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CHARACTERIZATION OF THE LOW ALLOY STEEL MODIFIED SUPERFICIALLY WITH IONS OF TITANIUM AND NITROGEN

CARACTERIZACIÓN DEL ACERO DE BAJA ALEACIÓN MODIFICADO SUPERFICIALMENTE CON IONES DE TITANIO Y NITRÓGENO

CARACTERIZAÇÃO DO AÇO DE BAIXA LIGA ALTERADO SUPERFICIALMENTE COM IONES DE TITÂNIO E NITROGÊNIO

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ABSTRACT

The three-Dimensional Ion Implantation technique (3DII) causes ions to collide with a solid surface in a perpendicular way regardless of the geometry of the solid (Khvesyuk & Tsygankov, 1997; Dugar-Jabon, Dulce-Moreno & Tsygankov, 2002). The steel AISI SAE 4140 has been used as substrate because of its wide use in the transport of oil industry, the specimens of steel underwent surface modification with Titanium ions (Ti) and the combined ions of titanium and nitrogen (Ti+N), with energy of 10keV for 5min and 10min. The superficially modified and unmodified specimens were characterized by Electrochemical Impedance Spectroscopy (EIS), noting among the most outstanding results the electrochemical double layer system capacitive characteristics under all tested conditions, the best corrosion performance was obtained for implanted substrates with Ti ions for 10min, having a charge transfer resistance much higher than those implanted with Ti+N and the non-implanted. Additionally it was determined that all the implanted systems can be simulated by using one equivalent circuit with a constant phase element instead of a capacitor and the non implanted substrate from day 15 can be simulated through another equivalent circuit.

Keywords: Chromium-molybdenum steels, Corrosion, Spectroscopy ionic implantation.

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RESUMEN

La técnica de implantación iónica tridimensional (3DII) consigue que los iones a implantar colisionen perpendicularmente con la superficie sin importar la geometría del sólido (Khvesyuk & Tsygankov, 1997; Dougar-Jabon *et al.*, 2002). Se ha usado un acero de herramientas AISI SAE 4140 como sustrato por su amplio uso en la industria del transporte de hidrocarburos. Las probetas de acero se sometieron a la modificación superficial con iones Titanio (Ti) y Titanio+Nitrógeno (Ti+N) a energía de 10keV durante 5min y 10min. Los sustratos modificados y no modificados superficialmente fueron caracterizados mediante la técnica de espectroscopia de impedancia electroquímica (EIS), observando entre los resultados más resaltantes que la doble capa electroquímica del sistema en estudio presenta características capacitivas en todas las condiciones evaluadas, donde el mejor desempeño frente a la corrosión lo presentan los sustratos implantados con iones de Ti durante 10min, debido a que poseen una resistencia a la transferencia de carga mucho mayor que los implantados con iones de Ti+N y los no implantados. Adicionalmente se determinó que todos los sistemas implantados pueden ser simulados mediante el uso de un circuito equivalente, el cual posee un elemento de fase constante en lugar de un capacitor y que el sustrato sin implantar a partir del día de inmersión 15 se puede simular a través de otro circuito equivalente diferente.

Palabras clave: Aceros cromo-molibdeno, Corrosión, Espectroscopia, Implantación iónica.

RESUMO

A técnica de implantação iônica tridimensional (3DII) consegue que os íons a serem implantados colidam perpendicularmente com a superfície sem importar a geometria do sólido (Khvesyuk & Tsygankov, 1997; Dougar-Jabon *et al.*, 2002). Foi usado um aço de ferramentas AISI SAE 4140 como substrato sendo que ele é extensivamente utilizado na indústria de transporte de hidrocarbonetos. Os espécimes de aço foram submetidos à alteração superficial com íons de Titânio (Ti) e Titânio + Nitrogênio (Ti+N) com energia de 10keV durante 5 min e 10 min. Os substratos alterados e não alterados superficialmente foram caracterizados mediante a técnica de espectroscopia de impedância eletroquímica (EIS), verificando-se entre os resultados mais importantes que a dupla capa eletroquímica do sistema em estudo apresenta características em todas as condições avaliadas, onde o melhor desempenho diante da corrosão é conseguido com os substratos implantados com íons de Ti durante 10 min, por conta deles possuírem uma resistência à transferência de carga muito maior do que aqueles implantados com íons de Ti+N e com os não implantados. Adicionalmente, determinou-se que todos os sistemas implantados podem ser simulados mediante o uso de um circuito equivalente, que possui um elemento de fase constante ao invés de um capacitor e que o substrato sem implantar a partir do dia de imersão 15 pode ser simulado através de outro circuito equivalente diferente.

Palavras-chave: Aços cromo-molibdênio, Corrosão, Espectroscopia, Implantação iônica.

