



Effect of Cadmium on Corrosion Performance of Ni-Mo Alloy Coating

Akshatha R Shetty*, A. Chitharanjan Hegde

Department of Chemistry, National Institute of Technology Karnataka, Surathkal, India

*Corresponding author: Akshatha R Shetty, Department of Chemistry, National Institute of Technology Karnataka, Surathkal, India, Tel: 08242474046, E-mail: acrhegde@gmail.com

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Abstract

In this article an attempt was made to increase the corrosion resistance character of Ni-Mo alloy coatings on addition of small quantity of CdCl₂ into its bath. The limitation of high limiting current density (iL) of Ni in both Ni-Mo and Ni-Cd baths due to inherent induced and normal type of codeposition has been successfully alleviated by addition of 1g/L of CdCl₂. The Ni-Mo-Cd alloy coatings of high corrosion resistance was developed by decreasing the iL of Ni. The advent of induced and normal type of codeposition that the individual binary baths follow have been used advantageously to optimize the Ni content of the ternary deposit for better corrosion stability. The composition vs. current density (c.d.) plots of all coatings have been studied, and thereby optimal iL of Ni in all baths were assessed. Experimental investigation revealed that (Ni-Mo-Cd)_{6.0} Adm-2 coating shows the highest corrosion resistance by reducing iL of Ni, on addition of Cd⁺² ions into the bath. The improved corrosion resistance of Ni-Mo-Cd alloy coatings were explained in the light of diffusion limited deposition of Ni⁺² ions. The experimental results are discussed based on the data obtained from different analytical techniques, like Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and X-Ray Diffraction (XRD) techniques.

Keywords: Effect of Cd, Ni-Mo, Ni-Cd, Ni-Mo-Cd, corrosion study

Introduction

Ni-Mo alloy coating is of great interest due to their good corrosion resistance and their ability to catalyse the hydrogen evolution reaction in alkaline solutions. Owing to their high corrosion resistance, they are considered to be a potential replacement for hard chrome coating at lesser cost. Similarly, Ni-Cd alloy coatings finds its extensive applications in decorative finishing of metals and protection from corrosion. It is well known that Ni-Mo and Ni-Cd alloy coatings follow induced and normal type of codeposition, respectively. In both type of alloy deposition, change in the composition of alloy coating is negligible, or unpredictable with plating variables. From a theoretical point of view, in Ni-Mo alloy coating pure molybdenum (Mo) having $E_{0Mo} = -0.20$ V cannot be electrodeposited from their aqueous solution, and it can be codeposited easily with iron group metals such as nickel (Ni) forming an alloy. In Ni-Mo alloy coating, Ni stimulate the deposition of reluctant metal Mo, which do not deposit by itself. Ni-Cd alloy coating follows normal type of codeposition, where Ni deposits preferentially than Cd, which characterized by the preferential deposition of noble Ni ($E_{0Ni} = -0.25$ V) instead of Cd ($E_{0Cd} = -0.40$ V). In induced type of electroplating, plating variables, like current density (c.d.), temperature, agitation, pH etc. has vagarious dependency on the composition of the bath compared to other normal types of codeposition [1].

Our preliminary study on electrodeposition of Ni-Mo and Ni-Cd alloy coatings showed two peculiarities, in terms of their Ni content: i) Change of Ni content in Ni-Mo alloy coating with c.d. is insignificant due to induced type of codeposition, and ii) Ni content of Ni-Cd alloy decreases with increase of c.d., due to normal type of codeposition. Even at higher concentration of Ni in the bath, Ni content of the alloy decreased with increase of current density, pH and temperature, reasoned by the preferential deposition of less noble metal (Cd). In

other words, Ni content of the coating is extremely low compared its content in the bath. In general, corrosion protection ability of both Ni-Mo and Ni-Cd alloy coatings depends upon their Ni (noble metal) content [11-14], and in alloy plating, the metal content depends on the limiting current density (iL) of the metal. Hence, it is very unlikely to develop coatings of high corrosion protection from both Ni-Mo and Ni-Cd alloy baths, by changing the c.d. Hence, to alleviate the problem associated with limiting c.d. of Ni in baths of Ni-Mo and Ni-Cd to develop coatings of high corrosion resistance, electrodeposition of their ternary alloy coating, i.e. Ni-Mo-Cd coating has been tried. The advent of induced and normal type of codeposition that the individual binary baths followed have been used advantageously to optimize the Ni content of the ternary alloy for better corrosion stability. The experimental results are presented, with an account responsible for improved corrosion resistance of ternary alloy coatings. The optimal Ni content of the alloy coatings have been identified. Experimental observations are compared, in relation to its binary alloy coatings, and results are discussed [2].

