

Effect of MoS₂ Concentration on Microstructure and Tribological Behavior of Electrophoretic-Electrodeposited Ni-Co-Al₂O₃-MoS₂ Composites

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Ni-Co-Al₂O₃-MoS₂ composite coatings were prepared on the surface of LY12 aluminum alloys by electrophoresis-electrodeposition with different MoS₂ concentrations. The microstructure, morphologies and composition of Ni-Co-Al₂O₃-MoS₂ composites were characterized by X-ray diffractometer (XRD) and scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS). The micro-indentation hardness as well as friction and tribological properties of the coatings were tested by micro-hardness tester and friction and wear tester separately. Results revealed that the composite coating fabricated at 1.0 g·L⁻¹ MoS₂ achieved dense structure, and the average thickness of the coating was 39.820 μm. The micro-indentation hardness of the composite coating was decreased from 578 HV to 465 HV with the increase of MoS₂ concentration. Also, the composite coating synthesized at 1.0 g·L⁻¹ MoS₂ had the lowest friction coefficient and wear rate.

Keywords: Ni-Co matrix, Molybdenum sulfide, Alumina, Microstructure, Anti-friction characteristics.

1. Introduction

Aluminum alloy, with numerous advantages such as light weight and excellent mechanical strength, has been widely used in aerospace, automobile, machinery manufacturing, shipbuilding and chemical industry. However, the poor hardness and wear resistance of aluminum alloy are the bottleneck of its wide application, so improving its mechanical properties has always been a research hotspot¹⁻⁵.

Electrophoresis-electrodeposition technology^{6,7}, which can embed various particles such as Al₂O₃⁸, MoS₂⁹, TiC¹⁰, TiO₂¹¹, ZnO¹², ZrO₂¹³ and SiC¹⁴ into metal or alloy matrix to form cermet composite coating with excellent mechanical and electrochemical performance, has drawn great attention. Among the numerous embeddable particles, MoS₂ with layered structure is an ideal solid lubricant for the preparation of coatings with excellent friction and wear resistance¹⁵. The introduction of solid lubricant can obviously improve the wear resistance of the material, thus prolonging the service life of the material¹⁶⁻¹⁸. Furthermore, Ni-Co matrix has aroused growing interest due to its exceptional comprehensive properties¹⁹⁻²². Ma et al.²³ presented the influence of the duty cycle and frequency on the microstructures and properties of SiC reinforced Ni-Co matrix nanocoating, and it is proposed that the composite coating has high hardness and excellent wear resistance under the optimized parameters. Li and Zhang²⁴ prepared ZrO₂ doped Ni-Co matrix composite coating by one-step DC electrodeposition process. The results show that the Ni-Co-ZrO₂ composite coating has excellent mechanical properties and corrosion resistance.

Based on our previous research and a large number of literatures^{25,26}, it is shown that the mechanical properties

of the composite coating can be effectively improved by adding Al₂O₃ to Ni-Co alloy or adding two kinds of ceramic particles (Al₂O₃ and MoS₂) to Ni matrix. However, there are few studies on embedment Al₂O₃ and MoS₂ particles into Ni-Co alloy by a low energy consumption and high efficiency liquid deposition technology and expect to obtain new or excellent anti-friction and wear resistance. Therefore, we try to prepare Al₂O₃ and MoS₂ particle reinforced Ni-Co matrix composites with the required properties by the above methods, and evaluate its microstructure, friction and wear behavior.

In this work, we aimed to prepare Ni-Co-Al₂O₃-MoS₂ composites on the surface of LY12 aluminum alloys by electrophoresis-electrodeposition method. The effects of MoS₂ concentration in EPD bath on microstructure, micro-hardness and tribological properties (involving friction coefficient, wear rate and worn scars of the coatings) were comparatively investigated.

2. Experimental Details

2.1 Pretreatments

The substrates, LY12 aluminum alloy, were cut into size of 15 mm×15 mm×10 mm samples. The chemical compositions of the substrate are listed in Table 1. The average particle sizes of alumina (α-Al₂O₃, 99.99% in purity, ST-NANO, China) and molybdenum sulfide (99.99% in purity, ST-NANO, China) are 150 nm and 200 nm, respectively. The substrates were polished with SiC paper, and then washed in alkaline solution composed of 50 g·L⁻¹ sodium hydroxide, 10 g·L⁻¹ triphosphate and 30 g·L⁻¹ sodium silicate to remove surface oil contamination. The samples were immersed

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Table 1. Chemical composition of LY12 aluminium alloys.

| Composition (wt%) | Si | Fe | Cu | Mn | Mg | Cr | Ni | Zn | Ti | Al |
|-------------------|-----|-----|---------|-------|---------|------|------|------|------|------|
| | 0.5 | 0.5 | 3.8-4.9 | 0-0.9 | 1.2-1.8 | 0.10 | 0.50 | 0.25 | 0.15 | rest |

in 25% sulfuric acid medium to eliminate the oxide film. After each step, the substrates were rinsed with distilled water to avoid contamination of the pretreatment solution.

