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Research Paper

Effects of Cr Concentration on the Morphology and Corrosion Resistance of Ni-Cr Coatings Prepared by the Electrodeposition Process on 25CrMo4 Steel

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Abstract: In the current study, Ni-Cr coatings with a various Cr concentration of 0.1 wt.% Cr, 0.2 wt.% Cr, 0.3 wt.% Cr and 0.4 wt.% Cr were performed on the 25CrMo4 low steel, using the electrodeposition process. The deposition of the Ni-Cr coatings was carried out at temperature of 30°C, direct current density of 2 A.dm^{-2} for 40 min, and a bath containing a $Cr_2(SO4)_3$ as source of chromium ions, Cr^+. However, the effects of the Cr concentration on the morphology and the quality of the film were investigated. SEM, EDS and XRD analysis were used to characterize the Ni-Cr coatings. The SEM morphology analysis exhibited that the Ni-Cr with 0.2% Cr showed the lowest network of cracks and porosity with uniform and compact structure. The corrosion resistance of the Ni-Cr coatings has been investigated in 3.5 wt.% NaCl aqueous solution, using polarization curves and electrochemical impedance spectroscopy. According to the results, it was found that the variation of Cr con-tent in the Ni-Cr coating greatly affected the microstructure and the corrosion resistance of the coating. More-over, the coating developed with 0.2 wt.% of chromium (Cr) can give excellent results as to quality, morphology, and corrosion resistance in 3.5 wt.% NaCl environment. The results of polarization showed that 0.2 wt.% Cr layer had the more important resistance with an E_{corr} of 0.488 mV, I_{corr} of -418 µm.cm^{-2} and Rp of 84 kΩ.cm^{-2}. Therefore, EDX analysis confirmed that Chromium (Cr) oxides were formed, which contribute to its resistance.

Keywords: Ni-Cr coating, 25CrMo4, Cr concentration, Morphology, Corrosion, Electrodeposition

1. Introduction

Many studies were investigated to improve the properties of low steels and to enhance their surface characteristics by several techniques. Among these techniques, we can mention the electrodeposition process. Thus, this surface treatments allow to the surface treated to resist to the corrosion and wear. Current industries are interested in the chromium (Cr) and nickel (Ni) Coatings by the electrodeposition process. However, the development of the electrodeposition gave not only a coating such as Chromium, Nickel and zinc deposits but also binary coatings like Chromium-Nickel (Cr-Ni) forming an alloy on the substrate surface [1,2].

Usually, the surface coated with only chromium (Cr), showed some defects such as microcracks and porosity [3,4]. Consequently, these defects can affect the microstructure of the coatings and its properties [5,6]. Therefore, studies by E O Cobo et al. revealed in salt solution the microcraks allowed the penetration of this solution until the substrate where, the coating did not resist and gave a similar electrochemical behavior as the uncoated substrate [1]. The electrodeposition of Cr alloys can improve the properties of the Chromium (Cr) Coating, using iron family metals such as Fe, Co and Ni [7-9]. In addition, the nickel is applied in the Cr-Ni to resist to acids and to hot oxidation [10].
Extensive research into the deposit of chromium (Cr) was carried out. For instance, Edigaryan et al. [11], they investigated the effect of current density and concentration of the electrolyte (containing chromium trivalent sulfate $Cr_2(SO4)_3$) components on the composition of the deposit alloys. It has

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been reported that Cr content decreased as the current density increased, increasing concentration of Ni ions in the electrolyte. Besides, Survilienne et al. [12], they have deposited Ni-Cr from a bath containing oxalate and chromium trivalent sulfate $Cr_2(SO4)_3$. They have examined the Ni-Cr surface using XPS to identify the chemical composition of the coatings. He et al. [13], they have reported that the addition of various concentrations of Ni to the Cr baths, revealed different results caused by the Ni ions. However, The Ni ions in too low concentration of Ni play the part of a catalytic on the deposition of Cr, where high concentrations of Ni allow the depositing of the Ni ions, more than Cr ions. In the other hand, the chromium ions (III) show usually, a complex nature of chemistry in aqueous solution which prevents its deposition on the surface of the Ni-Cr coating [14,15]. To ensure the quality of the Ni-Cr film electroplated, it is important to choose a suitable solution, current density, and parameters for the Ni-Cr deposition process [15,16].

In the other hand, the most crucial factor that may affect and reduce the efficiency and performances of the Ni-Cr electroplated is the corrosion. To avoid corrosion from occurring on the surface of the materials and without destroying the Ni-Cr layer, an appropriate study of the bath parameters must be applied for the Ni-Cr electrodeposition process. However, it was demonstrated in study by Sheibani et al. [17,18], that the electrodeposited Ni-Cr coatings at various Chromium (Cr) concentrations of 20.7 Cr, 39.8 % Cr and 50 % Cr revealed a different corrosion resistance where the coating with 38.9 Cr content showed the high resistance corrosion. They related this behavior to the formation of an equilibrium Cr phase structure and a dense network of microcracks.