Experimental

Two binary alloy baths, namely Ni-Mo and Ni-Cd were optimized using conventional Hull cell method as described elsewhere. The composition and processing parameters of Ni-Mo, Ni-Cd and Ni-Mo-Cd alloy baths used in the present study is given in Table 1. Electrolytic baths were prepared using LR grade (Merck, Mumbai, India) chemicals, as they are purchased without further purification, using double distilled water. The desired pH's were maintained using Micro pH Meter (Systronics, 362), by proper addition of either NH₄OH or HCl depending on the requirement. All depositions reported here were carried out at constant temperature (303K), under condition of constant agitation on copper substrate (7.5×3.0 cm), used as cathode. The copper substrates, polished metallurgically to get

mirror finish were used for electroplating. It is degreased (trichloroethylene), and then pickled (1:1 HNO₃) before to immersing it into electrolyte Table 1.

Chemical Constituents (g/L ⁻¹)	Ni-Mo Bath	Ni-Cd bath	Processing parameters
Nickel sulphate [NiSO ₄ .6H ₂ O]	18	18	Anode : Nickel plate
Sodium molybdate [Na ₂ MoO ₄]	48	-	Cathode: Copper
Sodium citrate [Na ₃ C ₆ H ₅ O ₇]	105	105	c.d. range: 1.0 - 6.0 Adm-2
Cadmium chloride [CdCl ₂]	-	1	Temperature: 303K, pH: 9.0, Duration: 10 min

Table 1: Composition and processing parameters of baths used for electrodeposition of Ni-Mo, Ni-Cd and Ni-Mo-Cd alloy coatings under constant condition of temperature and pH.

The depositions were carried out on known active surface area (3.0×3.0cm), leaving the other region covered by cellophane tape. Both binary and ternary alloy electrodepositions were carried out under same geometric conditions of anode and cathode, using 300 mL capacity cubic customized electrochemical cell as shown in the Figure 1.

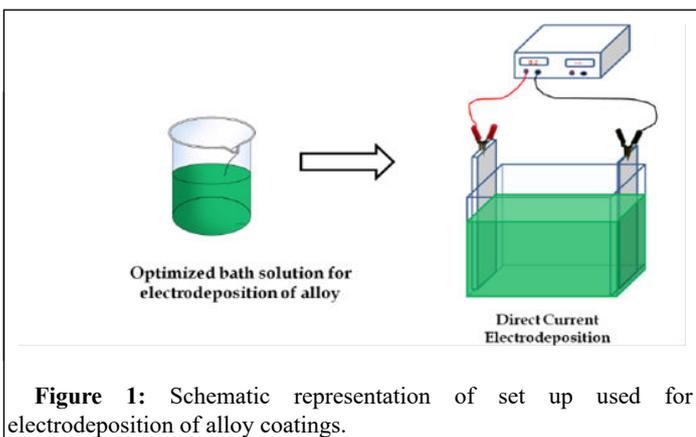


Figure 1: Schematic representation of set up used for electrodeposition of alloy coatings.

Electrodepositions were made at desired c.d.'s using DC power source (Power Analyzer, N6705A, Agilent Technologies, USA). The pure nickel plate was used as anode.

All depositions were carried out for 10 min. The cathode and anode were placed parallel at 5 cm distance, during deposition. After electrodeposition, all coatings were washed using distilled water followed by air drying. The coatings were then analysed.

