

Microstructural and Corrosion Behavior of A413/Al₂O₃ Metal Matrix Composites in 3.5 wt.% NaCl Solution

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A study on the microstructure, microhardness, and corrosion behavior of A413/Al₂O₃ composites has been carried out. Composites with 5, 10, and 15 wt.% of Al₂O₃ were produced by powder metallurgy (PM) method. Electrochemical techniques including potentiodynamic polarization curves, linear polarization resistance and electrochemical impedance spectroscopy were used to evaluate the corrosion behavior in 3.5 wt.% NaCl solution. Detailed electronic microscopy studies revealed that the surface of composites has a fairly uniform distribution of Al₂O₃ particles and a slight porosity content ranging from 2.59 to 3.83 % whereas X-ray diffraction patterns indicated the presence of the main phases Al, Si, along with reinforcement Al₂O₃ phase. The microhardness of the samples improved with the addition of alumina as reinforcement, obtaining the highest microhardness value for the composite A413-15 wt.% Al₂O₃ (104.5 HV). Electrochemical tests showed that the corrosion rate of the samples increased as the weight percent of Al₂O₃ increased, where the unreinforced A413 alloy exhibited the lowest corrosion rate. Corrosion process was charge controlled and remained unaltered by the addition of reinforcement. After the corrosion test, the composites evidenced a type of localized corrosion such as crevice due to the galvanic effects between the A413 alloy matrix and the Al₂O₃ particles.

Keywords: A413/Al₂O₃ composites, microstructure, microhardness, corrosion behavior.

1. INTRODUCTION

A413 aluminum alloy is a special type of aluminum alloy with high levels of silicon (Si) that exhibits excellent properties such as light in weight, pressure tightness, high strength, high corrosion

resistance, and good electric conductivity [1, 2]. This type of alloy can be used in the aerospace, marine, cycling, and automotive industries to manufacture constituent parts that are very strong and also very light at the same time. The most important applications of A413 alloy are focused on the production of pistons, manifolds, connecting rods, housings, engine cylinders, air cylinders, and marine products [3, 4].

However, the properties of A413 aluminum alloy can be enhanced with the addition or combination of carbides, oxides, and intermetallic particles to produce the called aluminum metal matrix composites (AMMCs). The reinforcements commonly employed by many researchers [5-9] to produce AMMCs are B_4C , SiC , Al_2O_3 , TiC , and SiO_2 . Aluminum oxide (Al_2O_3) belongs to the most used oxide ceramics, and it has been selected as a reinforcing material in this investigation due to its high hardness, low density, electrical resistivity, high melting point, wear resistance, high refractoriness, and corrosion resistance.

Hardness and corrosion resistance of AMMCs can either increase or decrease depending upon the manufacturing technique, operating parameters, size, content, and type of reinforcing material. AMMCs can be fabricated in many ways, including squeeze casting [10], stir casting [11], liquid infiltration [12], powder metallurgy [13], and mechanical alloying [14]. Powder metallurgy (PM) is one of the best techniques to prepare AMMCs that employed powders as raw materials to produce net shapes, reducing secondary operations and waste. One of its main advantages is the improved homogeneous distribution of the reinforcement phase that with other techniques is difficult to achieve.

During the selection of materials, not only its physical and mechanical properties must be taken into account, but must also include the electrochemical properties which together help to choose the proper areas for AMMCs and know their limits to ensure good functionality. Most information available on AMMCs reinforced with alumina has focused on their mechanical behavior which has been persistent improved depending on the weight percent of reinforcement [15-17]. However, from the literature review on corrosion behavior of AMMCs reinforced with alumina in 3.5% NaCl solution, the results have not been persistent, showing variations or contradicting their corrosion behavior. For example, Oraei et al. [18], reported the effect of Al_2O_3 reinforcement particles on the corrosion behavior of Al(Zn) solid solution matrix produced by mechanochemical synthesis. The results of the impedance and polarization tests showed that the presence of Al_2O_3 particles in the Al(Zn) matrix significantly increases the corrosion resistance of the system. Niveen et al. [19], studied the electrochemical corrosion behavior of (AA6061) Al/ Al_2O_3 composites in 3.5% NaCl solution. Samples were produced by liquid metallurgy technique. They concluded that (AA6061) Al/ Al_2O_3 composites have lower corrosion resistance than AA6061 aluminum alloy due to the corrosion rate increased by increasing the weight percent of the Al_2O_3 particles. Mostafa et al. [20], studied the corrosion behavior of Al based nanocomposite foams reinforced with alumina nanoparticles produced by powder metallurgy-based sintering-dissolution process. The electrochemical test results determined that increasing the Al_2O_3 content reduced the corrosion rate. From the literature review, there are several aluminum metal matrix composites with reinforcements combinations that may display distinct corrosion properties.

