Plating & Surface Finishing

Electroplating of Cd-Ni Alloy from Gluconate Baths

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The electroplating of Cd-Ni alloys onto steel substrates from baths containing $CdSO_4$, $NiSO_4$, sodium gluconate and boric acid (pH ~ 5) has been investigated. The influences of bath composition, cathodic current density and temperature on the electrodeposited Cd-Ni alloys have been examined. The study included the effect of these variables on cathodic polarization, cathodic current efficiency of co-deposition, chemical composition, surface morphology and structure of the deposits. The baths are characterized by high cathodic efficiency for co-deposition of Cd-Ni alloys. The co-deposition process shows an anomalous type of deposition with cadmium being preferentially deposited. The linear anodic stripping for Cd-Ni alloy shows the presence of two anodic peaks which are attributed to α-phase and ß-phase.

Keywords: *Alloy plating, cadmium-nickel alloys, gluconate plating bath*

Introduction

The electrodeposition of cadmium-nickel alloys has many technical applications, such as protection of jet-engine parts from corrosion, manufacture of electrical contacts and battery applications. The Cd-Ni alloy can be electrodeposited from various electrolytes, such as acidic sulfate,[1] sulfate-chloride,[2,3] sulfamate[4] and acetate[5] baths as well as from alkaline baths.[6] These investigations showed that the appearance of the alloy depends on the percentage of each metal in the deposit. However, the electrodeposition process is of the anomalous type. The improvement of the properties of electrodeposited Cd-Ni alloys is of great interest and requires more study. The literature shows that gluconate electrolytes have been used to electrodeposit metals such as nickel,[7] copper,[8] tin[9] and zinc,[10] but they are not currently utilized for electroplating binary alloys. The present investigation is aimed at developing baths from which a sound bright Cd-Ni alloy can be electrodeposited from gluconate solutions. The advantages of gluconate baths are their relatively low cost and environmentally friendliness. The present work studies the influences of bath composition, pH, temperature and current density on cathodic polarization,

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Experimental

The plating solutions were freshly prepared from purity-grade chemicals and doubly-distilled water. The numbers and chemical composition of these baths and operating conditions (cathodic current density, temperature and pH) are given in Table 1.

The experimental set up for the electrodeposition process has been described elsewhere.[11] In each run a mild sheet was placed midway between two parallel-plate platinum anodes. All electrodes had the same geometrical area (2.5 × 3.0 cm). Before using the mild steel cathode, it was degreased in petroleum ether for 24 hr, washed with water and dried. It was then polished with fine grade emery paper, washed with water, dried and weighed. The cathodic polarization during the deposition process was measured by using an EG&G 273A potentiostat / galvanostat connected to a computer. The potentials were measured relative to a saturated calomel electrode (SCE). The linear anodic stripping voltammetry was taken after the Cd-Ni alloys were deposited from some selected baths on platinum sheet under potentiostatic conditions (constant potentials) for constant time (t = 60 sec). At the end of the deposition, the deposits were anodically stripped under potentiodynamic conditions without withdrawing the electrodes or changing the solution. The potential was linearly swept at a scan rate of 5 mV/sec to more positive potentials. The cathodic current efficiency of the deposited alloys (F %) were determined by a recommended method.[12] The partial current efficiencies of the parent metals in the alloy F_1 and F_2 were determined from the relations:

$$F_1 = \frac{w_1}{w_t} \times 100 \quad (1)$$

$$F_2 = \frac{w_2}{w_t} \times 100 \quad (2)$$

Where W_1 and W_2 are the practical weight of cadmium and nickel respectively, which are determined by atomic emission spectroscopy. The theoretical weights of both cadmium and nickel can be calculated from the relation:

$$W = Q \times I \times t \quad (3)$$

where ($I \times t$) is the amount of electricity passed through the cell in A-sec and Q is the electrochemical equivalent of the metal. The total efficiency of the alloy is equal to sum of F_1% and F_2%. The surface morphology of the as-deposited Cd-Ni alloy on steel was examined using scanning electron microscopy (JEOL, JSM-T20). X-ray diffraction analysis [Philips PW 1390 diffractometer (40 kV, 25 mA) with a Ni filter and Co-Kα radiation] was used to examine the crystal structure of the as-deposited Cd-Ni alloy.

