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The Influence of Directly Plating Time on the Structure of Electroless Nickel on AZ91

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In this paper, the influence of deposition time on the surface morphology, composition, structure, grain size and hardness of electroless nickel plated directly in neutral pH bath on AZ91 magnesium alloy are discussed by using SEM, EDS, XRD and microhardness test machine measurements. The results show that with the increasement of the plating time, the surface tends to be smooth, dense, and complete. The coating is composed of Ni and P and the content of P increases with the increasing the plating time. Furthermore, the XRD peaks for the coatings shift to the right and broaden gradually with prolonged plating time, which indicates that the interplanar spacing of the crystal and the grain size are simultaneously decreased. At the same time, the microhardness of the samples is also increased with increasing plating time.

Keywords: electroless nickel plating, magnesium alloy, plating time.

1. INTRODUCTION

Magnesium has a high strength to weight ratio, with a density that is 2/3 of that of aluminum, and 1/4 of that of iron. Magnesium also has high thermal conductivity, high dimensional stability, good electromagnetic shielding characteristics, high damping characteristics, good machinability, and is easily recycled [1]. These properties make it valuable in a number of applications, including automobile [2] and computer parts, aerospace components, mobile phones, sporting goods, handheld tools, and household equipment [3].

However, the poor anti-corrosion properties of magnesium alloys mean that they are not widely applied. Several surface coating technologies have been developed to improve the corrosion resistance, wear resistance and thermal resistance of Mg alloys, such as electroless plating, conversion coatings, anodizing, gas-phase deposition and thermal spraying [4]. Electroless nickel coatings are widely studied and used due to their special advantages, such as uniform deposition, good corrosion and wear resistance, good electrical and thermal conductivity, and good solder ability [5]. According to Ayushi Thakur [6] Ni/Au deposits on magnesium substrates for aerospace applications have also been witnessed recently [7], and Ni electroless deposits have also been serviceable on engine cranksets, fuel pump cover and shell components in aeronautical applications [8]. The most widely used are electroless nickel–phosphorous alloys (Ni–P) that are obtained from hypophosphite-reduced baths [9, 10].

There are two main types of magnesium alloy nickel plating processes [11 – 17]. One is the magnesium alloy undergoing zinc immersion before electroless nickel plating. The zinc layer is used to prevent the re-oxidation of the magnesium alloy surface [18]. Nevertheless, the zinc immersion procedure is complicated and harmful to health [11]. The other one is that magnesium alloy is pickled by chromic acid, activated by hydrogen fluoride, and then proceeds to directly electroless nickel plating [6]. Generally, nickelous sulfate, nickelous chloride, nickelous acetate and basic nickelous carbonate are usually used as metal ion sources in the electroless plating bath [19 – 22]. However, SO_4^{2-} and Cl^- are corrosive to magnesium substrate, especially in an acidic bath. So, the environmental friendly technology or changing the plating bath pH has been extensively studied to electroless nickel plating.

Recently, Zhao [11] proposed an organic coating as the interlayer between Ni–P coating and AZ31 magnesium alloy substrate. Rajabalizadeh [23] applied the titanate conversion coating as CrO_3/HF free pretreatment for the electroless Ni-P plating on AM60B magnesium alloy. Many other researches on the parameters for pretreatment of magnesium alloy and electroless plating such as the pH, metal ion sources in the electroless plating bath have been presented too [4, 13, 24 – 27]. Among them, acidic and alkaline bath attracts much attention of researchers improving adhesion between coating and magnesium alloy and to improving the coating microstructure. Nevertheless, the bath of neutral pH is somewhat ignored and all these attempts are found to be sound but make the plating process complicated than before. The production cost is increased as well.

Furthermore, Hu [28] indicates that the initial process for the deposition of electroless nickel on a magnesium alloy surface could be described as five continues steps. The plating time is therefore one of the most important parameters affecting the thickness, structure, and properties of the coating.

The dependence of thickness and corrosion properties

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of electroless Ni–P coatings on deposition time has been discussed in our previous study [29, 30]. The structural comparisons of electroless Ni–P coatings obtained from baths of different composition, at different pH and temperature are also discussed by my research fellows [30].

