Insight into the Mechanism of the Fe-Ni Alloys Co-Deposition from Poly-Nuclear Complexes

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Electrodeposition of the Fe-Ni alloys in the citrate bath was anomalous co-deposition, but normal co-deposition in the citrate-ammonia bath. The Fe and Ni elements were uniformly distributed in the Fe-Ni deposit, which were highly alloyed. These results cannot be simply explained by the hydroxide deposition model or the competitive adsorption of intermediate model, which proposed in literature. A poly-nuclear complex co-deposition mechanism of Fe-Ni alloy was proposed in the above two baths, which can explain better electro-deposition behavior of the Fe-Ni co-deposition and the experimental phenomena we observed. © The Author(s) 2018. Published by ECS. This is an open access article distributed under the terms of the Creative Commons Attribution 4.0 License (CC BY, http://creativecommons.org/licenses/by/4.0/), which permits unrestricted reuse of the work in any medium, provided the original work is properly cited. [DOI: 10.1149/2.044181jes]

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Fe–Ni alloys have many desirable properties, such as low thermal expansion coefficient, good soft magnetic properties and protective properties.1–3 They have been widely used in computer storage devices, magnetic simulators, magnetic shielding, and solar cells, basal,4–7 etc. Many methods are used to prepare Fe-Ni alloys, including mechanical-alloying method,8,9 microwave plasma method10 and electroplating method.11 Where, electroplating method is the most cost-effective to prepare thin strip and thin film on a variety of surfaces with varied geometries. The properties of Fe-Ni alloy are affected greatly by its composition which determined by electrodeposition process. Therefore, it is of great significance to make clear the mechanism of Fe-Ni deposition process.

It has been well known that the deposition behavior of Fe-Ni alloys showed a typical feature of anomalous co-deposition, in which the less noble Fe is preferentially deposited on electrode in comparison with the noble Ni.12–19 But some researchers20–22 reported that anomalous co-deposition of Fe-Ni alloys can be changed to normal co-deposition by adjusting the electroplating conditions, such as the use of reverse pulse electro-deposition,20 or by increasing the plating temperature to 353K,21 or by using ionic liquids in the electrolyte solution.22

The mechanism of Fe-Ni anomalous co-deposition has been studied by many researchers.23–30 The hydroxide deposition model23 and the competitive adsorption of intermediate model30 have been well accepted so far to explain the anomalous co-deposition of Fe-Ni. In hydroxide deposition model,23 it said that noble Ni deposition was strongly suppressed in the presence of Fe(OH)2, which preferentially formed and adsorbed on the cathode. While in competitive adsorption of intermediate model,30 it said that two-step reaction process existed in deposition of Fe-Ni alloy, first, the univalent cation ion of metal hydroxide (FeOH+ and NiOH−) formed and adsorbed on electrode, then they were further reduced to metal alloy. As FeOH+ is more preferentially adsorbed on electrode than NiOH−, Ni deposition was inhibited. This model was confirmed by Baker and West,31 Harris and Clair.25 However, all of these models could not explain the reason why anomalous co-deposition of Fe-Ni alloys changed to normal co-deposition by adjusting the electroplating conditions.

In this paper, we reported normal co-deposition of Fe-Ni alloys in the citrate-ammonia bath and proposed a poly-nuclear complexes co-deposition model in Fe-Ni alloys electrodeposition process. It can explain why Fe-Ni showed anomalous co-deposition in some conditions but normal co-deposition when changed conditions. It is of great significance for the further study of the mechanism of electrodeposition of metal alloys.

Experimental

Electroplating bath.—The blank citrate bath was composed of 0.5M H3BO3, 0.25M Na3Cit•2H2O and 0.1M H3Cit•H2O; the Fe, Ni, Fe-Ni citrate bath contained 0.2M FeSO4•7H2O, 0.2M NiSO4•6H2O, (0.16–0.004M) FeSO4•7H2O and (0.04–0.16M) NiSO4•6H2O respectively. Adding 0.5M NH4H2O into the above baths, the corresponding citrate-ammonia baths were generated. The pH values of all baths were adjusted to 7.5 with 20% NaOH and 10% H2SO4 solutions. All plating baths were prepared using deionized water and analytical grade reagents that were used as received without further purification.

