

Electrochemical Behavior of Corrosion on Borided and Non-borided Steel Immersed in NaCl 3.5% and HCl Solutions

Sarra Laid, Mohamed-Elamine Djeghlal, and Hichem Salhi

Abstract– This paper assesses the ability of borided and non-borided Z80 WCrV 18-04-01 steel to resist corrosion by employing linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS) methods. The steel samples were subject to boriding using the powder boriding method for different durations (1 hour, 2 hours, and 8 hours) at a temperature of 1223 K. Structural analysis of the borided steel surfaces revealed the presence of a double layer of FeB / Fe₂B on the Z80 WCrV 18-04-01 steel. The properties of linear polarization resistance and electrochemical impedance spectroscopy were evaluated on the surfaces of both borided and non-borided steels amply by immersing them in corrosive solutions of 1M HCl (pH = 0.13) and 3.5% NaCl saline. The measurements of electrochemical impedance spectroscopy were analyzed over a 7-day exposure period in both corrosive solutions. Equivalent electrical circuits were used to model the impedance curves acquired for both the borided and non-borided steel samples. The findings from both electrochemical methods suggest that the boride layers formed on the surfaces of the steel provide effective protection against corrosive effects in both environments, but this protection is observed only during the first day of immersion.

Keywords– Steel, corrosion mechanism, corrosion resistance, EIS technique.

I. INTRODUCTION

Boriding involves the thermal diffusion of boron into the surface layer of the part to be treated. The relatively small size and high mobility of boron atoms enable their diffusion into the substrate material, resulting in the formation of a metal-boride layer on the surface. This layer can be either single-phase or double-phase, depending on the conditions of the boriding process. Unlike many other surface treatments, boron can diffuse into most alloys and metals to produce hard boride layers. Boriding ferrous materials leads to the formation of a well-defined composition layer, which can be either a single phase (Fe₂B) or a double phase (FeB/Fe₂B). This formation occurs as a result of the boriding process. The determination of the case depth, which refers to the measurement of the thickness of each layer, holds significant importance in understanding the mechanical and chemical properties of borided steels. The thickness of the boride layer is influenced by various factors, including the temperature and duration of the boriding process, the chemical composition of the steel, and the concentration of boron in the vicinity of the sample surface [1, 2, 3].

Corrosion resistance is considered one of the properties significantly enhanced by boriding. In recent years, numerous studies have been conducted to evaluate the corrosion

resistance of borided steels using alternative methods beyond conventional continuous and cyclic immersion corrosion tests [4, 5].

Materials with Surface coatings are frequently evaluated for their corrosion resistance using electrochemical techniques. In their study, Tavakoli and Mousavi Khoie [6] employed electrochemical techniques to evaluate the corrosion resistance of various borided steels types in a 3% NaCl solution. Impedance spectroscopy, an electrochemical test, was conducted for exposure periods of 1 and 4 hours, as well as 2 to 4 days, and the results clearly demonstrated that the presence of boride layers significantly substantially enhanced the corrosion resistance within the tested range of boriding conditions. Similarly, in the study conducted by Jiang et al. [7], the corrosion behavior of low-carbon borided steels immersed in a 3.5% NaCl solution was evaluated using both the polarization method and electrochemical impedance spectroscopy (EIS) technique. However, the exposure duration of borided steels to the acidic solution was not specified in this particular study). The outcomes of the electrochemical tests indicated significant improvement in the corrosion resistance of low-carbon borided steel due to the presence of Fe₂B on its surface, in comparison to the untreated steel. Similar findings were observed in the boriding of AISI 316L stainless steel using the powder-pack method [5]. Electrochemical experiments were carried out to evaluate the corrosion behavior of the material under various conditions. Tafel extrapolation and linear polarization methods were employed in 1 mol/dm³ HCl, 1 mol/dm³ NaOH, and 0.9% NaCl solutions. The material was exposed to each solution for 1 and 168 hours at a temperature of 310 K. The existence of FeB/Fe₂B layers on the surface of borided stainless steel resulted in a reduction of the estimated corrosion rate when exposed to the HCl solution. Conversely, the corrosion resistance of the borided stainless steel showed a decrease when exposed to the NaOH and NaCl solutions. A noticeable

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trend of increasing corrosion resistance in the borided samples was observed with longer exposure periods in all tested solutions. The polarization resistance technique is used to measure the corrosion resistance of materials by applying a small potential perturbation, E , to the working metal electrode. This perturbation is typically around ± 10 mV from the open circuit potential value, and it is applied at a fixed potential scan rate. The current response obtained from the experiments is documented, and the linear polarization resistance (LPR) is defined as the ratio between the applied potential and the corresponding current response. It is important to note that LPR is inversely correlated with the corrosion current [8]. Electrical impedance spectroscopy (EIS) is widely utilized and highly effective technique in the field of corrosion studies. Typically, the Analysis of impedance measurements involves the utilization of equivalent electrical circuits consisting of passive elements such as resistors, capacitors, inductors and constant phase elements. These circuits are used to extract valuable information about the electrochemical properties of the system, especially those associated with corrosion and its underlying mechanisms.