Therefore, in this paper, traditional pretreatment procedure and electroless bath with neutral pH are used to fabricate the electroless nickel phosphorous coating. Although using sodium hypophosphite as reducing agent, as well as the dependence of the incorporated phosphorus concentration on the deposition time are well known and described elsewhere in the scientific literature, the microstructure and the structure of the coatings are general. Ni–P coating is always named Ni–P alloy in literature. However, the relationship between the Ni atoms and P atoms is one of the most important for the structure of coating. Therefore, in this paper, the influence of the directly electroless deposition time on the morphology, composition, structure, grain size and hardness of the nickel coatings and the format of combination between Ni atoms and P atom are discussed.

2. EXPERIMENTAL DETAILS

Casting AZ91 magnesium alloy with a size of 15 mm × 15 mm × 15 mm was used. The samples were rubbed with sandpaper, and then kept in the dehumidifier for use. After ultrasonic cleaning using acetone and alcohol for 10 minutes separately, caustic washing using aqueous solution with composition of NaOH 45 g/L and $NaH_2PO_4 \cdot H_2O$ 5 g/L for 10 minutes at 60 °C, acid cleaning with CrO_3 125 g/dm^3, HNO_3 (65 %) 100 cm^3/dm^3 for a half minute at room temperature, and activating treatment using HF (40 %), 350 ml/L for 15 minutes, the samples were coated at 82 ± 2 °C using in-solution electroless nickel plating, as shown in Table 1. The pH of the plating solution is 7. The plating times used was 1 hour, 1.5 hours, 2 hours, and 2.5 hours, respectively.

The morphological investigations of Ni–P coatings on magnesium alloy substrate were performed by using S-3000N Scanning Electron Microscope (SEM) made by HITACHI company and the composition of the coatings was analysed using Energy Dispersive Spectrometer (EDS). The crystalline structure of the coatings is analysed using Rigaku.D/Max 2500 X-ray Diffraction (XRD). The hardness of the coatings is tested by using HXD-1000TM microhardness test machine and taking the mean value of 5 different points as the microhardness of the coatings.

3. RESULTS AND DISCUSSION

It is clear from Fig. 1 a that the surface of the AZ91 sample for plating for 1 hour is a uneven, with obvious pits and bumps present. With the extension of the plating time, the surface of the sample gradually tends to be stoical, smooth and complete, as shown in Fig. 1 b and d.

In addition, it can be seen that surface of the coatings is grainy distributed, and that the large spherical particles include several small particles. This is due to the fact that deposition of nickel on the surface of the magnesium alloy can be expressed as the following five steps.

Firstly, the dissolution of surface oxides or hydroxides resulting in the exposure of the magnesium alloy takes place. Secondly, reaction on the local anode (base phase) and local cathode taks place. Thirdly, the reaction is still ongoing at the site of Ni_0 generated on the cathode. As the growth point, where gradually growing the nucleus of Ni particles. Fourthly, when two adjacent small particles grow to join each other, because of a squeeze between each other, the small particles gradually merge into larger particles and the boundary between the two small particles become a straight dividing line. Finally, with the extension of the plating time, the small particles gradually grow up, squeezing, fusing, re-squeezing, re-fusing, and eventually make the magnesium alloy surface completely covered by the coating, the coating surface also gradually becomes smooth, dense with the extension of the coating time.

The coatings deposited for 1 hour, 1.5 hours, 2 hours, and 2.5 hours were analyzed using EDS. The results (Table 2) show that the main component of the coating is Ni. There are also P atoms in the coating and the P content is increased with increasing plating time. This is because in the electroless depositing process Ni^{2+} is reduced to Ni by the reducing agent NaH_2PO_2; this process is represented in Eq. 1 and Eq. 2. In addition, because Ni has catalytic abilities, the reduction will proceed spontaneously after nickel is deposited on the surface of the magnesium alloy AZ91 sample [31].

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + 2H^+ + 2e\,; \tag{1}$$

$$Ni^{2+} + 2e \rightarrow Ni \downarrow. \tag{2}$$

At the same time, a small amount of NaH_2PO_2 participates in the redox reaction to produce P and NaH_2PO_3. The reaction process is described in Eq. 3.

$$3H_2PO_2^- + 2H^+ \rightarrow H_2PO_3^- + 2P \downarrow + 3H_2O. \tag{3}$$

As, P is deposited on the surface of the magnesium alloy along with Ni, the Ni/P coating is therefore produced. With increasing plating time, the amount of NaH_2PO_2 participating in the redox reaction is increased. So the amount of P in the coating is increased, too. The EDS measurements did not detect any magnesium.

