

Article

Corrosion Resistance of SAE 5160 Steel Deposited by Duplex Simultaneous Treatment with Hastelloy Cathodic Cage

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Abstract: SAE 5160 steel, classified as high-strength, low-alloy steel, is widely used in the automotive sector due to its excellent mechanical strength and ductility. However, its inherently low corrosion resistance limits its broader application. This study explores the application of the cathodic cage plasma deposition (CCPD) technique to enhance the corrosion resistance of SAE 5160 steel. The treatment was performed using a Hastelloy cathodic cage under two atmospheric conditions: hydrogen-rich (75% H_2 /25% N_2) and nitrogen-rich (25% H_2 /75% N_2). Comprehensive analyses revealed significant improvements in surface properties and corrosion resistance. The hydrogen-rich condition (H25N) facilitated the formation of $Cr_{0.4}Ni_{0.6}$ and CrN phases, associated with a nanocrystalline structure (37.6 nm) and a thicker coating (45.5 μm), resulting in polarization resistance over 290 times greater than that of untreated steel. Conversely, nitrogen-rich treatment (H75N) promoted the formation of Fe_3N and Fe_4N phases, achieving a dense but thinner layer (19.6 μm) with polarization resistance approximately 20 times higher than that of untreated steel. These findings underscore the effectiveness of CCPD as a versatile and scalable surface engineering technique capable of tailoring the properties of SAE 5160 steel for use in highly corrosive environments. This study highlights the critical role of optimizing gas compositions and treatment parameters, offering a foundation for advancing plasma-assisted technologies and alloying strategies. The results provide a valuable framework for developing next-generation corrosion-resistant materials, promoting the longevity and reliability of high-strength steels in demanding industrial applications.

Keywords: cathodic cage; corrosion; duplex simultaneous; Hastelloy; SAE 5160 steel



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1. Introduction

SAE 5160 steel is classified as a high-strength low-alloy (HSLA) steel. It is widely used in the automotive industry, particularly in the manufacturing of springs, due to its excellent properties, such as mechanical strength and ductility [1]. This steel is also used to produce knives, bearings, and wagon wheels, among other applications [2]. However, low-alloy steels exhibit poor corrosion resistance, necessitating the use of surface treatment methods to enhance this property. The application of coatings through surface engineering techniques is a way to increase corrosion resistance, employing methods such as magnetron sputtering [3–6], anodic plasma electrolytic nitriding [7–12], electrodeposition [13–18], hot-dip coating, and plasma nitriding. These surface engineering techniques enable the formation of protective coatings that improve corrosion resistance and contribute to increased durability. The selection of the most appropriate technique depends on the operating conditions and the specific requirements of each application, making it essential to consider factors such as adhesion, coating thickness, and cost-effectiveness [19–22].

Da Silva Filho et al. [14] investigated the electrodeposition process of ASTM A36 steel substrates, depositing pure nickel via the galvanostatic method and nickel combined with myristic acid using the potentiostatic method. The coated samples exhibited hydrophobic behavior, particularly those incorporating myristic acid into the bath, which displayed superhydrophobic behavior (contact angle $> 150^\circ$), thereby contributing to enhanced corrosion protection. The dual-coating sample, first treated by the galvanostatic method and then by the potentiostatic method, exhibited superior corrosion resistance characterized by a lower corrosion current density, a more positive (nobler) corrosion potential, and a higher charge transfer resistance. This improved anticorrosive performance is attributed to hydrophobicity and optimized surface morphology.

Kusmanov et al. [7] applied anodic plasma electrolytic nitriding, followed by quenching, to low-alloy 40Cr steel to investigate the influence of temperature (between 650°C and 850°C) on corrosion and wear resistance. The resulting layers primarily consisted of iron nitride ($\text{Fe}_2\text{-}_3\text{N}$), iron oxide (Fe_3O_4), and martensite. All treatment temperatures enhanced corrosion and wear resistance; lower temperatures provided better wear resistance, while higher temperatures offered superior corrosion resistance.

Costa et al. [23] studied the effect of plasma deposition with a Hastelloy cathodic cage on the corrosion resistance of AISI D6 and AISI 304 steels. The samples were analyzed using potentiodynamic polarization curves, and the treatments proved effective in corrosion protection when conducted at 400°C , showing increased corrosion potential and reduced corrosion current, particularly for D6 steel. Plasma deposition with a cathodic cage is a viable option for corrosion protection. This PVD (Physical Vapor Deposition) technique involves sputtering the target, a cylindrical cage with equidistant holes, surrounding the sample to be coated. This method enables the deposition of nitrides and oxides of metallic elements, depending on the cage material—which serves as a source of metallic elements—and the composition of the treatment atmosphere.

This study applied the cathodic cage deposition technique to SAE 5160 steel to enhance its corrosion resistance. We used a Hastelloy alloy cage containing chromium (Cr) and nickel (Ni) elements, and treatments were conducted under two distinct atmospheric conditions: a hydrogen-rich atmosphere (75% H_2 /25% N_2) and a nitrogen-rich atmosphere (75% N_2 /25% H_2). The cathodic cage deposition technique was applied without an insulator, meaning the samples were subjected to cathodic potential. This configuration yields thicker coatings [24,25], particularly when compared to other techniques, such as hot-dip coating or magnetron sputtering. This treatment configuration closely resembles plasma nitriding; however, the presence of the Hastelloy cage (screen) enables the incorporation of elements such as chromium and nickel into the coating [26,27]. In the literature, this treatment setup

is also known as a duplex simultaneous treatment [25]. Nishimoto et al. (2013) [25] applied plasma nitriding with an active screen (ASPEN) to SACM 645 steel, simultaneously obtaining a TiN coating and a nitrogen diffusion zone on samples treated under cathodic potential.