II. EXPERIMENTAL WORK

Material and boriding process

The powder-pack boriding process was conducted on steel Z80WCrV 18-04-01. Chemical composition of this steel is: 0.81 wt.% C, 0.17 wt.% Mn, 0.23 wt.% Si, 0.019 wt.% S, 0.018 wt.% P, 0.08 wt.% Ni, 4.25 wt.% Cr, 0.09 wt.% Mo, 1.08 wt.% V, 17.60 wt.% W and 0.05 wt.% Co. Prior to boriding, The specimens underwent a series of preparation steps to ensure their cleanliness and quality. Firstly, they were carefully polished to achieve a smooth surface. Subsequently, they were cleaned using an alcoholic solution and deionized water for a duration of 15 minutes, employing ultrasound to enhance the cleaning process. After cleaning, the samples were thoroughly dried and stored under clean room conditions to maintain their integrity. The specimens were enclosed within a sealed cylindrical container made of AISI 304 steel. This container contained a mixture consisting of 5% B₄C, 5% NaBF₄, and 90% SiC. The entire assembly was then subjected to a temperature of 1223 K for durations of 1 hour, 2 hours, and 8 hours. Upon completion of the heat treatment, the container was carefully extracted from the furnace and allowed to cool gradually until it reached room temperature. The borided specimens were cut into cross-sections and then embedded in Bakelite for metallographic preparation. The preparation process involved grinding the samples using SiCs and paper with grit sizes ranging from 240 to 4000. The depths of the boride layers were examined using a Scanning Electron Microscope (SEM) under clear field observation. The microstructure of the boride layers developed on the surface of Z80 WCrV 18-04-01 steel was verified using X-ray diffraction (XRD) with a standard 2θ - ω scan procedure. X-ray diffraction analysis was conducted using an XPERT PRO analytical instrument equipped with Cu K α radiation at a wavelength of 0.154 nm was employed for this analysis.

III. RESULTS AND DISCUSSION

Optical microscopy (OM)

Micro-structural observations of transversal sections of borided samples of Z80 steel were performed by using an optical microscope (TESCAN SEM instrument).

It should be noted that the observations were carried out on transversal sections previously polished with an abrasive paper with granularity increasing from 240 to 4000 and a finishing with a diamond solution of 9 and 3 μ m. The obtained results are illustrated in figure 1.

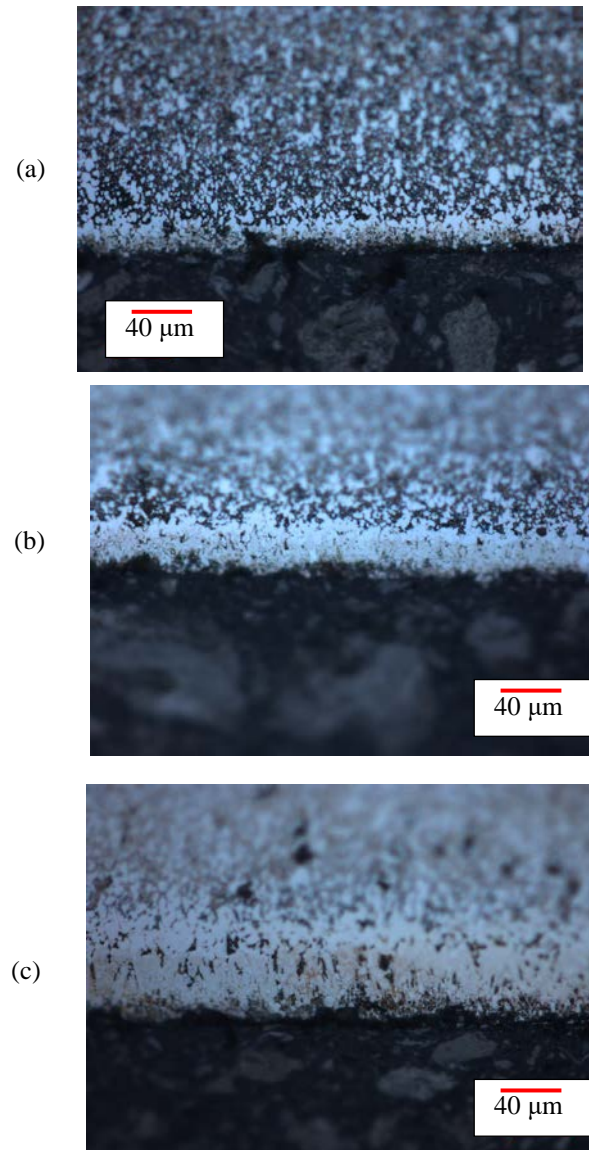


Fig.1: Optical micrographs carried out on transversal sections of borided samples at: (a) 1 hour, (b) 2 hours, (c) 8 hours.

