Study of the Corrosion of Nickel–Chromium Alloy in an Acidic Solution Protected by Nickel Nanoparticles

Mohammad N. Majeed, Qahtan. A. Yousif, and Mahmoud A. Bedair*

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ABSTRACT: This study uses nickel nanoparticles coated on the nickel–chromium (Ni−Cr) alloy by the electrodeposition technique to protect the alloy against corrosion. An open-circuit potential and potentiodynamic and linear polarization resistance in a 1 M H$_2$SO$_4$ solution saturated with carbon dioxide were used to study the anticorrosion performance of nanoparticle coatings. When coated with nanomaterials, the corrosion rate of Ni−Cr alloy was lower than when it was bare, and the potential for corrosion increased from −0.433 V for uncoated Ni−Cr alloy to −0.103 V when the electrodes were exposed to saturated calomel. Electrochemical experiments show that nickel-coated Ni−Cr alloy corrosion in sulfuric acid media has high protective characteristics, with an efficiency of 83.69% at 0.165 mA/cm$^2$ current density when pH = 1 is used. As demonstrated by the results of this research, the nickel−chromium alloy can be protected from corrosion in acidic media by a low-acidity bath coating layer. Surface morphologies have shown that coatings at different acidic scales may be able to resist an acid attack because of their excellent adherence to the nickel−chromium alloy surface. Measures for determining and studying the composition of the alloy surface’s protective covering were improved using X-ray diffraction (XRD).

INTRODUCTION

Corrosion is a crucial process that affects every country’s economic structure.$^{1,2}$ As a result, treating various alloys separately becomes crucial.$^3$ Additionally, corrosion of steel and nickel alloys is a significant industrial hazard that has garnered considerable attention.$^{4,5}$ Due to their high permeability and low coercivity, nickel alloys are well suited for soft magnetic applications.$^{6−9}$ While nickel−iron alloys corrode easily when exposed to certain acidic media, these acidic media are consumed during various manufacturing processes.$^9$ As a result, maintaining the corrosion resistance of nickel alloys is a critical and time-consuming work duty.$^{10}$ Numerous strategies are used to protect the metal surface against destructive attacks (anticorrosive approach); nevertheless, corrosion inhibitors are considered viable to protect the metal surface, especially in an acidic medium.$^{11−15}$ Anions such as sulfates, nitrates, chlorides, and thiosulfates cause corrosion damage to iron alloys in the industrial environment, significantly lowering their operational life.$^{14−16}$ Among the several corrosion protection techniques available, the most successful is the application of inhibitors and coatings.$^{17−19}$ Organic corrosion inhibitors, on the other hand, might be harmful to the environment, producing cytotoxic and mutagenic damage to biological systems.$^{20}$ Surface coating with micro-nano structures can provide good corrosion protection for metallic matrices.$^{21}$ The weak link between the brittle oxide layer and the natural covering on the metal surface provides insufficient protection against long-haul consumption.$^{22,23}$ Coatings composed of oxide nanoparticles are frequently used to protect metal substrates against
corrosion in harsh environments.\textsuperscript{24,25} The electrodeposition approach has the following competitive advantages: (a) low energy consumption for environmental sustainability, (b) rapid industrial scale-up, (c) simple, cost-effective fabrication, and (d) improved performance and adaptability due to controllable process parameters such as voltage, deposition time, current density, bath composition, pH, and temperature.\textsuperscript{26–28} Because of their tribological and corrosion-resistant capabilities, Ni coatings have attracted a lot of interest in the industry.\textsuperscript{29} Electrodeposition of nickel (Ni) and reinforcing nanoparticles was a promising technique for fabricating Ni-based coatings, with significant advantages such as simple, low-temperature processing, no need for templates, versatility, cost-effective, rapid process, mass production, and no need for final product machining.\textsuperscript{30–32} In this study, the nickel alloy turbine blade specimens were coated with nickel nanoparticles using an electrodeposition technique at pH scales of 1 and 6. Regrettably, the turbine blades rusted when cleaned with water in the presence of gaseous byproducts of heavy fuel burning. It is both economically and environmentally beneficial. The protection of a nickel alloy was investigated using electrochemical methods such as open-circuit potential (OCP), potentiodynamic (PD), electrochemical impedance (EIS), and linear polarization resistance at a constant temperature (298.15 K) (LPR). To determine the nature of the coated surface, two surface morphology techniques, field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectroscopy (EDS), were applied. For this reason, X-ray diffraction (XRD) was used to supplement the other tests because it is a critical nondestructive technique for evaluating the alloy’s protective film composition.

