Production of layered coatings of Fe-Ni alloy for enhanced corrosion protection

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Layered Fe-Ni alloy coatings have been galvanostatically developed on copper using dual and triple square current pulse from acid sulfate bath. The cyclic cathode current density (CCCD) and number of layers have been optimized for enhanced performance of the coatings against corrosion. Corrosion behavior of the coatings is evaluated by potentiodynamic polarization and electrochemical impedance spectroscopy methods in 1M HCl solution. The polarization study confirms that coatings developed using triple square pulses are more corrosion resistant than those deposited by dual square pulses. The improved corrosion behavior of layered coatings is due to the changed intrinsic electric properties evidenced by the electrochemical impedance spectroscopy. Under optimal conditions, the coatings developed using dual and triple square current pulse have been found to be by about 23 and 57 times, respectively, more corrosion resistant than the monolithic alloys. Surface morphology and layered coatings have been examined by scanning electron microscopy. Saturation of corrosion resistance at a high degree of layering was found, which is attributed to a shorter relaxation time for redistribution of metal ions during plating.

Keywords: CMMA, Fe-Ni, corrosion resistance, intrinsic electric property.

УДК 541.138

INTRODUCTION

One of the promising areas in nano-materials research is electrodeposition of metals/alloys with a spatially periodic composition in alternate passion (explain it, please, or use a synonim). These types of materials are known as a composition modulated multilayer alloy (CMMA) coating [1]. In CMMA coatings, thin layers of pure metals/alloys exhibit a wide range of applications owing to their unique technological properties, different from pure metals and homogenous alloys. In addition, due to the increased effect of surface or interface arising from the exceptional thinness of the layers, there can be large deviations from bulk behavior, and leads to promising mechanical strength, wear, elasticity, ductility, electrical, optical and corrosion properties for CMMA coatings [2].

There are different methods for producing CMMA coatings; physical vapor deposition (PVD), chemical vapor deposition (CVD) and electrodeposition etc. Electrodeposition was revived as a method of multilayer fabrication by Brenner's work on copper/bismuth multilayers with micron scale repeated lengths [3]. Electrodeposited CMMA can be produced by two methods, known as single-bath techniques (SBT) and dual-bath techniques (DBT). In SBT, the layers are obtained in one bath, and the alloy composition can be controlled by changing the cathodic current density (c.d.). The DBT is carried out from separate plating baths by transfer of the substrate from one bath to another. However, this technique is more time-consuming. Moreover, an oxide layer can be formed on the substrate during transport between the baths, which is another drawback of this method [4–5]. Both techniques are known to have their own advantages and disadvantages. In most cases, the drawbacks of the DBT have been considered to outweigh the benefits, so that the SBT approach is used instead.

Multilayers have been extensively studied because their properties differ from those of their bulk constituent owing to the two-dimensional structure of the film, and the high density of interfaces. The multilayer structures typically characterized by many successive layers, achieved by repeating length or period of deposition. The repeating length or period of deposition and modulation of applied c.d. are two parameters often used to define the periodic structure of CMMA coatings. The repeating length of deposition represents the thickness of each layer, and the applied c.d. is a measure of the interlayer composition variation. The engineering of alloys with specific properties, therefore, depends on the ability to tailor the compositional structure through the control of processing variables. Hence the composition and thickness of each individual layer can be manipulated to optimize the desired property of the deposit or coating. The overall property of the deposit is, then, a function of the individual components, the modulation thickness, as well as the structure of the interface.

Though gradation in composition is possible through many means such as modulation in cathode c.d., agitation, temperature etc., it can be better controlled with a great degree of accuracy and reproduc-

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ibility using microprocessor controlled power sources [6–7]. In principle, this technique is straightforward to fabricate multilayer alloy coatings. The multilayered alloy coating can also be developed from SBT using dual and triple square current pulses. Accordingly, it is possible to tailor the properties of coatings to meet the desired engineering application by proper manipulation of current/voltage pulse patterns. Considerable amount of work have already been reported to support the enhanced corrosion resistance of CMMA coatings of Zn-M (where M = Fe, Co and Ni) alloy using SBT [8]. Development of multilayered Zn-Ni alloy coatings, using square current pulses for the improved corrosion resistance have been reported [9]. But no work is reported with regard to development of corrosion resistant multilayer coatings of mutual alloys Fe-group metals, such as Fe, Ni and Co using dual and triple current pulses. In this direction, the present paper illustrates the optimization of deposition condition for the development of layered Fe-Ni alloy coatings on copper using dual and triple current pulses, while demonstrating the improved corrosion protection, due to an increased number of interfaces.

