

Article

Corrosion Behavior of Advanced High-Strength Steels (AHSS) in Chloride Solutions for Automotive Applications

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Abstract

The automotive industry utilizes high-strength low-alloy (HSLA) steels and advanced high-strength steels (AHSS) to manufacture various components, including front and rear rails, chassis, and roll bars, among others. In countries where de-icing salts are used, these steels are exposed to a localized corrosive environment. This research aims to characterize the corrosion behavior of AHSS [dual-phase (DP), ferrite-bainite (FB), and complex-phase (CP)] using electrochemical techniques such as cyclic potentiodynamic polarization (CPP) curves and electrochemical noise (EN), by immersing the steels in NaCl, CaCl₂, and MgCl₂ solutions. Optical microscopy (OM) is used to observe the microstructure of the tested samples. The CPP corrosion behavior of AHSS exposed to chloride solutions exhibits corrosion densities in the range of 10⁻² and 10⁻³ mA/cm². The results generally indicated that AHSS are susceptible to localized corrosion due to the presence of positive hysteresis in the CPP. Zn results show that DP780 presented higher corrosion resistance, with 845 Ω·cm², whereas FB780 presented 253 Ω·cm² when exposed to NaCl. Additionally, the type of corrosion is localized.

Keywords: corrosion; AHSS; electrochemical noise; wavelets; polarization



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1. Introduction

In the automotive industry, the use of advanced high-strength steels (AHSS) continues to grow as they are low-density alloys, a result of increasing demands for passenger safety, vehicle performance, and fuel economy. These steels are explicitly used in suspension structures, chassis components, and bodywork, among other applications. These steels combine high strength and formability to meet external design requirements, while maintaining a lightweight and rigid structure that is sufficient for vehicle maneuverability [1,2].

AHSS are multiphase metal alloys that exhibit ductility not found in conventional mild steels and low-alloy high-strength steels, while also providing a greater balance of

mechanical strength [1]. The type of AHSS encompasses the following generic categories: dual-phase (DP), ferrite–bainite (FB), transformation-induced plasticity (TRIP), complex-phase (CP), and martensitic steels (MART), as well as hot-formed (HF) and other types [3–5]. Depending on the type of AHSS steel, these can have elongations of less than 30% and reach tensile stresses of up to 1700 MPa, compared to conventional steels that typically have values less than 800 MPa and exhibit deformations of around 65% [5–7].

Advanced Applications of High-Strength Steel: Stamping Design and Process Guides, published by the World Steel Association, details part design and geometry, as well as secondary metallurgy processes such as stamping and forming, which result in indicators of mechanical properties [5].

Dual-phase steels have a microstructure composed of bainite or martensite in a ferritic matrix. The microstructure of DP steels can be improved by intercritical heat treatment, where rapid cooling promotes the transformation of austenite into a martensitic phase. To increase the hardness of DP steel (from 228 to 317 HV), a tempering heat treatment is typically performed between 740 and 820 °C to increase the percentage of the martensite phase [4,5].

AHSS of CP grade (complex-phase) exhibit excellent mechanical properties. This steel is used in impact areas, such as the car chassis, due to its high toughness, which allows for the absorption of energy. It is also used in panel interiors and safety cage components [8,9]. Complex-phase steel has a mixed microstructure of fractions of austenite, martensite, and pearlite retained (ferrite “ α ” + iron carbide “ Fe_3C ”) on a ferrite–bainite matrix. The grain size must be refined to achieve good properties through delayed recrystallization, resulting in a fine microstructure with small grains, which provides high tensile strength (UTS), fatigue, and wear resistance [10,11].

FB grade AHSS have a microstructure with a ferrite matrix and a dispersed bainite phase. These phases are obtained by a heat treatment of tempering, austenizing the FB steel at a temperature of 1100 °C for a residence time between 20 and 60 min, followed by air cooling and immersion in a salt bath at temperatures of 500 to 800 °C for a time of 1 h and ending the treatment in a rapid cooling in water [7–9]. The good mechanical properties of these ferritic–bainitic steels are due to grain refinement, the formation of dislocations and precipitates; in addition, they have approximately 22% elongation, while dual-phase steels have only 8% [10–12].

Abedini et al. indicated that an increase in austenitization temperature during the heat treatments of AHSS results in enhanced corrosion resistance, which, of course, depends on the phases transformed as a result of annealing or quenching heat treatments [13]. In contrast, dual-phase (DP) steels exhibit greater corrosion resistance than conventional steels, primarily due to the galvanic coupling between phases, such as martensite and ferrite, or pearlite and ferrite, with the latter phases being more susceptible to attack. Conversely, ferrite–bainite (FB) steels show lower corrosion resistance, as these galvanic interactions are more detrimental than in DP steels. Nevertheless, refining the grain size can improve their resistance, also contributing to better performance against pitting and intergranular corrosion. Alloying elements, such as silicon, contribute to enhancing corrosion resistance; however, their corrosion mechanisms are not well understood, and information on corrosion kinetics is limited [14–19].

The use of electrochemical techniques has enabled significant contributions to various studies on corrosion in AHSS, providing insight into the corrosion kinetics of these alloys in different corrosive environments. Electrochemical techniques polarize the material under study to obtain electrochemical parameters that interpret the dissolution process of steel in the presence of an electrolyte. These conventional techniques include linear polarization resistance (LPR), galvanodynamic polarization (GP), potentiodynamic polarization (PP),

and electrochemical impedance spectroscopy (EIS) [17]. However, there is electrochemical noise (EN), where measurements do not disturb the system under study, and fluctuations in the voltage and current signals occur at the electrode surface due to corrosion processes. The anodic and cathodic reactions are related to the transients and respond to deterministic processes (nucleation and growth of pits) or stochastic processes (rupture and repassivation of the passive layer) [20–25].

In several studies, it has been noted that the corrosion kinetics obtained from electrochemical techniques in AHSS exposed to a 3.5% by weight NaCl solution indicate that when dual-phase steels (DP, ferrite–martensite) or carbon steels (ferrite–pearlite) are present, localized galvanic couples form. The corrosion rate decreases [26–29]. A similar behavior for different dual-phase (DP) steels in the presence of chlorides was reported by authors such as Montoya, M., and Nagiub, A.M. [17,30]. Galvanic corrosion is common in AHSS due to the susceptibility of each phase present [29]. Dual-phase steels exhibit corrosion potentials between those of carbon and martensitic steels. The formation of galvanic couples between the martensite and ferrite phases favors localized corrosion in the ferritic phase when immersed in a sodium chloride solution [31]. Other studies have reported that the corrosion rate in dual-phase DP600 and DP800 steels immersed in a 3.5% NaCl electrolyte indicates that the corrosion resistance of DP800 steel was superior to that of DP600 steel. This assertion is corroborated by the impedance diagrams (Nyquist and Bode), and the corrosion current densities are on the order of 12 and 17 $\mu\text{A}/\text{cm}^2$; these values were determined from potentiodynamic polarization curves, respectively [30]. Other authors have found from their research that dual-phase steel with non-metallic inclusions (manganese sulfides) has the lowest resistance to pitting corrosion in chloride electrolytes [31–33].

This research work aimed to study the corrosion behavior of AHSS (ferritic–bainitic (FB), dual-phase (DP), and complex-phase (CP)) steels using electrochemical techniques, including cyclic potentiodynamic polarization curves (CPP) and electrochemical noise analysis in both the time and frequency domains. The steels were tested in different concentrations of MgCl_2 , NaCl, and CaCl_2 . Microstructural characterization is performed by scanning electron microscopy (SEM). AHSS are used in the automotive industry in environments with heavy snowfall and the use of de-icing salts.

2. Materials and Methods

2.1. Materials

The AHSS used are currently commercially available, have a yield strength (YS) of 780 MPa, and are identified as ferritic–bainitic (FB), dual-phase (DP), and complex-phase (CP) steels. The accepted practice corresponding to the specification of yield strength is as follows: XX aaa (where XX is the type of steel, aaa is the minimum YS in MPA), for example, DP 780 designates dual-phase steel with 780 MPa minimum yield strength. AHSS derive their properties from a multiphase, complex microstructure; FB steel is composed of ferrite–bainite, DP is composed of ferrite–martensite, and CP contains small amounts of martensite, retained austenite, and pearlite within a ferrite–bainite matrix. The chemical compositions of the FB, DP, and CP AHSS used in this study were determined by X-ray fluorescence (Olympus DELTA XRF, Houston, TX, USA) and are listed in Table 1.

