



The study of electroless nickel plating directly on magnesium alloy

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ABSTRACT

Magnesium alloy was disposed by three kinds of acid pickling formula and activation formula and the effect of the three kinds of acid activation formula on magnesium alloy was studied by contrast experiment. The experimental results indicated that after disposed by acid pickling formula of HNO_3 25 ml/L, H_3PO_4 25 ml/L, room temperature and activation formula of $\text{NH}_4\text{H}_2\text{PO}_4$ 80–100 g/L, NH_4F 30–50 g/L, room temperature, magnesium alloy could realize electroless nickel plating directly. The properties of the nickel plating layer and activation layer were researched by electrochemical polarization curves, X-ray diffraction and scanning electronic microscope and its energy spectrometer. The results showed that the structure of Ni–P coating was amorphous, the Ni–P coating was very meticulous and uniform, the activation coating was mainly MgF_2 and $\text{Mg}_2\text{P}_2\text{O}_7$, and comparing with magnesium substrate, the corrosion potential of magnesium alloys increased by about 1.1 V and the corrosion current density declined obviously. Tested by thermal shock test and file test, the adhesion of magnesium alloy and Ni–P coating was good.

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1. Introduction

Magnesium alloy has been praised as “green engineering material” in the 21st century and is widely used in many fields such as aerospace, automotive, electronic, national defense, and so on [1]. In some ways, it appears that the trend is that magnesium alloy will gradually replace steel and aluminum alloy. However, the magnesium alloy has a fatal drawback, that is, its standard potential is extremely negative and is prone to be corroded in the environment, which limits its use. Electroless nickel plating, as one of the magnesium alloy surface treatment means [2–6], has good corrosion resistance, abrasion resistance and magnetic resistance, thus has the favor of people.

At present, there are two main types of magnesium alloy chemical nickel plating processes [7–10]. One is magnesium alloy is dipped in zinc or other metals, then proceeds to electroless nickel; another is that magnesium alloy is pickled by chromic acid, activated by hydrogen fluoride, then proceeds to directly electroless nickel plating. The two processes prove to be more mature, but either the process is too complicated, or the pretreatment solution contains harmful chemical substances to the environment and the human body.

This paper studied the influence of the different pickling and activation processes to magnesium alloy substrate surface structure, and the experiment adopted suitable pretreatment processes, namely directly electroless plating in the surface of magnesium alloy, magnesium alloy chemical plating of coating uniformity and good adhesion strength

prepared. The purpose of this study was to find a pretreatment process that was suitable for magnesium alloy and caused no harm to the environment and human body.

2. Experimental methods

This experimental material was AZ91D, its chemical composition (mass fraction) is as follows: 93% Mg, Al 6.5%, and others 0.5%. This experiment adopted the technological process of electroless nickel plating: preliminary grinding sample, alkaline degreasing, pickling etching, activating, directly electroless nickel plating (deionized water washing between each step).

Oil removal process: NaOH 15 g/L, Na_2CO_3 25 g/L, and 0.5 g/L sodium dodecyl sulfate, at a temperature of 60–70 °C for 2 to 3 min. The purpose of oil removal was to degrease, remove dirt and other attachments on the surface of the specimen and make the magnesium alloy expose clean surface.

The purpose of pickling was to remove oxide on the surface of the magnesium alloy and appropriately coarsen the surface, then make the coating and the magnesium substrate produce “interlocking” effect to improve the ability of combination [11]. The traditional pickling process was chromic acid pickling. Because this process can form a kind of chromate conversion coating in the magnesium alloy surface, it would not cause too much corrosion to magnesium alloy. Because of great toxicity, it did not conform to the requirements of environmental protection. This study compared the three kinds of pickling technology. The formulas were: I, H_3PO_4 25 ml/L, HNO_3 25 ml/L, room temperature, time of 30 s; II, 30–50 g/L lactic acid, room temperature, time of 60 s;

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and III, 15 g/L lactic acid, H_3PO_4 0.04 g/L, the F^- 0.4 g/L, room temperature, time of 60 s.

