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Nucleation and Growth of a Binary Electrodeposited Ni-Co Thin Film

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Abstract- The microscopic arrangement of alloys has a significant influence on their electrical, thermal, mechanical, and catalytic properties. In this respect, we have developed nickel-cobalt alloy films by electrodeposition on an ITO glass substrate while examining nucleation and growth processes during the first steps of electrocrystallization. Energy Dispersive X-ray Spectroscopy (EDX) approved stoichiometry (1,1) of the alloy elaborated. X-ray diffraction (XRD) showed that the Co-Ni binary electrodeposited at different potentials crystallizes in a cubic structure that belongs to the Pm3m space group. From chronoamperometry and cyclic voltammetry curves, we concluded that the Co-Ni/ITO system is quasi-reversible, with diffusion of Ni²⁺ and Co²⁺ cations being the controlling step. The ascending part of the current-time transients investigation has shown that the Ni-Co electrodeposition mechanism is characterized by instantaneous nucleation in line with 3D growth. This result is confirmed by comparison of the experimental chronoamperometry data with the theoretical models of Scharifker and Hills on the one hand, and Bewick, Fleischmann, and Thirsk on the other.

Keywords- Nickel; Cobalt; Electrodeposition; Nucleation; Chronoamperometry; Cyclic Voltammetry

1. INTRODUCTION

An intriguing alloy, nickel-cobalt has distinctive qualities including high wear resistance [1], excellent electrical and thermal conductivity, high corrosion resistance (about 50 k Ω cm⁻²), and specific magnetic properties [2]. These qualities make it a great choice for a variety of applications in both the scientific and industrial domains. It was used for catalysis [1], electrocatalytic hydrogen evolution reaction [3,4], anti-corrosion applications, magnetic cards, and recording devices. The binary Ni-Co nanospheres / Nickel foam can be used as a negative electrode for energy storage/conversion in batteries [2] and supercapacitors with an excellent energy density of 34.5 Wh/kg at a power density of 683 W/kg [5-8]. Thanks to its thermal and electrochemical stability, the Ni-Co alloy can also be used in solid oxide fuel cells (SOFC) [5].

The properties of Ni-Co materials are influenced by the conditions and production method. Ni-Co alloys have been synthesized using a variety of methods, including physical ones like co-evaporation, and mechanical alloying and chemical ones like hydrothermal reduction, the sol-gel process, the microemulsion method, the magnetic field-assisted, solvothermal process, and electrochemical methods, or more specifically electrodeposition [7,8]. Due to its ease of use, affordability, and capacity to create pure, adherent, homogenous, and stable deposits, the electrodeposition has attracted a great deal of attention [9-11]. Moreover, it provides an in-situ opportunity to investigate the characterization and growth of deposited films. In addition, the Ni-Co alloy proprieties can easily controlled by the electrodeposition conditions, such as electrolyte composition, applied voltage, temperature and the nature of substrate material [9]. Also, the electrodeposition technique was chosen to investigate the nucleation and growth mechanism of the electrodeposited material [10,11]. Nucleation refers to the initial formation of a new phase, while growth refers to the increase in size of the newly formed phase [12]. However, Understanding the nucleation and growth mechanisms of Ni-Co alloys during electrodeposition is essential for optimizing the deposition process and enhancing the properties of the deposited alloy [13].

The purpose of this work is to investigate the growth and nucleation mechanisms of nickel, cobalt, and nickel-cobalt alloys electrodeposited on indium tin oxide (ITO) substrate, as well as the impact that applied potential has on both these processes and the materials' properties.

2. EXPERIMENTAL SECTION

2.1. Electrodeposition

The electrochemical experiment was conducted using a classic three-electrode cell that had an aqueous solution of 0.01 M hexahydrate nickel chloride (NiCl₂.6H₂O) and 0.01 M hexahydrate cobalt chloride (CoCl₂.6H₂O) prepared in 0.2M potassium chloride medium (KCl) as a supporting electrolyte. The working electrode used is a rectangular slide of glass

covered with indium tin oxide (ITO) layers. The counter electrode is platinum (Pt) grid and the saturated calomel electrode (SCE) was used as classic reference (all given potentials refer to this electrode). A Versa STAT 3 potentiostat-galvanostat associated with Versa Studio software was utilized for the measurement of Cyclic voltammetry (CV) and chronoamperometry (CA) at pH=6.5 and room temperature (20° C). The ITO electrode was ultrasonically pre-cleaned with acetone during 15 minutes, followed by 10 min of ethanol and distilled water prior to each experiment. The substrate area that emerged in the electrolytic solution was 1 cm².