1. INTRODUCTION

The low alloy tool steel AISI SAE 4140 contains chromium, molybdenum and manganese as main alloying elements, which confers high resistance to fatigue, abrasion, impact and torsion, in addition, it can achieve high hardness when processed under a suitable heat treatment. The chromium content provides a good resistance to pitting and molybdenum ensures high strength and uniform hardness. Common uses of tool steel AISI SAE 4140 mainly include industrial applications; small pinions, scissors, high strength screws, guides, cam followers, reducing axes, chisels, blocks of plates for filler brackets, punch holder for structural parts, clamps, gauges moulds, brake dice templates, and high strength parts (Peña *et al.*, 2009; V. Niño & Dougar-Jabon, 2006; Valbuena-Niño, Dulcé & Dugar-Zhabon, 2010).

In the energy sector, wear and corrosion are main causes of failure of industrial parts and tools. AISI SAE 4140 steel is not exempted from suffering these phenomena, although the alloy contains chromium, it is not enough to achieve properties of stainless steels. A common way or alternative to improve the resistance to corrosion and wear is to modify the surface by applying a coating (Peña *et al.*, 2009; V. Niño & Dougar-Jabon, 2006; Valbuena-Niño *et al.*, 2010).

Ion implantation is a process by which ions are accelerated to make them collide with a solid surface due to the kinetic energy acquired, these ions penetrate into the crystal lattice causing a series of mutually related processes that alter the physical and chemical behavior of the surface. The 3DII allows ions collide perpendicularly with the solid surface regardless geometry, preserving the monoenergetic characteristics of ions, which offers significant advantages over conventional methods of implantation (Khvesyuk & Tsygankov, 1997; Dougar-Jabon *et al.*, 2002).

3DII technique is performed at very low pressures (about 0.6Pa), which decreases the risk of contamination and the plasma generated in the high voltage discharge is self sustained (not need support plasma (Khvesyuk & Tsygankov, 1997; Dougar-Jabon *et al.*, 2002) that behaves as an ion source, which acquire energy of

kilo-electron-volts (*keV*) range (Dulcé-Moreno *et al.*, 2011; Parada-Becerra *et al.*, 2012; V. Niño, Peña, Reyes & Dugar-Zhabon, 2013; V. Niño, Salinas, Peña & Chinchilla, 2011). This research focuses on studying the electrochemical interaction surface-electrolyte of the steel AISI SAE 4140 implanted and non-implanted with Ti and Ti+N ions, using the Electrochemical Impedance Spectroscopy (EIS) technique.

2. METHODOLOGY AND MATERIALS

The substrates of steel AISI SAE 4140 with disk geometry were superficially implanted by hybrid discharges, high voltage and electric arc at low pressures with Ti and Ti+N ions for 5 and 10min to voltage of 10keV (Dugar-Zhabon, Dulcé-Moreno, Garnica-Villamizar & Valbuena-Niño, 2012; Tsygankov *et al.*, 2011; V. Niño *et al.*, 2012), taking into account the same procedure conducted in the previous research (Hernández *et al.*, 2014).

Electrochemical tests were performed with a potentiostat/galvanostat Gamry Interface 1000 and an electrochemical cell of three electrodes, comprising a bubbler, pH and temperature sensor, the working electrode (anode), against graphite electrode (cathode), a calomel reference electrode and 3.5%wt NaCl as electrolyte. Samples were immersed for 28 days and analyzed at day 0, 7, 15, 21 and 28, by EIS technique in 100kHz to 10mHz frequency range. Disturbance voltage was 10mV.

3. RESULTS AND DISCUSSION

The EIS is a non-destructive technique used to characterize the behavior of an electrochemical electrode-electrolyte interface, in this technique a harmonic voltage signal with amplitude on the order of millivolts and whose frequency *f* is applied generally within a range from several millihertz to some tens of kilohertz (Gil, Jiménez & Staia, 2006), the current flowing through the circuit is detected and recorded to analyze and interpret the complex impedance of the system (Gil *et al.*, 2006). Figure 1 show the Nyquist plot of the substrate in steel AISI SAE 4140 non-implanted and immersed from 0 to 28 days in brine solution.

