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A protocol for fast electroless Ni-P on Al alloy at medium-low temperature accelerated by hierarchically structured Cu immersion layer

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ABSTRACT

Electroless Ni-P coating is helpful to improve the corrosion resistance and mechanical properties of the Al alloys. However, the electroless Ni-P plating is usually operated at higher temperature (above 80 °C, even up to 90 °C) in order to achieve appropriate deposition rate. Herein it is demonstrated that a novel Cu immersion layer on Al alloy with a hierarchical structure can significantly accelerate the electroless Ni-P process in a wide range of temperature. The hierarchically structured Cu layer is deposited on Al alloy through galvanic replacement deposition from an environment-friendly deep eutectic solvent comprising choline chloride and ethylene glycol. The hierarchical structure of Cu immersion layer strongly affects the nucleation and growth of Ni-P, which is beneficial for enhancing the deposition rate of electroless process. This protocol might be of interest in the fast fabrication of electroless Ni-P deposition on Al alloys at medium and low temperature (50 °C ~ 70 °C).

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1. Introduction

Owing to the remarkable properties, such as high hardness, solderability, as well as excellent resistance to wear and corrosion, the electroless Ni-P coatings are widely used in automotive, aerospace, electronics, chemical process, tooling, printing, and other industries [1–6]. Electroless Ni-P on Al alloys is particularly important because the coatings are very helpful to improve the corrosion resistance and mechanical properties of the Al alloys [7–10].

In order to achieve appropriate deposition rate, the electroless Ni-P plating is usually operated at the higher temperature (above 80 °C, even up to 90 °C) [11–13]. The deposition rate greatly decreases with a decrease in the operating temperature and closes to zero at around 40 °C. There are several inherent problems for the high temperature electroless Ni-P process, including high-energy consumption, poor control of the process and short service life of the bath resulting from the solution volatile. Specific to the electroless Ni-P on Al alloys, there exist some other deficiencies, such as the complicated Zn immersion pretreatments to remove the naturally formed oxide film and the pollution of the plating bath caused by the dissolution of immersion layers [14,15], etc.

Over the past years, great efforts have been made to achieve fast electroless Ni-P at lower temperature (less than 70 $^\circ C)$ and some

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strategies have been proposed. One of the main strategies is additivesactivated electroless, such as accelerators catalysis and organic acid complexing agent [16,17]. Another strategy is external energy-assisted electroless, including ultrasonic assistance, photocatalytic, [18–20] etc. Although these strategies are promising to achieve the appropriate deposition rate at lower temperature to some extent, they also give rise to some side-effects, for example, complicated operation process, co-deposition of other foreign elements, deterioration of the coating properties, and so on [17].

Here, we presented a novel and simple protocol to achieve fast electroless Ni-P process on Al alloys at medium-low temperature. In our strategy, a Cu immersion pretreatment in a deep eutectic solvent instead of conventional Zn immersion pretreatment was carried out on Al alloys before the electroless Ni-P process. For Cu immersion, a deep eutectic solvent based on choline chloride (ChCl) and ethylene glycol (EG) eutectic mixtures was used as deposition media. One of the most prominent characteristics of ChCl-EG deep eutectic solvent is capable of facilitating sustained galvanic replacement deposition with high uniformity, which is in contrast to the aqueous reaction [21]. Our previous studies have shown that a compact and uniform Cu layer on Al (99.5%) substrates has been obtained in the ChCl-EG deep eutectic solvent [22]. Other advantages of this deep eutectic solvent include that it is non-corrosive, environmentally friendly and easily prepared with high purity [23,24]. We found that the Cu immersion layer obtained from this deep eutectic solvent had hierarchical structure, which could significantly enhance the deposition rate of electroless Ni-P process in a wide range of temperature.

Table 1 Chemical composition of Al alloy.