Results and discussion

Cathodic polarization

Figure 1 shows potentiodynamic cathodic polarization curves for Cd, Ni and Cd-Ni alloy electrodeposition onto steel substrates from gluconate bath numbers 9, 11 and 1 (Table 1). The polarization curves were scanned from the zero current potential of the substrate to more negative values at a scan rate of 5 mV/sec at a bath temperature of 25°C.

In the gluconate solution, Ni^{+2} and Cd^{+2} are predominantly present as $[Ni\ G]^+$ and $[Cd\ G]^+$ soluble complex ions, respectively. The gluconate $[G^-]$ is attached to either Ni^{+2} or Cd^{+2} by coordination through the carboxyl group and one of the adjacent hydroxyl groups.[13] Therefore, Ni^{+2} and Cd^{+2} ions can be electrolytically reduced from either complexed or uncomplexed species simultaneously with hydrogen evolution from an independent side reaction.[7] It is seen from Fig. 1 that nickel electrodeposition commences at -600 mV_{SCE}. Further increases in cathodic potential cause an increase in current density, indicating that the deposition of nickel proceeds via kinetic control. However, cadmium

Table 1
Chemical composition and operating conditions of the baths used in this study

Bath Number	$CdSO_4 \cdot 8H_2O$ (g/L)	$NiSO_4 \cdot 6H_2O$ (g/L)	Sodium Gluconate (g/L)	H_3BO_3 (g/L)	Temp. (°C)	pH	Current Density (A/dm^2)
1	10	30	20	50	25	5	0.2
2	5	30	20	50	25	5	0.2
3	10	50	20	50	25	5	0.2
4	10	30	20	50	25	5	0.133
5	10	30	20	50	25	5	0.06
6	10	30	40	50	25	5	0.2
7	10	30	20	30	25	5	0.2
8	10	30	20	50	60	5	0.2
9	10		20	50	25	5	0.2
10	10		40	50	25	5	0.2
11		30	20	50	25	5	0.2
12		30	40	50	25	5	0.2
13	2	30	20	50	25	5	0.2
14	12	30	20	50	25	5	0.2
15	10	10	20	50	25	5	0.2
16	10	30	5	50	25	5	0.2
17	10	30	30	50	25	5	0.2
18	10	30	10	50	25	5	0.2

electrodeposition begins at a more negative potential (-700 mV_{SCE}) with a rapid increase in current density up to a certain limiting current density, indicating that the deposition of cadmium in this case occurs under diffusion control. The cathodic polarization curve of Cd-Ni alloy electrodeposition is similar to that of pure cadmium, but it takes place at a less negative potential value than either the parent metals. Therefore, one can expect preferential deposition of cadmium and production of cadmium-rich alloys. This expectation is valid with the present system, which is the so-called anomalous co-deposition according to Brenner's classification.[14]

It has been reported that the single deposition of metals occurs via a two-step mechanism with one electron transfer in each step as follows:[15]

$$M^{+2} + e^- \rightarrow M^+ \quad (4)$$

$$M^+ + e^- \rightarrow M \quad (5)$$

Where M is either cadmium or nickel and M^+ is the intermediate species, which may or may not be adsorbed on the electrode surface. The formation of a mixed intermediate surface compound $(Cd\ Ni)_{ads}^{+}$ adsorbed on the cathode surface is also possible.[16] This intermediate can be spontaneously decomposed on the alloy surface as:

$$(Cd\ Ni)_{ads}^{+} + e^- \rightarrow Cd + Ni \quad (6)$$

It is probable that the codeposition of Cd-Ni alloy occurs via deposition of each parent metal independently through two successive one-electron transfer steps.[17]

Figures 2 and 3 show the effect of increasing the concentration of Cd^{+2} and Ni^{+2}, respectively, on the cathodic polarization curves for Cd-Ni codeposition on steel from gluconate baths at 25°C. It is clear that an increase in the concentration of either Cd^{+2} or Ni^{+2} in the bath increases

