 g/L MgSiF ₆ | 4.7 | 44.75 ± 2.21* |
| 30 g/L MgSiF ₆ | 4.7 | 31.44 ± 2.04* |
| 40 g/L MgSiF ₆ | 4.7 | 28.04 ± 2.59 |
| Control group | 4.7 | 27.08 ± 3.16 |

* $p < 0.05$, compared to the control group.

strength, and (3) possess excellent biocompatibility. In this study, different concentrations of MgSiF₆ were used to evaluate their effects on the coating's physicochemical properties and titanium-porcelain bonding strength. We found that MAO electrolyte concentration could significantly affect the coating physicochemical properties and subsequent titanium-porcelain bonding strength. The appropriate electrolyte concentration would be necessary to form a porous and homogeneous MAO coating which could effectively prevent oxygen diffusion and enhance bonding strength. However, excessive electrolyte concentration would impact MAO coating homogeneity. The data demonstrated that MAO could improve titanium-porcelain bonding strength only with appropriate electrolyte concentration.

The MAO electrolyte concentration could affect the coating morphology. In theory, increasing the electrolyte concentration could significantly enhance the bombarding energy of anions because of increased anion volume and electrolyte conductivity [13]. In this study, we found that pore sizes on the coating of the 20 g/L MgSiF₆ group were larger than that of the 10 g/L MgSiF₆ group, indicating the higher bombarding energy of the 20 g/L MgSiF₆ group. However, excessively high MgSiF₆ concentration would impair the coating function. For example, we found many cracks on the coating of the 40 g/L MgSiF₆ group, implying that an overly high bombarding energy of anions can damage the integrity of MAO coating. On the other hand, few pores were found on the 30 g/L MgSiF₆ and 40 g/L MgSiF₆ groups, indicating that the pores were covered by the excessive fused mass. Moreover, the MAO electrolyte concentration could affect the coating's chemical proportion. As the MAO electrolyte concentration was increased, a higher weight percentage of fluorine was found in the MAO coating, indicating that more anions were fused into the MAO coating. However, the weight percentage of silicon decreased with increasing MgSiF₆ concentration, which may be caused by the high atom ratio of fluorine and silicon in the anion.

Compared with the control group, the 20 g/L MgSiF₆ group exhibited bond strength enhancements of 65.3%. This