The surface morphology, composition and phase structure of Ni-Mo, Ni-Cd and Ni-Mo-Cd alloy coatings were analyzed using Scanning Electron Microscopy (SEM), interfaced with Energy Dispersive X-ray spectroscopy(EDS)(Oxford EDS(X-act), and X-ray diffraction study(XRD)(Rigaku- miniFlex 600), respectively. Binary and ternary alloy coatings deposited at different c.d.'s are conveniently represented as (Ni-Mo) and (Ni-Cd) and (Ni-Mo-Cd) alloys, with subscription right hand side, indicating the c.d. at which they are deposited [3].

Results and Discussion

Electrodeposition of alloy coatings

Electrodeposition of binary (Ni-Mo and Ni-Cd) and ternary Ni-Mo-Cd alloy coatings were carried out at different c.d.'s (ranging from 1.0 - 6.0 Adm-2) using electrolytic baths, shown in Table 1. The composition and corrosion protection efficacy of alloy coatings corresponding to different c.d.'s are given in Table 2. The actual metal contents in Ni-Mo, Ni-Cd and Ni-Mo-Cd baths, calculated from the weight of Ni, Mo and Cd salts are also shown in Table 2.

Induced codeposition of Ni-Mo alloy

To begin with, electrodeposition Ni-Mo alloy coatings were carried out at four different c.d.'s (ranging from 1.0-6.0 Adm-2), using bath shown in Table 1. From the composition data, shown in Table 2 it may be noted that there is no significant change in the Ni content of alloy with c.d. However, a small decrease of Ni content with c.d. clearly indicates that the bath follows induced type of codeposition.

Coating configuration	wt. % Ni	wt. % Mo	wt. % Cd	CR×10-2 (mmy-1)
Induced type of codeposition				
(Ni-Mo) 1.0 Adm-2	70	30	-	51.6
(Ni-Mo) 2.0 Adm-2	69	29	-	40.5
(Ni-Mo)4.0 Adm-2	68.2	31.8	-	36.5
(Ni-Mo) 6.0 Adm-2	67	33	-	30.7
Normal type of codeposition				
(Ni-Cd)1.0 Adm-2	21	-	78	44.74

(Ni-Cd)2.0 Adm-2	56.9	-	43.1	42.94
(Ni-Cd)4.0 Adm-2	79.5	-	20.5	28.72
(Ni-Cd)6.0 Adm-2	76.7	-	23.3	39.7
Ternary alloy deposition				
(Ni-Mo-Cd)1.0 Adm-2	37	5.8	58	29.2
(Ni-Mo-Cd)2.0 Adm-2	27	5.4	60.8	24.4
(Ni-Mo-Cd)4.0 Adm-2	17.1	4.9	68.6	16.5
(Ni-Mo-Cd)6.0 Adm-2	13.9	4.4	70.7	15.4
Metal content in the bath				
Wt. of M in the bath	17.2	80.2	2.6	-

Table 2: Composition and corrosion performance of Ni-Mo, Ni-Cd and Ni-Mo-Cd alloy coatings deposited from their optimal baths at different c.d.'s.

It may be further noted that wt. % Mo in the deposit is in the range of only ~ 30%, over the entire range of c.d. studied compared to its 80.2 % in the bath. At the same time, wt. % Ni in the deposit is found to be much higher (~70 %), compared to that in the bath (17.2 %). A small variation of Ni and Mo content in Ni-Mo alloy coatings, over the range of c.d. studied was found (Table 2). It is the testimony for induced type of codeposition, enabled due to possible complexation of Mo and Ni ions with citrate ions [15]. Thus, from the corrosion data reported in Table 2, it may be noted that a bright and uniform coating of Ni-Mo alloy coating corresponding to 6.0 Adm-2, represented as (Ni-Mo)6.0 Adm-2 coating is the most corrosion resistant, among others.

Normal codeposition of Ni-Cd alloy

Similarly, Ni-Cd alloy coatings depositions were carried out at four c.d.'s, like 1.0, 2.0, 4.0 and 6.0 Adm-2, using Ni-Cd bath shown in Table 1. The optimized bath allowed the deposition of variety of coatings, depending on the c.d. employed. The composition analysis data of coatings, deposited at different c.d.'s are reported in Table 2, with their corrosion rates. It is important to note that over entire range of c.d. studied, wt. % of Cd (less noble metal) in the deposit is much more than that in the bath [16], compared to only 2.6 % in the bath (Table 2).

