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Electrochemical deposition and characterization of nanocrystalline Fe-Ni coatings

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1. Introduction

Mild steel is an excellent construction and building material for less cost and easy processing. However, its inherent corrosion behavior received much attention in both industrial and academic arenas. Of many techniques available, formation of a thin adherent film on the surface of the metal is most effective in corrosion prevention of mild steel [1-5]. Research on coatings and surface modification of mild steel is dominated by zinc based deposits due to their easy formability and cost effectiveness. Extensive research has been carried out in this direction. Zn-Ni and Zn-Co codeposits possess the properties capable of replacing cadmium and chromium based toxic coatings [6-8].

But the main drawback of Zn-based coatings is their low hardness and mechanical processability. This has led the industry to seek new coatings that are better in physico-mechanical properties and economically viable for protection of mild steel [7]. Fe based coating systems have attracted much attention mainly due to their cost effectiveness and properties. Fe-Ni alloy exhibits excellent corrosion resistance, magnetic properties, ductility and appearance [8-10]. It has been widely used in the area of memory devices for computers, laser components and precise instruments [11]. Electrodeposition is the most popular method used to prepare Fe-Ni alloy, since it allows easier control of thickness and availability of small crystal size compared with the conventional rolling processes [12]. The binary alloys of

iron group metals, namely Fe-Ni, Co-Fe and Ni-Co electrodeposition exhibits peculiar phenomenon of 'anomalous codeposition'. This term introduced by Brenner is being used to describe the preferential deposition of the less noble metal than more noble metal [13-15, 22].

Research on Fe-Ni alloys is mainly focused on their deposition behavior with variation of plating parameters like complexing agent, operating temperature, pH and composition on the final properties of the films formed. The properties of the materials are known to change with the change of their matrices from micro to nano scale. Recently, research on obtaining nanostructured coatings of Fe-Ni alloys is gaining importance among researchers. Literature on nanocrystalline deposits of Fe-Ni alloy with extensive study on their properties is not widely available [16-19].

In present study, we report the experimental process and proceedings of electrodepositing nanocrystalline Fe-Ni alloy on mild steel substrates. The physico-mechanical and electrochemical properties of the materials like phase structure, surface morphology, thickness, hardness and corrosion protection ability were analyzed and results are reported. The factors leading to observed behavior are discussed.

2. Materials and Methods

Plating solutions were prepared from reagent grade chemicals and distilled water. All depositions were carried out at 303 K. MS plates of 50 mm × 20 mm × 2 mm were used as substrates (cathode), and pure nickel plate of same dimension used as anode with same exposed anodic area. The MS substrates were polished mechanically and then cleaned electrochemically. An acid sulfate solution was used as the electrolyte for deposition of Fe-Ni films. Bath composition and

operating parameters used in the present study is shown in *Table 1*. Ascorbic acid (AA) was used as antioxidant (to avoid anodic oxidation of Fe⁺² to Fe⁺³) and Sodium lauryl sulphate (SLS) as surfactant.

Tab. 1: Optimized bath composition and operating parameters

Component	Concentration (g/L)	Operating parameters
NiSO ₄ ·6H ₂ O	100.0	Cathode: Mild Steel (MS)
FeSO ₄ ·7H ₂ O	25.0	Anode: Pure nickel
H ₃ BO ₃ (BA)	20.0	Temperature: 303 K
Ascorbic acid (AA)	5.0	pH: 3.5
Sodium lauryl sulphate (SLS)	1.0	c.d.: 1.0 to 7.0 Adm ⁻²

The initial studies were focused on optimization of bath through standard Hull cell method, at 2A cell current, pH 3.5 and temperature 303 K. Varieties of deposits having grayish white/bright/semi bright/porous/black powdery appearance were obtained over wide range of c.d. 1.0 - 7.0 Adm⁻². Effect of each constituent, including pH and c.d. were tested while keeping other variables constant. The Hull cell panels were examined for their appearance, brightness and surface morphology and the one with best visual appearance was taken as the optimal. Thus optimal bath composition and operating parameters are arrived by standard Hull cell method [20].