2.2 Electrophoresis-electrodeposition progress

To electrophoretic deposition Al_2O_3 and MoS_2 , ethanol was acted as the dispersant according to our initial exploration. The electrophoresis was implemented in an EPD bath containing $10 \text{ g}\cdot\text{L}^{-1}$ alumina, $0\text{-}2.0 \text{ g}\cdot\text{L}^{-1}$ molybdenum disulfide, $0.5 \text{ g}\cdot\text{L}^{-1}$ hydrated magnesium chloride. The voltage, temperature and time of electrophoresis were kept at 80 V, 65°C and 5 min, respectively. The chemicals used in the experiment are analytical grade. After electrophoresis, Ni-Co alloy was deposited on the sample surface by pulse electrodeposition. The pulse electroplating solution consists of $300 \text{ g}\cdot\text{L}^{-1}$ nickel sulfate, $60 \text{ g}\cdot\text{L}^{-1}$ nickel chloride and $30 \text{ g}\cdot\text{L}^{-1}$ cobalt sulfate. And the voltage, PH, stirring speed, temperature and electrodeposition time were kept at 1.72 V, 3-6, 350 rpm, 50°C and 50 min, respectively. The schematic of pulse-electrodeposition process is shown in Figure 1.

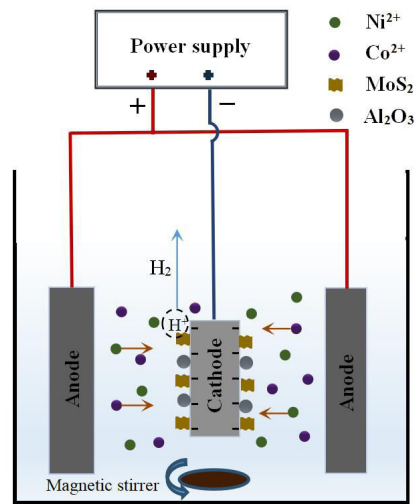
2.3 Characterizations

The surface and cross-section morphologies of as-fabricated Ni-Co- Al_2O_3 - MoS_2 composites were viewed by SEM (JSM-6390A, Japan). The crystalline structure of the coating was estimated by XRD (XRD-7000, Japan) with Cu K α filtered radiation ($\lambda=1.54 \text{ \AA}$) generated at 40 kV and 30 mA, and the affiliated EDS was used to determine the composition of the composite coating. The micro-indentation hardness of the coating surface was measured by micro Vickers hardness tester (EM-1500L, China) for 5 s under a load of 10 N. The distance between test points is 40 μm , and the final micro-indentation hardness is the average of five measurements. The wear resistance of the composite coating was investigated under dry sliding conditions in air at room temperature by the ball-on-disk method, using a high-temperature friction and wear tester (HT-1000, China). The test was performed with a disc speed of 560 rpm and a track radius of 5 mm, using a bearing GCr15 ball (6 mm in diameter) under a load of 5 N for 10 minutes. The test is compatible with ASTM G99. Each sample was tested three times under the same conditions, and the average value was selected as the final value. The worn surface of the sample was wiped with alcohol and the wear morphologies of the coating were observed by SEM. Adhesion tester characterizing bonding strength was carried out with a load of 0.1 N and a speed of 5 mm/min.

3. Results and Discussion

3.1 Morphologies and composition

Figure 2 exhibits the cross-section and surface morphologies of Ni-Co- Al_2O_3 - MoS_2 composites synthesized at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 . From Figure 2a, the dark gray area on

**Figure 1.** Schematic of the pulse-electrodeposition setup.

the left is the aluminum alloy substrate, the thin gray layer in the middle area is flash-plated Ni-Co layer to prevent the substrate from corrosion in EPD bath, and the gray region on the right is Ni-Co- Al_2O_3 - MoS_2 composite coating. The surface morphology of the composite coating is shown in Figure 2b, and it can be seen that the coating surface presents a mushroom-like structure. This phenomenon can be attributed to the preferential reduction of Ni^{2+} and Co^{2+} on MoS_2 particles with good conductivity or the convex regions of as-deposited metal matrix²⁷. The magnified image of cross-section morphology and the EDS spectrum of as-fabricated Ni-Co- Al_2O_3 - MoS_2 composites are exhibited in Figure 2c and d, respectively. It is proved that the Ni-Co- Al_2O_3 - MoS_2 composite coating was well synthesized on the aluminum alloy substrate, there are no obvious cracks and delamination at the interface, and the deposited particles are homogeneously dispersed in the Ni-Co matrix without obvious agglomeration.

The energy spectra of Ni-Co- Al_2O_3 - MoS_2 composites are shown in Figure 3. Obviously, there are two kinds of different particles (Al_2O_3 and MoS_2) in the composite coating. Figure 3b, c and d show the energy spectrum analysis of different regions. Results show that the light gray continuous region is Ni-Co matrix. The uniformly distributed black particles are Al_2O_3 particles and the light gray stripe particles are MoS_2 particles.

Figure 4 represents the XRD patterns of the composite coatings. The diffraction peaks of the composite coating exhibit polycrystalline orientation. It can be seen from Figure 4a that the composite coating deposited at $0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 contains Ni, Co and Al_2O_3 , and the diffraction peaks of each phase are obvious. As seen in Figure 4b, the characteristic peaks of MoS_2 are also found at $2\theta=14.533^\circ$, corresponding