2.2. Microstructural Characterization

Using a metallographic technique, samples of AHSS steel were prepared [34]. Different grades of SiC sandpapers, ranging from 280 to 4000, were used. Polishing of the samples was performed using a 0.1 μm alumina solution. Microstructure revelation was carried out with 5% weight Nital solution. Finally, scanning electron microscopy (SEM, JEOL-JSM-

5610LV, Tokyo, Japan) at $2000\times$ magnification. A secondary electron (SE) detector (JEOL, Tokyo, Japan) was used to investigate the microstructure of the samples.

Table 1. Chemical composition of different AHSS (wt.%).

Element	Dual-Phase DP780	Ferritic–Bainitic FB780	Complex-Phase CP780
Fe	Balance	Balance	Balance
C	0.10	0.09	0.09
Mn	2.61	1.73	1.669
Cr	0.420	0.640	0.771
Mo	-	0.006	-
Si	0.510	0.300	0.511
Ti	0.080	0.021	0.007
Nb	-	-	0.045

2.3. Electrochemical Technique

Electrochemical measurements were conducted to determine the corrosion behavior of AHSS using CPP curves and electrochemical noise. Samples were exposed to three electrolytes (all solutions were prepared with distilled water and contained the specified weight percentage of each chemical compound): 3.5% weight NaCl, 2% weight CaCl₂, and 2% weight MgCl₂, at room temperature, with an exposed area of 1.0 cm². A potentiostat/galvanostat/ZRA (produced by Solartron 1287A, Bognor Regis, UK) was used. A corrosion cell with a three-electrode (Figure 1a) configuration was employed: the working electrode (WE) was made of AHSS steel, the reference electrode (RE) was a saturated calomel electrode (SCE), and the auxiliary electrode (AE) was a platinum mesh. Corrosion tests were performed in duplicate at room temperature. The samples were a plate of AHSS.

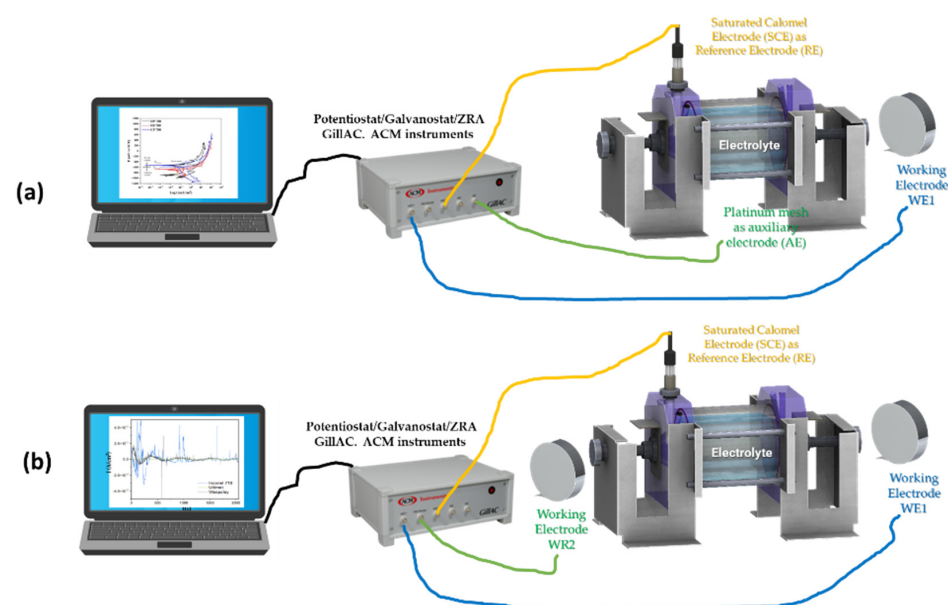


Figure 1. Three-electrode cell for: (a) cyclic potentiodynamic polarization curves (CPP), and (b) electrochemical noise (EN) measurements.

CPP tests [35–37], were performed in accordance with ASTM G61-86 [36], initially measuring the open-circuit corrosion potential (E_{corr}). Once the potential is stable, it is polarized within a range of -1 V to $+1.2$ V relative to the equilibrium potential (E_{corr}) scanned at 60 mV/min.

At room temperature, EN measurements were conducted using a standard three-electrode configuration (Figure 1b). Two identical samples were designated as the working electrodes (WE1 and WE2), and a saturated calomel electrode functioned as the reference electrode (RE). All measurements followed the guidelines established in ASTM G199-09 [38–41]. A scan rate of one data point per second was used to measure 1024 data points in each test. To process the data analysis obtained from the EN measurements, a program developed at the CIIIA eCorrosion Laboratory was used, using MATLAB 2018a software (MathWorks, Natick, MA, USA).

This type of research aims to analyze situations where corrosive environments, such as those caused by de-icing salts, lead to deterioration due to localized pitting corrosion in advanced high-strength steels.

3. Results and Discussion

3.1. Microstructure

Figure 2 shows the microstructures of AHSS. Figure 2a Hard islands martensite, or bulk martensite, and a soft ferrite phase, or black grains, make up the microstructure of DP780 steel. Figure 2b shows the microstructure of FB780 steel, which is composed of fine bainite (bright grains) and a soft ferrite matrix (dark grains) [42–45]. Figure 2c shows the microstructure of CP780 steel, where there is the martensite structure, the retained austenite, and the ferrite–bainite matrix [42–48].

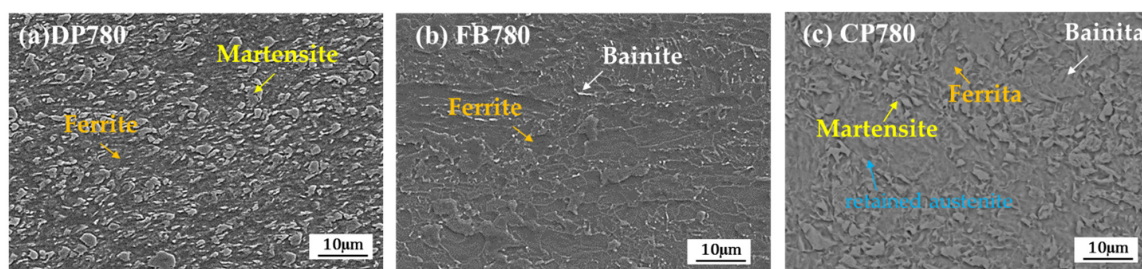


Figure 2. SEM—SE microstructures of (a) DP780, (b) FB780, and (c) CP780 steels.

3.2. Cyclic Potentiodynamic Polarization

The corrosion behavior of AHSS was analyzed using CPP, where oxidation and reduction reactions were studied to obtain characteristic electrochemical parameters, including corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic-cathodic potential ($E_{\text{a-c}}$), pitting potential (E_{pit}), and, of course, corrosion rate. Corrosion kinetics were calculated using the Tafel extrapolation technique, where the analysis was performed within a range of ± 300 mV in the linear section of the potentiodynamic polarization curves, at least a decade of current to obtain resistance to polarization (R_p) [49–55].

The results of this study on AHSS show typical polarization curves, which are Potential (E) vs. $\log(i)$. In the CPP, a positive hysteresis loop is observed as the potential sweep is reversed, indicating that AHSS are susceptible to localized corrosion. The localized corrosion of these steels represents their susceptibility to dissolution, which can be expressed through the breakdown potential (E_{pit}), the point at which localized corrosion begins, and the repassivation potential (E_r), also known as the protection potential (E_p). E_{pit} is often associated with the current density when it suddenly increases and with the rupture of its passive surface film; this is called transpassivation. The higher the value of the potential (more noble), the lower the probability that the steel will have the appearance of localized corrosion. At the repassivation or protection potential (E_p), the pits stop [4,17,51,56].

Figures 3–5 show the electrochemical behavior through the cathodic and anodic reactions of the CPP curves. Figure 3 shows all AHSS exposed to 3.5 wt % NaCl. Only

activation regions are observed in the anodic regions, and a small concentration effect is observed in the cathodic regions, possibly due to oxygen evolution. The hysteresis is positive (regressing in the cyclic potentiodynamic polarization curves), indicating that AHSS are susceptible to localized corrosion. Current density values, i_{corr} , are in the range of 10^{-2} and 10^{-3} mA/cm². Corrosion potential values (E_{corr}) are active in the range of -525 to -883 mV relative to the standard calomel electrode (SCE). DP 780 exhibits a distinct behavior, characterized by a double reaction in the anodic branch. This is associated with the formation of an unstable oxide layer on the surface, resulting in pitting and re-passivation. It also occurs due to changes in electrolyte concentration at the bulk scale, which is associated with pH and the reduction in oxides. All the anodic sites presented this unstable behavior.