Based on Figure 1, it becomes evident that with a

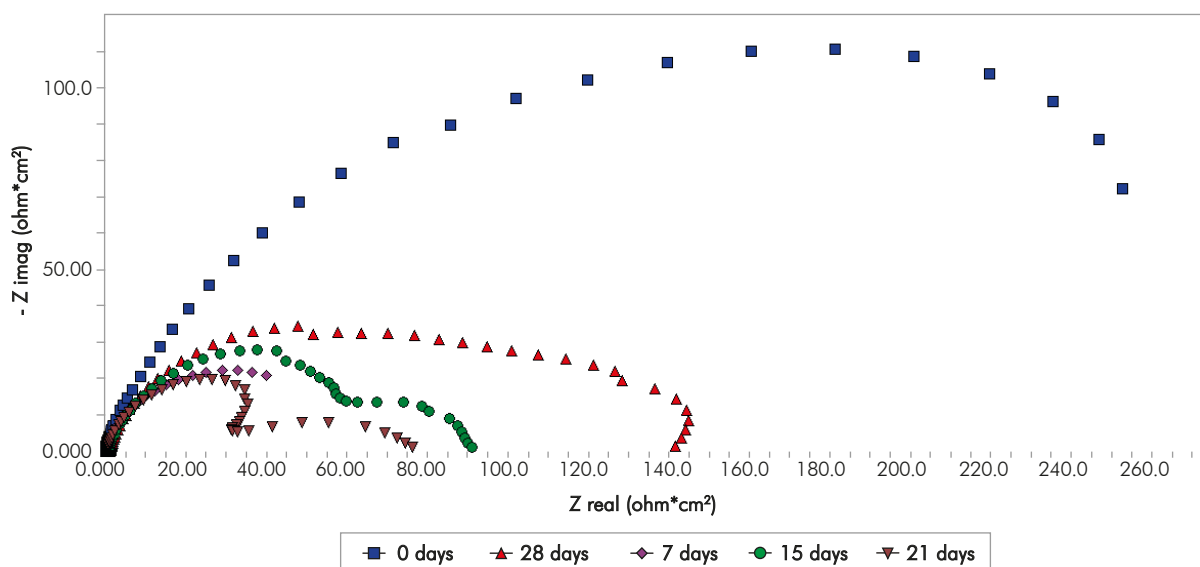


Figure 1. Nyquist plot for non-immersed substrates at different immersion times studied.

15-day immersion, the non-immersed samples behavior of the impedance at low frequencies corresponds to a pseudo-inductive effect, possibly caused by the reactions of adsorption/reabsorption of chemical species in the metal surface resulting in a change in the corrosion rate at that frequency (Escobar *et al.*, 2013). This phenomenon is not seen in the first seven days of immersion.

Next in Figure 2, the Nyquist plot corresponding to the specimens implanted with Ti+N to 10kV for 5min

is presented. The occurrence of one semicircle in the Nyquist plot indicates that the electrochemical reactions occur with only one time constant τ (Escobar *et al.*, 2013). Also a reduction of the semicircle diameter with respect to day 0 can be seen, from which a decrease in the charge transfer resistance R_{ct} can be verified after a prolonged immersion.

Figure 4 shows the Nyquist plots for samples implanted with Ti ions to 10kV for 5 and 10min. The implantation condition of 10min has the singularity that

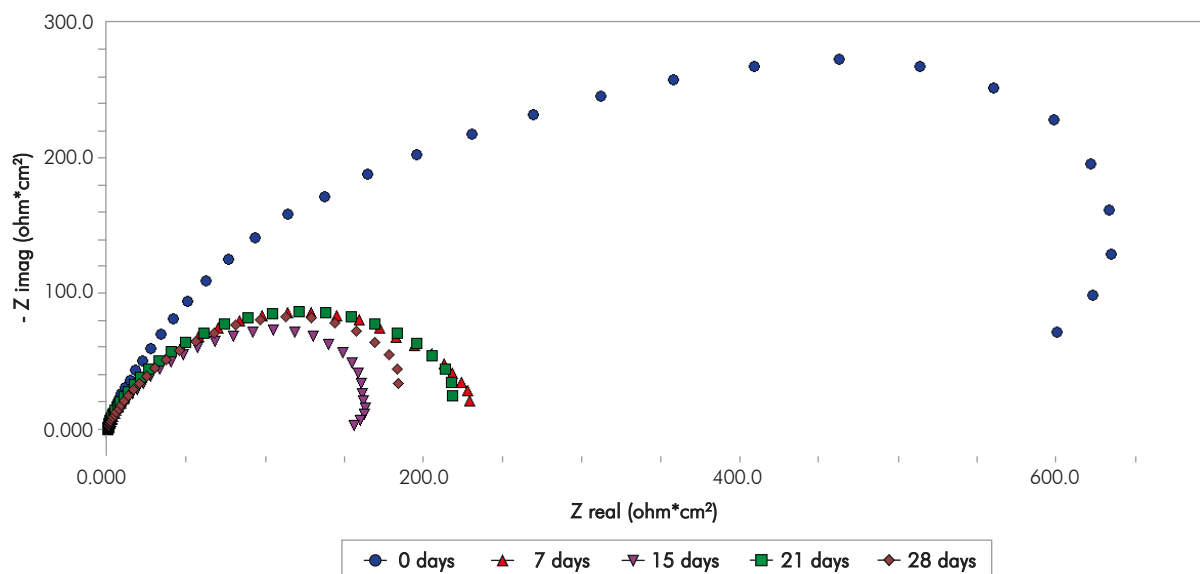


Figure 2. Nyquist plot for specimens implanted with Ti+N 10kV for 5min, at different immersion times studied.

