Self-lubricating nanocomposite coatings using MAO to improve tribological properties of 6061 aluminum alloy

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Abstract
This study aimed to prepare self-lubricating micro-arc oxidation (MAO)/graphite composite coatings on 6061 aluminium alloy by adding graphite nanoparticles into an alkaline electrolyte during the MAO process. The microstructure, phase composition and element distribution of MAO coatings were studied using Scanning Electron Microscope (SEM), x-ray diffraction (XRD) and Energy Dispersive Spectrometer (EDS). The results showed that graphite nanoparticles can be coated uniformly in the MAO coatings, and the porosity and surface roughness of the MAO coatings were reduced by the addition of graphite nanoparticles. However, when the content of graphite nanoparticles was more than 6 g l⁻¹, graphite aggregation occurred. The wear resistance of the coatings was investigated using the ball-on-disk friction tester. After incorporating the graphite nanoparticles, the MAO coatings showed excellent self-lubricating properties and a reduction in the friction coefficient during the wear process. In addition, the MAO coatings showed the best result with the addition of 4 g l⁻¹ graphite nanoparticles compared with all the other composite coatings under investigation. In conclusion, the incorporation of graphite nanoparticles improved the wear properties of the MAO coatings.

1. Introduction

Aluminum and its alloys are widely used in the manufacturing, aerospace and automotive industries due to their low density, high strength-to-weight ratio, and excellent high atmospheric corrosion resistance [1–3]. However, their shortcomings in mechanical and tribological properties are obvious, which seriously restrict their applications [4, 5]. Surface treatments such as hard anodic oxidation, micro-arc oxidation (MAO), electrodeposition, laser processing, and so forth, are effective ways to improve the surface hardness and tribological behavior of aluminum and its alloys obviously [6–8]. Among various surface processing techniques, MAO showed high productivity, ecological friendliness and economic efficiency [9, 10]. In addition, the coatings exhibited excellent bonding onto the substrate and higher hardness simultaneously [11].

Recently, several studies have indicated that self-lubricating nanocomposite coatings with lubricant nanoparticles embedded in hard coatings can provide a superior self-lubrication effect. Nie et al [12] studied a duplex coating with an oxide underlayer and a lubricant graphite composite top coating to reduce the friction coefficient. The duplex coating exhibited a low friction coefficient and excellent tribological performance during dry tribological tests. However, the duplex coating was inconvenient due to the two-step coating process. Wu et al [13] proved that the graphite/oxidation composite coating could be obtained through one-step MAO. Graphite nanoparticles were uniformly added to the electrolyte during the MAO process. However, research on the tribological behavior of the graphite/oxidation composite coatings is lacking.

The present study, attempted to prepare graphite/oxidation nanocomposite coatings through one-step MAO on 6061 aluminium alloy to improve the surface hardness and tribological behavior. The effects of the concentration of graphite on the growth behavior, phase composition, microstructure, and tribological
properties of coatings were studied, and the lubrication mechanism was discussed. These nanocomposite coatings were able to adjust their surface composition and morphology automatically and reversibly through multiple mechanisms, which was promising for the reduction of friction and wear over broad ranges of ambient conditions [9, 14, 15]. As a pilot study, these findings might provide preliminary guidance and theoretical support for preparing self-lubricant composite coating on aluminum and its alloys.

2. Experimental details

2.1. Materials and coating procedure

The circular samples with dimensions of (Φ35 × 3 mm) were cut out from 6061 aluminum alloy. The chemical composition of the 6061 aluminum alloy samples used in the present study was as follows (in wt. %): (0.8–1.2 Mg, 0.4–0.8 Si, 0.7 Fe, 0.15–0.4 Cu, 0.25 Zn, 0.04–0.35 Cr, 0.15 Ti, 0.15 Mn, and balance Al). Prior to the MAO treatment, all specimens were ground using emery papers of up to 2000 grit, the surface roughness, Ra, was 0.2 μm as provided. The specimens were cleaned with distilled water and air-dried. A pulsed electrical source of 10 kW power was used for MAO experiments. The specimens and a stainless steel container with a water cooling system were used as the anode and the cathode, respectively. The temperature of the electrolyte was kept below 25°C. The electrolyte solution consisted of 30 g l⁻¹ (NaPO₃)₆, 5 g l⁻¹ Na₂SiO₃, and (0, 2, 4, 6 and 8 g l⁻¹) graphite nanoparticles with an average size of 50 nm. The MAO coatings fabricated with adding different contents of graphite were named as C0, C2, C4, C6 and C8. The MAO treatments were carried out for 20 min with a current density of 6 A dm⁻², frequency of 500 Hz and duty cycle of 5%. The ultrasonic-assisted process was used in the whole MAO process to improve the dispersion effect of graphite. After the MAO treatment, the sample was washed with alcohol in ultrasonic waves for 10 min and then dried in warm air.

