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Electrochemical behavior of gold (III) in cyanide-free bath with 5,5'-dimethylhydantoin as complexing agent

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ABSTRACT

Gold electrodeposits are prepared in a cyanide-free bath with 5,5'-dimethylhydantoin (DMH) as complexing agent. The electrochemical behavior of the electrodeposition is then investigated together with the influence of additive A (pyridyl-compound) as an additive on the nucleation and growth of gold using electrochemical techniques on gold working electrode at different temperatures. Cyclic voltammogram consists of a single cathodic reduction wave at -0.62 V which corresponds to the reduction of Au(III) to Au without anodic oxidation wave observed. The diffusion coefficient of Au(III) in the bath is found to be $\sim 10^{-6}$ cm²/s and the energy of activation (43 kJ/mol) is deduced from the cyclic voltammograms at different temperatures. The nucleation and growth of gold on gold working electrode is investigated by chronoamperometry. The progressive nucleation mechanism is found for gold deposition using Scharifker–Hills' model with three-dimensional (3D) diffusion-controlled growth nucleation. The introduction of the additive A does not influence this mechanism. The gold electrodeposits are characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and cathodic polarization measurements. Experimental results indicate that additive A increases the cathodic polarization of bath, refines the grains of electrodeposit and changes the preferred orientation of electrodeposit from [1 1] direction to [2 0 0] direction.

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1. Introduction

Gold electrodeposits have a wide application in microelectronic, optoelectronic and microsystem industry [1-3]. They can be generally classified into soft and hard gold. Soft gold electrodeposition is usually done using cyanide or sulfite electrolytes, but these electrolytes have their respective inherent problems of poisonousness, process incompatibility or instability [4-6]. Commercial sulfite baths have solved the stability problem by being operated at pH 8 or incorporating proprietary stabilizing additives such as ethylenediamine, which makes it possible to operate the bath in a lower pH range of 5-8. More recently, 2,2'-bipyridine has been used to stabilize sulfite bath, possibly because it forms a complex with Au⁺. Furthermore, polyamine, such as ethyldiamine, and aromatic nitro compound such as nitrobenzene have also been simultaneously used to stabilize Au(I)-sulfite complex so that the bath can be operated at an even lower pH of 4-6.5 [6-8]. A mixture of sulfite and thiosulfate is less susceptible to degradation, but sulfur contamination is inevitable, significantly degrades gold deposit in terms of softness [9,10]. This is why researchers are now searching for alternative cyanide-free gold plating electrolytes, and gold plating bath with $C_5H_8N_2O_2$ has been proposed [11]. Hydantoins, known as drugs and agrochemicals, are weak acids $(pK_a = 9.1)$ and can complex a number of metal ions such as Cu⁺ and Ag⁺. The ionization equation of DMH follows: $DMH = H^+ + DMH^$ and researchers have found that gold (III) forms a four-coordinate light-stable complex with DMH⁻ [12]. However, the main problem encountered in replacing gold cyanide baths is the very high stability of gold-cyanide complex. So in our present investigation, DMH is also used as complexing agent to get a cyanide-free gold plating bath with high stability. The properties and quality of electrodeposits in this kind of electrolyte are directly determined by the nucleation and growth mechanism of electrocrystallization. Therefore, it is of great significance to study the nucleation and growth during gold electrochemical deposition for the purpose of improving the properties and quality of electrodeposit.

To the best of our knowledge, not much work has been done on the electroplating mechanism of gold electrodeposit. Liew et al. [13] used standard rotating disk and cyclic voltammetry to investigate the electrochemical behavior of gold thiosulfate–sulfite system, and found that the diffusion coefficient of the mixed ligand electrolyte is 1.2×10^{-6} cm²/s, which lies somewhere between the diffusivities of gold thiosulfate and gold sulfite electrolytes. The electrodeposition of gold from a new electroplating system for

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gold with iodide and thiosulfate has been studied using rotating disk voltammetry [14]. The cathodic electron transfer is slow, with a transfer coefficient of $\alpha = 0.76 \pm 0.02$. Chronoamperometry based on current transient has been used to determine the nucleation mechanism and nucleation rate [15]. The mechanism of gold alloy electrodeposition from acid baths in the presence of a trace amount of Tl⁺ is investigated using chronoamperometry [16]. Analysis of chronoamperometric measurements shows a progressive and instantaneous nucleation and growth mechanism at low and high cathodic potentials, respectively. As a fundamental research, it is meaningful to investigate the nucleation and growth mechanism of gold deposition in cyanide-free bath and the influence of additive on the electrodeposition process.

