Influence of solid lubricant WS$_2$ on the tribological properties of plasma electrolytic oxidation coating of ZL109

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Abstract

Ceramic coatings with solid lubricant were prepared on the surface of ZL109 by plasma electrolytic oxidation (PEO) and burnishing technologies. The tribological properties of composite coatings were investigated by using a ball-on-plate friction and wear tester. The wear morphologies were tested and analyzed by SEM and EDS. The results showed that the discharge micro-pores and the micro-dimple defects on the ceramic coating surface were effectively filled with WS$_2$ nanoparticles during the burnishing process. The grounding procedure before burnishing can decrease the friction coefficient. The composite coating prepared by grounding and burnishing after PEO process has a lowest friction coefficient of 0.07. The coupling ball of the composite coating prepared by only burnishing after PEO process has a lowest wear rate of $4.97 \times 10^{-6} \text{mm}^3 \text{N}^{-1}\text{m}^{-1}$. The interaction between the hard substrate of the PEO coating and the solid lubricant stored in the porous layer of the PEO coating can explain the anti-friction mechanism of the composite coating.

1. Introduction

Metal alloys such as magnesium, aluminum, titanium and their composite materials have numerous advantages, such as low density and high specific strength. Due to such characteristics, they are often used to replace traditional materials, such as high-strength steel, to make relative moving parts, which play an important role in aerospace, water transportation, automobile, and other energy and emission-reduction industries [1–3]. However, due to its disadvantages, for example having relatively active chemical properties, poor stability and low hardness, their use are limited at higher friction temperatures and contact stress conditions which can cause high friction coefficient and serious adhesive wear [4]. Therefore, several surface technologies including anodization [5], chemical conversion coating [6], electroplating [7], PVD [8], nitriding [9, 10], and PEO [11–13] are utilized to expand their use.

The use of PEO technology to form a ceramic film with high bonding strength, anti-wear and corrosion resistance properties on the surface of ZL109 alloy is currently recognized as a surface treatment technology with great prospects [4]. Nevertheless, the volcanic-like morphology formed at the surface discharge micro-pores during the PEO process leads to an increase in the roughness of the ceramic coating. A high friction coefficient under dry sliding condition not only increases frictional power consumption, but also exacerbates component failure due to wear of the two coupling materials [14, 15]. Therefore, the PEO composite layer is widely studied for improving the anti-friction performance of the ceramic coatings.

At present, the techniques for improving the anti-friction performance of PEO ceramic coatings can be classified into two categories.

First, the one-step method involves directly adding functional micro-nano particles into the electrolyte and can be incorporated into the ceramic coating under the action of electrophoresis force of the PEO process to become a discrete phase in the hard ceramic phase. When employed, the release of the incorporated micro-nano particles creates an anti-friction effect. Zhang et al [16] formed a PTFE/PEO composite coating on the aluminum alloy prepared by introducing 5 wt% of PTFE into the electrolyte, and found that the composite coating not only has better tribological performance, but also has an excellent anti-corrosion property. Chen

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et al [17] added different concentrations of graphene particles into the electrolyte and showed that the addition of graphene particles can reduce the breakdown voltage and promote the reaction of PEO of aluminum alloy. Meanwhile, the thickness and hardness of the PEO coating also increase. Zuo et al [18] displayed that the composite coatings incorporating graphene oxide possess compact micro-structures and better tribo-corrosion resistance in the NaCl aqueous solution. Besides this, different micro-particles with special functions, such as ZrO2 [19], CeO2 [20], MoS2 [21] Al2O3/TiO2 [22], MoO3 [23], SiC [24, 25], and Si3N4 [26] also are reported.

