

A Method for Electroless Nickel Plating on Aluminum Alloy Surface

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Abstract: According to the following plating processes: deoiling → washing → pickling → washing → drying → weighing → electroless plating → washing → drying → weighing. It can obtain Ni-P deposit on aluminum alloy. It studied the influence of bath composition and process parameters on the composition and deposition rate of the alloy coating. Under temperature of 85~90°C, nickel sulfate hexahydrate 15~35g/L, sodium hypophosphite 10~30g/L, sodium citrate 5~10g/L, tartaric acid 1~2g/L, pH 3~5, reaction time 30~60min, load factor 1.0~2.0dm²/L. The microstructure, surface morphology, composition and valence of the elements in the alloy coating were studied by metallographic microscope, SEM, EDS and other modern analytical methods. The size of spherical grain was below 1um and compact distribution. The chemical coatings were mainly composed two elements, which were phosphorous and nickel. The mass percentage of phosphorous was about 15%, and the other one was about 80~85%. The corrosion resistance of the alloy coating was studied by CASS [1] test method, through 80h CASS test detection, the protection class of chemical coating can be reached 5. The relevant evaluation criteria can be referred to GB/T 6461-2002 [2]. The results show that the Ni-P binary amorphous alloy can be successfully prepared by this process.

Keywords: Electroless Plating, Alloy, Plating, Ni, P, Amorphous Alloy

1. Introduction

Amorphous alloy [3, 4] is applied to ultra-rapid cold solidification, during which the atoms cannot be arranged in an orderly manner to crystallize. The solid alloy obtained is a long-range disordered structure, and there is no grain or grain boundary of crystalline alloy. In 1911, G. T. Bly et al. theorized that amorphous alloys could be made from melt quench. Amorphous alloys are a new type of metal materials developed in the 1970s. In 1946, Brenner and Riddel [5, 6] obtained the first electroless Ni-P amorphous coating in a solution consisting of nickel salts and sodium hypophosphite salts, and it was widely used in the PCB [7], cast iron [8], glass microspheres [9, 10], carbon [11], titanium [12], SiC ceramic [13], magnesium alloys [14-16], PVC [17].

There are many researches on electroless nickel plating on aluminum matrix in China [18-20], but the content of phosphorous element is not high, and the anti-corrosion ability

is also poor.

This paper mainly studies electroless nickel plating on aluminum alloy surface to improve its hardness and corrosion resistance, expand its application range and strengthen its service capability.

2. Experimental Conditions and Methods

Materials and composition: 6061 aluminum alloy (magnesium0.8-1.2%, ferrum0.7%, silicon0.4-0.8%, copper0.15-0.4%, zinc0.25%, chromium0.04-0.35%, manganese0.15%, titanium0.15%).

Experimental condition: Structure analysis of electroless Ni-P alloy coating with 6061aluminum alloy plate of 15cm×7cm×0.1cm as substrate.

Plating process: deoiling (20g/L NaOH, 20g/L Na₂CO₃, ultrasonic stirring 5min, room temperature)→ washing (DI water, room temperature)→ pickling (20%H₂SO₄, ultrasonic stirring 5min) → washing (DI water, room temperature) → drying (blow

dry, room temperature) → weighing → electroless plating (Ni₂SO₄·6H₂O 15~35g/L, Na₂HPO₂ 10~30g/L, complexing agent A and B, 85~90°C, pH3~5, 40min, load factor 1.0~2.0dm²/L)→ washing (DI water, room temperature) → drying (blow dry, room temperature) → weighing.

Composition of deoiling solution: 20g/L sodium hydroxide and 20g/L sodium carbonate solution; Composition of acid lotion: sulfuric acid solution with 20% mass content. Bath formula: nickel sulfate hexahydrate 15~35g/L, sodium hypophosphite 10~30g/L, complexing agent A: sodium citrate

0~10g/L, complexing agent B: tartaric acid 0~2g/L, complexing agent C: EDTA 0~2g/L, complexing agent D:

Reactive condition: 85~90°C, pH: 3~5, load factor: 1.0~2.0dm²/L.