Therefore, DMH is selected in this paper as complexing agent, and pyridyl-compound as additive in a cyanide-free gold electroplating bath. The electrodeposition behavior, rate controlling step, and the nucleus growth mechanism of gold on gold electrode are investigated using cyclic voltammograms (CV) and chronoamperometry (CA) techniques. The influence of additive on the nucleation mechanism and the properties of the gold electrodeposits are also discussed.

2. Experimental

2.1. Gold electrodeposition

Electrolyte was prepared by dissolving salts in distilled water. The basic gold electrodeposition bath was: 0.03 mol/L HAuCl4, 0.5 mol/L DMH, 0.2 mol/L K₃PO₄, 3.5 mmol/L additive A and calculated amount of KH₂PO₄ solution (the solution was prepared by first dissolving 1 mol/L KH₂PO₄ in deionized water. Then, it was added to gold plating bath in some volume ratio for the pH maintaining and used as a buffer). It contained HAuCl4 as the main salt, DMH as the complexing agent and additive A as the main brightener. The pH of the bath was adjusted to 9.0 using 30% KOH. The HAuCl₄ was fully complexed with DMH and formed a DMH-gold ligand through the following reaction:

$$[\operatorname{AuCl}_4]^- + 4\operatorname{DMH}^- \rightleftharpoons [\operatorname{Au}(\operatorname{DMH})_4]^- + 4\operatorname{Cl}^- \tag{1}$$

All the chemicals used in this work were analytical grade. The bath under investigation was modified according to different experimental requirements. The bath components were modified while pH was kept constant.

Gold electroplating experiments were conducted under galvanostatic conditions $(1 \text{ A/dm}^2, 323 \text{ K} \text{ and } 10 \text{ min})$ in different baths with mild agitation to study the surface morphology and crystal structure. A cell with an anode of $2.5 \text{ cm} \times 2.5 \text{ cm}$ platinized titanium sheet and a copper sheet cathode of $1 \text{ cm} \times 1 \text{ cm}$ was employed. The copper sheet samples of $1 \text{ cm} \times 1 \text{ cm}$ were Ni electroplated followed by gold electroplating. The purpose of Ni undercoat deposition was to prevent the diffusion of Cu into Au top layer. Prior to Ni electrodeposition, samples were polished with emery silicon carbide paper ($8-4 \mu \text{m}$ grain size), thoroughly cleaned using a solution of HCl and distilled water with the volume fraction of 1:1, and rinsed with distilled water.

2.2. Electrochemical evaluation

Electrochemical evaluation was carried out using cyclic voltammetry, chronoamperometry and cathodic polarisation measurements in a three-electrode glass cell on a GAMRY Reference 600 electrochemical workstation. A saturated calomel electrode (SCE) was used as the reference electrode and a platinum foil (99.99%, 2 cm²) was employed as the counter electrode. A gold electrode with a working surface of 0.07 cm² (Ø 0.3 cm) was used as the working electrode. Before every electrochemical measurement,

Fig. 1. Cyclic voltammograms of $[Au(DMH)_4]^-$ in electrolyte with additive A recorded on gold electrode with T=318 K at scan rate 50 mV/s.

the working electrode was polished successively with increasingly finer grades of diamond polish powder, and finally to a mirror finish with aqueous slurry of 0.15 μ m alumina, rinsed with distilled water and thoroughly dried before each experiment. After the electrochemical experiments the electrode was rinsed with distilled water and dried with an air blast at room temperature. For the electrochemical experiments, the bath temperature was kept at 303–323 K by a thermostat. The cathodic polarisation curves were recorded in a potential range from the open-circuit potential to -1.5 V (vs. SCE) at a scanning rate of 1 mV/s.