Second, the two-step method involves filling functional materials into the microporous structure of the PEO ceramic coating by some post-treated techniques to form a functional composite coating. Qin et al [27] burnished a thin MoS2 film on the PEO coating of Ti6Al4V substrate with surface texture and found that the composite coating has a much longer life and lower friction. Lu et al [28] impregnated PTFE on PEO coating of aluminum alloy at a vacuum condition to prepare a self-lubricating composite coating, which has an excellent friction coefficient and relatively lower wear rate. Li et al [29] fabricated self-lubricating composite coatings via immersing the PEO coatings into molten liquid paraffin and found that the composite coatings exhibit a lower friction coefficient and lower wear rates. Ma et al [30] prepared a composite coating with WS2 particles by electrophoresis deposition on PEO coating, which exhibits excellent anti-wear and self-lubricating properties. Furthermore, thermal spraying [31], electrostatic powder spraying [32], texture filling [33], grafting [34] are also employed on the PEO coatings.

The above studies showed that the self-lubricating particles stored in the porous structure of the PEO ceramic film not only can increase the abrasion resistance and corrosion resistance of the composite film layer, but also can reduce the friction coefficient. However, for the first category, the low content and unevenly distributed micro-nano particles that doped into the PEO ceramic layer leads to limited anti-friction effect. For the second category, the processing method is relatively complicated, although the friction reducing effect is better. Therefore, a method that directly burnsishes the micro-nano solid lubricant on the surface of the PEO ceramic coating by some post-treated techniques to form a functional composite coating.

2. Experimental details

2.1. Preparation of PEO coating

The chemical composition of aluminum alloy (ZL109, 40 mm × 10 mm × 10 mm) is shown in table 1. Prior to PEO processing, the surfaces of the specimens were sequentially grounded by 600 #, 800 #, 1000 #, 2000 # abrasive papers, and then polished to obtain a lower roughness surface (Ra 0.12). Lastly, they were ultrasonically cleaned with acetone and ethanol for 10 min separately before drying in warm air.

An alkaline aqueous electrolyte was obtained from a solution of Na2SiO3 (8 g l−1), Na2WO4·2H2O (5 g l−1), KOH (2 g l−1) and EDTA-2Na (2.5 g l−1). The power supply chose constant voltage mode [35], and the electrolyte temperature was controlled at 30 °C ~ 40 °C during the PEO process. Other primary electrical parameters, for instance, voltage, frequency, duty cycle, pulse ratio and oxidizing time were +420 V, −120 V, 500 Hz, 20%, 1:1 and 30 min, respectively. All these electrical parameters as well as the composition and concentration of the electrolyte were optimized by previous works [36, 37].

2.2. Preparation of composite coating

The preparation process was shown in table 2. The PEO specimen was ultrasonically cleaned with ethanol for 15 min and used as the control sample A # after drying. The PEO specimen was sequentially grounded again by

| Table 1. Composition of ZL109 cast aluminum alloy. |
|-----------------|-----------------|-------------|-------------|-------------|-----------------|
| **Composition** | **Si** | **Cu** | **Mg** | **Nbi** | **Al** |
| **Content(wt%)** | 11.0 ~ 13.0 | 0.5 ~ 1.5 | 0.8 ~ 1.3 | 0.8b ~ 1.5 | the rest |


Based on this process, the surface of the sample B was burnished with WS$_2$ nanoparticles which was sprayed on the polishing cloth with a certain pressure and certain speed for 10 min to obtain the sample C. In addition, the sample D was prepared by directly burnishing WS$_2$ nanoparticles on the surface of the PEO sample. The WS$_2$ nanoparticles (Diameter: 50 nm, purity: 99.9 wt%) were detected by a particle size analyzer, and the particle size was 90% distributed in the range of 50 nm. The results as shown in figure 1.

### Table 2. Preparation process of composite coatings.

| Samples | PEO | Grounding | Burnishing |
|---------|-----|-----------|------------|
| A#      | 30 min | —         | —          |
| B#      | 30 min | 5 min     | —          |
| C#      | 30 min | 5 min     | 10 min     |
| D#      | 30 min | —         | 10 min     |

2.3. Examination of the specimen

The ball-on-plate friction and wear tester (Lanzhou Huahui Instrument Technology Co., Ltd) was adopted to evaluate the tribological behavior of the specimens. The test sample was used as the lower part, and the grinding pair was a stainless steel ball (AISI 304, Diameter 4 mm, HRC38). The test was carried out under dry sliding conditions with a normal load of 10 N at the speed of 400 rpm and an oscillating amplitude of 5 mm for 20 min at room temperature (25 °C) and humidity (60%). The schematic diagram of the tester is as shown in figure 2.

