

Enhancement of Corrosion Resistance of Hot-Dip Galvanized Carbon Steel by Low Amount of Ni Addition

Rafael Gonzalez-Parra, Arturo Barba-Pingarrón, Raúl Valdez- Navarro, Alba CoveloVillar, Miguel A. Hernández-Gallegos

Centro de Ingeniería de Superficies y Acabados (CENISA). Departamento de Ingeniería de Diseño y Manufactura. División de Ingeniería Mecánica e Industrial. Facultad de Ingeniería. UNAM. 2o Nivel. Edificio "T" (Bernardo Quintana). Circuito Exterior. Ciudad Universitaria. Coyoacan, 04510, Ciudad de México.

rafael.parra@yandex.com, arturo.barba@ingenieria.unam.edu, raulgvaldez@hotmail.com,
acovelov@gmail.com, mahg@yahoo.com

Javier Cervantes-Cabello

Centro de Ingeniería Avanzada. Departamento de Ingeniería de Diseño y Manufactura. División de Ingeniería Mecánica e Industrial. Edificio X. 2o Piso. Circuito Exterior, Ciudad Universitaria. Coyoacan, 04510, Ciudad de México. cercab2@yahoo.com.mx

Abstract

Hot Dip Galvanizing is one of the oldest and more used methods to protect steels substrates from corrosion due to the good corrosion resistance of the Zn coatings. This work studies the effect of the addition of small amounts of Ni to hot-dip galvanizing bath on the corrosion behaviour of hot-dipped Zn rich coating using open circuit potential (OCP), electrochemical noise (ENM) and electrochemical impedance spectroscopy (EIS). The results show that the Zn-Ni coating alloys exhibit nobler potentials and low current density indicating the increase of the corrosion resistance of the sample due to the protective effect provided by the oxides formed on Zn-Ni coating alloys. However, the corrosion products allow the mass transfer of aggressive species showing a semi-infinite behaviour. The protective effect shows a relationship with the Ni amount employed. The hot-dip method for deposition of ZnNi coatings using 0.5% Ni exhibit the best performance.

Keywords

Zn-Ni coatings, Corrosion resistance enhancement, Hot Dip Galvanizing, corrosion protective properties.

1. Introduction

One of the oldest ways to protect steel or iron from corrosion is by immersion in a molten zinc bath known as the hot-dip galvanizing process. The hot-dip galvanizing process is commercially relevant and widespread in the industry. The treatment consists of submerging steel on a Zn rich bath to obtain a coating over the surface (Abdel Hamid et al. 2010). To improve the corrosion resistance of Zn coatings the research on this field is open (Belleze et al. 2006).

The basis of this galvanizing process lies in obtaining a series of layers of intermetallic compounds, produced by the diffusion that takes place when the piece is submerged in the bath, mainly constituted of molten Zn, although it can count on small amounts of other elements. The corrosion protection that hot-dip galvanizing can provide is proportional to the thickness of the coating, therefore, the life span of galvanized steel depends on the thickness and the environmental conditions in which the coating is located. The objective of hot-dip galvanizing is to obtain a series of intermetallic layers, from the diffusion of the molten Zn on the surface of iron or steel.

Furthermore, Zn-Ni coating alloys have been reported to provide higher corrosion resistance, better properties of machining such as weldability and formability, and an increase in temperature resistance in comparison to pure Zn and other coatings formed by Zn alloys (Vourlias et al. 2001).

1.1 Objectives

The aim of this work was to determine the effect of a low Ni amount on corrosion behaviour of Zn-Ni alloys obtained by hot-dip galvanizing process, under influence of chloride ions employing electrochemical impedance spectroscopy and polarization resistance techniques. This allows us to determine if the addition of a low Ni amount contributes to the corrosion resistance of hot-dip galvanized samples.

2. Literature Review

The addition of nickel modifies the kinetics of growth of intermetallic phases, morphology and thermodynamic equilibria (Pistofidis et al. 1998) leading to a decrease of the Zn-Fe reactivity, while allow maintaining the minimum coating thickness (Belleze et al. 2006).

The works reported by Fratesi et al. (2002) and Shibli and Manu (2005) suggest that the addition of Ni causes a secondary reaction forming a ternary Zn-Fe-Ni intermetallic compound between the Zn- ζ interface, which limits the growth of phase ζ . It is known that this intermetallic could function as a mechanical barrier and diffusion of Zn to adjacent layers, thus limiting the growth of the so-called Irving-Williams Series (Irving and Williams 1948).

