

Study on Nano-deposited Ni-based Electrodes for Hydrogen Evolution from Sea Water Electrolysis

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Abstract: Ni-based nano-layers were electrodeposited on low alloy carbon steel substrate using alkali electrolyte at current density 20 mA/cm² for 30 min. The effect of co-deposited elements (Fe, Mo) on the morphology, mechanical and electrochemical properties of deposited layer, has been investigated. SEM image of the deposited films exhibited a nodular structure having a grain size in the range of ~4- 7 nm. Based on EDAX analysis, the pure Ni layer was electrodeposited from pure electrolytes containing Ni-salts. The Ni-Fe alloy was composed of 96% Ni, 3% Fe, and 1% O. On the other hand, the chemical analysis of Ni-Mo binary alloy had the following analysis; 65% Ni, 33% Mo, 2% O. The ternary alloy, Ni-Mo-Fe contained 46% Ni, 49% Mo, 1% Fe, and 4% O. The oxygen content detected, increased with increasing the molybdenum content in the electrodeposited layer. According to XRD, multiple peaks representing pure Ni, Ni- and Mo solid solutions (ss), as well as intermetallic compounds were detected. The degree of crystallinity of Ni-based nano-deposits was decreased by the presence of alloying elements. The hydrogen evolution on Ni-Mo-Fe nano-deposit examined in sea water electrolytes was accompanied by low cathodic Tafel's constant β_c (50.2 mV/decade), and low over-potential (-850 V). This leads to high hydrogen evolution capability, where the volume of hydrogen evolved by cathodic electrolysis of sea water was 8 mL/hr. This ternary Ni-Mo-Fe deposit showed high corrosion resistance (1.7x 10⁻³mm/y), in sea water.

Keywords: Nanodeposits, electrodeposition, Ni-based alloys, HER, sea water electrolysis.

1. Introduction

Nickel based electrodes have been tried as cathodes for the production hydrogen by water electrolysis. This has attracted research interest since hydrogen would be the fuel of the future energy [1]. Catalytic materials are developed to minimize the overvoltage needed for hydrogen evolution reaction (HER), so that high energy consumption for the process may be reduced. It is well established that that Pt metal is the best electro-catalyst for hydrogen evolution. This is because the rate of adsorption of protons on the surface is almost equal to the rate of desorption or recombination to form hydrogen gas with $\Delta G = -0.1$ eV. Next ideal metal is Ni having $\Delta G = -0.28$ eV, followed by Mo with $\Delta G = -0.36$ eV [2].

In order to produce cheaper hydrogen by electrolysis, new electrode materials with improved electrocatalytic activity for hydrogen have to be developed. Successful electrode materials for the HER have to satisfy a number of criteria: They have to be stable with no signs of corrosion, large surface area of electrodes, and improvement of electrocatalytic activity of electrodes. It should be mentioned that economic reasons are in favor of cheaper materials, while environmental concern requests the use of non-polluting electrodes [3, 4].

Nanostructural materials are important for energy-based applications. New desirable properties are available at the nanoscale and not found in conventional 3D materials, such as: Higher surface area to promote catalytic interactions independent control of nanomaterials parameters (activity for example), which depend on each other for 3D materials [4]. Baeck et al [5] stated that electro deposition is proved to be a cost-effective and non-equipment-intensive method for the preparation of nano phase and nanocrystalline metallic materials, with a remarkable degree of reproducibility.

Recently, electrodeposited cathode binary and ternary coatings like Ni- S, Ni-Mo [6-8], Ni-Ti, Ni-Mo-Si, and Ni-Fe-Mo-Zn were and studied for hydrogen production. Alloying nickel with left transition metals (W, Mo and Fe) proved to increase the intrinsic electrocatalytic activity in the HER compared to pure nickel [9].

A series of ternary Ni alloys such as Ni-Mo-Fe, Ni-Mo-Cu, Ni-Mo-Zn, Ni-Mo-W, Ni-Mo-Co and Ni-Mo-Cr were studied for hydrogen evolution and the authors reported that the best and most stable cathode was Ni- Mo-Fe [4-12]. High stability of amorphous Ni-Mo-Fe electrode was also confirmed by other authors who tested the electrode with current interruptions [13].

Electrolysis of seawater, as an abundant available resource on earth, is one of the most promising ways to produce hydrogen. The electrolysis of seawater using nanostructured catalysts has received increasing attention recently [14, 15].

The aim of this work is the electrodeposition of nanocrystalline Ni-based alloys using alkaline solutions. The effect of adding Mo and Fe element on the morphology, chemical composition, mechanical properties and corrosion resistance will be investigated. In addition, the electrocatalytic activity for hydrogen production in sea water will be studied.

2. Experimental

The substrate used in the electrodeposition process is carbon steel 4130 delivered in the form of sheets 0.5 mm thick with the chemical analysis shown in the table 1. The specimens were machined to be of 6 cm long and 1.5 cm width. The specimens were ground with a grit sand paper, then it was pickled in hydrochloric acid of (20% wt) for 10 seconds and rinsed with distilled water followed by air drying. During electrodeposition, the current density applied was 20 mA/cm², for 30 minutes at 50 °C. The anode was made out of 5 cm × 5 cm × 0.635 cm nickel plate (99.5% Ni) supplied by Alfa Aesar, Germany. A power supply of type GW Instek GPC-3030DQ – Taiwan, was used in the process, which provides maximum voltage of 30 volts and current of 6 amperes. The solution composition and electrodeposition working conditions are presented in table 2.