2. Materials and Methods

SAE 5160 steel samples with a diameter of 19.05 mm and a thickness of 6 mm were ground up to 1200 mesh grit and subsequently polished using an alumina suspension (0.3 μm). The samples were commercially obtained with the following chemical composition range (wt.%): 0.56–0.64% C, 1.35–1.65% Mn, 0.70–0.90% Cr—with Fe as the balance. The samples were directly placed on the sample holder (cathode) without the use of any insulating material and were surrounded by the cathodic cage. The cage employed in the treatments was fabricated from Hastelloy C-276 nickel alloy, with the following composition (wt.%): 47.80% Ni, 22.00% Cr, 9.00% Mo, 1.50% Co, 18.50% Fe, 0.50% Mn, 0.60% W, and 0.10% C. The equipment used in this study consisted of a plasma reactor coupled with peripheral devices for monitoring and control, including a power supply, a vacuum pump, and mass flow controller, as described in previous studies [28,29].

The samples were cleaned via sputtering in an atmosphere composed of 50% H_2 and 50% Ar (with a total flow rate of 90 sccm) for 30 min at a temperature of 350 °C. The treatment conditions are presented in Table 1. The total volumetric gas flow rate in both treatments was 60 sccm ($\text{N}_2 + \text{H}_2$).

Table 1. Treatment conditions.

Sample	Temperature	Time	Gas Ratio	Pressure
H25N	450 °C	4 h	25% N_2 + 75% H_2	260 Pa
H75N			75% N_2 + 25% H_2	120 Pa

For the characterization of the samples, roughness measurements, X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), and electrochemical impedance spectroscopy (EIS) were employed. EIS tests were conducted in triplicate, whereas the remaining analyses were duplicated. Roughness measurements were performed using a portable Mitutoyo profilometer, specifically model SJ-210. Six measurements were taken from each sample, with an evaluation length of 4.0 mm and a cut-off length of 0.8 mm. XRD analysis was performed on a SHIMADZU XRD-6000 diffractometer using $\text{Cu-K}\alpha$ radiation, with a 2θ scan range from 10° to 110° at a scan rate of 1°/min, operating at 40 kV and 30 mA. SEM and EDS analyses were conducted using an FEI Company Quanta FEG 250 microscope with a Bruker XFlash 5010 detector.

Corrosion tests in a 3.5% NaCl solution were conducted at 298 K in an electrochemical cell, which contained a platinum plate (200 mm^2) as the counter electrode and a saturated $\text{Ag(s)}|\text{AgCl(s)}|\text{Cl}^-$ (KCl-saturated) wire as the reference electrode. Untreated samples (base) and coated samples (H25N and H75N) were used as working electrodes with exposed areas of 283 mm^2 . The corrosion behavior of the samples was evaluated through EIS measurements, performed over a frequency range from 20 kHz to 6 mHz with 10 points per frequency decade and a sinusoidal amplitude of 10 mV relative to the open circuit potential (OCP). Before each measurement, OCP values were recorded for a sufficient period (3600 s) to ensure the variation rate ($d\text{WE}/dt$) was below 10^{-6} V/s, indicating stabilization before EIS testing. Impedance data were acquired, and spectra were analyzed using Nova[®] 2.1 software. A Metrohm Autolab PGSTAT-204 potentiostat/galvanostat was employed for corrosion tests.

The impedance spectra data were modeled using equivalent electrical circuits with the EIS Spectrum Analyser[®] software. Finally, polarization curves were obtained within a

potential range of -200 mV to $+700$ mV relative to the OCP at a scan rate of 1 mV/s. All experiments were conducted inside a Faraday cage, with at least three repetitions for each condition evaluated.

3. Results and Discussion

Figure 1 shows the X-ray diffraction patterns of the untreated, H25N, and H75N samples, respectively, along with the calculated profiles obtained through Rietveld refinement and the difference between the calculated and experimental patterns. It also shows the Bragg peak positions of the phases present in the coating. The Rietveld refinement method was employed to identify and quantify the volumetric fraction of each phase. The software used for the Rietveld refinement was ReX 0.9.2.

