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Immersion gold deposition from a chloroauric acid–choline chloride solution: Deposition kinetics and coating performances

Yurong Wang [a], Xiaoyun Cao [a], Wenchang Wang [a], Naotoshi Mitsuzak [c], Zhidong Chen [a,b,*]

[a] School of Petrochemical Engineering, Changzhou University, Changzhou 213164, China
[b] School of Material Science and Engineering, Jiangsu Key Laboratory of Material Surface Technology, Changzhou University, Changzhou 213164, China
[c] Qualtec Co., Ltd, Osaka 590-0906, Japan

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ABSTRACT

A simple, green, stable, and acidic solution was developed based on choline chloride (ChCl) and chloroauric acid ($HAuCl_4$) for immersion gold deposition onto electroless nickel substrate by galvanic displacement process. The relationships between gold (Au) deposition rate and solution parameters were investigated. The optimal conditions were determined as 1.0 $g \cdot L^{-1}$ $HAuCl_4$, 500 $g \cdot L^{-1}$ ChCl, 80 °C, pH 0.5 ~ 2.5. The kinetics equation of IG deposition process was deduced and the corresponding activation energy was calculated to be 28.03 $kJ \cdot mol^{-1}$. At the same time, the performances of the coatings were investigated. The results revealed that Au coatings prepared from chloroauric acid–choline chloride (Au(III)-ChCl) solution had excellent properties on solderability and corrosion resistance.

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1. Introduction

Electroless nickel/immersion gold (ENIG) is gaining in popularity in electronic industries, especially for surface finishing or coating for printed circuit boards (PCBs) to prevent the oxidation of nickel-phosphorus (Ni-P) coating deposited on copper (Cu) surface [1] providing excellent corrosion-resistance and good solderability along with good electrical conductivity [2,3]. Three kinds of immersion gold (IG) deposition technologies have been applied to gold (Au) coatings preparation on Ni-P coating, namely, galvanic displacement, substrate-catalyzed, and autocatalytic processes [4]. Autocatalytic process involves the oxidation of a reductant on the catalytically active surface of the metal being deposited [5]. Although the Au coatings can be formed without a limitation on the thickness, the plating solution became unstable due to the presence of reductant [6]. Potassium dicyanoaurate ($KAu(CN)_2$) was always applied as the source of Au during IG because of its high stability [1,5,7]. However, considering the toxicity of cyanide to human health and the environment, non-cyanide Au plating solutions have been developed, such as Au(I)-thiosulfate complex solution and Au(I)-thiosulfate-sulfite mixed ligand solution [4,6]. Nevertheless, some severe problems still exist. For instance, the compositions in plating solution were relatively complicated. The quality of Au coating did not match the demand of the application. The base bath solution had low compatibility with the commonly used photoresist, which was favored in acidic solutions.

Choline chloride (2-hydroxyethyl)trimethylammonium chloride, ChCl) as a quaternary ammonium salt is widely applied to metal [8] and alloy electrodeposition [9–19] in the form of deep eutectic solvents (DESs) [20–22]. However, relatively low attention was received in metal electroless deposition from ChCl-based DESs [23,24]. Recently, we applied ChCl, serving both as solvent and ligand, to perform sustained electroless silver (Ag) [25] and tin (Sn) [26] deposition from ChCl-H_2O solutions. The plating solution for Ag and Sn deposition was very simple and stable. Vreese et al. [27] also reported that crystals of $CuCl_2 \cdot 2H_2O$, $Cu(choline)Cl_3$, $[choline]_4[Cu_4Cl_{10}O]$, and $[choline]_3[CuCl_4][Cl]$ were formed in the ionic liquid ChCl-$CuCl_2 \cdot 2H_2O$, indicating that ChCl performed as ligand.

Up to now, researches conducted in the application of ChCl to electroless IG deposition have not been reported, so the goal of this work is to develop a non-cyanide, simple, stable, and acidic IG deposition solution for ENIG coating preparation by galvanic displacement process using ChCl as ligand and chloroauric acid ($HAuCl_4$) as the source of Au. Studies were carried out on investigating the effects of the solution compositions and the operation conditions of Au plating process on the deposition rate, and characterization of the coating structures and morphologies. Also, the kinetics equation of IG deposition was deduced, and the performances such as the stability of the Au(III)-ChCl solution, corrosion resistance, and solderability of Au coating were evaluated.