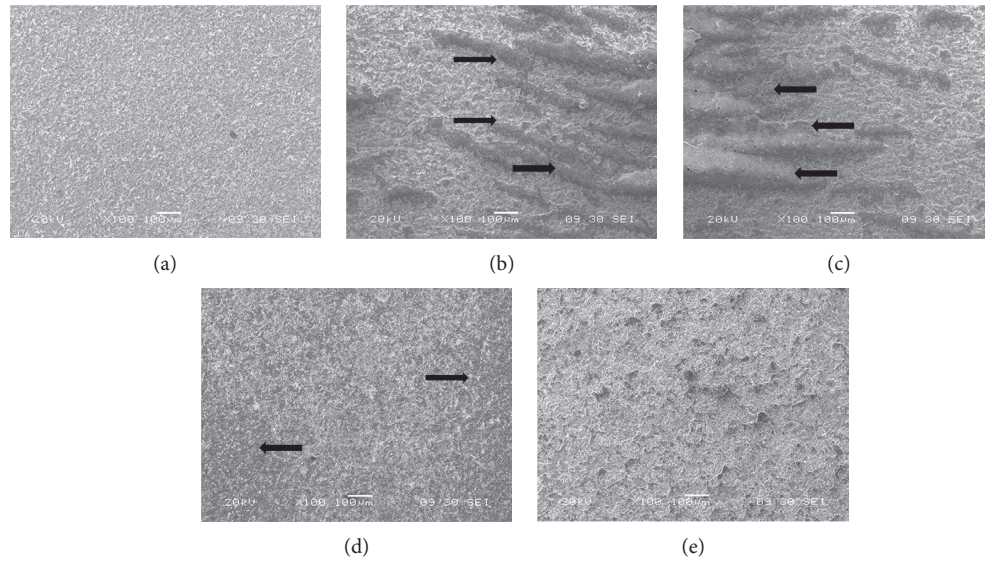


FIGURE 2: SEM microphotographs of the fracture surface of titanium specimens: (a) control group, (b) 10 g/L MgSiF_6 group, (c) 20 g/L MgSiF_6 group, (d) 30 g/L MgSiF_6 group, and (e) 40 g/L MgSiF_6 group. Bar = 100 μm . The residual porcelain is indicated by arrows.

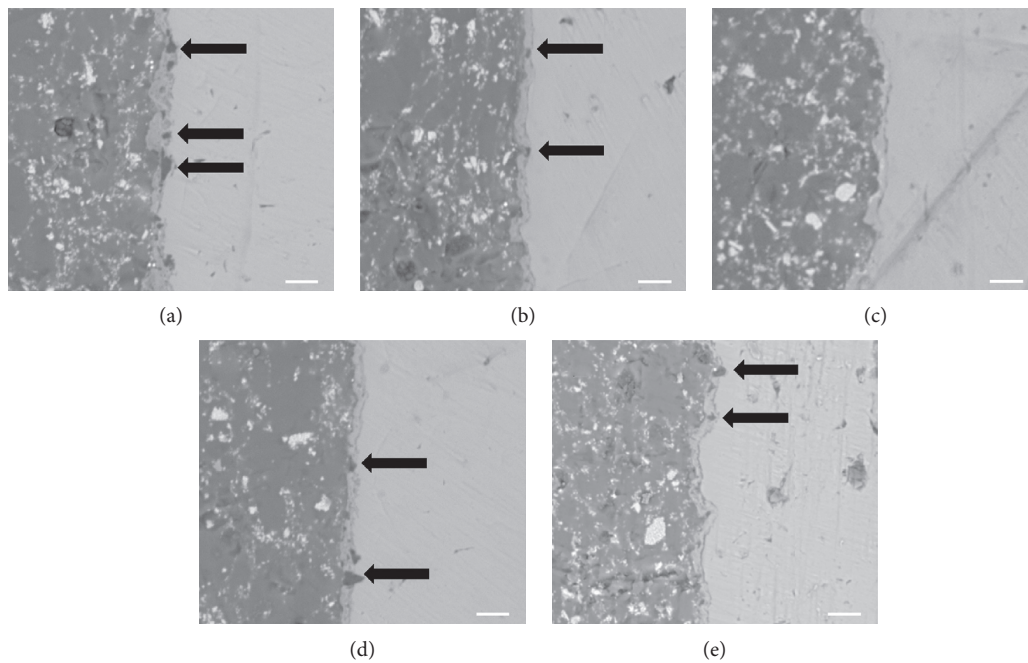


FIGURE 3: SEM microphotographs of titanium-porcelain interface: (a) control group, (b) 10 g/L MgSiF_6 group, (c) 20 g/L MgSiF_6 group, (d) 30 g/L MgSiF_6 group, and (e) 40 g/L MgSiF_6 group. Bar = 10 μm . The pores and cracks are indicated by arrows.

result can be indirectly proved by the fracture surface observation. The titanium-porcelain fracture of the 20 g/L MgSiF_6 group occurred in the interior of porcelain. It demonstrates that the bonding strength of the 20 g/L MgSiF_6 group had exceeded the mechanical strength of bonding porcelain. The improved bonding strength of the 20 g/L MgSiF_6 group should stem from the coating morphology, prevention of oxygen diffusion, and coating chemical composition. First, the improved bonding strength can be attributed to the porous morphology of MAO coating. The pores (approximately 1-2 μm diameter) on the MAO coating

of the 20 g/L MgSiF_6 group would enhance the interlock between titanium and porcelain. According to results on the coating's morphology, more pores and larger pore diameters were found on the MAO coating of the 20 g/L MgSiF_6 group, compared to the other groups. Other studies have proved that porcelain can fuse into the porous coating and physically interlock with titanium substrate [6]. In addition to this, studies have demonstrated that interlocking is an important bonding force for titanium-porcelain restorations [14, 15]. Second, the improved bonding strength may stem from the protective effect of MAO coating on the oxygen

