

Article

Effects of Graphene on the Wear and Corrosion Resistance of Micro-Arc Oxidation Coating on a Titanium Alloy

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Abstract: In order to improve the wear and corrosion resistance of micro-arc oxidation (MAO) coating on a Ti-5Al-1V-1Sn-1Zr-0.8Mo alloy, 0–0.20 g/L graphene was added to the electrolyte to prepare micro-arc oxidation coating. The thickness, roughness, micro-morphology, and composition of the MAO coating were characterized, and the wear and corrosion resistance of the coating was tested and analyzed. The results show that with 0.05 g/L of graphene in the electrolyte, the roughness of the coating decreased from 56.76 μm to 31.81 μm . With the increase in the addition of graphene, the microstructure of the coating became more compact, the diameter of micro-holes and micro-cracks decreased, and the corrosion resistance of the coating improved. The wear tests showed that the mass loss of the coating at the early wear stage (0–100 revolutions) was greater than that at the later stage (100–250 revolutions), and the wear resistance of the coating obtained by the addition of 0.10 g/L of graphene was the highest. With 0.10 g/L of graphene, the adhesion force between the coating and the substrate alloy is the largest, reaching 57.1 N, which is 9.98 N higher than that without graphene. After salt spray corrosion for 480 h, the coating with graphene has better corrosion resistance than that of a graphene-free coating.

Keywords: micro-arc oxidation; graphene; Ti-5Al-1V-1Sn-1Zr-0.8Mo alloy; wear resistance; corrosion resistance



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1. Introduction

Titanium alloy is a lightweight alloy. It is widely applied to aerospace, machinery, automotive, petroleum, and other fields due to its low density and high specific strength [1–4]. However, titanium alloys still have some insufficient properties, such as poor wear resistance and poor thermal conductivity. In order to improve the properties of the titanium alloy, surface treatment is usually carried out [5–8]. Micro-arc oxidation (MAO) is a technology that uses an in situ growth of ceramic coating directly on Al, Mg, Ti, and Zr surfaces. The oxide coating obtained by MAO is bonded to the metal closely, and the coating has good wear resistance, corrosion resistance, and electrical insulation properties [9–11].

It has been found that electrolyte compositions play a crucial role in the MAO process. The substrate alloy is melted by the micro-arc and solidified by the electrolyte. During this process, the conductivity of the electrolyte and composition of the coating is changed, which means that the performance of the coating is changed. Therefore, composite coatings are prepared by adding the nano-particles (such as ZrO_2 , and graphene) into the electrolytes to improve the density, hardness, corrosion, and wear resistance of the coatings [12–17].

According to the filling and sealing effect of graphene on the micro holes of MAO coatings, graphene has been shown to be effective in the micro-arc oxidation of other metals. However, it is rarely used as an additive in the micro-arc oxidation of titanium

alloys, especially of the Ti-5Al-1V-1Sn-1Zr-0.8Mo alloy. In this study, graphene was added to the electrolyte. The influence of graphene on the morphology, wear resistance, and corrosion resistance of the coating was studied.

2. Experimental

2.1. Materials and Preparation

The sample for micro-arc oxidation is Ti-5Al-1V-1Sn-1Zr-0.8Mo alloy (Northwest Institute for Non-ferrous Metal Research, Xi'an, China). The samples were cut into a flake-like 30 mm × 20 mm × 5 mm and were then polished by sandpaper, step by step, and washed with acetone. In order to reduce the edge effect phenomenon during the MAO process, the edges of the specimen were polished and smoothed. The specimens were cleaned with acetone in an ultrasonic shock cleaning machine (Fuyang Technology Group Co., Ltd., Shenzhen, China) for 15 min, respectively. The electrolyte solution was comprised of 16 g·L⁻¹ of Na₂SiO₃, 2.0 g·L⁻¹ of Na₂EDTA, and 10.0 g·L⁻¹ of Na₂HPO₄. In order to study the influence of graphene on MAO coatings, they were selected as shown in Table 1. The graphene was dispersed by ultrasonic shock for two hours before being added into the electrolyte (Tulinjinhua Technology Co., Ltd., Shenzhen, China). The details of the graphene are shown in Table 1.

Table 1. The details of the graphene.

Number of Layers	Particle Size	Density	Moisture Content
1–3	7–12 µm	0.01–0.02 g/L	≤2%

The electrolyte containing graphene was stirred with a stirrer throughout the MAO process. The electrolyte, by a circulating water cooling system, was controlled between 20 °C and 30 °C. The applied voltage, frequency, duty cycle, and oxidation time are also shown in Table 2.