1. MATERIALS AND METHODS

Plating solutions were prepared from reagent grade chemicals and distilled water. Electroplating was carried out at pH 3.5 and temperature was maintained at 303 K. Copper panels having an exposed area of 7.5 cm^2 were used as the cathode, and pure nickel plate with the same exposed anodic area. Before electroplating the copper substrates were polished mechanically and then cleaned electrochemically. A sulphate bath having $FeSO_4.7H_2O$ (16 gL^{-1}), $NiSO_4$. $6H_2O$ (100 gL^{-1}), H_3BO_3 (30 gL^{-1}), L-Ascorbic acid (8 gL^{-1}), and sulphanilic acid (1 gL^{-1}) was used for the deposition of Fe-Ni alloy coatings.

All coatings, viz. monolayer and CMMA were carried out using power source (N6705A, Agilent Technologies) for 10 minutes. Electrochemical studies were performed using Potentiostat/Galvanostat (VersaSTAT^3, Princeton Applied Research) in a three-electrode configuration cell using a saturated calomel electrode (SCE) as a reference electrode. The corrosion behaviors of the coatings were evaluated, in 1M HCl solution by potentiodynamic polarization and Electrochemical Impedance spectroscopy (EIS) method. Polarization study was made in potential limit of ± 250 mV from the open circuit potential (OCP), at scan rate of 1 mVs^{-1}. EIS studies were carried out in the frequency range, from 100 KHz to 10 mHz with perturbing signal of 10 mV. Scanning Electron Microscopy (SEM) with Energy dispersive X-ray (EDX) analyzer facility (Model JSM-6380 LA from JEOL, Japan) was used for examining the layer formation. The phase structures of the coatings were identified with X-ray Diffractometer (XRD) (JEOL JDX-8P), using Cu Kα (λ = 1.5406 Å) radiation in continuous scan mode at scan rate of 2°min^{-1}.

2. RESULTS AND DISCUSSION

2.1. Fabrication of Fe-Ni alloy coatings

Before developing the layered coatings, the effect of (c.d.) on composition of Fe-Ni alloys, consequently on its phase structure was initially investigated. The EDX technique, used for compositional analysis of the coatings revealed that monolayer Fe-Ni alloys show about 62–86 wt. % of Ni variation over range of c.d. studied i.e. 2.0–8.0 Adm^{-2}. The preferred orientation of electrodeposited Fe-Ni alloy coating at different c.d. was characterized by XRD analysis. The identification of the phases was obtained from the peak profiles of the X-ray reflection plotted as a function of 2θ is shown in Fig. 1.

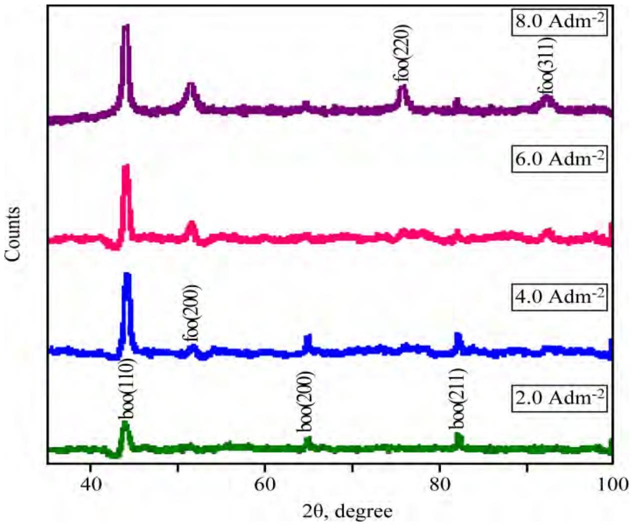

Fig. 1. X-ray diffraction peaks of Fe-Ni alloy deposited at different c.d. from same bath.