X-Rays Diffraction (XRD)

The X-ray diffraction (XRD) technique was employed to analyze the microstructure of the boron layers formed on the surface of Z80 alloyed steel. The analysis included both non-borided samples and samples subjected to boriding for 1 hour, 2 hours, and 8 hours. The XRD analysis was conducted using an XPERTPRO analysis instrument with CuK α radiation, which has a wavelength of $\lambda = 0.154$ nm. The scanning procedure followed the regular 2θ - ω scanning pattern. The angular step size (2θ) during XRD digitization was 0.0260, and the temporal step size was 245.9154 seconds.

The X-ray diffraction (XRD) analysis carried out on the surface of the borided steel Z80 (as shown in Figure 3) confirmed the presence of the FeB-Fe2B layer.

Electrochemical study

a) Corrosion in 3.5% NaCl solution

The effect of the addition of boron atoms on the corrosion behavior of borided Z80 steel was evaluated by performing electrochemical tests in 3.5% NaCl solution. No mechanical load was applied in this case. The measurement chain is used in potio-dynamic mode, that is mean the current is measured following a scanning around the thermodynamic equilibrium potential. The scan rate was 0.5 mV / s. The impedance diagrams presented in this work were obtained by using "Volta-lab.PG.Z 402" impedance-measurement equipment. Data acquisition and treatment is done through Voltmaster and EClab software.

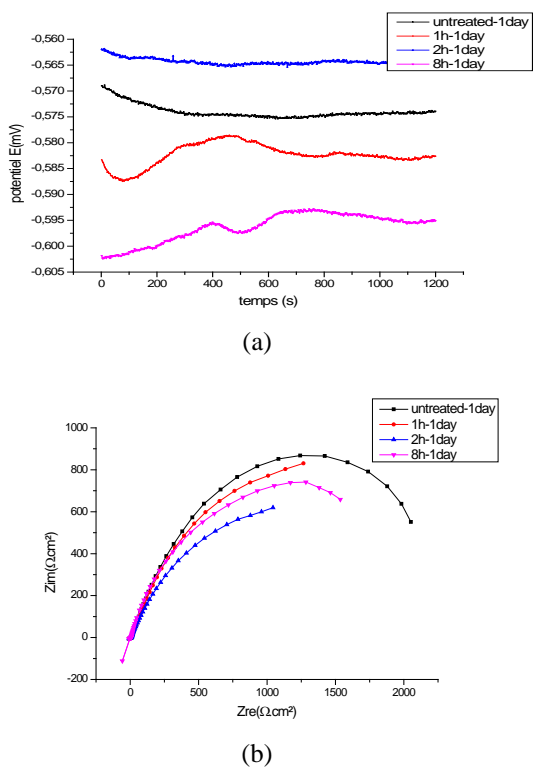


Fig.5: Evolution of the open-circuit potential, EOC (a), and electrochemical impedance spectroscopy (Nyquist format) (b) of Z80 steel according to the immersion time in a 3.5% NaCl solution of non-borided and borided steel at 1, 2 and 8 hours.

The Nyquist plots obtained for borided and non-borided Z80 steel are shown in Fig.5, the results are summarized in the Table 1 and Fig.9. The corrosion resistance of Z80 steel has reached a maximum value of 2590 Ω.cm² during the 08 hours of exposure to treatment based on linear polarization resistance testing (LPR). According to electrochemical impedance spectroscopy (EIS) the maximum value is 2580 Ω.cm² for a treatment that lasted 8 hours, which confirms the tests of the linear polarization resistance.

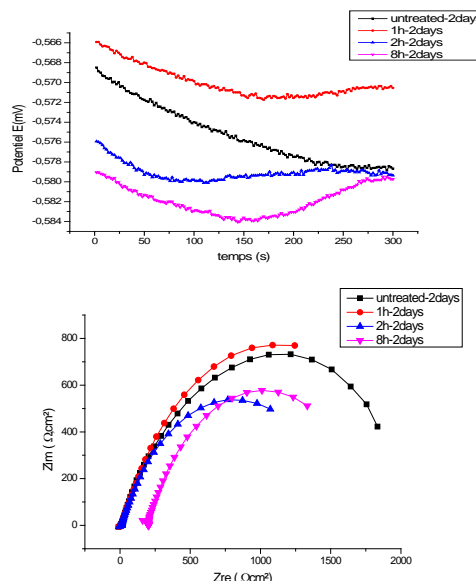


Fig.6: Evolution of the open-circuit potential, EOC (a), and electrochemical impedance spectroscopy (Nyquist format) (b) of Z80 steel according to the immersion time in a 3.5% NaCl solution of non-borided and borided steel at 1h, 2h and 8h after 2 days of immersion in NaCl

The Nyquist plots obtained for the borided and non-borided Z80 steel, which remained in the NaCl solution for 2 days, are shown in Fig 6, the results are summarized in the Table 2 and Fig 10. The corrosion resistance of Z80 steel reached a maximum value of 1370 Ω.cm² for untreated steel according to linear polarization resistance tests and according to electrochemical impedance spectroscopy the maximum value is 1310 Ω.cm², which confirms the linear polarization resistance tests. So, it is concluded that the untreated steel has a better resistance against corrosion, and this is due to the degradation of the surface layer (FeB / Fe₂B) which leaves a rough surface (the dendritic form of the double FeB layer / Fe₂B) which causes corrosion.