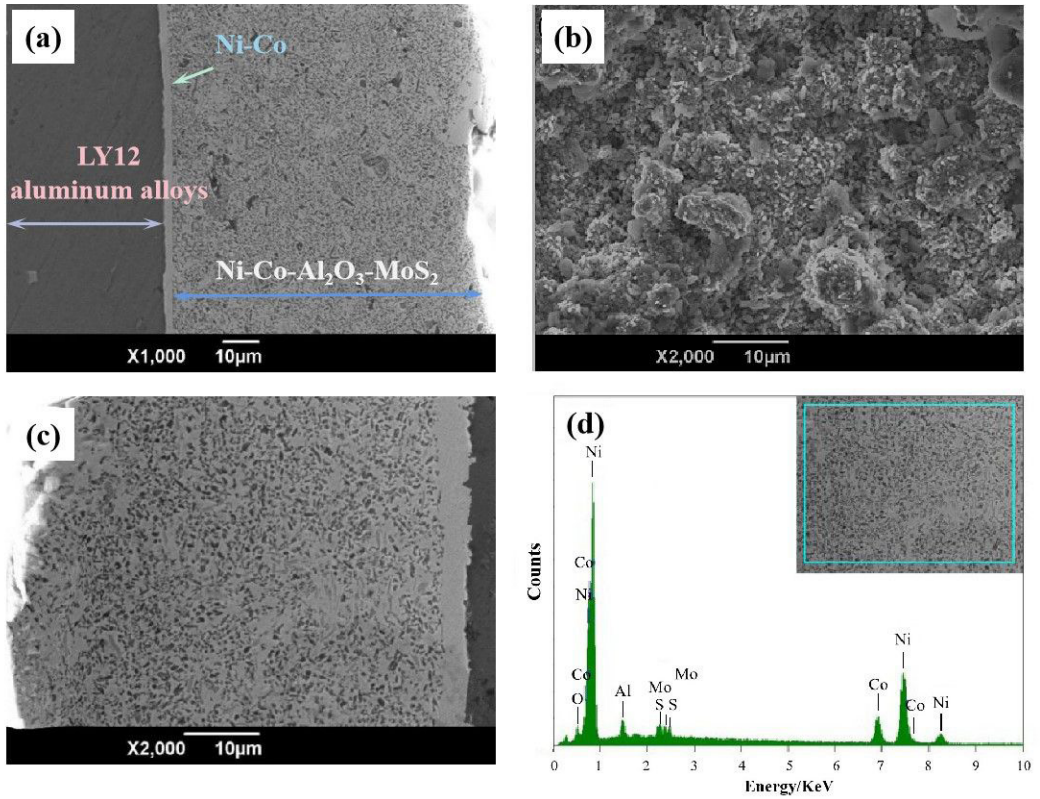


Figure 2. Composition and morphologies of as-prepared Ni-Co matrix composites.

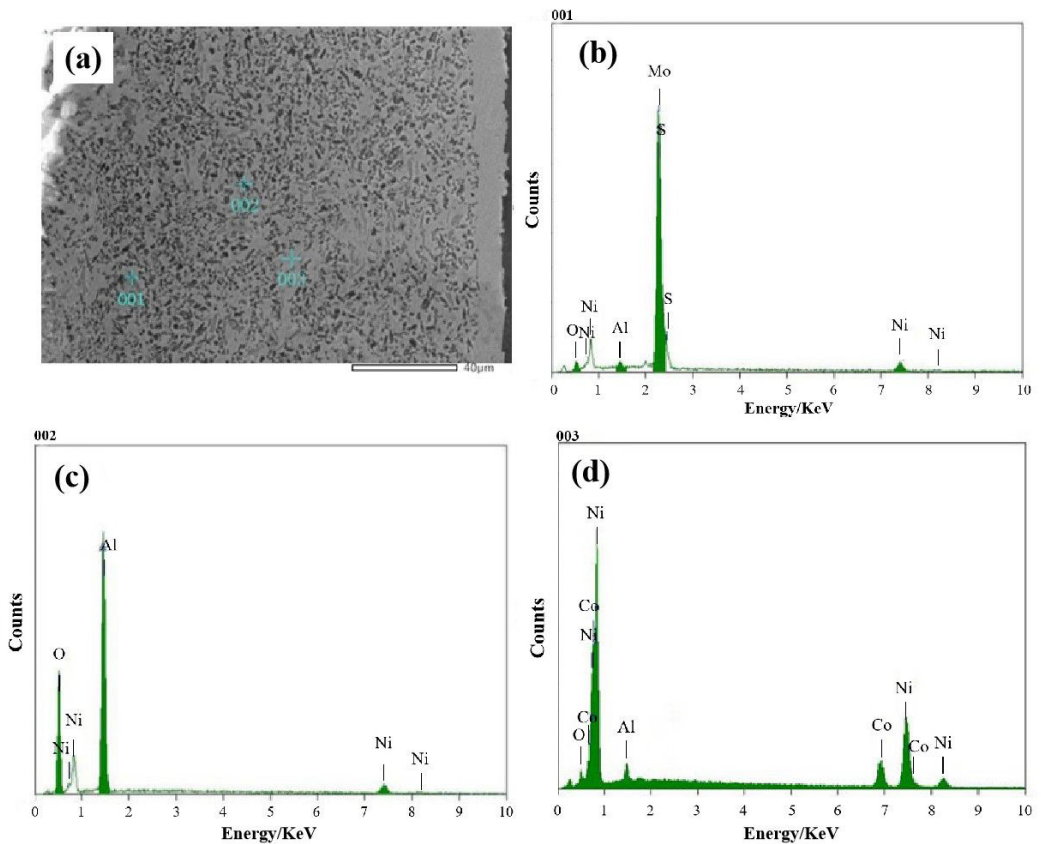


Figure 3. Energy spectrum of the as-deposited composites.

(002) plane. Therefore, the composite coating synthesized at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 contains Ni, Co, Al_2O_3 and MoS_2 .