Table 2. The specimens code and MAO parameters.

Specimens Code	Graphene Addition (g/L)	Conductivity of Electrolyte (S/m)	Forward/Negative Voltage (V)	Frequency (Hz)	Duty Cycle (%)	Oxidation Time (min)
S0	0	11.42	420/80	100	50	20
S1	0.05	12.67				
S2	0.10	14.41				
S3	0.15	14.58				
S4	0.20	14.65				

2.2. Characterization

The thickness of the coating was measured by an eddy current thickness meter (HCC-25, Hitachi Analytical Science Ltd., Shanghai, China). The average value was measured by 10 points at different positions on the front and back of the sample. The morphology images of the coating were analyzed by a scanning electron microscope (QUANTA FEG 650, FEI, Hillsboro, OR, USA), and the images were used to measure porosity with Image J software (Version 1.8.0, National Institutes of Health, Bethesda, MD, USA). Laser confocal microscopy (LSM700, Zeiss, Oberkochen, Germany) was used to measure the roughness (Ra) and 3D morphology. Six equal distance locations of the X-axis and Y-axis were selected for line scanning from an area of 1.3 mm × 1.3 mm. To measure the roughness of the coatings, the average of six data was taken as the roughness result of the coating. The X-ray diffraction (XRD) analysis was performed on an X-ray diffract meter (PW1700, Cu Kα radiation, Rigaku, Tokyo, Japan) to identify the different phases present in the coatings. The scans were acquired from 20° to 80° at a step scan of 0.02°. XPS analysis was conducted with ESCALAB-250Xi X-ray photoelectron spectroscopy instrument (Thermo

Fisher Scientific, Waltham, MA, USA). The test parameters were: monochrome Al K α ($h\nu = 1486.6$ eV), and fixing through an analyzed energy of 20 eV.

The adhesion between coating and alloy was tested by a coating adhesion automatic scratch instrument (WS-2005, ZhongkeKaihua Technology Development Co., Ltd., Lanzhou, China). The wear experiments were performed by mass-loss on a sandpaper-on-flat reciprocal tribometer (DZ-322TABER, Dazhong Instrument Co., Ltd., Dongguan, China). The wear time was 5 min with the revolving speed of 50 r/min, and the load was 2.5 N. The mass loss after every 50 revolutions was calculated. The corrosion resistance of the coating was tested by a salt spray cabinet (Zhongkehuaishi Instrument Co., Ltd., Beijing, China), which was in accordance with the China standard (GB T10587-2006), as shown in Table 3.

Table 3. Details of the corrosion resistance test.

Corrosive Liquid	Temperature	Corrosion Time	pH
NaCl (5%)	35 ± 2 °C	680 h	6.8–7.2

3. Results and Discussion

3.1. Roughness and Thickness

The effects of graphene addition on the thickness and roughness of MAO coatings are shown in Figure 1. As can be seen, the roughness of the coating is $56.76 \mu\text{m}$ (Ra) with no graphene in the electrolyte, and the roughness is significantly reduced with the addition of graphene. With the 0.05 g/L of graphene addition, the roughness is the lowest, with Ra at $31.81 \mu\text{m}$. It can be observed that the thickness of the MAO coating increased with the increase in the graphene addition. The MAO coating is formed by the molten substrate alloy during micro-arc discharge and then by contacting the electrolyte and cooling. The molten alloy is spewed out with micro-arc discharge, which is closely related to the roughness of the coating. The roughness of the MAO coating is mainly associated with the intensity of the micro-arc reaction. The graphene as an additive is moved to the substrate and coating surface by the electric field action and the stirring of the electrolyte and deposited into the coating in the form of physical encapsulation. The probability of micro-arc discharge is increased. Under the condition of invariable voltage during the MAO process, the number of micro-arcs increased and the energy of a single arc decreased. As the intensity of the reaction decreases, the degree of MAO is more gentle, and the roughness of the coating is smaller. In addition, the graphene is deposited into the micro-arc discharge channel continuously, which can fill the micro holes and cracks during melting, cooling, and nucleation processes. Thus, the roughness of the coating layer is reduced [18].