XRD signals of Fe-Ni coating at 2.0 Adm^{-2} showed single phase bcc related to Fe crystal structure. As the cathode c.d. increased, the wt. % of Ni in the deposit increased. Accordingly an increase in fcc phase structures corresponding to increased Ni content was observed. The XRD signals corresponding to 4.0 Adm^{-2} show an intermittent transitional phase from bcc to fcc. During this transition, the Fe content tends to decrease, while the Ni content tends to increase. Ni being nobler (please use the synonym) compared to Fe, a decrease of corrosion rate (CR) was observed to 4.0 Adm^{-2} as shown in Table 1. However, at a higher c.d. an increase of CR was observed. This is attributed to high porosity of the coatings, as a consequent to the increase in Ni content.

2.2. CMMA Fe-Ni alloy coatings

2.2.1. Optimization of cyclic cathode current densities (CCCD's)

Based on the composition studies of monolayer Fe-Ni alloy coatings, CMMA Fe-Ni alloys were electrodeposited using dual and triple square current pulses at different combinations of c.d.'s known as CCCDs. To find the corrosion resistance of Fe-Ni CMMA coatings, initially coatings were developed at different sets of CCCDs with 10 layers (arbitrarily chosen), and corresponding corrosion data are reported in Table 2. Among the various sets tried, the less CR was observed in coatings produced at 2.0–4.0 Adm^{-2} and 2.0–4.0–6.0 Adm^{-2} for dual and triple square current pulses, respectively, as shown in Table. 2.

Table 1. Corrosion data of monolayer Fe-Ni alloys deposited at different c.d.

c.d. /Adm^{-2}	$-E_{corr}$ vs SCE/V	i_{corr} /μA cm^{-2}	CR mm y^{-1}
1.0	1.0559	1644.5	18.49
2.0	0.9958	1046.9	11.77
3.0	0.8658	860.7	9.68
4.0	0.9137	650.3	7.40
5.0	0.8337	942.6	10.73
6.0	0.9341	1080.1	12.13
7.0	1.0775	1339.2	15.06
8.0	1.0275	1458.4	16.40

Table 2. Corrosion rates of Fe-Ni coatings at different sets of CCCDs (with 10 layers each)

Coating configuration	$-E_{corr}$ vs SCE/V	i_{corr} /μA cm^{-2}	CR mm y^{-1}
CMMA coatings using dual pulses			
2.0 Adm^{-2} difference			
$(Fe-Ni)_{2/4}$	0.887	229.43	2.57
$(Fe-Ni)_{4/6}$	0.859	267.47	3.00
$(Fe-Ni)_{6/8}$	0.860	284.63	3.20
$(Fe-Ni)_{3/5}$	0.883	333.96	3.75
4.0 Adm^{-2} difference			
$(Fe-Ni)_{2/6}$	0.993	306.47	3.44
$(Fe-Ni)_{2/6}$	0.869	395.18	4.44
$(Fe-Ni)_{2/6}$	0.993	426.20	4.79
CMMA coatings using triple pulses			
2.0 Adm^{-2} difference			
$(Fe-Ni)_{2/4/6}$	0.894	103.46	1.16
$(Fe-Ni)_{1/3/5}$	0.946	138.68	1.55
$(Fe-Ni)_{3/5/7}$	0.875	207.56	2.33
$(Fe-Ni)_{4/6/8}$	0.862	240.06	2.70
3.0 Adm^{-2} difference			
$(Fe-Ni)_{2/5/8}$	0.885	177.52	1.99
$(Fe-Ni)_{1/4/7}$	0.866	266.38	2.96

2.2.2. Optimization of number of layers

The properties of multilayer coatings including their corrosion behavior can be improved by a proper manipulation of layer configurations i.e. by controlling the nucleation process during successive layering. A reduced grain size and large number of interfaces may lead to the changed intrinsic electric properties of coatings. This may lead to a substantial improvement in corrosion resistance of the coatings, usually up to an optimal limit. Therefore by selecting 2.0–4.0 Adm^{-2} and 2.0–4.0–6.0 Adm^{-2} as two sets of CCCDs for dual and triple square current pulses, CMMA Fe-Ni alloy coatings having 10, 20, 60, 120, 300, 400 and 600 layers were developed, and their corrosion parameters are listed in Table 3.