* Corresponding author at: School of Petrochemical Engineering, Changzhou University, Changzhou 213164, China. Tel.: +86 579 86330239; fax: +86 519 86330239.
E-mail address: zdchen.lab@gmail.com (Z. Chen).

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2. Experimental

2.1. Materials and methods

Cu sheets (10 mm × 20 mm × 0.2 mm) were used as the substrates for ENIG deposition. All the chemicals were from Sinopharm (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) with analytic grade. All the solutions were prepared with distilled water. Before electroless plating, the substrates were immersed successively in basis solution (10 $g \cdot L^{-1}$ NaOH, 20 $g \cdot L^{-1}$ Na_2CO_3, 5 $g \cdot L^{-1}$ Na_3PO_4) at 60 °C for 5 min, acid solution (10 wt.% H_2SO_4) at 30 °C for 1 min, and activating solution (0.1 $g \cdot L^{-1}$ $PdCl_2$ and 10 $ml \cdot L^{-1}$ HCl) at 30 °C for 1 min. After each of these pre-treatment steps, the substrates were rinsed with distilled water and dried under air flow. Then electroless nickel (EN) plating was performed in the EG plating solution (25 $g \cdot L^{-1}$ $NiSO_4 \cdot 6H_2O$, 30 $g \cdot L^{-1}$ $NaH_2PO_2 \cdot H_2O$, 30 $g \cdot L^{-1}$ sodium citrate, 20 $g \cdot L^{-1}$ CH_3COONa, pH 4.5) at 80 °C for 40 min to obtain the Ni-P coatings (P content of 8.0 wt.%) with the thickness of about 3.5 ~ 4.0 μm. Finally, the Ni-P plated Cu sheets were immersed in the Au plating solution to perform IG deposition. The plating solution composition and the operating conditions for IG deposition are listed in Table 1.

2.2. Characterization of the coatings

The deposition rate was defined as the increased thickness of as-plated coatings in unit time ($nm \cdot min^{-1}$). The thickness of Au coating deposited on Cu/Ni-P surface was measured by energy-dispersive X-ray spectroscopy (EDX, EDX1800, Skyray Instrument Co. Ltd., China). Every sample was tested for 9 random points, and every point was replicated for 3 times. The deposition rate is the average results of 9 points.

Structure and morphology of coatings 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 $6° \cdot min^{-1}$, 2θ from 20° to 80°), and scanning electron microscope (SEM, JSM-6360LA, Japan Electron Optics Laboratory Co. Ltd., Japan), respectively.

Solderability of coatings was determined by a wetting balance using solderability tester (SKC-8H, Shanghai Runpu Inspection Equipment Co. Ltd., China). Sn63Pb37 was used as eutectic solder, and the liquid solder was kept at a constant temperature of 235 °C during tests [28].

Corrosion resistance was performed by potentiodynamic polarization technique on an electrochemical analyzer (RST-5200 F, Suzhou Risetest Instrument Co. Ltd, Suzhou, China) in 3.5 wt.% NaCl solution. The counter and the reference electrodes were platinum wire electrode and saturated calomel electrode (SCE), respectively. For comparison, the working electrode was Ni-P/Au coated and Ni-P coated Cu sheets. The area of working electrode exposed to solution was 1 cm^2 and the scan rate was 10 $mV \cdot s^{-1}$.

2.3. Spectroscopic measurement

UV-vis optical absorption spectra were measured on a spectrophotometer (UV mini 1240, Shimadzu Corporation, Japan), controlled by UV Data Manager software version 1.02, for the investigation on the lifetime of Au(III)-ChCl plating solution. Measurements were taken from 600 to 200 nm in 1 mm and 0.01 mm quartz cuvettes (suprasil) at a scan speed of 400 $nm \cdot min^{-1}$ with a spectral bandwidth of 5 nm. Because the absorbance of the original solution was beyond the detection range, the plating solution was diluted to one hundred times with distilled water before testing.