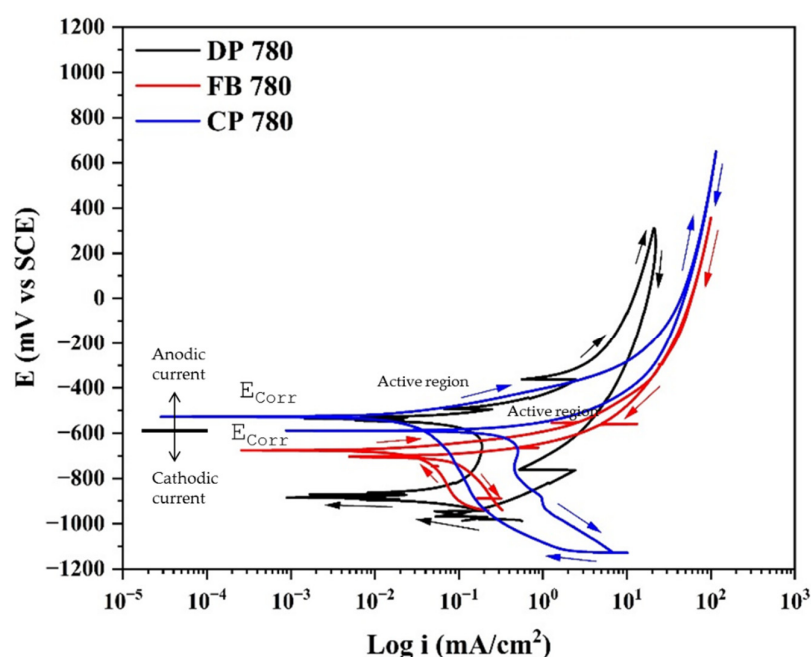


Figure 3. Potentiodynamic polarization curves for AHSS, exposure in NaCl solution.

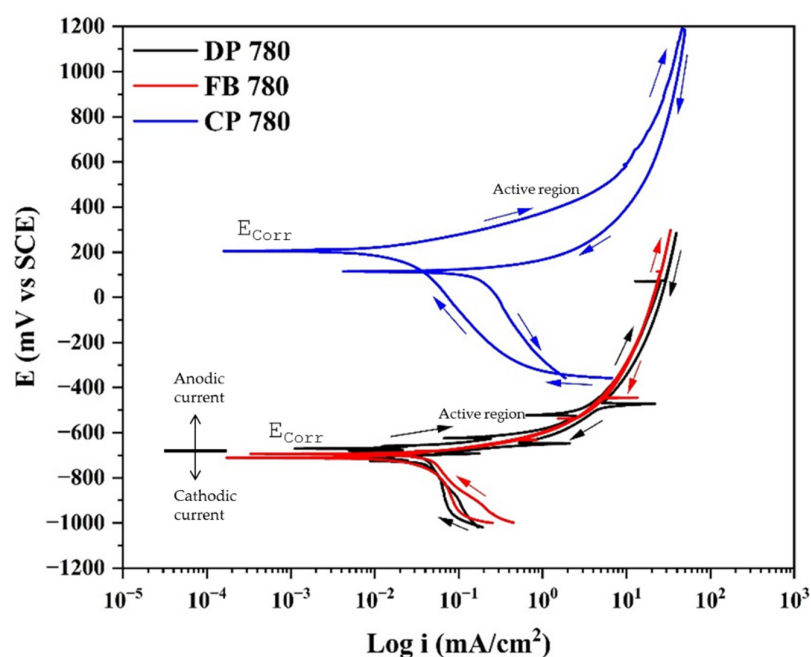


Figure 4. Potentiodynamic polarization curves for AHSS exposed in CaCl₂ solution.

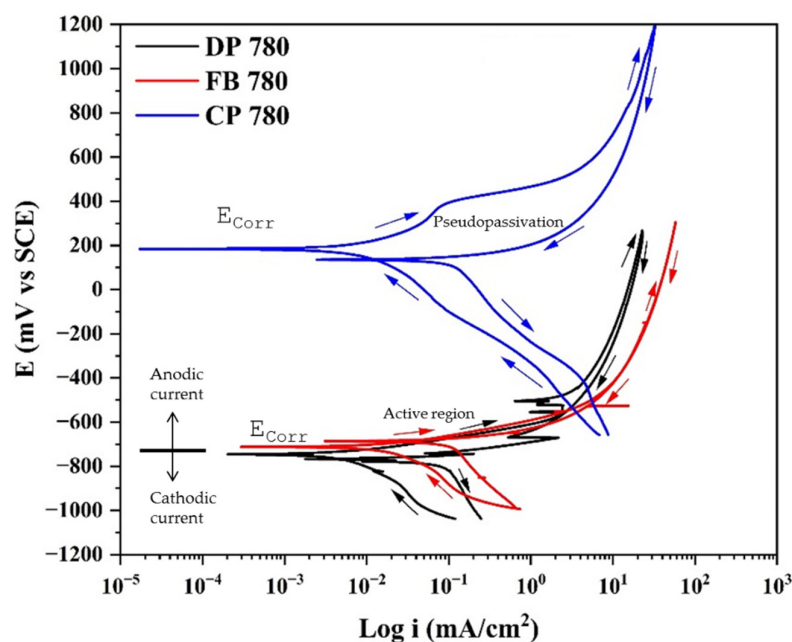


Figure 5. Potentiodynamic polarization curves for AHSS exposed in MgCl_2 solution.

Figure 4 shows all AHSS exposed to 2% wt. CaCl_2 . Mixed behavior is observed in the anodic regions for cathodic and anodic reactions, with activation regions in the anodic region. The hysteresis is positive, indicating that AHSS are susceptible to localized corrosion; the current density values, i_{corr} , are in the range of 10^{-2} and 10^{-3} mA/cm^2 . The corrosion potential values (E_{corr}) for FB 780 and CP 780 steels are active in the range of -670 to -710 mV (see Table 2). In contrast, CP 780 steel has a low corrosion potential of $+200$ mV vs. SCE.

Table 2. Obtained electrochemical parameters for AHSS exposure to NaCl, CaCl_2 , and MgCl_2 solutions.

AHSS Steels	Solution	E_{corr} (mV vs. SCE)	R_p ($\Omega \cdot \text{cm}^2$)	E_{pit} (mV)	i_{corr} (mA/cm^2)	Hysteresis
DP 780	NaCl	-883	38	297	1.69×10^{-2}	Positive
FB 780		-678	9.62	335	1.98×10^{-2}	Positive
CP 780		-525	1133.1	638	2.20×10^{-3}	Positive
DP 780	CaCl_2	-670	24	275	2.18×10^{-2}	Positive
FB 780		-710	28.7	296	1.30×10^{-2}	Positive
CP 780		205	1754.6	1173	1.63×10^{-3}	Positive
DP 780	MgCl_2	-748	41	255	3.76×10^{-3}	Positive
FB 780		-709	16.5	303	1.502×10^{-2}	Positive
CP 780		184	3595.3	1178	1.62×10^{-3}	Positive

Figure 5 shows the corrosion behavior of all AHSS exposed to 2% wt. MgCl_2 . In the anodic regions, a mixed behavior is observed in the cathodic and anodic reactions, with activation regions present only in the anodic region for FB 780 and CP 780 steels. However, CP 780 steel exhibits activation in the anodic region followed by pseudopassivation. The hysteresis is positive, indicating that AHSS are susceptible to localized corrosion; the current density values, i_{corr} , are in the order of 10^{-2} and 10^{-3} mA/cm^2 . The corrosion potential (E_{corr}) values for FB 780 and CP 780 steels range from -709 to -748 mV, with CP 780 steel having a low corrosion potential of $+184$ mV vs. SCE.

Almost all the samples from Figures 3–5 (except CP 780 from Figure 5) presented an oxygen reduction reaction controlled by mass transportation. For that reason, the current increase is abrupt.

3.3. Electrochemical Noise

3.3.1. Time-Domain Analysis

The EN signal consists of random, stationary, and direct current (DC) components. To properly analyze EN data, it is essential to isolate the DC part from the random and stationary components, since the DC contribution introduces false frequencies and disrupts visual, statistical, and PSD evaluations. By eliminating the DC, the corrosion information observed at low frequencies is preserved [48]. EN can be expressed using Equation (1) [57–59]:

$$x(t) = m_t + s_t + Y_t \quad (1)$$

$$y_n = x_n - \sum_{i=0}^{p_0} a_i n^i \quad (2)$$

where $x(t)$ is the time-series, m_t is the DC signal, s_t is the stationary signal, and Y_t is the dynamic component. Within the polynomial framework, the noisy signal (x_n) is represented by a polynomial of degree n (p_0), where the coefficient of the n th term is denoted as a_i at time n . y_n is the signal after the polynomial filter.