The Mild steel (MS) plates were pickled with acid and cleaned with distilled water just before dipping into electrolyte. PVC cell of 200 ml capacity was used as plating bath with 5 cm separation between electrodes. DC Power Analyzer (Agilent N6705) was used for all electrochemical deposition of the coatings. The deposition was carried out from 1.0 to 7.0 Adm⁻² cathodic c.d. for 600

seconds in all cases for comparison purpose. The pH of bath solution before and after every deposition was measured and the change of pH was recorded (μpH System-362, Systronics). Solution pH was adjusted to 3.5, using dil. H_2SO_4 /dil. NaOH . The MS panel, after deposition was rinsed with distilled water and then air dried. The thickness of each coating was calculated from the weight of deposit, using Faraday's law. The hardness of the deposit was measured by Vickers method using Microhardness Tester (CLEMEX).

Current efficiency (C.E.) at each applied c.d. was calculated using the formula

$$\text{C.E.} = \frac{\text{Measured mass gain}}{\text{Theoretical mass gain}} \times 100 \quad [1.1]$$

Theoretical mass gain could be obtained by

$$\text{Theoretical mass gain} = \frac{Eq.wt \times I \times t}{96485} \quad [1.2]$$

Where *Eq.wt* is equivalent weight of the alloy, *I* is the total current (strength) passed and *t* is the deposition time.

The corrosion behaviors of coatings were evaluated in 5% HCl and 5% KOH medium by potentiodynamic polarization and EIS techniques, using Potentiostat/Galvanostat (VersaSTAT3, Princeton Applied Research), keeping open to air at room temperature. The media were selected to replicate aggressive acid and alkaline environments in lab conditions and are in accordance with ASTM standards for laboratory corrosion tests. A saturated calomel electrode (SCE) was used as reference electrode. Polarization study was carried out in a potential ramp of ± 250 mV from open circuit potential (OCP) at scan rate of 1 mVs^{-1} . EIS signals were recorded using AC signal of 10 mV amplitude, at a frequency range from

100 KHz - 100 mHz. Surface morphologies and compositions of the Fe-Ni alloy coatings were examined using FESEM, interfaced with EDXA facility (Carl Zeiss Neon 40). The phase structures of the coatings were identified with XRD (JEOL JDX-8P), using $\text{Cu K}\alpha$ ($\lambda = 1.5406 \text{ \AA}$) radiation in continuous scan mode at scan rate of 2° min^{-1} .

3. Results and Discussion

3.1. Alloy composition

Standard Hull cell method was used to optimize the chemical combination/composition and operating parameters for galvanostatic deposition of uniform and bright Fe-Ni alloy coatings on mild steel panels. It was found that c.d. plays a very important role in deciding the appearance and thickness of the deposits. Ascorbic acid (AA) was used to impart stability to the bath by preventing the oxidation of Fe^{+2} to Fe^{+3} . Boric acid (BA) was used as a buffering agent to prevent variation of pH during the deposition. Sodium lauryl sulphate (SLS) was included to decrease the surface tension of the electrolyte, consequently served as a grain refining and brightening agent by improving the surface interaction at metal-electrolyte interface. The deposit over wide range of c.d. (from 1.0 to 7.0 Adm^{-2}) on Hull cell panel was found to be bright, except at extremely high c.d., and chemical composition of electroplated coatings were determined by EDX analysis. The variation in the wt. % of Fe in the deposit at different c.d. is shown in *Figure 1*. The amounts of Ni and Fe in the bath are found to be 81.6% and 18.3% respectively, shown by horizontal lines in *Figure 1*. It may be noted that wt. % Fe in the deposit at all c.d. is much higher than in the bath (lower horizontal line) indicating that the bath follows anomalous type of codeposition, as envisaged by Brenner [14]. The increase of Fe content with c.d. up to 4.0 Adm^{-2} indicates that the bath tends

to deposit in normal type, and further decrease is due to anomalous type of codeposition. The increased anomalous type of deposition at high c.d. is due to depletion of more readily depositable metal (Fe) at cathode film.

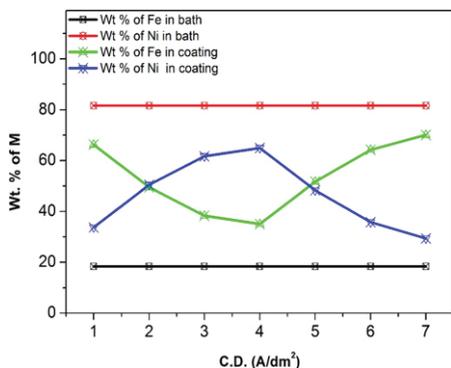


Fig.1: Variation of metal contents in the deposit with applied c.d.