\section*{EXPERIMENTAL PART}

The Ni–Cr alloy samples were composed of the elements mentioned in Table 1. A suitable sample with the dimensions (1 cm $\times$ 1 cm) and thickness specified below was constructed (0.5 cm). All specimens were polished to a mirror-like finish using emery sheets of various sizes and then lubricated with a soft cloth saturated in the diamond paste. The working electrode of the Ni–Cr alloy was coated with epoxy resin to keep the exposed area of 1 cm$^2$ away from electrolyte solutions. An electrolyte solution of 1 molar sulfuric acid in deionized water saturated with carbon dioxide was used to study the protective effects of nickel nanoparticles (pH = 1 and 6) on the working electrode surface. The experiments were conducted at a temperature of 298.15 K.

A nickel layer was electrodeposited on Ni–Cr alloy using a glass cell with a volume of 250 cm$^3$ and a working electrode sandwiched between two parallel platinum metal electrodes. The electrodes were spaced 0.5 cm apart. A magnetic stirrer was used to agitate the electrolyte, which comprised 40 g/L of nickel chloride hexahydrate, 210 g/L nickel sulfate, and 25 g/L boric acid.\textsuperscript{33} Sodium hydroxide was used to adjust the electrolyte’s pH to 6. The electrodeposition was carried out for 5 min at a steady DC voltage of 40 V. The electrolyte was kept at a temperature of 308.15 K. The samples were dried in the air for 24 h at ambient temperature before being stored in a desiccator until further testing. The Ni layer was deposited onto the Ni–Cr alloy using the identical technique, except that the electrolyte pH was changed to 1 using nitric acid. The applied voltage was increased to 60 V for 1.5 min to create a black nickel layer. The corrosion electrochemical cell was connected to a potentiostat device to determine full electro-chemical parameters. FESEM (ZEISS Gemini, Germany, Ltd. Company) was employed to characterize specimens. The chemical components’ compositions were determined using EDS spectra. The XRD patterns were recorded by the Bruker D6 Advanced diffraction equipment with a Cu radiation source.

\section*{RESULTS AND DISCUSSION}

\textbf{Open-Circuit Potential (OCP).} The fluctuation can identify the Ni–Cr alloy coating in OCP as a function of immersion time. The evolution of the open-circuit potential with time for Ni–Cr alloy in 1 M H$_2$SO$_4$ solution without and with coated protection is depicted in Figure 1. The curves show that the nanomaterial protective layer causes considerable changes in the OCP’s temporal behavior. A cathodic displacement of Ni alloy is found in the absence of protection. However, with nickel layer protection at pH = 6, the potential change in the cathodic (active) direction is more significant until the 200s. The decline in OCP is greater at pH = 6 than unprotected, most likely because the protective layer adequately isolates the Ni–Cr alloy surface from the corrosive environment. At pH = 1, the profiles of the OCP curves with nickel coating show typical anodic tendencies. However, in the latter situation (pH = 1), the OCP values remained constant, drifting slightly into the 100s. This could indicate that an effective protective coating adheres to the alloy’s surface.

\textbf{Potentiodynamic Polarization (PDP).} Figure 2 depicts the potentiodynamic polarization curves of an uncoated Ni–Cr alloy and a nickel-coated at different pH scales. From these curves, the corrosion potential ($E_{cor}$), current density value ($I_{cor}$), anodic Tafel constant ($\beta_a$), cathodic Tafel constant