Electroplating, composition and microstructure of Fe-Ni alloy.—Fe-Ni alloys were electroplated on 316 stainless steel plates for 30 min at 7 A/dm2, 75°C in the baths. Under the above experimental conditions, the smooth and good adhesion coatings can be obtained. The peeled deposit was dissolved in HCl (1:1) + H2O2 solution. The content of Fe and Ni were determined by WFX-1C atomic absorption spectrophotometer. The morphology of the Fe-Ni alloy were characterized by an environmental SEM FEI QUANTA 200 and EDAX attachment produced by EDAX. The X-ray diffraction patterns for Fe-Ni alloys were recorded using a D/max-rA diffractometer with nickel-filtered and graphite-monochromatized Cu Kα radiation operating at 50kV and 100 mA over 20 range of 5–80°.

EQCM measurement.—For the EQCM, an AT-cut 9 MHz gold-coated electrode quartz crystal(area of 0.29cm2) was used as working electrode, an Ag/AgCl electrode (saturated KCl) served as the reference electrode and all potentials reported versus this scale, and a platinum wire served as the counter electrode.

Mass changes during deposition and dissolution result in frequency shift which can be described by the Sauerbrey’s equation:33

\[
\Delta f = \frac{-2f_0^2 \Delta m}{A \sqrt{\rho \kappa}} \tag{1}
\]

Where, \(\Delta f\) is the resonant oscillator frequency shift of the quartz crystal which caused by mass change (Hz); \(f_0\) is the resonant frequency of the quartz crystal (Hz); \(\Delta m\): the mass change; A: the effective working area of quartz crystal (cm²); \(\mu_s\) is the shear modulus of quartz crystal (GPa); \(\rho\): the density of quartz. For the same quartz, \(f_0\), A, \(\mu_s\) and \(\rho\) are constant. So

\[
\Delta f = -K \Delta m \tag{2}
\]

K is constant for the given quartz crystal.

The resonant frequency was measured using an HP4395A impedance analyzer and the mass change was calculated according to Equation 2. Electrochemical results were performed using CHI600A  

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Results and Discussion

The anomalous and normal co-deposition behavior of Fe-Ni alloys.—In the study process of Fe-Ni electrodeposition, we found that Fe-Ni alloy deposited in the citrate bath exhibited anomalous co-deposition, but in the citrate-ammonia bath, it showed normal co-deposition. Fig. 1 shows the Fe and Ni content in Fe-Ni alloys obtained by electrodeposition in the citrate and citrate-ammonia baths. In the citrate baths, as the content of Ni in the deposits was less than that in the electrolytes, it was anomalous co-deposition. In the citrate-ammonia baths, as the content of Ni in the deposits was greater than that in the electrolyte, it was normal co-deposition.

Morphology, composition, and microstructure of the Fe-Ni deposit.—Fig. 2 shows the morphology of Fe59.091Ni40.9 alloy obtained in the citrate bath (0.1M FeSO4 and 0.1M NiSO4). 13μm thickness of Fe-Ni deposit was smooth, no crack and pit. It was tightly adhered to the substrate without gaps.

FeNi3 (XRD JCPDS data file No. 38-0419) phase was observed in the citrate-ammonia bath. No peaks of pure Fe or pure Ni phases were observed, it means Fe-Ni deposits were highly alloyed. In Fig. 3A, the 2θ angles of Fe59.1Ni40.9 were 43.3°, 50.3° and 74.6°. In Fig. 3B, the 2θ angles of Fe18.7Ni81.3 were 43.8°, 51.0° and 75.6°. These 2θ angles changes obeyed Vegard’s law.

