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ABSTRACT

Nanostructured Ni-Fe alloy coatings was developed on mild steel panels using electrolytic sulphate bath. The composition of the electrolytic bath and the experimental parameters were optimized. Corrosion behaviour of the developed Ni-Fe alloy coatings were evaluated in 3.5% NaCl solution using potentiodynamic polarization and electrochemical impedance spectroscopic techniques. Highest corrosion resistance was observed for coatings at 3 A dm⁻². Surface Morphology of the coatings was also characterised by SEM.

Keywords: Ni-Fe alloy, SEM, Corrosion resistance.

1. INTRODUCTION :

The study of Nanocrystalline materials reported by earlier researches was a great emphasis due to their attractive properties [1, 2]. Numerous techniques like ball milling, electrochemical deposition, sputtering was studied for the development of these materials [3-8]. Electrodeposition method was recognized as appropriate technique for the development of such nanostructured materials. It is known that Alloys and intermetallic has more potential towards structural applications compared to it conventional metals, because of their high strength, modulus and oxidation resistance [9, 10]. The advent in research has directed its attention towards the study Ni-Fe alloy due to its excellent mechanical and magnetic properties [11-13]. For over 100 years polycrystalline nickel-iron (NiFe) alloys was used in a larger scale for engineering applications [14]. This was due to the unique and excellent mechanical properties of Ni-Fe alloy which are reliant on its alloy composition like permalloy (82Ni-18Fe), Hypernik (50Ni-50Fe). The nanocrystalline forms of these Ni-Fe alloys will have exceptional effect on its coating properties and its corrosion behavior. Several previous studies on the corrosion behavior of the electrodeposited metal coatings has specified the importance of grain size of the coatings on its corrosion behavior [15-17]. With this understanding, the present work deals with the development of nanostructured Ni-Fe alloy coatings and evaluation of their anti-corrosive properties.

2. MATERIALS AND METHODS :

A new electrolytic bath was prepared with nickel sulfate (NiSO₄.7H₂O), ferrous sulfate (FeSO₄.7H₂O) as salts, boric acid as a buffer and Cetyl Trimethyl Ammonium Bromide (CTAB) as an additive. This sulphate electrolytic bath was prepared using laboratory graded chemicals and distilled water. The optimization of bath composition and the experimental condition for the development of bright Ni-Fe alloy coatings was carried by the conventional Hull cell method. The optimized bath composition along with the experimental parameters is given in table 1.

The electrodeposition of Ni-Fe alloy was carried out on mild steel panel cathode, taking nickel as anode. The coatings were deposited on the cathodic surface area of 7.5 cm², with same anodic surface area. Before electrodeposition, mild steel panels were polished mechanically using emery papers of various grit size (from 100 to 2500) and then cleaned electrochemically. During electrodeposition both anode and cathode were placed parallel to each other on PVC cell containing 250 cm³ of electrolytic bath solution. Electrodeposits were developed for various current densities (c.d.) from 1.0 A dm⁻² to 6.0 A dm⁻² using direct current power source, N6705B, Keysight Technologies for 10

minutes. The resultant coatings were then cleaned with distilled water and air dried. The surface morphological study of the developed coatings was carried out through Scanning electron microscope (SEM, ZEISS model). The average surface roughness of the Ni-Fe coatings was evaluated by Atomic Force Microscope (AFM, Bruker). The corrosion resistance performance of the electrodeposits was evaluated in 3.5% NaCl by Electrochemical Impedance Spectroscopy (EIS) and Tafel extrapolation method with three electrodes set using CHI608D electrochemical workstation (CH Instruments, Austin, USA). The developed coatings with exposed 1 cm² surface area was used as working electrode. The Ag/AgCl electrode and platinum electrode were used as a reference electrode and counter electrode respectively.