2.2.1 Surface Morphology and Phase Analysis: SEM (JEOL JXA-840A) was used to demonstrate the microstructure and surface morphology of the electrodeposited layers. The X-Ray diffractometer used for phase analysis is (D8 ADVANCE X-Ray DIFFRACTOMETER)–Germany. The numerical procedures were facilitated by the use of computer software (PSI-Plot, ply software international, U.S.A. The crystalline diameter (L) was estimated by the Scherer's equation using the peak broadening of the most intense peak.

2.2 Micro-hardness: The hardness values were taken in Vicker's scale, using micro-hardness machine (Shimadzu Hardness tester HMV-2t), with a load of 970 mN. An average of five values on a straight line was taken for each specimen.

2.3 Potentiodynamic Corrosion Test: Potentiodynamic polarization measurements were conducted using a standard three-electrode cell with the coated samples as working electrode, Pt as auxiliary electrode, and saturated calomel electrode as reference electrode. Only 0.2 cm² of the Ni-based samples was subjected to 3.5% NaCl electrolyte at 25°C. The three electrodes were connected to Volta Lab 10 potentiostat/galvanostat. Computer software (Volta Master 4) was used to draw Tafel extrapolation and to get the corrosion current and corrosion potential. All experiments were conducted using a scanning range from -800 mV to 200 mV at a rate of 100 μV/sec.

2.4 Sea Water Electrolysis: The electrocatalytic activity of specimens for hydrogen evolution using sea water was measured using three techniques; the first method [16] is the comparison of the potential of different Ni-based electrodeposited nanocrystalline alloys recorded at constant current density of -0.03 mA/cm² in linear polarization curves (*I-V*). The second method [17] is the comparison of the slope of the cathodic region of Tafel polarization curves (β cathodic). The third method is the actual measurement of volume of hydrogen evolved during sea water electrolysis using Hofmann apparatus. The cell consisted of identical electrodes of 0.6 cm² area, for each, immersed in 3.5% solution. The same described above power supply was utilized. The experiments were conducted at 6 volts for 30 min, at room temperature.

3. Results

3.1 SEM Analysis: Figures 1 illustrates the surface morphology of different Ni-based layers being electrodeposited on steel substrate using current density 20 mA/cm², for 30 min at pH 9.8. The pure nickel layer (Figure 1-a) illustrates elongated areas, containing some rounded particles. Figure 1-b shows the surface morphology of Ni-Fe nano deposit, which is characterized by granular appearance with a relatively smaller grains compared to other types of deposits. The Ni-Mo layer (Figure 1-c) was characterized by fine particles embedded in larger nodules. In addition, grain boundaries could be easily identified. Figure 1-d illustrates a dense adherent nodular structure of Ni-Fe-Mo deposited layer. The grains display a spherical shape, while very fine particles grow preferentially out of the surface, producing a large number of non-equally grains with spherical shape. Also some boundaries between clusters can be seen.

3.2 EDAX Chemical Analysis: The chemical analysis of different Ni-based nano deposits are presented in Figure 2 and table 3. The Ni nano-deposit is composed of 100% Ni. The Ni-Fe deposit contains 3% Fe and 1% O and the balance is Nickel. On the other hand, Ni-Mo nano-deposits proved to have 65%, 33% Mo and 2% O.

The Ni-Mo-Fe nano-deposit; has 46% Ni, 49% Mo, 1% Fe and 4% O. The oxygen content increased, by increasing the molybdenum content.

3.3 XRD Characterization: The phases presented in the electrodeposited films as well as the crystallite size at current density 20 mA/cm² for 30 min in alkaline solution, were identified by XRD. In case of Ni-nanodeposits, only peaks for pure nickel are shown (Figure 4-a). In case of Ni-Fe nanodeposits, multiple peaks for pure Ni as well as Ni₃Fe intermetallic compounds, were detected (Figure 4-b). The Ni-Mo nano-deposits were characterized by the presence of XRD peaks for Ni, Ni_{0.91}Mo_{0.09} ss and Ni₄Mo intermetallic compounds. Figure 4-d shows that XRD pattern for a Ni-Mo-Fe film contains narrow peaks for pure Ni, as well as NiMo ss, NiFe ss, as well as Ni₄Mo intermetallic compound, in addition a small peak for FeO. The Ni (111) preferred orientation is more pronounced while the other peaks are not clear.

The crystal size was calculated from the line broadening using Sherrer's equation. The crystal size was determined as 4.3, 4.6, 4.8 for Ni, Ni-Fe, Ni-Mo, while the size was 6.7 nm for Ni-Mo-Fe nano deposits.

3.4 Micro-Hardness measurements: The microhardness values obtained at load 980 mN for different Ni-based nano deposits are illustrated in Figure 4. The hardness of nano-deposits layers are 596.2, 715, 627.4 and 498.8 (VHN) for Ni, Ni-Fe, Ni-Mo and Ni-Mo-Fe deposits, respectively. The highest hardness (715 VHN) was found for Ni-Fe nano-deposit due to the presence of Ni₃Fe intermetallic compounds [18]. A relatively low hardness (498.8 VHN) was registered for Ni-Mo-Fe nano-deposit. This could be due to grain size increment detected (6.7 nm).

3.5 Roughness Measurements: Figure 5, shows the roughness of different Ni-based nano deposits layers. Clusters of non-uniform nodules presented in case of the SEM for pure Ni (Figure 4-a), reflects the highest roughness 1.4208 μm. The Ni-Mo-Fe nano deposit had the lowest roughness 1.0288 μm.