Several research have been investigated on the Ni-Cr coatings by the electrodeposition process, but limited studies on the variation of the chromium (Cr) concentration in the solution of Ni-Cr coating and its effect on the morphology and the corrosion resistance. Hence, the intent of this study is to investigate the effect of the chromium (Cr) concentration on the Ni-Cr electroplated layer from a bath containing chromium trivalent sulfate ($Cr_2(SO_4)_3$) as source of Cr ions. As a result, this study examines the effect of the Chromium (Cr) concentration on the morphology and quality of the Ni-Cr electroplated surface. As part of this study, the Ni-Cr layers electroplated on the 25CrMo4 substrates were investigated for corrosion resistance and stability in 3.5 wt.% NaCl environment to demonstrate the performance and the resistance of these coatings. Further, to optimize their parame-ters for industrial applications in a natural environment. The results of this study can be used to explain the relationship between the chromium (Cr) content, morphology, and corrosion resistance of Ni-Cr coatings.

2. Experimental Methods

The low steel 25CrMo4 substrate was used in this study with 10 x 20 x 5 mm^3 dimensions. All the substrates were grounded with SiC abrasive paper until 1000 grit size, then they were cleaned ultrasonically for 10 min in 99 vol. % acetone solution. After being cleaned in acetone, they were cleaned by distilled water and dried. Afterwards, begin the electrodeposition process by immersion the 25CrMo4 surface in a bath containing nickel chloride ($NiCl_2$. $6H_2O$), nickel sulfate ($NiSO_4$. $6H_2O$), boric acid (H_3BO_3), tri-sodium citrate ($Na_3C_6H_5O_7$. H_2O) and chromium trivalent sulfate ($Cr_2(SO_4)_3$). Therefore, the electrodeposition was carried out with a various Chrome (Cr) concentration, using $Cr_2(SO_4)_3$ as a main source of chromium ions. Chromium (Cr) concentrations were varied by 0.1 wt.% Cr, 0.2 wt.% Cr, 0.3 wt.% Cr and 0.4 wt.% Cr, whereas all the other elements concentrations remained the same. Furthermore, a Ni-Cr layer was electrodeposited on the surface at 2 A.dm^{-2} direct current density and 30 ± 1 ◦C temperature, maintaining the bath at pH of 4.

The pH value and the Temperature have been used in accordance with research in the literature. On the other hand, the value of the current density and the potential were set using the cyclic voltammetry (CV). In order to determine the solution potential values, the 25CrMo4 substrate was immersed in the appropriate electrolyte and scanned over the Potential range of -1 V to -1 V and scan rate of 0.05

$V.S^{-1}$, by using a Potensiostat type Parstat 3000A-DX where a current density (A) has been taken into account when measuring the Potential E (V).

Table 1 represents the composition and the molarity of the Ni-Cr solution with the various Cr concentrations. The surface morphology and the elemental distribution of the Ni-Cr layer were characterized by scanning electron microscope (Gemini SEM 300), using the EDS system under an acceleration voltage of 20 kV. In other hand, the composition of the phases formed was identified by x-ray diffraction (XDR), using Cu−kα and 2 theta. Electrochemical corrosion tests were performed on all coatings at various Cr concentrations, where the potensiodynamic polarization was carried out in an aerated 3.5 wt.% NaCl aqueous solution at 25°C and at a scan rate of 1 mV/s. In addition, an Ag/AgCl electrode was used as a reference electrode to maintain a stable potential during electrochemical measurements. After polarization in 3.5 wt.% NaCl, the surface composition of the coatings was analyzed by performing EDX measurements in order to provide a detailed understanding of the elemental distribution resulting from the corrosion process.

Table 1. Bath Composition of the Ni-Cr coating at various Cr concentrations.

Component	Concentration (M)			
	Bath 1	Bath 2	Bath 3	Bath 4
$Cr_2(SO_4)_3$	0.1	0.2	0.3	0.4
$NiCl_2 .6\ H_2O$	0.1	0.1	0.1	0.1
$NiSO_4 .6\ H_2O$	0.4	0.4	0.4	0.4
H_3BO_3	0.3	0.3	0.3	0.3
$Na_3C_6HO_7 \cdot H_2O$	0.1	0.1	0.1	0.1

3. Results and Discussion

3.1. Comparison of Surface Morphology

Figure 2 shows the SEM images for the Ni-Cr at various Cr concentrations. As observed, all the Ni-Cr coatings revealed a dark grey morphology; except the Ni-Cr 0.2 wt.% Cr coating (Figure 2c), is darker in color. As a result, coatings with Cr concentrations of 0.1 wt.% Cr and 0.3 wt.% Cr presented a uniform and slightly lighter morphology, and almost the same grain size distribution (wide grains), what is the opposite with the 0.4 wt.% Cr coating, which represents an irregular morphology (Figure 2g), and it is due to the nucleation mechanism. However, a severe network of cracks on these Coatings was observed. Hence, the cracks damaged the layer by breaking the continuous structure of the layer deposited and formed voids (Figure 2e and f), which deteriorates the layer. The Cracked morphology can be related to the low dissolved of Ni^{2+} ions, which promotes the deposition of the Cr ions [19]. In the other hand, it can be caused by the internal stress of the coating developed on the surface [4,20]. Comparing these results with those obtained in 0.2 wt.% Cr concentration (Figure 2c), it was found that the coatings present a regular, slightly darker morphology and a narrow grain size. Thus, it was seen that the Ni-Cr coatings with 0.2 wt.% Cr, revealed an extremely low cracked zone and it is quite evident that the coating with 0.2M chromium content does not reveal any porosity, precipitation, or segregation on the film.