Table 3. Corrosion rate of CMMA Fe-Ni with different number of layers

Coating configuration	$-E_{corr}$ vs SCE/V	i_{corr}/μA cm^{-2}	CR mm y^{-1}
$(Fe-Ni)_4$	0.9137	650.38	7.40
CMMA coatings using dual pulse deposited at 2/4 Adm^{-2}			
$(Fe-Ni)_{10}$	0.887	229.43	2.57
$(Fe-Ni)_{20}$	0.904	131.46	1.47
$(Fe-Ni)_{60}$	0.894	97.1	1.09
$(Fe-Ni)_{120}$	0.914	75.6	0.84
$(Fe-Ni)_{300}$	0.924	28.7	0.32
$(Fe-Ni)_{400}$	0.913	46.3	0.52
$(Fe-Ni)_{600}$	0.914	54.1	0.60
CMMA coatings using triple pulse deposited at 2/4/6 Adm^{-2}			
$(Fe-Ni)_{10}$	0.894	103.46	1.16
$(Fe-Ni)_{/20}$	0.903	83.8	0.94
$(Fe-Ni)_{/60}$	0.912	35.7	0.40
$(Fe-Ni)_{120}$	0.919	21.3	0.24
$(Fe-Ni)_{300}$	0.920	12.0	0.13
$(Fe-Ni)_{400}$	0.911	14.5	0.16
$(Fe-Ni)_{600}$	0.913	17.7	0.19

2.3. Potentiodynamic polarization studies

Potentiodynamic polarization behavior of CMMA Fe-Ni coatings deposited using triple square current pulse is shown in Fig. 2. It was observed that CR decreased with an increase in number of layers up to 300, and then increased at higher number of layers. The decrease of CR at a higher degree of layering is attributed to the lesser relaxation time for redistribution of metal ions (Fe^{2+} and Ni^{2+}) at the diffusion layer during the deposition. At high degree of layering, the deposition time for each layer is very small, and hence the metal ions could not relax (against diffusion under given c.d.), and get deposited on the cathode with different compositions [10]. Hence, at high degree of layering modulation in composition is not likely to take place. Consequently, CMMA deposit tends to become monolithic, showing less corrosion resistance.

Further, the shape of cathodic polarization curves in Fig. 2 reveals that the decrease of corrosion rate with an increase in the number of layers is due to suppression hydrogen evolution reaction. The exchange current density (i_0) equivalent to the reversible rate at equilibrium for reaction, $2H^+ + 2e \leftrightarrow H_2$ is found to be decreased as the number of layers increased. The CMMA coating having $(Fe-Ni)_{2/4/6/300}$ configuration is found to exhibit about 57 times higher corrosion resistance than monolayer

$(\text{Fe-Ni})_{4.0}$ alloy coating, developed from same bath. The improved corrosion resistance of the CMMA coatings may be explained in terms of the failures like pores and crevices occurring in the single layer will be covered by the successively deposited coating layers, and thus the corrosion process is blocked, or extended [11]. Hence, as the number of the layers increased, the corrosive agent needs more time to penetrate through the coating, and then into the substrate material than in the case of monolithic coating.

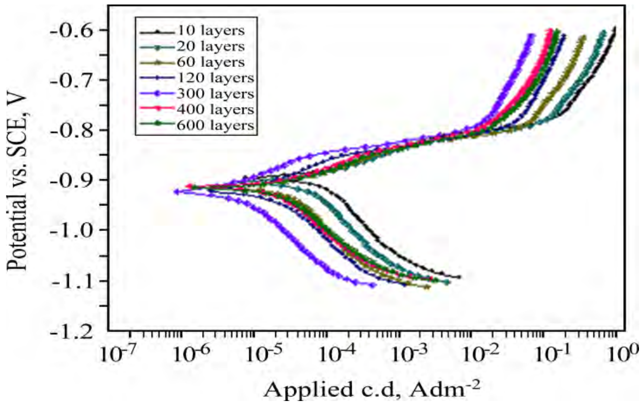

Fig. 2. Potentiodynamic polarization plots of CMMA Fe-Ni alloys deposited using triple square current pulse.