3.2. Cathode current efficiency, thickness and hardness

Cathode current efficiency (CCE) is an important parameter in galvanostatic deposition of metals/alloys. In all deposition conditions, calculated CCE is above 80%, which is in terms with commercial requirement. Usually in the absence of conduct-

ing salt, the CCE values are expected to be less than 70%. However in present composition, the lack of conducting ions is fulfilled by high concentration of SO_4^{2-} ions. Addition of SLS has contributed to improve the CCE by reducing the hydrogen evolution. Thus the synergistic effect of all the components resulted in high CCE of the bath.

The thickness of the films formed at different c.d. is reported in *Table 2*. Following to high CCE, a bright and sound deposit was obtained at all c.d.. The thickness of the coatings were measured using thickness tester (Coatmeasure-400), verified by calculation using Faraday Law. It was found that coating thickness varied as a function of applied c.d., and observed a maximum thickness of 16 μm at 7.0 Adm^{-2} . The highest rate of film formation of present electrolyte was found to be $\sim 96 \mu\text{m} / \text{hour}$, which is in agreement with industrial deposition processes.

The hardness of the coatings was evaluated by Vickers method. The coatings were found to possess high hardness values as the requirement for many advanced applications. A comparative account of hardness of the electrodeposited Fe-Ni alloy coatings at different c.d. are given in *Table 2*. It may be noted that hardness of Fe-Ni coatings

Tab. 2: Physico-mechanical characteristics of Fe-Ni coatings at different c.d. from same bath

C.d. Adm^{-2}	Eq. Wt. of the alloy	CCE	Wt. of deposit in 25 cm^2 (mg)	Vickers micro hardness (V_{500})	Thickness (μm)
1.0	28.39	91.3	40.2	280	10
2.0	28.62	92.4	82.2	325	10
3.0	28.78	94.1	126.3	340	11
4.0	28.83	94.4	169.2	500	12
5.0	28.59	91.3	202.4	435	14
6.0	28.42	87.1	230.9	440	14
7.0	28.33	85.9	264.8	450	16
Hardness of pure Ni: 240 V and pure Fe: 210 V					

is much higher than individual Fe and Ni coatings deposited from same electrolyte, leaving the other salt, respectively. It suggests that alloy formation has bestowed with better properties due to different phase structure of the alloy. It is to be noted that the weight of deposits is obviously proportional to total charge passed (applied c.d.). However, towards high c.d., the amount of the alloy deposited has decreased due to increased evolution of H_2 gas, and hence CCE decreased. However increase of thickness at high c.d. is due to increased metal hydroxide formation at cathode film as explained by Brenner [14]. In other words, the coating is more porous.

3.3. Surface morphology

The Hulls cell panels are screened by visual inspection in the beginning. Coatings at all c.d. appear bright and uniform. The surface morphology of representative coatings at very low (1.0 Adm^{-2}) and medium (4.0 Adm^{-2}) is shown in *Figure 4*. The topographical image of the coatings demonstrates the basic principles of electrocrystallization process taking place during plating. Crystallization in an electrodeposition process usually involves two steps. The first step is about the discharge of metal ions and the generation of metal atom. There are two scenarios in the second step: (1) the incorporation of metal atom into the crystal and crystal growth; and (2) the formation of new nucleus when the rate of crystal growth may not be sufficient to cater for generation of atom. It has been proved that the critical radius of the surface nucleus, r is a function of the overpotential [23]. The overpotential is proportional to the applied c.d. and hence effects the grain size. High Fe content in the deposits causes better grain refinement and reduction of grain size [12]. In coatings at low c.d., the surface is smooth because of more number of smaller grains. This could be attributed to better nucleation at low c.d. values.

Available adatoms will majorly contribute to nucleation because of better relaxation time. The dendrite like nano crystals in *Figure 2a* are similar to dendritic structures observed in undercooled Fe-Ni alloys. At 4.0 Adm^{-2} , grain size being in nano range, an agglomerated spheres were observed in the coatings. The crystal structure is transforming from needle like to spherical shape. At higher c.d. (7.0 Adm^{-2}), a discrete spherical crystals were found (not shown in *Figure 2*). It may be attributed to fact that surface is flooded with new ions at higher rate. Due to less relaxation time for nucleation process, the ions preferentially grow on existing crystals thereby increasing their size. This results in roughness of the coatings.