Figure 5 shows the cross-section morphologies of the composite coatings synthesized at various MoS_2 concentrations. It can be seen from Figure 5 that the composite coating is well bonded with the substrate, and there is no crack and void at the interface. The average thickness of the Ni-Co intermediate layer is about $5.571 \mu\text{m}$. By introducing $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 particles, the composite coating is dense and uniform, and the average

thickness of the coating reached a minimum of $39.820 \mu\text{m}$ (Figure 5b). This may be attributed to the stable suspension of MoS_2 and Al_2O_3 particles in the bath, and then the suspended particles are uniformly deposited on the cathode surface, resulting in the uniform and dense microstructure of the coating. If the MoS_2 concentration is not optimal ($1.0 \text{ g}\cdot\text{L}^{-1}$), numerous pores appear in the composite coating. As seen in the Figure 5d, there are particularly large pores in the composite coating. Because MoS_2 particles are hydrophobic

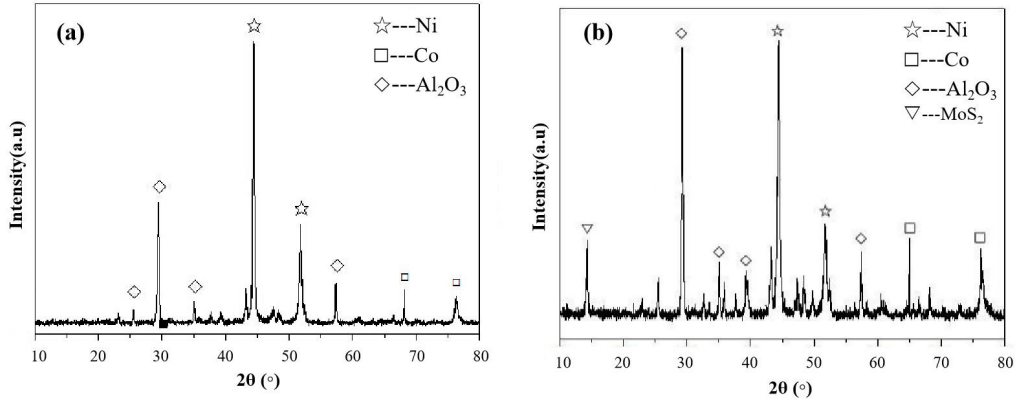


Figure 4. XRD patterns of the electrophoretic-electrodeposited composites: (a) without adding MoS_2 , (b) adding $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 .

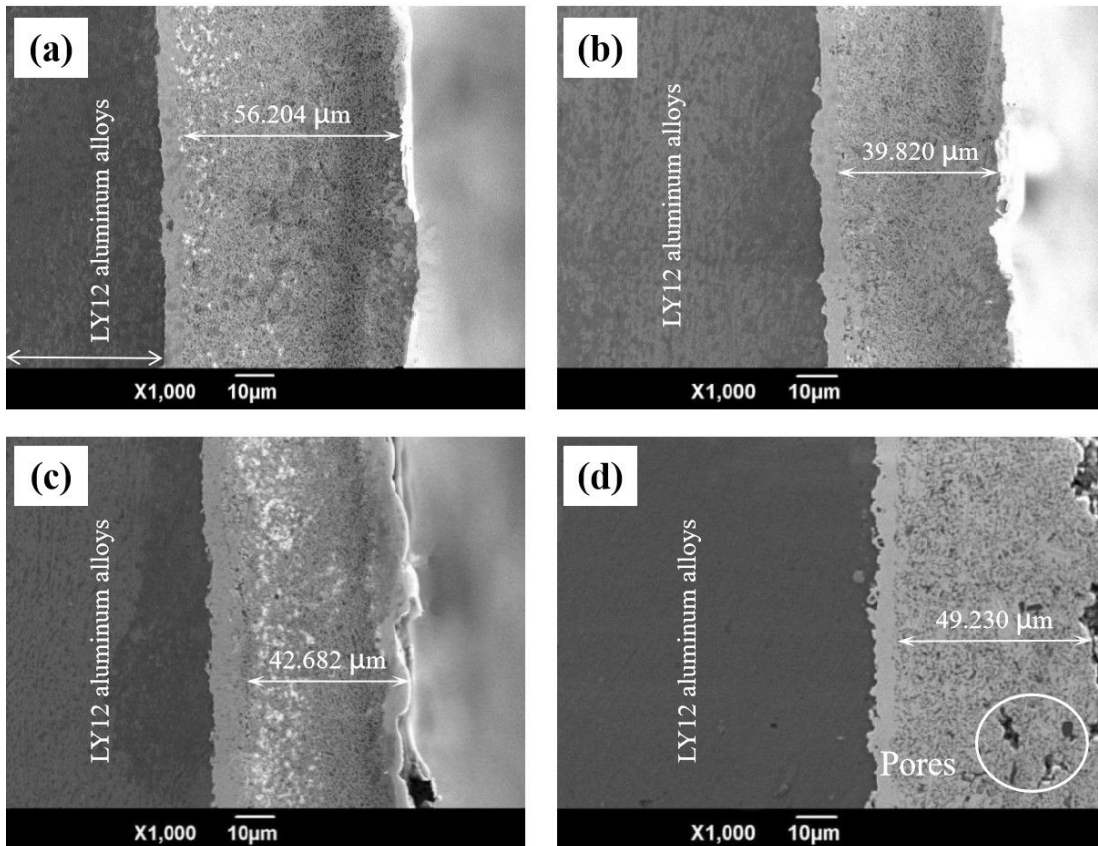


Figure 5. Cross-section morphologies of the coating with various MoS_2 concentrations: (a) $0.5 \text{ g}\cdot\text{L}^{-1}$; (b) $1.0 \text{ g}\cdot\text{L}^{-1}$; (c) $1.5 \text{ g}\cdot\text{L}^{-1}$; (d) $2.0 \text{ g}\cdot\text{L}^{-1}$.

and with a larger wetting angle in water, the hydrophobicity of MoS₂ particles directly affects the deposition of Ni-Co metal matrix. Ni-Co matrix could not completely fill the pores between the particles, which lead to a large number of pores in the coating.