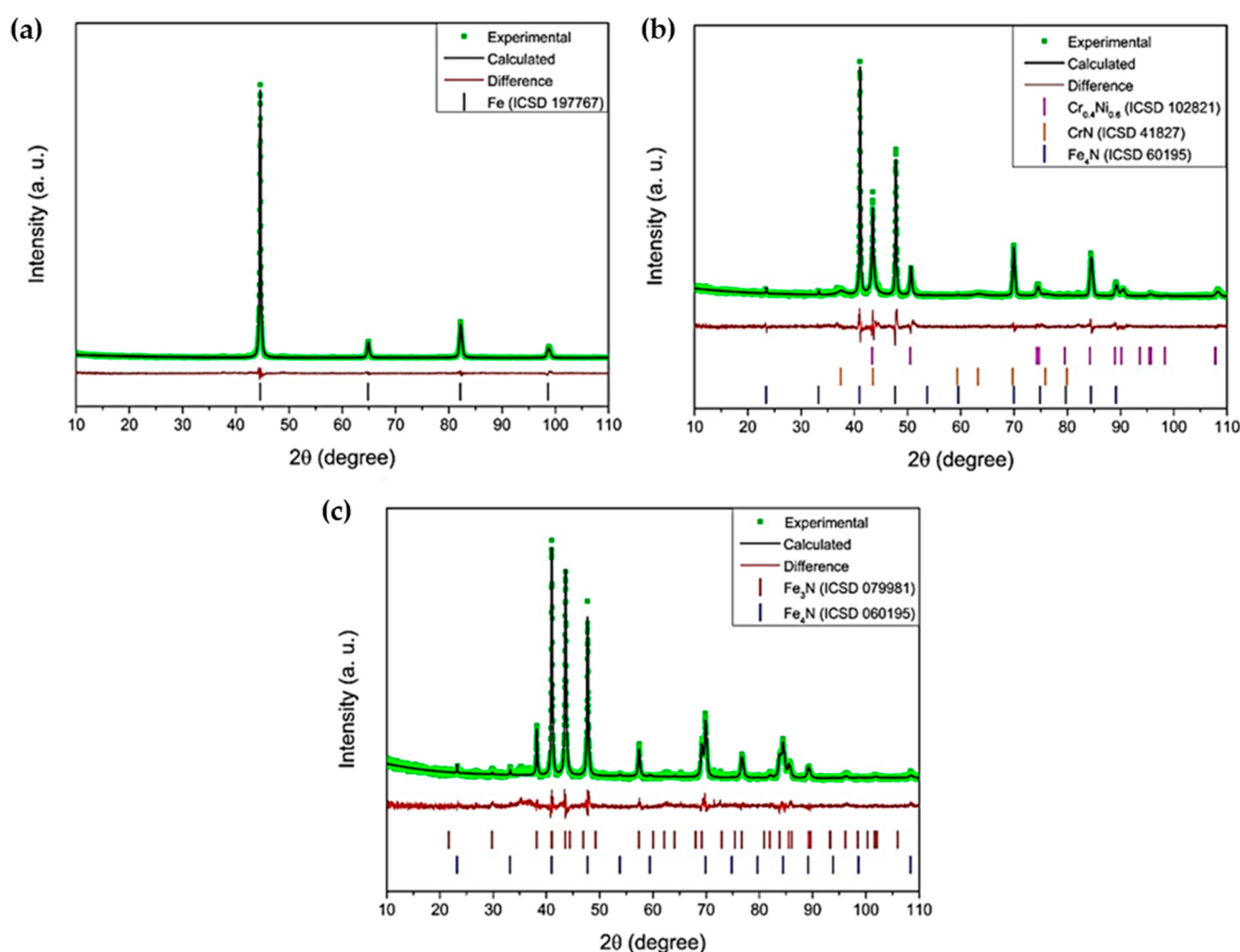


Figure 1. Bragg positions and experimental and calculated diffractograms of the samples: (a) base; (b) H25N; (c) H75N.

The X-ray diffraction pattern of the untreated sample (Figure 1a) shows the characteristic diffraction peaks, corresponding exclusively to the Fe- α phase (ICSD 197767) [30]. According to the XRD pattern of the H75N sample treated in a nitrogen-rich atmosphere (Figure 1c), only iron nitrides, Fe_3N (ICSD 079981) [31] and Fe_4N (ICSD 060195) [32], were detected. This result indicates a more efficient nitriding process, where the higher concentration of iron nitrides, particularly Fe_3N , contributes to increased surface hardness and enhanced corrosion resistance. Additionally, this treatment condition does not promote the formation of phases containing elements diffused from the Hastelloy cage (Cr, Ni,

Mo). Conversely, the H25N sample (Figure 1b) exhibited the presence of $\text{Cr}_{0.4}\text{Ni}_{0.6}$ (ICSD 102821) [33] and CrN (ICSD 041827) [34] phases, in addition to Fe_4N (ICSD 60195), evidencing the diffusion of alloying elements from the Hastelloy cage into the steel surface. The presence of protective phases such as Fe_4N (ICSD 60195) and CrN (ICSD 041827) suggests improved surface corrosion resistance. These results indicate that cathodic cage deposition in controlled atmospheres effectively modifies the surface of SAE 5160 steel, forming phases that enhance its corrosion resistance efficiently. Figure 2 presents the phase percentages obtained through Rietveld refinement for each phase in the coatings.

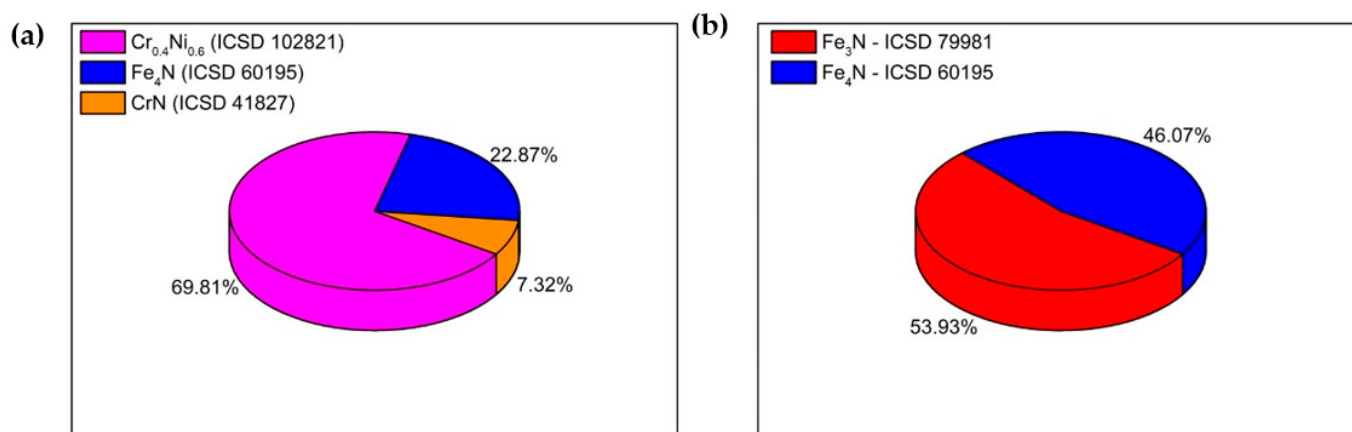


Figure 2. Percentage of the phases present in the coatings of the treated samples (a) H25N and (b) H75N.