2.2. Characterization of MAO coatings

X-ray diffraction (XRD-7000 s, Cu Kα radiation, accelerating voltage: 40 kV, tube current: 40 mA) analysis was used to determine the phase composition of the MAO coatings. The scans were performed at a grazing angle of 2°, and the scan speed was 10° min⁻¹. The microstructural and elemental compositions of the MAO coatings were investigated using a high resolution scanning electron microscope (SEM, JEM-6510, Japan Electronics Co. LTD) with an energy dispersive spectrometer (EDS) system. The thickness and roughness of the MAO coatings were tested using an eddy current thickness meter (Helmut Fischer GMBH, Germany) and a surface roughness tester (TR-210, China), respectively. The hardness of the coatings was measured with a depth-sensing micro-indentation tester (CSM Instruments Micro-Combi Tester) using a standard Vickers indenter. According to the Oliver–Pharr method [16] indentation hardness from the cross sections was calculated; 100 mN was the maximum load, and the loading and unloading rates of the indenter were both 200 mN min⁻¹ during indentation. Ten indentations were measured for each coating to minimize the standard deviation. The coating porosity was tested according to surface SEM pictures with the aid of Image J 6.0 software.

2.3. Evaluation of tribological behavior

According to the ASTM-G99 standard, the tribological performance of the MAO coatings was evaluated by the ball-on-disk testing method in a wear tester (XLG1200, China) under dry sliding conditions. The GCr15 balls with a diameter of 5 mm were adopted as couple materials, and all samples were tested at 0.42 m s⁻¹ linear speed.

Figure 1. Voltage and time variations during MAO processing at a constant current density of 6 A dm⁻² with different concentrations of graphite particles.
and a rotating diameter of 20 mm for 8000 laps with a load of 4 N. All the tests were completed at room temperature, and the relative humidity was approximately 64%–72%. The friction coefficient was recorded with a computer during the wear tests. The width and depth of wear tracks were measured at five different positions, and the average was obtained to estimate the wear volume. The wear rate is the ratio of the volume of the worn coating to sliding distances and the load. After the sliding tests, the morphology characterization of wear tracks was conducted by FESEM analysis.

3. Results and discussion

3.1. Process voltage-time diagrams

Figure 1 represents voltage and time variations during the MAO processing at a constant current density of 6 A dm$^{-2}$ with different concentrations of graphite nanoparticles. It conforms to the typical trend followed by the variation in MAO voltage. In the initial stage, the current-voltage variation was subjected to Ohm’s law to some extent with and without the addition of graphite nanoparticles. When the system was energized, the oxyacid anions under the impact of voltage formed a condensation layer of aluminized hydrate at the interface of the sample/solution [17]. The condensation layer of the aluminized hydrate was deposited on the surface of the sample rapidly with the increase in voltage, resulting in the formation of a barrier layer on the surface of the sample. The sedimentary layer dehydrated under the action of Joule heating. As the voltage broke through the limits of the Faraday region into the micro-arc discharge region, the sedimentary layer was polarized to its dielectric breakdown voltage. Also, MAO coatings were formed in the micro-discharge area. The final oxidation voltage slightly decreased with the addition of graphite nanoparticles. As graphite owns excellent electrical conductivity, the conductivity of the electrolyte after the addition of graphite nanoparticles (0, 2, 4, 6 and 8 g l$^{-1}$) addition was measured, and is listed in Table 1. The addition of graphite nanoparticles increased the conductivity of the electrolyte slightly. Besides, the addition of graphite nanoparticles might also contribute to the electrical conductivity of the graphite/oxidation nanocomposite coatings. This finding was consistent with that of a previous study by Sarkar and Das [18] showing that the conductivity of MWCNTs/alumina composite improved with the increase in the concentration of CNTs.

| Sample | C0 | C2 | C4 | C6 | C8 |
|--------|----|----|----|----|----|
| Conductivity (mS/cm) | 10.19 ± 0.01 | 10.24 ± 0.02 | 10.30 ± 0.02 | 10.36 ± 0.02 | 10.41 ± 0.02 |

Figure 2. Images of MAO coatings with different concentrations of graphite particles: (a) 0 g l$^{-1}$, (b) 2 g l$^{-1}$, (c) 4 g l$^{-1}$, (d) 6 g l$^{-1}$, and (e) 8 g l$^{-1}$.
3.2. Discharge characteristic of the micro-arc

The images of MAO coatings with different concentrations of graphite nanoparticles are shown in figure 2. The color of the MAO coatings became darker with the increase of graphite nanoparticles. Nevertheless, when the concentration in graphite was up to 6 g l$^{-1}$, uneven island was observed at the edge of the sample as shown in figure 2(d), when the concentration of graphite was up to 8 g l$^{-1}$, the number of islands increased as shown in figure 2(e).