The present study aims to investigate the microstructure, microhardness, and corrosion behavior of A413/ Al_2O_3 composites with different contents of Al_2O_3 produced by powder metallurgy technique.

The composites were evaluated by electrochemical techniques in 3.5 wt.% NaCl solution. Thus, this study is an effort to contribute important information on the behavior of these A413/Al₂O₃ composites.

2. EXPERIMENTAL PROCEDURE

2.1. Testing materials

In this study, A413 aluminum alloy powder, with particle size less 250 μm and chemical composition as given on Table 1, was used as a matrix material. Aluminum oxide powder (primarily α phase, Sigma-Aldrich, 99.5% purity, and 10 μm particle size) was used as reinforcement for the synthesis of A413/Al₂O₃ composites.

Table 1. Nominal composition (wt. %) of A413 aluminum alloy powder.

Al	Si	Fe	Zn	Mn	Mg	Cu	Ti	Cr	Ni	Pb
Balance	11.69	0.75	0.28	0.23	0.06	0.28	0.027	0.021	0.015	0.005

2.2. Synthesis of the composites

The A413/Al₂O₃ composites were synthesized through the powder metallurgy (P/M) route with different weigh percentages (5, 10, and 15 wt.%) of Al₂O₃. The preparation consisted of mixing the matrix (A413 powders) with the Al₂O₃ particles in suitable proportions. The powders mixtures were milled using a planetary high-energy ball mill (Fritsch-Pulverisette 7) at 300 rpm for 5 h under argon atmosphere with a ball to powder ratio of 5:1. Stearic acid (2 ml) was used in each vial as a process control agent (PCA) to prevent excessive cold welding. The milled A413/Al₂O₃ powders were cold compacted in a cylindrical die setup at 680 MPa resulting in green compacts. The sintering of the green products was done at 500 °C for 4 h under an argon gas atmosphere.

Table 2. Composition used for the aluminum matrix composites.

Sample designation	A413 alloy (wt. %)	Al ₂ O ₃ (wt. %)
A413	100	0
A413-5% Al ₂ O ₃	95	5
A413-10% Al ₂ O ₃	90	10
A413-15% Al ₂ O ₃	85	15

For comparative purposes, an unreinforced A413 aluminum alloy was also prepared under the same conditions. The composition used for the aluminum matrix composites is shown in Table 2. The

X-ray diffraction (XRD) patterns were recorded on a D2 Phaser diffractometer. Archimedes principle was used to determine the experimental density of the samples and the mixing rule to calculate the theoretical density. The theoretical and experimental densities were used to calculate the percent porosity by [21] Equation 1.

$$\text{percent porosity} = \{(\rho_{\text{Th}} - \rho_{\text{Ex}}) \div \rho_{\text{Th}}\} \times 100 \% \quad (1)$$

where ρ_{Th} = Theoretical Density (g/cm^3), and ρ_{Ex} = Experimental Density (g/cm^3).

The microhardness of the samples was evaluated using a Shimadzu HVM Microhardness Tester and was performed using an indentation load of 0.9807 N with a dwell time of 15 seconds. The average value of five measurements for each sample was reported.