Activation was the key step in the directly electroless nickel plating. At present, the activation fluid was given priority to hydrogen fluoride. It can form a MgF_2 conversion coating in the magnesium alloy surface and prevent the corrosion of magnesium alloys in electroless nickel plating solution. But the fluoride layer had no catalytic activity to nickel deposit. There were a lot of researches on how the nickel was to start the initial deposition. Due to various reasons, fluoride membrane was porous and no dense membrane, actually for a mixture of MgF_2 with MgO . Magnesium oxide in the film was acted as active sites of electroless plating. So the initial deposition of nickel came true. Then the initial deposition of nickel particles was regard as the center and the coating began to grow, gradually spread, and finally covered the entire matrix [12]. According to this study, three kinds of activated formulations were designed to replace the hydrogen fluoride.

- i. $\text{NH}_4\text{H}_2\text{PO}_4$ 80 g/L–100 g/L, NH_4F 30–50 g/L, room temperature, time of 10 min
- ii. Na_3PO_4 80 g/L–100 g/L, K_2ZrF_6 15 g/L–25 g/L, room temperature, time of 10 min
- iii. $\text{Zn}(\text{H}_2\text{PO}_4)_2$ 50 g/L–70 g/L, sodium potassium tartrate 20 g/L–30 g/L, K_2MnO_4 20 g/L–30 g/L, room temperature, time of 5–10 min

Electroless nickel plating solution was composed of the following: nickel sulfate 25 g/L, lactic acid 25 g/L, sodium hypophosphite 30 g/L, stabilizer 1 mg/L, pH 5.0–6.5, and a temperature of 85 °C.

The surface and cross section morphology of the coating was observed by S-4800 ice emission scanning electron microscope, which was produced by Hitachi. The sample surface and the coating composition and phase composition were tested by the D/Max-2200 type X-ray diffractometer. The steady-state current–potential polarization curve (I–E) of the coating was tested by CHI 760C electrochemical workstation, produced by Shanghai Chenhua Instrument Company. The binding force of the coating was tested with the scratch test and thermal shock test.

3. The experimental results and discussions

3.1. Effects of acid pickling on the surface morphology of magnesium alloys

After chemical degreasing, Magnesium alloy respectively was put into pickling formula I, II, and III for pickling to remove the oxide film on the surface of magnesium alloy. The reaction of magnesium alloy in acid lotion I was violent, in acid lotion II was mild, while in acid lotion III the reaction, originally, was violent, and after a certain period of time, was moderate. Fig. 1 showed the SEM images of magnesium alloy using different pickling solutions.

The morphologies in Fig. 1(a)–(c) respectively were magnesium alloy after acid pickling by acid wash I, II, and III. The conclusion from Fig. 1(a) was that the surface of the scale of magnesium alloy after phosphoric acid and nitric acid treatments not only was fully removed, but also had certain microroughness. This can provide good adhesion strength for the plating. Following the process conditions, magnesium alloy surface corrosion phenomenon was not found. The morphology in Fig. 1(b) was magnesium alloys after pickling in acid solution. Lactic acid belonged to organic acid. Comparing with the inorganic acid, lactic acid corrosion of magnesium alloys was much more modest. From the figure we can see that lactic acid corrosion of magnesium alloys was relatively uniform and the surface was smooth. Besides, other impurities or phase precipitation can also be seen, which may be that lactic acid corrosion of magnesium alloys was selective, namely lactic acid corrosion of α -Mg phase was stronger than the β -Mg phase. From the microscopic roughness speaking, the effect of acid pickling with lactic acid was worse than formula I. Fig. 1(c) showed the morphology of magnesium alloy after the acid treatment in the solution