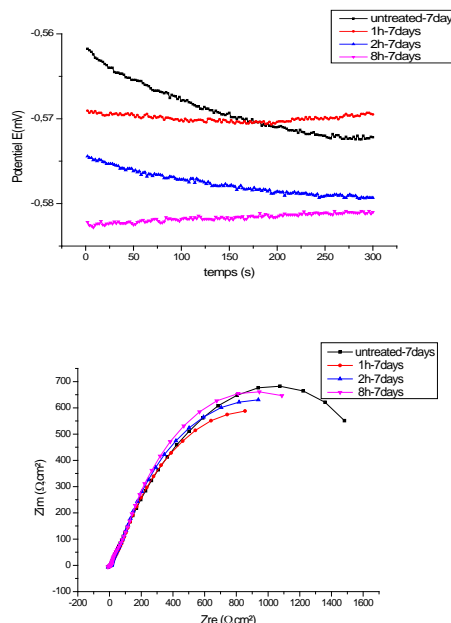


Fig.7: Evolution of the open-circuit potential, EOC (a), and electrochemical impedance spectroscopy (Nyquist format) (b) of Z80 steel according to the immersion time in a 3.5% NaCl solution of non borided steel and borided at 1h, 2h and 8h after 7 days of immersion in NaCl.

The Nyquist plots obtained for borided and non borided Z80 steel are shown in Fig.7, the results are summarized in the Table 3 and Fig 11. The corrosion resistance of Z80 steel reached a maximum value of 1240 Ω.cm² for untreated steel based on linear polarization resistance tests. According to electrochemical impedance spectroscopy the maximum value is 1200 Ω.cm² also for untreated steel, which confirms the linear polarization resistance tests.

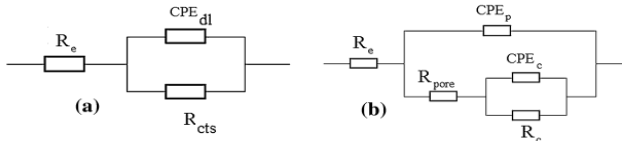


Fig.8: EECs used for numerical fitting of impedance data: (a) Z80 non-borided, and (b) Z80 borided steel

Table 1: Results of electrochemical impedance spectroscopy (EIS) in 3.5% NaCl solution

Non-borided steel	R_e [$\Omega.cm^2$]	R_{cts} [$\Omega.cm^2$]	CPE_{dl} [$\mu F/cm^2$]	N_{cts}			
Z80 WCrV18-04-01	6.707	1220	121.4	0.7873			
Borided steel Z80 WCrV18-04-01	R_e [$\Omega.cm^2$]	R_{pore} [$\Omega.cm^2$]	CPE_p [$\mu F/cm^2$]	N_{pore}	R_c [$\Omega.cm^2$]	CPE_c [$\mu F/cm^2$]	N_c
1 hour	5.92	1580	365	0.6465	9.776	567	0.7542
2 hours	5.44	1520	681	0.5251	40.7488	385.4	0.7875
8 hours	1.422	2580	445.2	0.7317	329.1	587	0.6023

Linear polarization resistance (LPR) gave the following results:

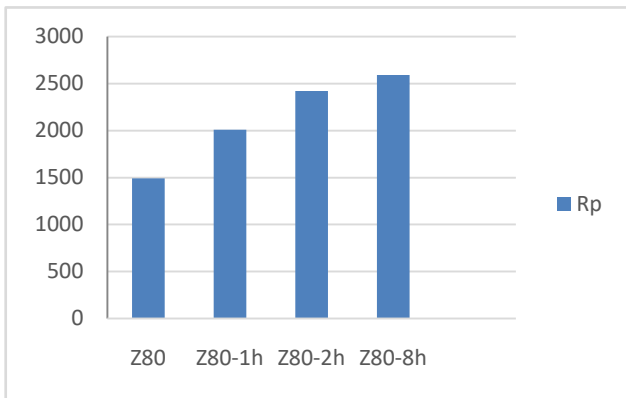


Fig.9: Evolution of the polarization resistance of Z80 steel immersed in a 3.5% NaCl solution of non-borided and borided steel at 1, 2 and 8 hours.