Moreover, the addition of Ni to the Zn coating provides higher corrosion resistance (Yu et al. 2020), better properties of machining such as weldability and formability, and an increase in temperature resistance in comparison to pure Zn and other coatings formed by Zn alloys (Vourlias et al. 2001). Recently, these coatings are developed through thermal spray, low-pressure cold spray (Bai et al. 2017) or electrodeposition (Choudhary et al. 2017) technologies. The advantages and disadvantages of these production methods are discussed elsewhere (Kwon et al. 2016). However, hot-dip galvanizing is the most employed process in the industry, but the amount of Ni is limited due to the low solubility showed on the Zn-Ni phase diagram. Moreover, the corrosion resistance developed depends practically on the Ni content.

To understand the corrosion behaviour of Zn-Ni coating alloy, some studies have been carried out. Guosheng et al. (2013) reported that the nickel amount present on the coating decreases the corrosion rate, but it could have a negative effect if the nickel content is too high. Bai et al. (2017) in the study of a composite Zn-Ni coating determined that the content of Ni for the best corrosion protection is 20% weight. Conde et al. (2011) in the study of Zn-Ni coatings produced by electrodeposition and LPCS points out that the protective characteristics shift with the phase structure and their morphology. Despite the efforts of several researchers, there is a lack of information about the behaviour of galvanized coating added with low Ni amount and obtained by the hot-dip galvanizing process. In the best knowledge of the authors, just a few works deals with this topic, Vourlias et al. (2004) have used the maximum composition of Ni employed in our work, and Abdel Hamid et al. (2016) reported a similar concept but diminishing the amount of Sn in the galvanizing bath. Thus, this study fulfils the lack of information about the effect of a low Ni addition on galvanized steel.

3. Methods

The coatings were generated using the hot-dip galvanizing process. The substrate employed was AISI-SAE 1015 steel sheets of $9 \times 10^{-4} \text{ m}^2$ that were immersed on Zn-Ni bath at 450 °C for 15 s. The zinc bath composition was modified to compare the influence of Ni amount on the corrosion resistance, the set of samples used are composed of Zn, Zn-0.1%Ni and Zn-0.5%Ni coating alloys. The chemical composition of the AISI-SAE 1015 is 0.15 C, 0.42 Mn, 0.030 P, 0.050 S and 0.040 Si. Also, the purity level of the Zn and Ni employed was 99%. The nickel amount was established due to the maximum solubility level to avoid the formation of intermetallic on the Zn-Ni phase diagram.

Before immersion in the zinc bath, the surface preparation was carried out. The substrate was mechanically cleaned, degreased at room temperature conditions with perchloroethylene and rinsed in distilled water. Later, the oxides were removed by a solution of 16 % of HCl in distilled water and rinsed again in distilled water. Finally, the surface was activated by the immersion of the samples on a solution at 70 °C of 1.76 M ZnCl_2 and 2.99 M NH_4Cl . Once the surface cleaning has finished the substrates were ready to be immersed in the galvanizing bath.

4. Data Collection

The data acquisition was performed using a set of surface and electrochemical techniques. Briefly, the morphology of the coating was observed by means of scanning electron microscopy (SEM) through a JEOL JSM 5900 with an acceleration voltage of 20 kV, the image was obtained with backscattered electrons. The composition of the coating

formed was determined through XRD using a Rigaku Ultima IV with a radiation Cu ($\lambda_{\alpha} = 1.596 \text{ \AA}$) and the sweep from 10° to 65° .

The electrochemical characterization has been carried out with a potentiostat/galvanostat Autolab model PGSTAT 205, the cell configuration was a classical three-electrode arrangement inside a Faraday cage. The samples were immersed in the electrolyte and employed as the working electrode. The potential was referred to versus a saturated calomel electrode (SCE) and a large-graphite sheet was employed as counter-electrode (CE). The samples were immersed in 0.1 M NaCl solution for 336 hours, the volume of solution employed was 100 mL at room temperature and the controlled area exposed was $1.39 \times 10^{-4} \text{ m}^2$.