In this study, a rectangular stainless-steel plate was used as the cathode with a larger area than the anode. The phenomenon of micro-arc discharge was observed at different times, as shown in figures 3(a) and (b). This phenomenon was more evident at the edge of the sample, perhaps because of the distribution state of the electric field. In addition, to test this conjecture, a needle-shaped cathode structure was designed specifically. In addition, to test this conjecture, a needle-shaped cathode structure was designed specifically, the distance between the poles was 20 mm, as shown in figure 3(c). The micro-arc discharge from the central area corresponding to the needle-shaped cathode had a higher density and energy. Figures 3(d) and (e) reveals the distribution of electric field under cathodes of different shapes. The electric field strength at the edge of the sample was higher than that in the central area. When the cathode had a larger area compared with that of the anode, a large number of OH$^-$ ions moved to the marginal region rapidly, which contributed to the formation of an electron avalanche, thereby promoting the violence of the reaction of MAO to some extent.

There are two different discharge modes in the MAO processes. A kind of type of discharges derives from the breakdown in one strong electric field. While another kind of type discharges is caused by the gas discharges. When the critical breakdown voltage of the oxide ceramic coating exceeded by the applied voltage, the surface passivation film of the substrate would be punctured, and a large number of micro arc discharge zones appeared on the surface of the substrate. As the discharge reaction further, the micro-arc discharge channel formed. The gas discharges with the complex plasma chemical and electrochemical reaction proceeded in the discharge.
Both the crater-like surface morphology of coatings and the formation of crystalline $\alpha$-$\text{Al}_2\text{O}_3$ with higher melting points confirmed that the temperature in the discharge channels was very high.

### 3.3. Phase composition of the coatings

Figure 4 shows the XRD patterns of the MAO coatings with different concentrations of graphite particles. The diffraction peaks of aluminum were detected in each sample, because the x-rays passed through the porous MAO coatings to the substrate and the crystalline phases of substrate were detected. As expected, the crystalline phase of the graphite was detected in the graphite-added MAO coatings, indicating that graphite nanoparticles were successfully incorporated into the MAO coatings. According to the literature [19], when the temperature is below 700 °C, amorphous alumina is the predecessor of $\gamma$-$\text{Al}_2\text{O}_3$, $\gamma$-$\text{Al}_2\text{O}_3$ transforms into $\alpha$-$\text{Al}_2\text{O}_3$ at 1200 °C–1300 °C irreversibly, and Gibbs free energy goes down in the process. The instantaneous temperature of plasma discharge was as high as $10^4$–$10^5$ K in the MAO process [20]. Therefore, the formation of $\gamma$-$\text{Al}_2\text{O}_3$ and $\alpha$-$\text{Al}_2\text{O}_3$ in the MAO process was predictable. The intensity of $\gamma$-$\text{Al}_2\text{O}_3$ and $\alpha$-$\text{Al}_2\text{O}_3$ peaks decreased gradually with the addition of graphite. Particles having a high melting point can be considered as sintering additive to inhibit the formation of coatings because melted particles can either raise sintering temperature or reduce the speed of sintering [21].

### 3.4. Microstructure

Figure 5 reveals differences in the surface morphology of coatings with different concentrations of graphite nanoparticles. The size became smaller in terms of micropores of the MAO coatings with the increase in graphite nanoparticles. This achievement could be attributed to the increase in graphite nanoparticles which promoted the uniformity of electrode distribution on the surface of coatings. On the contrary, as the voltage decreased gradually, the discharge energy of micro-arc monopulse decreased, the extent of reaction of MAO reduced, and the diameter of micropores on the coatings decreased. Moreover, the graphite nanoparticles followed the electrolyte into the discharge channel and the molten oxide was ejected from the micropores which were blocked by the graphite nanoparticles during the MAO process. The nucleation rate of molten oxide inside the micropore was promoted, and many closed and semi-closed micropores were formed on the surface of the ceramic coatings. The diameter of the micropores on the surface of coatings was reduced to a certain extent.