The estimated crystallite size of the treated sample layers was determined using the full width at half maximum (FWHM) values of the three most intense diffraction peaks in the Scherrer equation (Equation (1)) [35].

$$D_{hkl} = \frac{K \cdot \lambda}{\beta \cdot \cos \theta'} \quad (1)$$

where D_{hkl} is the crystallite size, K is the Scherrer constant (0.91), λ is the wavelength of the instrument's radiation (0.154 nm), β is the full width at half maximum (FWHM) of the peaks, and θ is the diffraction angle. The results revealed that the deposited layers exhibited nanocrystalline structures with average sizes of 37.5 ± 6.2 nm for the H25N sample and 62.7 ± 5.9 nm for the H75N sample.

Figure 3 presents the SEM and EDS images of the treated sample surfaces. The presence of elements originating from the cathodic cage, comprising primarily nickel, is observed, demonstrating the efficiency of the deposition technique [36]. SEM image analysis also reveals that the H75N sample exhibits a more homogeneous surface than H25N.

The distribution of Ni and Cr elements in the EDS images of the H25N sample (Figure 4) reinforces the presence of the $\text{Cr}_{0.4}\text{Ni}_{0.6}$ phase with nucleation regions of this phase.

Figure 5 shows that the deposition treatments increase roughness (R_a and R_q) compared to the untreated polished 5160 steel substrate. The H25N sample exhibited higher roughness than H75N, a fact corroborated by the SEM images of the sample surfaces (Figure 3). These higher roughness values may be associated with the higher treatment pressure [37], as shown in Table 1.

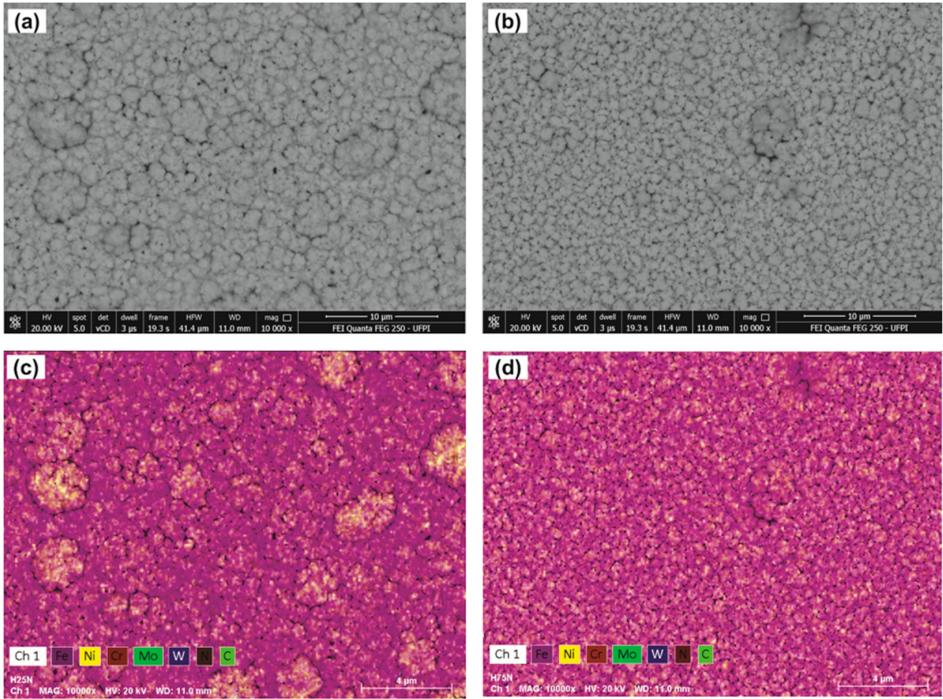


Figure 3. SEM and EDS images of the surface of the treated samples: (a) SEM image—H25N; (b) SEM image—H75N; (c) EDS—H25N; (d) EDS—H75N.

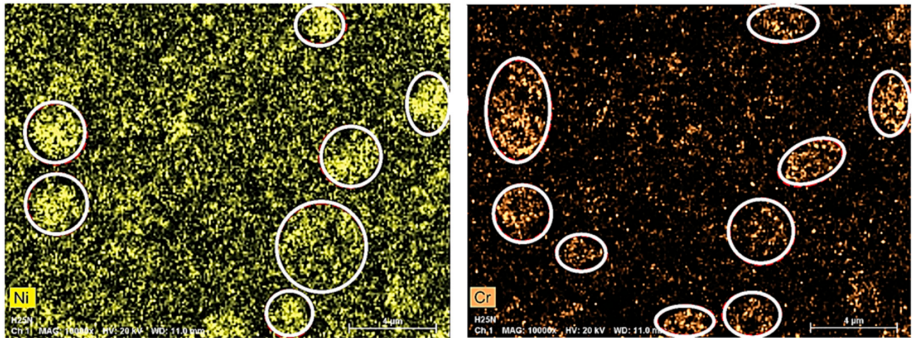


Figure 4. Ni (yellow) and Cr (orange) distribution in sample H25N.

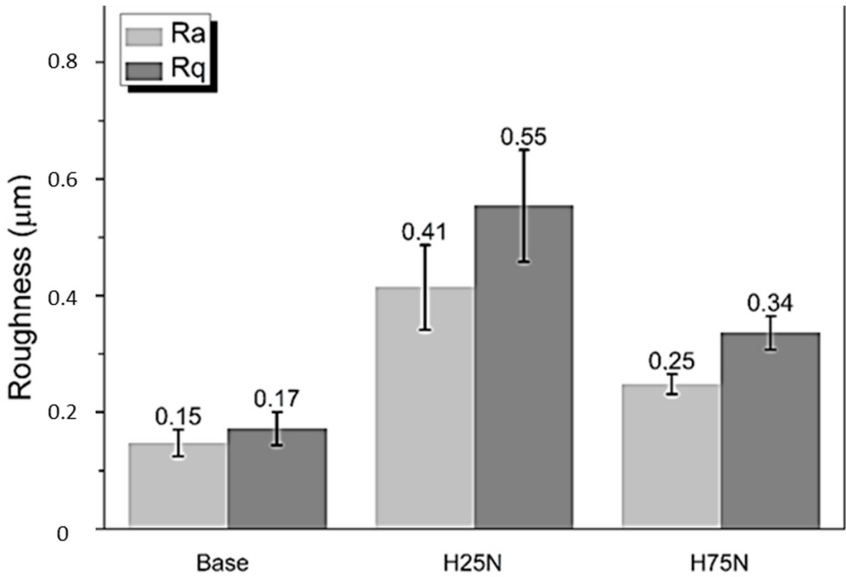


Figure 5. Roughness of the base, H25N, and H75N samples.