For electrochemical impedance spectroscopy (EIS) the amplitude of the sinusoidal signal was 10 mV and a frequency sweep between 10^5 to 10^{-2} Hz was employed. Furthermore, the polarization resistance (R_{pol}) was obtained, applying a potential sweep of 20 mV since the corrosion potential.

5. Results and Discussion

5.1 Result

The morphology of the layers developed is shown in Figure 1 for (a) hot-dip galvanizing without Ni addition, (b) hot-dip galvanizing + 0.1%Ni and (c) hot-dip galvanizing + 0.5%Ni. The sample coated only with Zn, Figure 1 (a), exhibit a homogeneous coating layer characterized by the formation of four distinct layers (γ , δ , ζ , η) over the substrate. The addition of a low amount of Ni (0.1% Ni) to the hot-dip galvanizing bath, Figure 1 (b), modifies the layer structure developing a bi-layered structure coating with a homogeneous appearance. The increase of the Ni amount on the hot-dip bath until 0.5% Ni, Figure 2(c), modifies the morphology of the coating developing cracks at the inner layer of the coating. Also, the transition region exhibits a bigger range. The concentration of 0.5 % Ni added promote the formation of a three-layered coating; this behaviour is related to the accumulation of Ni atoms at an intermediate distance between the substrate and the surface. Similar results were observed by Vourlias et al. (2001) for galvanized samples which indicate that the addition of Ni leads to the formation of a stratified coating.

Figure 1 (d) shows the diffraction pattern of the samples coated with Zn, Zn - 0.1%Ni and Zn-0.5%Ni, alloy after 336 hours of immersion on NaCl 0.1 M solution. The characteristic peaks obtained indicated the presence of the products such as zinc hydroxide (Zn(OH)_2), simonkolleite ($\text{Zn}_5(\text{OH})_8\text{Cl}_2 \cdot \text{H}_2\text{O}$), metallic Zn and Ni. These types of oxides are reported to increase the corrosion resistance of Zn alloy coatings due to their low solubility that allowing them to precipitate over the surface (Barranco et al. 2004).

Thus, the surface characterization evidence that the Ni addition promotes the change of the coating morphology, decreasing the number of layers developed. However, the composition of the samples remains almost constant despite the amount of Ni added.

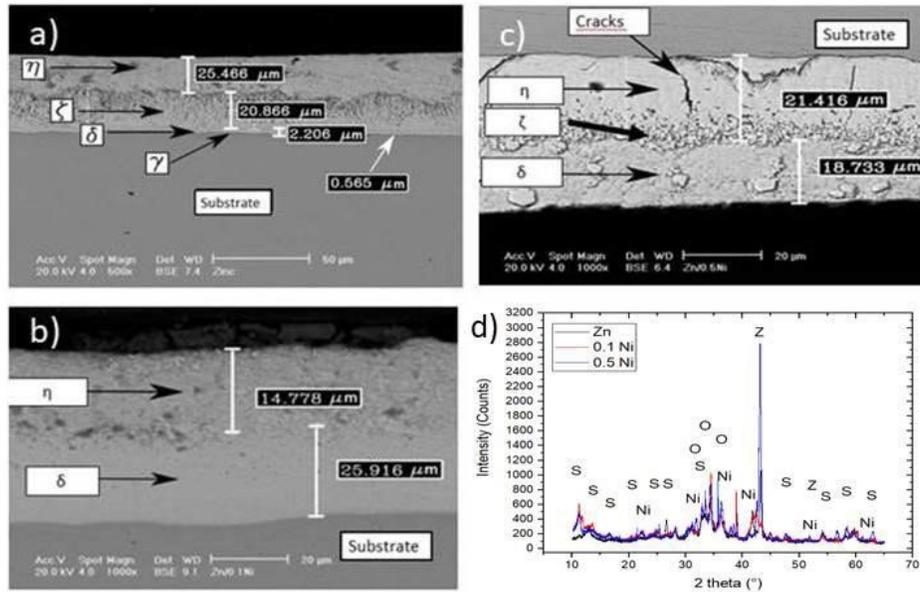


Figure.1 Cross-section of the hot-dip galvanizing coating, (a) pure Zn, (b) Zn-0.1%Ni, (c) Zn-0.5%Ni and (d) XRD pattern of galvanized samples. Where, S: Simonkolleite, O: Zinc Hydroxide, Z: Zinc, Ni: Nickel, C:

Figure 2 show the Nyquist diagrams obtained for the Zn - Ni alloy coating submerged during 336 hours in 0.1 M NaCl solution. In general terms, the diagrams exhibit the presence of at least two electrochemical processes evidenced by two resistive capacitive loops during the first immersion hours. However, as immersion time proceeds the system evolves showing the presence of a diffusive behaviour.