The composition data of Cd in the bath indicates that the bath follows normal type of codeposition with preferential deposition of noble metal (Ni) with increase c.d.[17]. The corrosion data reported in Table 2 reveals that corrosion rate of Ni-Cd alloy coatings decreases with increase of Ni content, affected at higher c.d. In other words, corrosion rate decreased with increase of Ni content. However, at 6.0 Adm-2, i.e. corresponding to (Ni-Cd)6.0 Adm-2 CR was found to increase once again. It may be attributed to the fact that applied c.d. has exceeded the limiting c.d. of Ni, for bath under operation. Thus a bright and uniform Ni-Cd alloy coating corresponding to 4.0 Adm-2, represented as (Ni-Cd)4.0 Adm-2 was found to be the most corrosion resistant compared to all other coatings [4].

Codeposition of Ni-Mo-Cd alloy

Based on the corrosion performance of Ni-Mo and Ni-Cd alloy coatings, and their dependency with c.d., an attempt was made

develop ternary alloy coatings of higher corrosion resistance by proper manipulation of its Ni content. In this direction, a ternary Ni-Mo-Cd alloy coating was attempted by adding small quantity (1.0 g/L) of CdCl₂ into optimized Ni-Mo bath. The composition and operating variables used for deposition of Ni-Mo-Cd alloy coating is shown in Table 1. Ternary alloy coatings were electroplated at various c.d.'s, like 1.0, 2.0, 4.0 and 6.0 Adm-2, and their corrosion performance were evaluated. The composition and corrosion rates of ternary Ni-Mo-Cd alloy coatings corresponding to different c.d.'s are reported in Table 2. Hence, from corrosion data reported in Table 2, it may be inferred that Ni-Mo-Cd alloy coating corresponding to 6.0 Adm-2, represented as (Ni-Mo-Cd)6.0 Adm-2 is the most corrosion resistant.

Corrosion behaviour

Potentiodynamic Polarization Study

The corrosion behaviour of electroplated binary and ternary alloy coatings were studied by potentiodynamic polarization method and is shown in Figure 2.

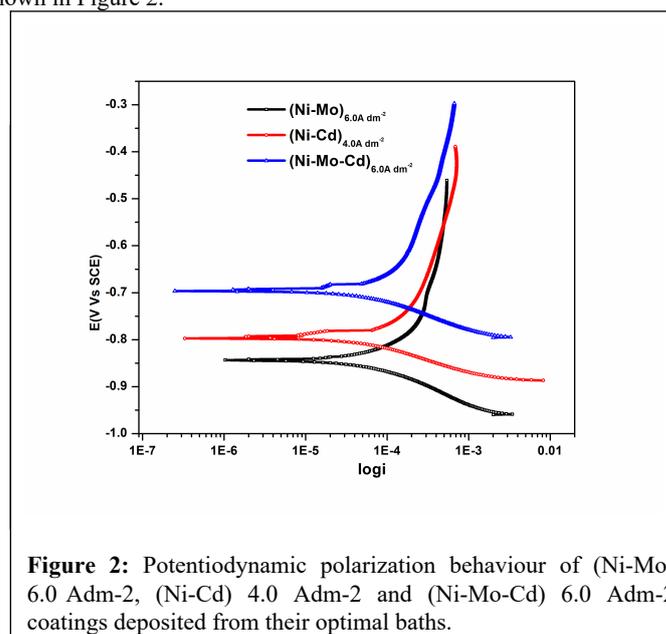


Figure 2: Potentiodynamic polarization behaviour of (Ni-Mo) 6.0 Adm-2, (Ni-Cd) 4.0 Adm-2 and (Ni-Mo-Cd) 6.0 Adm-2 coatings deposited from their optimal baths.