To determine noise resistance (R_n), the standard deviation of the time-series data must be calculated, as this statistical parameter provides insight into corrosion kinetics and mechanisms. Cottis and Turgoose [60] reported that both variance and standard deviation increase with higher corrosion rates. The standard deviation (σ) is obtained using Equation (3), where the average ($\bar{x}$) is calculated for each value (x_i) and divided by the number of data points. At the same time, R_n can be derived from the EN time series (EPN and ECN) and sample areas (A) through Equation (4).

$$\sigma_x = \sqrt{\overline{x^2}} = \sqrt{\frac{\sum_1^N (x_i - \bar{x})^2}{N}} \quad (3)$$

$$R_n = \frac{\sigma_v}{\sigma_I} \times A \quad (4)$$

Since R_n and R_p are correlated, the Stern–Geary equation (Equation (5)) can be applied as an analog relation to evaluate corrosion kinetics. In this Equation, B is a constant, typically assigned values of 0.026 V for active corrosion and 0.052 V for passive corrosion [17,60–62].

$$R_n = \frac{B}{i_{\text{corr}}} = \frac{(\Delta E)}{(\Delta i)} \quad (5)$$

Several studies have linked statistical analyses of EN signals to corrosion behavior on metal surfaces [30,63–65]. The I_{rms} is determined by Equation (4), where X represents the average of the EN data, n is the number of data points, and σ is the standard deviation.

$$\text{rms} = \sqrt{Xn^2 + \sigma^2} \quad (6)$$

With standard deviation and I_{rms} , the localization index can be calculated:

$$\text{LI} = \frac{\sigma_i}{I_{\text{rms}}} \quad (7)$$

Values obtained can be associated with the system's corrosion type [66,67]. This research will consider additional parameters to determine the type of corrosion (Table 3).

Table 3. Corrosion types evaluated by the Localization Index (LI).

Corrosion Type	LI
Localized	1.0–0.1
Mixt	0.1–0.01
Uniform	0.01–0.001

In this work, skewness and Kurtosis were used to determine the type of corrosion. The localization index (LI) was not considered, as Mansfeld and Sun [68] demonstrated in 1995 that LI may present limitations and should be applied cautiously. Later, in 2001, Reid and Eden [69] patented a method for corrosion classification based on statistical moments, specifically skewness and Kurtosis (Equations (8) and (9)), which correspond to the third and fourth moments [70].

$$\text{skewness} = \frac{1}{N} \sum_{i=1}^N \frac{(x_i - \bar{x})^3}{\sigma^3} \quad (8)$$

$$\text{kurtosis} = \frac{1}{N} \sum_{i=1}^N \frac{(x_i - \bar{x})^4}{\sigma^4} \quad (9)$$

It is essential to note that statistical calculations are accompanied by a standard error (SE), which introduces uncertainty. This error can be expressed as a function of N, the number of data points analyzed [71]. Consequently, as the dataset size increases, the standard error decreases, thereby improving the reliability of the results, see Equation (10).

$$\text{SE} = \sqrt{\frac{24}{N}} \quad (10)$$

With an SE of 0.153, this value was adopted as the measure of uncertainty for the reported results. Table 4 provides the classification of corrosion type as determined from Kurtosis and skewness:

Table 4. Corrosion types evaluated by Kurtosis and skewness [69].

Corrosion Type	Potential		Current	
	Skewness	Kurtosis	Skewness	Kurtosis
Uniform	<±1	<3	<±1	<3
Pitting	<−2	>>3	>±2	>>3
Transgranular (SCC)	4	20	−4	20
Intergranular (SCC 1)	−6.6	18 to 114	1.5 to 3.2	6.4 to 15.6
Intergranular (SCC 2)	−2 to −6	5 to 45	3 to 6	10 to 60

Figure 6 shows the electrochemical potential and current noise of AHSSs exposed to NaCl. Figure 6a shows the electrochemical potential noise (EPN), which indicates the behavior of all the alloys presented. However, CP780 presented a higher amplitude of potentials, with values of 2×10^{-2} V. On the other hand, DP780 and FB780 presented amplitude values of 3×10^{-3} V, indicating that electrolytic transference is lower. The ECN of Figure 6b shows that CP780 obtained amplitude values of 1×10^{-4} A/cm², indicating that its corrosion kinetics are higher than those of DP780 and FB780, which obtained values of 2×10^{-6} A/cm². Table 5 presents the results of statistical parameters, indicating that the LI of all the alloys ranged from 0.016 to 0.081, suggesting that corrosion is mixed on the surface. The values obtained for skewness indicated that DP780 and FB780 exhibited uniform corrosion, whereas CP780 showed localized corrosion by this method. The results

of R_n showed that CP780 presented $81 \Omega \cdot \text{cm}^2$, indicating that its corrosion resistance is the lowest of the three alloys. FB780 presented the highest value, at $584.81 \Omega \cdot \text{cm}^2$.

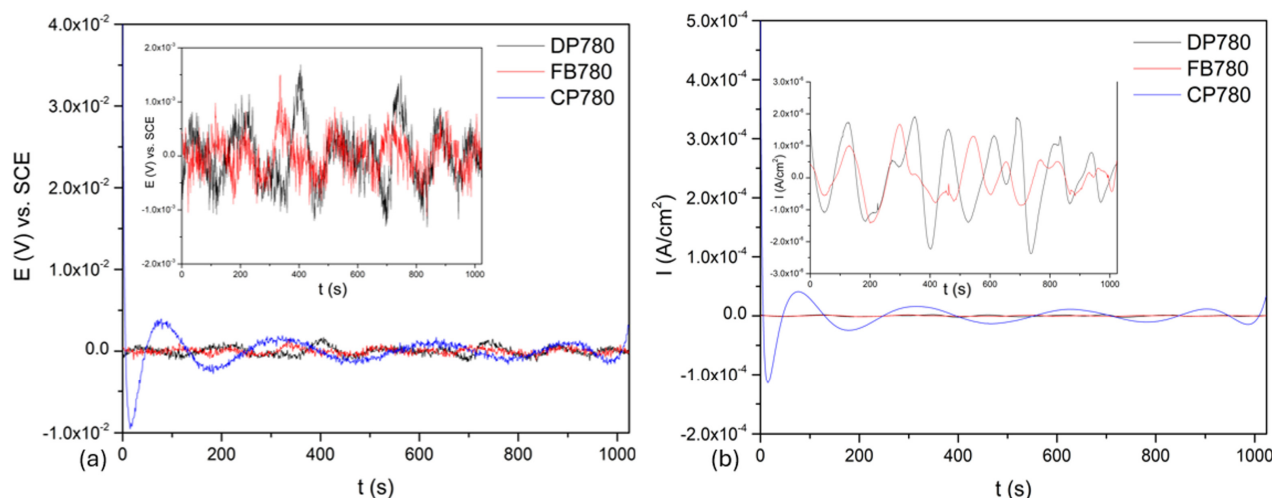


Figure 6. Electrochemical current and potential noise-time series for AHSS in potential (a) and current (b) exposure to NaCl solution.

Table 5. Parameters obtained by statistical analysis.

Alloy	LI	Kurtosis	Skewness	$R_n (\Omega \cdot \text{cm}^2)$
NaCl				
DP780	0.016	2.3	−0.17	531
FB780	0.037	2.9	0.3	584
CP780	0.081	147	8	81
CaCl ₂				
DP780	0.022	1.98	−0.06	384
FB780	0.008	2.09	0.07	1026
CP780	0.007	2.9	0.01	209
MgCl ₂				
DP780	0.01	2.4	−0.53	972
FB780	0.17	5.9	1.17	700
CP780	0.016	10	0.26	124

Figure 7 shows the EPN and ECN of AHSS exposed to CaCl₂. Figure 7a shows the EPN of AHSSs; all the alloys presented a similar behavior with fluctuations. However, the ECN from Figure 7b shows that CP780 presented a higher amplitude with $10 \times 10^{-6} \text{ A/cm}^2$, indicating a higher corrosion kinetics. R_n supports this, with a value of $209 \Omega \cdot \text{cm}^2$. In this electrolyte, FB780 presented a resistance of $1026 \Omega \cdot \text{cm}^2$, indicating the highest value and suggesting high corrosion resistance. It is worth noting that the type of corrosion in this electrolyte is predominantly uniform. LI showed results of 0.022 for DP780 (mixed corrosion) and 0.007, which indicated uniform corrosion. Skewness presented values ranging from 0.07 to −0.06, indicating a uniform corrosion process on the material surface.