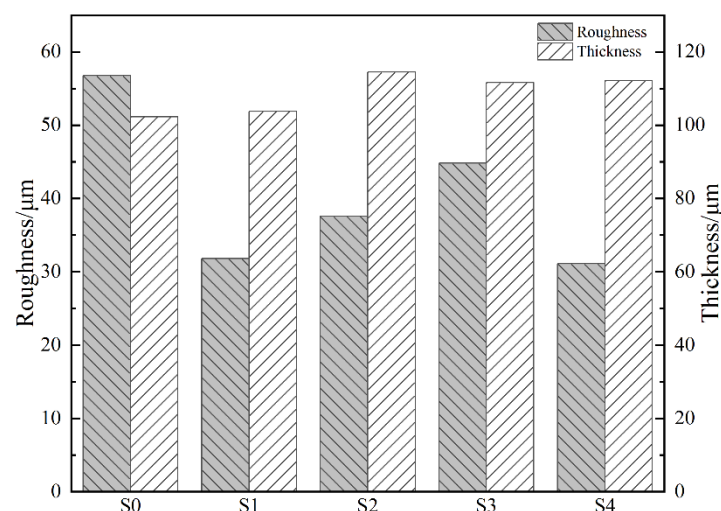


Figure 1. Thickness and roughness of MAO coatings.

Graphene's effect on the roughness of the coating can also be confirmed from the 3D morphology shown in Figure 2. As can be seen in Figure 2a, the altitude variation of the coating between the “high altitude area” and the “concave area” is about 1000 μm . With 0.05 g/L and 0.10 g/L of graphene in the electrolyte, the surface of the coatings became smooth, and the linear scanning results showed that the altitude variation of the coatings was reduced, as shown in Figure 2b,c. As the graphene addition is 0.15 g/L and 0.20 g/L, the variation of the coating is slightly increased, but the roughness is still small.