For the information on the distribution of elements through the surface (especially C), EDS maps of the coated surface were obtained (figure 6). The surface EDS maps detected graphite in the coatings with the addition of graphite nanoparticles in the electrolyte as shown in figure 6(c). The coating without graphite nanoparticles (figure 6(a)) showed a distribution of element C on the surface. It may be that the carbon dioxide has not been completely vacuumed during the detection process. However, the specific content of graphite was not detected in figure 6(b), probably because the content was too less. Proper addition of graphite nanoparticles did not change significantly the Ra of the MAO coatings. Ra was $1.32 \pm 0.18 \ \mu\text{m}$, $1.24 \pm 0.10 \ \mu\text{m}$ and $1.19 \pm 0.09 \ \mu\text{m}$ for C0, C2 and C4 respectively. When the content of graphite nanoparticles added was up to 6 g l$^{-1}$, the aggregation of graphite occurred and the roughness of the coating(C6) increased obviously to $3.22 \pm 0.82 \ \mu\text{m}$.

Graphite is a kind of excellent lubricant, due to the binding energy between the C atom density layers is low, and planar slip occurred between the layered C atoms. The addition of graphite particles changed the conductivity of the electrolyte because of excellent electrical conductivity of graphite. When graphite nanoparticles entered the discharge channel with the electrolyte, the plasma discharge occurred in the discharge channel. Both the crater-like surface morphology of coatings and the formation of crystalline $\alpha$-$\text{Al}_2\text{O}_3$ with higher melting points confirmed that the temperature in the discharge channels was very high.

**Figure 4.** XRD patterns of MAO coatings with different concentrations of graphite particles.
channel and the plasma high temperature will evaporate the surrounded electrolyte, the graphite particles in the electrolyte are adsorbed in the discharge channel. The graphite nanoparticles were coated in the coatings finally.

The results of cross-sectional SEM micrographs of the MAO coatings with different concentrations of graphite nanoparticles are presented in figures 7(a)–(d). The porosity of the coatings decreased slightly with the addition of graphite nanoparticles, and was consistent with the decrease in the micropore size on the surface. Based on the cross section of the MAO coatings, the growth rate of the coatings reduced with the addition of graphite nanoparticles, to a certain degree, and the thickness of the coatings corresponding to the final voltage.

The cross-sectional elemental mapping of C, Al, and O is indicated in figures 7(e)–(g) to investigate the distribution of element in the coatings. The concentration gradient of C, Al, and O in the coatings was examined.

**Figure 5.** Surface morphologies of MAO coatings with different concentrations of graphite particles: (a and b) 0 g l⁻¹, (c and d) 2 g l⁻¹, (e and f) 4 g l⁻¹ and (g and h) 6 g l⁻¹ and (b and d), (f and h) are partial magnifications of (a and c), (e and g), respectively.
by line scanning. The graphite nanoparticles were found to be homogenously distributed in the coatings, and did not aggregate in the coatings. After graphite nanoparticles entered the discharge channel with the electrolyte, a complex series of chemical/plasma reactions with the substrate occurred. The melted alumina cooled in the electrolyte rapidly and was deposited on the surface of the MAO coatings with graphite nanoparticles. Subsequently, the weakest part of the coating was broken down under a high voltage and the molten oxide was ejected from the micropores, forming a new discharge channel; the process was repeated. The graphite nanoparticles were coated and ejected in the process repeatedly, and hence, homogenously distributed in the coatings.

3.5. Tribological behavior
The variations in the friction coefficient with respect to the sliding laps for the respective coating against GCr15 balls obtained by the ball-on-disk testing method in the wear tester is shown in figure 8. The friction coefficient
increased with the increase in wear time and sliding laps for the coatings without graphite nanoparticles. However, the friction coefficient did not increase significantly for the coatings with graphite nanoparticles because the abrasive particles of coatings participated in the friction process, changing the mode of original sliding friction into the mixed mode of sliding friction and rolling friction. On the contrary, the contact area increased gradually due to abrasion. Nevertheless, the effect of self-lubrication alleviated the disadvantages of abrasion, and the van der Waals forces between graphite layers were so weak that interfacial slippage occurred under the transverse shear forces during the friction process. The aggregation of graphite was caused due to the edge effect, and the roughness and friction coefficient of the MAO coating (C6) increased significantly.