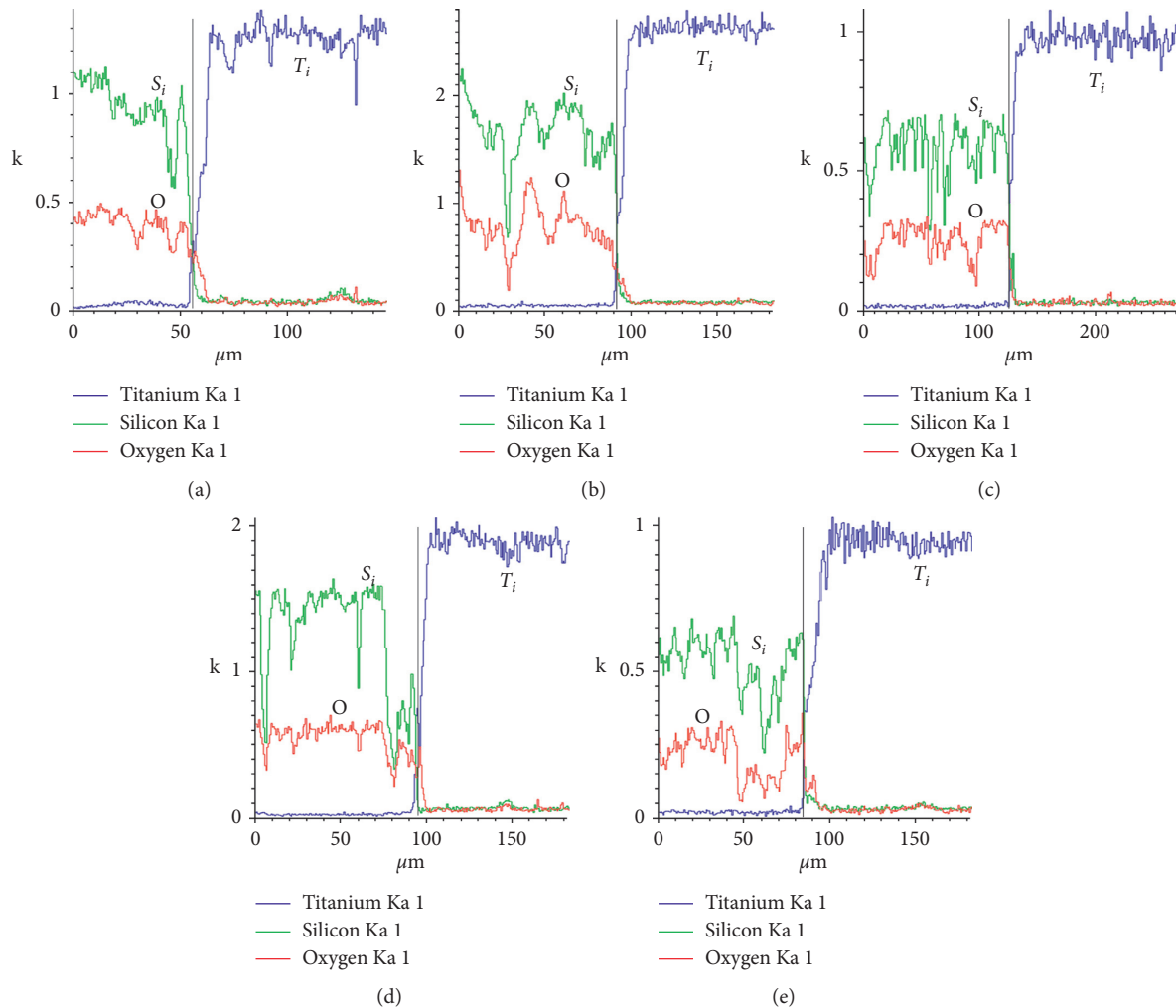


FIGURE 4: Elemental line scanning of titanium-porcelain interface: (a) control group, (b) 10 g/L MgSiF_6 group, (c) 20 g/L MgSiF_6 group, (d) 30 g/L MgSiF_6 group, and (e) 40 g/L MgSiF_6 group.

diffusion. As shown, the oxygen curves of the 20 g/L MgSiF_6 group declined rapidly in a narrow range after crossing the interface, indicating that the MAO coating effectively controlled the diffusion of oxygen into titanium. The oxygen-diffusion barrier of the 20 g/L MgSiF_6 group may have resulted from the impact structure at the titanium-porcelain interface. For example, many pores and cracks were found at the interface of other groups. In the porcelain application, the oxygen could diffuse into the titanium substrate through these structural defects between titanium and porcelain. Third, the improved bonding strength may result from chemical changes caused by anions bombarding at the titanium-porcelain interface. After MAO treatment, silicon and fluorine were found on the surface of titanium substrate. It was found that the same element both in coating and porcelain would improve chemical bonding between titanium and porcelain, for example silicon. The chemical bonding formed during porcelain sintering and played an important role in the titanium-porcelain bond. For example, Lin found a mutual diffusion of silicon formed at the titanium-porcelain interface. The elemental silicon that diffused

to the titanium substrate comes from the silica coating made by sol-gel dipping [5].

Furthermore, excessively high MgSiF_6 concentrations would impair the bonding strength between titanium and porcelain. When the MAO concentration of MgSiF_6 was above 20 g/L, the bonding strength decreased with increasing MAO concentration. The weakened bonding strength may result from changes to the coating's chemical composition. Chemical bonding has been proved to be the major bonding force of titanium-porcelain restorations [16]. In our previous study, we