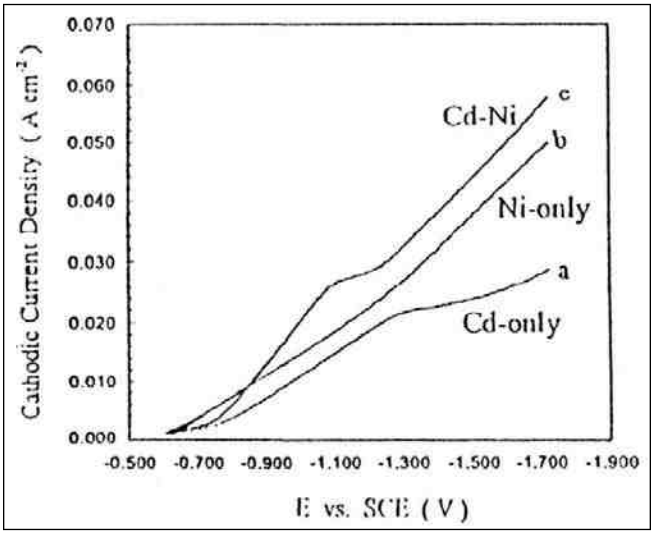

Figure 1 - Potentiodynamic cathodic polarization curves for electrodeposition: (a) 10 g/L $CdSO_4 \cdot 8H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid; (b) 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid; (c) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid at 0.2 A/dm²; scan rate = 20 mV/sec and T = 25°C.

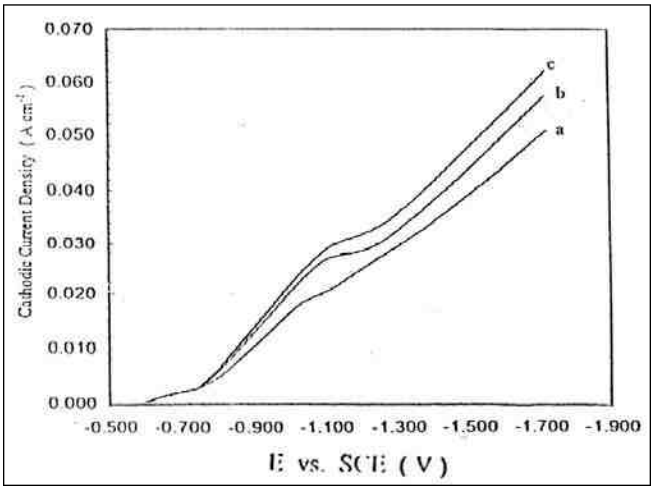

Figure 2 - Effect of $CdSO_4$ concentration on potentiodynamic cathodic polarization curves for Cd-Ni alloy electrodeposition: (a) 2 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid; (b) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid; (c) 12 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid at 0.2 A/dm²; scan rate = 20 mV/sec and T = 25°C.

the limiting current density and decreases the cathodic polarization of the Cd-Ni co-deposition. This can be explained in light of the fact that increasing the concentration of parent metal ions in the bath would result in lowering the concentration polarization type for electrodeposition of the alloy.[18]

Figure 4 shows the influence of the sodium gluconate content in the bath on the cathodic polarization curve for Cd-Ni alloy electrodeposition. It is obvious that increasing the gluconate concentration leads to a shift of the cathodic polarization curve to less negative potential values. This behavior could be related to an increase in the conductivity of the bath.

Figure 5 represents the cathodic polarization curves for Cd-Ni alloy electrodeposition at different electrolyte temperatures ranging from 25 to 60°C. Inspection of the curves indicates that increasing temperature results in increasing limiting current density and shifts the cathodic polarization of the alloy to less negative potential values. This is attributable to a decrease in activation polarization of both alloy co-deposition and hydrogen evolution. In addition, an increase in temperature enhances the rate of diffusion of the reducible species to the cathode surface and consequently concentration polarization also decreases.

Deposit composition

The composition of Cd-Ni alloys was analyzed by energy dispersive x-ray analysis (EDX). The percentage of Cd and Ni in both the plating solution and the deposit, the partial cathodic current efficiency of H_2 evolution and the total current efficiency of Cd-Ni alloy deposition are listed in Table 2. It is worth noting that the amount of cadmium in the