2.3. Characterizations of gold electrodeposits

Gold electrodeposits were characterized by SEM and XRD. SEM observations were performed under high vacuum by field emission scanning electron microscopy (FE-SEM, Hitachi S4700) at 25 kV working voltage. XRD analysis was done using a D/max-3C X-ray diffractometer at a scanning rate of 0.02° /s with Cu K α radiation.

3. Results and discussion

3.1. Analysis of cyclic voltammograms

Transient cyclic voltammogram is an efficacious method used to study the reactions on the working electrode surface. In order to study the cathode electrode reaction in the electrodeposition process of gold, electrochemical study was carried out using cyclic voltammograms in a conventional three-electrode system. As shown in Fig. 1, the voltammogram of $[Au(DMH)_4]^-$ in electrolyte exhibits one prominent reduction wave and an area of hydrogen evolution. No anodic dissolution peak but an area of oxygen evolution can be observed on the positive scan. It can be seen from Fig. 1 that onset potential (Eonset) of Au deposition on gold electrode occurs immediately at $E_{onset} = -0.45$ V. A cathodic reduction peak can be observed at -0.62 V, which corresponds to the reduction of Au(III) to Au. The scanning area beyond -1.1 V (vs. SCE) on the cathodic side of cyclic voltammogram may be due to hydrogen evolution. During the potential scanning from -0.85 V to -0.59 V, the inverse current is smaller than the direct one, which indicates that the deposition process is controlled by mass transport [17]. A "hysteresis loop" appears in the range of potential from -0.59 V to -0.19 V, which means the three-dimensional growth of nuclei of gold occurs on the gold electrode [18]. The scanning area beyond





Fig. 2. Cyclic voltammograms of $[Au(DMH)_4]^-$ in gold plating bath with additive A recording on gold electrode at different scan rates at 323 K: (a) 10 mV/s; (b) 20 mV/s; (c) 40 mV/s; (d) 60 mV/s; (e) 80 mV/s; (f) 100 mV/s.

+1.1 V (vs. SCE) on the anodic side of the cyclic voltammogram may be due to oxygen evolution. No anodic dissolution peak is observed on the positive scan, which is attributed to the gold electrodeposit cannot be dissolved in DMH bath.

As shown in Fig. 2, all the CV results are obtained on the gold electrode covered with a thin gold film, and there is no "hysteresis loop" on the cyclic voltammograms. Cathodic peak current I_p increases and cathodic peak potential E_p shifts negatively with the increase of scan rate, which means the reduction of Au(III) on the gold electrode is not reversible [19]. It can be seen from Fig. 3 that peak potential E_p plotted vs. log ν gives a linear response at all the temperatures, which is characteristic to an irreversible electrode process for the reduction of Au(III). Charge transfer coefficient (α , 0.1 $\leq \alpha \leq 0.9$), a measure of symmetry barrier in non-reversible (quasi- and irreversible) electrode processes, can be determined from the shift in E_p per 10-fold increase in scan rate as shown below:

$$\Delta E_{\rm p} = \frac{1.15RT}{(\alpha n_{\alpha}F)} \tag{2}$$



Fig. 3. Variation of cathodic peak potential E_p vs. log ν for reduction of Au(III) at different temperatures.



Fig. 4. Cathodic peak currents $I_{\rm p}$ vs. $\nu^{1/2}$ for reduction of Au(III) at different temperatures.

where n_{α} is the number of electrons involved in the rate determining step, *R* is the gas constant, *F* is Faraday constant, and *T* is the absolute temperature in K [19]. The value of αn_{α} is 0.63 for the measurements from Eq. (2) at 323 K.