found that, excluding oxygen, silicon (21.2%) and fluorine (2.6%) are major nonmetallic elements in the bonding porcelain [10]. However, the weight percent of silicon in the 40 g/L MgSiF_6 group (0.76 ± 0.13 wt %) was far lower than that in the bonding porcelain. Moreover, the weight percent of fluorine in the 40 g/L MgSiF_6 group (71.43 ± 0.65 wt%) was far higher than that in the bonding porcelain. Change to the chemical proportion would increase the mismatch of thermal expansion coefficient (TEC) between titanium and porcelain. It was demonstrated that the TEC mismatch would decrease the

bonding strength between titanium and porcelain [17]. On the other hand, the weakened bonding strength may stem from the morphology of the 40 g/L MgSiF₆ group. In contrast with the 20 g/L MgSiF₆ group, the coating of the 40 g/L MgSiF₆ group was uneven with numerous cracks and plaques distributed. Decreased morphological integrity would increase oxygen diffusion into titanium and impair the bonding strength.

5. Conclusions

In this study, we found that different concentrations of the MAO electrolyte can influence the morphology of the MAO coating and result in different effects on the bonding strength. MAO treatment with 20 g/L MgSiF₆ can significantly improve the titanium-porcelain bonding strength.

Although more anions were fused into the MAO coating with increasing electrolyte concentration, the bonding strength decreased when the concentration exceeded 20 g/L. In addition, it was demonstrated that overly high MgSiF₆ concentrations have a negative influence on the structural homogeneity of the coating, interface integrity, and oxygen diffusion. Therefore, titanium-porcelain restorations treated by MAO are suitable for clinical use when appropriate MgSiF₆ concentrations are used.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Authors' Contributions

The authors Jing Lv and Mujie Yuan contributed equally to this work.

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