Study of Fe-Ni electro-deposition behavior by EQCM.—EQCM technique was used to study electrochemical behavior and mass changes of Fe-Ni deposition process in different electroplating baths. The electro-deposited Fe was completely dissolved into electrolyte during the anodic stripping process, so the proportionality coefficient K in Equation 2 can be determined by the deposition and dissolution process of Fe. According to the measurement, the proportionality coefficient K value was 1.18ng·Hz−1. The mass changes calculated from the frequency shift according to Equation 2. The partial current densities of Fe, Ni and Fe-Ni deposits can be derived via Faraday’s law from the deposition rate (dm/dt). For the alloy deposition, the composition changes during the potential sweep and could not be determined. We reckon Fe59.9Ni40.1 in the citrate bath and Fe18.7Ni81.7 in the citrate-ammonia bath according to the results of Fig. 1.

The mass changes, total current densities and Fe, Ni and Fe-Ni alloy partial current densities versus scanning potential in the citrate and citrate-ammonia electrolyte baths are shown in Fig. 4A-(I, II, III), Fig. 4B-(I, II, III) and Figs. 4C-(I, II, III) respectively, where curve a and b are from the citrate bath and curve c from the citrate-ammonia bath. The insert graphs in Figs. 4A, 4B, 4C-I are the local derivative curves of total and partial current densities-voltage curves.

In Fig. 4A-I, the maximum amount of the Fe deposits were 22.80μg·cm−2 in the citrate bath and 59.53μg·cm−2 in the citrate-ammonia bath. Obviously, the amount of Fe deposit obtained in the citrate-ammonia bath was higher than that in the citrate bath, which meant ammonia accelerated the deposition of Fe. The same results were obtained by Osseo-Asare.34 When swept back to −0.76 V, the Fe deposits began to dissolve in both baths. The mass change was close to zero when the potential swept back to −0.558V in the citrate bath and to −0.385V in the citrate-ammonia bath, which meant Fe deposits had been completely dissolved in the baths.

From the insert graphs of Fig. 4A-II and Fig. 4A-III, in the Fe citrate bath, the total current density increased at −0.98V and the Fe partial current density increased at −1.10V; and in the Fe citrate-ammonia bath, the total current density increased at −0.99V and the Fe partial current density increased at −1.10 V. The potential difference in Fig. 4A-II and Fig. 4A-III indicated hydrogen evolution or other side reaction occurred. The current efficiency of Fe deposition were 4.43% in the citrate bath and 5.71% in the citrate-ammonia bath. As the deposition potential of Fe did not change in both electrolytes, the deposition mechanism of Fe in both baths may not change. The
The plating velocity order was: Fe<Fe-Ni<Ni. While in the citrate bath, the plating rate order was: Ni<Fe-Ni<Fe. The current efficiency of Fe, Ni, Fe-Ni deposition were improved by adding ammonia. The ranking of amplitude increases was Ni>Fe-Ni>Fe. The change of deposition potential of Ni and Fe-Ni in both baths means the change of deposition processes. Although the extent of the positive shift of potential for side reaction occurrence was larger than that of the Fe-Ni deposition, the current efficiency of Fe-Ni deposition was still greatly improved. This means the reduced complex must be electro-active.

**UV-visible absorption spectroscopy of different electrolyte baths.**—Fig. 5 shows the UV-visible absorption spectra of the various baths. In Fig. 5A, in the citrate bath, when increasing the concentration of Ni (II), the absorption intensity increased and the absorption peak gradually blue shifted (comparing curve b and curve g, the absorption peak blue shifted about 26nm, 684nm→658nm). The shift of the absorption peak indicated that, in the Fe-Ni bath, Fe (II) and Ni (II) did not exist as a separate complex but formed new complexes. In the Fe citrate bath, the predominant species was the \(\text{Fe}_x\text{Ni}_y(\text{cit})_3(\text{OH})_4\) species, which could be calculated by software Hydra/Medusa (see Fig. 6). In the Ni citrate bath, a poly-nuclear complex of the composition \(\text{Ni}_x(\text{cit})_y(\text{OH})_2\) formed in the weakly alkaline solutions. The shift of the absorption peak in Fig. 6A showed that Fe(II) and Ni(II) in the Fe-Ni citrate bath interacted to form a new poly-nuclear \(\text{Fe}_x\text{Ni}_y(\text{cit})_3(\text{OH})_4\)^− species.