Figure 2 (a) show the Nyquist diagrams, the first semicircle displayed in the plot is attributed to the resistance of the coating and the capacitive effect of the protective layer, while the second semicircle is associated with the charge transfer resistance coupled to the double layer capacitance (Guosheng et al. 2013). After 24 hours, Figure 2 (b) show the presence of two semicircles, already discussed. Although, the samples galvanized with Zn and Zn - 0.1%Ni displays a notable decrease of the amplitude that can be associated with the diminishment of the protective properties of the galvanized layer. The diminish of protective properties may be related to the porosity of Zn corrosion products that does not inhibit the metallic dissolution of Zn to Zn²⁺ or the access of aggressive species such O₂ or Cl⁻ (Trabelsi et al. 2004). Figure 2 (c) shows the response of the samples at 48 hours of immersion. The amplitude of these semicircles considerably decreased in comparison with the behaviour exhibited at the beginning of the immersion; this is associated with the loss of the protective performance on the sample. It is worth to mention, that the Zn galvanized coupon show the presence of one semicircle in the high-frequency region, while at lowfrequency an ill-defined tail is observed. Barranco et al. (2004) in the study of a Zn and Zn-Al coating exposed to chloride media suggests that the first semicircle is related to the charge transfer resistance and the double-layer capacitance, while the ill-defined tail can be related with a diffusion process associated with the mass transfer of oxygen, zinc and aggressive ions (Wang et al. 2014). After 168 hours of immersion, Figure 2 (d), all the samples exhibits the change of their behaviour displaying the presence of two semicircles and a tail in the low-frequency region. This trend prevails until 336 immersion hours, Figure 2 (e), where the two processes and the diffusive behaviour are well determined. Moreover, the tail evidenced in the low-frequency region is related to the presence of a semi-infinite diffusion process.

Therefore, results acquired indicate that the galvanized coatings exposed to the NaCl environment provide a protective effect that decreases as immersion time proceeds. Also, the formation of a Zn corrosion products layer and mass transport processes are evidenced. The mass transport mechanism is carried out in a complex interface formed by the electrolyte-corrosion products-substrate (Barranco et al. 2004). It is worth mentioning that enhance of the impedance behaviour of the oxide layer developed is related to the amount of Ni added.