3.6 Corrosion Properties

Figure 6 illustrates the potentiodynamic polarization curves for the electrodeposited layers, at current density 20 mA/cm² for 30 min at pH 9.8 examined in 3.5% NaCl. The cathodic reaction zone is characterized by a plateau with low slope. Meanwhile, the anodic polarization part is characterized by two anodic active zones with increasing slopes. The potentiodynamic corrosion properties for Ni-based nanodeposits in the above solution of are summarized in Table 4. The maximum corrosion rate was obtained for Ni-Mo deposit (~26x10⁻³ mm/y). The minimum corrosion rate was obtained for Ni-Mo-Fe deposit (1.7x 10⁻³ mm/y).

3.7 Electrocatalytic Behavior and Hydrogen Evolution: The electro-catalytic behavior of different Ni-based nano deposits in sea water has been evaluated by three methods (Table 5). The first method was the comparison of the volume of hydrogen evaluation on Ni-based nano deposits, during seawater electrolysis. The rate of hydrogen gas evolution was 2.5, 10, 5 and 8 mL/h for Ni, Ni-Fe, Ni-Mo and Ni-Mo-Fe nano deposits, respectively.

The second method is to evaluate the slope of the cathode region of the polarization curves (β_c) for different Ni-based nano deposits. The measured values of β_c were 50.9, 93.1, 189 and 50.2 mV/decade for Ni, Ni-Fe, Ni-Mo and Ni-Mo-Fe nano deposits respectively.

The third method was the over-potential of the specimens for hydrogen evolution in 3.5% NaCl electrolyte, taken at constant current density of -0.03 mA/cm² on the I-V curves.

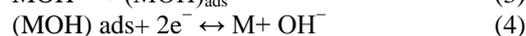
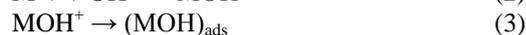
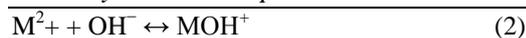
The measured over-potential values were -1000, -950, -880 and -850 mV for Ni, Ni-Fe, Ni-Mo and Ni-Mo-Fe nano-deposit, respectively. The Ni-Mo-Fe nano-deposit has relatively lower over-potential, which corresponds to high hydrogen evolution capability.

4. Discussion

Ni-based nano-layers were electrodeposited on low alloy carbon steel substrate using alkali electrolyte at current density 20 mA/cm² for 30 min. The effect of co-deposited elements (Fe, Mo) on the morphology, mechanical and electrochemical properties of deposited layer, has been investigated.

The co-deposition of iron-group metals (e.g. Fe, Co and Ni) is widely explained as an anomalous electroplating type. The sequence of metal hydroxide ions with respect to increase in the adsorption ability is Ni(OH)⁺ < Fe(OH)⁺. The reaction mechanism is generally processed as follows (Yin and Lin 1996; Harris et al 1999):





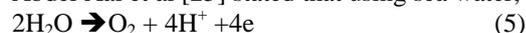
Where M indicates iron and nickel atoms. Although many attempts have been done to explain the anomalous co-deposition of alloys, there is still no universally accepted approach [20, 21].

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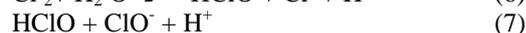
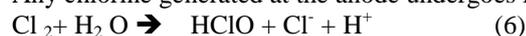
Nickel is a metal widely applied as the cathodic material in water electrolysis, due to its good catalytic activity and stability in alkaline as well as neutral solutions, and even in seawater [22]. Concerning the electrochemical reactivity for hydrogen evolution and according to this work, Ni-Mo-Fe nano-deposit had relatively low Tafel's constant β_c (50.2 mV/decade), and low over-potential which corresponds to high hydrogen evolution capability (VH₂=8 mL/hr), accompanied by the high corrosion resistance compared with other binary Ni-based deposits. This relatively low β_c for Ni-Mo-Fe corresponds to a good hydrogen evolution capability, compared to a value of 30 mV/decade, for Pt [23].

According to Lupi et al [24], the binary nickel-molybdenum coating electrodes showed low hydrogen overpotential and very good time stability under continuous electrolysis. However, the activities for hydrogen evolution reaction (HER) are easily lost after intermittent operation, especially after a long period because of the degradation due to the oxidized dissolution of Mo for nickel-molybdenum alloys. According to Jianga et al [15], hydrogen evolution reaction in 3.5 wt% NaCl (simulated seawater) was investigated using electrodeposited Ni-Fe-C cathode. The hydrogen overpotential was only about 65 mV, which was tested in the 3.5 wt% NaCl of 90 °C at pH 12.

Abdel Aal et al [25] stated that using sea water, both oxygen and chlorine evolution generates H⁺ as follows:



Any chlorine generated at the anode undergoes immediate hydrolysis which also generates H⁺ as follows:



As the anode becomes more acidic, the thermodynamic voltage for oxygen evolution becomes more anodic, a trend favoring chlorine evolution [25].

5. Conclusions

This study on electrodeposition of nano layers of Ni-based alloys has shown some results and conclusions, which may be summarized as:

1. According to XRD-analysis, the phases detected are as follows; Ni, Ni-Mo ss, Ni-Fe ss as well as intermetallic compounds. The grain sizes were ~ 4-8 nm.
2. From SEM micrographs, Ni-based nano-deposits showed a compact surface with nodular morphology.
3. The Ni-Mo-Fe deposit (~ 46% Ni, 49% Mo, 1% Fe, and 4% O) is characterized by low surface roughness (Ra= 1.0288 μm), with micro-hardness level of 498.8HVN.
4. Ni-Mo-Fe nano-deposit had relatively low Tafel's constant β_c (50.2 mV/decade), and low over-potential which corresponds to high hydrogen evolution capability (VH₂=8 mL/hr), accompanied by the high corrosion resistance compared with other binary Ni-based deposits.