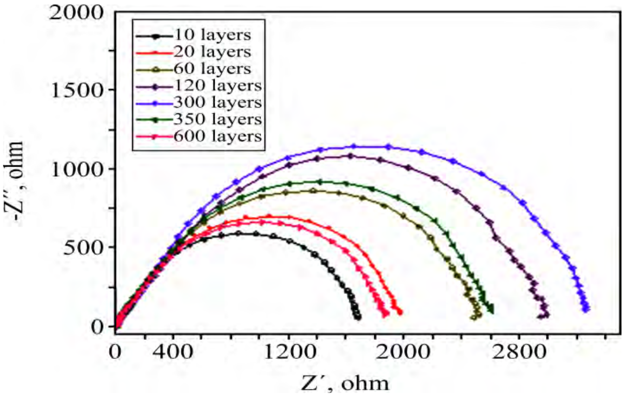

Fig. 3. Nyquist plots of CMMA Fe-Ni alloys deposited using triple square current pulse.

2.4. Electrochemical impedance study

EIS is one of the most powerful techniques for investigation and prediction of corrosion protection. EIS could provide information about the changes in coating and metal interface performance long before the visual changes can be observed using traditional exposure test [12]. The impedance behavior of CMMA Fe-Ni alloys, deposited using triple square current pulses is shown in Fig. 3. Impedance diagrams with only one depressive loop over the whole frequency range indicate that the capacitance of the double layer is responsible for improved corrosion protection of the deposits. Nyquist response of multilayer coatings indicated that the decrease of corrosion rate with layering is due to decrease of charge transfer resistance, R_{ct} between metal and medium.

Fitting the EIS plots of CMMA Fe-Ni alloy coating has attempted using different electrochemical equivalent circuits. The best agreement between experimental and fitting data was obtained with the equivalent circuit shown in Table 4 and Fig. 4. Two (RQ) parallel circuit combinations are used to represent the electrochemical activities of the oxide layer and the coating/solutions interface. In Fig. 4, R_{ct} and constant phase element (CPE) Y_{01} are the charge transfer resistance and the oxide layer capacitance. R_c and Y_{02} are the coating resistance and capacitance, or the resistance and capacitance of the space charge layer. The time constant at high-frequency range originated from the R_{ct} Y_{01} combination, while at low-frequency range, it initiated from the R_c Y_{02} combination. A CPE replaced the capacitance of the double layer (C_{dl}).

$$Z_{CPE}(\omega) = Y_0^{-1}(j\omega)^{-1} \tag{1}$$

where, Y_0 is a constant that is independent of frequency, ω is the angular frequency, $j = \sqrt{-1}$, and n is the exponential index which represents a dispersion of relaxation. When n equals 1, CPE represents an ideal capacitor; when n equals 0, CPE acts as a pure resistor. The mathematic expression of the impedance of the electrode system can be described by the following equation [12]:

$$Z_{CPE}(\omega) = j\omega L + R_s + \\ + \frac{1}{\frac{1}{R_{ct}} + Y_{01}(j\omega)^{n1}} + \frac{1}{\frac{1}{R_c} + Y_{02}(j\omega)^{n2}}. \tag{2}$$

The oxide layer resistance, R_c increased with the increase in number of layers and reached the maximum of 1709 Ω cm^2 for $(\text{Fe-Ni})_{2.0/4.0/6.0/300}$ then began to fluctuate. Increased R_c indicating the good corrosion protection properties. In addition, according to the following equation [13]:

$$c = \frac{\varepsilon\varepsilon^0}{d} \tag{3}$$

where, ε is the dielectric constant, ε^0 is the permittivity of free space, and d is the thickness of the film. A decrease in the capacitance (C) may result from either an increase in the film thickness or a decrease in the dielectric constant. A decrease in the dielectric constant was consistent with a decrease in the defect density within the film [13]. Within the course of EIS test, the changes in the capacitance (C), accompanied by a fluctuation in film resistance R_c, may result from the different variation extent in the film thickness and defect density.