Research method: the quality ratio, nickel salt content, sodium hypophosphite content and polishing performance indexes of different complexing agents at different time were investigated. The surface grain morphology and element content of the coating were detected by Su-70 scanning electron microscope and energy spectrum.

3. Results and Discussions

3.1. Effect of Coating Time on Coating Quality

Table 1. The coating time and coating quality.

Coating time (min)	6	18	33	40	60
Weight quality Δm (mg)	10	70	90	90	170
Film deposition rate (mg/h·cm ²)*	0.65	1.52	1.06	0.88	1.10
The mass fraction of the Ni element (%)	23.09	67.33	81.02	82.11	84.08
The mass fraction of the P element (%)	6.52	13.56	15.76	15.83	15.33
The mass fraction of the Al element (%)	69.17	18.59	2.66	0	0

*Note: The effective area of the deposit was 11cm×7cm×2=154cm², the same as below.

The coating time has a great influence on the deposition of the coating. Table 1 studies the deposition rate of the coating at different times of 6~60min and the contents of Ni and P elements in the coating: it found that when the content is less than 33min, the content of Ni element in the coating is less than 81% and the content of P element is less than 15%. The reaction time was extended to 60min, and the Ni content in the coating was 84%, which was 3.8% higher than that in 33min. The content of P element was 15.33%, which was 2.7% less

than that at 33min. If the reaction time is too long, it will lead to the consumption and decomposition of the plating solution, while if the reaction time is too short, Ni-P deposition cannot be formed. The reaction time of 40min is appropriate, which can ensure that the P content in the coating is at a high value of 15.83% and the Ni content is above 82%. After 40min, the surface of aluminum alloy are completely covered by Ni atoms and P atoms, so the content of Al element will not be detected.

3.2. Effect of Complexing Agent on Coating

Table 2. The mass ratio of complexing agent and coating quality.

The mass ratio of complexing agent A: B	A: 5g/L B: 1g/L 5: 1	A: 5g/L B: 2g/L 5: 2	A: 7g/L B: 2g/L 7: 2	A: 10g/L B: 2g/L 10: 2
Weight quality Δm (mg)	160	120	110	110
Film deposition rate (mg/h·cm ²)	1.56	1.17	1.07	1.07

Note: The nickel salt 15g/L, sodium hypophosphite 10g/L, the reaction time is 40min, and temperature is 85-90°C.

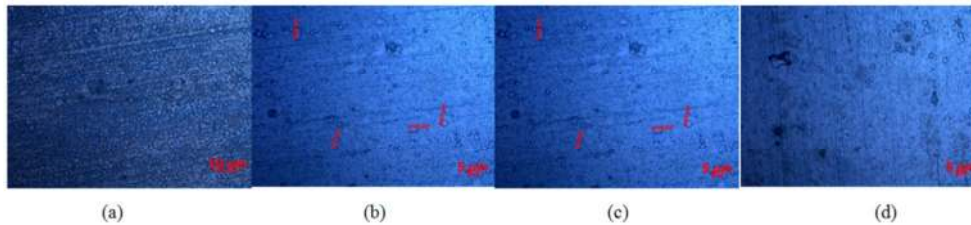
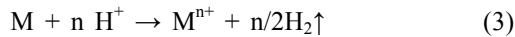
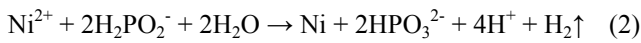
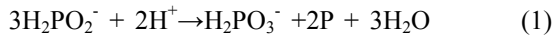


Figure 1. Ni-P coating morphology under different proportions of complexing agent A and B (1000 times).

(a) Complexing agent A and B=5: 1; (b) complexing agent A and B=5: 2; (c) Complexing agent A and B=7: 2; (d) complexing A and B=10: 2.

As can be seen from table 2 and figure 1, the ratio of complexing agent is adjusted when the main salt and reducing agent are unchanged and the mass ratio is 15: 10. When the mass ratio of sodium citrate and tartaric acid is 5: 1, the

coating is more uniform, dense and the deposition rate is the maximum, so it can get the best condition while the ratio of complexing A and complexing B is 5: 1.