The significantly low cracked zone can be explained by the process of the electrodeposition where the gas does not generate a high internal stress in the Ni-Cr coating [6,17,20]. As well as for decomposition Cr hydride during the Ni-Cr electroplating process [21].

The thicknesses of the Ni-Cr coatings were measured with the optical microscopy by taking 3 measurements for each coating and were confirmed with a contact-profilometer by using a diamond stylus which it moves vertically and laterally in contact with the surface's coating to measure the thickness. However, a thickness that varies between 40 and 50 nm has been observed and calculated for the coatings with the different concentrations.

3.2. Comparison of XRD Results

Figure 1 represents the XRD Pattern of the electrodeposited Ni-Cr coatings. Well also, a result of XRD pattern of Ni coating has been added to the superposition to understand and identify the phases. The results showed various diffraction peaks at 2 θ = 44.83, 52.07, 65.095, 76.62, 82.421, 98.96 and 116.24, which the peaks having miller indices (111), (110), (200), (220) (211), (311), (310). The XRD analysis indicates that the coatings at various Cr concentrations contained a few phases, where a single fcc Nickel phases was observed. Furthermore, Ni phase has a crystalline structure [22-24], which has been demonstrated by Ohgai et al. [25], as well as Demir et al. [26]. The micro-hardness, wear and corrosion resistance of the Ni-Cr layer can also be improved by this crystalline structure [27].

All diffractograms present an intense X-ray peak around 2θ of 44.83° is assigned to (110), indicating that the coatings were successfully obtained with all Cr concentrations used hence correspond to Cr and Ni. Thus, extra peaks were also observed and were indexed to fcc Ni and bcc Cr, indicating an amorphous structure of the coatings and the existence of a chromium-in-nickel solid solution. When compared to the pure Ni coating, the diffraction peak (200) decreases in intensity, while the peak (111) increases. Peak intensities increase as Cr concentrations increase and Ni decreases intensity for all the Ni-Cr Coatings. As compared to other, the Ni-Cr coating with 0.2 wt.% chromium concentration showed a significant decrease in peak intensities due to its compact structure.

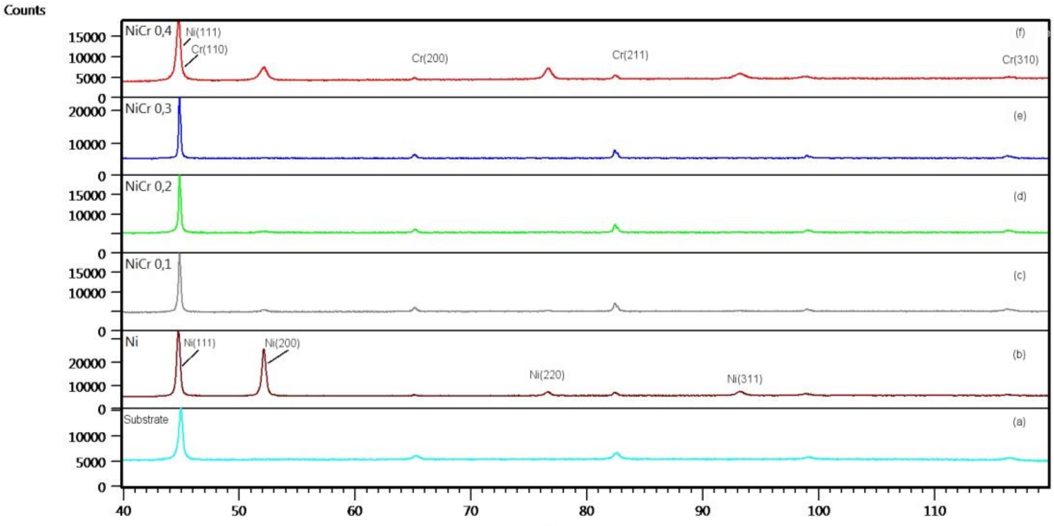

Figure 1. The superposition of XRD Patterns of: a) 25CrMo4 Substrate, b) Ni Pure, c) 0.1 wt.% Cr, d) 0.2 wt.% Cr, e) 0.3 wt.% Cr, f) 0.4 wt.% Cr.