In the SEM images of the cross-section of the treated samples (Figure 6), the treatment with a lower proportion of nitrogen (H25N) resulted in the most significant layer thickness, measuring $45.48 \pm 1.90 \mu\text{m}$. The H75N sample showed a thickness of $19.60 \pm 1.09 \mu\text{m}$.

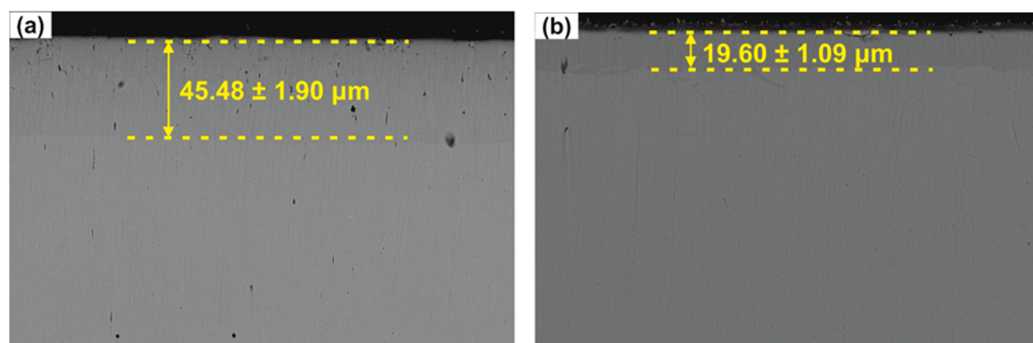


Figure 6. SEM images of the cross-section of the treated samples. (a) H25N, (b) H75N.

Figure 7 illustrates the variation in OCP values of the samples over 60 min. For all samples, fluctuations in corrosion potential are induced by the evolution of thin oxide films formed during monitoring [38]. However, these values tend to stabilize by the end of the monitoring period.

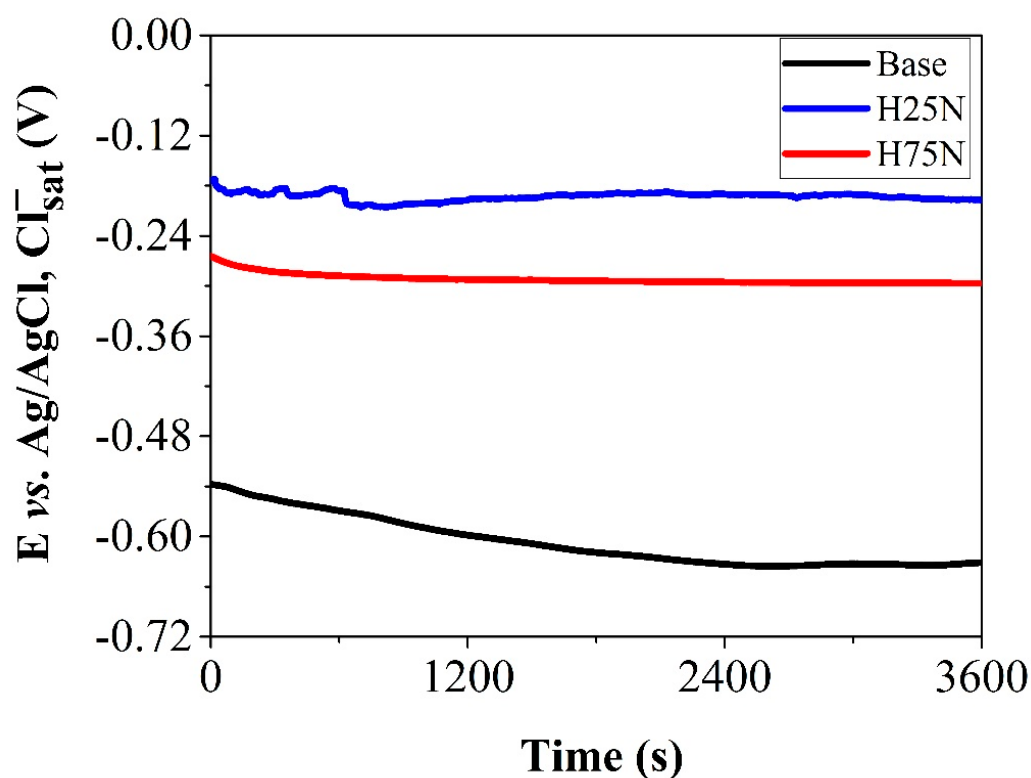


Figure 7. Monitoring the open circuit potential of the base, H25N, and H75N samples in a 3.5% NaCl solution.

Compared to the untreated sample (Base), the H25N and H75N samples exhibited more positive OCP values (Table 2), indicating the formation of a less porous and more stable oxide layer, resulting in lower electrochemical activity in the deposited layers [39,40]. The higher nobility of the H25N sample can be explained by the presence of more noble phases, such as $\text{Cr}_{0.4}\text{Ni}_{0.6}$ and CrN.

Table 2. Values of open-circuit potential (OCP), solution resistance (R_s), constant phase element (CPE), non-ideality factor (n), and polarization resistance (R_p) were obtained from fits of experimental data using the proposed modified Randles circuit.