| Table 1. Composition of Ni–Cr Alloy Specimen |
|---------------------------------------------|
| elements | C | Mn | Al | Si | Mo | Fe | Cr | Ni | wt % |
|-----------|---|----|----|----|----|----|----|----|------|
|           | 0.05 | 0.12 | 0.18 | 2.26 | 0.03 | 3.21 | 15.30 | balance |
A thin nickel layer coating applied to the Ni–Cr alloy surface in 1 M sulfuric acid solution decreased the cathodic and anodic slopes, suggesting that the hydrogen production mechanisms were affected by applying a thin protective film on the alloy surface. Polarization curves show that both the anode Tafel slopes ($\beta_a$) and the cathodic ones ($\beta_c$) decrease significantly by applying the Ni coating at different pH values, which suggests that Ni coating mainly inhibits both the anodic dissolution of the Ni–Cr alloy and the cathodic hydrogen reduction process to some extent. It is clear that the hydrogen evolution reaction and the metallic dissolution could be controlled, and the mechanism of the proton discharge reaction differed depending on the technique of protection utilized.

**Linear Polarization Resistance (LPR).** The well-known linear polarization resistance (LPR) method determines the corrosion current density. The Stern–Geary equation was utilized to determine the polarization resistance ($R_p$) of Ni–Cr alloy coatings. From the ($R_p$) values obtained from linear polarization data, the coating protection ($LP$) was estimated using eq 2 as illustrated in Table 3, when a protective layer is added to the surface, the values of polarization resistance increase.

$$\%LP = \frac{[R_p^0 - R_p]}{R_p^0} \times 100$$  

*Figure 2.* Potentiodynamic curves without and with coating layers of nickel at pH = 1 and 6 on the Ni–Cr alloy surface.

**Table 2. Parameters of Potentiodynamic Curves without and with Coating Layers of Nickel at pH = 1 and 6 on the Ni–Cr Alloy Surface**

| coating types | $\beta_a \times 10^{-3}$ (V/decade) | $\beta_c \times 10^{-3}$ (V/decade) | $I_{corr}$ (mA/cm$^2$) | $E_{corr}$ (mV vs SCE) | corrosion rate (mpy) | %PE |
|--------------|----------------------------------|----------------------------------|-----------------|-----------------|-------------------|------|
| blank        | 682.8                            | 295.5                            | 1.65            | -486.0          | 753.7             | 88.48|
| pH = 1       | 234.6                            | 115.6                            | 0.19            | -135.0          | 114.30            | 80.42|
| pH = 6       | 111.9                            | 222.0                            | 0.29            | -262.0          | 132.78            | 82.42|

(\(\beta_c\)), and corrosion rate (CR) are calculated. Table 2 shows the results of this calculation. It is common knowledge that samples having a higher positive corrosion potential are more inert.\(^{15,32}\) It is clear from Table 2 that the blank shows an $E_{corr}$ value of -486.0 mV (vs saturated calomel electrode (SCE)), while the Ni coatings shift to a more positive $E_{corr}$. The high $E_{corr}$ values of -135.0 mV (vs SCE) and -262.0 mV (vs SCE) are obtained for the Ni coatings prepared at pH = 1 and 6, respectively, indicating their resistance to corrosion. It was revealed that the current density values of the coated sample were lower than those of the uncoated Ni–Cr alloy, indicating their high corrosion resistance.\(^ {33}\) The corrosion potential of the coated sample is shifted from the cathodic to the anodic direction. This result demonstrates the resistance of the coating to corrosive media. Based on observations of corrosion current density, the following equation can be used to calculate the percentage of protection efficiency\(^ {34}\) (%PE).

$$\%PE = \left[ \left( I_{corr} - I_{coating,corr} \right) / I_{corr} \right] \times 100$$ \hspace{1cm} (1)

where $I_{corr}$ and $I_{coating,corr}$ are the corrosion current densities values in uncoated and coated states, respectively.

Corrosion current densities reduced from 1.65 mA/cm$^2$ for the untreated Ni–Cr alloy to 0.12 and 0.29 mA/cm$^2$ for Ni, pH = 1 and Ni, pH = 6, respectively, for the coating types. The corrosion rate (CR) of nickel coating layers at 132.78 and 114.30 mpy is less than that of the uncoated surface. The Ni coatings highly reduced the corrosion rate of the Ni–Cr alloy by acting as a protective layer between the Ni–Cr alloy surface and the 1.0 M of H$_2$SO$_4$ solution.\(^ {35}\) As indicated, the corrosion current density of the nickel-coated surface at pH = 6 is less than that of the Ni-coated surface at pH = 1, and the percent PE is more significant. As a result, the enhanced corrosion protection provided by nickel coatings may be due to the nickel nanoparticles charged adhering to the electrode surface, shielding it from corrosive substances such as chloride ions, hydrogen, and oxygen gas.