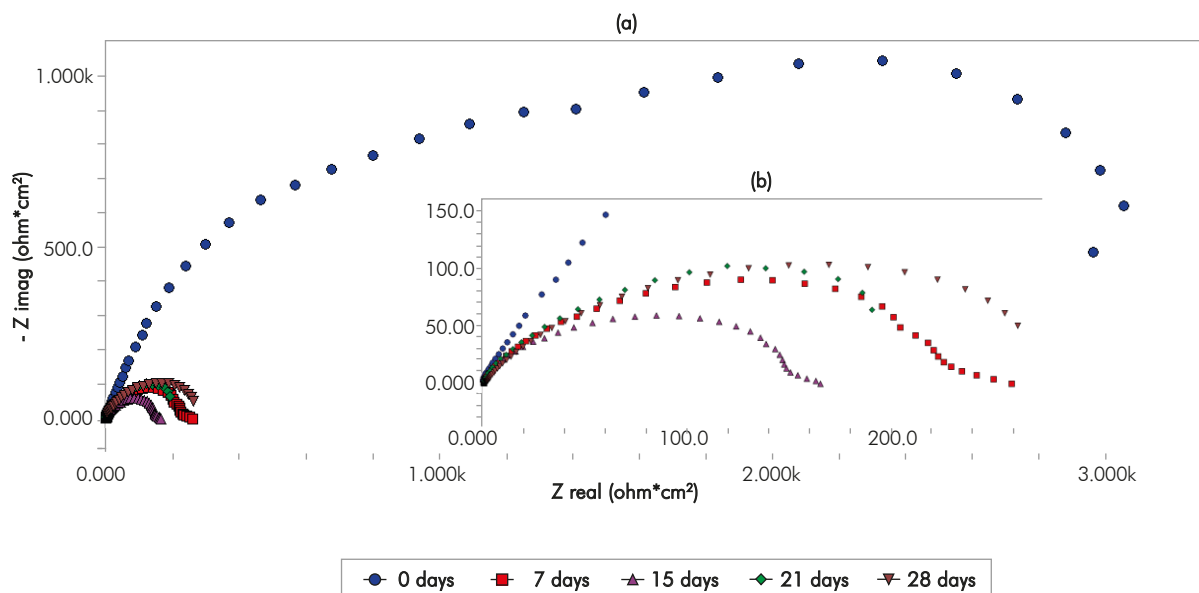


Figure 3. Nyquist plots of the samples implanted with Ti+N to 10kV for 10min (a) for all immersion times studied, (b) to observe Zoom detail Nyquist curves.

in the 28th immersion day the semicircle was enlarged and this allows us to observe an increased resistance to charge transfer at the end of the time evaluated in this study.

Before fitting the equivalent circuits, the data was validated using the Kramers-Kronig relations, always obtaining excellent χ^2 values $<10^{-3}$. The equivalent circuit simulation obtained for both implanted and non-implanted surfaces was performed with the Simplex method in the software V6.11 Gamry Echem Analyst, which simulates all the implanted systems behavior through one simple equivalent electric circuit and the non implanted substrate behavior from day 15 through another equivalent circuit; Figure 5 provides details on the two types of circuits that were used for the simulation. The simulation had $\chi^2 < 10^{-3}$ (goodness of the fit) in almost all the conditions studied.

In circuit (b) CPEads and Rads were used to simulate the pseudo-inductive behavior possibly caused by the reactions of adsorption/reabsorption of chemical species in the metal surface. Figure 6 shows some of the curve fitting for Kramers-Kronig validation and equivalent circuit simulation.

Tables 1 to 5 provide the parameters corresponding to the EIS test applied to steel AISI SAE 4140 modified

and not modified superficially with Ti and Ti+N ions. Note that the alpha parameter α indicates that the electrochemical double layer may have a resistive or capacitive behavior, therefore in almost all conditions studied, the electrochemical double layer formed on the interface between samples and the surrounding electrolyte, has a capacitive characteristic because the values of α were always higher than 0.5.

Figures 7 and 8 show that the capacitance of the electrochemical double layer (CPE) in all the samples increases with immersion time; one order of magnitude for the reference substrate (not modified superficially) and up to three orders of magnitude for implanted substrates, note that in the samples implanted with Ti ions for 10min a sudden drop in capacitance is recorded in the 28th day of trial, which reaches a decrease of 9 orders of magnitude.