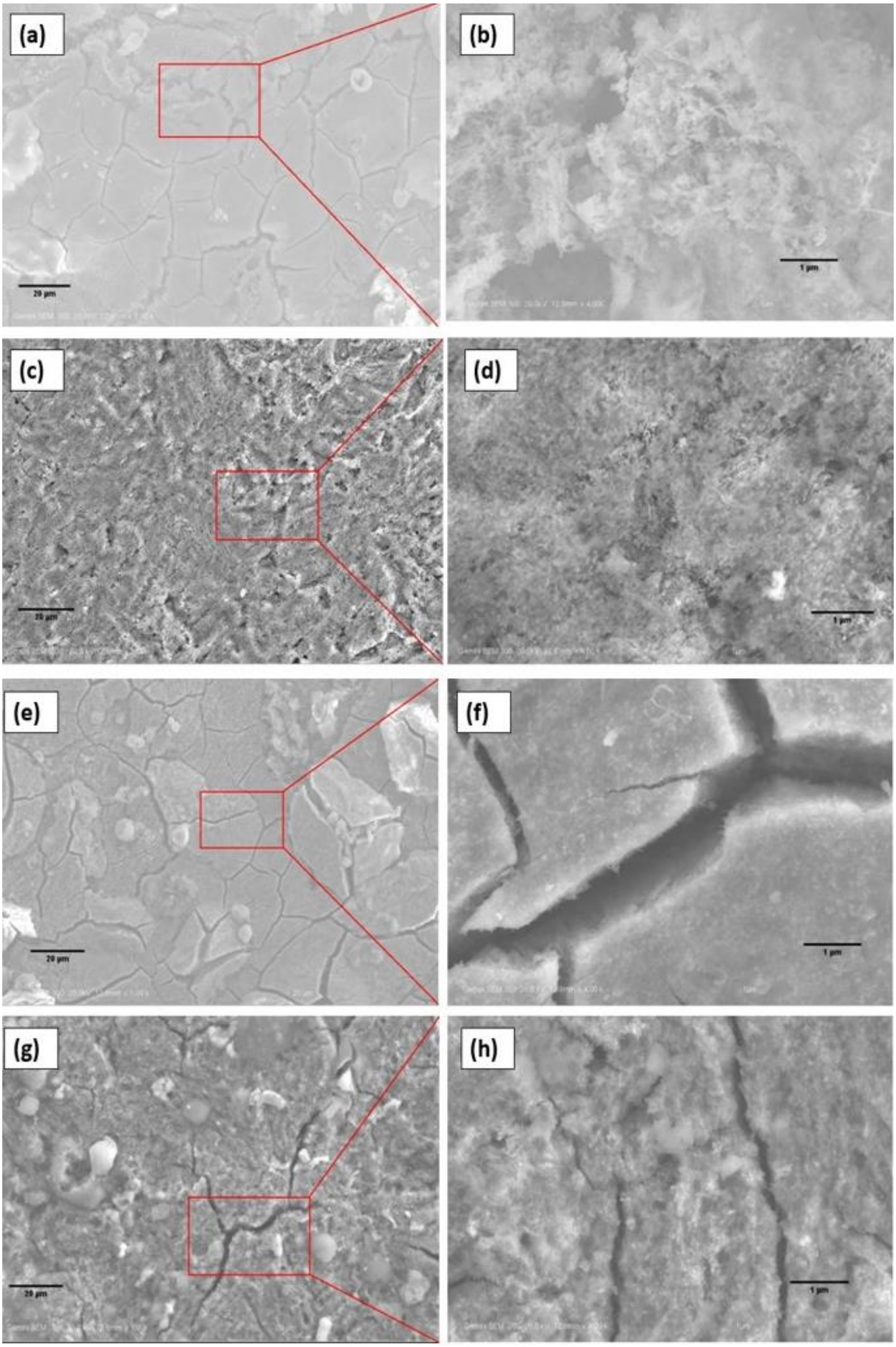

Figure 2. Surface Morphology of electrodeposited Ni-Cr at various Cr concentrations a-b) 0.1 wt.% Cr; c-d) 0.2 wt.% Cr; e-f) 0.3 wt.% Cr; g-h) 0.4 wt.% Cr.

3.3. Electrochemical Corrosions Analysis

The Figure 3 illustrates the electrochemical behavior of Cr-Ni coatings in 3.5 % NaCl aqueuse solution, at room temperature, a scan range from -1 to 0.1 V and a scan rate of 1mV/s. It appears that all curves at the interface (Figure 3A) of the potentials studied showed the same appearance. Although, it is readily apparent that the anodic current densities increase, and it has a tendency to stabilize. However, this stability indicates the formation of a corrosion layer, and corrosion products on the surface of the Ni-Cr coatings. As the Cr content in the Ni-Cr coating increases, the corrosion potential (E_{corr}) increases as well, and towards more negative values. In addition to that, the corrosion potential (E_{corr}) increases with increasing of Cr content except for 0.2 wt.% Cr coating. The results are shown in Table 2.

Table 2. Corrosion potential (E_{corr}), corrosion current density (I_{corr}), and the polarization resistance (Rp) measured in 3.5 wt-.% electrolytes.