**Table 3. LRP Method Information without and with Coating Layers of Nickel at pH = 1 and 6 on the Ni–Cr Alloy Surface**

| coating types | $I_{corr}$ (A/cm$^2$) | $-E_{corr}$ (mV) | $R_p$ (\(\Omega\) cm$^2$) | corrosion rate (mpy) | %LP |
|--------------|-----------------|-----------------|-----------------|-------------------|-----|
| blank        | 1.012 $\times 10^{-3}$ | 433.1 | 25.74 | 462.5 | 83.69 |
| Ni, pH = 1   | 165.9 $\times 10^{-6}$ | 103 | 157 | 75.82 | 83.69 |
| Ni, pH = 6   | 225.4 $\times 10^{-6}$ | 247 | 115 | 103 | 77.61 |
where $R_p^0$ and $R_p$ are the polarization resistances with and without coating layer, respectively. The polarization resistance ($R_p$), the corrosion potential ($E_{corr}$), the corrosion current density ($I_{corr}$), and the corrosion rate are determined by doing a linear regression on a current density versus potential curve near the corrosion potential. Thus, the corrosion rate of a Ni–Cr alloy with a coating layer is 75 mpy (at pH = 1), while the corrosion rate of the alloy surface without a coating is around 462.5 mpy.

According to Table 3, the nickel coating is more resistant to corrosion at a low pH value than its Ni-alloy counterpart at a high acidic value, owing to the higher corrosion at a low pH value than its Ni-alloy counterpart at a

$\text{corrosion rate of the alloy surface without a coating is around}$

$\text{near the corrosion potential. Thus, the corrosion rate of a Ni}$

$\text{a linear regression on a current density versus potential curve}$

$\text{equation was used to determine the percentage protection}$

$\text{without coating layers, respectively.}$

$\text{Electrochemical Impedance Measurements (EIS).}$

Electrochemical impedance measurements were made to better understand the Ni–Cr alloy surface electrode’s behavior. Figures 3 and 4 show the Bode and Nyquist plots for a Ni–Cr alloy surface immersed in 1 M H$_2$SO$_4$ and nickel layers plated on the Ni-alloy surface. The diameter of the semicircle fluctuates, and modifications are made in response to changes in the coating pH scale. The deviation from the perfect semicircle is typically caused by frequency dispersion, surface inhomogeneity, coating grain boundaries, and solution contaminants. Figure 5A illustrates the equivalent circuit used to fit the electrochemical impedance spectroscopy of Ni–Cr alloy in 1 M H$_2$SO$_4$ with and without the coated layer.

Table 4 summarizes the impedance characteristics of the electrolyte “solution” resistance ($R_s$), pore resistance ($R_{pore}$), and charge transfer resistance ($R_c$). The constant phase parameters $Y_{coating} = m$ and $Y_{corr} = n$ refer to the coating layer and corrosion reaction “double layer,” respectively. The following equation was used to determine the percentage protection efficiency $\%E_{pf}$.

$$\%E_{pf} = \left(\frac{R_{ct,c} - R_{ct}}{R_{ct,c}}\right) \times 100$$

where $R_{ct,c}$ and $R_{ct}$ are the charge transfer resistance with and without coating layers, respectively.

Additionally, Table 4 provides the double-layer capacitance per unit electrode area ($C_d$) and the double-layer capacitance per unit electrode area ($C_{dl,coating}$) determined from the curves presented in Figure 5. The $n$ exponent values in Table 4 range between 0.704 and 0.978, demonstrating nonideal capacitance behavior induced by the heterogeneity of Ni–Cr alloy surfaces generated by the stiffness of the surface coating layer. The resistances have changed because $R_{pore}$ replaces the protective layers and $R_c$ is increased and lowers the $R_s$ value. It indicates the protective layer’s thickness. It reduces $C_d$ by increasing the thickness of the electrical double layer, suggesting that the nickel layer at pH = 1 provides the optimum protection for the alloy under optimal conditions. As depicted in Figure 3, the Bode diagram demonstrates that as the protective coating layer changes, the IZI and phase angles rise, implying that it supports a single charge transfer mechanism. At pH = 1, the capacitance of the double layer decreases, indicating that the amount of Ni–Cr alloy components dissolving has been reduced.