2.5. Morphological studies

Surface morphology and formation of layers are shown in Fig. 5(A) and 5(B) respectively. The high

Table 4. Equivalent circuit fitment data of CMMA Fe-Ni with different number of layers

Coating configuration	*L* (Hcm^2) × 10^{-6}	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	Y_{01} (Ω^{-1}cm^{-2}S^{-n}) ×10^{-6}	*n*1	R_c (Ωcm^2)	Y_{02} (Ω^{-1}cm^{-2}S^{-n}) ×10^{-5}	*n*2
CMMA coatings using dual pulse deposited at 2/4 Adm^{-2}								
(Fe-Ni)$_{10}$	0.166	16.42	793.4	0.206	0.7	141.9	1.501	0.8
(Fe-Ni)$_{20}$	7.001	5.76	1135	0.200	0.8	136.5	1.370	0.8
(Fe-Ni)$_{/60}$	7.461	7.28	1673	0.145	0.8	188.9	1.029	0.8
(Fe-Ni)$_{120}$	1.138	9.69	1746	6.844	0.8	225.0	0.823	0.8
(Fe-Ni)$_{300}$	1.518	12.92	2218	5.156	0.8	306.2	0.764	0.8
(Fe-Ni)$_{400}$	7.465	8.09	1174	6.751	0.9	33.0	1.917	0.7
(Fe-Ni)$_{600}$	4.447	2.95	841.8	0.267	0.8	55.1	1.808	0.8
CMMA coatings using triple pulse deposited at 2/4/6 Adm^{-2}								
(Fe-Ni)$_{10}$	6.545	5.81	1616	0.218	0.8	88.05	1.341	0.8
(Fe-Ni)$_{20}$	6.906	6.22	1862	0.141	0.8	130.1	0.944	0.8
(Fe-Ni)$_{60}$	1.188	10.41	2370	0.116	0.8	153.1	0.718	0.8
(Fe-Ni)$_{120}$	1.757	9.44	3154	0.161	0.8	502.0	0.691	0.8
(Fe-Ni)$_{300}$	1.271	11.29	3137	0.107	0.8	1709	0.013	0.8
(Fe-Ni)$_{400}$	9.087	8.18	2449	0.112	0.8	171.4	0.718	0.8
(Fe-Ni)$_{600}$	8.451	4.37	1979	0.238	0.8	63.6	0.700	0.7

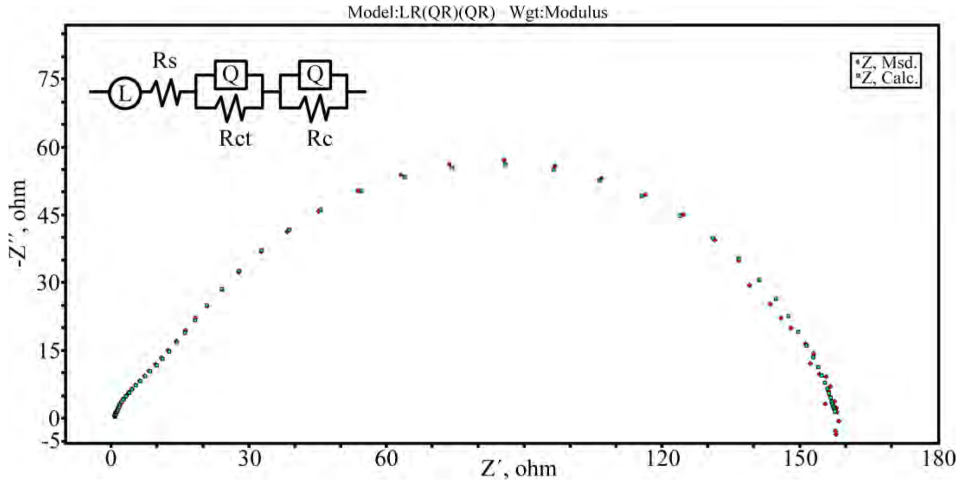

Fig. 4. Equivalent circuit fitment of CMMA Fe-Ni alloys deposited using triple square current pulse.