	0.1 %C	0.2 %C	0.3 %C	0.4 %C

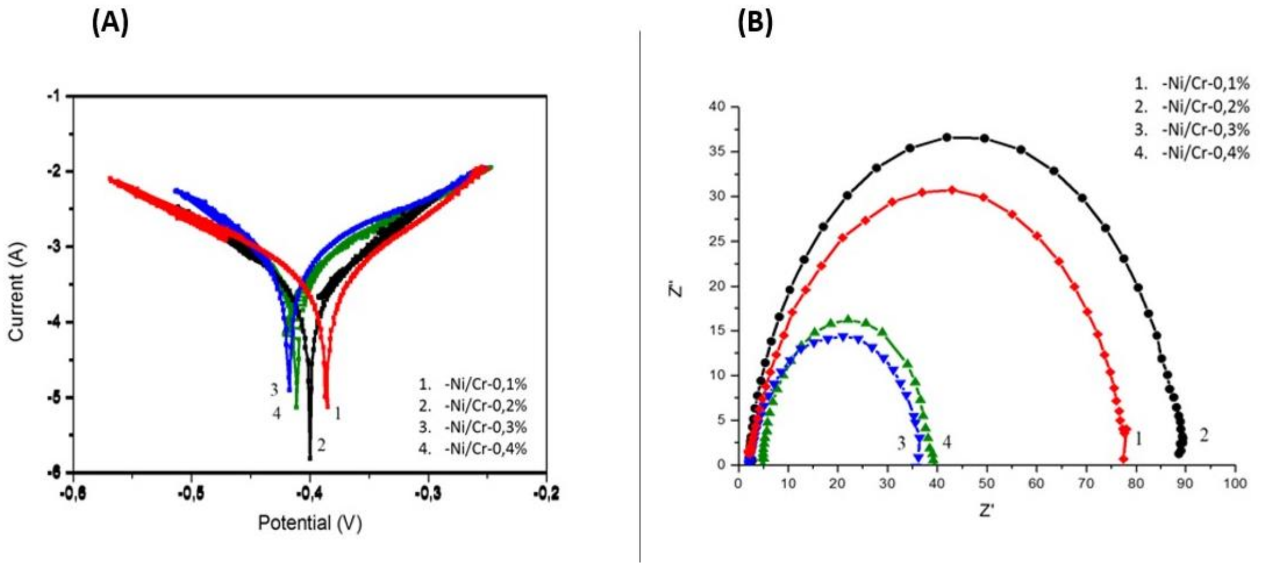

Figure 3. (A) potentiodynamic polarization curves obtained for various coatings in 3, 5 wt.% NaCl solution. (**B)** impedance curves of the Ni-Cr coatings.

Through the superposition of the curves (Figure 3a) and in agreement with the data represented in Table 3, it is evident that the coating with 0.3 wt.% Cr shows the most vulnerable structure at 3,5% NaCl environment (Polarization resistance Rp of 32 KΩ.cm^{-2}. It is due to the progressive accumulation of corrosion products; hence chlorine anions leak out of the pores and aggressively attack the Ni-Cr coating. Then it can reach the substrate's surface. On the other hand, the most adequate one corresponds to the energy of 0.2 wt.% Cr coating with a Polarization resistance (Rp) value of 84 KΩ.cm^{-2}.

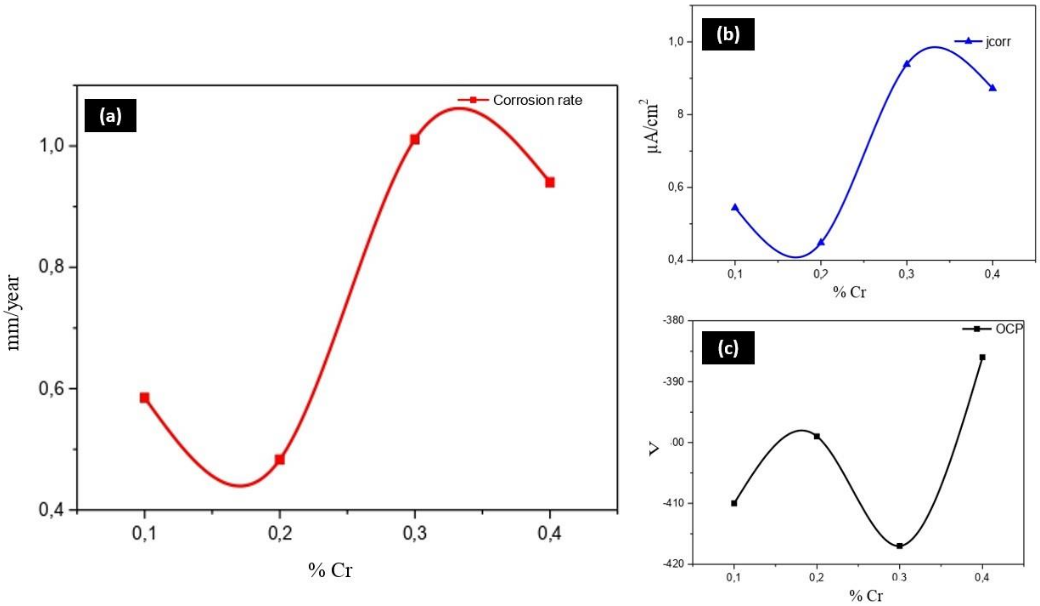

Figure 4. Electrochemical impedance spectroscopy data of the Ni-Cr coatings**:** (a) corrosion rate, (b) corrosion current density, (c) open circuit potential (OCP).

Electrochemical impedance spectroscopy of the coatings with a various Cr concentration in 3.5% NaCl solution is shown in Figure 3b. The frequency range used is between 100 kHz and 0.01 Hz, using peak-to-peak signal amplitude of 10 mV. The results revealed the same chemical process for all coatings with different Cr content, forming a loop and namely the active dissolution of the metal hence the diameter of this loop is proportional to the transfer resistance of the coating in the 3.5 wt.% NaCl solution.

Table 3. Electrochemical impedance spectroscopy measurements.