The corrosion rates of electrodeposited coatings are determined by Tafel's extrapolation method, and CR's are reported in Table 2. The polarization behaviour of Ni-Mo, Ni-Cd and Ni-Mo-Cd coatings, deposited only under optimal conditions are shown in Fig. 2, for comparison purpose. From nature of polarization plot, it may be noted that (Ni-Mo-Cd)6.0 Adm-2 coatings is more corrosion resistant than binary alloy coatings. This is further supported by its highest Ecorr values as seen in Fig. 2. The nature of the anodic curves of all three alloy coatings indicates that the corrosion is more an anodic controlled.

Electrochemical Impedance Spectroscopy (EIS) study

EIS technique is used for ranking coatings, quantifying coating breakdown, assessing interfacial reactions and for predicting the lifetime of coating and/or metal systems. The EIS responses of electrodeposited Ni-Mo, Ni-Cd and Ni-Mo-Cd coatings, corresponding to their optimal conditions are shown in Figure 3. It may be seen confirmed from larger diameter of the (unfinished) semicircle corresponding to (Ni-Mo-Cd)6.0Adm-2 coatings is more corrosion resistant than binary alloys.

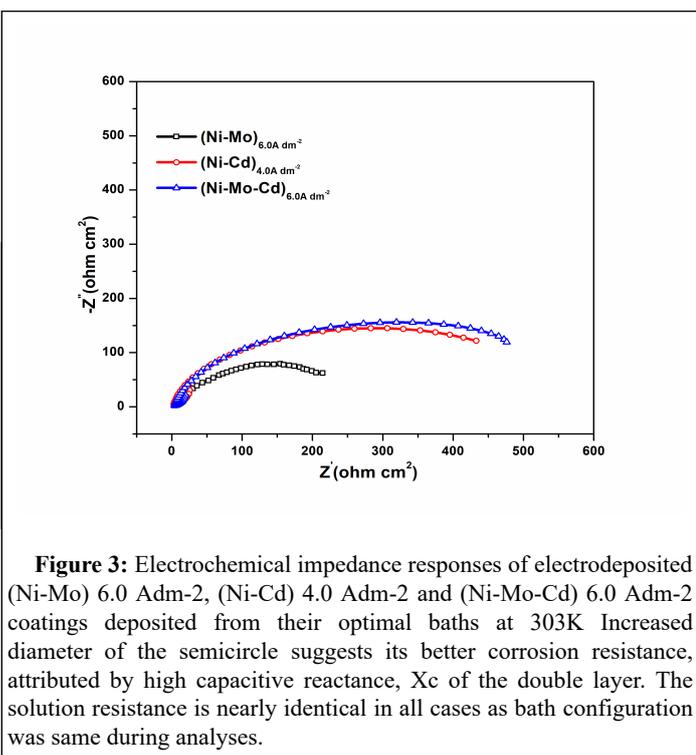


Figure 3: Electrochemical impedance responses of electrodeposited (Ni-Mo) 6.0 Adm-2, (Ni-Cd) 4.0 Adm-2 and (Ni-Mo-Cd) 6.0 Adm-2 coatings deposited from their optimal baths at 303K. Increased diameter of the semicircle suggests its better corrosion resistance, attributed by high capacitive reactance, X_c of the double layer. The solution resistance is nearly identical in all cases as bath configuration was same during analyses.

Surface morphology and XRD study

The corrosion performance of any coatings largely depends on the surface morphology and their phase structures. The microstructure of (Ni-Mo) 6.0 Adm-2, (Ni-Cd) 4.0 Adm-2 and (Ni-Mo-Cd) 6.0 Adm-2 alloy coatings, showing the highest corrosion resistance of respective baths are shown in Figure 4. The poor corrosion resistance behaviour of (Ni-Mo) 6.0 Adm-2 alloy coating may be due to its rough surface, having granular structure as may be seen in Fig. 4a. The better corrosion resistance of (Ni-Cd) 4.0 Adm-2 alloy coating, compared to that of (Ni-Mo) 6.0 Adm-2 coating is due to decrease of surface roughness, seen in Fig. 4b. The least corrosion rate of (Ni-Mo-Cd) 6.0 Adm-2 coating may be attributed to increased smoothness of coating,

compared to both binary alloys as seen in Fig.4c. From the composition data (Table 2), it may be noted that smoothness of coatings has inverse dependency with Ni content of the alloy. Thus, high corrosion resistance of (Ni-Mo-Cd) 6.0 Adm-2 alloy coating is not attributed by its Ni content of alloy.