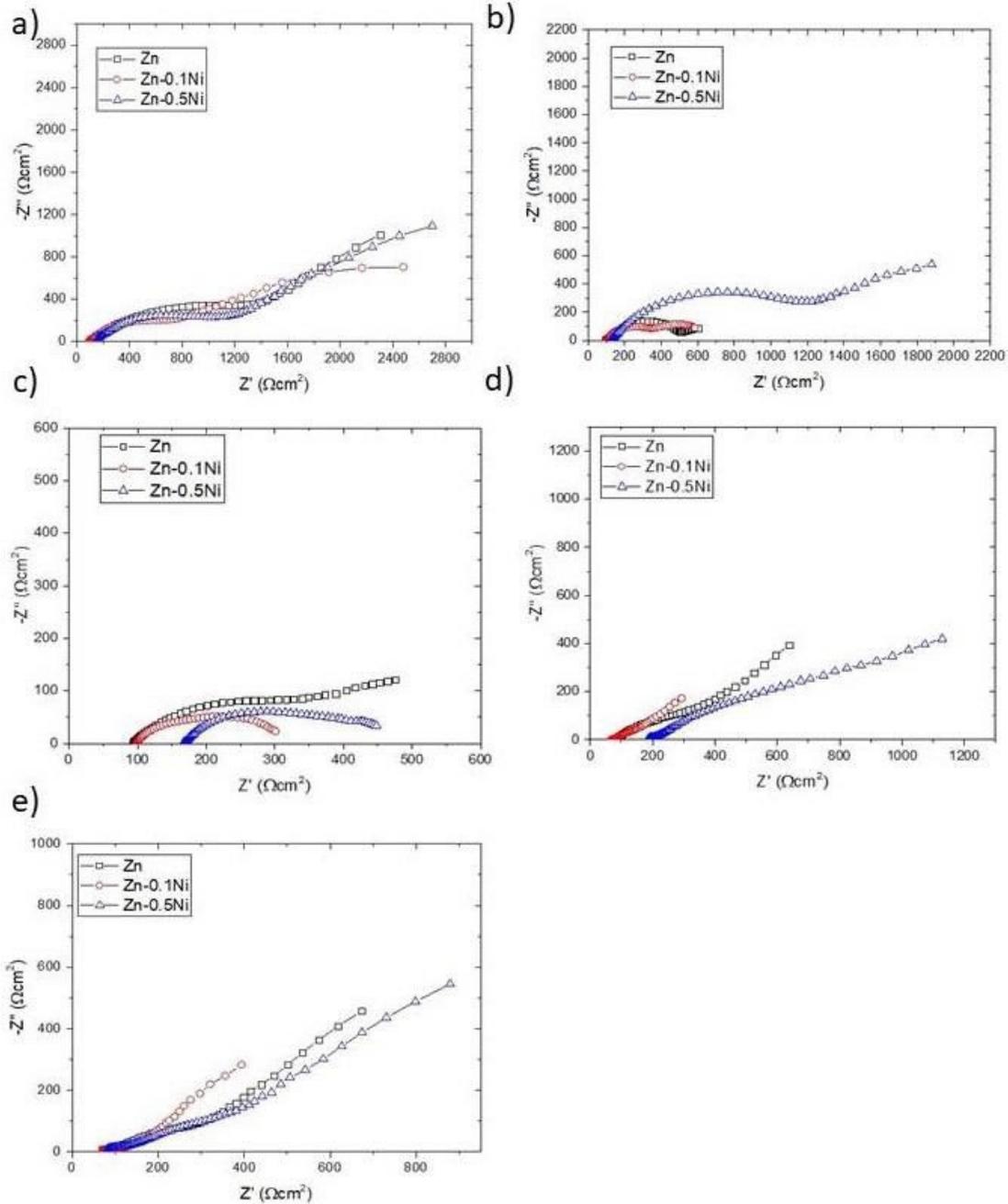


Figure. 2 Nyquist diagrams for the evolution in time of the Zn and Zn-Ni galvanized systems at A) 0 hours, B) 24 hours, C) 48 hours, D) 168 hours, and E) 336 hours of constant immersion.

The Nyquist plots were fitted to the electric equivalent circuits (EEC) displayed in Figure 3 where the parameters represent in these circuits R_{sol} represents the solution resistance; CPE1 is a constant phase element employed to represent the capacitive behaviour of the coating, in parallel, R_1 is the resistance of the coating, CPE2 represents the double-layer capacitance and R_2 symbolizes the charge transfer resistance (Hu et al. 2003). The CPE elements can be influenced by the surface roughness using the n parameter that can be evidenced by the depression of the semicircle on the Nyquist diagrams (Mahato et al. 2011). Moreover, the W-Yo represents the admittance of the diffusive element attributed to the mass transfer at low frequencies. This parameter is associated with the Warburg coefficient (σ_w) (Bard et al. 2000).

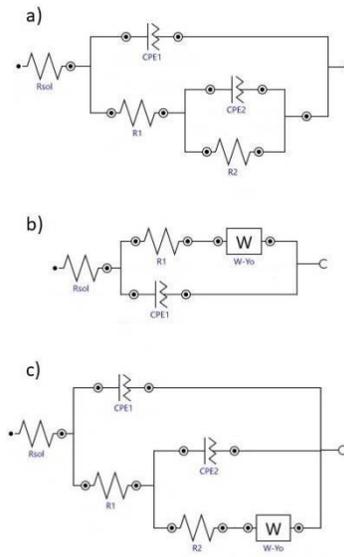


Figure. 3 Electric equivalent circuits employed to the fitting of Nyquist diagrams.

The evolution of the parameters and the electric equivalent circuit employed to fit the results are shown on Table 1.

Table 1. Fitted parameters of the electric equivalent circuits.