Figure 6 presents the surface morphologies of as-synthesized Ni-Co-Al₂O₃-MoS₂ composites. As can be seen, with the increase of MoS₂ concentration to 1.0 g·L⁻¹, the composite coating with uniform, flat and fine surface morphology is observed (Figure 6b). The conductive MoS₂ particles adsorbed on the cathode surface increase the reaction area of nickel ions and cobalt ions and provide more nucleation sites during the pulse electrodeposition process, thus the coating surface presents a relatively smooth and dense morphology²⁸. As shown in Figure 6c and Figure 6d, with the increase of MoS₂ concentration to 1.5 g·L⁻¹ and 2.0 g·L⁻¹ respectively, rough mushroom-like microstructure is formed on the coating surface. This is mainly due to the agglomeration of MoS₂ in the electrophoresis bath, and then the deposition of these aggregates on the cathode surface. During the subsequent

pulse electrodeposition process, the applied current is more concentrated on the conductive particles, which leads to the priority reduction of Ni²⁺ and Co²⁺ on the raised aggregates. At the same time, because of the electric field repulsion between the protrusions, the reduction of metal ions in the grooves is hindered, which exacerbates the roughness of the composite coating and then forms a mushroom-like structure.

Table 2 tabulates the EDS results of Ni-Co-Al₂O₃-MoS₂ composite coatings. With the increase of MoS₂ concentration, the contents of Ni and Co in the composite coating are gradually decreased. When the MoS₂ concentration is low (< 1.0 g·L⁻¹), the relative contents of Al and O present an increasing trend. When the MoS₂ particles increased to 1.5 g·L⁻¹ and 2.0 g·L⁻¹, the proportion of Al and O elements showed a downward trend, indicating that the introduction of MoS₂ particles inhibited the deposition of Al₂O₃ particles.

3.2 Micro-indentation hardness

The micro-indentation hardness of the Ni-Co-Al₂O₃-MoS₂ composites is provided in Figure 7. With increasing

Table 2. Results of energy spectrum analysis of the coatings.

| MoS ₂ | Ni/at% | Co/at% | Al/at% | O/at% | Mo/at% | S/at% |
|-----------------------|--------|--------|--------|-------|--------|-------|
| 0.5 g·L ⁻¹ | 36.27 | 21.12 | 14.57 | 20.34 | 3.11 | 4.59 |
| 1.0 g·L ⁻¹ | 35.96 | 20.66 | 15.80 | 19.88 | 4.66 | 5.04 |
| 1.5 g·L ⁻¹ | 35.90 | 19.81 | 12.12 | 17.97 | 5.99 | 8.21 |
| 2.0 g·L ⁻¹ | 35.17 | 19.72 | 12.25 | 16.91 | 6.09 | 9.86 |

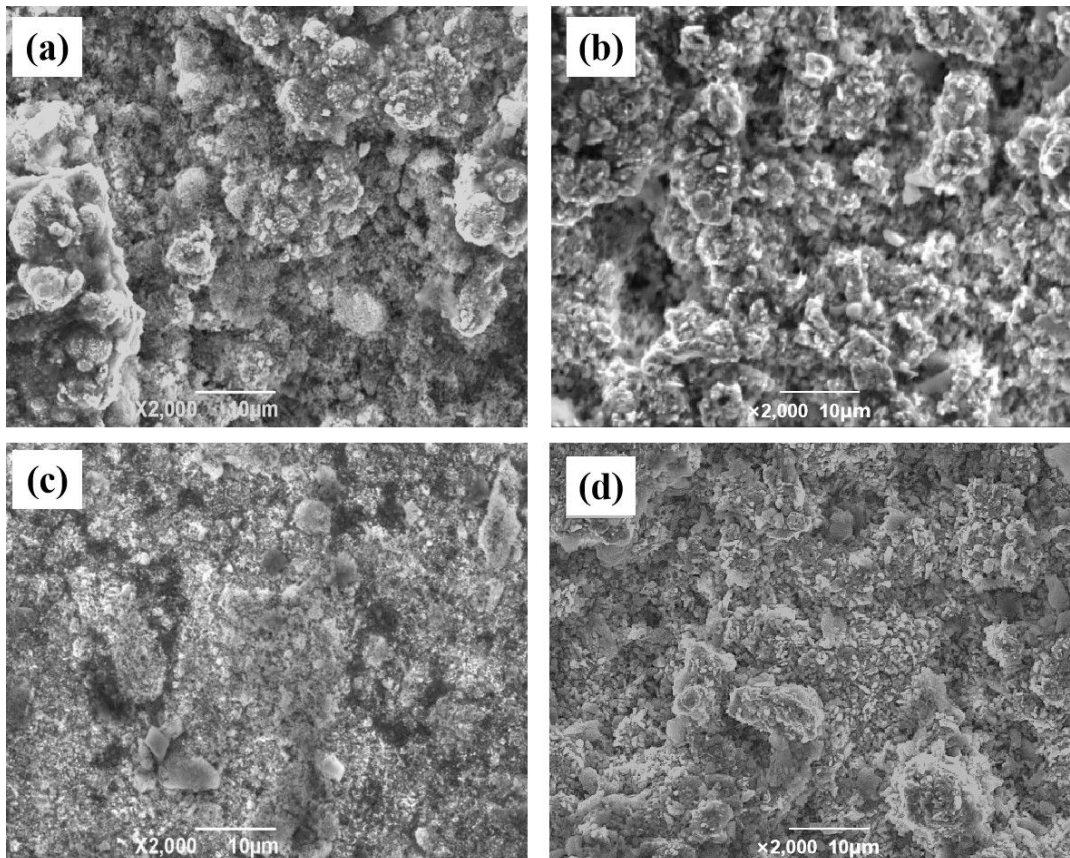


Figure 6. Surface morphologies of the composite coating with different MoS₂ addition: (a) 0.5 g·L⁻¹; (b) 1.0 g·L⁻¹; (c) 1.5 g·L⁻¹; (d) 2.0 g·L⁻¹.