The phase analysis of all coatings were carried out using XRD technique. The plot of intensity versus 2θ (position) values helps to assess the crystallinity of coatings corresponding to different metals. It may be noted that there is no change in the position of the XRD peaks corresponding to (Ni-Mo) 6.0 Adm-2, (Ni-Cd) 6.0 Adm-2 and (Ni-Mo-Cd) 6.0 Adm-2 coatings, except their intensity as may be seen Figure 4. All most same type of XRD patterns were demonstrated for binary (Ni-Mo) 6.0 Adm-2, (Ni-Cd) 4.0 Adm-2 and ternary (Ni-Mo-Cd) 6.0 Adm-2 coatings. The constancy of diffraction angles in all XRD patterns of all alloy coating (both binary and ternary) suggests that coatings form the solid solutions of individual metals. Hence, it may be inferred that alloy coatings are likely formed in baths having complex ions, due to citrate salt. Solid solutions are characterized by the fact that atoms in the lattice of a metal are substituted by atoms of another metal. This substitution changes the dimension of the unit cell but does not change the type of cell, and this substitution may occur either over a limited range of composition or over the complete range of composition from one pure metal to the other. A slight shift of diffraction lines, corresponding to different alloy coatings may be noted as may be seen in Figure 4b. This may be attributed to lattice expansion of metals affected due to difference in size of atoms [5].

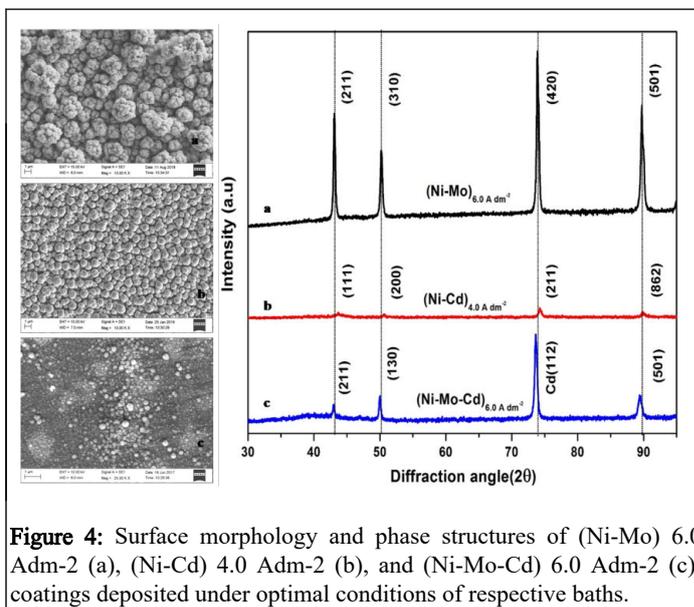


Figure 4: Surface morphology and phase structures of (Ni-Mo) 6.0 Adm-2 (a), (Ni-Cd) 4.0 Adm-2 (b), and (Ni-Mo-Cd) 6.0 Adm-2 (c), coatings deposited under optimal conditions of respective baths.

Effect of c.d.

Variation in wt. % of M(metal) content (Ni, Mo and Cd) in binary and ternary alloy coatings over range of c.d. studied is shown in Figure 5. It may be noted that in Ni-Mo alloy coating, Ni content of the deposit is slowly decreasing with increase of c.d., as expected in induced type codeposition. Corrosion data, reported in Table 2 clearly shows that c.d. slightly effects the Ni content, and hence corrosion resistance of the alloy coating as mentioned earlier. But in case of Ni-Cd alloy coatings, Ni and Cd content has large dependency with c.d. A sudden increase of Ni content was found in the range of 2.0 - 4.0 Adm-2 (Figure 5). The maximum Ni (noble metal) content

corresponding to 4.0 Adm-2 indicates that it is its approximate limiting current density for deposition of Ni in that bath. However, it is important to note that in ternary Ni-Mo-Cd alloy coating Ni content tends to decrease gradually (or increase of Cd) with c. d., as shown in composition vs c.d. plot in Figure 5.