Zn									
Time	Rsol	R1	CPE1	n1	R2	CPE2	n2	σ_w	EEC
h	$\Omega \text{ cm}^2$	$\Omega \text{ cm}^2$	$S^{-1} \cdot s^n$		$\Omega \text{ cm}^2$	$S^{-1} \cdot s^n$		$\Omega \cdot s^{-1/2}$	
0	95.7	1430	2.08×10^{-4}	0.521	4050	0.00375	0.662	-	a)
24	98	170	2.40×10^{-4}	0.692	450	0.00125	0.27	-	a)
48	91.7	311	0.00129	0.56	--	--	--	32.1	b)
168	76.4	318	0.00134	0.509	--	--	--	110.1	b)
336	59.8	47	0.137	0.463	188	5.63×10^{-4}	0.601	115.3	c)
Zn-0.1%Ni									
0	83.8	950	2.36×10^{-4}	0.483	2600	0.00234	0.55		a)
24	101	272	3.24×10^{-4}	0.717	360	0.0069	0.599		a)
48	96.8	180	0.00239	0.565	289	0.0339	0.26	--	a)
168	63.9	12.7	2.02×10^{-4}	0.472	78.2	0.00219	0.547	549.8	c)
336	70	19.7	1.32×10^{-6}	0.902	57.4	8.25×10^{-4}	0.493	970.7	c)
Zn-0.5%Ni									
0	136	1110	6.46×10^{-5}	0.531	4050	0.00209	0.633		a)
24	129	3.64	2.35×10^{-5}	0.853	2070	3.65×10^{-4}	0.36		a)
48	168	7.3	1.22×10^{-4}	0.852	429	0.00169	0.36	--	a)
168	166	99.5	0.00162	0.234	660	8.81×10^{-4}	0.506	2548.3	c)
336	83.8	101	2.18×10^{-4}	0.393	212	5.58×10^{-4}	0.589	3205.2	c)

In general terms, results obtained are similar to those observed on the Nyquist diagrams; during the first immersion hours, the plots exhibit a higher resistive behaviour (R1 and R2) that decreases as corrosion time proceeds, evidencing the loss of the protective properties and the increase of the diffusive phenomena. Additionally, is observed that the sample with 0.5 Ni exhibits a higher resistance R1 and R2 than those displayed for the pure Zn and Zn-0.1%Ni indicating that this amount of Ni added provides better corrosion resistance.

Despite the degradation of the Zn-Ni galvanized samples, results acquired indicate that the low amount of addition of Ni enhances the corrosion resistance of the samples. This is in agreement with the work reported in the literature (Guosheng et al. 2013) and with the results determined on this work.

5.3 Validation

To validate the results and the behaviour determined, the impedance modulus in the low-frequency region (Figure 3 (a)) and the linear polarization resistance (Figure 3 (b)) were plotted. The results display a similar behaviour amongst both techniques indicating that the corrosion protection decreases as immersion time proceeds. Figure 3 (a) evidences that the Ni addition enhances the impedance modulus leading to higher corrosion protection of the samples. On the other hand, the linear polarization technique Rpol shows a minor contribution of the Ni addition to the corrosion performance of the samples but the trend prevails along immersion time.

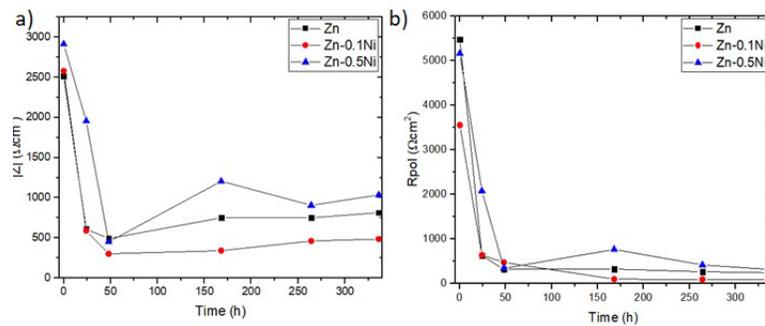


Figure 3 (a) Impedance modulus in the low frequency and (b) linear polarization resistance of the hot-dip galvanized samples.

6. Conclusion

The corrosion characterization of hot-dip galvanized steel samples covered with Zn, Zn-0.1%Ni and Zn0.5%Ni was performed and several conclusions can be derived. The results obtained indicate that the coating displays a stratified developed by the intermetallic phases η , ζ , δ and γ related with the Zn diffusion process. Also, the existent phases changes with the Ni amount added leading to the formation of a two and three-layered structure as Ni amount increase. The composition of the coating remains almost constant, evidencing the presence of crystalline phases attributed to simonkolleite, zinc hydroxide, zinc and Ni.