Increased capacitance is possibly due to the penetration of electrolytes through the corrosion products that are formed in the surface of the specimens (Escobar *et al.*, 2013). Other authors describe the increase in capacitance as the result of an increased porosity of the layers of the corrosion products presented (Cottis & Turgoose, 1999), whereas the decreased capacitance of the Ti substrate implanted with ions during 10min on the 28th day of immersion may be related to changes

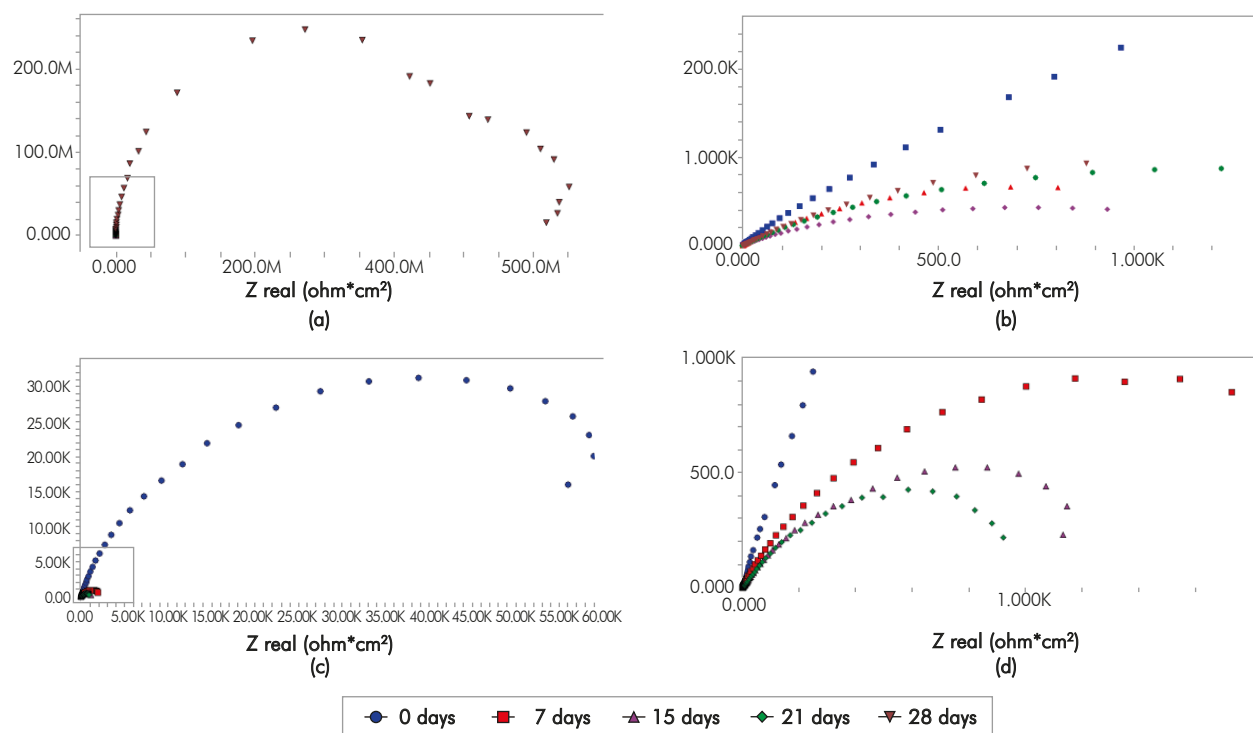


Figure 4. Nyquist plots for samples implanted with Ti 10kV (a) for 10min, (b) for 5min, (c) Zoom of insert (a), (d) Zoom of insert (c).

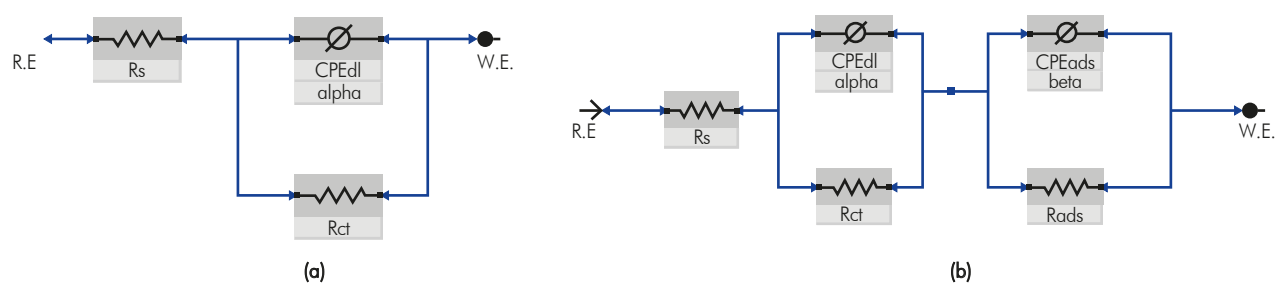


Figure 5. (a) Equivalent circuit used to simulate the implanted samples, and non implanted samples immersed 0 days and 7 days. (b) Circuit used to simulate the behavior of the non implanted substrate from 15 to 28 days of immersion..