2.3. Electrochemical measurements

Corrosion behavior of composites was investigated by using electrochemical techniques such as potentiodynamic polarization curves, linear polarization resistance (LPR), and electrochemical impedance spectroscopy (EIS) measurements. Electrochemical tests were done by using an ACM Instruments potentiostat Gill AC controlled by a desktop computer. Electrochemical measurements were carried out in a conventional cell composed of three electrodes using a 3.5% NaCl solution at room temperature. Samples were welded to a copper wire and then encapsulated with commercial epoxy resin to be used as working electrodes (WE). A graphite rod was used as a counter electrode (CE), and a saturated Ag/AgCl was used as the reference electrode (RE). Before testing, each working electrode surface was prepared by ground with silicon carbide papers from 240 down to 600 grit in agreement with ASTM [22] standard, then rinsed with distilled water, degreased with acetone, and dried with cold air. Polarization curves were carried out using a sweep rate of 1 mV/s and polarizing the samples from -600 mV up to +1500 mV with respect to the open circuit potential (OCP). LPR measurements were performed by polarizing the samples ± 10 mV versus the open circuit potential value at a sweep rate of 1 mV/s, taking readings every 60 minutes during 24 hours. EIS measurements were carried out at the OCP value by using a signal with an amplitude of 10 mV RMS, and the frequency spectrum was scanned between 0.1 Hz to 100,000 Hz. Each different sample was repeated three times to guarantee the reproducibility of the data. The samples were analyzed before and after the corrosion test by scanning electron microscope (SEM), JCM-6000 Plus, equipped with EDX analysis.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Fig. 1(a-d) shows the microstructure of A413 base alloy and A413/ Al_2O_3 composites with different concentrations of Al_2O_3 particles as 5, 10, and 15 wt. % before the corrosion test.

It can be seen that the microstructure of the samples is composed of a white phase (Al_2O_3 particles) and a gray zone corresponding to the A413 aluminum alloy matrix.