When electrode reaction is controlled by diffusion processes, the relationship between peak current I_p and scan rate (ν) of CV curves can be expressed as shown below [20]:

$$I_{\rm p} = 0.4958nF^{3/2}CAD_0^{1/2} \left(\frac{\alpha n_{\alpha}}{RT}\right)^{1/2} \nu^{1/2}$$
(3)

where A is the electrode area in cm^2 (0.07 cm^2), C is the gold concentration in mol/cm³, D_0 is the diffusion coefficient in cm²/s, *n* is the number of exchanged electrons, and v is the potential sweep rate in V/s. As shown in Eq. (3), there is a linear relationship between I_p and $\nu^{1/2}$. It can be seen from Fig. 4 that the plots of $I_{\rm p}$ against $\nu^{1/2}$ at all the temperatures are linear and show that the electrodeposition of gold is controlled by the diffusion process [21]. So, from the above discussion it can be concluded that the reduction of Au(III) to Au on a gold electrode is an irreversible and diffusion-controlled process. However, the qualitative and quantitative nature of gold involved in the reduction is not known exactly in the present study. Therefore, the total concentration of gold taken for voltammetric measurements is substituted in Eq. (3) to obtain an approximate diffusion coefficient and this method can also be found in [19]. It can be seen from Table 1 that the diffusion coefficient mounts with an increase in the temperature and the values are of the same order as the diffusion coefficient of gold sulfite electrolyte reported in [13].

The diffusion coefficient is related to the temperature by Arrhenius equation shown in Eq. (4):

$$D = Ae^{(-E_a/RT)} \tag{4}$$

Table 1

Diffusion coefficients of Au(III) in electrolyte obtained from cyclic voltammetry at various temperatures.

Temperature (K)	Diffusion coefficient of Au(III) in electrolyte, $D \times 10^{-6} \text{ cm}^2/\text{s}$
303	2.30
313	4.27
323	6.55

 $[Au] = 0.03 \text{ M}; A = 0.07 \text{ cm}^2.$



Fig. 5. Comparison of cyclic voltammograms of $[Au(DMH)_4]^-$ in gold plating bath with additive A at various temperatures and scan rate: 60 mV/s.

where *A* is the pre-exponential factor, and E_a is the energy of activation, which can be obtained from the plot of $\ln D$ against 1/T. It is seen from Fig. 5 that I_p increases and the peak potential E_p shifts towards positive potential with an increase in the temperature. It can be seen from Fig. 6 that the energy of activation for the diffusion of Au(III) is 43 kJ/mol.

3.2. Nucleation mechanism

Chronoamperometry is an electrochemical technique commonly used to study the mechanism of electrodeposition of metals. During chronoamperometry experiments, current–time transients are recorded as the potential is stepped down from the open–circuit potential (OCP) to the potential at which the electrodeposition of metals occurs [22,23]. A common feature of *I*–*t* curves is that in a very short period of time during the potential step, due to the double-layer charging, the current increases rapidly and diminishes further. Then the current increases again and reaches its maximum, following by subsequent diminution because of the nucleation and the growth of new phase [24]. Due to the charging of double layer and the formation of first nuclei on the gold working electrode, the



Fig. 6. Arrhenius plot for diffusion of Au(III) in gold plating bath with additive A.



Fig. 7. Current-time transients resulting from chronoamperometry experiments recorded for Au(III) reduction in gold plating bath without additive A on gold working electrode at 323 K and applied overpotentials of (a) -0.84 V; (b) -0.86 V; (c) -0.88 V; (d) -0.90 V.

transients shown in Fig. 7 are characterized by an initial decrease of negative current which is followed by an increase in the negative current which leads to maximum value (i_m) at time t_m . This can be attributed to the three-dimensional growth of gold over the nuclei. Maximum diffusion current (i_m) increases with negative step potential at the initial stage of gold electrocrystallization. Therefore the shape of potentiostatic current density transients is a typical 3D growth nucleation process [25]. The transient current reaches its maximum value followed by a negative current decay with time; finally the current becomes a constant.

Much work has been done on the modelling of nucleus growth mechanisms, and the most commonly accepted is Scharifker–Hills' models [26], one instantaneous nucleus growth mechanism and one progressive nucleus growth mechanism. One convenient method to examine the experimental results is to obtain a linear fit for the ascending current with respect to $t^{1/2}$ for instantaneous nucleation and $t^{3/2}$ for progressive nucleation. In our case, it is obvious that a good linear relationship of $I-t^{3/2}$ curves is shown in Fig. 8. Therefore, it is reasonable that the progressive nucleation control for the growth of gold from additive-free bath on working electrode takes place. All current transients can be presented in a non-dimensional form by plotting the normalized current $(I/I_m)^2$



Fig. 8. *I* against $t^{3/2}$ from rising portion of current transient at different applied overpotentials: (a) -0.84 V; (b) -0.86 V; (c) -0.88 V; (d) -0.90 V.