2.2. Characterization

Utilizing an X-ray diffractometer (Broker D8 Advance) fitted with a graphite monochromator, a Lynx-Eye detector, and parallel beam optics Cu K_{α} radiation ($\lambda = 1.5411$ Å), electrodeposited nickel-cobalt binary thin films were examined. A Philips XL30FEG scanning electron microscope (SEM) was used for the morphology examination. The stoichiometry of the Ni-Co thin films was examined by energy dispersive X-ray spectroscopy (EDX).

3. RESULTS AND DISCUSSION

3.1. Cyclic voltammetry

Cyclic voltammetric method (CV) is one of the most suitable electrochemical techniques for studying the behaviour of redox systems and the oxidation and reduction processes of the corresponding species in an electrolytic solution. In fact, this technique was chosen in this work to study the electrodeposited cobalt-nickel binary film on ITO glass substrate. Before starting the binary co-deposition, it seemed necessary to study each metallic element alone. Therefore, four solutions were investigated using cyclic voltammetry in the voltage rang of -0.6 to -1.5 V vs SCE with a scan rate of 20mV s⁻¹. Figure 1 shows the obtained voltammograms for KCl, Ni²⁺; Co²⁺ and mixture (Ni²⁺, Co²⁺) solutions.

First, the lack of a peak in the blank voltammogram produced for the KCl solution indicates that the ITO substrate is stable in the supporting electrolyte. It was also found that each voltammogram shows a cathodic peak and a crossing between the back and forth scans in potential which leading to a nucleation loop characterizing to the deposition of a new phase [14].

For the solution containing only Ni^{2+} cations, the cathodic peak C_1 appearing in the vicinity of -1.10 V/SCE can be attributed to the deposition of nickel on the working electrode following reaction mechanism described by equation (1) [10].

$$Ni^{2+} + 2e^{-} = Ni$$
 (1)



Figure 1. Cyclic voltammograms of Ni and Co electrodeposition on ITO substrate at scan rate of 20 mV s⁻¹ (a) supporting electrolyte (blank) (b) Ni²⁺ (c) Co²⁺ and (d) mixture (Ni²⁺, Co²⁺) solutions

When dealing with a solution that solely contains Co^{2+} , the cathodic peak C₂ is found at a potential near -1.04 V/ECS, which is in line with the value determined by the Nernst formula for the Co²⁺/Co pair. It may be related to the cobalt (II) reduction as indicated in the reaction below (equation (2))[15].

$$Co^{2+}+2e^{-} = Co$$
 (2)

The corresponding voltammogram for the binary mixture (Co^{2+} , Ni^{2+}) displays a broad, more intense cathodic peak C₃ distributed over the range of potentials containing two peaks seen in situations where the ions are examined independently. This could be attributed to the superposition of the peak from the transforamtion of Co^{2+} ions to metallic Co and that from the reduction of Ni²⁺ ions to metallic Ni according to equation (3).

$$yCo^{2+} + xNi^{2+} + 2(x+y)e^{-} = Ni_xCo_y$$
 (3)

In addition, we observed that the onset of reduction of the binary system takes place at a less negative potential than the potentials of the separate metal deposits studied, which is in line with the theory of the co-deposition process [16]. This shift enables us to reduce the release of dihydrogen gas and thus obtain more homogenous films, as well as optimizing electrodeposition yields. So, the electrodeposition is an appropriate method to prepare the binaries films of cobalt and nickel by a reduction voltage ranging from -0.9 V to -1.15 V vs. SCE.

Next, we investigate this binary system's reversibility and the kind of control that goes into co-deposition. The cyclic voltammetry of the ITO substrate immersed in NiCl₂ (0.01M) and CoCl₂ (0.01M) in a KCl (0.2 M) solution selected as the supporting electrolyte will be investigated by studying the impact of scan rate in order to identify the mode that restricts the

transfer of nickel and cobalt (II) ions to the working electrode. Figure 2 exhibits the various voltammograms acquired for the four scan rates 10, 20, 30 and 40 mV/s.