Acknowledgment

This paper is extracted a thesis submitted to the faculty of Engineering-Cairo University as partial fulfillment of the M.Sc. degree by one of the authors. (A. Abdel-Fatah)

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Table 1: Chemical analysis of 4130 carbon steel substrate.

Element	C	P (max)	S (max)	Mn	Si	Mo	Cr	Ni	Fe
Wt%	0.28-0.33	0.035	0.04	0.4-0.6	0.2-0.35	0.15-0.25	0.8-1.1	0-0.25	Rem.

Table 2 Electroplating bath and working condition [3]

Compound	Weight of compound, (gm/lit)

Study on Nano-deposited Ni-based Electrodes for Hydrogen Evolution from Sea Water Electrolysis

	Ni	Ni-Fe	Ni-Mo	Ni-Fe-Mo
NiSO ₄ .7H ₂ O	6.5	6.5	6.5	6.5
NiCl ₂	3.5	3.5	3.5	3.5
Na ₂ MoO ₄ .2H ₂ O	---	---	1.0	1.0
FeCl ₂ .4H ₂ O*	---	1.0	---	1.0
Na ₃ C ₆ H ₅ O ₇ .H ₂ O	6.0			
Time, min	30			
pH= 9.8				
Working Temperature 50°C				
Magnetic Stirring at 300 rpm				

Table 3 Chemical analysis of different Ni-based nano- deposits.

Alloy	Chemical Analysis
Ni	100% Ni
Ni-Fe	96.26% Ni, 3.10% Fe , 0.64% O
Ni-Mo	64.67% Ni , 33.21% Mo, 2.01% O
Ni-Mo-Fe	46.33% Ni, 48.91% Mo, 1.14% Fe, 3.61% O

Table 4 Corrosion properties of various Ni-based nano-deposits, tested in 3.5% NaCl.

Deposit	E_{corr} (mV)	I_{corr} (μA/cm²)	Corrosion Rate (mm/Y)*10⁻³
Ni	-463.9	0.4569	5.97
Ni-Fe	-444.9	0.3993	3.08
Ni-Mo	-280.6	3.3618	26
Ni-Mo-Fe	-360.1	0.0762	1.71

Table 5 Electrocatalytic properties of various Ni-based nano-deposits, tested in 3.5% NaCl.

Deposit	β_c (mV/decade)	Overpotential,- mV	Vol. of H₂ (ml/h)
Ni	50.9	1000	2.5
Ni-Fe	93.1	950	10
Ni-Mo	189	880	5
Ni-Mo-Fe	50.2	850	8