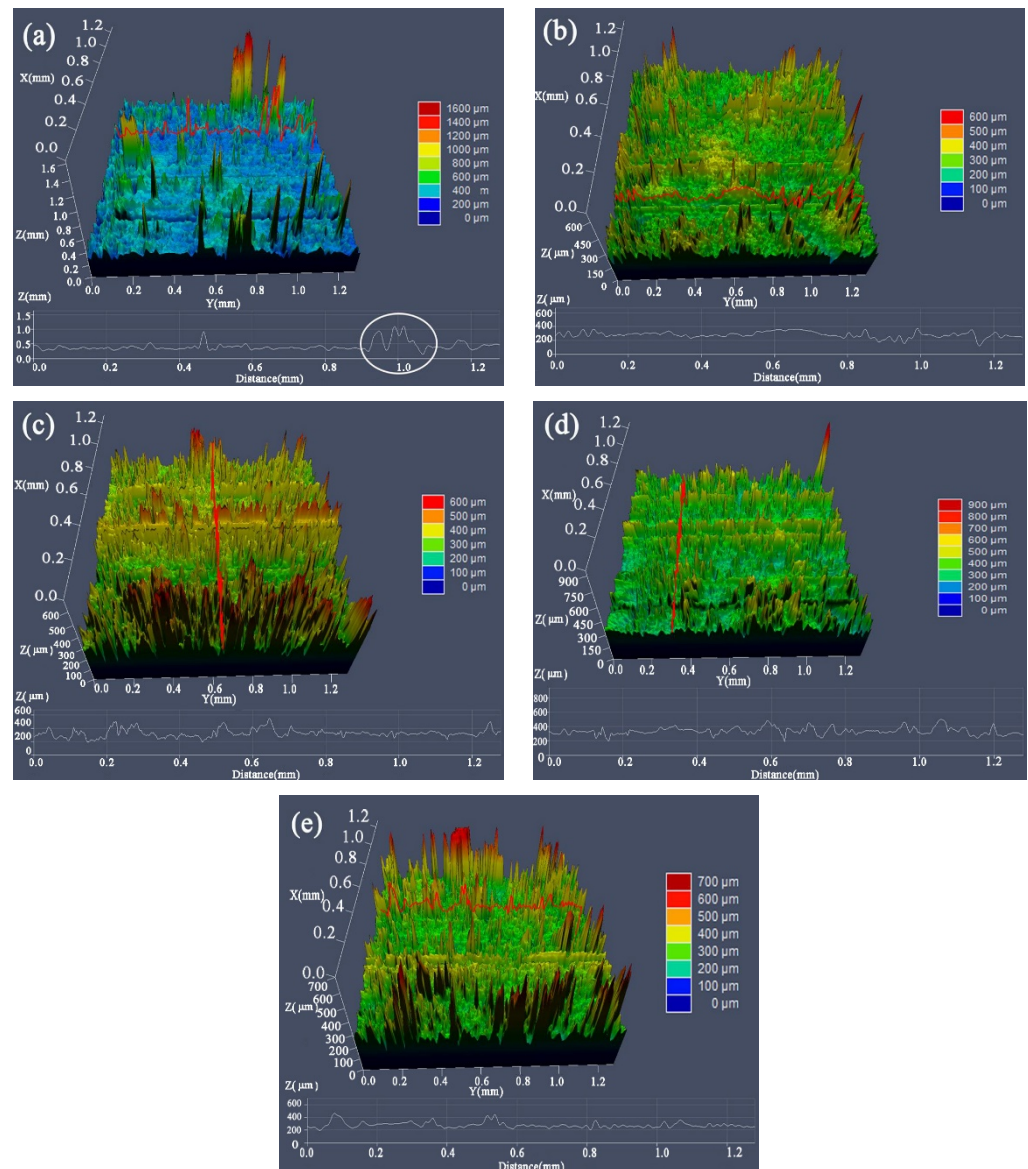


Figure 2. The 3D morphology of MAO coatings. (a) S0; (b) S1; (c) S2; (d) S3; (e) S4.

3.2. Morphology

Figure 3 shows the surface morphology of the MAO coating. It can be observed in Figure 3a there are large-diameter micro-pores and cracks on the coating surface, which were prepared without graphene in the electrolyte. The micro-cracks were caused by the shrinkage of molten metal during rapid solidification, and the micro-pores are the channels for discharge breakdown. With the graphene in the electrolyte, the crater morphology on the surface of the coating can still be seen, but the diameter of the micro-pores is significantly reduced, and some micro-pores are filled by the generated oxides, as can be seen in Figure 3d. Because of the addition of graphene, the conductivity of the electrolyte is increased, as shown in the table. In such a constant voltage condition, the equivalent

resistance (consisting of the electrolyte, the sample, and the electrolytic cell) decreases, resulting in an increase in current. This means that the energy for coating formation is increased. However, the graphene could increase the number of micro-arcs, which causes a decrease in the diameter of a single arc and the shortening of the duration of discharge. Therefore, the energy generated by arc discharge can only melt the newly formed oxides at the interface between the coating and the substrate, resulting in the reduction in the diameter of the micro-pores.

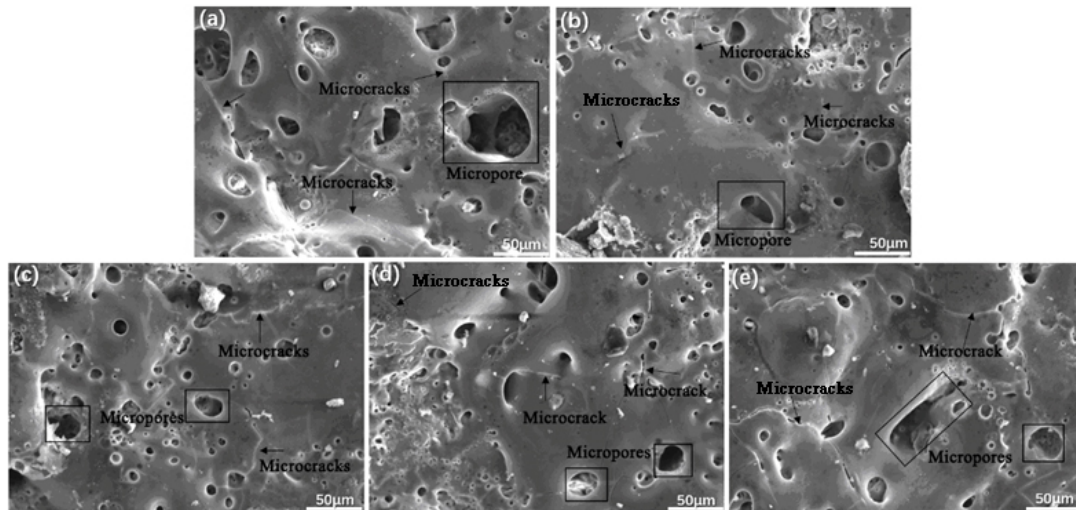


Figure 3. Surface morphology of MAO coatings (a) S0; (b) S1; (c) S2; (d) S3; (e) S4.

The porosity of different coating surfaces is shown in Figure 4. The porosity is 4.386% for a coating prepared by an electrolyte without graphene. The porosity of S2 is the minimum at 3.188% with 0.10 g/L graphene in the electrolyte. The decrease in porosity contributes to the improvement of the corrosion resistance of the coating. The compact coating is mainly due to the following two reasons: Firstly, the addition of graphene leads to a smaller diameter of pores and fewer cracks in the coating during the MAO process. Secondly, the graphene as an additive is moved to the substrate surface, and deposited into the coating in the form of physical encapsulation.