Table 1. Content of electroless plating bath

Compositions	$NiSO_4 \cdot 6H_2O$	$NaC_2H_3O_2$	$NaH_2PO_2 \cdot H_2O$	NH_4HF_2	HF (40 %)	Thiourea
Content	15 g/L	14 g/L	13 g/L	8 g/L	12 mL/L	1 mg/L

Table 2. Ni and P contents for different plating times (mean value of 5 points for each samples wt.%)

	Plating for 1 h	Plating for 1.5 h	Plating for 2 h	Plating for 2.5 h
Ni	96.77	96.47	95.80	93.56
P	3.23	3.53	4.20	6.44

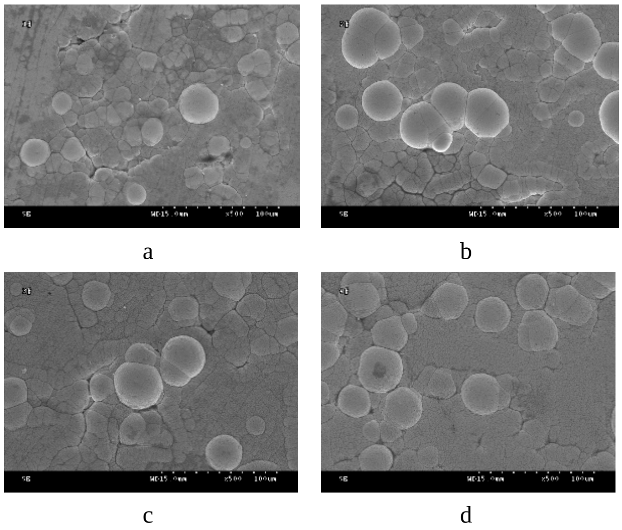

Fig. 1. Morphology of electroless Ni coatings: a – plating for 1 h; b – plating for 1.5 h; c – plating for 2 h, d – plating for 2.5 h

That is, the magnesium alloy is completely covered by the Ni–P coating. It was verified by the XRD results (Fig. 2).

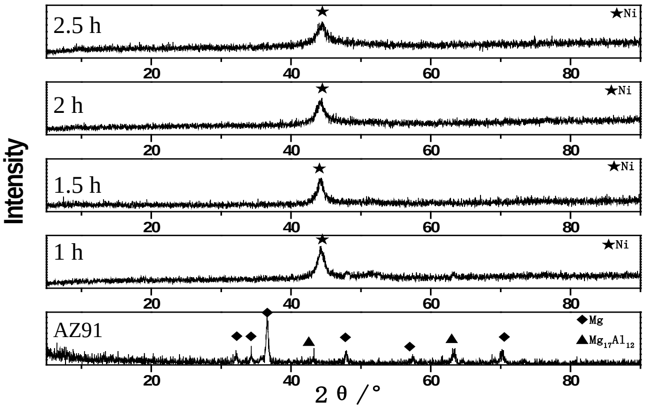

Fig. 2. XRD patterns of samples with different plating time

Table 3. FWHM and grain size determined using Lorentzian fitting

	Plating for 1 h	Plating for 1.5 h	Plating for 2 h	Plating for 2.5 h
$2\theta_B$, °	44. 254	44. 302	44. 357	44. 451
B_θ, rad	1.2944	1.6092	2.0342	2.3634
t, nm	66.24762	53.3006	42.17289	36.31075

From Fig. 2, it can be seen that the sharp diffraction peaks associated with the Ni/P coating are characteristic of a face-centered cubic crystal for the (111) planes of Ni. In addition, when the plating time is increased, the 2θ value for the diffraction peak increases from 44.25° to 44.30°, to 44.36°, to 44.45°, which is close to the position of metal Ni for the (111) planes.

As mentioned above, the coating includes Ni and P. The 2θ value for P should be 37.98°. However, there is no diffraction peak at this position. That is to say, the P element could not exist in the form of elementary P. Moreover, there is no other diffraction peak in Fig. 2 elsewhere. So, Ni_3P or other type of compound of Ni-P hasn't produced and the Ni and P formed a new solid solution [32].