Table 2: Results of electrochemical impedance spectroscopy (EIS) after 2 days of immersion of the samples in the NaCl solution

Non-borided steel	R_e [$\Omega.cm^2$]	R_{cts} [$\Omega.cm^2$]	CPE_{dl} [$\mu F/cm^2$]	N_{cts}			
Z80 WCrV18-04-01	5.78	1310	26950	0.8163			
Borided steel Z80 WCrV18-04-01	R_e [$\Omega.cm^2$]	R_{pore} [$\Omega.cm^2$]	CPE_p [$\mu F/cm^2$]	N_{pore}	R_c [$\Omega.cm^2$]	CPE_c [$\mu F/cm^2$]	N_c
1 hour	6.63	1030	232	0.9389	443.9	873.4	0.7427
2 hours	6.30	1150	30.65	1	10.59	385.4	0.6914
8 hours	126.08	1100	902.2	0.7504	12.09	0.5086	0.7115

After 2 days of immersing the samples in the NaCl solution, the linear polarization resistance (LPR) gave the following results:

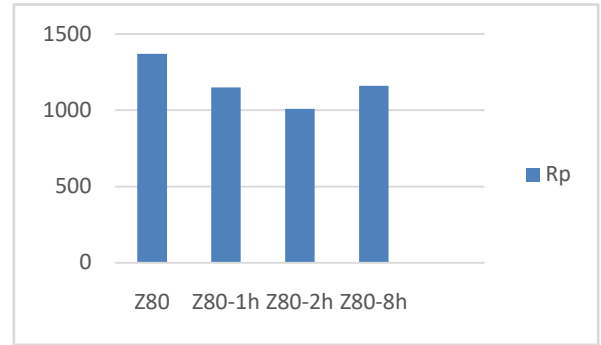


Fig.10: Evolution of the polarization resistance of Z80 steel immersed in a 3.5% NaCl solution of non borided and borided steel at 1h, 2h and 8h after 2 days of immersion in NaCl

Table 3: Results of electrochemical impedance spectroscopy (EIS) after 7 days of immersion of the samples in the NaCl solution

Non-borided steel	R_e [$\Omega.cm^2$]	R_{cts} [$\Omega.cm^2$]	CPE_{dl} [$\mu F/cm^2$]	N_{cts}			
Z80 WCrV18-04-01	7.22	1200	386.3	0.7282			
Borided steel Z80 WCrV18-04-01	R_e [$\Omega.cm^2$]	R_p [$\Omega.cm^2$]	CPE_{pore} [$\mu F/cm^2$]	N_{pore}	R_c [$\Omega.cm^2$]	CPE_c [$\mu F/cm^2$]	N_c
1 hour	7.4624	1010	653.5	0.8358	138.56	963.7	0.7085
2 hours	6.83	1010	587.5	0.8684	176.32	909.6	0.7331
8 hours	10.848	998	937.6	0.8255	108.73	0.4349	0.9074

After 7 days of immersing the samples in the NaCl solution, the linear polarization resistance (LPR) gave the following results

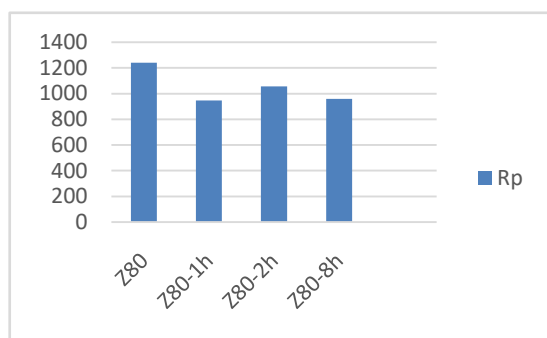


Fig.11: Evolution of the polarization resistance of Z80 steel immersed in a 3.5% NaCl solution of non-borided and borided steel at 1h, 2h and 8h after 7 days of immersion in NaCl

From Fig.7, we notice that the potential of Z80 steel decreases with the increase of the boriding treatment time, it means that steel becomes less noble, thus reducing their resistance to corrosion (Fig 11). This tendency can be attributed to the high porosity content and micro-cracks formed in the FeB and Fe2B layers as well as the rough appearance of the borided layer which is dependent on saw-tooth in the FeB-Fe2B inter-phase [8]. It should be noted that the aggressive Cl⁻ ions can diffuse into the cracks of the boron layers, reaching the base metal, resulting in film degradation, dissolution corrosion of the base steel and formation of ferrous ions [8]. The reaction of OH⁻ produced by the reduction of rust on the coating surface [9], accordingly to the electrochemical process:



Re [Ω.cm²): electrolyte resistance;

Rcts[Ω.cm²): resistance of the electron charge transfer of the substrate;

Rpore[Ω.cm²): resistance of the pore ;

Rc[Ω.cm²): resistance of the boride layer;

CPEp[μF/ cm⁻²): capacitance of the pore;

CPPEdl[μF/ cm⁻²): electrochemical double layer of the substrate;

CPEc[μF/cm²): capacitance of the boride layer.

b) Corrosion in HCl 1M solution

The corrosion assessment for both treated and non-treated Z80 steel immersed in a 1M HCl solution was conducted through two methods: monitoring the open circuit potential (EOCP) over time, and carrying out the EIS technique by using an electrochemical workstation of the “Volta-lab.PG.Z 402” brand.