Fig. 9. Comparison of experimental data obtained from chronocurrent transients of gold electrodeposition process from bath without additive A with instantaneous and progressive nucleation models for overpotentials: (a) -0.84 V; (b) -0.86 V; (c) -0.88 V; (d) -0.90 V.

vs. time t/t_m . The theoretical transients of instantaneous and progressive nucleus growth, with 3D under diffusion control, are given by [26]:For instantaneous nucleation,

$$\left(\frac{I}{I_{\rm m}}\right)^2 = 1.9542 \left(\frac{t}{t_{\rm m}}\right)^{-1} \left\{1 - \exp\left[-1.2564\left(\frac{t}{t_{\rm m}}\right)\right]\right\}^2 \tag{5}$$

And for progressive nucleation,

$$\left(\frac{I}{I_{\rm m}}\right)^2 = 1.2254 \left(\frac{t}{t_{\rm m}}\right)^{-1} \left\{1 - \exp\left[-2.3367 \left(\frac{t}{t_{\rm m}}\right)^2\right]\right\}^2 \tag{6}$$

where *I* and *t* are the current density and time, respectively, and I_m and t_m are the maximum values of current transients. Our experimental results are compared with the theoretical dimensionless current–time transients derived from progressive and instantaneous nucleation growth models. It can be seen from Fig. 9 that the experimental results show a good agreement with the theoretical curves for progressive nucleation under different overpotentials, which suggests the electrochemical deposition of gold is followed by the progressive nucleation. It can be seen from Fig. 10 that the nucleation mode of gold is not changed by the additive. The deposition of gold is still followed by the progressive nucleation in the presence of additive. Therefore, it can be concluded from the CV and CA results that the reduction of Au(III) on a gold electrode corresponds to a three-dimensional progressive nucleation growth process with diffusion-controlled growth.

3.3. Effect of additive A on properties of gold electrodeposits

Bright gold electrodeposits are obtained from the bath modified by adding additive A 3.5 mmol/L. To investigate the influences of this additive in the bath, the cathodic polarization was studied with or without additive in electrolyte. As shown in Fig. 11, there is a single wave for potentials higher than -1.2 V vs. SCE in the gold plating region, and so, it is likely that the cathode reaction is a single step process [27]:

$$Au(III) + 3e^{-} \rightarrow Au(0) \tag{7}$$

The suitable range for gold plating in the baths is then about -0.65 to -1.2 V vs. SCE. At a potential higher than -0.65 V vs. SCE, the gold plating rate is very low. Hydrogen evolution also begins at a potential lower than -1.2 V vs. SCE, and results in a lower current



Fig. 10. Comparison of experimental data obtained from chronocurrent transients of gold electrodeposition process from bath with additive A with instantaneous and progressive nucleation models for overpotentials: (a) -0.88 V; (b) -0.90 V; (c) -0.92 V.

efficiency. Compared with the curve of the bath without additive shown in Fig. 11a, cathodic polarization is improved by adding additive as shown in Fig. 11b. The adsorption of additives at the interface influences the nucleation overpotential [28]. As shown in Fig. 11, a higher overpotential is required for the gold deposition in the bath with additive A, may be due to the strong adsorption of additive A at the cathode. Fine gold grains would be obtained at such a high overpotential as shown in Fig. 12.