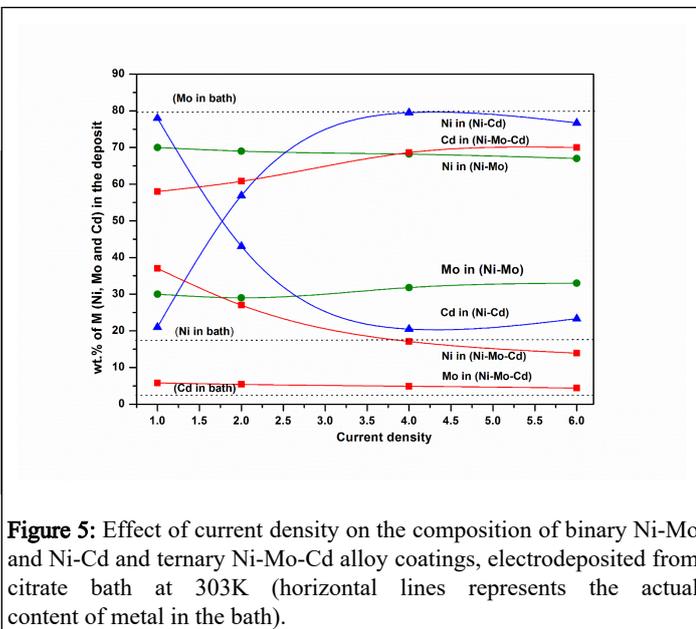


Figure 5: Effect of current density on the composition of binary Ni-Mo and Ni-Cd and ternary Ni-Mo-Cd alloy coatings, electrodeposited from citrate bath at 303K (horizontal lines represents the actual content of metal in the bath).

Effect of addition of Cd+2

The essence of electroplating is charge transfer across the electrode-electrolyte interface, and most importantly the composition of deposit depends on the i_L of ions involved. Therefore, a profound understanding of the structure of Electrical Double Layer (EDL) is important in electroplating, in terms the effect of c.d. on the diffusion of ions and thickens of the EDL. It is well known that in regular codeposition, effects of plating variables on composition of the deposit are determined by changes in the concentrations of metal ions in the cathode diffusion layer, and is predictable from simple diffusion theory. Therefore, at i_L , the rate at which metal ions in Ni-Mo-Cd alloy can deposit may be determined by diffusion and electrical migration of the ions, and is proportional to their concentrations. Thus by taking the advantage of diffusion theory based i_L of metal ions, it is possible to control the Ni content of the alloy coatings, by proper modulation of charge transfer process at EDL. In this study, the i_L of Ni was decreased to large extent by addition of small quantity of Cd+2 ions into Ni-Mo bath. The CdCl₂ added in small quantity (1 g/L) decreased the i_L of Ni, and allowed the deposition of ternary alloy coating of high corrosion resistance as explained below.

It may be recalled that i_L of a metal is the current density at which rate of its deposition is maximum. At i_L the concentration of metal ions near the electrode drops to zero, and it depends only on the bulk concentration. Therefore, i_L of a metal in an electrolytic bath may be written in the form of Eqn. (1),

Where n is the number of electrons per ion being transferred, F the Faraday constant (96,490 C/equivalent). CB is the concentration of metal ions in bulk electrolyte and δ is the thickness of EDL (diffusion layer). From the above relation, it may be noted that in a given bath (as CB is constant), i_L is changing only by altering the thickness of the EDL. Thus, in the light of above discussion, the inverse dependency of