Additionally, the capacitance value of the coating is significantly lower, demonstrating the coating’s compact nature. As shown in Table 4, as the pH of the coating bath was dropped to prepare the layers on the working electrode surface, the protective efficiency increased. On the other hand, these findings show that the nickel alloy surface coating’s barrier properties are strengthened, consistent with potentiodynamic and linear polarization data investigations.

Surface Analysis Techniques. Figure 6 shows FESEM images and an EDX spectrum of the uncoated alloy and nickel coatings at different pH scales for the Ni–Cr alloy electrode surface. Image A demonstrates uncoated Ni–Cr alloy. The nickel layer deposited on the surface alloy at pH = 1 is shown in Figure 6b. It demonstrates that the coatings created on the nickel alloy surface are more homogeneous and denser. The coating is of such high quality that no cracks or separation of the coatings with visible superficial cracks is due to the density of the deposited layer. Image C demonstrates that the nickel coating (pH = 6) is brittle and insufficiently cohesive, revealing a change in the acidity of the electroplating bath; it is assumed that this resulted in insufficient adherence to the alloy’s surface.
The EDS elemental analysis shown in Figure 6B indicates a higher Ni content related to the type of coating layer. It is relatively lower in the presence of a coating layer at pH = 6. Besides, the Ni element was detected by the EDX analysis in the uncoated alloy related to the composition of the alloy without any percent for the chromium element.

**X-ray Diffraction (XRD).** The X-ray diffraction peaks were recorded and are presented in Table 5 to identify the protective layer’s porous alloy surface and crystalline phases. The peaks at 36.47, 44.31, 51.48, 54.94, and 75.73°, respectively, can be indexed to the diffractions of (202), (111), (200), (242), and (220) Miller planes, as shown in Figure 7.

The EDS elemental analysis shown in Figure 6B indicates a higher Ni content related to the type of coating layer. It is relatively lower in the presence of a coating layer at pH = 6. Besides, the Ni element was detected by the EDX analysis in the uncoated alloy related to the composition of the alloy without any percent for the chromium element.

**Table 4. EIS Data of without and with Coating Layers of Nickel at pH = 1 and 6 on the Ni−Cr Alloy Surface**

| coating types | blank | Ni, pH = 1 | Ni, pH = 6 |
|---------------|-------|------------|------------|
| $R_s$ (Ω)     | 4.102 | 3.094      | 3.76       |
| $R_{pore}$ (Ω)| 15.62 | 88.57      | 3.099      |
| $R_c$ (Ω)     | 23.07 | 118.7      | 78.38      |
| $Y_{coating}$ ($S$ $\times$ $10^{-6}$) | 7.874 | 104.6      | 18.01      |
| $n$ $\times$ $10^{-3}$ | 704   | 882.4      | 987        |
| $Y_{corr}$ ($S$ $\times$ $10^{-6}$) | $1.226 \times 10^{-3}$ | $2.124 \times 10^{-3}$ | $708.2 \times 10^{-6}$ |
| $m$ $\times$ $10^{-3}$ | 492   | 556        | 678        |
| $C_\text{dl}$ ($\mu$F/cm$^2$) | $3.259 \times 10^{-3}$ | $1.80 \times 10^{-5}$ | $3.79 \times 10^{-5}$ |
| $%E_{pr}$ | 80.564 | 1.80 $\times$ $10^{-5}$ | 70.566 |

The pattern shows that the porous material is made of nickel at a high intensity due to the high solubility of chromium atoms in the nickel matrix. Figure 7 shows the XRD patterns of nickel plating at pH = 1 and 6. The thin crystallinity and purity of the nickel nanoparticles were further confirmed by the diffraction peaks’ vulnerability and the absence of any diffraction peak matching any impurity.