Energie (%)	0.1 %Cr	0.2 %Cr	0.3 %Cr	0.4 %Cr
Rs (Ω)	2.62	5.41	2.11	2.62
Rp (KΩ.cm^{-2})	34	84	32	81
C (µF)	1.50	1.02	1.58	1.53

The corrosion current density (I_{corr}), the corrosion rate and open circuit potential (OCP) were evaluated and represented. The results are shown in Figure 4. Among the comparison of the results (Figure 4), it appears that the corrosion resistance increase for the coating with 0.2 wt.% Cr. Whereas the coating with 0.3 wt.% shows the low resistance in the 3.5% NaCl , probably it is related to the presence of stress relief on the layer deposited and the hydrogen in the Cr content which leads to achieve a high corrosion current density and then a low corrosion resistance in the 3.5 wt.% NaCl aqueous solution [17], confirmed that the 0.3 wt.% Cr coating was unable to provide an excellent protection to the substrate in this corrosive environment.

Figures 5 and 6 show the Scanning electron microscope (SEM) micrographs and the elemental analysis (EDX) of the Ni-Cr coatings after the corrosion tests in 3.5% NaCl environment. It is observed that the 0.1 wt.% Cr and 0.3 wt.% Cr concentrations revealed a severe layer degradation and intergranular attack, forming path corrosion, cracks, and fracture (Figure 5A-(a), 5A-(b) and 6A). The 0.1 wt.% Cr content showed a pull-of the Ni-Cr layer which exposes the 25CrMo4 surface to corrosion, and then accelerates the loss of the material (Figure 5A) and this is most probably due to the cracks exist in the coatings before the corrosion tests and for the heterogonous microstructure of the coating [21].

Corrosion products were mostly shaped as lepidocrocite grey and brighter in color, either evenly distributed across the surface as seen in Figures 6B (a, b). Therefore, some forms were shaped as goethite as seen in Figure 5A-(a). The different morphologies observed after the corrosion tests are caused by the different behavior of the surface towards the corrosive environment.

The EDX spectrum shown in Figures 5 and 6 represent the distribution of the elements on the surface of the Ni-Cr after polarization in 3.5 wt.% NaCl solution. Energy dispersive X-ray spectroscopy (EDX) was conducted in duplicate (two different areas: spot 1 and spot 2) on each coating to analyze the elements after corrosion and identify the distribution of the formed oxides.