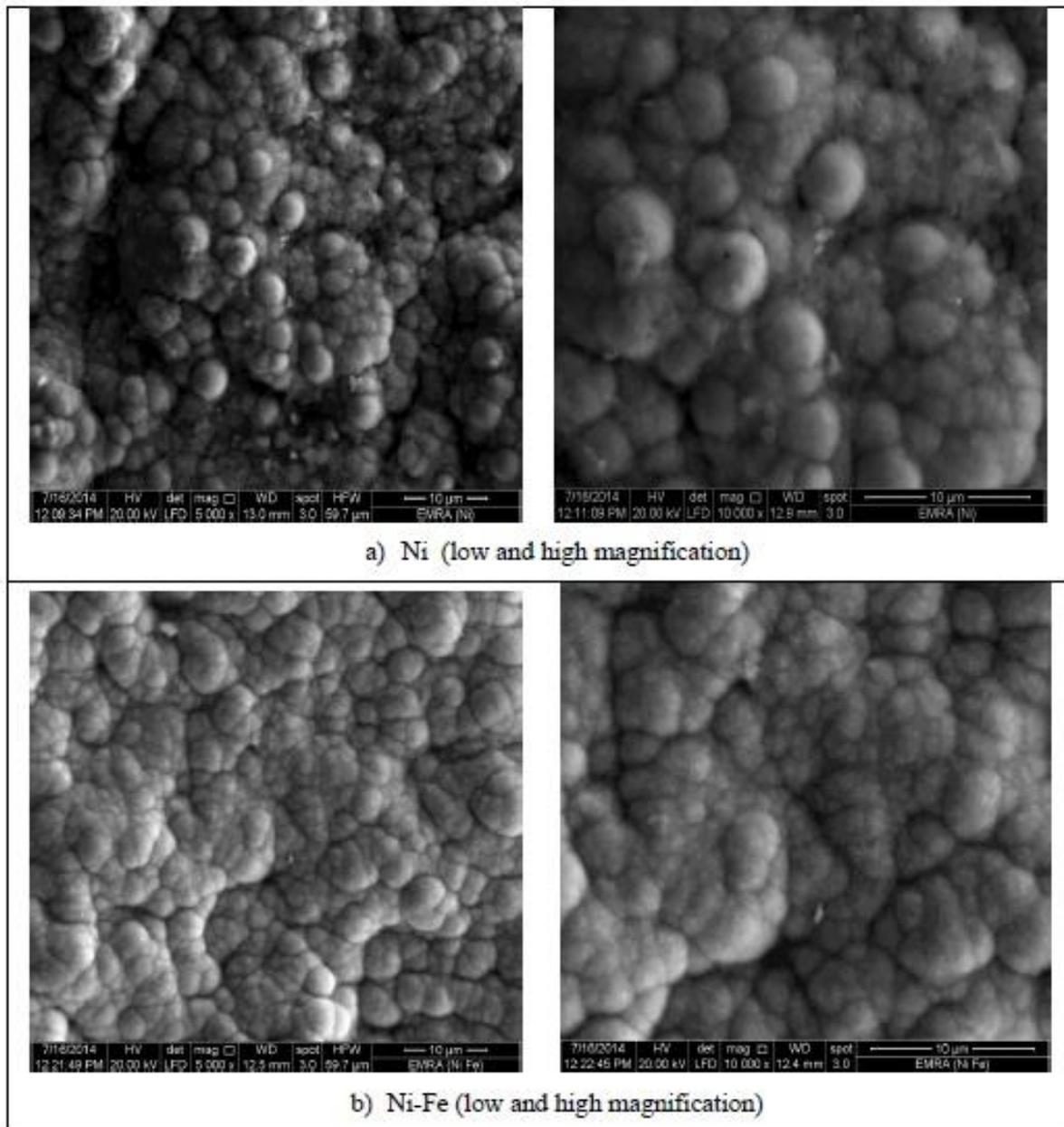


Figure 1 SEM images of Ni-based nanodeposits prepared by D.C electroplating at current density current density 20 mA/cm² for 30 min in alkaline solution.

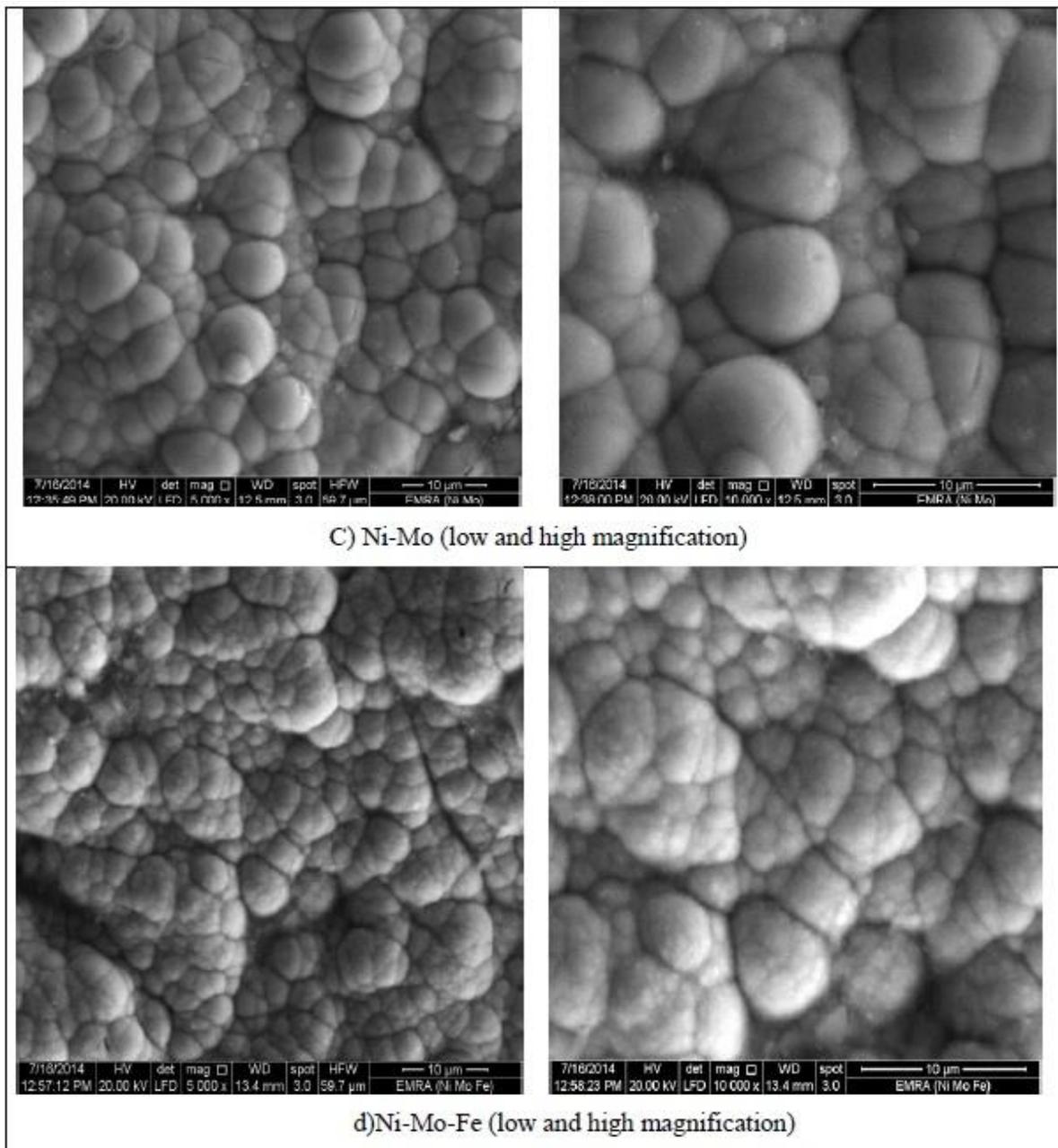


Figure 1 SEM images of Ni-based nanodeposits prepared by D.C electroplating at current density current density 20 mA/cm² for 30 min in alkaline solution. (continue)