After the test, the diameter of wear spot and morphology of the coupling steel ball were analysed by a scanning electron microscopy (TESCAN VEGA3). The wear rate was utilized to evaluate the wear performance and calculated from the following equation:

$$R = \frac{V}{N \times L}$$  (1)

Where $R$ is the wear rate, $V$ is the wear volume ($\text{mm}^3$), $N$ is the normal load (N), and $L$ is the sliding distance (m). In addition, because the hardness of the ceramic film was much higher than that of coupling steel ball, the wear rate was mainly characterized by the wear volume of the coupling steel ball which was calculated by the diameter of the wear spot.

Roughness tester (TR200) was used to examine the surface roughness of the coating. The morphologies of wear scar, surface and cross-section and the element composition in specific area of the sample were observed and analyzed by a scanning electron microscopy (TESCAN VEGA3) equipped with an energy dispersive spectrometer (OXFORD).
3. Results

3.1. Microstructure characterization

Figures 3(a)–(d) show the SEM morphologies of the prepared specimens. As well, the results of the EDS analysis at selected areas of samples C# and D# are displayed in figures 3(e) and (f), respectively. The typical morphology of PEO coating, such as irregular volcanic-like micro-pores and pancake structures, are observed in figure 3(a). The diameter of the pores is about 1–5 μm, and the surface roughness is about Ra 3.5. After grounded with abrasive paper, the surface of sample B# becomes relatively smooth, and the surface roughness is greatly reduced to Ra 0.75. However, there are still some small amounts of micro-dimple defects, as shown in area marked in yellow of figure 3(b), which is considered to be the bottom of volcanic-like micro-pores after grounding. For sample C#, the micro-dimple defects on sample B# almost disappear after being burnished by WS2 nanoparticles. Furthermore, the surface roughness is dramatically reduced to Ra 0.44 which is about 41.3% lower than sample B#. The result of the EDS analysis at the yellow square of figure 3(c) indicates that most of the surface is covered by WS2 solid particles except the black smooth regions (PEO ceramic substrate). The content of W and S elements are 16.8 wt% and 11.2 wt%, respectively. It is considered that WS2 nanoparticles encapsulate and fill the bottom pit of volcanic-like micro-pores during burnishing. The surface morphology of sample D# is shown in figure 3(d). The porous phenomenon and PEO ceramic substrate both almost disappear when the WS2 nanoparticles are directly burnished. Besides this, the roughness became Ra 2.3 which is about 34.2% lower than sample A#. The result of the EDS analysis at the yellow square of figure 3(d) also shows that the surface is completely covered by WS2 nanoparticles. Meanwhile, W and S elements content increase to 37.7 wt% and 29.1 wt%, respectively. It is considered that the rough porous surface of PEO ceramic layer creates a better natural environment for introducing solid lubricant. Under this condition, the solid nanoparticles can enter the PEO loose layer more easily during the burnishing process.

Figure 4 represents the backscattered electron (BSE) micrograph of the cross-section of the prepared samples. It can be seen that there is no obvious micro cracks between the PEO ceramic coating and the aluminum matrix. The smooth transition indicates the better quality of the bonding strength. Besides this, all the coatings present the typical structural properties of conventional PEO coatings, for example, the porous outer layer and dense layer, which are analogous to previous studies [21–26]. For sample A#, it can be seen from figure 4(a) that the thickness of the dense layer is about 50 μm, and the thickness of the loose layer is about 20 μm. After grounding, the porous layer structure is polished away, leaving only the dense layer on the matrix which has a thickness of about 30 μm, as shown in figure 4(b). However, a small amount and discontinuous WS2 layer can be found on the surface of the dense layer the area marked in yellow in figure 4(c)) after being burnished by the WS2 nanoparticles. Nevertheless, the WS2 on the dense layer are partially transferred to the resin material during the inlaying process, indicating that the bonding strength between them is not large enough. For sample D#, both the dense layer and the loose layer of the PEO ceramic film still exist. The WS2

![Figure 2. Schematic diagram of friction and wear tester.](image-url)
nanoparticles are burnished into the micro-pores in the loose layer and the quantity is much greater compared with sample C#, as shown in figure 4(d).