Figure 8 shows the EPN and ECN of AHSS exposure to MgCl₂. Figure 8a presents the EPN signal, and FB780 presents various steps, which are associated with the degradation of the material. In ECN, the behavior is the same, indicating that the material presented pitting. The results of the LI for this sample are 0.17, which is associated with localized corrosion. The skewness is at 1.17, which also indicates localized corrosion. Additionally, CP780 exhibited lower corrosion resistance with a value of $124 \Omega \cdot \text{cm}^2$. DP780 exhibited higher corrosion resistance, with an electrical resistance of $972 \Omega \cdot \text{cm}^2$.

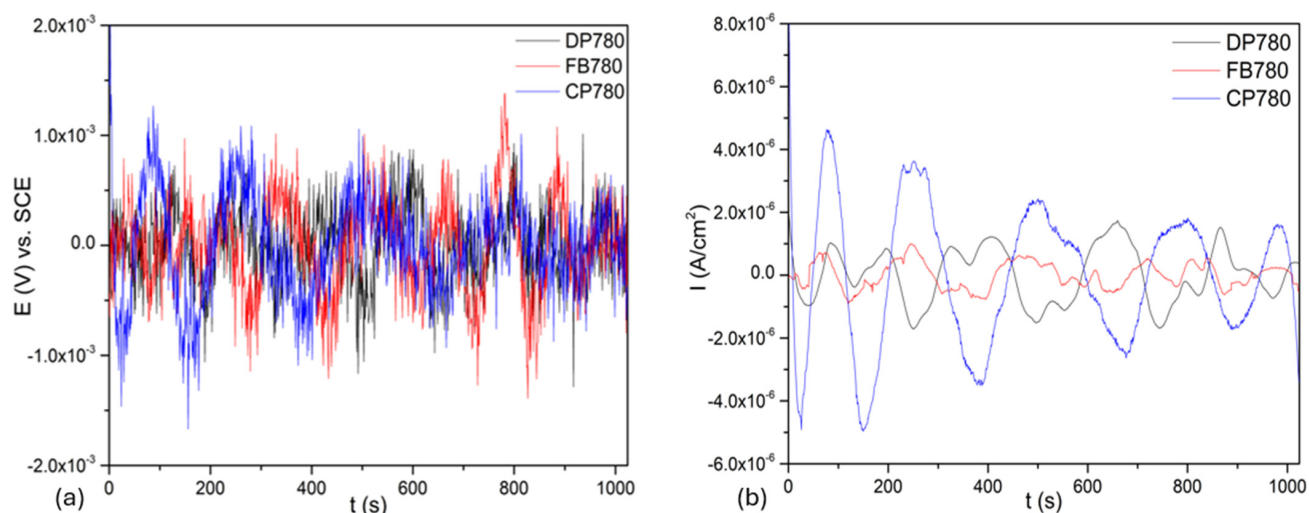


Figure 7. Electrochemical current and potential noise-time series for AHSS in potential (a) and current (b) exposure to CaCl_2 solution.

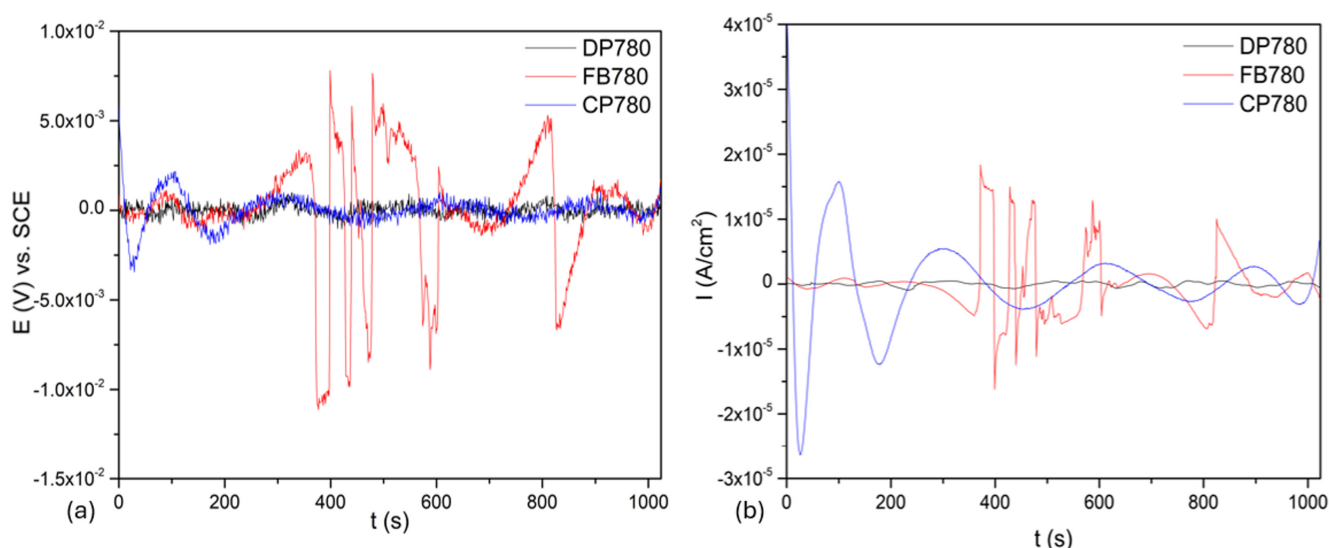


Figure 8. Electrochemical current and potential noise-time series for AHSS in potential (a) and current (b) exposure to MgCl_2 solution.

3.3.2. Frequency Domain Analysis

Given the correlation observed in the EN signal (after applying a polynomial filter), PSD analysis requires converting the time-domain EN data into the frequency domain through FFT. Once the spectral density is obtained, Equations (11) and (12) can be applied [71–73], where $R_{xx}(m)$ represents the signal correlation.

$$R_{xx}(m) = \frac{1}{N} \sum_{n=0}^{N-m-1} x(n) \cdot x(n+m), \text{ when values are from } 0 < m < N \quad (11)$$

$$\Psi_x(k) = \frac{\gamma \cdot t_m}{N} \cdot \sum_{n=1}^N (x_n - \bar{x}_n) \cdot e^{\frac{-2\pi i k n^2}{N}} \quad (12)$$

In the analysis of the PSD, the limit frequency is taken as the cutoff frequency. This parameter defines the start and end points of a slope, which helps characterize the corrosion mechanism. The cutoff frequency also provides insight into the sample's behavior after it has been subjected to pitting. The slope, detailed in Equation (13), is denoted as β_x [74].

$$\log \Psi_x = -\beta_x \log f \quad (13)$$

The zero-frequency limit (ψ^0) yields information about material degradation, since the power of the PSD is directly related to the total energy in the system. It is important to note that only the current PSD reflects material dissolution. To classify corrosion phenomena on material surfaces, Mansfeld et al. [75–78] proposed in 1998 the use of Table 6, where data are adjusted to decibel units.

Table 6. Electrochemical parameters (β intervals to indicate the type of corrosion).

β Intervals	Corrosion Type					
	Uniform		Pitting		Passive	
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum
$\text{dB(V)} \cdot \text{Decade}^{-1}$	0	−7	−20	−25	−15	−25
$\text{dB(A)} \cdot \text{Decade}^{-1}$	0	−7	−7	−14	−1	1

Figure 9 shows the PSD in potential and current. The results indicate that at low frequencies, the CP780 exhibited a higher potential. The behavior remains the same; the CP780 presented a ψ^0 of $-85 \text{ dBi (A}^2 \cdot \text{Hz}^{-1})$, as shown in Table 4, indicating that electronic transference is higher, which means a faster corrosion kinetic. The FB780 presented a lower value of ψ^0 , resulting in homologs to ECN.