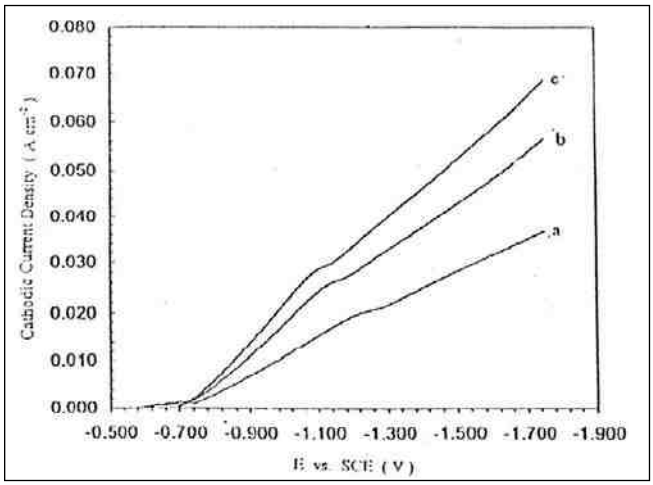

Figure 3 - Effect of $NiSO_4$ concentration on potentiodynamic cathodic polarization curves for Cd-Ni alloy electrodeposition: (a) 10 g/L $CdSO_4 \cdot 8H_2O$, 10 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid; (b) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid; (c) 10 g/L $CdSO_4 \cdot 8H_2O$, 50 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid at 0.2 A/dm²; scan rate = 20 mV/sec and T = 25°C.

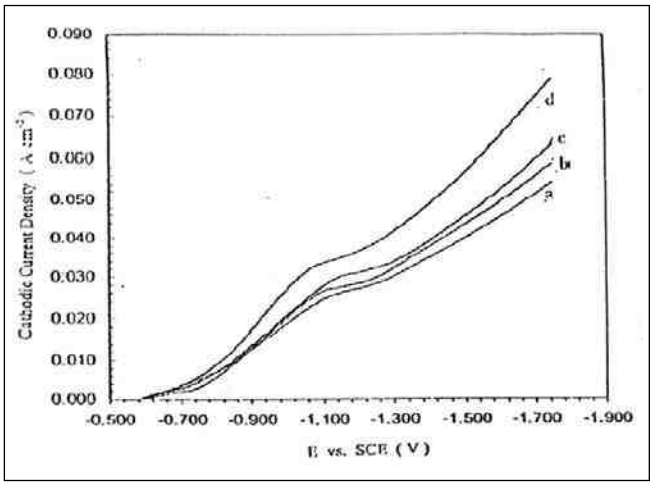

Figure 4 - Effect of sodium gluconate concentration on potentiodynamic cathodic polarization curves for Cd-Ni alloy electrodeposition: (a) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 5.0 g/L sodium gluconate and 50 g/L boric acid; (b) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 10 g/L sodium gluconate and 50 g/L boric acid; (c) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 30 g/L sodium gluconate and 50 g/L boric acid; (d) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid at 0.2 A/dm²; scan rate = 20 mV/sec and T = 25°C.

Cd-Ni alloys is larger than its composition in the respective bath indicating that cadmium is preferentially deposited, and thus there is anomalous codeposition.[13]

The most accepted mechanism for anomalous co-deposition[3,14] includes the assumption that the pH in the vicinity of the cathode surface is raised to permit the precipitation of metal hydroxide on the cathode surface. However, the anomaly of the Cd-Ni alloy process can be assigned to the inhibitory action of adsorbed $Cd(OH)_2$ on the cathode surface. The adsorption of $Cd(OH)_2$ on one hand, accelerates the discharge of cadmium and on the other hand, may suppress the deposition of nickel by blocking its discharge sites. Nevertheless, it is found that the cadmium composition in the alloy deposit increases with increasing total metal content in the bath. An increase in the total amount of metal ions in the diffusion layer favors the increased deposition of the preferentially deposited metal.[14]

It is also clear from the data in Table 2 that the cathodic current efficiency of Cd-Ni alloy deposition is less than 100% as a result of simultaneous evolution of H_2. The data reveal that the efficiency increases with increasing concentration of Cd^{+2}, Ni^{+2} and gluconate ions.