3.2. Tribological properties

The evolution of the friction coefficient versus sliding time for the specimens is shown in figure 5. The friction coefficient of the sample A# rises sharply at the beginning, yielding a friction coefficient of 1.2 after 50 s later. But it stabilizes at about 1.5 after 280 s. The rising trend of the friction coefficient of the sample B# is similar to that of the sample A#. The friction coefficient increases to 0.8 after 250 s under the same condition and is stabilized at this value until the end. However, the friction coefficient of the sample C# shows different trend. In the initial stage, the friction coefficient experiences a small decrease and then comes to a stable state. The value of
Figure 4. SEM of different layers’ cross section; (a) Sample A#; (b) Sample B#; (c) Sample C#; (d) Sample D#.

Figure 5. Friction coefficient of different samples.
friction coefficient is extremely low, observed to be about 0.07, which is about 91.2% lower than that of the sample B#. However, in the later stage, followed by a brief fluctuation, the value shows a sharp upward trend and comes to 1.45 at 1060 s. While in steady state, with a slight rise during the test, the friction coefficient of the sample D# rises from 0.3 to 0.45, which is about 75% lower than that of sample A#, and about 81.3% more than the initial stable value of sample C#.

Combined with the surface roughness of prepared samples, it is known that the surface roughness and WS2 solid lubricant which is used when burnishing may be the key factors affecting the friction coefficient in the test. The friction coefficient of the grounded samples B# and C# are lower than those of the ungrounded samples A# and D#, respectively. The burnished samples C# and D# are lower than those of the samples B# and A#, respectively.

The width of wear scar (WWS) of coating surfaces, wear spot diameter (WSD) of coupling steel balls is shown in figure 6. Specific wear rates of different specimens is displayed in figure 7. Under the same test condition, the width of wear scar of sample A# is about 1268.16 μm and the wear spot diameter of coupling steel ball is about 1206.73 μm, which exhibits the highest wear rate of $32.66 \times 10^{-6}$ mm$^3$ N$^{-1}$ m$^{-1}$, as shown in figures 6(a), (b). For sample B#, as shown in figures 6(c), (d), the width of wear scar of sample is relatively narrow, about 1026.62 μm and the wear spot diameter of coupling steel ball is about 1067.75 μm. As well, the wear rate is $19.95 \times 10^{-6}$ mm$^3$ N$^{-1}$ m$^{-1}$, which is reduced by about 38.9% compared with the sample A#. This
demonstrates that the grounding process has a better influence on anti-friction performance. Furthermore, due to the burning of WS$_2$ nanoparticles, the surface roughness of sample C# is very small, inducing in greatly reduced wear degree of sample C# after the test. Compared with the sample B#, the width of wear scar of sample C# is relatively small, about 827.72 μm. The wear spot diameter of coupling steel ball also declines to 824.38 μm with the wear rate being $7.11 \times 10^{-6}$ mm$^3$ N$^{-1}$m$^{-1}$, which is decreased by 63.4% compared with sample B#. For sample D#, it can be seen from figures 6(g), (h) that the width of wear scar on sample surface is the narrowest, about 687.25 μm, and the wear spot diameter of coupling steel ball is about 712.35 μm. Consequently, the wear rate is extremely low, only $4.97 \times 10^{-6}$ mm$^3$ N$^{-1}$m$^{-1}$, which is significantly decreased by 84.8% compared with sample A#. The reason for this result may be that plenty of solid lubricant WS$_2$ on the surface can reduce the friction coefficient. The data of samples C# and D# demonstrate that burning of WS$_2$ nanoparticles has a positive influence on anti-friction performance.