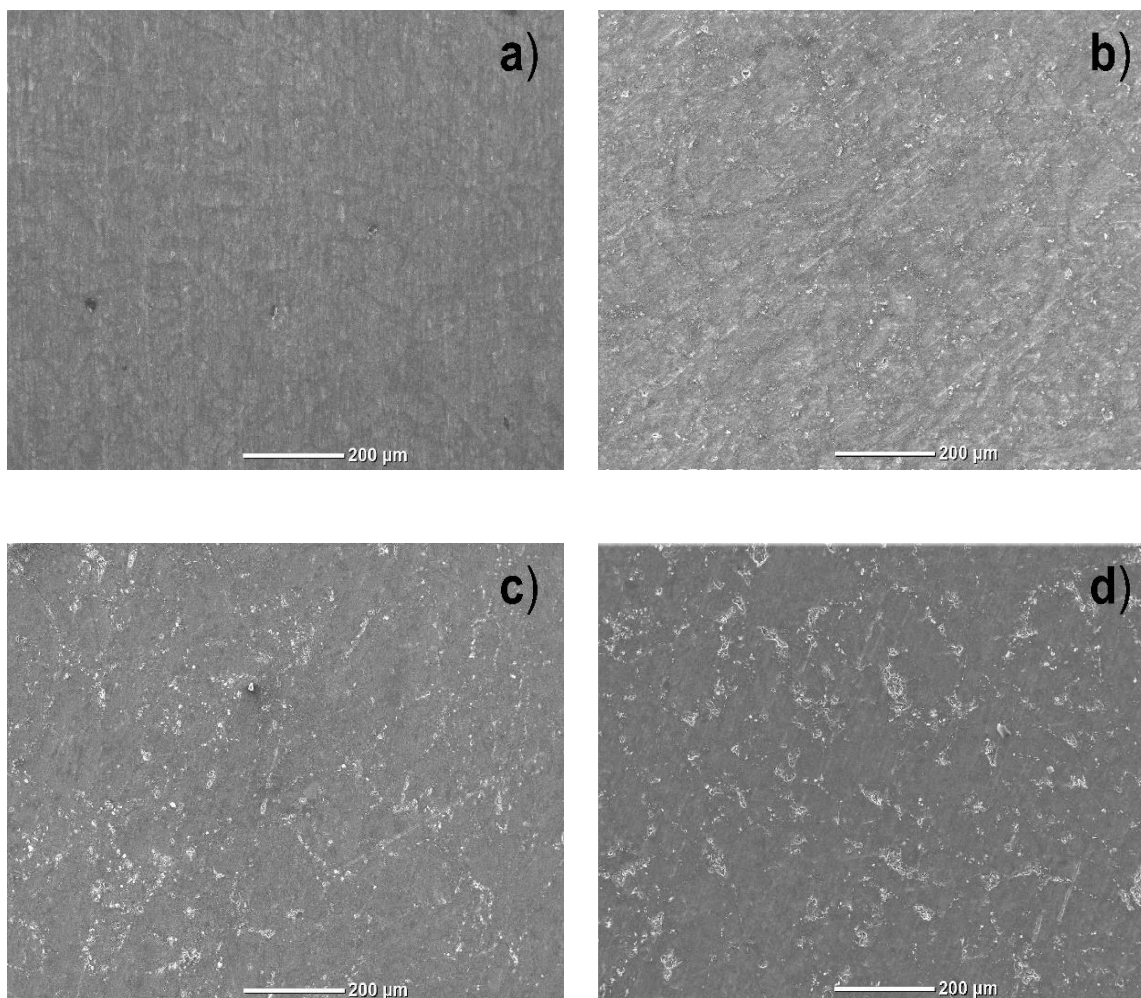


Figure 1. Microstructure of A413/ Al_2O_3 composites containing (a) 0, (b) 5, (c) 10, and (d) 15 wt.% Al_2O_3 .

In Fig. 1(a) it is clearly seen that the unreinforced A413 alloy does not show the presence of Al_2O_3 particles. However, Fig. 1(b), 1(c), and 1(d) show that the presence of Al_2O_3 is visible and more evident on the surface of the composites as its weight percent increased, showing a fairly uniform distribution, and well bonded to the A413 matrix. The surface microstructure of the samples reveals slight porosity content, which is a characteristic of samples produced by the powder metallurgy technique. Also, as the content of Al_2O_3 particles increases, the porosity of the composites is more visible on their surface, it can be attributed to the hard Al_2O_3 particles that minimize the compressibility of the powders, promoting the porosity increases.

3.2. Density, porosity, and microhardness

The density, porosity, and microhardness results for the base A413 alloy and composites are shown in Table 3. It can be seen that the density, porosity, and microhardness of the composites increased with the increase of Al₂O₃ particles in the A413 matrix alloy.

Table 3. Comparison of density, porosity, and microhardness of the composites.

Sample designation	Theoretical density, g/cm ³	Experimental density, g/cm ³	Porosity, %	Hardness (Hv)
A413	2.66	2.591 ± 0.02	2.59	66.4 ± 6
A413-5% Al ₂ O ₃	2.711	2.633 ± 0.06	2.87	71.4 ± 5
A413-10% Al ₂ O ₃	2.763	2.673 ± 0.03	3.25	93 ± 6
A413-15% Al ₂ O ₃	2.814	2.706 ± 0.03	3.83	104.5 ± 7

The theoretical and experimental density values increased, due to the density of Al₂O₃ (3.22 g/cm³) is higher as compared to the matrix alloy (2.66 g/cm³). However, the experimental density showed lower values as compared to theoretical density due to the presence of porosity ranging from 2.59 to 3.83 %.

The microhardness results show that composite with 15 wt.% Al₂O₃ particles exhibited the highest value of 104 HV, which represents an improvement of 57.3 % as compared to the unreinforced A413 alloy. The improvement of microhardness is due to the increases and uniform distribution of hard Al₂O₃ particles into the matrix alloy. However, it was found that microhardness is related to the crystallite size [23, 24]. Thus, the microhardness of composites tends to increase as the crystallite size decreases.

3.3. X-ray diffraction analysis

Fig. 2 presents the XRD patterns of the A413 aluminum alloy and composites with different concentrations of Al₂O₃ particles. The XRD patterns of the composites show the characteristic peaks of aluminum (Al), silicon (Si), and alumina (Al₂O₃), while the XRD pattern of the unreinforced alloy only shows the main phases of aluminum (Al) and silicon (Si). It was observed that the intensity of Al₂O₃ peaks (located at 2θ values of 25.57, 35.13, 43.33, and 57.47°) increased with the increase of Al₂O₃ particles in the composites, while the Al peaks (located at 2θ values of 38.52, 44.76, 65.13, 78.24 and 82.37°) and Si peaks (at 2θ values of 28.44, 47.27 and 56.07°) became broader and their intensities decreased, such behavior is related to the reduction on the crystallite size [25].

The crystallite size was determined using Scherrer equation [26] and the full peak width at half maximum (FWHM) of Al (111), Al (200), Al (220), and Al (311) peaks. The crystallite size values of the base A413 alloy and composites with 5, 10, and 15 wt.% Al₂O₃ are 68.24, 66.76, 36.24, and 23.55

nm, respectively. The results confirm the reduction of the crystallite size as the amount of Al_2O_3 particles increases.