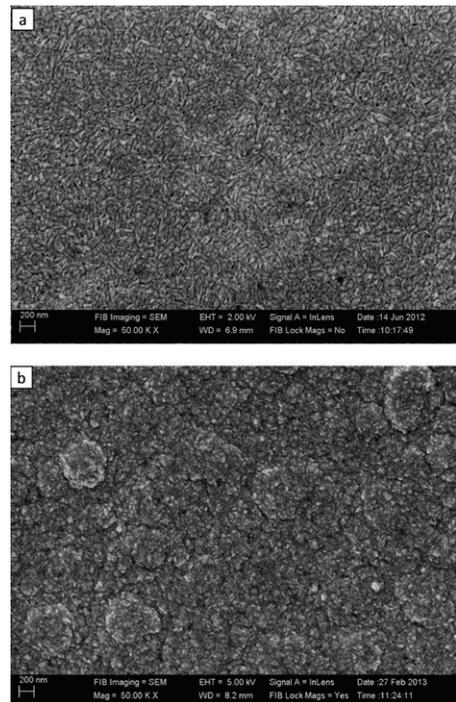


Fig. 2: FESEM micrographs of Fe-Ni coatings at:
a) 1.0 Adm^{-2} and
b) 4.0 Adm^{-2}

3.4. XRD analysis

The phase composition of the nanocrystalline Fe-Ni alloys was determined by powder XRD analysis. The characteristic XRD peaks of coatings deposited at different c.d. are shown in *Figure 3*.

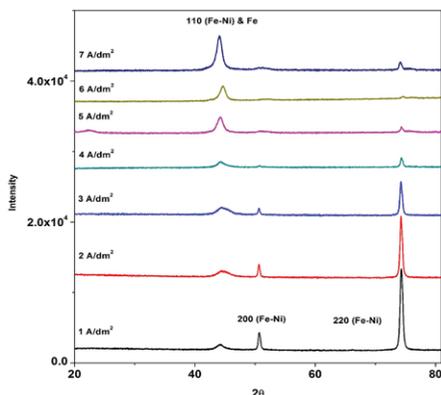


Fig. 3: Powder XRD patterns of Fe-Ni coatings at different c.d.'s from same bath.

It is clearly seen that c.d. influences significantly the phase composition of the coatings. At low c.d. the (220) and (200) FCC phases of Fe-Ni alloy are dominant in the coating. There is also presence of small amount of pure Fe phase. As the c.d. is increased, gradual decrease in FCC phases are observed, and the (110) BCC phase of Fe-Ni along with pure Fe phase gain intensity. At high c.d., the coatings contain very small amount of FCC phase. It could be visualized as shift of preferential orientation from FCC phase to BCC phase. As Ni-Fe form solid solutions the texture coefficient is known to vary with applied c.d.. At high c.d., the coatings contain very small amount of FCC phase. The reason for phase change could be attributed to rate and mechanism of nucleation which decide the coating phase structure. Thus, c.d. plays a decisive role in the phase composition of the coatings as it is dictated by the rate of nucleation [12, 15]. It may be noted that the dominant

(220) and (200) FCC phase of Fe-Ni alloy observed at low c.d. range gradually diminished as the c.d. increased, and was replaced by (110) as shown in *Figure 3*. The grain sizes were calculated using the Full Width at Half Maximum (FWHM) values and size distribution was found to be 6-24 nm for all the coatings.