In Fig. 5B, in the citrate-ammonia bath, similar phenomenon had been observed, with the increasing of the Ni (II) concentration, the absorption peak red shifted about 11 nm (658nm→669nm) and the absorbance increased, which meant that Fe(II) and Ni(II) in the Fe-Ni citrate bath interacted to form a new poly-nuclear \(\text{Fe}_x\text{Ni}_y(\text{cit})_3(\text{OH})_4\)^− species. In the citrate-ammonia bath, the absorption peak red shifted about 11 nm (658nm→669nm) and the absorbance increased, which meant that Fe(II) and Ni(II) in the Fe-Ni citrate bath interacted to form a new poly-nuclear \(\text{Fe}_x\text{Ni}_y(\text{cit})_3(\text{OH})_4\)^− species.

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**Figure 3.** XRD patterns of electrodeposited Fe, Ni and Fe-Ni alloy (A) in the citrate bath and (B) in the citrate-ammonia bath.
Figure 4. Measured mass changes (Δm) (I), cyclic voltammograms (II) and partial current density (III) in different baths: (A) Fe, (B) Ni and (C) Fe-Ni; curve a represent results obtained in the citrate bath and curve b in the citrate-ammonia bath; the insert graphs in Figure (I) are the enlargement of the mass change curves; the insert graphs in Figure (II) show the derivation of the total current density versus potential (di/dE); the insert graphs in Figure (III) display the derivation of the partial current density versus potential (di/dE); total [M(II)] = 0.2 M, in Fe-Ni bath, Fe(II)/Ni(II) = 1, pH 7.5, scan rate 5 mV s⁻¹, the arrow indicates the potential scanning direction.
New poly-nuclear complex deposition mechanism model.—For Fe-Ni alloy, the deposition potential was not equal to single Fe or Ni indicates a new species different from individual Fe and Ni formed and participated the electrochemical reactions. However, the present hydroxide deposition and competitive adsorption of intermediate models didn’t deal with the Fe-Ni alloy deposition potential and also cannot explain the phenomenon that Fe and Ni elements were highly alloyed and uniformly dispersed in the Fe-Ni coatings. A new poly-nuclear complex deposition mechanism model was proposed for the Fe-Ni alloy electro-deposition process.

In the Fe-Ni citrate bath, the following cathodic reactions occurred:

\[ \text{H}_2\text{O} + e^- \rightarrow \text{H}_{\text{ads}} + \text{OH}^- \quad [3] \]

\[ 2\text{H}_{\text{ads}} \rightarrow \text{H}_2 \uparrow \quad [4] \]

\[ [\text{Fe}_{x}\text{Ni}_{y}(\text{cit})_3(\text{OH})_5]^{(x+y=4)} + 8e^- \rightarrow \text{Fe}_{x}\text{Ni}_{y} + 3\text{cit}^{3-} + 4\text{OH}^- \quad [5] \]

The Fe-Ni alloy electro-deposition was obtained through the discharge of the poly-nuclear complex \([\text{Fe}_{x}\text{Ni}_{y}(\text{cit})_3(\text{OH})_5]^{(x+y=4)}\), where the values of \(x\) and \(y\) were related to the concentration of Fe(II) and Ni(II) in the bath, this can be confirmed by the results in Fig. 5A. Because the \(\text{OH}^-\) ion favored association with the \(\text{Fe}^{2+}\) more than with the \(\text{Ni}^{2+}\), the value of \(x/y\) was greater than that of \(c_{\text{Fe}^{2+}}/c_{\text{Ni}^{2+}}\) in the electrolytes, e.g. \(c_{\text{Fe}^{2+}}/c_{\text{Ni}^{2+}} = 0.1\), \(x/y > 1\).