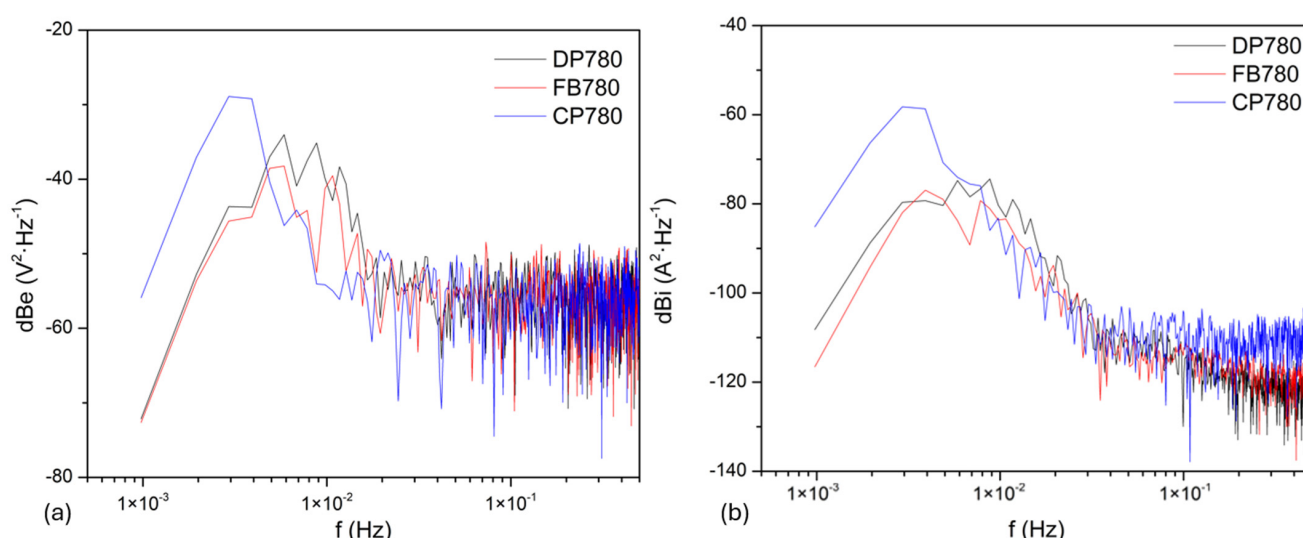


Figure 9. Power spectral density (PSD) in potential and current for AHSS in potential (a) and current (b) exposure to NaCl solution.

Figure 10 shows the PSD in potential and current for AHSS exposure to CaCl_2 . The behavior of Figure 10a in potential presents a similar behavior with a slope near 0, indicating that the corrosion behavior is stable. Figure 10b shows the PSD in current, where CP780 presents a value of $102 \text{ dBi (A}^2 \cdot \text{Hz}^{-1})$, indicating a higher corrosion kinetic. To support these results, it is necessary to evaluate the noise impedance (Z_n).

For MgCl_2 , the PSD (Figure 11) results show that CP780 continuously presents the highest corrosion kinetic with a ψ^0 of $-93 \text{ (A} \cdot \text{Hz}^{-1})$, indicating that this alloy is the most susceptible to corrosion among all the samples. DP780 alloy presented a lower corrosion kinetic rate of $-118 \text{ (A} \cdot \text{Hz}^{-1})$. The results of the next section support this behavior.

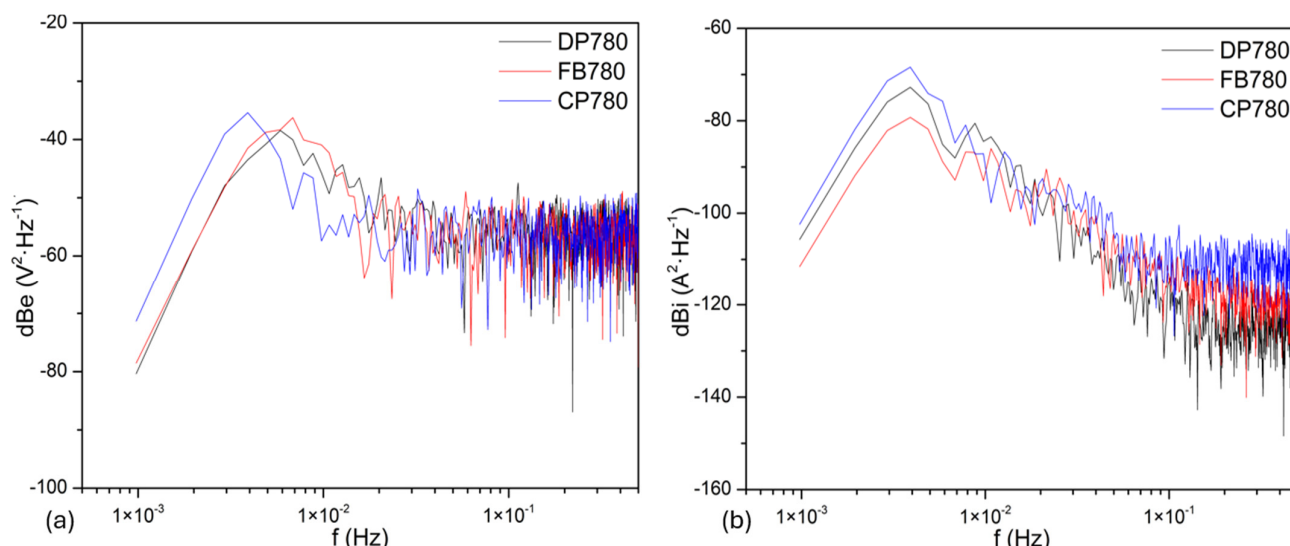


Figure 10. Power spectral density (PSD) in potential and current for AHSS in potential (a) and current (b) exposure to CaCl_2 solution.

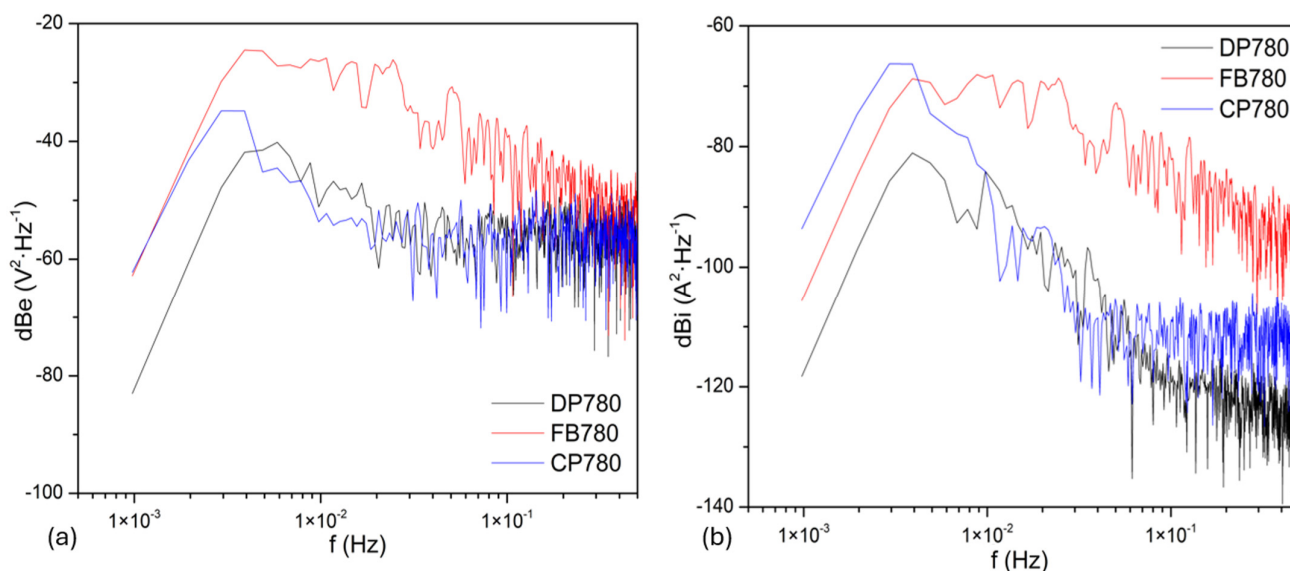


Figure 11. Power spectral density (PSD) in potential and current for AHSS in potential (a) and current (b) exposure to MgCl_2 solution.

Noise Impedance

The noise impedance, $Z_n(f)$, also referred to as spectral noise resistance, is defined in Equations (14) [79,80].

$$Z_n = \sqrt{\frac{\psi_V(f)}{\psi_I(f)}} \quad (14)$$

It is determined by dividing the PSD of potential by the square root of the PSD of current. The electrochemical noise impedance at zero frequency Z_{n0} is associated with the material's corrosion resistance, see Table 7 [81–83].