the concentration of MoS_2 particles, the micro-indentation hardness of the composite coating tends to decrease. Due to introducing soft particles of MoS_2 , the micro-indentation hardness of the composite coating is correspondingly reduced. In addition, the addition of MoS_2 hinders the deposition of Al_2O_3 particles and weakens the strengthening effect of hard particles in the coating, which is consistent with EDS results.

3.3 Friction and wear

It is generally considered that low friction coefficient and wear rate represent excellent friction and wear resistance. Figure 8 shows the friction coefficient of Ni-Co matrix

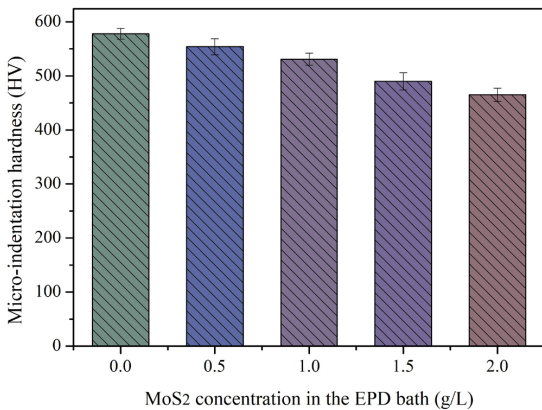


Figure 7. Micro-indentation hardness of the composite coatings.

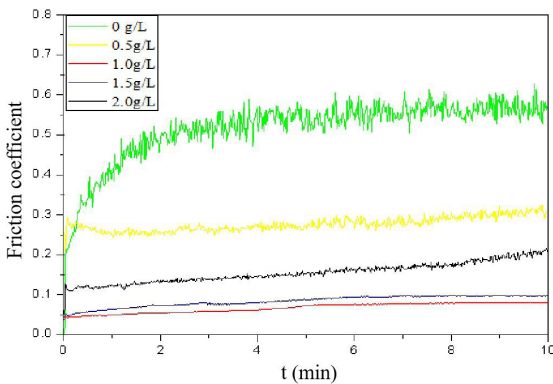


Figure 8. The effect of MoS_2 addition on the friction coefficient of the composite coating.

composite coatings. It can be seen that the friction coefficient of the coating deposited at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 reaches the lowest value, which indicates that the coating has excellent anti-friction performance. The micro-hardness of the composite coating without introduction of MoS_2 is higher because it has more Al_2O_3 particles. As the friction continues, the Al_2O_3 particles in the coating are gradually exposed and even fall off. The exfoliated Al_2O_3 particles act as abrasive particles in the process of wear, so that the original adhesive wear changes to abrasive wear, which further aggravates the wear²⁹. Therefore, the friction coefficient increases quickly and then remains at a high level of friction balance in coefficient state. With the introduction of MoS_2 particles into the composite coating, the friction coefficient of the composite coating is lower than that without adding MoS_2 particles, which proves that MoS_2 plays a lubricating role to a certain extent. As the concentration of MoS_2 was increased to $1.0 \text{ g}\cdot\text{L}^{-1}$, the composite coating became extremely smooth and dense, and the friction coefficient of the coating decreased significantly. This phenomenon may be attributed to the formation of lubricating film on the coating surface, which effectively improves the wear resistance of the coating³⁰. If the MoS_2 concentration exceeds $1.0 \text{ g}\cdot\text{L}^{-1}$, many bumps appear on the coating surface, and the surface quality of the coating deteriorates obviously. In addition, the uneven distribution of particles and the appearance of defects such as holes and cracks cause the coating structure to be extremely uneven, which leads to the increase of friction coefficient.

The schematic of the friction mechanism of the coating are shown in Figure 9. At the beginning of friction, MoS_2 particles are difficult to fully play the role of lubrication between friction pairs due to insufficient MoS_2 stripping. With the continuous friction and wear, a large number of MoS_2 particles are pulled out from the composite coating, forming a layer of self-lubricating film between the friction pairs, which significantly improves the wear resistance of Ni-Co- Al_2O_3 - MoS_2 composite coatings. In addition, the uniformly distributed hard particles have “pinning effect” on the Ni-Co matrix, which effectively hinders the plastic deformation and exfoliation of the matrix during the process of friction and wear.

The wear rate of Ni-Co- Al_2O_3 - MoS_2 composite coatings with various MoS_2 concentrations is presented in Figure 10. The minimum wear rate of the Ni-Co- Al_2O_3 - MoS_2 composites fabricated at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 is recorded, testifying the superior wear resistance. This is attributed to the uniform and dense

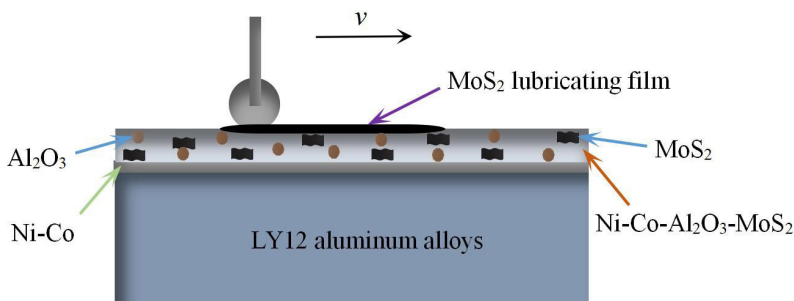


Figure 9. Schematic diagram of friction process of Ni-Co- Al_2O_3 - MoS_2 composite coating prepared at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 .