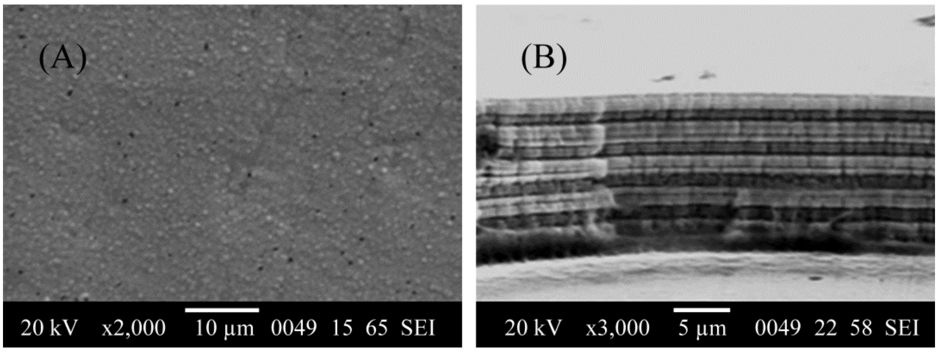

Fig. 5. SEM image showing (A) surface morphology and (B) formation of layers.

corrosion prevention of multilayer coating is due to the fact that one layer of alloy having one type of failures (like pores, crevices or columnar structure), deposited at one c.d. will be covered successively by another layer of alloy having different composition than the previous one, as shown in Fig. 5(B). Thus the coating possesses alternate layers having differrent degree of failures, and thus the corrosion agents' path is longer or blocked.

3. CONCLUSIONS

1. The multilayer Fe-Ni alloy coating showing excellent corrosion resistance were successfully deposited on copper using dual and triple square current pulse.

2. XRD study of monolayer Fe-Ni alloys revealed that at low c.d. volume fraction of bcc predominates and at high c.d. fcc predominates. There exists an intermittent transitional phase from bcc to

fcc. During this transition, the Fe content tends to decrease and Ni content tends to increase.

3. The corrosion resistances of coatings was found to increase with the number of layers up to a certain degree of layering, and then decreased. The decrease of corrosion resistance at high degree of layering is attributed to the lower relaxation time for redistribution of solutes in the diffusion double layer, during plating.

4. The results of SEM analysis confirm the formation of layers. Further, the corrosion protection of CMMA (Fe-Ni)$_{2.0/4.0/6.0/300}$ coatings having more number of layers is due to successive deposition of layers having different compositions. Consequently, corrosive agent path is supposedly extended or blocked. However, the decrease of corrosion protection at more number of layers is due to diffusion of layers.

5. The electrochemical corrosion study showed that the corrosion resistance of CMMA (Fe-Ni)$_{2.0/4.0/6.0/300}$ coating is ~ 57 times higher than that of monolayer Fe-Ni obtained from the same bath for the same time.

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Реферат

Многослойные Fe-Ni покрытия были получены на меди гальваностатическим способом с применением двойного и тройного квадратного импульса тока из серной кислоты. Плотность циклического катодного тока (ПЦКТ) и количество слоев было оптимизировано в целях повышения устойчивости покрытия к коррозии. Коррозионное поведение покрытий оценивалось методами потенциодинамической поляризациии и электрохимической импедансной спектроскопии (ЭИС) в 1М HCl растворе. Изучение поляризации показало, что покрытия, образованные при тройных квадратных импульсах, являются более коррозионностойкими, чем полученные при двойных квадратных импульсах. Улучшенное коррозионное поведение многослойных покрытий является результатом изменения собственных электрических свойств, что подтверждается ЭИС. При оптимальных условиях покрытия, созданные с использованием двойных и тройных импульсов тока, превышают коррозийную стойкость монолитного слоя примерно в 23 и 57 раз, соответственно. Морфология поверхности и многослойные покрытия были изучены с использованием сканирующей микроскопии (СЕМ). Было обнаружено насыщение коррозионного сопротивления при высокой степени наслаивания, что объяснялось меньшим временем релаксации для перераспределения ионов металла в процессе покрытия.

Ключевые слова: CMMA, Fe-Ni, коррозионная стойкость, собственные электрические свойства.