Figure 12 shows the noise impedance of AHSS's alloys exposed to NaCl. Table 5 shows the results of Z_n corresponding to this Figure. FB780 presented a higher impedance noise resistance, with $845 \, \Omega \cdot \text{cm}^2$, indicating that it is more resistant to corrosion. DP780 presented a resistance of $253 \, \Omega \cdot \text{cm}^2$, representing a 70% reduction in corrosion resistance. In contrast, CP780 exhibited lower corrosion resistance, with $88 \, \Omega \cdot \text{cm}^2$, corresponding to

89% less corrosion resistance compared to the standard. Those results correspond to those obtained by statistical analysis.

Table 7. PSD and Z_n parameters.

Alloy	ψ^0	Z_n0
NaCl		
DP780	−108	253
FB780	−116	845
CP780	−85	88
CaCl ₂		
DP780	−105	48
FB780	−111	158
CP780	−102	117
MgCl ₂		
DP780	−118	225
FB780	−105	716
CP780	−93	124

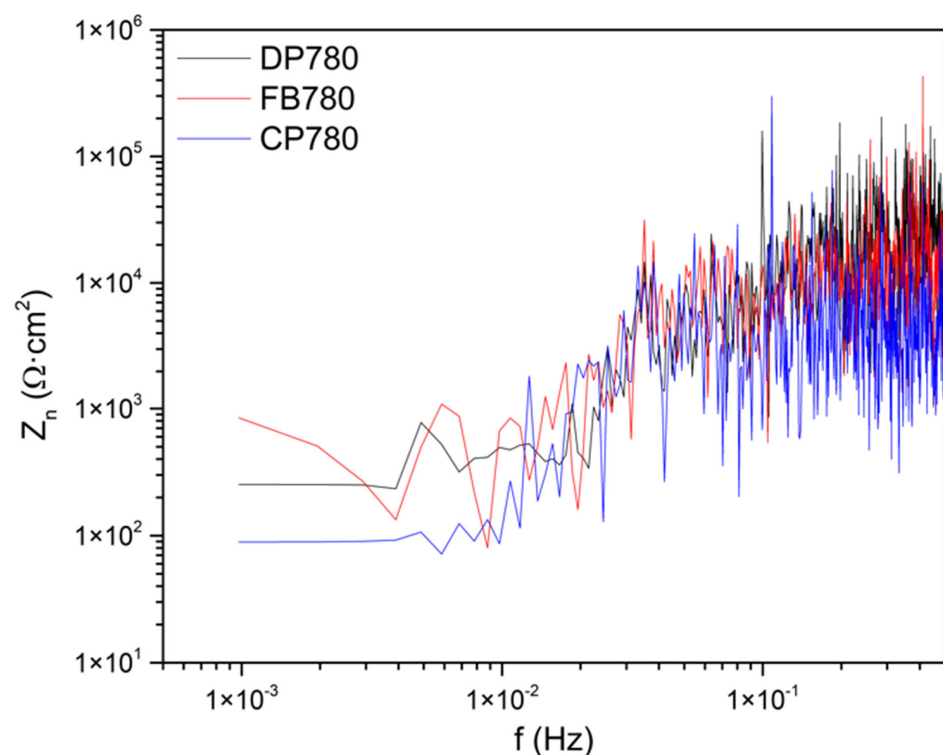


Figure 12. Noise impedance (Z_n) for AHSS exposed to NaCl electrolyte.

Figure 13 shows the noise impedance for AHSSs exposed to CaCl₂. These results show that DP780 exhibited lower corrosion resistance, with a value of 48 $\Omega \cdot \text{cm}^2$, whereas CP780 exhibited a value of 117 $\Omega \cdot \text{cm}^2$. The higher noise impedance value, 158 $\Omega \cdot \text{cm}^2$, for FB780 indicates high corrosion resistance. It is worth noting that the three alloys exhibited similar behavior in this medium.

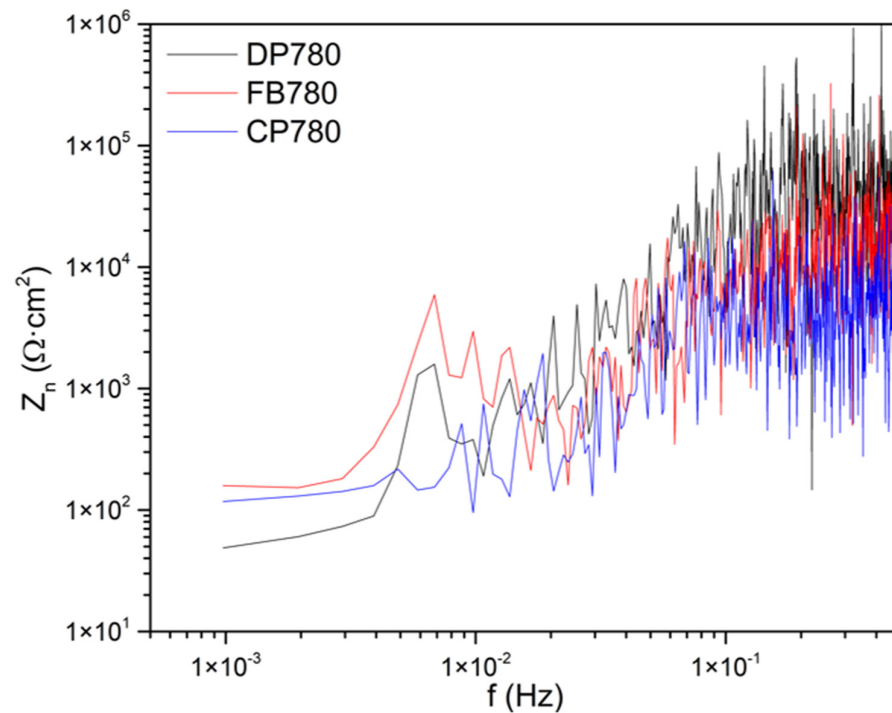


Figure 13. Noise impedance (Z_n) for AHSS exposed to CaCl_2 electrolyte.

Figure 14 shows the noise impedance of AHSS exposed to MgCl_2 . This media shows that CP780 presented a noise impedance of $124 \Omega \cdot \text{cm}^2$, DP780 obtained a value of $225 \Omega \cdot \text{cm}^2$, and FB780 obtained $716 \Omega \cdot \text{cm}^2$. That means that CP780 presents the lower corrosion resistance, while FB780 exhibits the highest corrosion resistance. However, FB780 exhibited a regular behavior across all frequencies, indicating that its corrosion behavior is consistent.

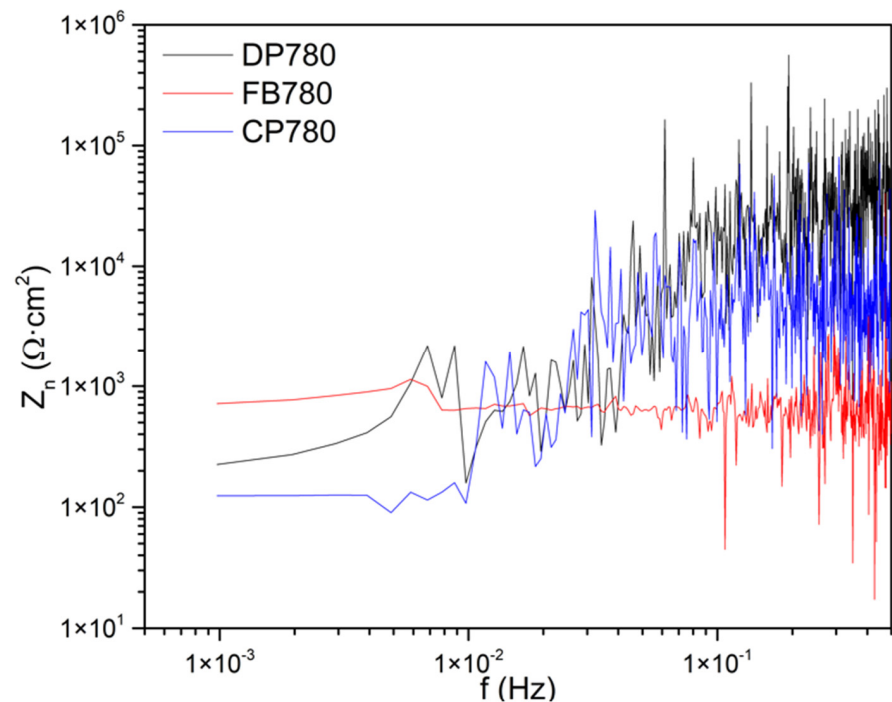


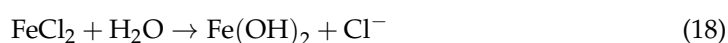
Figure 14. Noise impedance (Z_n) for AHSS exposed to MgCl_2 electrolyte.