Table 1. EIS corresponding values of the parameters for the reference substrate.

Parameter	Days									
	0	±error	7	±error	15	±error	21	±error	28	±error
R_{ct} (ohm*cm ²)	244.30	2.48	51.34	0.77	62.19	1.49	37.67	0.51	64.61	8.61
CPE (S*s ^α)/(cm ²)	5.88E-A	4E-06	7.13E-04	1E-05	1.02E-03	2E-05	8.09E-04	2E-05	1.05E-03	4E-05
α	9.04E-1	7E-06	8.83E-01	3E-03	8.25E-01	4E-03	8.69E-01	4E-03	8.11E-01	1E-02
Xi squared	3.10E-2		1.84E-03		3.42E-03		1.82E-02		2.15E-03	
KramersKronig Xi ²	1.51E-3		9.10E-04		2.07E-03		1.10E-02		1.47E-03	

Table 2. EIS corresponding values of the parameters for samples implanted with Ti+N for 5min

Parameter	Days									
	0	±error	7	±error	15	±error	21	±error	28	±error
R_{ct} (ohm*cm ²)	620.90	5.94	236.30	2.31	188.30	1.68	221.60	2.24	212.90	2.85
CPE (S*s ⁰)/(cm ²)	3.99E-04	5E-06	2.28E-03	3E-05	4.14E-03	4E-05	7.23E-03	6E-05	1.19E-02	1E-04
α	8.64E-01	5E-03	7.99E-01	2E-03	7.23E-01	2E-03	8.57E-01	2E-03	8.16E-01	2E-03
Xi squared	2.67E-02		6.03E-04		6.98E-03		3.44E-03		1.71E-03	
KramersKronig χ_i^2	6.21E-04		1.51E-04		7.12E-03		1.26E-03		1.35E-03	

Table 3. EIS corresponding values of the parameters for samples implanted with Ti+N for 10min.

Parameter	Days									
	0	±error	7	±error	15	±error	21	±error	28	±error
R_{ct} (ohm*cm ²)	2415.00	21.62	226.90	2036.00	144.50	1306.00	190.00	2432.00	316.60	4270.00
CPE (S*s ⁰)/(cm ²)	9.00E-05	9E-07	1.35E-03	2E-05	1.95E-03	2E-05	4.83E-03	5E-05	7.31E-03	6E-05
α	8.76E-01	1E-03	8.22E-01	2E-03	8.72E-01	2E-03	1.00E+00	2E-03	7.24E-01	2E-03
Xi squared	8.03E-02		5.68E-03		8.92E-03		5.73E-02		1.95E-03	
KramersKronig χ_i^2	9.71E-04		2.73E-04		3.30E-03		5.64E-02		9.42E-05	

Table 4. EIS corresponding values of the parameters for samples implanted with Ti for 5min.

Parameter	Days									
	0	±error	7	±error	15	±error	21	±error	28	±error
R_{ct} (ohm*cm ²)	325200.00	3289.00	2146.00	52.16	1611.00	36.92	3420.00	128.40	4567.00	277.80
CPE (S*s ⁰)/(cm ²)	5.98E-06	4E-08	1.86E-03	1E-05	3.57E-03	2E-05	3.72E-03	2E-05	4.19E-03	3E-05
α	8.11E-01	9E-04	7.21E-01	2E-03	6.70E-01	1E-03	7.09E-01	1E-03	7.26E-01	2E-03
Xi squared	4.24E-02		2.38E-02		1.18E-02		1.14E-02		1.92E-03	
KramersKronig χ_i^2	7.52E-04		2.96E-04		6.66E-05		4.45E-05		3.26E-05	

Table 5. EIS corresponding values of the parameters for samples implanted with Ti for 10min.

Parameter	Days									
	0	±error	7	±error	15	±error	21	±error	28	±error
R_{ct} (ohm*cm ²)	67940.00	734.10	1898.00	20.59	1828.00	39.46	1578.00	38.19	5.78E+08	4.7E+06
CPE (S*s ⁰)/(cm ²)	2.98E-05	2E-07	1.40E-03	1E-05	2.55E-03	2E-05	3.44E-03	3E-05	1.10E-11	2E-13
α	8.85E-01	9E-04	8.90E-01	1E-03	6.68E-01	2E-03	6.53E-01	2E-03	9.68E-01	1E-03
Xi squared	3.76E-02		5.24E-02		5.43E-03		8.37E-02		5.90E-03	
KramersKronig χ_i^2	2.86E-04		4.32E-04		2.17E-04		5.60E-05		4.30E-04	