Sample	E_{corr} (mV)	R_s ($\Omega \text{ cm}^2$)	CPE		R_p ($\text{k}\Omega \text{ cm}^2$)
			Y_0 ($\mu\text{F cm}^{-2} \text{ s}^{n-1}$)	n	
Base	-630 ± 20	2.8 ± 2.1	42.75 ± 5.12	0.72 ± 0.02	3.28 ± 0.18
H25N	-195 ± 15	2.5 ± 2.2	0.15 ± 0.06	0.88 ± 0.03	960.52 ± 0.34
H75N	-295 ± 10	3.1 ± 2.3	0.43 ± 0.11	0.79 ± 0.02	62.02 ± 0.25

The corrosive behavior of the samples was investigated through impedance spectra (EIS), represented by Nyquist (Figure 8) and Bode (Figure 9) plots. For all samples, the Nyquist plots exhibit only one capacitive arc, as confirmed by the Bode plots, which show a single peak in the phase angle. A single arc in the EIS diagrams suggests that only one interfacial process occurs, attributed to the charge transfer phenomenon between the electrode and the electrolyte [41]. Furthermore, qualitatively, Nyquist diagrams with larger diameter arcs and Bode diagrams with higher impedance moduli indicate coatings with greater resistance to charge transfer. Thus, the H25N sample is more resistant to corrosion.

However, for quantitative analysis, the experimental results of the EIS spectra were adjusted using the modified Randles circuit (Figure 10), and the quality of the experimental adjustments (χ^2) was always under 10^{-3} . In this proposed circuit, R_s represents the resistance of the solution, R_p is the polarization resistance of the coating, and CPE is the constant phase element, related to the capacitance of the electrical double layer. CPE, instead of a capacitor, enables the compensation of deviations from ideal dielectric behavior on the inhomogeneous surface, caused by surface roughness and adsorption effects.

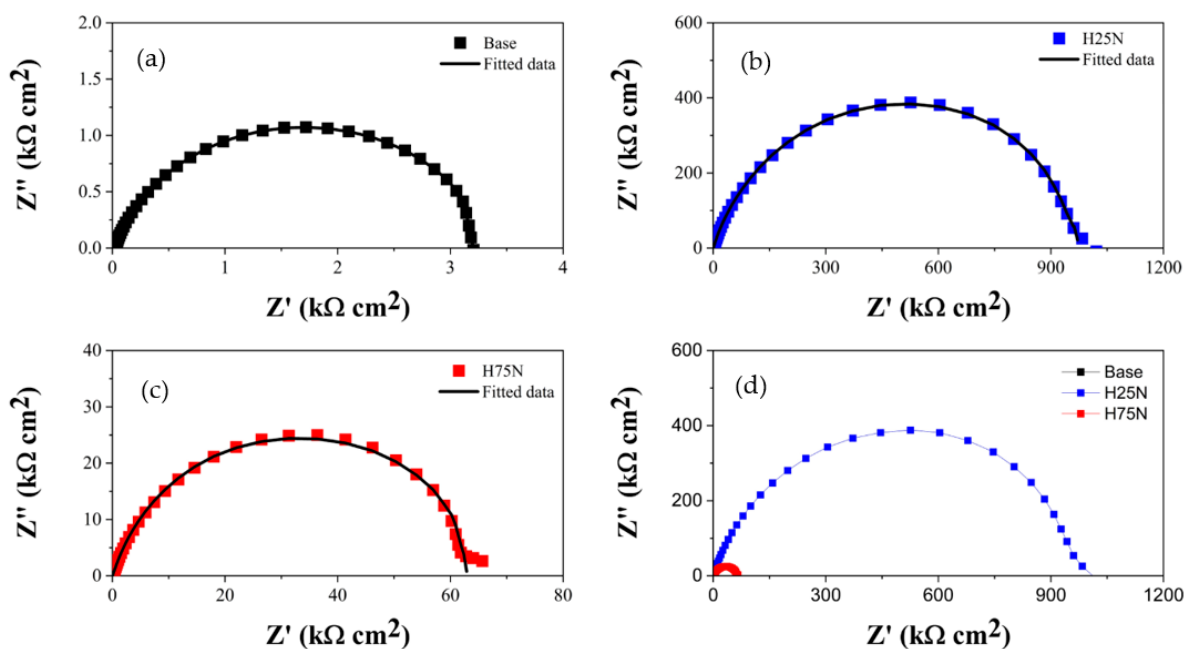


Figure 8. Impedance diagrams of Nyquist representation in 3.5% NaCl solution of (a) base, (b) H25N, (c) H75N, and (d) all samples.

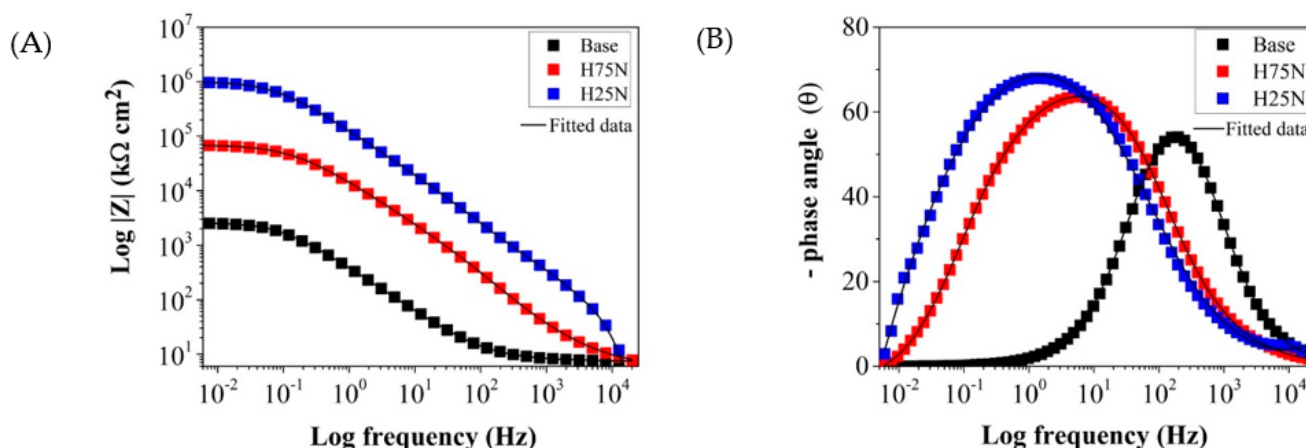


Figure 9. Electrochemical impedance diagrams of 3.5% NaCl solution of the base, H25N, and H75N samples: (A) impedance modulus Bode; (B) phase angle Bode.