Table 1: Bath composition and parameters

Bath Components		Composition (gL ⁻¹)
NiSO ₄ .7H ₂ O		90
FeSO ₄ .7H ₂ O		12
H ₃ BO ₃		35
Cetyl Trimethyl Ammonium Bromide (CTAB)		1.0
Electrodeposition Parameters		
c.d.	1 Adm ⁻² to 6 Adm ⁻²	
pH	3.5	
Temperature	300K	

3. RESULTS AND DISCUSSION :

Ni-Fe alloy coatings were deposited on mild steel panels by electrodeposition method from the optimized electrolytic bath given in table 1. During the optimization of the bath, hydrogen was liberated by consumption of the given electric current. Due to this, there was variation in the pH of the solution. To avoid this disturbance and to obtain good coatings without such interruption, boric acid, H₃BO₃ was used as a buffer to maintain the pH throughout the electrodeposition process. The surfactant CTAB was used as an additive to obtain bright Ni-Fe coatings with good corrosion resistance. CTAB is a cationic surfactant which reduces the surface tension of the bath and accelerates the ion transfer from bath to the cathode resulting in good quality coatings [18].

The surface morphology of the developed Ni-Fe alloy electrodeposits was evaluated using SEM. SEM micrographs of the coatings developed at 1 Adm⁻², 3 A dm⁻² and 5A dm⁻² is given in Fig 1(a), (b) and (c). From the figures it was observed that the Ni-Fe alloys are deposited on mild steel in the form of spherical grains. In 1 A dm⁻² the grains deposited were larger and unequal in size and their distribution is not uniform. But with the increase the c.d.'s the grain size was observed to decrease and its distribution is uniform throughout the coatings as observed in 3 A dm⁻². The decrease in size and the grain refinement may be the indication of increase in Fe content in the coatings [19]. The smaller and uniform grains result in the smoother surface of the coatings which may strengthen the corrosion resistance of the coatings. From the Fig 1(c) it is worth notice of the secondary grain development above which the size decreased grains are located. This further increase in grains increases the surface

roughness of the coatings which in turn consequences to the higher corrosion rate (CR). From the surface analysis of the developed Ni-Fe alloy coatings developed at 3 A dm^{-2} was suspected to have higher corrosion resistance ability than the coatings developed at any other c.d.'s.

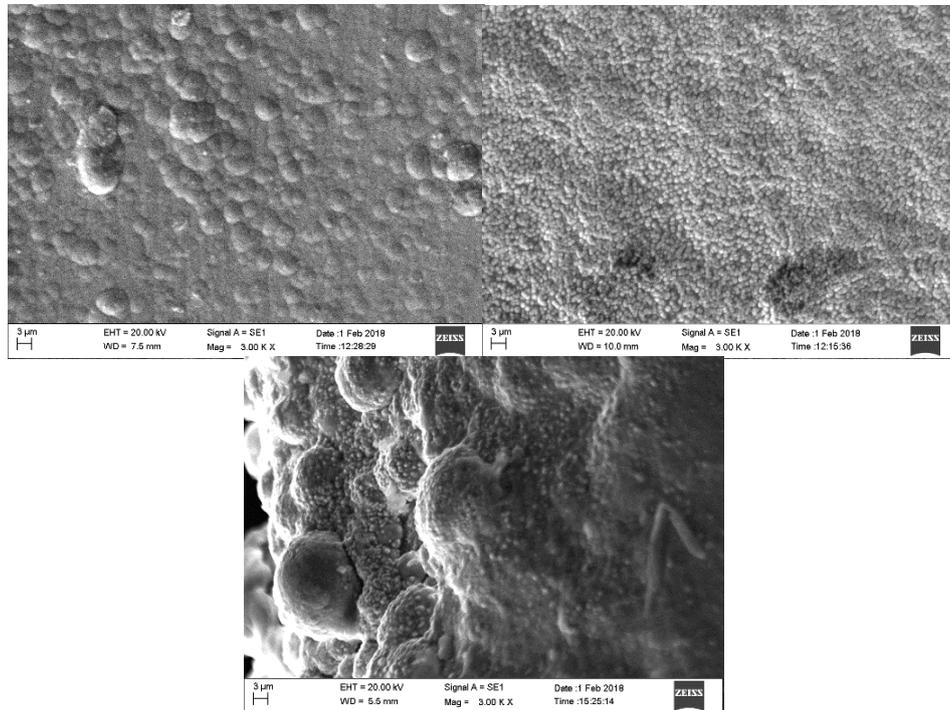


Fig. 1: SEM images of the Ni-Fe alloy coatings at (a) 1 A dm^{-2} , (b) 3 A dm^{-2} and (c) 5 A dm^{-2}