Figure 2. (a) Cyclic voltammograms of ITO in a solution of NiCl₂ (0.01M) and CoCl₂ (0.01M) / KCl (0.2 M), pH =7; T= 25 °C at various scan rates; (b) plot of (j_{Peak}) as a function the scan rate square root ($v^{1/2}$)

From Figure 2(a), we can see that the cathodic peak current density j_{Peak} becomes more intense as the scan rate increases, with a slight shift in peak potentials towards more negative potentials, wish was typical behaviour of quasi-reversible reaction [17,18]. Indeed, the electrochemical parameters are extracted and listed in Table 1.

Table 1. Peak current density j_{peak} , peak potential E_{peak} and half-peak potential of cathodic peak as a function of scan rate deduced from voltammograms in chloride solution (Figure 2 (a))

V	E _{peak}	j _{peak}	E _{peak/2}	$ \mathbf{E}_{\mathbf{Peak}} - \mathbf{E}_{\mathbf{peak}/2} $
(mV/s)	(V/ECS)	$(mA cm^{-2})$		(V/SCE)
10	-1.14	-2.72	-1.04	0.10
20	-1.17	-2.95	-1.04	0.13
30	-1.21	-3.16	-1.06	0.15
40	-1.24	-3.34	-1.07	0.17

We can see from the above table (Table 1) that the gap between the peak and half-peak potentials is greater than the theoretical value corresponding to a reversible reaction, which is of the order of $\frac{\text{RT Ln 10}}{2 \text{ F}} = 29 \text{mV}$ at 298 K. We can therefore deduce that the electrodeposition of binary cobalt- nickel on ITO substrate is not a perfectly reversible process, which is in line with the results of cyclic voltammetry.

In Figure 2(b), we represented the variation of the cathodic peak current density's absolute value as a function of the scan rate's square root, in accordance with the Randles-Evčík equation [14], in order to gather additional information regarding the limiting mechanism of the Co-Ni electrodeposition. With a correlation coefficient of 0.996, it was discovered that the cathode peak current density, j_{Peak} (mA/cm²), varied linearly as a function of v^{1/2} (V/s)^{1/2}. This means that diffusion control governs the electrodeposition reaction of Co²⁺ and Ni²⁺ ions on ITO substrate [15,16].

3.2. Chronoamperometry

The chronoamperometric curves measured at potentials -1.02, -1.04, -1.06 and -1.08V/SCE are plotted in Figure 3. It is evident that following a brief double layer charging period, the current density rapidly rises in absolute value until it reaches a maximum value j_{max} at t_{max} , at which point it gradually falls once more. This corresponds to a typical pattern of j-t transients characterising diffusion-controlled electrodeposition processes [19]. Furthermore, the time during which the current density maximum is reached as the imposed (cathodic) potential increases in absolute value. Hence, the rate of deposition formation increases with the applied potential.



Figure 3. Transients current density for Co-Ni binary electrodeposition on ITO substrate at different deposition potentials

The first investigation of these chronoamperemetric responses consists of exploiting the last part which tends towards a constant current indicating diffusion control. So the Cottrell equation (4) [20] can be tested to confirm this finding by plotting current densities evolution in the last part as function of $t^{-1/2}$:

$$j = zFC[\frac{D}{\pi t}]^{1/2} \qquad (4)$$

where zF is the molar transferred charge, C is the concentration, D is the diffusion coefficient, and t is time.

The obtained figure 4 shows a linear dependence of the downstream current density between 12 and 15 seconds of different transients obtained, with correlation coefficients higher than 0.99. This result is in agreement with Cottrell's relationship [20] and also confirms that the electrodeposition of Ni-Co binary on ITO substrate is controlled by a diffusion process.