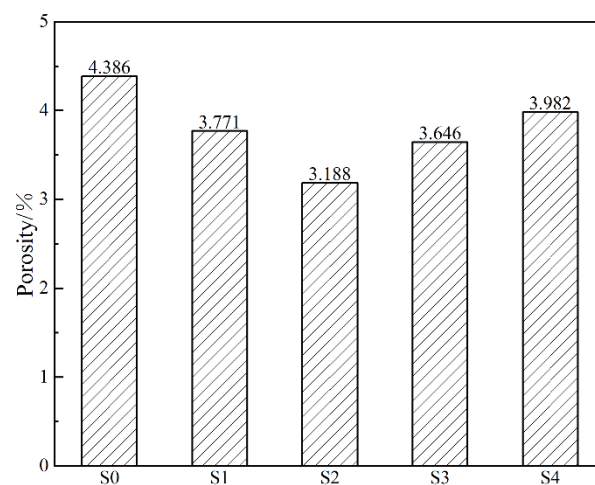


Figure 4. Porosity of MAO coatings.

The cross-section morphologies of MAO coatings are illustrated in Figure 5. As shown, the MAO coatings (inside the red dotted line) consist of an outer porous layer and an inner compact layer. Figure 5a shows that the number and diameter of micro-pores are more and larger, compared with other coatings with graphene in the electrolyte. The coating

thickness and compactness exhibit an increase with the increase in the graphene addition. The increase in thickness and compactness is also helpful for improving the corrosion resistance of the coating.

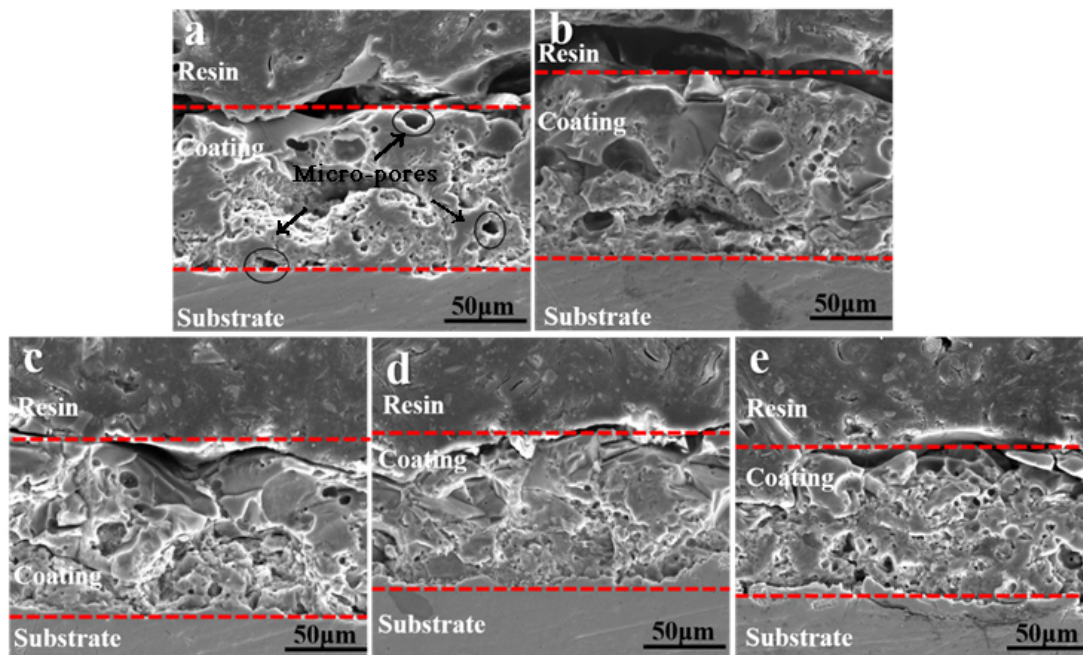


Figure 5. Cross-section morphology of MAO coatings (a) S0; (b) S1; (c) S2; (d) S3; (e) S4.