In the Fe-Ni citrate-ammonia bath, the following cathodic reactions occurred:

\[ \text{NH}_4\text{OH} \Rightarrow \text{NH}_4^+ + \text{OH}^- \quad [6] \]

\[ \text{NH}_4^+ + e^- \rightarrow \text{NH}_3 + \text{H}_{\text{ads}} \quad [7] \]

\[ [\text{Fe}_{x}\text{Ni}_{y}(\text{cit})_3(\text{NH}_3)_3]^{(x+y=4)} + 8e^- \rightarrow \text{Fe}_{x}\text{Ni}_{y} + 2\text{cit}^{3-} + 4\text{NH}_3 \quad [8] \]

Here, the Fe-Ni alloy electro-deposition resulted from the discharge of the poly-nuclear complex \([\text{Fe}_{x}\text{Ni}_{y}(\text{cit})_3(\text{NH}_3)_3]^{(x+y=4)}\), where the values of \(x\) and \(y\) were related to the concentration of Fe(II) and Ni(II) in the bath, this can be confirmed by the results in Fig. 5B. Due to the \(\text{NH}_3\) coordinated better with \(\text{Ni}^{2+}\) than with \(\text{Fe}^{2+}\), the value of \(x/y\) was less than that of \(c_{\text{Fe}^{2+}}/c_{\text{Ni}^{2+}}\) in the electrolytes, e.g. \(c_{\text{Fe}^{2+}}/c_{\text{Ni}^{2+}} = 0.1\), \(x/y < 1\).

Generally, the poly-nuclear complex has a high degree of entropy value and high internal energy, and the chemical bond is easily broken, so the Fe-Ni poly-nuclear complexes are electro-active and easy to be reduced. These were confirmed by the experimental results showed in Fig. 4.

In summary, the poly-nuclear complex deposition mechanism model. It was demonstrated that Fe and Ni were simultaneously reduced by cathodic reduction in the ratio of \(x/y\). The content of Fe and Ni in the deposit was determined by the ratios of \(x\) and
y. As the deposition process proceeded, reduced Fe and Ni elements were highly alloyed and uniformly dispersed in the coatings. In the Fe-Ni citrate bath, the stoichiometry ratio \( x/y \) of the poly-nuclear complex \( \left[ Fe_yNi_x(cit)_y(\text{OH})_z \right]^{2-} \) was greater than that of the \( c_{Fe^{II}}/c_{Ni^{II}} \) in the electrolytes, then, exhibited the typical anomalous co-deposition. In the Fe-Ni citrate-ammonia bath, the stoichiometry ratio \( x/y \) of the poly-nuclear complex \( \left[ Fe_yNi_x(cit)_y(\text{NH}_3)_z \right]^{2-} \) was much faster than that in the Fe-Ni ammonia citrate bath, so the electro-deposition rate of the Fe-Ni alloy was determined by the composition of the electrolytes, then, exhibited the typical anomalous co-deposition. In the Fe-Ni citrate bath, the stoichiometry ratio \( x/y \) of the poly-nuclear complex \( \left[ Fe_yNi_x(cit)_y(\text{OH})_z \right]^{2-} \) was with only one negative charge. The latter complex diffused faster to the cathode surface, so the electro-deposition rate of the Fe-Ni alloy in the Fe-Ni citrate-ammonia bath was much faster than that in the Fe-Ni citrate bath.

Conclusions

In this reported study, a new mechanism model for the co-deposition of Fe-Ni alloys from poly-nuclear complexes was developed. The results of this effort showed that anomalous and normal co-deposition of Fe-Ni depended on the composition of the electroactive poly-nuclear complex in the plating solution. Simultaneous deposition of Fe and Ni was determined by the composition of the complex. Highly alloyed, uniform Fe-Ni deposits were obtained. The rate deposition in the Fe-Ni ammonia-citrate bath was found to be faster than that in the Fe-Ni citrate bath, which may be caused by the difference in the negative charge number of poly-nuclear complexes.

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