As seen in Table 5, the Ni coating layers were responsible for the high diffraction peaks at $\approx$44, $\approx$51, and $\approx$75°. The XRD pattern (Figure 7) of pH = 1 displays the same diffraction peaks as pH = 6 with different intensities, as shown in Table 5. At pH = 1 and 6, nickel nanoparticles had particle diameters of 24.78 nm and 24.76 nm, respectively. The findings demonstrated no further variations in the particle sizes of the protective layer generated on the Ni−Cr alloy surface using the electrodeposition approach, even though previous electrochemical methods revealed an obvious shift in protection.

**CONCLUSIONS**

The investigations described in this work indicate that nickel nanoparticles with a pH of 1 are an excellent coating that was successfully formed on a Ni−Cr alloy specimen using the electrodeposition method, exhibiting excellent corrosion resistance in sulfuric acid solution (1 M) saturated with CO$_2$. Electrochemical studies revealed that nickel coatings on the alloy surface reduce corrosion current densities and
increase polarization resistance in corroded acidic solutions. Under fixed voltage, it takes less time to achieve a uniform, compact, and adherent nickel coating. Electrochemical impedance spectroscopy measurements were used to determine the resistance of the protective layer coating. The values for the coatings are significantly higher than those for the bare alloy. The EIS results are consistent with those obtained from potentiodynamic and linear polarization measurements. This

Table 5. XRD Analysis Peaks of Ni–Cr Alloy Surface and Coating Layers of Nickel at pH = 1 and 6

| peak position (2θ) | FWHM (β) | theta (θ) | intensity | crystallite size D (nm) | Miller index |
|--------------------|-----------|-----------|-----------|-------------------------|--------------|
| 36.473             | 1.180     | 18.236    | 0033      | 07.23                   | 202          |
| 44.313             | 0.344     | 22.156    | 1164      | 25.42                   | 111          |
| 51.488             | 0.393     | 25.744    | 0532      | 22.87                   | 200          |
| 54.949             | 0.590     | 27.474    | 0033      | 15.48                   | 242          |
| 73.733             | 0.246     | 37.866    | 0568      | 41.75                   | 220          |
| Ni, pH = 1         |           |           |           |                         |              |
| 44.2089            | 0.3444    | 22.10445  | 1501      | 25.41                   | 111          |
| 51.4195            | 0.3936    | 25.70975  | 656       | 22.87                   | 200          |
| 75.6341            | 0.3936    | 37.81705  | 568       | 26.08                   | 220          |
| Ni, pH = 6         |           |           |           |                         |              |
| 44.0136            | 0.3444    | 22.0068   | 3118      | 25.40                   | 111          |
| 51.2041            | 0.3936    | 25.60205  | 1319      | 22.84                   | 200          |
| 75.5337            | 0.3936    | 37.76685  | 568       | 26.06                   | 220          |

Figure 6. FESEM images and EDX of blank (A) and coating layers of nickel at pH = 1 (B) and pH = 6 (C) on the Ni–Cr alloy surface.

Figure 7. X-ray diffraction pattern of Ni–Cr alloy surface and coating layers of nickel at pH = 1 and 6.
study demonstrates that nickel coatings with a pH of 1 layer exhibit excellent corrosion resistance and can be considered a potential coating material for protecting Ni–Cr alloy from corrosion in a 1 M H2SO4 solution. These observations are confirmed by FESEM, EDX, and XRD analyses.

**AUTHOR INFORMATION**

Corresponding Authors
Qahtan. A. Yousif – College of Engineering, Department of Materials Engineering, University of Al-Qadisiyah, Al Diwaniyah S8001, Iraq; Email: qahtan.adnan@qu.edu.iq
Mohammad A. Bedair – College of Science and arts, University of Bisha, Al-Namas 61977, Saudi Arabia; Department of Chemistry, Faculty of Science (Men’s Campus), Al-Azhar University, 11884 Cairo, Egypt; orcid.org/0000-0002-2236-7837; Email: mbedair@ub.edu.sa, m_bedier@azhar.edu.eg, m_bedier@yahoo.com

Author
Mohammad N. Majeed – College of Education, Department of Chemistry, University of Al-Qadisiyah, Al Diwaniyah S8001, Iraq

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.2c02679

Notes
The authors declare no competing financial interest.

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