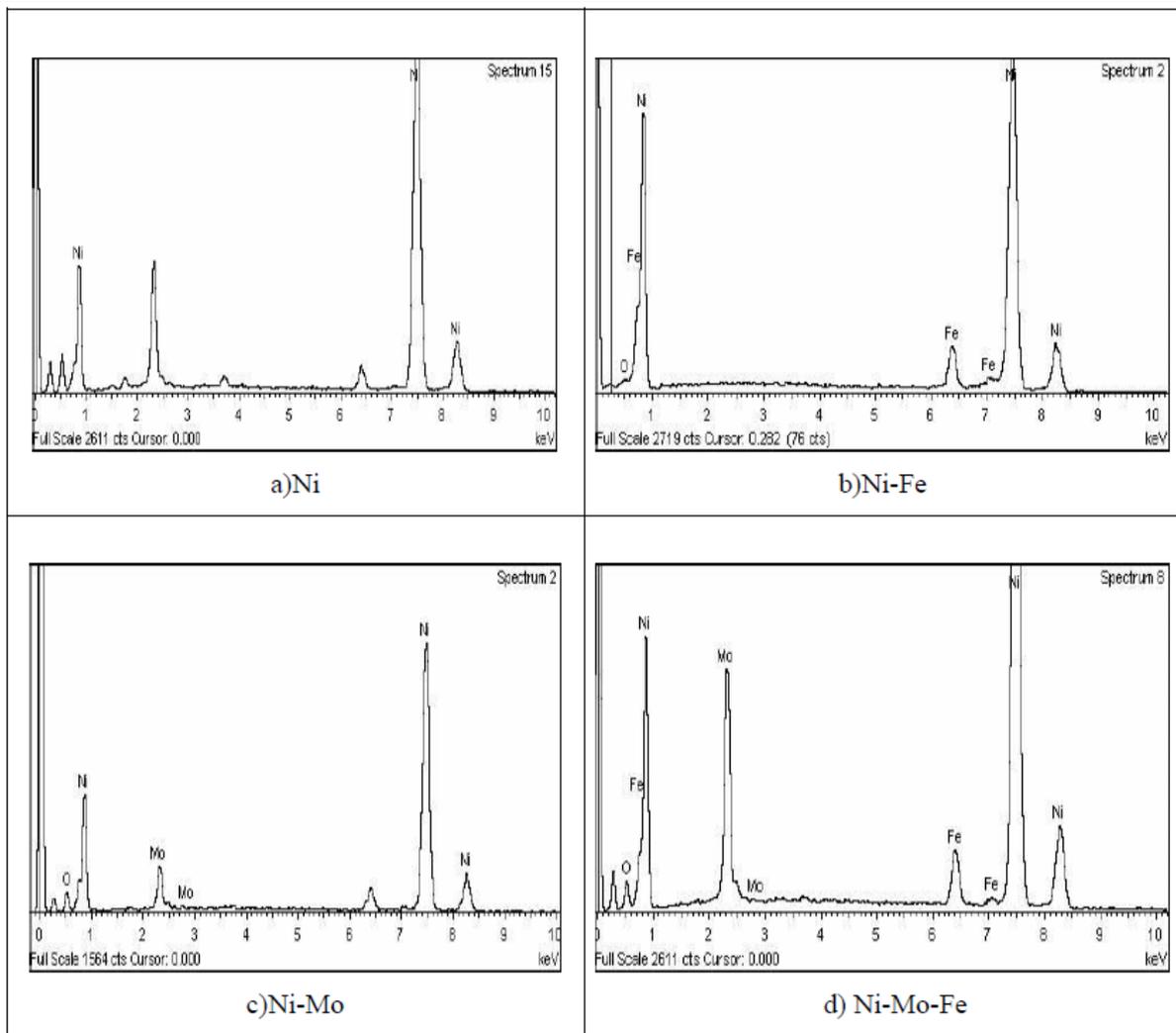


Figure 2 EDAX Analysis of Ni-based nano-deposits prepared by D.C electroplating at current density current density 20 mA/cm² for 30 min in alkaline solution.