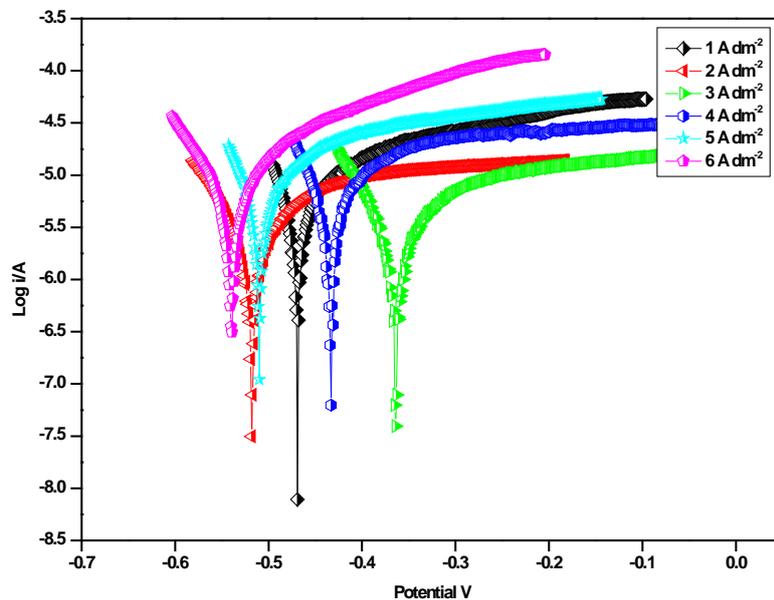


Fig. 2 : Potentio-dynamic polarization curves of the developed Ni-Fe alloy coatings

Table 2 : Corrosion data obtained from potentiodynamic polarisation method

c.d. (Adm ⁻²)	-E _{corr} (V vs SCE)	i _{corr} (µA cm ⁻²)	β _a (V/dec)	β _c (V/dec)	C.R. (mpy)
1.0	0.4690	6.500	5.901	3.679	2.858
2.0	0.5180	2.971	2.955	16.855	1.306
3.0	0.3640	2.948	3.013	17.909	1.296
4.0	0.4330	4.872	2.999	28.210	2.142
5.0	0.5100	5.850	3.188	27.30	2.572
6.0	0.5390	7.975	4.538	16.061	3.507

Potential-dynamic polarization method was employed to study the corrosion behavior of the developed Ni-Fe alloy coatings. The coatings developed at 1 A dm⁻² to 6 A dm⁻² was tested at in 3.5% NaCl solution. The resulting corrosion parameters like corrosion current (i_{corr}), corrosion potential (E_{corr}), corrosion rate (CR) are summarized in table 2.

From the table 2 it was highest positive potential E_{corr} (0.3640 V) and lowest corrosion current i_{corr} (2.948 µA cm⁻²) was observed for coatings developed at 3 A dm⁻², this direct towards the good corrosion resistance ability of this coating. Moreover, the CR was decreased as the c.d. increased. This indicates that the good corrosion protective coatings are obtained as c.d. was increased. At 3 A dm⁻² the CR was least (1.296mpy). This observation can be attributed to the surface morphology of the coatings. The uniform and fine surface of the coatings at 3 A dm⁻²

Results in the good quality coating with highest corrosion resistance. Above 3 A dm⁻² the surface of the coatings was coarse due to the development of secondary grain size as discussed above. Hence there observed a rise in CR.