The corrosion behaviour of the samples shows that the samples exhibit at least two electrochemical processes related to the charge transfer and the protective behaviour of the coating and the oxide layer. Additionally, the mass transport is evidenced attributed to the displacement of aggressive species through the corrosion products layer developed. The impedance modulus and the polarization resistance slightly increase with the amount of Ni added. The protective effect of the layer decreases in a short time but trends to stabilize as immersion time proceeds.

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Biographies

Rafael González-Parra is a professor in the Division of Mechanical and Industrial Engineering and collaborator with the Surface Engineering Center in the Department of Design Engineering and Manufacture at the National Autonomous University of Mexico, Mexico City, Mexico. He earned his bachelor degree in Mechanical Engineering, his Masters in Materials Science and Engineering and his PhD in Materials Science and Engineering from National Autonomous University of Mexico, Mexico. He has published journal and conference papers. His research interests are corrosion, oxides and coatings. He is member of SMEQ and REDISYT.

Arturo Barba-Pingarrón is a Professor in the Division of Mechanical and Industrial Engineering and is Surface Engineering Coordinator of Surface Engineering Center in the Department of Design Engineering and Manufacture at the National Autonomous University of Mexico, Mexico. He earned his B.S. in Metallurgical Engineering from National Polytechnic Institute, Mexico, Masters in Materials Science and Engineering from National Autonomous

University of Mexico, Mexico and PhD in Chemistry of Materials, Metals Science from Barcelona University, Spain. He has published journal and conference papers and taught courses in surface engineering for graduate and undergraduate students. His research interests are thermochemical treatments and thermal spray techniques. He is member of REDISYT.

Raul Valdez-Navarro is a full-time associate professor at the Faculty of Engineering, Coordinator of the Advanced Manufacturing Laboratory and the Conventional Manufacturing Laboratory of the same institution. He earned his B.S. in Mechanical Engineering and Master of Materials Science and Engineering from the National Autonomous University of Mexico, Mexico. He has published journal and conference papers. His research topics are included in the development of thermal spray, electroless nickel plating and austempered ductile iron coatings for surface improvement, resistance to wear and corrosion.

Alba Covelo-Villar is a professor in the Division of Mechanical and Industrial Engineering and leader to generate and characterize superficial treatments of materials of the Surface Engineering Center in the Department of Design Engineering and Manufacture at the National Autonomous University of Mexico, Mexico. She got her PhD at the same university in Technology Materials with the development and characterization of nanocoatings Sol-gel on aluminum alloys. She carried out a postdoctoral research at the CENISA group of the National University of Mexico from 2012-2014 developing hybrid sol-gel coatings doped with nanocontainers and corrosion inhibitors loaded in nanofibers produced by the electrospinning technique. She has published journal and conference papers. Her expertise area comprises conversion coatings to protect metal/alloys against corrosion phenomena.

Miguel A. Hernández-Gallegos is a professor in the Division of Mechanical and Industrial Engineering at the Engineering School of the National University of Mexico, Mexico. He got his PhD in materials engineering at the Chemistry School of the National University of Mexico, Mexico studying the electrochemical and the anti-corrosion properties of waterborne organic coatings applied on steel substrates. He accomplished several fellowship jobs during his career. In 2003 he was at the Ecole Polytechnique Federal de Lausanne (EPFL), Switzerland with Prof. D. Landolt. From 2008-2010 and in 2019 he was at the University of Vigo, Spain with Prof. R. Nóvoa developing solgel coatings on aluminum alloys and hydrophobic anodized layers. In 2010 he also stayed with Prof. M. Zheludkevich at the University of Aveiro, Portugal studying localized corrosion measurements on AA2024 alloys. He has published many journal and conference papers. His expertise area relies on the electrochemical-corrosion properties of materials mainly metals/alloys.

Javier Cervantes-Cabello is a professor in the Division of Mechanical and Industrial Engineering of the National University of Mexico, Mexico. He earned his B.S. in Mechanical and Electric Engineering, his Master in Mechanical Engineering and his PhD in Engineering from National Autonomous University of Mexico, Mexico. He has published journal and conference papers. His research interests are advanced manufacture techniques and materials characterization.