Figure 4. Transients current density (j) versus $t^{-1/2}$ of the last part at different potentials

The j-t transients may be utilised to identify the nucleation and deposit growth mechanisms during the first instants [21]. This method consists of comparing the experimental data with theoretical models developed by Scharifker and Hills for 3D-instantaneous (Equation (5)) and 3D-progressive (Equation (6)) nucleation processes, and those developed by Bewick, Fleischmann and Thirsk for 2D-instantaneous (Equation (7)) and 2D-progressive (Equation (8)) nucleation processes [22].

$$\left(\frac{j}{j_{max}}\right)^{2} = 1.9542\left(\frac{t_{max}}{t}\right)\left\{1 - \exp\left[-1.2564\left(\frac{t}{t_{max}}\right)\right]\right\}^{2}$$
(5)
$$\left(\frac{j}{j_{max}}\right)^{2} = 1.2254\left(\frac{t_{max}}{t}\right)\left\{1 - \exp\left[-2.3397\left(\frac{t}{t_{max}}\right)^{2}\right]\right\}^{2}$$
(6)
$$\left(\frac{j}{t_{max}}\right) = \left(\frac{t}{t_{max}}\right)\exp\left\{\frac{1}{t_{max}}\left[1 - \left(\frac{t}{t_{max}}\right)^{2}\right]\right\}$$
(7)

$$\left(\frac{J}{j_{max}}\right) = \left(\frac{J}{t_{max}}\right) exp\left\{\frac{1}{2}\left[1 - \left(\frac{J}{t_{max}}\right)\right]\right\}$$
(7)

$$\left(\frac{j}{j_{max}}\right) = \left(\frac{t}{t_{max}}\right)^2 exp\left\{\frac{2}{3}\left[1 - \left(\frac{t}{t_{max}}\right)^3\right]\right\}$$
(8)

Where, j_{max} is the maximum current intensity and t_{max} is the corresponding time. The Figure 5 shows the plots $(j/j_{max})^2$ and (j/j_{max}) as a function of (t/t_{max}) and the theoretical models for the four imposed potentials.



Figure 5. Comparison of theoretical non-dimensional plots for instantaneous and progressive nucleation to experimental transients current density: (a) 3D nucleation (b) 2D nucleation

An analysis of Figure 5 shows that the experimental curves deviate significantly from the theoretical curves for the two 2D nucleation processes. Whereas, the experimental $(j/j_{max})^2$ curves show a correlation with the instantaneous three-dimensional nucleation mode at short time and a slight deviation from the theoretical model after t_{max}.

To confirm the nucleation mechanism obtained by Scharifker-Hills model another analysis can be considered according to Astley approach[20,23,24]. The latter has described the variation of the current density in the ascending part of the current transients by the following equations in two limiting nucleation cases.

For instantaneous 3D nucleation/growth mechanisms, the equation (9) led to a linear variation $j-t^{3/2}$:

$$j(t) = zFD^{3/2}C\pi^{1/2}Nkt^{1/2} \quad (9)$$

For progressive 3D nucleation/growth mechanisms, (equation (10)) [25] a linear correlation between $j-t^{1/2}$ must be obtained.

$$j(t) = \frac{1}{2} z F D^{3/2} C \pi^{1/2} N_0 k' t^{3/2} \quad (10)$$

Where N is the number of nuclei and N₀ is the number of accessible active sites.

We plotted the variation curves of $j-t^{1/2}$ (Figure 6(a)) and $j-t^{3/2}$ (Figure 6(b)) for the ascending part before the current maximum in Figure 6 in order to investigate the theoretical models developed by Astley. The instantaneous nucleation mode, which was previously determined using non-dimensional graphs, is confirmed by these figures, which clearly demonstrate that linear regression of the experimental results yields a strong linear correlation for the $j-t^{1/2}$ curves.



Figure 6. Transients current density (j) (a) versus $t^{1/2}$ (b) versus $t^{3/2}$ of the second part at different potentials

3.3. Characterization

3.3.1. SEM analysis

Using a field emission scanning electron microscope, the structural morphology of Co-Ni binary nanowires grown on ITO substrate was examined. The cross-sectional SEM images were taken from Co-Ni/ITO sample prepared by chronoamperemetry at potential of -1.06 V/SCE during 5min of deposition. The surface of the ITO substrate is entirely covered in Co-Ni binary nanowires, as seen by the SEM image in Figure 7. This indicates that the nanowires have not been deposited uniformly, which is consistent with the 3D progressive nucleation found in the chrnoamperemetric study. Since two morphologic structures are observed, a smoother NiCo films consisting of well-organized micrometric grains, with the same sizes approximately is accompanied by 3D dendritic morphology which starts to nucleate in some locations of the substrate. The dendrites formed are heavily branched and composed of several hierarchical flowers. It grows from a central nucleus to which several sub-branches of different diameters and lengths are attached.