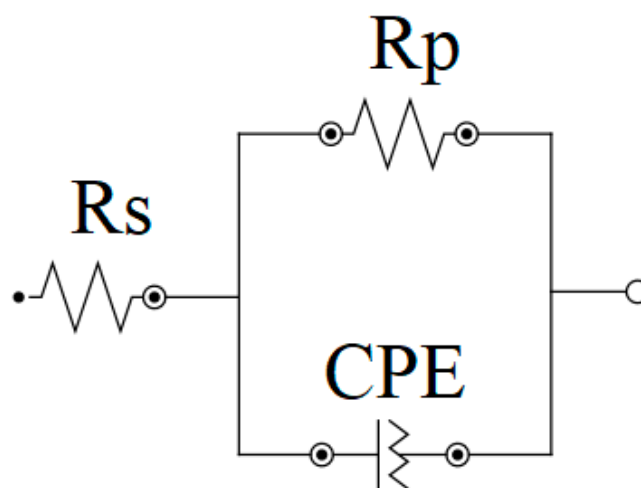


Figure 10. Modified Randles equivalent electrical circuit used to adjust the experimental results of the electrochemical impedance spectra of the base, H25N, and H75N samples.

Table 2 shows the values of the elements of the modified Randles circuit evaluated in each EIS spectrum. The data reveal that the polarization resistance (R_p) of the H75N sample is $62.02 \text{ k}\Omega \text{ cm}^2$, whereas for the untreated steel (base), this value is $3.28 \text{ k}\Omega \text{ cm}^2$, indicating approximately 19 times greater resistance. Boztepe et al. (2018) [42] studied the influence of plasma nitriding on the corrosive behavior of AISI P20 steel in 3.5% NaCl. The authors report the presence of nitrides such as Fe_3N and Fe_4N , which are noble phases and more positive than steel in the electrochemical series. The corrosion resistance value increases when these phases are present without defects in the compact layer.

Table 2 also shows that the polarization resistance of the sample (H25N) is $960.52 \text{ k}\Omega \text{ cm}^2$, making it 15 times more resistant than the H75N sample and 292 times more resistant than the untreated steel (base). It is also worth noting that the increase in R_p values is accompanied by a decrease in the CPE (Y_0) values, reaching $0.15 \mu\text{F cm}^{-2} \text{ s}^{-1}$ for the H25N sample, indicating a lower accumulation of charges on the electrode [41]. Furthermore, the better corrosive behavior of the H25N sample can also be seen in the shift of the maximum phase angle ($\theta_{\max}$) to the lower frequency region (Figure 9B). This value reaches 1.38 Hz for this sample, indicating lower charge transfer kinetics during the corrosion process of these layers [41].

The superior behavior of the H25N sample is attributed to the presence of nobler $\text{Cr}_{0.4}\text{Ni}_{0.6}$ and CrN phases, which were incorporated into the compact layer when the

chemical composition of the gas flow was richer in hydrogen (75%) and when the working pressure was increased to 260 Pa. This treatment condition also led to the formation of crystalline structures at approximately 37.6 nm. In contrast, under the deposition condition of the H75N sample, these structures were estimated to be approximately 62.6 nm, indicating that the reduction in crystallite size contributes to greater corrosion resistance. Similar results were reported by Escobar and Aperador (2014) [43] when they analyzed the effect of grain size on vanadium nitride (VN) and hafnium nitride (HfN) layers using atomic force microscopy (AFM) images. These authors estimated the grain sizes at 78 nm for the VN sample and 58 nm for the HfN samples. The EIS spectra showed that reducing the grain size increased corrosion resistance by 5 times. Another factor contributing to the increase in corrosion resistance is the thickness of the compound layer, which is approximately 19.6 μm for the H75N sample, whereas the layer thickness of the H25N sample is 45.5 μm , thereby presenting a greater barrier effect against chloride ion attack [44].

The polarization curves for SAE 5160 steel and the deposited samples (H25N and H75N) are plotted in Figure 11. Additionally, Table 3 lists the average values of the corrosion potential (E_{corr}) and corrosion current density (i_{corr}) obtained by extrapolating the straight line in the linear region of the cathodic branch to the line positioned at E_{corr} in the polarization curves [45]. The data indicate that for SAE 5160 steel, the corrosion potential was approximately -630 mV, while the corrosion current density was $38.95 \mu\text{A cm}^{-2}$. Notably, for this sample, active corrosion occurred on the electrode surface. Beyond -314 mV, the increase in current density became more pronounced with increasing potential, suggesting possible localized corrosion [46]. However, due to equipment limitations, the current density remained constant after -223 mV until the end of the experiment.