3.5. Corrosion study

3.5.1. Potentiodynamic polarization study

The property of a material changes when its size is reduced from micrometric to nanometric scale. Corrosion being a surface phenomenon, is also affected by such change. The nanocrystalline coatings were subjected to lab scale corrosion tests in acid and alkali media by potentiodynamic polarization method in voltage ramp of ± 250 mV around the open circuit potential. The corrosion behaviors of the coatings were evaluated in 5% HCl and 5% KOH. The corrosion rates (CR) expressed in mm per year (mmy^{-1}) was determined by Tafel's extrapolation method and all CR values were calculated with error margin of $\pm 2\%$. The polarization behaviors of the coatings in 5% HCl and 5% KOH are shown in *Figure 4a & 4b* respectively, and corresponding corrosion data are shown in *Table 3*. In both cases, a positive open circuit potential (OCP) value was observed, suggesting that the coatings are less prone to undergo dissolution [12, 13, and 15]. In 5% HCl acid medium, coatings at low c.d. are more prone to corrosion, and passivation at anodic region may be due to oxidation. This may be attributed to phase transformation observed at these c.d. and the finer grain size. The corrosion rate is lowest (2.19 mmy^{-1}) for coating at 6.0 Adm^{-2} . The finer interface presented to corrosive media and the presence of nickel rich preferential orientations at this c.d. is the reason for decreased corrosion rate.

It is important to note that in spite of the same composition of Fe-Ni alloy, deposited at 2.0 Adm^{-2} and 5.0 Adm^{-2} the coating exhibit different corrosion behavior as may be seen in Table 3. Hence it may be inferred that the corrosion behavior of the coating is more a function of crystal lattice than the composition of the alloy, evidenced by corresponding XRD peaks in Figure 3. In 5% KOH, all the coatings exhibited a passive region in the anodic region as may be seen in Figure 4b. It is due to the fact that at extreme anodic polarization, the metal hydroxide (or metal oxides) formed due to combination of metal ions with hydroxyl ions is responsible for formation of passive film. Due to this reason, all Fe-Ni coatings are very stable, regardless of the c.d. at which they are deposited. Further, the potentiodynamic polarization behavior of all coatings in 5% KOH medium indicates that same corrosion protection mechanism holds good for all coatings deposited at different c.d.. The coating at 3.0 Adm^{-2} exhibited the least corrosion rate (0.096 mmy^{-1}) compared to coatings at other c.d..

Tab. 3: Corrosion parameters of Fe-Ni alloy coatings in 5% HCl and 5% KOH medium

c.d. (Adm^{-2})	E_{corr} (V vs SCE)	I_{corr} (mA/cm^2)	CR (mmy^{-1})
5% HCl medium			
2	0.009	2.46	27.96
3	0.081	2.51	28.52
4	0.051	1.07	12.23
5	0.225	0.76	8.43
6	0.346	0.22	2.19
5% KOH medium			
1	0.164	0.04	0.48
2	0.150	0.03	0.38
3	0.192	0.008	0.09
4	0.172	0.057	0.65
5	0.171	0.031	0.35
6	0.165	0.035	0.40

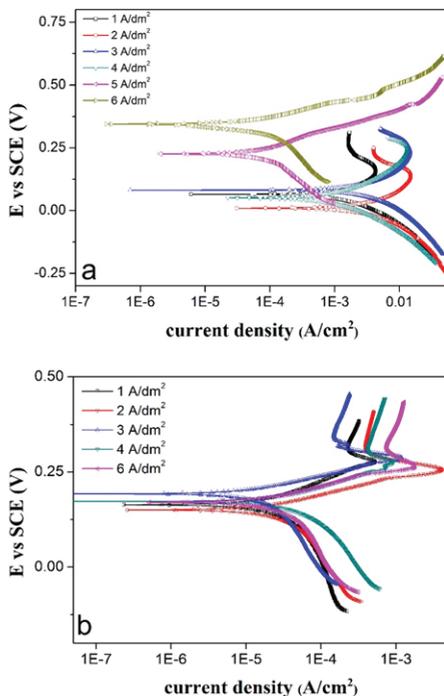


Fig. 4: Tafel plots for corrosion behavior of Fe-Ni coatings in a) 5% HCl and b) 5% KOH

3.5.2. Electrochemical Impedance Spectroscopy

Electrochemical impedance spectroscopy (EIS) provides information about the physical and chemical processes occurring on electrode surfaces and is useful to determine electrochemical information both qualitatively and quantitatively. The EIS study was carried out in the frequency range from 100 kHz to 100 mHz, using $\pm 10 \text{ mV}$ perturbing voltage at the open circuit potential using 5% HCl and 5% KOH solution, and the corresponding plots are shown in Figure 5. In present study, the Nyquist plots show different behavior in both acidic and alkaline medium, and hence corrosion rates were not deduced directly

from the plots. The corresponding data were fitted with equivalent circuits (using ZS_{imwin} Software) and the results of best fit are presented in *Figure 6a & 6b*. The possibility of more than one equivalent circuit for same plot hinders the deter-