The experiments were conducted at room temperature with the open circuit potential (EOCP) measured using a conventional three-electrode cell configuration. The cell consisted of a cylindrical platinum rod serving as the counter electrode, an Ag / AgCl mini electrode acting as the reference electrode, and the working electrode placed horizontally at the bottom of

the cell. The working electrode was affixed to an acrylic cylinder, exposing a surface area of 1 cm². Before initiating the electrochemical testing, the samples were submerged in the electrolyte solution for approximately 30 minutes to allow for the stabilization of the open circuit potential (EOCP) at room temperature. The electrochemical impedance spectroscopy (EIS) measurements were carried out over a period of 7-days while the samples were exposed to the acid solution. In this technique, a sinusoidal input signal $E_0 \sin(xt)$ was applied to the electrode system at a specific frequency, x . The measurements were conducted within a frequency range of 1 MHz to 10 MHz, using a sinusoidal alternating potential disturbance with amplitude of 10 mV (rms). To estimate the electrochemical parameters, the experimental data was fitted with a suitable Constant Phase Element CPE model.

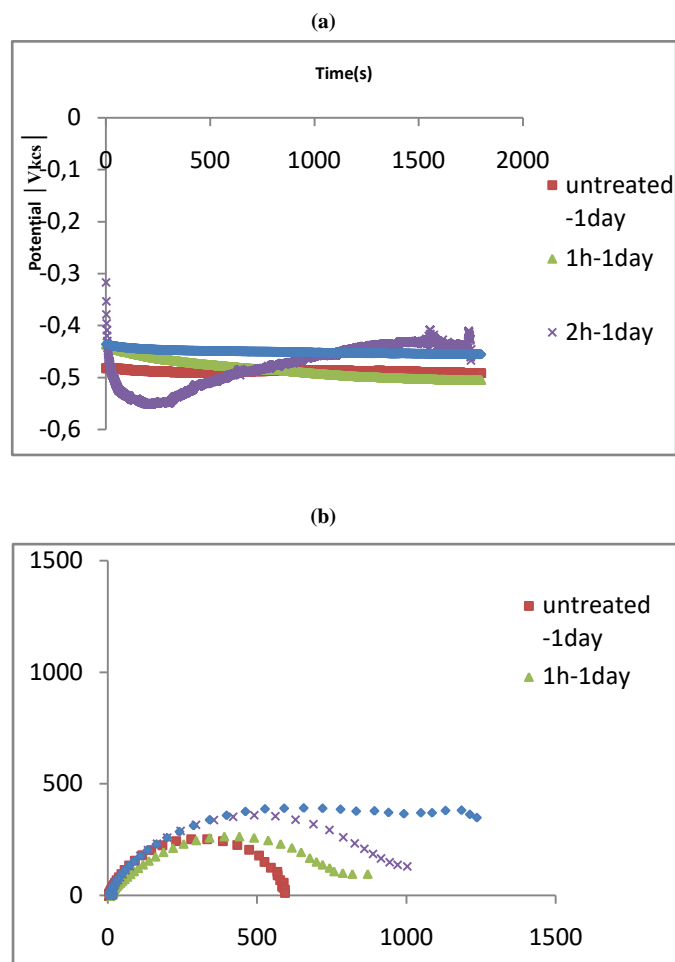


Fig.12: Evolution of the open-circuit potential, EOCP (a), and electrochemical impedance spectroscopy (Nyquist format) (b) of Z80 steel according to the time of immersion in a HCl 1M solution of non borided and borided steel at 1 hour, 2 hours and 8 hours.

Figure 12 displays the Nyquist plots obtained for both treated and non-treated Z80 steel, while table 4 and figure 14 summarize the results. The corrosion resistance of Z80 steel reached a maximum value of 1083 Ω.cm² during the 08 hours of exposure to treatment based on electrochemical impedance spectroscopy tests. The increase in corrosion potential values for Z80 borided steel proves that the layer of boron formed on the surface of the steel protects the steel against corrosion. By analyzing the data presented, the piece of steel immersed in a solution of hydrochloric acid for one hour was subjected to boriding at 950 °C, for boriding for 8 hours, it presents the lowest corrosion rate.