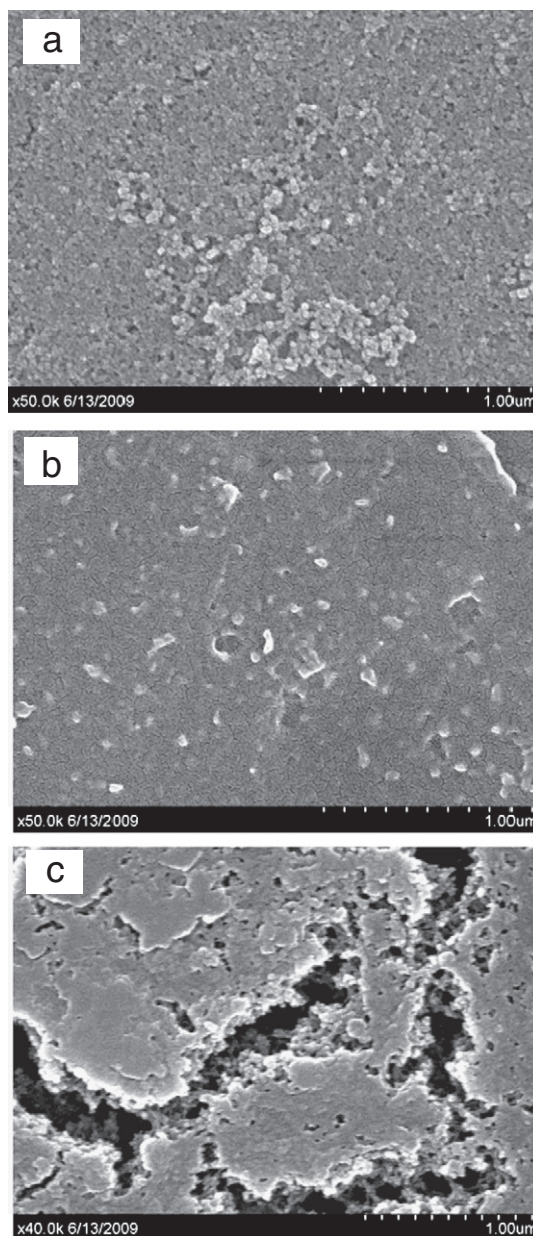


Fig. 1. SEM images of magnesium alloy using different pickling solutions. (a: $\text{HNO}_3 + \text{H}_3\text{PO}_4$, b: $\text{C}_3\text{H}_6\text{O}_3$, c: $\text{C}_3\text{H}_6\text{O}_3 + \text{H}_3\text{PO}_4$).

of lactic acid and phosphoric acid. The phenomenon of “big fissure” was very easy to be observed from the figure, which suggested that overetching had taken place in some areas on the surface of the magnesium alloy, making surface state of magnesium alloy show a great difference, which was harmful to the chemical deposition of nickel. Therefore, based on the analysis of the above, formula I was chosen as the acid lotion for magnesium alloy. The mechanism of the acid pickling formula I is shown below: Nitric acid was a strong oxidizing acid, and can make the substrate surface become rough. In acid liquid, nitric acid can remove oxide film on the surface of the magnesium alloy, and reacted with Mg : $4\text{Mg} + 10\text{HNO}_3 \rightarrow 4\text{Mg}(\text{NO}_3)_2 + \text{N}_2\text{O} + 5\text{H}_2\text{O}$ (1), so the magnesium alloy surface generated evenly corrosion. In acid liquid, phosphoric acid had a strong ability of passivation, its ability to dissolve metal oxide was weak, and reacted with Mg : $2\text{H}_3\text{PO}_4 + 3\text{Mg} \rightarrow \text{Mg}_3(\text{PO}_4)_2 + 3\text{H}_2\uparrow$ (2), which reduced the corrosion from nitric acid to magnesium alloys. So the surface oxidation film of magnesium alloy was removed and it was conducive to the combination of the coating film and magnesium substrate.

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