3.3. Phase Compositions

XRD patterns of the MAO coatings with different graphene additions in the electrolyte are shown in Figure 6. It is evident from the figure that the coatings were mainly composed of rutile TiO_2 and anatase TiO_2 . Notably, the chemical compounds associated with the graphene in the electrolyte and titanium were not detected in the coatings, which meant that the coatings were compact. The XPS results of MAO coating on S4 were shown in Figure 7. With graphene (0.2 g/L) in the electrolyte, a peak of carbon was detected, indicating that the C entered the coating. The peak binding energies of C1s are 284.72 eV, 286.79 eV, and 288.47 eV, respectively, corresponding to C-C, C-O-H, and O-C=O chemical bonds. This means that the C appeared as these chemical bonds in the MAO coating. This is due to the graphene's oxidation or reaction with the electrolyte during the micro-arc oxidation process. Rutile TiO_2 and anatase TiO_2 phases are different crystal types of TiO_2 . Rutile TiO_2 is a relatively stable crystal type, while anatase TiO_2 can be transformed into a more stable rutile TiO_2 under certain conditions. After the addition of graphene in the electrolyte, there was a slight change in the XRD patterns, as shown in the S1 pattern. The diffraction peak of rutile TiO_2 is stronger than others.

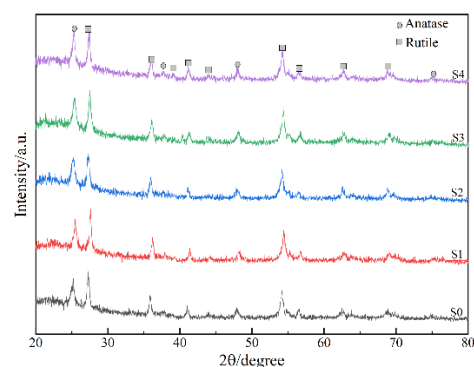


Figure 6. XRD pattern of MAO coating.

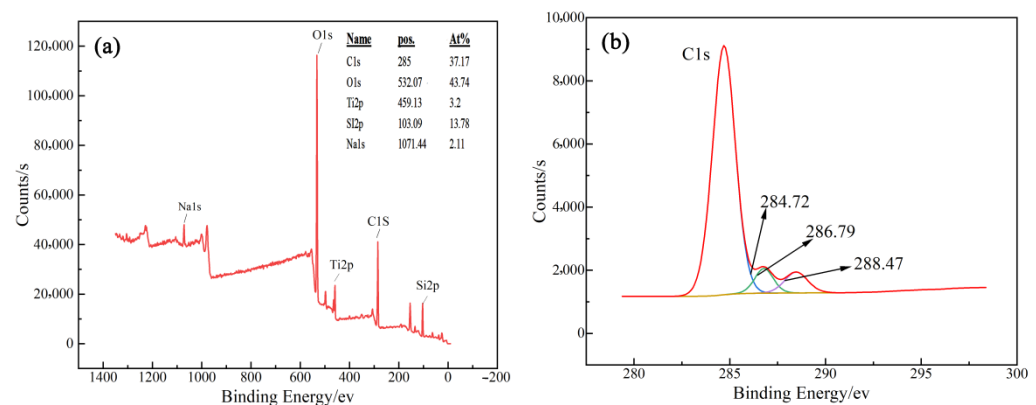


Figure 7. XPS analysis of MAO coating on S4 (a) XPS spectra of MAO coating, (b) XPS high resolution spectra of C detected.

3.4. Adhesion Force

The adhesion force between the substrate and coating was tested in the form of conductivity. When the applied load exceeded a certain critical value, the coating was scratched, and the conductivity value was mutated due to the conductivity of the titanium alloy substrate. This loading was recorded as the adhesion force of the coating. The scratch morphology and adhesion force are shown in Figure 8.

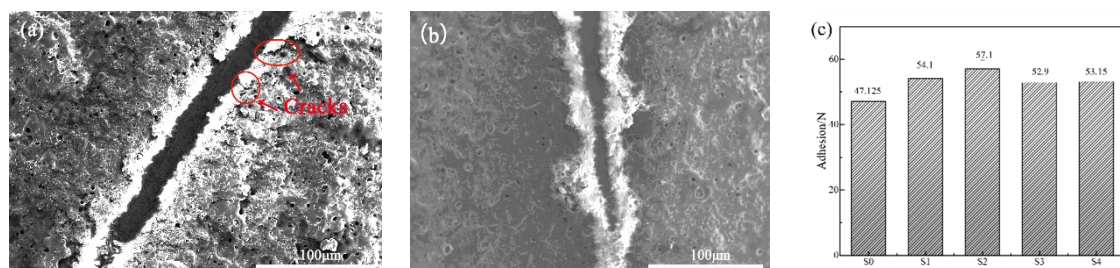


Figure 8. Scratch morphology and adhesion force of the MAO coating. (a) Scratch morphology of S0; (b) scratch morphology of S2; (c) adhesion force.