Linear anodic stripping voltammetry

Potentiodynamic linear anodic stripping voltammetry (LASV) was performed in the same plating bath to analyze *in situ* the Cd-Ni alloy deposited onto a platinum electrode at constant cathodic potential E_s. Figures 6 and 7 show the stripping response for Cd-Ni alloys deposited from Bath Nos. 1 and 6, respectively at E_s = -900 mV and 25°C. For comparison, stripping curves were recorded for pure cadmium and pure nickel under identical conditions. The

Table 2
The percentage of Cd and Ni in both the plating bath and the deposit, the partial cathodic current efficiency of H_2 evolution and the total current efficiency of the Cd-Ni alloy deposition

Bath Number	Metal in the bath (wt%)		Metal in the deposit (wt%)		Cathode current efficiency (*F*%)	
	Cd	Ni	Cd	Ni	Cd-Ni	H_2
1	19.26	80.74	75.79	24.21	93.28	6.72
2	10.65	89.35	64.38	35.62	88.61	11.39
3	12.52	87.48	69.00	31.00	94.01	5.99
4	19.26	80.74	75.79	24.21	99.31	0.69
5	19.26	80.74	97.44	2.56	97.43	2.57
6	19.26	80.74	53.68	46.32	95.21	4.79
7	19.26	80.74	83.85	16.15	95.01	4.99
8	19.26	80.74	86.51	13.49	92.48	5.62

LASV responses of pure cadmium and pure nickel have only one stripping peak. The curves of Figs. 6 and 7 reveal that the stripping peak of pure nickel is broad and appears at a more noble potential than that of either pure cadmium or Cd-Ni alloy. It is also seen that there are two anodic peaks for the electrodeposited Cd-Ni alloy. The peaks correspond to an α-phase (Cd-Ni) and a β-phase (Cd_5Ni) which appears at a more positive potential.[18,19] It is interesting to note that the quantity of charge consumed through the Cd-Ni alloy stripping peak obtained from Bath 6 is larger than that obtained from Bath 1. This result agrees well with the data of cathodic current efficiency listed in Table 2. Moreover, Fig. 8 shows that increasing the deposition potential E_s from -800 to -1200 mV_{SCE} shifts both Cd-Ni alloy stripping to more noble potentials and increases their sizes. These changes may be attributed to an increase in the stability of Cd-Ni deposits as well as the quantity of the deposit. The same finding can be obtained for Cd-Ni electrodeposited from Bath 6, with increasing deposition time at constant deposition potential E_s as shown in Fig. 9.

Surface morphology

The Cd-Ni alloy films plated onto steel and platinum substrates from gluconate baths are bright, smooth, compact and adherent to the substrate surface. The surface morphology of as-deposited Cd-Ni alloys from gluconate baths onto steel was examined by scanning electron microscopy (SEM). Figure 10 shows the SEM micrographs for Cd-Ni alloy as-deposited from Baths 1, 5, 6 and 8 (Table 1), respectively. The micrographs show that the deposits are dense and compact as well as free of pores and cracks. This is attributable to the high cathodic current efficiency of co-deposition and consequently low H_2 evolution. It is noted that the increase of cathodic current density from 0.06 to 0.2 A/dm^2 decreased the grain

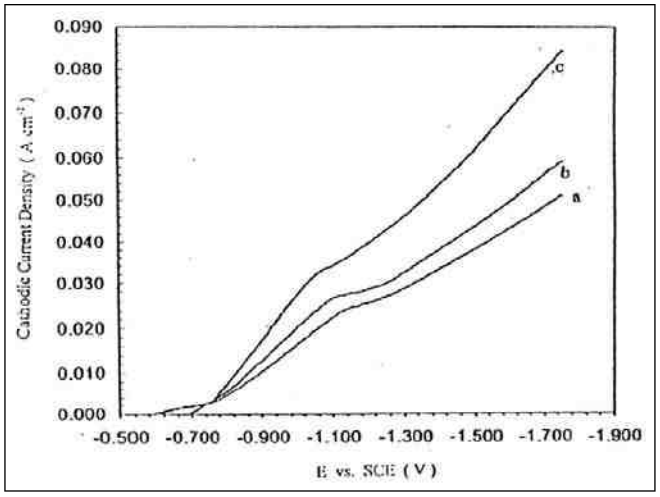

Figure 5 - Effect of temperature on potentiodynamic cathodic polarization curves for Cd-Ni alloy electrodeposition from a bath containing 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20.0 g/L sodium gluconate and 50 g/L boric acid at 0.2 A/dm^2 and scan rate = 20 mV/sec: (a) 25°C; (b) 40°C; (c) 60°C.