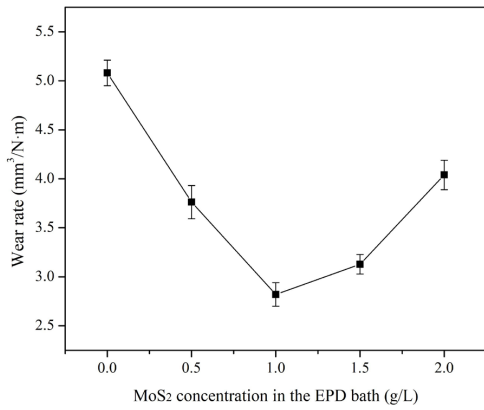


Figure 10. The variation of wear rate of the composite coatings prepared with different MoS₂ concentrations.

microstructure of the composite coating and the formation of solid lubricating film under this condition, which effectively alleviates the wear of the coating.

Figure 11 shows the worn scar morphologies of as-prepared composites. From Figure 11a concerning the worn scar morphology with 0 g·L⁻¹ MoS₂, it can be seen that the wear is serious comparatively. Figure 11b shows a shallower mark than Figure 11a. MoS₂ particles, between the friction pairs, play a lubricating role to a certain extent. As the concentration of MoS₂ particles was increased to 1.0 g·L⁻¹, a wide scratch appeared on the coating surface, and local peeling occurred due to adhesive wear (Figure 11c). This result seems to deviate from the low friction coefficient and wear rate. The reason may be due to the fact that the Ni-Co-Al₂O₃-MoS₂ composite coating fabricated at 1.0 g·L⁻¹ MoS₂ is even and dense (confirmed by Figure 5b), and the protrusion

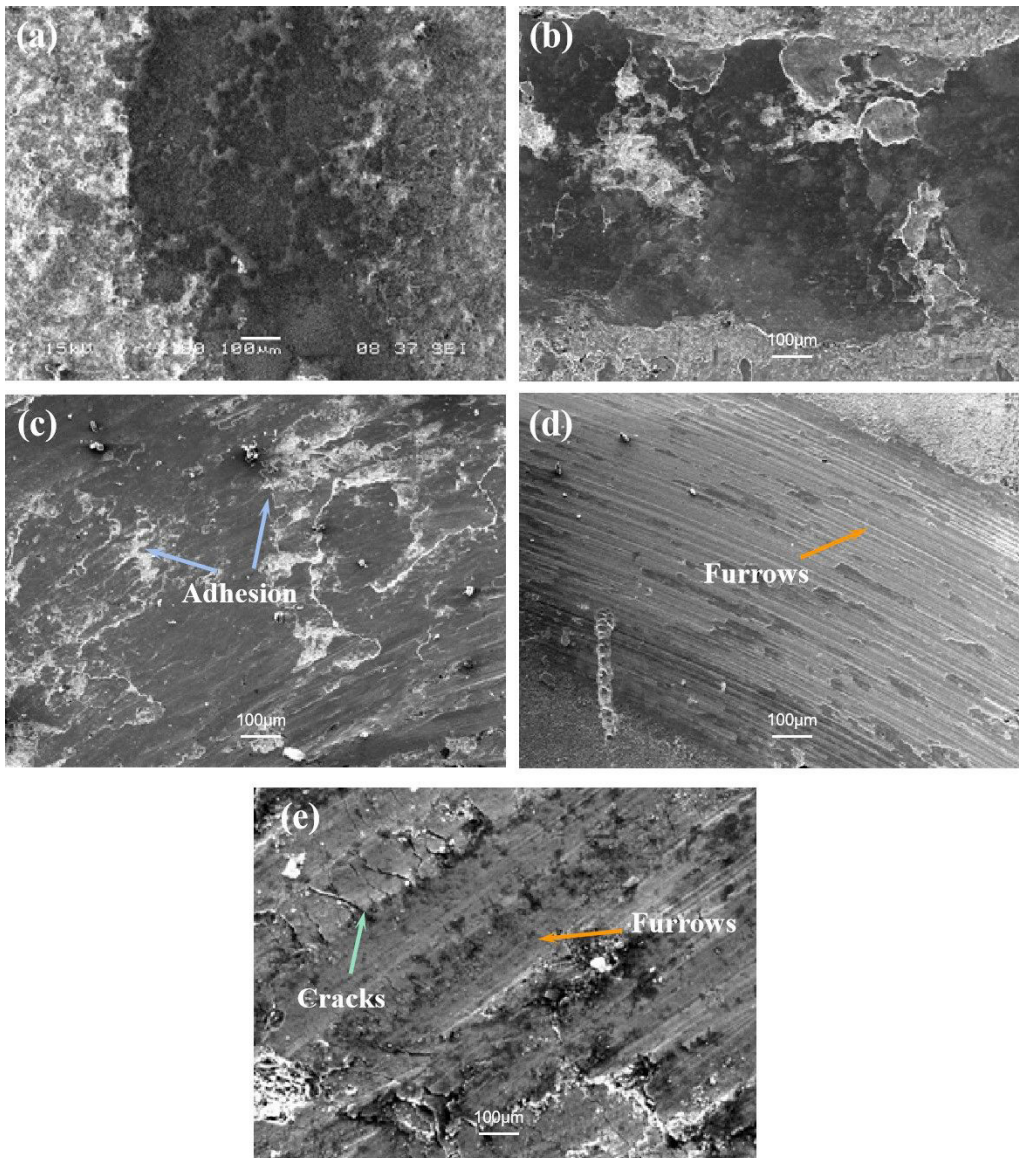


Figure 11. SEM images of worn morphology of as-fabricated composites: (a) without adding MoS₂ particles; (b) and (c) adhesive wear of the coatings with MoS₂ concentration of 0.5 g·L⁻¹ and 1.0 g·L⁻¹, respectively; (d) and (e) abrasive wear of the coatings with MoS₂ concentration of 1.5 g·L⁻¹ and 2.0 g·L⁻¹, respectively.