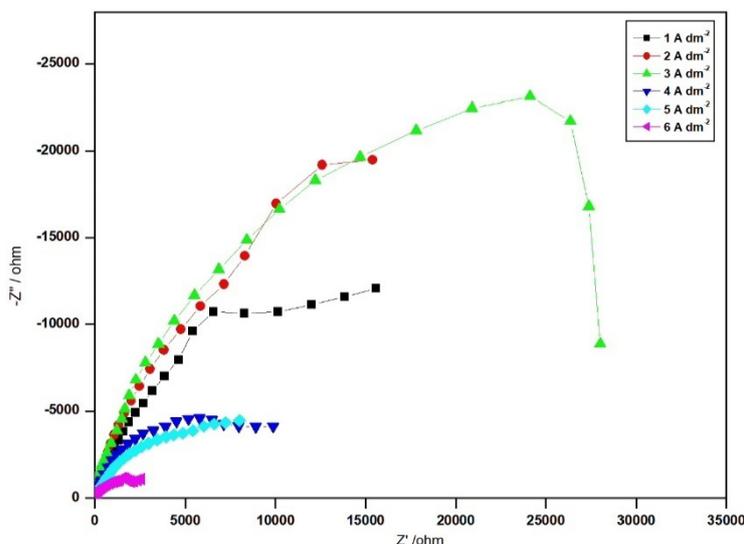


Fig.3 : Nyquist graphs for the developed Ni-Fe alloy coatings

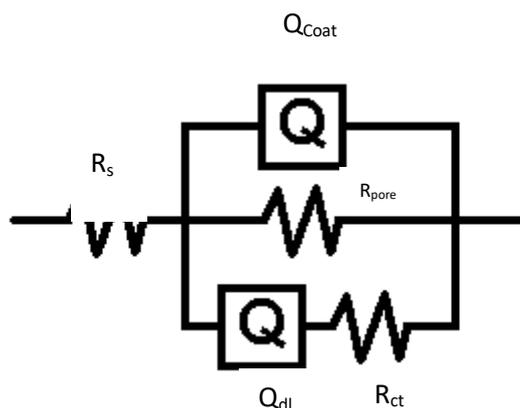


Fig. 4 : Equivalent circuit fit for the Nyquist graphs of Ni-Fe alloy coatings

Table 3 : Corrosion data obtained from Electrochemical impedance Spectroscopy

c.d. (A dm ⁻²)	R _s (Ω cm ²)	R _{pore} (Ω cm ²)	R _{ct} (Ω cm ²)	Q _{coat} -Y ₀ (μFcm ⁻²) CPE	n _c	Q _{dl} -Y ₀ (μFcm ⁻²) CPE	n _{dl}
1.0	2.981	1.194	32700	31.8	0.9024	14.83	0.8012
2.0	1.999	29.32	35600	22.45	0.2173	40.98	0.8841
3.0	5.264	5470	63300	8.429	1	11.64	0.6843
4.0	2.283	7128	10890	31.51	1	22.96	0.8787
5.0	4.697	2107	14210	30.87	0.9306	59.82	0.5843
6.0	0.899	1456	3377	44.49	1	128	0.7169

Electrochemical Impedance spectroscopy is a very useful technique to evaluate the barrier properties between the solution and the electrode as a function of frequency. This in turn helps the study of corrosion phenomena of the coatings. The Nyquist plot and the equivalent circuit for the Ni-Fe alloy coatings developed at various c.d.'s were given in Fig.3 and Fig.4. The corresponding fitment values of the circuit elements are given in table 3.

In the Fig.3 the graph obtained is a depressed semicircle which indicated the parallel CPE(Constant phase element Q) present in the circuit [20].In the circuit R_{ct} is the charge transfer resistance, R_s is the solution resistance and R_{pore} is the porosity resistance of the coatings, whereas Q_{coat}-Y₀ (CPE) and Q_{dl}-Y₀ (CPE) are the coating capacitance, and double layer capacitance.

In Fig.11, the charge transfer resistance R_{ct} is in series with the double layer capacitance Q_{dl}, whereas the coating capacitance Q_{coat} and porosity resistance R_{pore} are parallel to each other. This combination of connections is in series with the solution resistance R_s.

From the table 3, it was noticed that the coating capacitance Q_{coat} and the double layer capacitance Q_{dl}

value was least for the coatings at 3 A dm^{-2} , thus indicating the good corrosion protection of the coatings at this c.d. Furthermore, it is worth notice of the increase in R_{ct} value with the c.d. which indicates the increase in the resistance of charge transfer which in turn indicates the reduction in corrosion rate. R_{ct} value was highest for 3 A dm^{-2} which again decreases with further increase in c.d. This result is in agreement with the corrosion results obtained by Tafel method (potentiodynamic polarisation method).

4. CONCLUSION :

- New stable sulphate bath was proposed for the galvanostatic electrodeposition of Ni-Fe alloy monolithic coatings on mild steel using CTAB as additive.
- The electrochemical analysis of the corrosion resistance of the developed coatings confirms the least corrosion rate at 3 A dm^{-2} . This result was reflected in surface morphology of the coatings.

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