

Effects of choline chloride on electrodeposited Ni coating from a Watts-type bath

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ABSTRACT

Electrodeposition of bright nickel (Ni) was carried out in a Watts-type bath. Choline chloride (ChCl) was applied as a multifunctional additive and substitute for nickel chloride (NiCl₂) in a Watts-type bath. The function of ChCl was investigated through conductivity tests, anodic polarization, and cathodic polarization experiments. The studies revealed that ChCl performed well as a conducting salt, anodic activator, and cathodic inhibitor. The effects of ChCl on deposition rate, preferred orientation, grain size, surface morphology, and microhardness of Ni coatings were also studied. The deposition rate reached a maximum value of greater than 27 μm h⁻¹ when 20 g L⁻¹ ChCl was introduced to the bath. Using X-ray diffraction, it was confirmed that progressive addition of ChCl promoted the preferred crystal orientation modification from (2 0 0) and (2 2 0) to (1 1 1), refined grain size, and enhanced microhardness. The presence of ChCl lowered the roughness of the coating.

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1. Introduction

Electroplated nickel (Ni) is extensively used in engineering applications due to its excellent corrosion and wear resistance, easy mechanical operation, and good electromagnetic characteristics [1,2]. As is known to all, the structural characteristics and mechanical properties of Ni coating are closely related to the plating parameters, such as bath component [3], current density [4], additives [5–9], etc. Nevertheless, under certain suitable conditions, additives have a more profound influence on deposition properties than any other plating variables [5]. Thus, more effort has been placed toward applying new additives to Ni electroplating bath [10–15].

Oliveira et al. [10] studied the effects of glycerol, mannitol and sorbitol finding that the presence of polyalcohols in the electrolytic solution could significantly improve the stability of the bath and achieve good leveling properties to produce a

smooth film. Meng et al. [11] demonstrated that the addition of phytic acid favored the growth of nano-scale twins in the interior of the grains of Ni coating and could improve the corrosion resistance. Sezer et al. [12] revealed that using *N,N*-dimethyl-*N*-2-propenyl-2-propene-1-ammonium chloride-2-propenamide (PQ7) as a surfactant provided the possibility of obtaining Ni coatings with high plating rate, high leveling, and low particle size. The effect of the additive on Ni coating is highly dependent on the functional groups present in their structures. For examples, the additives containing hydroxyl groups, such as 2-butylene-1,4-diol [8] and glycerol [10], usually serve to brighten Ni coating. Compounds containing quaternary ammonium functional group, such as PQ7 [12] and benzyl-dimethyl-alkyl ammonium chlorides [16], play a role of leveling Ni coating surface and inhibiting the cathode Ni deposition. NiCl₂ behaves as a conducting salt while the chloride ion acts as an anodic activator is commonly applied in Ni electroplating bath [17]. In this context, this work aims to explore the feasibility of designing or screening a compound containing various functional groups to serve as a multifunctional additive to simplify the electroplating bath and improve the performance of Ni coating.

Choline chloride (ChCl, 2-hydroxyethyltrimethylammonium chloride, (CH₃)₃N(Cl)CH₂CH₂OH), a cheap, biodegradable, and safe quaternary ammonium salt, has been widely applied to metal Ni

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Table 1
Composition and operating conditions for Ni electroplating.

Bath composition		Operation conditions	
NiSO ₄ ·6H ₂ O (g L ⁻¹)	200	pH	4.0
H ₃ BO ₃ (g L ⁻¹)	30	Temperature (°C)	30
ChCl (g L ⁻¹)	0–200	Current density (A dm ⁻²)	2
		^a Time (min)	10

^a 60 min for microhardness test.

[18,19] and its alloys [20,21] electrodeposition in the form of deep eutectic solvents (DESS). Recently, our group applied ChCl both as a solvent and a ligand to conduct electroless silver [22], tin [23], gold [24], and palladium [25] deposition in ChCl-H₂O solutions. All the coatings exhibited bright surfaces and satisfactory performance. ChCl contains Cl⁻, quaternary ammonium and hydroxyl functional groups. Thus, it could be a good multifunctional additive to simplify the electroplating bath and improve the performance of Ni coating. Applying ChCl to Ni electrodeposition from aqueous solution has not reported up to now.

In the present work, ChCl has been introduced to the most widely used Watts-type bath as a novel multifunctional additive. The roles of ChCl in the plating bath and the effects on Ni coating have been investigated through the techniques of electrochemistry and surface analysis, respectively.

2. Experimental

2.1. Electrodeposition procedure of Ni coating

The electroplating was carried out in a Teflon rectangular cell (8 cm × 6 cm × 5 cm) containing Watts-type bath with different amount of ChCl. A Ni plate (50 mm × 150 mm × 2 mm) and a Cu sheet (20 mm × 50 mm × 0.2 mm) were used as the anode and cathode, respectively, at a distance of 80 mm. The plating bath composition and operating conditions are listed in Table 1. All solutions were prepared with analytic grade reagents and distilled water. Before electroplating, Cu sheets were treated in alkali and acid solutions to be degreased and micro-etched, respectively. After each of the pretreatment steps, the substrates were rinsed with distilled water and dried through air flow.