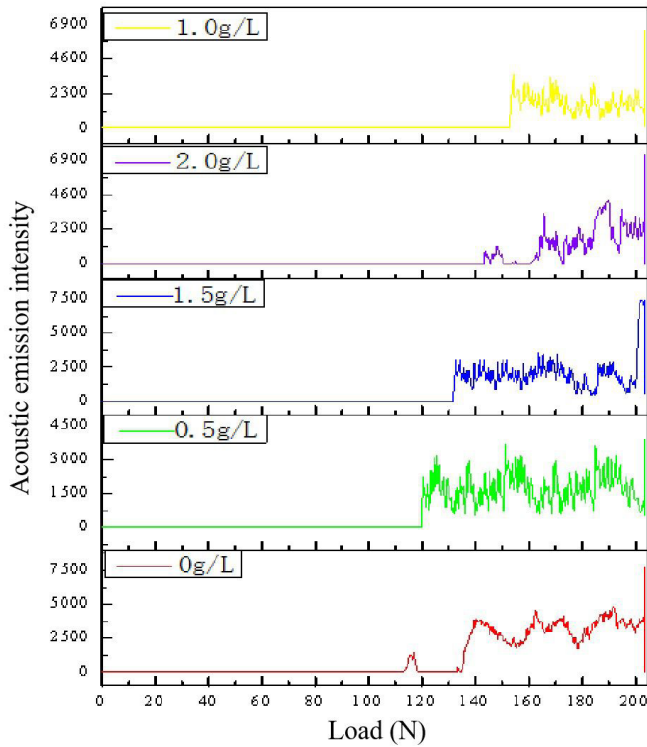


Figure 12. Acoustic emission spectra of Ni-Co matrix composite coatings.

formed on the coating surface is obviously reduced, thus losing the support function between the friction pairs, increasing the friction area, and finally leading to a wider wear mark. Simultaneously, the shortening of friction running in period because of the decrease of the protrusion and the formation of continuous lubricating film result in low friction coefficient and wear rate of the composite coating.

With the increase of MoS_2 concentration to $1.5 \text{ g}\cdot\text{L}^{-1}$, obvious furrows were observed by SEM (Figure 11d). This may be attributed to the uneven distribution of hard particles, and the agglomerated particles are prone to fall off. More abrasive particles are formed under the action of friction extrusion, which leads to serious abrasive wear on the coating surface. When the MoS_2 concentration is $2.0 \text{ g}\cdot\text{L}^{-1}$, many defects such as cracks and holes appear in the coating, which weakens the continuity and uniformity of the alloy matrix, and the coating surface becomes rough and uneven. During the process of friction and wear, a large number of cracks and furrows appear at the scratches, indicating severe wear conditions (Figure 11e).

3.4 The bonding force

The adhesion scratch test is designed to examine the bonding strength between coating and substrate. Figure 12 shows the curve of the acoustic emission signal-pressure loading (K-L) of the composite coatings. The acoustic emission spectrum is shown in red, corresponding to Ni-Co- Al_2O_3 composite coating. When the applied load reaches 117.8 N, the jumping increase of acoustic emission intensity indicates that the composite coating has failed. With the increase of MoS_2 concentration, the critical load initially increases

and then decreases. The critical load of the composite coating containing $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 reaches the highest value about 154 N, which means that the Ni-Co matrix composite coating embedded with Al_2O_3 ($10 \text{ g}\cdot\text{L}^{-1}$) and MoS_2 ($1.0 \text{ g}\cdot\text{L}^{-1}$) has a good combination with LY12 aluminum alloy substrate.

4. Conclusions

1. Ni-Co matrix composites were successfully synthesized on the surface of LY12 aluminum alloys. At the MoS_2 concentration of $1.0 \text{ g}\cdot\text{L}^{-1}$, Ni-Co- Al_2O_3 - MoS_2 composite coatings with uniform and compact microstructure were obtained, Al_2O_3 and MoS_2 particles were homogeneously embedded into the Ni-Co alloy matrix, and the average thickness of the coating was $39.820 \mu\text{m}$. As the MoS_2 concentration was increased to $2.0 \text{ g}\cdot\text{L}^{-1}$, numerous defects such as holes and cracks appeared in the coating.
2. With the introduction of $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 , the micro-indentation hardness of the coatings decreased slightly to 531 HV. In addition, the minimum friction coefficient and wear rate of the as-prepared composites were achieved at $1.0 \text{ g}\cdot\text{L}^{-1}$ MoS_2 , which originated from the “pinning effect” of Al_2O_3 particles on Ni-Co matrix and the formation of a complete self-lubricating film. Meanwhile, the adhesion between the composite coating and the substrate was good, without obvious cracks and delamination. The maximum bonding strength was recorded as 154 N.

5. Acknowledgments

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