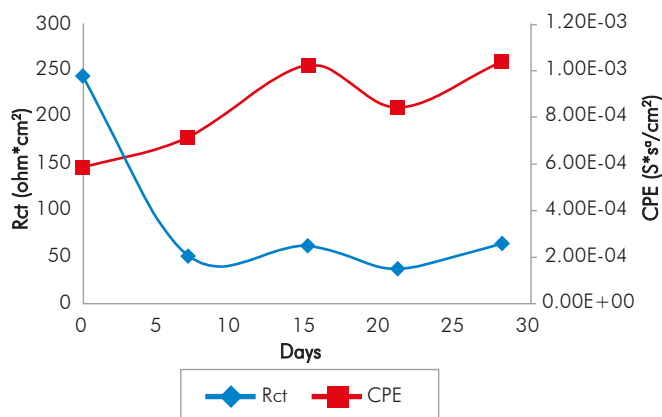


Figure 7. Resistance to charge transfer and capacitance versus immersion time for the reference substrate.

in morphology of corrosion products layers formed on the surface possibly becoming a homogeneous passive layer (Cottis & Turgoose, 1999). The resistance to charge transfer for the conditions studied, except for the implantation with Ti for 10min, has the characteristic of

starting in a maximum and then decrease and stabilize 1 order of magnitude below the initial value for the substrate, 2 orders of magnitude below the initial value to the Ti for 5min and Ti+N for 10min, and decrease 3 times under baseline conditions for the implantation of Ti+N for 5min.

The samples implanted with Ti ions for 10min have a different behavior, since the resistance to charge transference increases about 5 orders of magnitude between day 21 and day 28 and remains up to 4 orders of magnitude above the initial value. Increased resistance to charge transference indicates that the corrosion process is disadvantageous (Flores-Merino & Paucar-Cuba, 2003; Cabrera-Sierra, Marín-Cruz & González, 2007; Galván *et al.*, 2016; Chang, 2016) possibly due to the presence of a passive film that becomes more stable in time (Galván *et al.*, 2016; Vasilescu *et al.*, 2015).

The open circuit corrosion potential is one of the

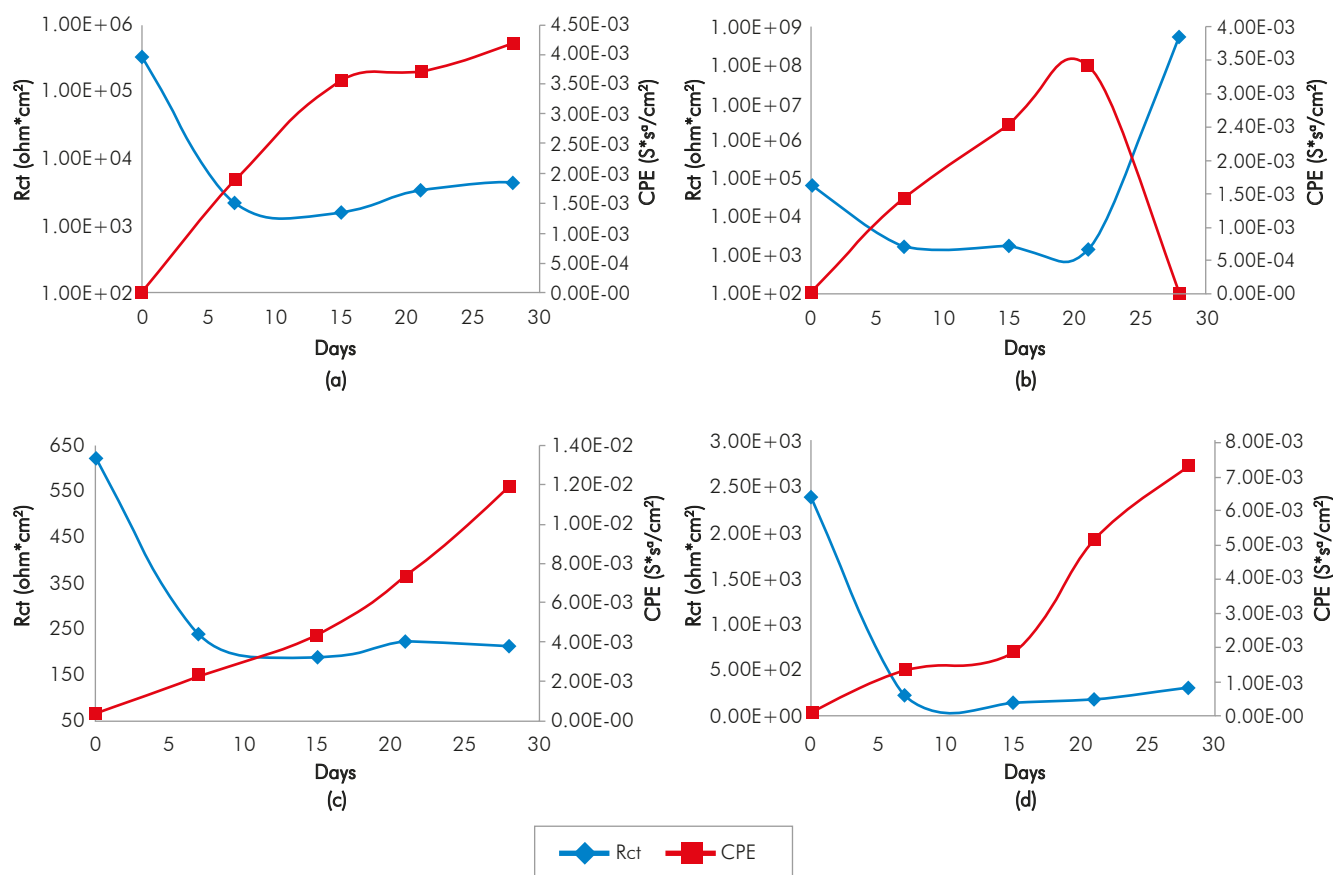
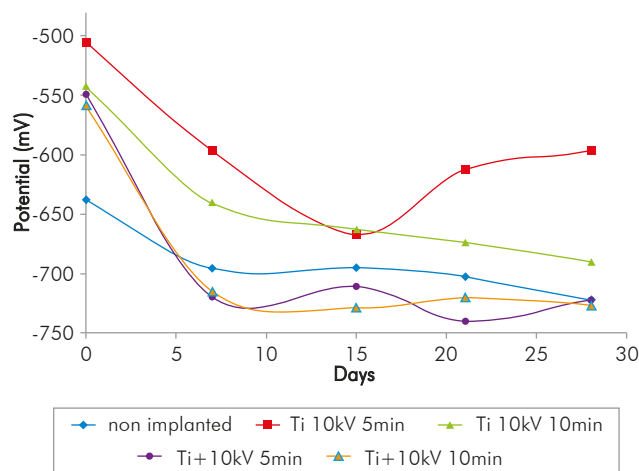