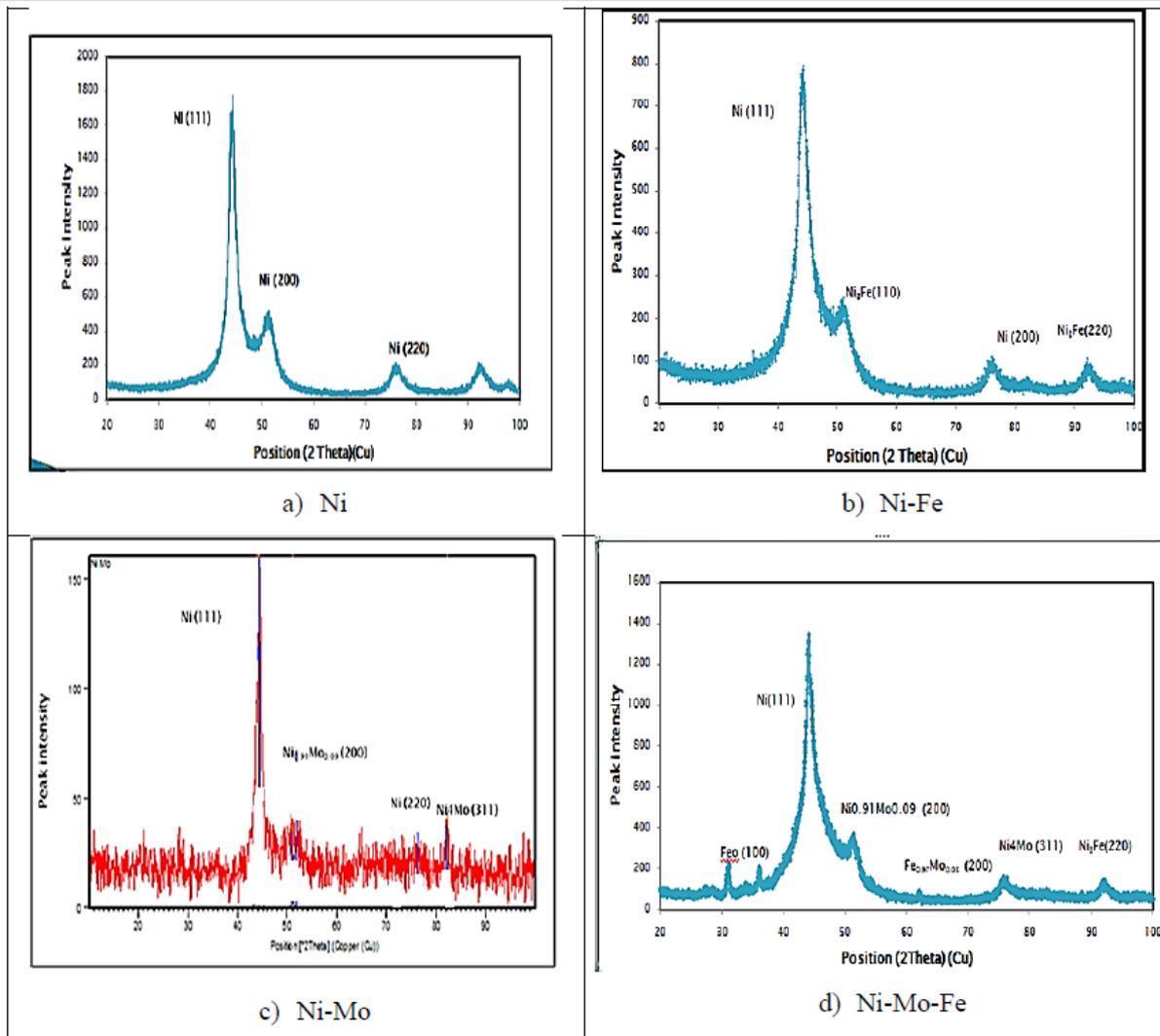


Figure 3 XRD-analysis of Ni-based nano-deposits prepared by D.C electroplating at current density current density 20 mA/cm² for 30 min in alkaline solution.

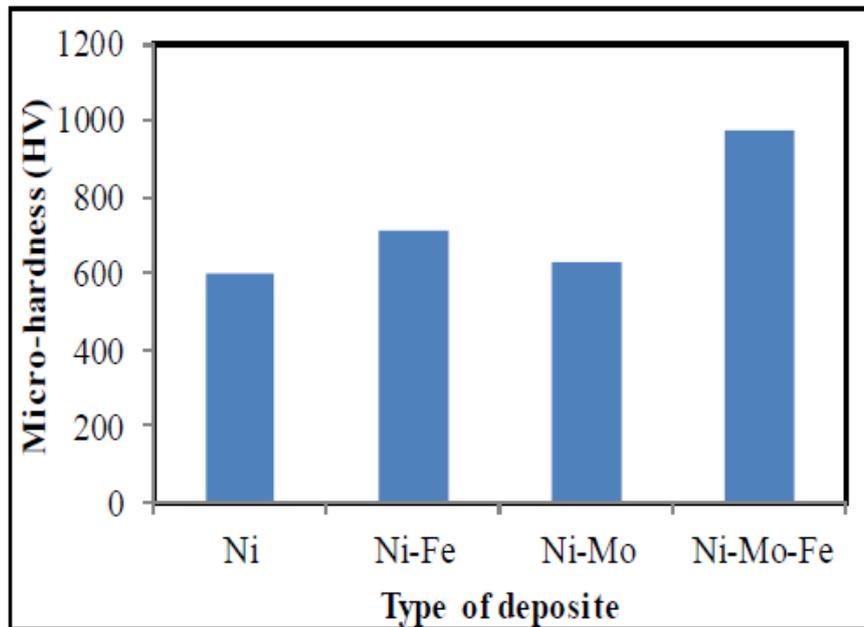


Figure 4 Micro-hardness of Ni-based nano-deposits at current density 20mA/cm² and deposition time 30 min.

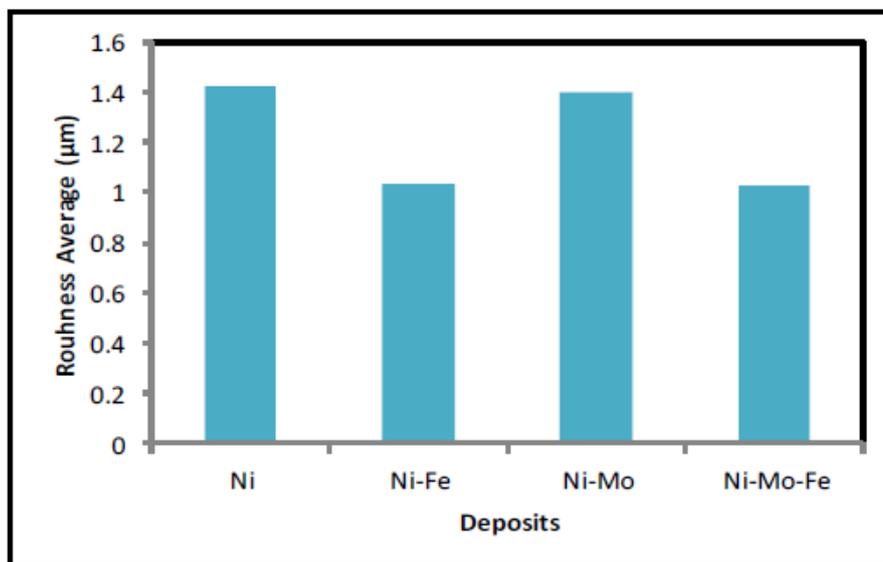


Figure 5 Roughness of Ni-based nano- deposits at current density 20 mA/cm² and deposition time 30 min.