From formula (2), it can be seen that the concentration of H^+

increases, which increases the concentration of products, and the equilibrium moves in the direction of the reverse reaction, inhibiting the deposition of Ni atoms. When the concentration of complexing agent B tartaric acid increased, the concentration of H^+ in the solution increased and the deposition rate of Ni decreased.

3.3. Effect of Nickel Salt Content on Coating

Table 3. The content of nickel salt and coating quality.

The mass ratio of Ni salt: P salt	Ni salt: 20g/L P salt:	Ni salt: 25g/L P salt:	Ni salt: 30g/L P salt:	Ni salt: 35g/L P salt:
	10g/L	10g/L	10g/L	10g/L
	20: 10	25: 10	30: 10	35: 10
The mass ratio of complexing agent A: B	5: 1	5: 1	5: 1	5: 1
Weight quality Δm (mg)	130	120	150	110
Film deposition rate ($\text{mg}/\text{h}\cdot\text{cm}^2$)	1.27	1.17	1.46	1.07

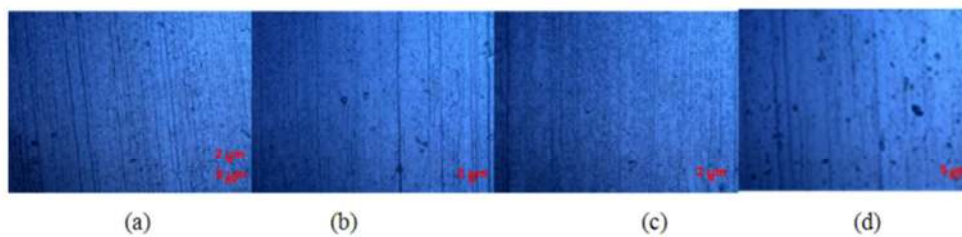


Figure 2. The morphology of Ni-P coating under different proportions of nickel and phosphorus salt (1000 times).

(a) The mass ratio of Ni salt: P salt=20: 10; (b) The mass ratio of Ni salt: P salt=25: 10; (c) The mass ratio of Ni salt: P salt=30: 10; (d) The mass ratio of Ni salt: P salt=35: 10.

The content of nickel salt has a great influence on the deposition rate of the coating, as can be seen from table 3: when the mass ratio of nickel phosphorus salt is 30: 10, the deposition rate of the coating reaches the maximum, reaching $1.46\text{mg}/\text{h}\cdot\text{cm}^2$. However, when the mass ratio of Ni/P is 15: 10, the deposition rate of the coating is the smallest, only $1.07\text{mg}/\text{h}\cdot\text{cm}^2$. A doubling of the nickel salt content increases the deposition rate of the coating by 36%, which is consistent with the principle of equilibrium movement: increase the concentration of the reactants, shift the equilibrium toward the products, and increase the reaction rate. As the concentration of the reactant Ni^{2+} increases, the reaction rate accelerates, which is conducive to the precipitation of Ni. When the content of nickel salt increases by 1 times, the concentration of nickel salt continues to increase, the deposition rate of the coating decreases instead, possibly because the plate surface has a certain thickness of Ni-P coating, at this time, because of the exclusion of Ni^{2+} by the atomic layer in the coating, the deposition rate of the coating decreases. As can be seen from figure 2, with the increase of nickel ion content, the coating becomes more uniform and denser. From above all, it can include that the best mass ratio of nickel salt and hypophosphite salt is 30: 10.

3.4. The Corrosion Resistance Testing

From Figure 4, the corrosion resistance of aluminum alloy surface coatings was tested by the 80hCASS test method, and the samples were rated after the corrosion test [19, 20]. The

final electroless plating process conditions are as follows: hexahydrate nickel sulfate 30g/L, sodium hypophosphite 10g/L, sodium citrate 5g/L, tartaric acid 1g/L, reaction time 40min, heating temperature $85\sim 90^\circ\text{C}$., load factor $1.0\sim 2.0\text{dm}^2/\text{L}$. The deposition rate could be up to $1.56\text{mg}/\text{h}\cdot\text{cm}^2$, the film owned the best quality. The coating had the best corrosion resistance and the protection level can reach 5.