Figure 8. Resistance to charge transfer and capacitance versus immersion time for substrates implanted with (a) Ti+N for 5min, (b) Ti+N for 10min, (c) Ti for 5min, and (d) Ti for 10min.

tools to find out the tendency of an electrochemical system to corrode or not, where higher thermodynamic potential tendency for low corrosion may take place and vice versa. Figure 9 shows that at 0 days samples implanted in all conditions have corrosion potential between 100 and 140mV higher than the non-implanted substrate, indicating that implanted samples have a lower thermodynamic tendency to corrosion as compared to substrates without implantation. However after 7 days immersion, there are two subgroups with different trends, the group formed by the samples implanted with Ti ions for 5 and 10min, whose potential is above the corrosion potential of the non-implanted substrate, meaning it is less prone to corrode. The second subgroup comprises the specimens implanted with Ti+N ions for 5 and 10min, whose potential is below the corrosion potential of the substrate of reference, indicating that the specimens implanted with Ti+N ions have a higher thermodynamic tendency to corrosion as compared to the non-implanted substrate.



4. CONCLUSIONS

- The EIS spectra of the substrates implanted and non-implanted presented only one time constant τ , which allows us to conclude that the implanted ions do not form a film as in a coating, but instead they fully integrate with the substrate, the behavior of the implanted samples can be simulated by using one equivalent circuit, which has a constant phase element instead of a capacitor, and the behavior of the non implanted substrates from day 15 to 28 can be simulated with one equivalent circuit, which has two constant phase elements instead of two capacitors.

- The electrochemical double layer formed on the interface between samples, implanted and non-implanted, and the surrounding electrolyte, showed a capacitive behavior, because the values of α obtained from the EIS test were between 0.5 and 1.
- According to the charge transference resistance, all surfaces implanted have best corrosion resistance when compared to the non-implanted surface. The decrease in the capacitance of samples implanted with Ti ions for 10min and 10kV on the 28th day, may be related to the changes in morphology due to corrosion products and the layers homogeneous passive characteristics of TiO_2 formed on the surface.
- According to the open circuit potential measurements the Ti ions implanted samples have less tendency to corrode. The R_{ct} of the specimens implanted with Ti ions showed a higher corrosion resistance than surfaces implanted with Ti+N and non-implanted. The best corrosion performance was obtained by specimens implanted with Ti ions for 10min and 10kV. Further studies will be conducted to analyze the morphology and composition of the surface and corrosion product layers before and after immersion, to confirm the layer passive characteristics.

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