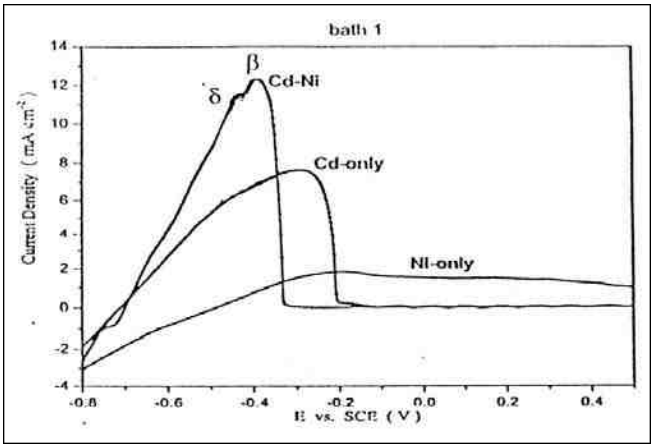

Figure 6 - Linear anodic stripping voltammetry (LASV) curves for cadmium, nickel and Cd-Ni alloy from a bath containing: (a) 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid; (b) 10 g/L $CdSO_4 \cdot 8H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid: (c) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid at 0.2 Adm^2, scan rate = 5 mV/sec, time = 160 sec and deposition potential = -0.9 V_{SCE}.

size and enhanced the smoothness of the deposit (Fig. 10a and b). On the other hand, the opposite was found when the temperature was increased from 25 to 60°C (Fig. 10c and d).

Conclusions

Electroplating of Cd-Ni alloys containing 64 to 97% Cd was obtained from baths containing $CdSO_4$, $NiSO_4$, sodium gluconate and boric acid at 0.2 A/dm^2 and 25°C. The effects of bath composition, current density and temperature on cathodic current efficiency of co-deposition, composition of alloy and surface morphology of the deposited alloy were examined. The observed anomalous type of Cd-Ni alloy electrodeposition could be attributed to the formation and adsorption of $Cd(OH)_2$ on the cathode surface. High current efficiency for Cd-Ni alloys with α-phase and ß-phase was observed.

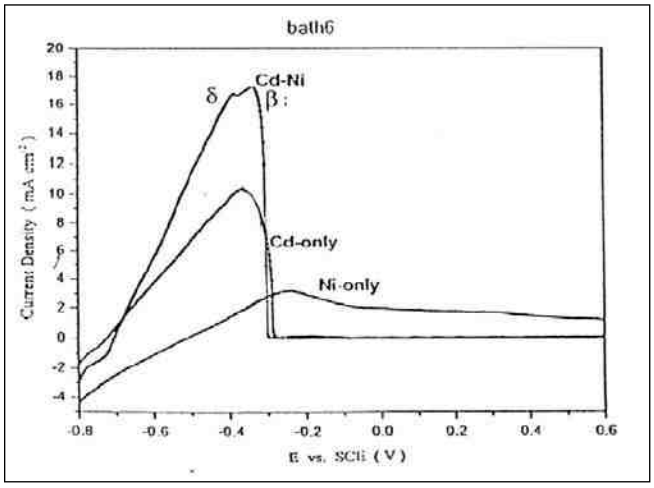

Figure 7 - Linear anodic stripping voltammetry (LASV) curves for cadmium, nickel and Cd-Ni alloy from a bath containing: (a) 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid; (b) 10 g/L $CdSO_4 \cdot 8H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid: (c) 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid at 0.2 Adm^2, scan rate = 5 mV/sec, time = 160 sec and deposition potential = -0.9 V_{SCE}.