Figure 7. SEM image of Ni-Co binary electrodeposited on ITO substrate by chronoamperemetry at potential of -1.06 V/SCE during 5 min

3.3.2. EDX analysis

Elemental analysis was done using an energy dispersive X-ray analyzer (EDX) attached onto the SEM. Based on EDX results, shown in Figure 8 the composition of electrodeposited binary Ni-Co coatings ITO substrate has been determined and described in table of Figure 8. It is apparent that deposits contain the same atomic percentage of nickel and cobalt. However, the other elements making up the substrate have also been identified. We then conclude that the use of a mixture of precursor ions with the same concentrations leads to the formation of a binary with Ni and Co stoichiometry. We also note that other studies have shown that under the effect of concentration several stoichiometries can be achieved.



Figure 8. EDS spectrum and elemental composition of Ni-Co binary electrodeposited on ITO substrate by chronoamperemetry at potential of -1.06 V/SCE during 5 min

3.3.3. XRD analysis

The X-ray diffraction technique was used to characterize the crystalline structure of the binary Ni-Co thin film potentiostatically deposited on the ITO substrate, at an applied potential of -1.06 V *vs*. SCE, during 15 minutes. The resulting XRD patterns are shown in Figure 9.

The characteristic peaks located at different diffraction angle (20) of 30.8° , 45.8° , and 66.7° corresponding to the (111), (110), (200) crystallographic planes of NiCo , as indexed with reference to JCPDS card (no. 73-1702) [26]. NiCo thin films exhibited a cubic crystal structure with the Pm3m space group [7]. Based on the peak positions, the lattice parameters were calculated as follows: a = b = c = 2.78 Å. The preferred orientation of the crystallites is along the (110) direction.



Figure 9. XRD patterns of the NiCo thin film electrodeposited on ITO substrate during 15 minutes

The crystallite size "D" was calculated using Scherrer's equation (11) [11]:

$$D = \frac{\kappa\lambda}{\beta\cos\theta} \tag{11}$$

where λ is the wavelength of the incident radiation ($\lambda = 1.5418$ Å), K = 0.9, β the full width at half maximum (FWHM) of the diffraction peak, θ : the Bragg diffraction angle. The dislocation density (δ) was calculated using the equation (12) [27].

$$\delta = \frac{1}{D^2} \tag{12}$$

The grain size (D) and the dislocation density (δ) of the crystallites in the NiCo films are tabulated in Table 2.

Planes (h k l)	Position (2θ) (Degree)	FWHM (Degree)	Grain size (D) (nm)	Dislocation density δ (× 10 ¹⁴) m ⁻²
(111)	30.8	0.157	52.526	3.624
(110)	45.8	0.250	34.523	8.390
(200)	66.7	0.187	50.897	3.860

Table 2. Calculated properties of NiCo cubic phase films deposited at 15 minutes

4. CONCLUSION

This study of the binary (Ni-Co) electrodeposition on the ITO substrate was carried out using cyclic voltammetry and chronoamperemetry. The typical behavior of a quasi-reversible reaction controlled by diffusion of Co²⁺ and Ni²⁺ was identified in cobalt–nickel codeposition process. These results were confirmed by the Cottrell model and scan rate effect. According to Scharifker, Hills, and Astley's theoretical model, the electrodeposition mechanism of nickel-cobalt codeposits during the initial stages demonstrated instantaneous nucleation and three-dimensional growth. The morphology of the Ni-Co deposits showed that the surface of the ITO substrate is entirely filled with binary Co-Ni hierarchical flowers, wish is in good agreement with the 3D instantaneous nucleation, as concluded by the chronoamperometric study. By using EDS analysis, it was found that the stoichiometry of binary (Ni-Co) deposit is (1.1).

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Declarations of interest

The authors declare no conflict of interest in this reported work.

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