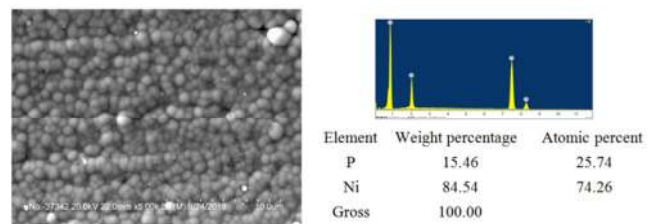


Figure 3. The microstructure and energy spectrum of Ni-P coating under SEM (5000 times).



Figure 4. The appearance of Ni-P coating before and after 80hCASS test.

After 146h NSS test, the surface of 6061 Al alloy appeared many tiny hole erosion, but 6061 Al alloy plate with Ni-P electroless plating had no from Figure 5. It shown that Ni-P electroless plating improved the resistance of corrosion of matrix. According to GB 6461, the protection of level can reach 5.



(a)



(b)



(c)

Figure 5. 146h NSS test.

(a) 6061Al; (b) Ni-P electroless plating film; (c) peel off the Ni-P electroless plating film.

3.5. The Roughness of Ni-P Electroless Plating

Table 4. The roughness of Ni-P electroless plating.

The sample category	6061Al alloy	Ni-P electroless plating
Roughness (um)	0.1-0.15	0.4-0.9

Before electroless plating, the roughness value of 6061Al alloy was only 0.1-0.15um, after electroless plating, the roughness value of Ni-P electroless plating could be up to 0.4-0.9um. Because the accumulation of atoms on the surface of aluminum alloy, which increased the thicker of coating. But the growth of membrane is irregular, which is subject to some external conditions. Temperature, time, ions concentration, mixing way, the placement state of sample in the bath and so on. It is impossible to achieve the absolute consistency of the surface film, so there are differences in roughness, but most are less than 1um. Generally, the roughness value is about 0.5um, which is five times of aluminum alloy.

3.6. The Hardness of Electroless Plating

Table 5. The hardness of electroless plating.

The sample category	matrix	Ni-P electroless plating
Hardness (HV)	100-110	120-140

From table 5. The hardness value of matrix was 100-110HV under pressure of a kilogram of force. After plating, the hardness value of Ni-P electroless plating was 120-140HV under same load. Comparing before and after plating data, the hardness value was increased by 20% to 30%. So it can be included that according to Ni-P electroless plating method, the hardness of 6061Al will be improved. According to GB/T 4340.1-2009 Metallic materials--Vickers hardness test--Part 1: Test method.

3.7. The Thickness of Plating

The aluminum alloy plate covering Ni-P coating was cut into a square of 1cm*1cm by the method of wire cutting. Then the surface of test sample was cleaned by ultrasonic in 75% ethanol solution for 5min, drying at room temperature.



Figure 6. The thickness of plating.

From Figure 6, the thickness of plating was test by

metallographic microscope. The reference standard for detection method was GB/T 6462-2005 Metallic and oxide coatings--measurement of coating inckness--microscopical method. The value of thickness was 1.5 to 2.2um.

4. Summary

1. The composition of bath (main salt, reducing agent and complexing agent) and process parameters (time and temperature) have great influence on the composition and deposition rate of the coating, which concentration of nickel sulfate hexahydrate is 30g/L, reducing agent is 10g/L, complexing agent sodium hypophosphite is 5g/L, complexing agent sodium citrate is 1g/L, reaction time is 40min, reaction temperature is 85~90°C, load factor is 1.0~2.0dm²/L.
2. The surface micromorphology of Ni-P alloy coating is spherical protuberance with particle size less than 1um. With the increase of Ni content, the coating becomes more uniform and denser.
3. After polishing, the surface roughness is improved, the surface area in contact with the plating solution increases, but the deposition rate was decreased.
4. According to the corrosion resistance test of 80h CASS and 146h NSS, both of the Ni-P electroless plating on the surface of 6061 aluminum alloy could be up to protection level 5. The test data show that it has good corrosion resistance.

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