4. Discussion

Two basic reactions dominate the corrosion process of AHSS.



These reactions lead to active oxidation; when oxygen diffuses onto the surface, the resulting corrosion products are porous and fail to provide passivation. This condition facilitates the penetration of Cl^{-} ions into the ferrite phase, resulting in localized corrosion and preventing surface passivation, which ultimately causes pitting within the ferrite region [84–87]. The chemical reactions involved are as follows:



The corrosion behavior of this alloy is strongly influenced by metallurgical heterogeneities within the metal matrix and the absence of a stable passive film [58,59]. Variations in phases, grain boundaries, impurities, and microstructural non-uniformity create anodic and cathodic regions that promote localized attack. In particular, phase differences contribute to galvanic corrosion [88]. The cathodic role of bainite and martensite, due to oxygen reception, made ferrite act as an anode due to its low oxygen content, making it susceptible to OH^{-} and Cl^{-} attacks [89].

For DP780 and FB780 steels, the previously described corrosion mechanism can be further characterized by uniform material dissolution when exposed to a chloride solution. This occurs as ferrite, martensite, and bainite interact with the solution, forming galvanic couples and undergoing auto-corrosion [90]. The preferential dissolution of ferrite generates active sites. At the same time, the uniformly distributed pits become interconnected, resulting in overall uniform material degradation, as illustrated in Figure 15. One of the important results obtained by CPP and EN showed that localized corrosion is the process that drives domain corrosion systems. The CPP revealed localized corrosion; several researchers have demonstrated how it is possible to detect localized corrosion using cyclic potentiodynamic polarization [91–93]. Additionally, standards such as ASTM G61 and F2129 recommend employing this technique to study localized corrosion in iron, nickel, or cobalt-based alloys [94–97].

EN results from statistical analysis, such as skewness and LI, showed results of uniform and mixed corrosion. Those results are associated with two processes that occur on the surface. The EN transients and process show how localized corrosion can occur; however, as pitting corrosion begins, after the pitting starts to diffuse on the surface, it can be considered a uniform pitting corrosion system. It is worth noting that CPP hysteresis is closely aligned with the active zone. If the interpreter lacks the sequence of data, it can be attributed to the presence of both corrosion processes. Sung Park and Jin Kim [98] mentioned that the potential difference between the ferritic matrix generates the anodic dissolution of ferrite. Salas Reyes et al. [95] noted that the anodic reaction is associated with oxygen reduction, a common phenomenon in chloride solutions.

Figure 15 illustrates the corrosion mechanism for FB780, DP780, and CP780, as suggested by the EN technique. The corrosion mechanism is almost the same, with the difference that in FB780, the phase that presents the corrosion is bainite, with ferrite serving as the cathode. For DP780 and CP780, the anode is the ferrite, and the martensite is the cathode. However, the corrosion is due to a galvanic attack by the phase difference. This behavior is consistent across the three solutions [91].

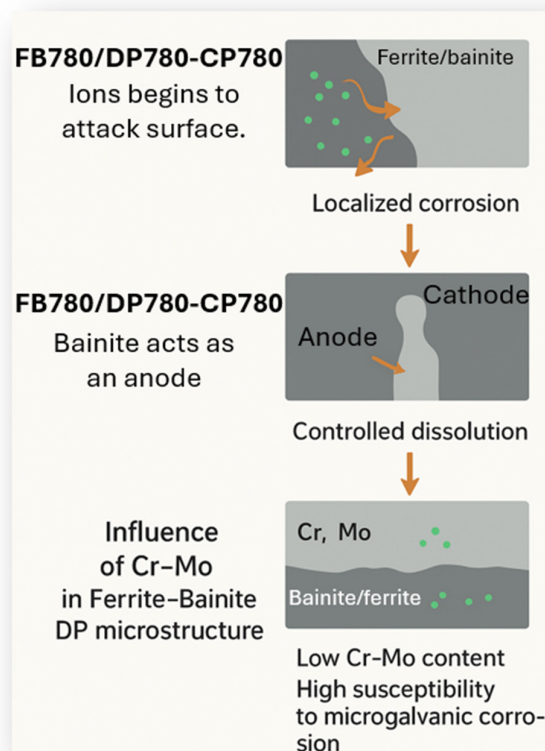


Figure 15. Schematic representation of corrosion for three alloys. FB presents bainite, whereas DP and CP present ferrite as the anode.

The corrosion behavior of FB780 steel with a ferrite–bainite microstructure in CaCl_2 and MgCl_2 solutions is characterized by localized attack, where the bainite phase dissolves more rapidly than the ferrite due to its anodic role. Iron carbides (represented as green dots) act as cathodic sites, as illustrated in Figure 15. The degradation of DP780 and CP780 steel with a ferrite–martensite microstructure is different: martensite, functioning as a cathode, serves as a corrosion barrier, while the continuous ferrite matrix limits the propagation of corrosion.

For ferrite–bainite dual-phase microstructures in Cr–Mo alloyed steels, the initiation of corrosion is strongly influenced by compositional variations. Regions with low Cr–Mo content contain more crystal defects, which reduces the electrochemical potential and increases susceptibility to microgalvanic corrosion. Conversely, at higher Cr–Mo concentrations, ferrite behaves as the cathodic phase, while bainite preferentially dissolves [92,93]. Mo helps produce MoO_2 and MoO_3 deposition, as well as some molybdate (MoO_4^{2-}), which repairs corrosion pits and acts as a barrier against attacks.

It is worth highlighting the aggressive role of Cl^- ions. Several studies have shown that when alloys are exposed to chloride-containing environments, Cl^- preferentially attacks the most vulnerable regions, leading to localized corrosion and intensifying the overall damage. In FB steel, the predominance of the ferritic matrix accelerates corrosion kinetics, since the larger anodic area reduces the material’s corrosion resistance.

Additionally, CaCl_2 was the most aggressive medium, presenting the lowest ZnO values; however, the alloy that showed the most susceptibility to corrosion was CP780. That behavior can be associated with a lower presence of Mn and a low presence of Ti. On the other hand, the high corrosion resistance of FB780 can be attributed to the presence of Mo in the alloy, which reduces the corrosion kinetics.

It is normal for some results obtained by CPP and EN to present differences. The techniques are different; CPP focuses on uniform corrosion processes and is a perturbative technique that induces an accelerated corrosion process. On the other hand, the EN technique is a non-perturbative method that facilitates the analysis of localized and pitting corrosion processes. In this research, the corrosion behavior of AHSS is dominated by localized corrosion. The EN technique helped determine the correct corrosion mechanism, as shown in Figure 15. The results from Tables 2 and 4 do not match in the i_{corr} , and R_p does not present a trend in the results of Z_n . However, as localized processes dominate the corrosion process, the correct technique to analyze the corrosion kinetics of AHSS is the EN.

5. Conclusions

- AHSS steels in their DP780, CP780, and FB780 grades presented galvanic pairs accompanied by a surface autocorrosion process. This process is generated by the diffusion of ions on the surface, resulting in uniform corrosion that degrades specific areas such as ferrite, inclusions, or some defects in the microstructure. With localized corrosion, chloride attack is encouraged, causing a uniform dissolution of the ferritic phase and resulting in pitting that is uniformly distributed and dissolves the material on the surface. The presence of inclusions influences the beginning of localized attacks.
- In CPP, the anodic breach reaction is associated with oxygen reduction, controlled by mass transfer.
- Both techniques demonstrated that localized corrosion is the predominant system in the AHSS.
- Results indicated that CPP revealed localized corrosion as the type of corrosion present in the system. This is due to the two phases that exist in the system.
- The results obtained by CPP determined that the E_{corr} of CP 780 is higher (noble values), indicating a higher energy required to begin the corrosion process.
- The difference between the results of CPP and EN is due to the mechanism of corrosion. CPP helps determine the corrosion kinetics when a corrosion uniform process dominates; however, as corrosion of AHSS is dominated by a nucleation pitting corrosion process, CPP does not apply to this analysis due to the Tafel law, which applies to uniform corrosion processes.
- Z_n showed that FB780 presented higher corrosion resistance when characterized by this method. That behavior is observable in all media and can be attributed to the presence of Mo in the alloy. On the other hand, CP780 exhibited lower resistance in various solutions, including R_n and Z_n .
- Samples presented mixed corrosion, with a predominance of uniform corrosion, which occurs when a pitting process is diffused. In this case, the diffusion provoked the distribution of pitting in the susceptible phases.

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