Table 1
Composition and operating conditions of IG deposition solution.

Chemicals	Concentration ($g \cdot L^{-1}$)	Condition
$HAuCl_4$°$4H_2O$	1.0	
ChCl	500	
pH (HCl)		2.0
Temperature		80 °C
Immersion time		5 min

3. Results and discussion

3.1. The effects of solution components and conditions on IG deposition and kinetics analysis

Here, we developed a simple IG plating solution containing only $HAuCl_4$, ChCl, H_2O, and pH adjuster. Thus, the influences of the plating parameters, such as the concentrations of $HAuCl_4$ and ChCl, the pH value, and the plating temperature on IG deposition were investigated. The relationships between the deposition rate and the plating parameters are shown in Fig. 1. The insert parts of Fig. 1(a), (b), and (d) are the logarithm of concentration of $HAuCl_4$ versus the logarithm of the deposition rate, the logarithm of concentration of ChCl versus the logarithm of the deposition rate, and the inverse of thermodynamic temperature versus the logarithm of the deposition rate, respectively.

As is shown in Fig. 1(a), the deposition rate increased dramatically with the increase of $HAuCl_4$ concentration. This could be explained by the fact that with the increase of $HAuCl_4$ concentration, the redox potential of Au moved to more positive values. Consequently, the speed of the reaction was promoted and rate of Au deposited on unit area increased. During the experiments, IG deposition could hardly take place with $HAuCl_4$ concentration lower than 0.5 $g \cdot L^{-1}$. In contrast, Au coatings would peel off from the substrate with $HAuCl_4$ concentration higher than 2.0 $g \cdot L^{-1}$. Considering the proper deposition rate and the amount of $HAuCl_4$, we choose 1.0 $g \cdot L^{-1}$ as the optimal concentration for $HAuCl_4$ in this work.

As can be seen in Fig. 1(b), the deposition rate decreased with the increase of ChCl concentration. The possible reasons could be attributed to two aspects. On the one hand, the viscosity of the Au(III)-ChCl solution becomes higher with the increase concentration of ChCl [29]. As a result, the ability of Au(III) migration decreased. Au(III) was less likely to be reduced on Ni-P surface causing the decline in the deposition rate. On the other hand, ChCl might perform as a ligand to form complex with Au(III). The supposition was verified by UV absorbance curves of $HAuCl_4$ in ChCl-H_2O solutions showing an increasing absorbance with the increasing ChCl concentration at the wavelength of 226 nm and 313 nm (not shown here). Coordination equilibrium between ChCl and Au(III) is speculated. A decrease in ChCl concentration led to a shift in equilibrium toward an increase of free Au(III) promoting the effective collision between Ni and Au(III) resulting in IG deposition. During the experiments, the Au coatings fell off easily when ChCl concentration was lower than 300 $g \cdot L^{-1}$. However, the deposition rate was too slow when ChCl concentration was higher than 700 $g \cdot L^{-1}$. Therefore, a concentration of 500 $g \cdot L^{-1}$ was chosen as the optimal concentration for ChCl.

Fig. 1(c) shows that there was less influence of pH value or the hydrogen ion (H^+) concentration on the deposition rate. It could be deduced that H^+ did not participate in the reaction. However, it surely affected the appearance of Au coatings. When pH value was above 2.5, black spots appeared on the coating. Here, pH values ranging from 0.5 to 2.5 were all suitable for IG deposition on Ni-P coating to get bright Au coatings maintaining stable solutions. In the following studies, pH value was set at 2.0.

Fig. 1(d) shows the relationship between the deposition rate and the plating temperature. The deposition rate increased with the increasing temperature, which was caused by two factors. One was the effective collision between Ni and Au(III) increased rapidly by the accelerated speed of molecular motion at higher temperature. Second, it was the migration of ions that was speeded up by the lowered viscosity of the ChCl solution resulted from the increased temperature [30]. The brightness of Au coatings increased with increase in plating temperature and appeared brightest at 80 °C to 90 °C. Taking the deposition rate,

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