In the beginning, due to surface protrusion of the object, the contact area is small and a large friction coefficient will be obtained. Then the protrusion was ground, the worn out surfaces become smooth with the increase of the wear. And then the wear area increased, the surface roughness of the coatings reduced gradually. As the wear increases further, there is a stable stage, at this stage the area of the contact surface tends to be constant, which causes the friction coefficient to be constant, this stage has the largest proportion in the whole process of wear [22]. Generally speaking, the larger the friction coefficient is, the greater the wear rate is. After graphite nanoparticles incorporation, the surface of the coating C4 is smoother than that of the coating C0 and C2, since the effect of effect of self-lubricating, the friction coefficient of C4 is smaller than that of C0 and C2. Then, the wear rate of C4 is smaller than that of C0 and C2. Therefore, the MAO coatings with graphite nanoparticles incorporation exhibit excellent tribological behavior. From the finite element analysis of the cross section, it can be seen that the coatings with graphite nanoparticles has a lower wear degree. The improvement of the wear resistance is due to the higher hardness of the ceramic coating and the lubricity of incorporation graphite nanoparticles [23, 24].

The micrographs of wear tracks and the corresponding worn balls slid against the GCr15 balls under a load of 4 N as shown in figure 9. Wide and rough wear tracks were seen on the worn surface without the graphite.
nanoparticle coating. Brittle fracture of the coating occurred due to the frictional shear stress between the coating and the steel ball. The hardness and volume wear rates of the MAO coatings are shown in Table 2. The images of the worn surface of the steel ball (the inset in figure 9(c)) showed a relatively flat and smooth surface; the worn surface of the coating was also smooth. The lubrication that graphite provided resulted in a low coefficient of friction for the coatings with graphite addition, the coatings and the steel ball both showed a lower worn rate. The synergistic effect of promoting the hardness and decreasing the friction coefficient was the main reason that caused improvement in the tribological behavior [25]. The coating without graphite nanoparticles addition exhibited deeper groove after the wear tear. Due to the role of protrusions and microholes on the coating surface, obvious scratches occurred on the surface of the couple materials in the wear process, as shown in the figure 9(a). The friction coefficient between the coating and the couple materials decreases after the addition of graphite, both the surface of the couple materials and the wear crack become smooth and flat, as shown in the figures 9(c) and (g).

In order to further explain the self-lubrication mechanism that the coating with graphite nanoparticles addition improves the wear resistance. The formation schematic of the transfer coatings during the wear test are illustrated in figure 10. The coating without graphite nanoparticles addition exhibited deeper groove after the wear tear. Under the effect of transverse shear force, the positions with protrusions were firstly ground, and the positions with microcracks crack, then the partial area shed from coatings. Wear mechanism of the wear resistance was also elaborated according to the [26–28]. However, after graphite nanoparticles incorporation, the microcracks and pores could be filled. The nano-addition could limit the formation of cracks [29]. In the process of the wear, graphite nanoparticles with layered structure destroyed under the wear load, and the separated layered structure was transferred on the surface of coatings and couple materials. That is to say, there is a transfer layer between coatings and couple materials, and the transfer layer reduced the friction coefficient. The tribological properties of the self-lubricating nanocomposite coatings are improved.

4. Conclusions

After incorporating graphite nanoparticles, the electrical resistance of MAO/graphite composite coatings declined, reducing the breakdown voltage of the coatings, which, in turn, slowed down the growth of the coatings. Graphite as inert materials of high melting point, combined with MAO coatings at the instant of the

| Sample | Thickness (μm) | Hardness (HV) | Wear rate (mm³ N⁻¹ m⁻¹) |
|--------|----------------|--------------|-------------------------|
| C0     | 22.3 ± 1.0     | 1128 ± 48    | 2.35E-5                 |
| C2     | 20.1 ± 0.8     | 1136 ± 36    | 1.84E-5                 |
| C4     | 19.2 ± 0.7     | 1235 ± 56    | 7.36E-6                 |
| C6     | 17.2 ± 1.1     | 836 ± 126    | 3.65E-4                 |

Figure 10. The formation schematic of the transfer coatings during the wear test.
discharge in the graphite nanoparticle clogged discharge hole, thus reducing the porosity of the MAO coatings. The graphite nanoparticles were coated and reserved in the coatings in this process because the MAO coatings were breakdowned repeatedly and molten alumina was constantly squirted and then deposited on the surface. Also, the distribution of graphite nanoparticles in the coatings was uniform. The dispersed graphite nanoparticles caused nailing and second phase strengthening in the composite coatings, improving the hardness of the composite coatings. In addition, the graphite nanoparticles detached from the coatings served as a transfer film with low shear strength due to self-lubrication at the interface during the sliding. The friction coefficient of self-lubricating nanocomposite coatings reduced by 50%, and the volume wear rate reduced by 68%.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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