It is well-known that solid solutions can be classified as either interstitial solid solutions, or substitutional solid solutions [33]. If there is a large difference between the radius of the solute atoms and that of the solvent atoms, the solute atoms will enter the crystal lattice and form an interstitial solid solution. The atomic radius of P and Ni are 1.3 Å and 1.24 Å, respectively [34]. Therefore, the similar radius makes it impossible for the atoms of P enter the crystal lattice to form an interstitial solid solution. The Ni and P in the coating formed a substitutional solid solution, with Ni as the solvent atom and P as the solute atom. In the face-centered cubic crystal of Ni, P replaced Ni in its position in the original crystal structure, and the structure of the face-centered cubic crystal is distorted. In addition, because the atomic radius of P is smaller than that of Ni, the Ni atoms around the P atoms would have shifted closer to P; this led to the decrease in the crystal plane spacing, the shift to the right of the X-ray diffraction peak, and the increase in the 2θ values.

The crystal plane spacing d can be obtained from the Bragg formula [35].

$$n\lambda = 2d \sin\theta. \tag{4}$$

For constant $n\lambda$, the interplanar distance d decreases monotonically. That is, the reason for the X-ray diffraction peaks of the Ni/P coatings shift to the right is the decrease in the interplanar distance.

The half-width for each coating was obtained from the XRD results, using Lorentzian fitting, and the grain size in the coating was calculated according to the Scherer formula [33, 36], as shown in Table 3.

Table 3 shows that the grains in the coating deposited on the surface of the magnesium alloy AZ91 by electroless nickel plating are nano-sized particles. The longer the plating time, the smaller the grains; this is the result of the increase in the amount of P in the coating produced by the increasing plating time. Because of the presence of P atoms, there is a lattice distortion. The result of this lattice distortion is that Ni-rich areas appear in the micro-regions near to the P atoms. At the same time, regions lacking Ni are produced around the Ni-rich areas. Because of these Ni-rich and Ni-poor regions, it is difficult for Ni atoms to diffuse and immerge freely over long distances, and this makes it hard for the Ni to deposit as a mega crystal. On the other hand, it is easy for Ni to deposit as fine grains. With increasing plating time, the amount of P in the coating increases gradually, and the lattice distortion arising from the presence of P also increases gradually. The grains of the Ni/P coating therefore became finer as the plating time was increased. If the amount of P in the coating is increased to a certain level, the micro-regions arising from the lattice distortion are numerous enough to hinder the long-range free diffusion of the Ni atoms, which led to the formation of a noncrystalline structure.

In summary, with increasing plating time, the P content in the coating increases monotonically. This causes lattice distortion in the Ni coatings, whose original structure is face-centered cubic. The interplanar spacing is therefore reduced, resulting in a shift of the XRD peaks to the right. Meanwhile, the increasing of the content of P and the lattice distortion means that the grains in the coating became finer with increasing plating time.

The microhardness of the samples with Ni coating versus plating time were tested and the results are shown in Table 4. It is known that the microhardness is monotonically increases with the extension of plating time. The hardness of AZ91 substrate alloy is only 79.5HV. However, the microhardness of the sample with 1 hour nickel plating soared to 418.3 HV, which is more than 5 times the value of AZ91. When the plating time was extended to 1.5 hours and 2.5 hours, the value of the microhardness reached 573.5 HV and 737.2 HV, respectively.

Table 4. Microhardness of samples plating for different time

Sample	AZ91	Plating for 1 h	Plating for 1.5 h	Plating for 2 h	Plating for 2.5 h
Micro-hardness, HV	79.5	418.3	573.5	624.7	737.2

The increase of hardness is the result of the three factors as described as follows. In the first place, in the initial stage of coating deposition, nickel atoms firstly deposit near the base phase and begin to grow to particles to cover the entire specimen surface. When the deposition time is too short, due to the fact that the limited amount of nickel cannot cover the specimen surface, there are small pores, small cracks and other surface defects in the coatings. With the increase of plating time, more nickel particles aredeposited. The particles squeeze each other and form a dense, pore free, and complete coating. On the other hand, the phosphorus content in the coating increased due to the extension of the plating time. Therefore, the hardness increases due to the effects of solid solution strengthening mechanism. The refinement tendency of grains also contributes the hardness increase with longer plating time.

4. CONCLUSIONS

1. The coatings deposited on the surface of the as-cast magnesium alloy AZ91 using direct electroless nickel plating in neutral pH have a composition of Ni and P. The content of P is increased when the plating time is prolonged. Ni_3P and elementary P are not found in the coatings
2. The Ni atoms and the P atoms form a substitutional solid solution, and with increasing plating time, the interplanar spacing of the crystal and the grain size are simultaneously decreased.
3. The microhardness of the coating is monotonically increased versus plating time because of the coaction of the increasing P content and much dense coating.

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