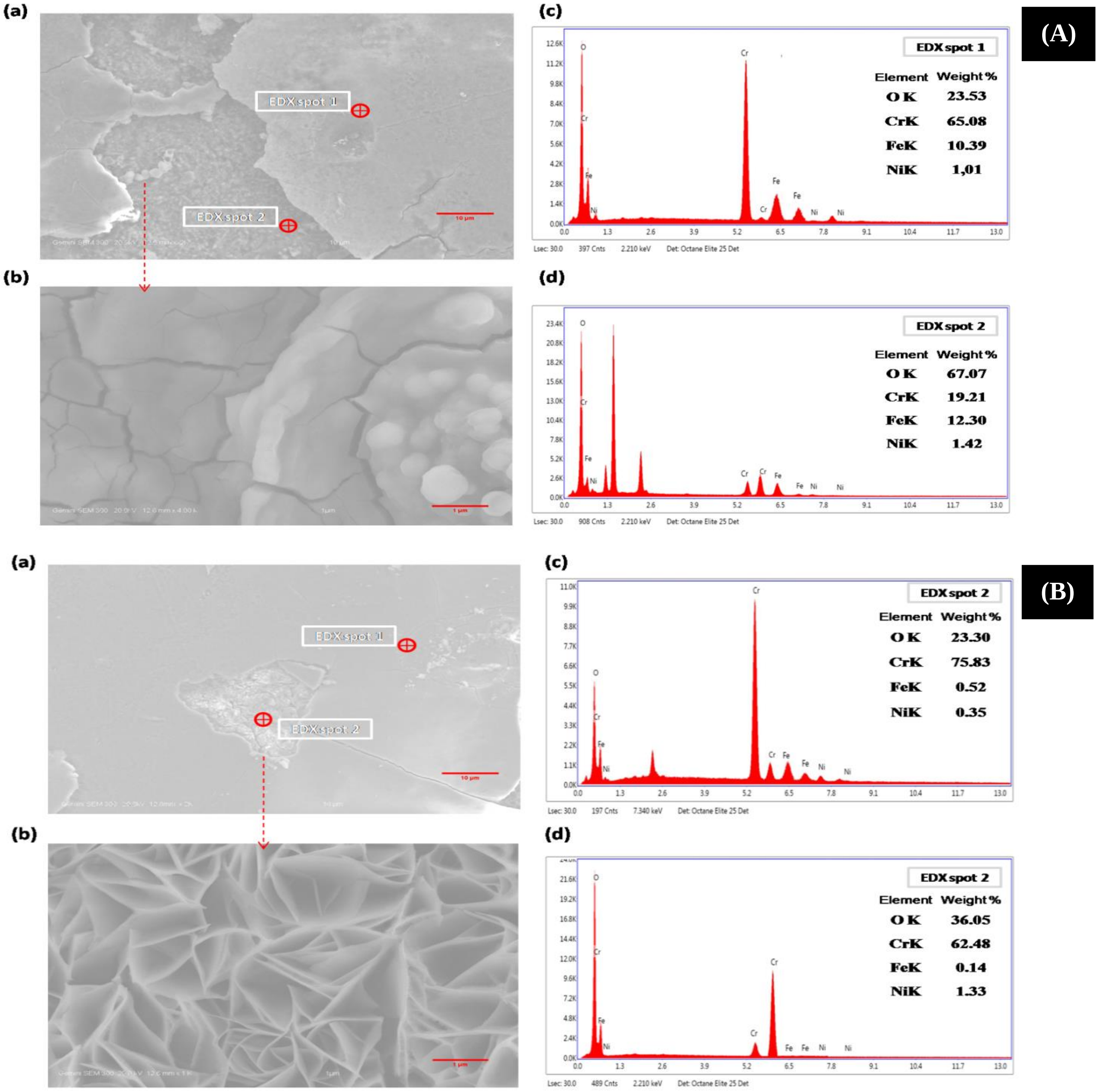

Figure 5. SEM morphologies (a-b) and EDX compositional analysis (c-d) of Ni-Cr coatings after polarization in 3.5 wt% NaCl solution: (A) Ni-0.1 wt.% Cr; Ni-0.2 wt.% Cr.

The entire EDX spectrums revealed the presence of Oxygen (O), chromium (Cr), nickel (Ni) and iron (Fe). However, the 0.3 wt.% Cr (Figure 6A-(c, d)) surface indicates a higher iron (Fe) content of about 53.60 wt.% and 65.11 wt.%, while the deposit with 0.2 wt.% Cr showed only negligible traces of iron (Fe) (Figure 5B-(c, d)).

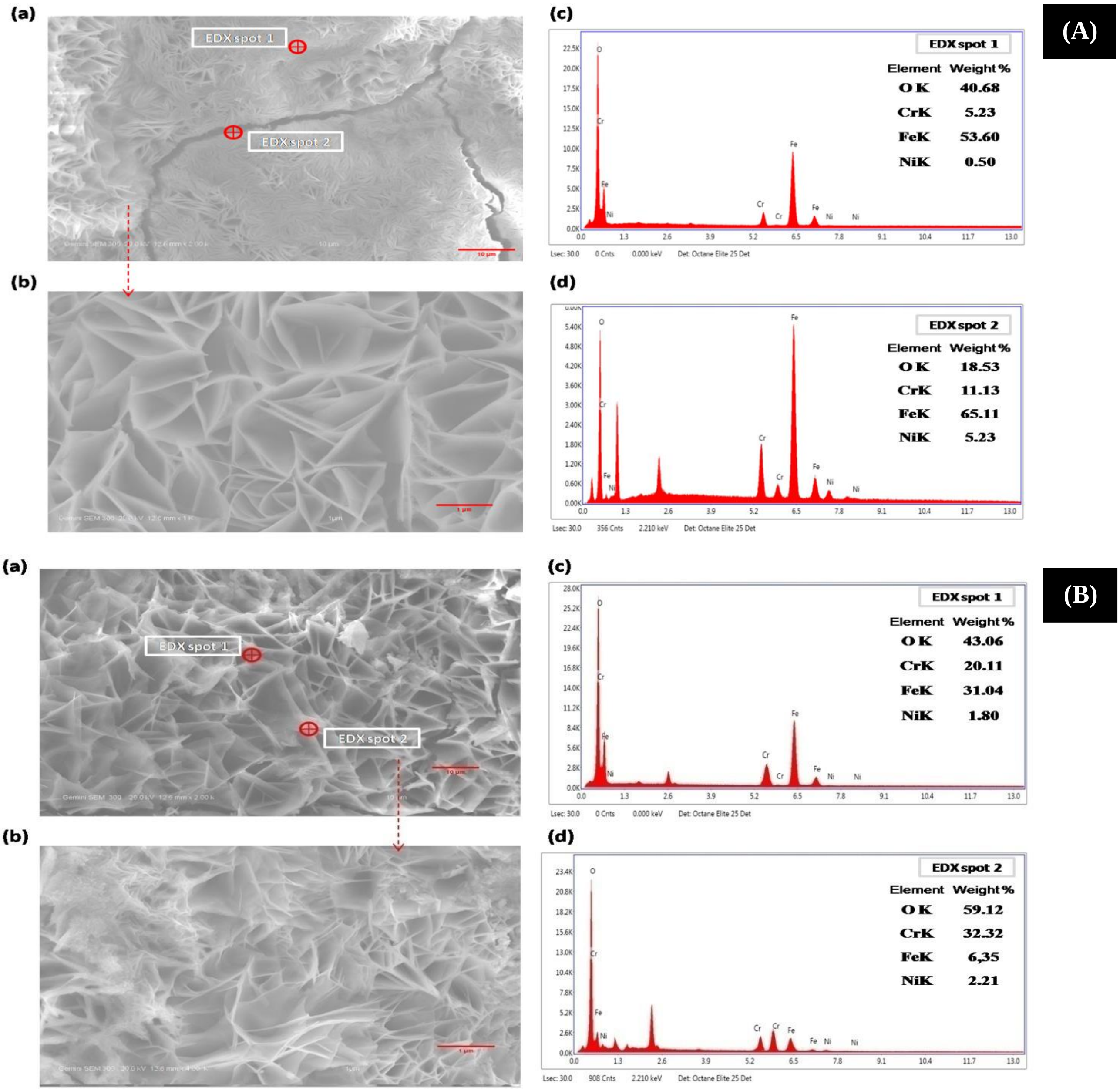

Figure 6. SEM morphologies (a-b) and EDX compositional analysis (c-d) of Ni-Cr coatings after polarization in 3.5 wt% NaCl solution: (A) Ni-0.3 wt.% Cr; Ni-0.4 wt.% Cr.