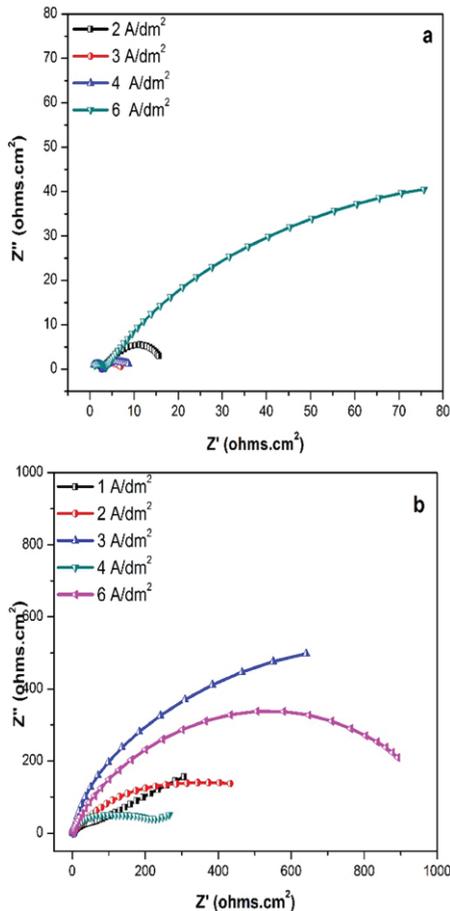


Fig. 5: Nyquist plots of nanocrystalline Fe-Ni coatings in a) 5% HCl and b) 5% KOH medium

mination of exact R_{ct} value and hence the plots of best fit circuits are only presented here. However, the impedance behaviors of the coatings are in compliance with experimental results obtained

by Tafel's extrapolation method. Further, the Nyquist plots corresponding to acid medium showed two capacitive loops indicating a separate corrosion mechanism different from that in alkaline medium.

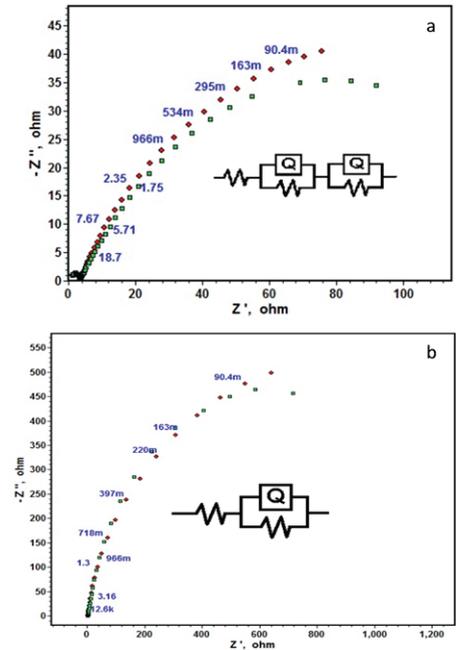


Fig. 6: Equivalent circuit fit results of Nyquist plots of Fe-Ni coatings obtained in a) 5% HCl and b) 5% KOH medium

Conclusions

1. A stable electrolyte composition for galvanostatic formation of Fe-Ni coatings on mild steel over a wide range of c.d. was deduced. At all c.d. the deposition followed anomalous behavior.
2. Electrodeposited coatings were subjected to different physico-mechanical and electrochemical tests. The alloy coatings were found to be harder and compact and CCE of deposition process was encouraging.

3. All coatings were uniform, free from cracks and grains were nanocrystalline as confirmed by FESEM analysis. Phase compositions of the coatings were found to be the function of applied c.d..
4. The corrosion behavior of the coating is more a function of crystal lattice than the composition of the alloy, evidenced by XRD peaks.
5. The coatings provided agreeable corrosion protection in 5% HCL medium. Highest corrosion rate observed at 6.0 Adm⁻² is due to presence of nickel rich preferential orientation in the phase composition of the alloy.
6. High corrosion resistance of Fe-Ni coatings deposited at 3.0 Adm⁻² in 5% KOH medium may be due to the formation of passive oxide film evidenced by anodic polarization curves.

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