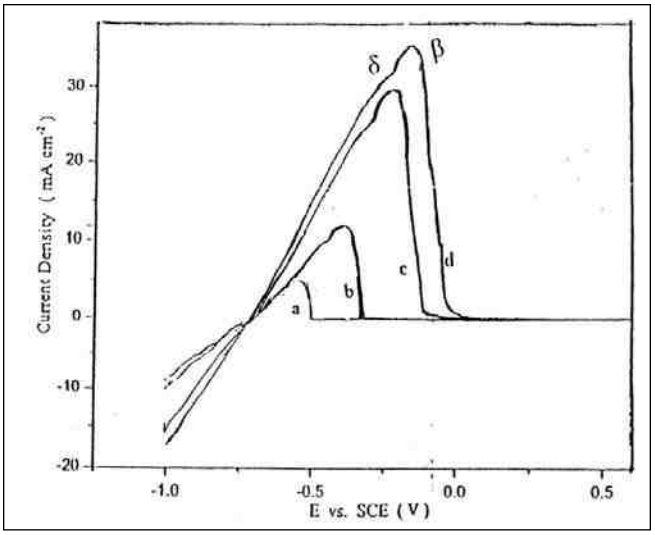

Figure 8 - Linear anodic stripping voltammetry (LASV) curves for a Cd-Ni alloy from a bath containing 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L sodium gluconate and 50 g/L boric acid at 0.2 Adm^2 at different deposition potentials: (a) -0.8 V_{SCE}; (b) -0.9 V_{SCE}: (c) -1.00 V_{SCE}; scan rate = 5 mV/sec and time = 160 sec.

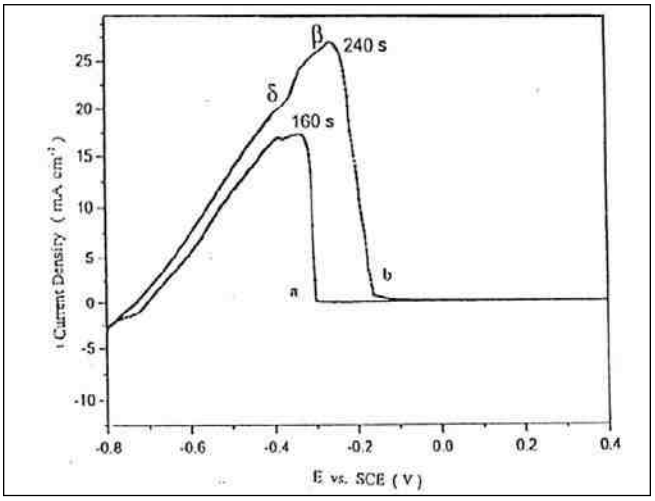

Figure 9 - Linear anodic stripping voltammetry (LASV) curves for a Cd-Ni alloy from a bath containing 10 g/L $CdSO_4 \cdot 8H_2O$, 30 g/L $NiSO_4 \cdot 6H_2O$, 40 g/L sodium gluconate and 50 g/L boric acid at 0.2 Adm^2 at different times: (a) 160 sec; (b) 240 sec; scan rate = 5 mV/sec and deposition potential = -0.9 V_{SCE}.

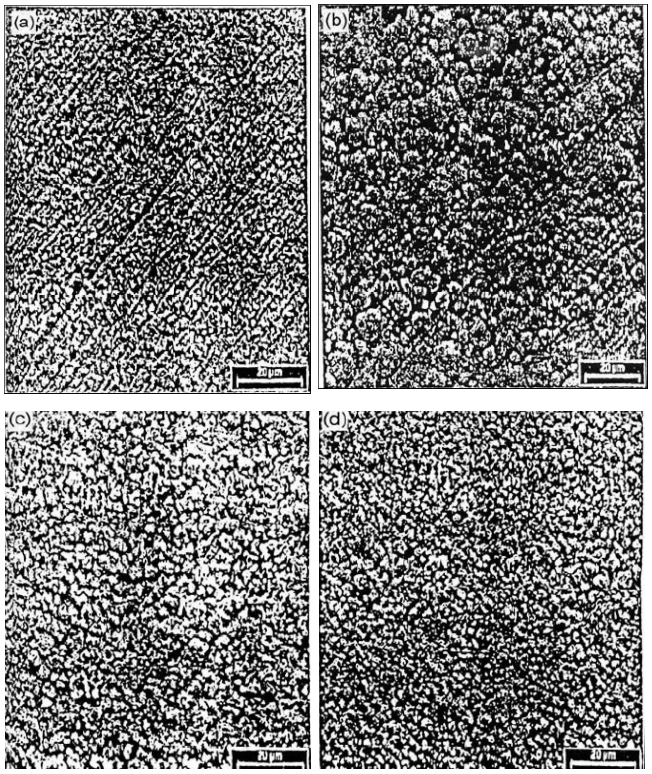

Figure 10 - SEM micrographs of electrodeposited Cd-Ni alloys: (a) 0.2 A/dm², 25°C, time = 15 min; (b) 0.065 A/dm², 25°C, time = 15 min; (c) 0.2 A/dm², 25°C, time = 15 min; (d) 0.2 A/dm², 60°C, time = 15 min.

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