Furthermore, the observations suggest that the oxides formed on the surface coatings with 0.1 wt.% Cr and 0.3 wt.% Cr were iron oxides, nickel oxides and Chromium oxides as important oxygen content was identified. The oxygen (O) reacts with the alloy matrix and formed iron oxide layers. Moreover, the SEM images (Figure 5A and Figure 6A) were confirmed the presence of cracking and material removal of the deposit, indicating that iron (Fe) may be immigrated from the substrate and interacted with oxygen (O) to result in iron oxide formation, which eventually led to degradation and spallation of the surface. Iron (Fe) oxide is considered as a non-protective oxide [28], and represented a chromium (Cr) depleted areas [29]. In accordance with the impedance and potentiodynamic polarization curves (Figure 4), this coating of 0.3 % Cr showed the greatest corrosion potential.

Chromium and nickel oxides formed, and it was confirmed by the chromium (Cr) and nickel (Ni) peaks identified by EDX analysis, where their formation was limited by iron oxide nucleation and growth, which excluded them.

On the other hand, it is found that the 0.4 wt.% Cr coating exhibited similar trends to those of 0.1 wt.% Cr and 0.3 wt.% Cr coating with a lower iron (Fe) content. It has been revealed that the formation of iron oxide is reduced in this surface, due to the presence of signification chromium peaks (Figure 6B) of about 20.11 wt.% and 28.01 wt.% , respectively. Moreover, Nickel oxide (NiO, $NiCr_3O_4$) is formed, where nickel has a better corrosion resistance to iron oxide (Fe_2O_3) [30]. Thus, it is well known that chromium enhances corrosion resistance through the formation of the chromium oxide (Cr_2O_3) [21].

The results of EDX analysis revealed the presence of both nickel (Ni) and chromium (Cr) content with important peaks of Chromium (Cr) (Figure 5B-(c, d)). The passive film formed on the surface of the 0.2 wt.% Cr was primarily composed of Chromium oxides and hydroxides, as opposed to nickel oxides and hydroxide and it is consistent with the previous results conducted by Zhang et al. [31]. According to Mahesh et al. [32] and HJ Zhang et al. [33], the Cr oxides such as Cr2O3 and oxides of Nickel like NiO and $NiCr_3O_4$ can be considered as protective and benefited oxides.

Further, the presence of the chromium (Cr) with an important content after polarization can play a crucial role in the formation and stabilization of the passive film (Cr_2O_3), leading to enhanced corrosion resistance in 0.2 wt.% Cr surface and reducing the rate of corrosion [21,34], where nickel (Ni) can attribute in the thickness of the surface oxide layer [35]. On the other hand, the good resistance of the 0.2 wt.% Cr coating to 3.5 wt.% NaCl may be explained by the excellent adhesion of the Ni-Cr layer towards the surface electroplated [33]. Corrosion is well known to be related to adherence [36].

4. Conclusions

Ni-Cr coating can be obtained from a bath containing 0. 1 wt.%, 0.2 wt.%, 0.3 wt.% or 0.4 wt.% of Chromium (Cr) concentration using a $Cr_2(SO_4)_3$ as a source of Cr ions, with the aid of either H_3BO_3, $NiSO_4$, $Na_3C_6HO_7$ and $NiCl_2$. Moreover, the coating with 0.2 wt.% Cr can give best results in terms of quality, adhesion, and corrosion resistance. Thus, a very low level of cracks and porosity were observed for this Ni-Cr layer with SEM analysis. An increase of Cr contents in the Ni-Cr layer deposited tends to lower Ni content of the coating, causing the deposition of Cr ions hence, the apparition of the cracks from the release of hydrogen during the electrodeposition.

The XRD confirmed the amorphous morphology and showed the presence of the Ni and Cr single phase peaks for all the coatings. Therefore, it has been shown that Changes in Cr concentration resulted in changes in the preferential intensity of the peak of the matrix.

Throughout the results, it was found that chromium (Cr) content variation affected Ni-Cr layer morphology and quality during the deposition process. Moreover, the corrosion resistance in NaCl environment can also be affected by Cr content, where it has been shown by the SEM images that the 0.3 wt.% coating had cracked and significant corrosion. In addition, EDX analysis confirmed the presence of oxides, where the coating with 0.3 w.t% Cr revealed a non-protective oxide of iron, whereas coating with 0.2% Cr content revealed protective oxides rich in Cr-Ni. In the coatings of 0,1 wt.% Cr and 0,4 wt.% Cr, iron oxide and chromium oxide were detected, but not in amounts greater than 0.3 wt.% Cr coating. However, 0.3 wt.% Cr is unable to tolerate the corrosion in 3.5 wt-.% NaCl and this is in opposition to the coatings with 0.2 wt.% Cr. Thus, it is particularly important to control the Cr content in the Ni-Cr to get excellent results in term of morphology and corrosion resistance.

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Authors’ Contributions

This paper presents research that was conceptualized by SD. The methodology was suggested by SD, DA, YM, AH, and ZA. The manuscript was prepared by SD, YM, DA, AH. The results were interpreted by SD, YM, DA, AH, and ZA. All the authors read and approved the final manuscript.

Competing Interests

The authors declare that they have no competing interests.

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