Ni content with c.d. due to addition of Cd+2 ions into Ni-Mo bath may be attributed to its decreased i_L of Ni. Thus, Cd+2 ions reduced the i_L of Ni greatly in Ni-Mo-Cd bath, in relation to that in (Ni-Mo) bath. In the present study, it may be concluded that wt. % of Ni in the deposit has decreased due to decrease of its i_L , due to addition of Cd+2 ions, and allowing the bath to undergo regular ternary alloy deposition. The decrease of i_L of Ni in Ni-Mo-Cd alloy bath is due to change in the charge transfer process at the EDL, affected due to different complexation of process compared to that in binary baths. Hence, least corrosion rate of ternary Ni-Mo-Cd coating is due to decreased i_L of Ni due to addition of Cd into bath. Hence, it may be noted that the corrosion performance of binary and ternary alloy coatings need not always depends more on the i_L of noble metal, rather than the wt.% of noble metal in the deposit. Therefore, it may be concluded that Cd+2 ions acted as such agency to decrease the i_L of Ni by increasing thickness of EDL.

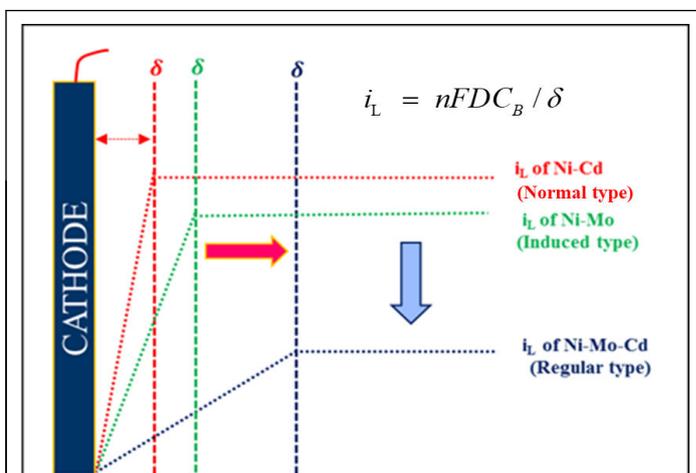


Figure 6: Representational diagram showing the decrease of limiting current density (i_L) of Ni responsible for regular type of codeposition in ternary Ni-Mo-Cd alloy plating affected due to addition Cd into Ni-Mo bath (induced type of codeposition), in relation to i_L of Ni-Cd bath (normal type of codeposition).

Representational diagram showing the decrease of i_L of Ni in Ni-Mo-Cd bath affected due to addition Cd+2 ions into Ni-Mo bath is shown in Fig. 6, in relation to its value in Ni-Cd bath, following normal codeposition; and Ni-Mo bath, following induced type of codeposition. It may be seen from Fig. 6 that the thickness of EDL (δ) corresponding to Ni-Mo-Cd bath is much larger than that for Ni-Mo and Ni-Cd baths. This increase of δ is due to change in the charge/mass transfer process at EDL due to addition of Cd+2 ions into the bath.

Conclusion

The experimental investigation to understand the effect of addition of Cd into Ni-Mo bath on the corrosion protection efficacy of Ni-Mo-Cd alloy coatings, following observations were made as conclusions: Ni-Mo-Cd alloy coatings of better corrosion performance, than both Ni-Mo and Ni-Cd alloy coatings can be developed on addition of small quantity CdCl₂ into Ni-Mo bath. The limitation of high limiting current density (i_L) of Ni of both Ni-Mo and Ni-Cd baths, due to inherent induced and normal type of codeposition has been successfully alleviated by adding of CdCl₂. The advent of induced and

normal type of codeposition of Ni-Mo and Ni-Cd baths, respectively have been used advantageously to optimize the Ni content of the ternary alloy for better corrosion stability. The composition versus current density (c.d.) plots of alloy coatings have been studied, and optimal i_L of Ni in three baths were assessed. Experimental investigation revealed that (Ni-Mo-Cd)6.0 Adm-2 coating shows highest corrosion resistance due to decrease of i_L of Ni, affected due to addition of Cd²⁺ ions into the baths. The decrease of i_L of is due to change of charge transfer process at the electrical double layer (EDL), affected by the addition of Cd ions into the bath. The corrosion performance of binary and ternary alloy coatings always depends more on the i_L of noble metal, rather than the wt.% of noble metal in the deposit.

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