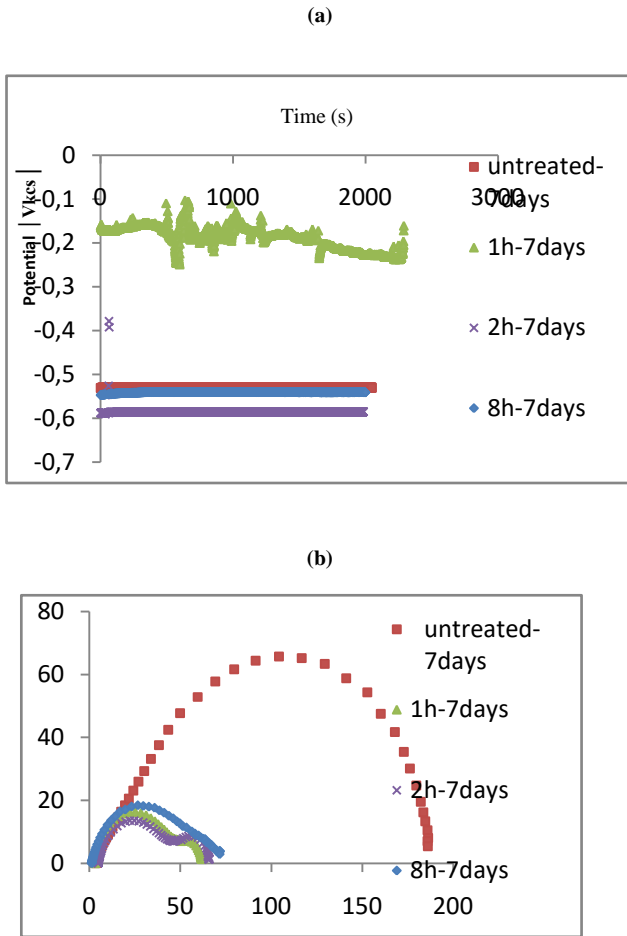


Fig.13: Evolution of the open-circuit potential, EOC (a) and electrochemical impedance spectroscopy (Nyquist format) (b) of Z80 steel according to the time of immersion in a HCl 1M solution of non borided and borided steel at 1 hour, 2 hours and 8 hours after 7 days of immersion in HCl.

Figure 13 displays the Nyquist plots obtained for both treated and non-treated Z80 steel, which remained in the 1M HCl solution for 7 days, the results are summarized in the Table 5 and Fig 15. Based on electrochemical impedance spectroscopy tests, the untreated Z80 steel exhibited a corrosion resistance with a maximum value of 142 Ω.cm². Therefore, it is concluded that the untreated steel has a better resistance against corrosion, and this is always due to the degradation of the surface layer (FeB/Fe₂B) which leaves a rough surface (the dendritic form of the double layer FeB/Fe₂B) which causes corrosion.

Table 4: Results of electrochemical impedance spectroscopy (EIS) in HCl 1M solution

	R _s [Ω.cm ²]	R _{dl} [Ω.cm ²]	Q _{dl} 10 ⁻³ [F.cm ²]	N
Z80	9,1565	594.94	0.0005	0.89281
Z80-1 hour	8.1834	790.97	0.00050472	0.74023
Z80-2 hours	5.6705	930.85	0.0005	0.84092
Z80-8 hours	5.3358	1083	0.00030028	0.79513

Linear polarization resistance (LPR) gave the following results

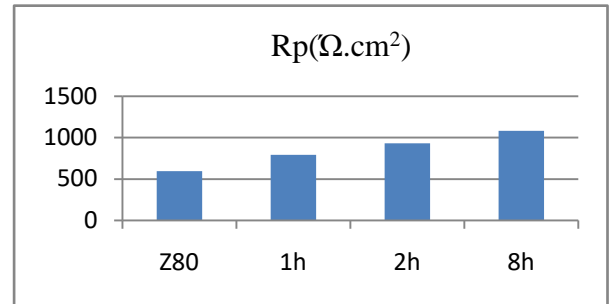


Fig.14: Evolution of the polarization resistance of Z80 steel immersed in a HCl 1M solution of non-borided and borided steel at 1 hour, 2 hours and 8 hours.

Table 5 : Results of electrochemical impedance spectroscopy (EIS) after 7 days of immersion of samples in HCl 1M solution

	R _s [Ω.cm ²]	R _{dl} [Ω.cm ²]	Q _{dl} 10 ⁻³ [F.cm ²]	n	P
Z80	35.747	142.85	0.0005	0.84384	0.0005
Z80-1 hour	3.1884	57.644	5.6924 E-05	0.61679	0.0005
Z80-2 hours	4.5478	40.734	/	0.76684	0.00021486

After 7 days of immersing the samples in the HCl solution, the linear polarization resistance (LPR) gave the following results:

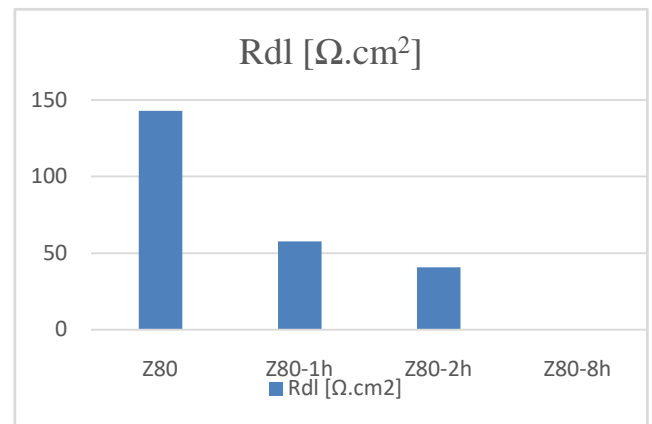
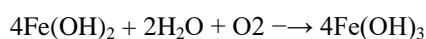
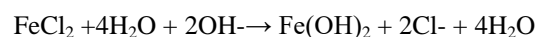
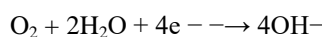
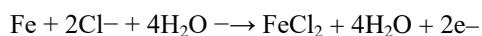


Fig.15: Evolution of the polarization resistance of Z80 steel immersed in a HCl 1M solution of non-borided and borided steel at 1h, 2h and 8h after 7 days of immersion in HCl.