	*	-							
Element	Al	Mn	Si	Fe	Cu	Ti	Zn	other	
(wt%)	97.19	1.17	0.88	0.54	0.10	0.04	0.01	0.07	

Table 2

Conditions of electroless Ni-P coating in alkaline solution.

Chemical composition		pН	Time
NiSO4+6H ₂ O Na ₃ C ₆ H ₅ O7+2H ₂ O NaH ₂ PO ₂ +H ₂ O NH ₃ +H ₂ O	10.5 g/L 23.5 g/L 17.5 g/L 37.5 mL/L	9.30 ± 0.05	0.5 h

Table 3

Conditions of electroless Ni-P coating in acidic solution.

Chemical composition	рН	Time	
NiSO ₄ •6H ₂ O Na ₃ C ₆ H ₅ O ₇ •2H ₂ O NaH ₂ PO ₂ •H ₂ O Lactic acid	15 g/L 10 g/L 20 g/L 5 mL/L	5.00 ± 0.05	0.5 h
CH ₃ COONa·3H ₂ O	20 g/L		

2. Experimental

2.1. Materials and pretreatment of Al alloy samples

Choline chloride [HOC₂H₄N(CH₃)₃Cl] (ChCl) (99%, supplied by Tianjin Guangfu Fine Chemical Research Institute, China), ethylene glycol (EG) (99%, supplied by Tianjin Baishi Chemical Co. Ltd., China), CuCl₂·2H₂O (99%, supplied by Tianjin Kaixin Chemical Co. Ltd., China) and thiourea (99%, Beijing Chemical Works, Beijing, China) were all used as purchased. The chemicals used for electroless Ni-P plating were all purchased from Xilong Scientific Co., Ltd. Al alloys were cut into blocks with 15 mm \times 10 mm \times 5 mm in size in the experiments. The chemical composition of Al alloy substrate used in the experiment was tabulated in Table 1. Prior to deposition, the specimens were mechanically polished using SiC abrasive papers (grade 600, 1000 and 1500) and then were cleaned by immersing sequentially in ultrasonic baths of distilled water and acetone for about 10 min, respectively. Al alloys always were covered with an oxide film even the freshly cleaned and etched surface would be oxidized rapidly. Hence, the Al alloy specimens were subjected to acid solution about 5 s, and then to acetone rinsing with ultrasonic before Cu and Zn immersion pretreatments. The acid solution was hydrofluoric acid (7% of volume percentage) aqueous solution. The Zn immersion pretreatment was used a relatively common procedure according to the literature [25].

2.2. Preparation of Cu immersion layer

The ChCl-EG deep eutectic solvent was formed by mixing ChCl and EG with the molar ratio of 1:2 at 65 °C until a homogeneous, colorless liquid was formed [21]. CuCl₂·2H₂O (0.15 M) and thiourea (0.15 M) were added to freshly prepared ChCl-EG deep eutectic solvent and stirred until fully dissolved. The Cu immersion process was carried out in the Cu²⁺-containing ChCl-EG solution at 30 °C for 2 h. The non-hierarchical Cu immersion layer was also obtained from the ChCl-EG deep eutectic solvent through galvanic replacement. Only CuCl₂·2H₂O (0.15 M) was added to ChCl-EG deep eutectic solvent and stirred until fully dissolved. Freshly pretreated Al alloy substrate was immersed in the Cu²⁺-containing (0.15 M) ChCl-EG solution at 30 °C for 2 h.

2.3. Preparation of Ni-P coatings

Ni-P coatings were plated at different temperature in commonly used alkaline and acidic solutions [25,26], the chemical composition and plating process parameters are listed in Tables 2 and 3. The



Fig. 1. Deposition rates (a, b) of electroless Ni-P on Al alloy with Cu and Zn immersion pretreatment in alkaline (a) and acidic (b) solution from 30 to 80 °C, (c, d) cross-sectional SEM images of the Ni-P coatings with Cu and Zn immersion pretreatment obtained from alkaline (c) and acidic (d) solutions for 2 h at 70 °C.

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