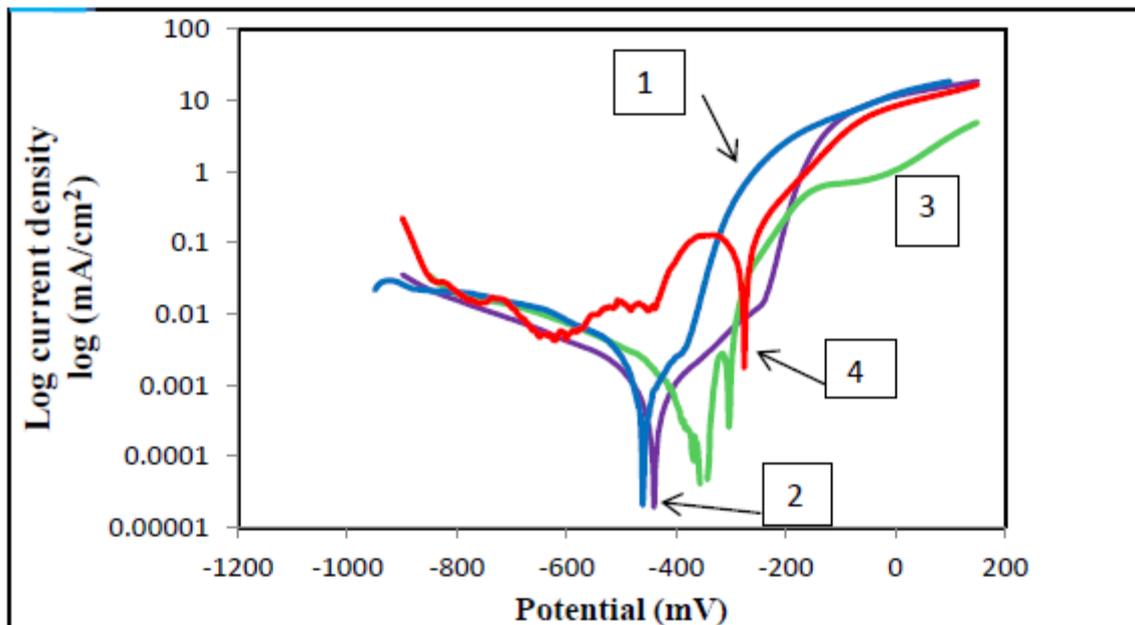


Figure 6 Polarization curves for various Ni-based nano-deposits. Curve 1: Ni nano-deposit; curve 2: Ni-Fe nano-deposit; curve 3: Ni-Mo nano-deposit; curve 4: Ni-Fe-Mo nano-deposit in 3.5%NaCl.

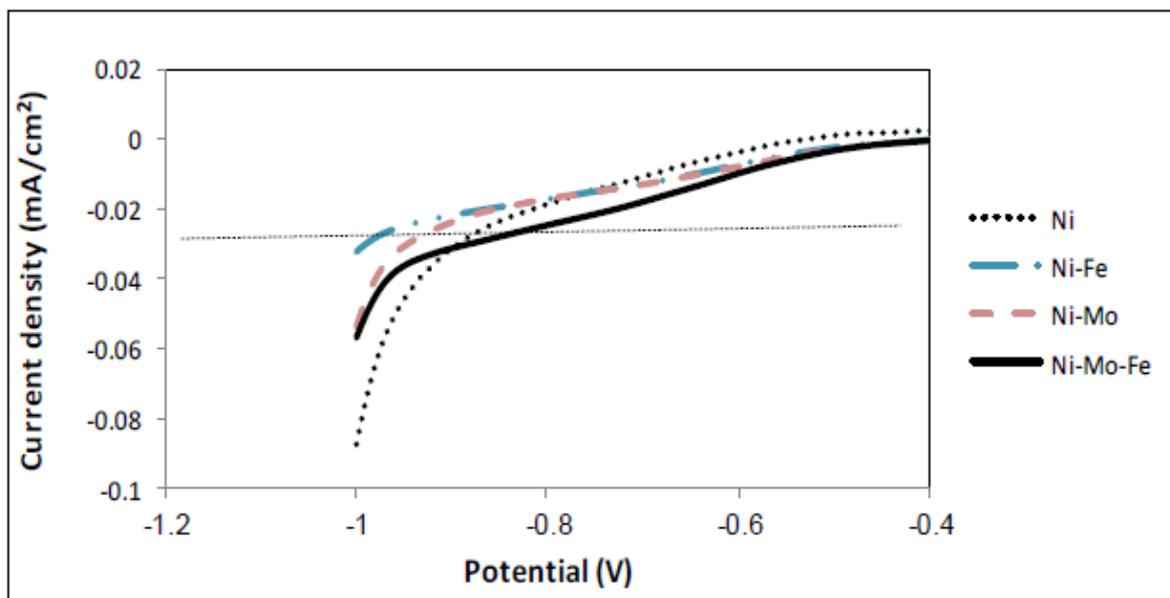


Figure 7 Over- potential of different Ni-based nano-deposits tested in sea water