2.2. Instruments and methods

Conductivity was measured using a conductivity meter (DDS-11A, Shanghai Kangning Electric Light Technology Co. Ltd., China). The microhardness property was analyzed using a microhardness tester (HXD-1000TMC/LCD, Shanghai Taiming Optical Instrument Co. Ltd., China) with a diamond pyramid indenter at a load of 200 g for 15 s.

Electrochemical measurements were conducted on an electrochemical workstation (CHI660D, Beijing Huake Putian Technology Co. Ltd., China). The anodic polarization and cathodic polarization experiments were conducted in the plating solution as was shown in Table 1. The counter electrode and the reference electrode were a platinum plate (10 mm × 10 mm) and saturated calomel electrode (SCE), respectively. The working electrode was a Ni plate anode and a platinum wire electrode for anodic polarization and cathodic polarization experiments, respectively. The potential was scanned at a constant scan rate of 5 mV s⁻¹.

The thickness of Ni coating was measured by energy dispersive X-ray spectroscopy (EDX, EDX1800, Skyray Instrument Co. Ltd., China). Structure and surface morphology of the deposited Ni coating were investigated by X-ray diffraction (XRD, Rigaku D/max 2500 powder diffractometer, Cu Kα (λ = 1.542 Å) radiation at 30 kV and 40 mA, scan rate at 6° min⁻¹, 2θ from 10° to 80°) and

scanning electron microscope (SEM, JSM-6360LA, Japan Electron Optics Laboratory Co. Ltd., Japan), respectively. The roughness of the Ni coatings was tested by optical stylus profilometer (Contour GT, Bruker Corporation, USA).

In order to describe the structure and estimate quantitatively the preferred orientation of the Ni deposits, the relative texture coefficient $RTC_{(hkl)}$ was calculated, which is defined as [14]:

$$RTC_{(hkl)} = \frac{I(hkl)/I_{(hkl)}^0}{\sum_1^3 I(hkl)/I_{(hkl)}^0} \times 100\% \quad (1)$$

where $I_{(hkl)}$ are the diffraction intensities of the (hkl) lines measured in the diffractogram of the deposit and $I_{(hkl)}^0$ are the corresponding intensities of a standard Ni powder sample randomly oriented from JCPDS 04-0850. The summation in the denominator is taken for the three “basic” lines visible in the diffraction pattern, i.e. (1 1 1), (2 0 0), and (2 2 0). A preferred orientation through an axis (hkl) is indicated by the value of $RTC \geq 16.7\%$.

The crystal grain size of Ni coating was calculated through the Scherrer equation [26] on the basis of the Ni (1 1 1) line from the XRD.

3. Results and discussion

3.1. Features of ChCl in plating bath

Firstly, as a quaternary ammonium salt, ChCl contains conducting power. Experiments were carried out to measure the conductivity of the plating bath with different ChCl concentrations (C_{ChCl}) at room temperature. The results showed that the conductivity increased linearly from 46.6 mS cm⁻¹ to 78.2 mS cm⁻¹ with the increasing C_{ChCl} from 0 to 50 g L⁻¹. The linear regression equation for the linear part of the plotted C_{ChCl} – Conductivity curve was established. The slope, intercept and square of the correlation coefficient of the equation were 0.622, 46.7 and 0.9989, respectively. In contrast, the conductivity of Watts bath containing varied NiCl₂·6H₂O concentration (C_{NiCl_2} , all the following mentioned NiCl₂ are referred to NiCl₂·6H₂O) from 0 to 50 g L⁻¹ was also tested. It was found that the conductivity increased with C_{NiCl_2} and reached the maximum of 75.0 mS cm⁻¹ when C_{NiCl_2} increased to 50 g L⁻¹, which is about 1.17 times as many the mole ratio of Cl⁻ in NiCl₂ as in ChCl.

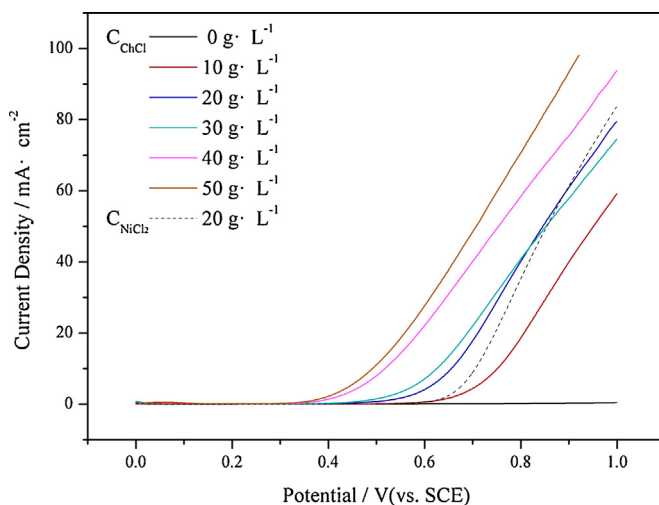


Fig. 1. Effect of ChCl on anode polarization of nickel electrodeposition in comparison with Watts bath containing 20 g L⁻¹ NiCl₂. Conditions are shown in Table 1.

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