The double boron layer (FeB/Fe₂B) has thermal expansion coefficients, which lead to the formation of compressive constraints leading to the creation of cracks. This type of defect allows chloride ions to penetrate through the formed boron layer thus migrating to the surface of the steel. Chloride ions diffuse through cracks in boron layers formed on the steel surface and form ferrous ions. These react with hydroxide ions to form iron hydroxides. The following equations summarize the recommended phenomenon:



R_s [$\Omega.cm^2$]: Resistance of the solution;
 R_{dl} [$\Omega.cm^2$]: Load transfer resistance;
 Q_{dl10-3} [$F.cm^{-2}$]: Constant phase element of the double layer / constant phase element of the deposit;
 n : The coefficient of frequency dispersion (It represents a measure of surface inhomogeneities).

c) Comparison between the behavior of Z80 steel in a HCl 1M solution and 3.5% NaCl

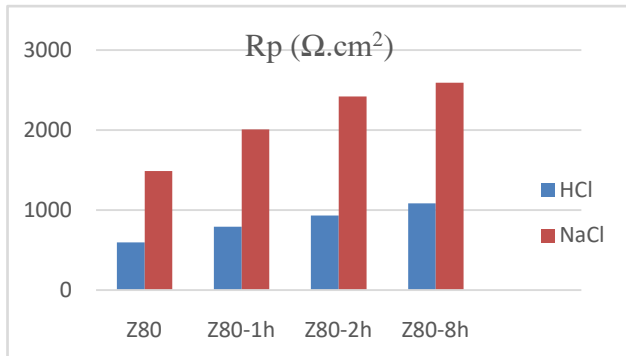


Fig.16: Evolution of the polarization resistance of Z80 steel immersed in a HCl 1M and 3.5% NaCl solution of non-borided and borided steel at 1 hour, 2 hours and 8 hours.

Note that the polarization resistance changes in the same way in both solutions, it increases with the increase of the surface treatment time. Accordingly to the analyzes of plots obtained from polarization resistance and electrochemical EIS tests, corrosion resistance increases with increasing boriding treatment time.

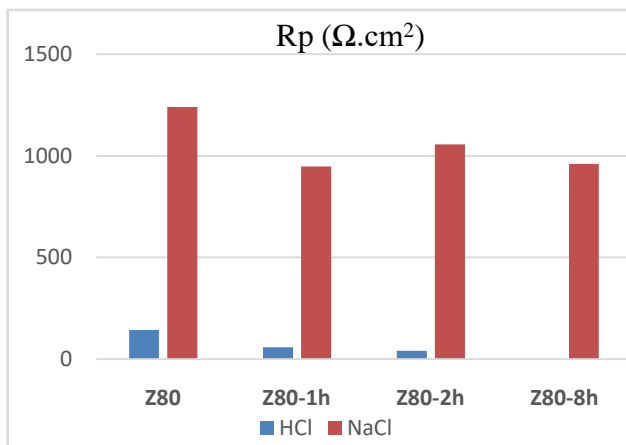


Fig.17: Evolution of the polarization resistance of Z80 steel immersed in a HCl 1M and 3.5% NaCl solution of non-borided and borided steel at 1 hour, 2 hours and 8 hours after 7 days of immersion in the solutions.

From Figure 16 and Figure 17, we can see that the polarization resistance changes in the same way in both NaCl and HCl solutions. Also, Z80 steel resists better in saline solution than in acidic solution. The formation of a bilayer (FeB/ Fe₂B) leads to the formation of cracks increasing the possibility for chloride ions to come into contact with the steel surface, accelerating the corrosion phenomenon. Corrosion resistance decreases with increasing sample immersion time in both 3.5% NaCl and HCl 1M solutions, which allows us to say that the surface layer of the samples deteriorates over time of exposure.

IV. CONCLUSION

Based on the results obtained, we can conclude with the following points:

- Aggressive ions, such as chloride ions (Cl⁻), have the ability to permeate through protective layers and initiate corrosion. These ions diffuse into the pores and eventually reach the steel substrate.
- Electrochemical Impedance Spectroscopy (EIS) is a highly effective and non-destructive technique used to investigate corrosion and various electrochemical phenomena occurring within metal samples.
- The corrosion rates of borided steel are typically influenced by the presence of micro-cracks and porosities within the coating. These porosities have a detrimental effect on the coating quality and significantly decrease its corrosion resistance.
- The presence of porosities within the study material can compromise the integrity of interfacial regions, creating vulnerable areas that can facilitate failure. These local defects can establish a direct pathway for corrosive environments to reach the boride layers.

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