4. Discussion

Figure 8 represents the morphologies of friction region and the composition at the selected region. The surface morphology and composition of the coupling steel balls are shown in figure 9. Some black smooth regions (the areas marked in yellow) can be seen from figure 8(a). The result of EDS analysis at the yellow square of figure 8(a) shows that Fe element content is 41.3 wt%, accompanied by a large amount of O element and a spot of Co element which come from the steel ball. The content of the main elements of the base alloy, Al, Si, etc is very low. It is considered that the steel ball substrate is transferred to the ceramic surface and a transfer film is formed on the black smooth region. The appearance of O element indicates that a high temperature oxidation reaction occurs during the transfer process. Furthermore, the transfer of the steel ball material may have some relationship with the porous layer of the PEO ceramic coating. Under dry sliding condition, the steel ball is in direct contact with the loose layer which has a high roughness. In addition, the irregular volcanic-like micro-pores and pancake structures on surface can produce a ploughing effect on the steel ball during the friction process [38], which can be seen from figure 9(a). Both reasons eventually lead to the sharp increase of the friction coefficient, the widest width of wear scar on the sample and the largest wear spot diameter of the steel ball.

But for sample B#, the porous layer of the PEO coating is removed by a grounding process. The friction surface becomes the dense layer of the PEO coating which has a lower roughness. The morphology of friction region also shows larger smooth areas (black area in figure 8(c)). The smaller roughness and the larger smooth areas may lead to the slow rise of the friction coefficient. The result of EDS analysis at the yellow square of figure 8(c) reveals that the content of Fe element is 25.9 wt%, which indicates a small decline, and the content of Al element is 15.7%, which indicates a small increase, compared with sample A#. The lower Fe content indicates that fewer wear debris of the steel ball are left on the coating surface. Besides this, fewer scratches and smaller amount of iron particles are shown on the wear spot of the steel ball, as shown in figure 9(c). The reason for that may be the decline of coating roughness reduces the damage of the steel ball. However, the direct contact occurring between the ceramic coating and the steel ball still causes a high friction coefficient and a relatively large width of wear scar on the sample and wear spot diameter of the steel ball.

For sample C#, the roughness of the surface is further reduced after being burned by WS$_2$ nanoparticles based on the grounding process. The morphology of friction region shows small smooth areas, as represented in figure 8(e). The result of EDS analysis at the yellow square of figure 8(e) shows that the content of Fe element is 15.2%, which is much lower than those of samples A# and B#. Apart from this, the W and S elements with a content of 3.5 wt% and 2.3 wt% emerge. It means a small amount of WS$_2$ nanoparticles remains at the bottom of the volcanic-like micro-pores (as shown in figure 3(b)) after burning. The lower Fe element and smaller smooth areas indicate that the degree of wear is greatly reduced due to the presence of WS$_2$ solid lubricant which acts as a friction reducer. In addition, the extremely low friction coefficient in the initial stage of the dry sliding process may be due to the further reduced roughness and the solid lubricant. However, the reason for the sharp increase in the friction coefficient after experiencing a fluctuation (as shown in figure 5) may be attributed to the continuous reduction of the remaining WS$_2$ solid lubricant. After a relatively long period of time, the wear of the steel ball occurs when the amount of WS$_2$ is basically consumed, producing some wear debris which comes from the steel ball, as shown in figure 9(e). Some of the steel debris enter the matching surface of the sample surface and steel ball, resulting in an instantaneous increase in the friction coefficient. However, the wear debris is flattened under the action of the shear force and is laid on the ceramic substrate, causing a decrease in the friction coefficient. However, adhesive wear finally happens between the steel ball and the PEO dense layer after a short period of dry friction due to the run out of the solid lubricant at that time, causing a sharp increase in the friction coefficient again. Nevertheless, a large extent in the reduction of the width of wear scar on the sample and wear spot diameter of the steel ball reveals that WS$_2$ plays a better role in anti-friction and wear-resistance properties during the friction process.
The roughness of the surface of sample D also declines after the ceramic coating is directly burnished by WS₂ nanoparticles. The morphology of friction region also shows small smooth areas, as displayed in figure 8(g). As well, the result of EDS analysis at the yellow square of figure 8(h) reveals that the content of Fe, W and S elements are 33.1 wt%, 5.1 wt% and 2.4 wt%, respectively. This indicates that a significant amount of WS₂ nanoparticles remains on the surface after test. The smaller smooth areas also indicate that the degree of wear is reduced significantly due to the presence of WS₂ solid lubricant. It can be considered that WS₂ nanoparticles enter into the volcanic-like micro-pores and pit defects (as shown in figure 3(a)) on the PEO ceramic coating, forming a thin solid lubricating film in the process of direct burnishing. The WS₂ nanoparticles stored in the micro-pores act as a solid lubricant to the friction surface and maintain the existence of the lubricating film. Therefore, no serious adhesive wear happens between the ceramic coating and steel ball in the early stage, and the friction coefficient is stable at a low level for a long time. Nevertheless, the SEM (figure 9(g)) and EDS (figure 9(h)) prove that the solid lubricant on the ceramic coating is transferred to the contact area of steel ball.
during the continuous action of the shear stress. Moreover, the friction reducing effect is more obvious as the solid lubricant content increases.