It can be seen from Fig. 12a and b that in the presence of additive A, the grains become smaller. Deposit a is golden-brown in color and has large gold grains of $0.5-1 \,\mu$ m in diameter as shown in Fig. 12a. Compared with deposit a, the addition of additive A to the bath causes an increase in cathodic polarization and results in smaller grains in deposit b. As shown in Fig. 12b, gold deposits are bright golden-brown in color and the structure of deposit b is different from that of deposit a. Large gold grains disappear, while deposit b has some smaller gold grains of $0.1-0.5 \,\mu$ m in diameter and some crystal bulks (less than 1 μ m in diameter). In general, a higher cathodic polarization accelerates the growth of finer grains,



Fig. 11. Effect of different concentrations of additive A added to gold plating bath: (a) 0 mmol/L; (b) 3.5 mmol/L.



Fig. 12. Effect of concentrations of additive A added to gold plating bath on surface morphology of gold deposits: (a) 0 mmol/L; (b) 3.5 mmol/L; (c) 7 mmol/L.

so the grains of deposit b are finer than those of deposit a when the cathodic polarization of bath in the presence of additive A is larger than that of bath in the absence of additive A in this case. It can be seen from Fig. 12b and c that the size of grains in deposit is not markedly affected and does not change significantly with the increasing concentration of additive A in the bath.

As shown in Fig. 13, the five obvious peaks at 2θ values of 38.3° , 44.8° , 64.7° , 77.7° and 81.9° can be indexed as the $1\,1\,1,2\,0\,0,2\,2\,0$, $3\,1\,1$ and $2\,2\,2$ peaks of the fcc structure of Au, respectively. The relative amplitude of Au diffraction peaks increases with the addition of additive A to the bath. As shown in Fig. 13a, due to the thinness or porosity of gold deposits, the peak at 52° can be indexed as peak $2\,0\,0$ of Ni element in Cu substrate with Ni plated. The diffraction peaks intensity which corresponds to the $[1\,1\,1]$ plane is larger than other peaks, which indicates that the electrodeposit has a well preferred orientation along $[1\,1\,1]$ direction in the absence of additive A in bath. As shown in Fig. 13b, the electrodeposit has a well preferred orientation along $[2\,0\,0]$ direction in the presence of additive A in bath.

3.4. Studies on aged bath

In order to induce accelerated aging, the fresh gold plating bath is used extensively in an industrial process (for over 1 week) during which period the equivalent gold is added according the consumption. The fresh bath is transparent with a faint yellowish color, and the aged bath has no discernible change in the color and no



Fig. 13. Effect of concentrations of additive A added to gold plating bath: (a) 0 mmol/L; (b) 3.5 mmol/L.

precipitates or turbidity is detected in the aged bath. In order to determine if there is any significant change in the electrochemical properties of fresh and aged baths, cyclic voltammograms are obtained at the scan rate of 20 mV/s on the gold working electrode

at 318 K. And the two CV curves exhibit similar characteristics just as curve b in Fig. 2. Then, it can be concluded that the aged bath is stable.

4. Conclusions

The electrochemical nucleation of gold from a cyanidefree stable bath containing 0.03 mol/L HAuCl4, 0.5 mol/L DMH, 0.2 mol/LK₃PO₄, 3.5 mmol/L additive A and calculated amount of KH₂PO₄ on a gold working electrode is investigated. The cyclic voltammograms of Au(III) in bath on gold working electrode at different scan rates and different temperatures are recorded. The prominent reduction wave observed at -0.62 V in cyclic voltammogram is due to the reduction of [Au(DMH)₄]⁻ to gold. No anodic wave is observed during the forward scan towards positive potentials, which means the absence of Au dissolution in the bath. Influence of the scan rate on Au(III) reduction in bath follows an irreversible diffusion-controlled process on the working electrode. The diffusion coefficient and energy of activation for the reduction of Au(III) is $\sim 10^{-6}$ cm²/s and 43 kJ/mol, respectively. The analysis of current-time transients indicates that the electrodeposition of gold on a gold electrode proceeds via progressive nucleation with 3D diffusion-controlled growth of nuclei, which is not influenced by adding additive. The results of cathodic polarization curves and SEM indicate that additive A exhibits an inhibition effect on the deposition of gold and brighter gold deposits can be obtained from the additive-containing bath. The XRD patterns of gold electrodeposits with or without additive A in electrolyte indicate that additive A also changes the preferred orientation of electrodeposits from [111] direction to [200] direction.

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