As can be seen from Figure 8a,b, some small and grinding ceramic particles were scattered on the surface of the scratch scar, indicating a typical plowing phenomenon. The cracks were also observed in Figure 8a with 0.10 g/L of graphene in the electrolyte, though the width of the scratches became narrow and the ceramic particles decreased, indicating that the coating was more difficult to cut through, seen in Figure 8b. The adhesion force of the coating without graphene is 47.125 N, and improved after graphene was added (illustrated in Figure 8c). The maximum adhesion force of the S2 coating prepared with the addition of 0.10 g/L of graphene is 57.1 N. The reason for the improvement is that the addition of graphene increases the density of the coating, requiring more force to break through the coating during testing.

3.5. Wear Resistance

Figure 9 shows the wear loss of the coating. The wear was the most serious on the first 100 revolutions and decreased during the 100th–250th revolutions. At the early stage of wearing, the friction pair was in contact with each other under the action of loading. There are two reasons for the increase in wear loss. Firstly, the outer layer of the coating is relatively loose. Secondly, the end of micro-arc discharge only occurs on the local surface of the coating, and high micro convex particles are formed. This caused the coating to fall off easier after contact with the sandpaper, and the wear loss was large. After 100 revolutions of wearing, the loose layer and higher micro-convex body had fallen off and the contact area between the friction pair increased, so the mass loss decreased gradually. By comparing the

mass loss before and after adding graphene, it can be found that graphene reduced the mass loss of the coating during the wearing process. The wear resistance of the coating obtained by adding 0.10 g/L graphene is the highest, and the mass loss with 250 revolutions was only 15.8 mg. There are two main reasons for the improvement of the wear resistance of the coating by graphene. First of all, the coating prepared with graphene in the electrolyte has higher compactness and wear resistance. Secondly, graphene enters into the coating during the MAO process, which can also play a certain role in lubrication [14]. In addition, there are holes formed by micro-arc discharge on the surface of the coating, which is beneficial to the storage of a lubricating medium in practical applications and can further reduce the wear caused by friction.

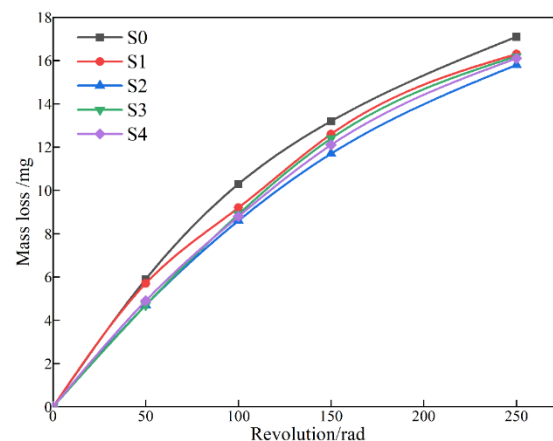


Figure 9. The mass loss of MAO coating.

3.6. Corrosion Resistance

Figure 10 shows the corrosion morphology of the salt spray corrosion coating for different durations. From the morphologies, the MAO coatings have excellent corrosion resistance in a salt spray corrosion environment. After 132 h, some black pitting corrosion appeared on the surface of the coating, especially appearing on the micro holes of arc discharge or the edges of the specimens. With the prolongation of corrosion time, as shown in Figure 10a, comparing the pitting corrosion at the same position of the coating on S0 after 480 h corrosion and 680 h corrosion, the diameter of small black spots gradually expanded. By comparison to the coating on S0, both the number and area of the pitting corrosion on the coating surfaces of S1–S4 were lesser. This indicated that the corrosion resistance of the coating was improved with graphene in the electrolyte.

The corrosion resistance of the MAO coating is mainly affected by the thickness and compactness of the coating. The corrosion medium of salt spray is Cl^- , which has a very small radius, which can pass through the coating and enter it, resulting in pitting corrosion. As different additions of graphene were added into the electrolyte, the pore diameter and cracks on the surface of the coatings were reduced. The graphene filled the pores, prevented the invasion of corrosive ions, inhibited the diffusion pathway of aggressive electrolytes, and proved a good barrier against ion penetration.