Figure 10 shows the friction mechanism between the samples and the steel ball. For samples A# and B#, the PEO ceramic coatings are not burnished by the WS2 solid lubricant, and thus the steel ball is in direct contact with the porous layer or the dense layer when the friction test is carried out under dry sliding condition. Therefore, the irregular volcanic rocks on the surface ploughs the steel counterpart, resulting in a rapid rise in the friction coefficient. Under such conditions, a three body abrasive wear occurs between the ceramic and the steel due to the debris generated from steel ball, which further aggravates the wear of the steel ball. This may be the main reason of high friction coefficient and large wear amount during the test. Besides that, part of the debris are squeezed into micro-holes in the PEO coating to form a relatively flat integrated surface under the action of
High shear force, resulting less ploughing effect and stable friction coefficient at the last stage of the test, as shown in figure 5.

However, when the WS2 solid lubricant is burnished on the surface of PEO ceramic coatings, such as samples C# and D#, the WS2 nanoparticles are reserved in the PEO self-generated micro-pores and pit defects. Therefore, when the steel ball is in contact with the hard layer of the ceramic coating, the ceramic coating acts as the hard substrate to improve load bearing capacity and the self-generated micro-pores act as the solid lubricant texture reservoir to release WS2 nanoparticles. The interaction of these two factors can reduce the friction and wear between the samples and steel balls. Only the WS2 lubricant is consumed or extruded out of the contact area after a long period of use, and as the steel ball begins to make direct contact with the ceramic coating, the adhesive friction occurs, as shown in the last two pictures in figure 10. Furthermore, it can be seen from figures 9(f), (h) that there is a mutual transfer of WS2 in addition to the transfer of the steel ball matrix.

5. Conclusion

The WS2 solid lubricant nanoparticles have been burnished on the PEO ceramic coating surface on ZL109. Afterwards, the self-lubricating film was prepared. The tribological performance was investigated by using the ball-disk friction and wear tester under dry sliding condition. The following conclusions are obtained:

1. Burnishing WS2 solid lubricant nanoparticles on the PEO ceramic coating surface can effectively reduce the roughness and the friction coefficient. Meanwhile, it can also significantly lower the WSD of the steel ball. The composite coating of C# has a lowest friction coefficient of 0.07.

2. The burnishing WS2 solid lubricant process shows better tribological property during the whole test, while the grounding process before burnishing solid lubricant only has a better influence on tribological properties in the early stage of the test. The coupling ball of composite coating of D# has a lowest wear rate of $4.97 \times 10^{-6}$ mm$^2$ N$^{-1}$ m$^{-1}$.

3. A friction mechanism concerned with PEO ceramic coating structure and solid lubricant is proposed. This involves the ceramic coating acting as the hard substrate to improve load bearing capacity and the self-generated micro-pores act as the solid lubricant reservoir to increase the volume content of solid lubricant and bonding strength, which can slowly release WS2 nanoparticles. The interaction of these two factors can effectively reduce the friction and wear properties of the samples and steel balls.

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