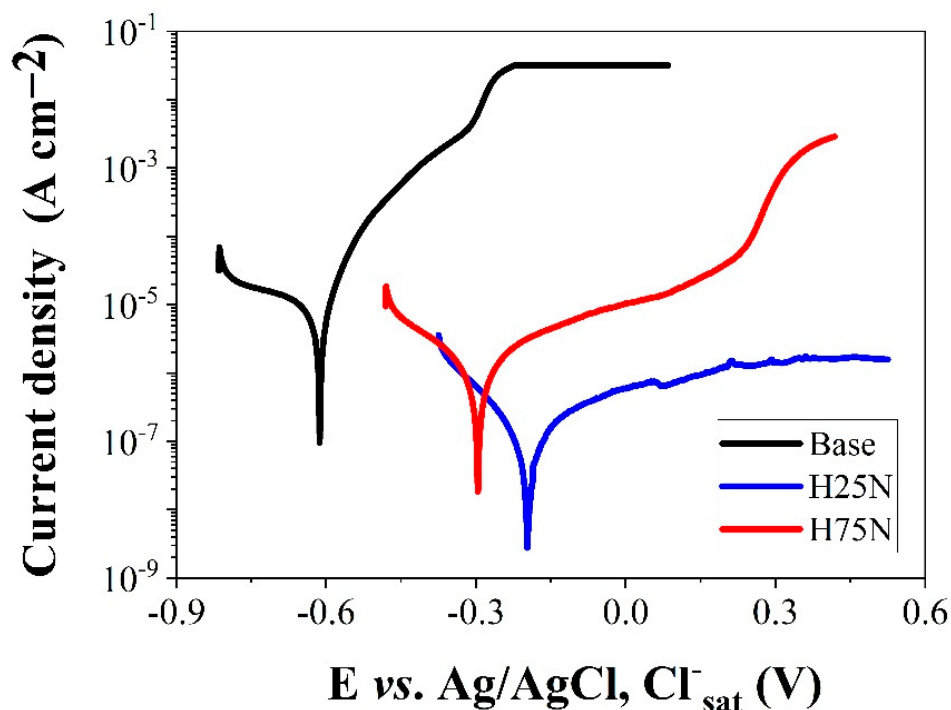


Figure 11. Polarization curves of the base, H25N, and H75N samples, all immersed in a 3.5% NaCl solution.

Table 3. Corrosion potential (E_{corr}) and corrosion current density (i_{corr}) values were obtained from linear polarization curves in a 3.5% NaCl solution.

Sample	E_{corr}/mV	$i_{corr}/\mu\text{A cm}^{-2}$
Base	−630	38.95
H25N	−195	0.25
H75N	−295	1.79

For the deposited sample (H75N), the polarization curve profile was not significantly altered; however, in this experiment, the corrosion potential shifted towards more positive values ($E_{corr} = -295$ mV). This behavior can be attributed to the precipitation of noble phases such as Fe_3N and Fe_4N , as identified by XRD (Figure 1c). Under this treatment, the corrosion current density was approximately $1.79 \mu\text{A cm}^{-2}$, representing a reactivity reduction of almost 22 times compared to SAE 5160 steel. Furthermore, for the anodic branch of the polarization curve corresponding to this sample (H75N), the increase in current density with increasing potential was less pronounced up to -195 mV compared to the anodic curve of SAE 5160 steel. However, beyond this potential, the current density sharply increased, indicating the onset of pitting corrosion [46].

Compared to the other conditions, the polarization curve profile was considerably altered for the deposited samples under condition (H25N). Firstly, the corrosion potential shifted to even more positive values ($E_{corr} = -195$ mV) compared to SAE 5160 steel and the deposited samples under condition H75N. This shift can be attributed to the presence of more noble chromium-rich phases in the compound layer, primarily comprising 69.8% $\text{Cr}_{0.4}\text{Ni}_{0.6}$ and 7.3% CrN (Figure 1b). Secondly, the corrosion current density for the H25N sample was approximately $0.25 \mu\text{A cm}^{-2}$, demonstrating a reactivity reduction of up to 155 times compared to SAE 5160 steel. Thirdly, within the potential range scanned in the anodic branch, the current density increased slowly with potential and remained almost constant from 362 mV to the end of the sweep (530 mV). This behavior indicates that this treatment condition led to the formation of a protective oxide film [47].

4. Conclusions

This study successfully demonstrated the enhancement of corrosion resistance in SAE 5160 steel through the application of cathodic cage plasma deposition (CCPD) using a Hastelloy cage under distinct atmospheric conditions. The findings underscore the significance of controlled deposition environments in tailoring surface properties for industrial applications.

The hydrogen-rich treatment (H25N) promoted the formation of $\text{Cr}_{0.4}\text{Ni}_{0.6}$ and CrN phases, coupled with a nanocrystalline structure averaging 37.6 nm. It resulted in a thick, compact layer ($45.5 \mu\text{m}$) that provided exceptional corrosion resistance—an improvement of 292 times over the untreated steel. This superior performance is attributed to corrosion-resistant chromium-nickel phases and the optimized microstructure, significantly reducing electrochemical activity and enhancing passivation behavior. In contrast, the nitrogen-rich treatment (H75N) led to the formation of Fe_3N and Fe_4N phases, yielding a thinner but dense coating ($19.6 \mu\text{m}$) with a 20-fold increase in polarization resistance. Despite its effectiveness, this treatment showed lower corrosion protection compared to H25N, emphasizing the critical role of chromium incorporation in the coating composition.

Electrochemical impedance spectroscopy (EIS) and polarization studies further validated the enhanced electrochemical stability of the coated samples, particularly that of H25N, which exhibited the lowest corrosion current density ($0.25 \mu\text{A cm}^{-2}$) and the highest polarization resistance ($960.52 \text{ k}\Omega \text{ cm}^2$).

These results highlight the effectiveness of CCPD as a scalable and versatile surface engineering technique for improving the longevity and performance of SAE 5160 steel in aggressive environments. Although the focus of this study was on corrosion resistance, the presence of phases such as CrN and the density of the coatings obtained suggest, based on the literature, the potential for superior performance in terms of wear resistance. Previous studies indicate that coatings containing CrN and Cr–Ni exhibit high hardness and good adhesion—desirable characteristics for applications subjected to tribocorrosion. Therefore, future investigations should include specific wear and tribocorrosion tests to evaluate the mechanical behavior of the developed coatings and their viability for applications where corrosion and wear act synergistically.

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