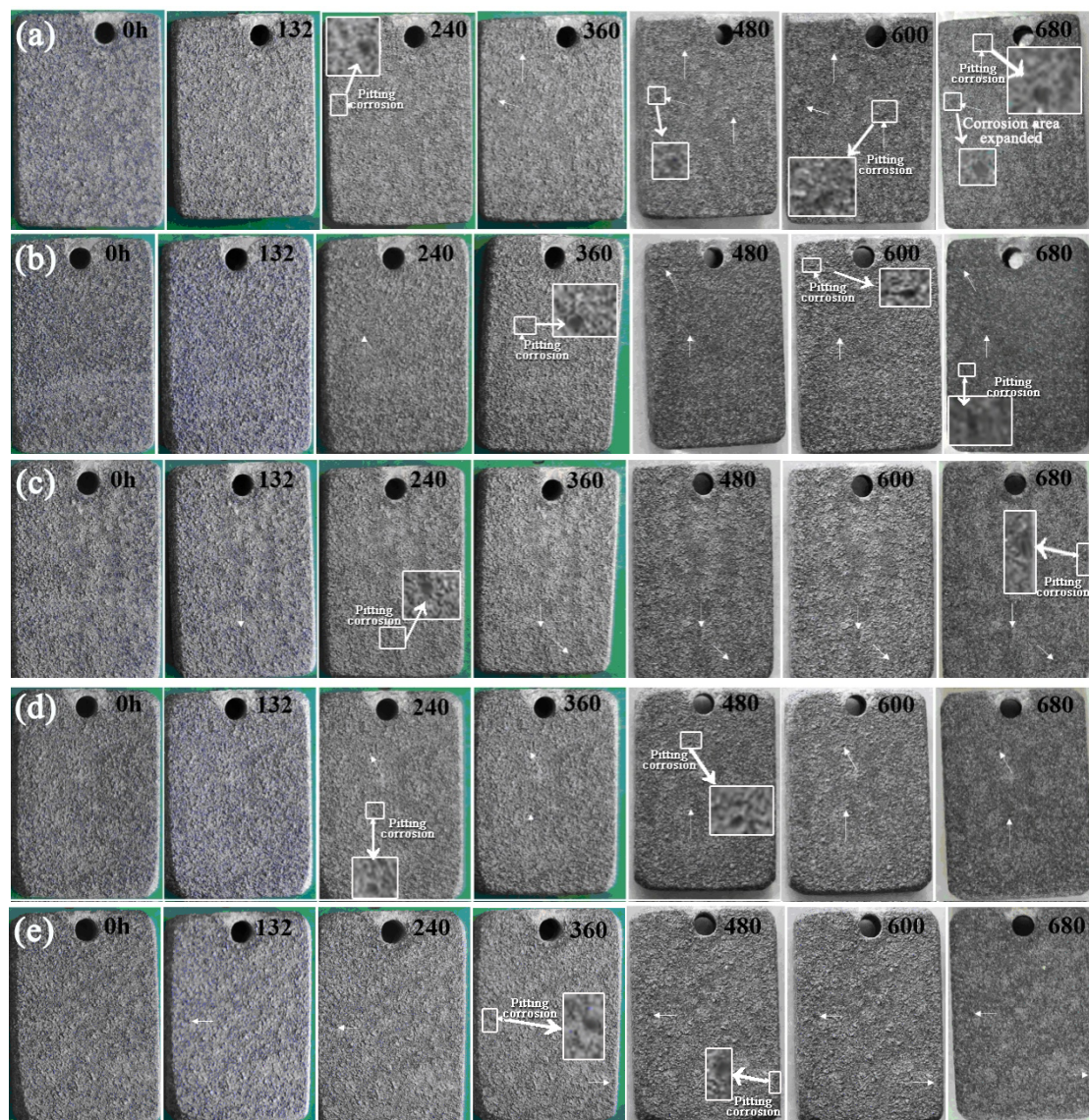


Figure 10. MAO coating after different time by salt spray corrosion. (a) S0; (b) S1; (c) S2; (d) S3; (e) S4.

4. Conclusions

The microstructure and resistances of the MAO coating obtained from a graphene electrolyte were investigated in this paper. In conclusion, the optimal additive amount of graphene is 0.10 g/L, and the main results are summarized as follows:

- (1) After adding graphene into the electrolyte, the roughness of the coating is significantly reduced. When the amount of graphene is 0.05 g/L, the lowest roughness is 31.81 μm .
- (2) With the increase in graphene content, the microstructure of the coating becomes more compact and the diameter of micro-pores and micro-cracks decreases. When the amount of graphene was 0.1 g/L, the lowest porosity was 3.188%.
- (3) The pitting corrosion of the coating without graphene increased after 480 h corrosion, and the corrosion area expanded after 680 h. The graphene filled the pores and prevented the invasion of corrosive ions, and the corrosion resistance of the coating was improved with graphene in the electrolyte.
- (4) When the amount of graphene was 0.10 g/L, the highest adhesion force of the S2 coating was 57.1 N. It exhibited the highest wear resistance, and the mass loss was only 15.8 mg after 250 revolutions of wearing.

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