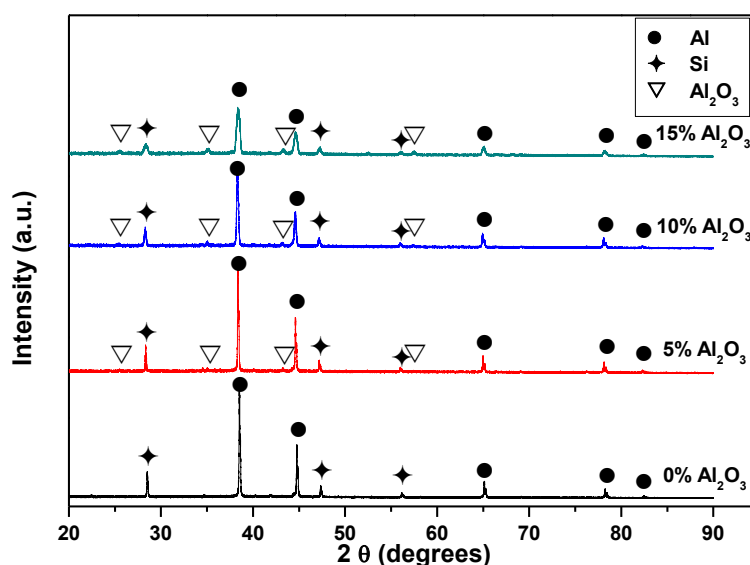


Figure 2. X-ray diffraction patterns of A413 aluminum alloy and the A413/ Al_2O_3 composites.

3.4. Corrosion results

3.4.1. Potentiodynamic polarization curves

Polarization curves for A413 aluminum alloy reinforced with different wt. % of Al_2O_3 particles in 3.5% NaCl solution are given in Fig. 3. It can be seen that the A413/ Al_2O_3 composites and the unreinforced A413 alloy displayed similar polarization curves showing an active-passive behavior, showing an increase in the anodic dissolution current as the applied anodic potential increases until a more or less stable, passive current density value is found, where the passive zone starts and it ends where a pitting potential value, E_{pit} , is reached. This E_{pit} value increases as the reinforcement Al_2O_3 particles increases. Unreinforced A413 aluminum alloy had the noblest free corrosion potential (E_{corr}) value, however, when the A413 alloy was reinforced with Al_2O_3 particles, the polarization curves exhibited a shift in the E_{corr} value towards more active values.

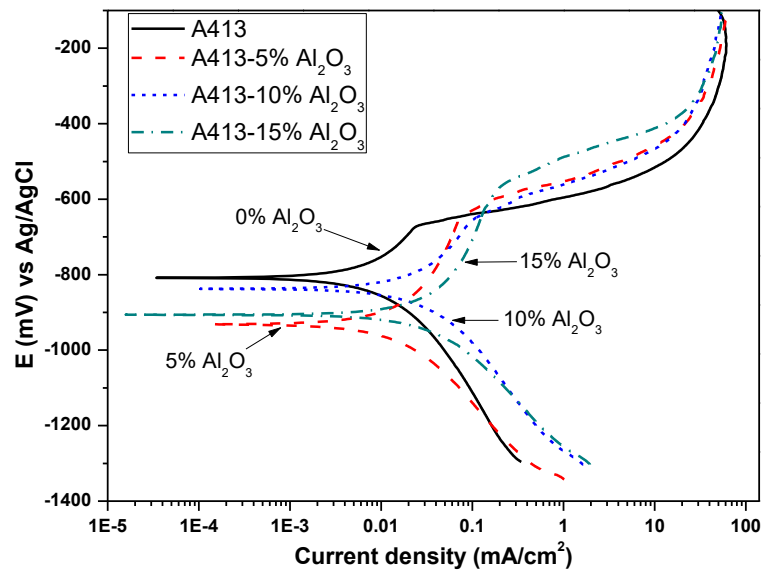


Figure 3. Polarization curves for A413 aluminum alloy and A413/Al₂O₃ composites in 3.5% NaCl solution.

Table 4. Electrochemical parameters obtained from the polarization curves.

Sample designation	E _{corr} (mV)	i _{corr} (mA/cm ²)	-b _c (mV/dec)	E _{pit} (mV)
A413	-808	0.0132	330	-660
A413-5% Al ₂ O ₃	-930	0.0178	270	-650
A413-10% Al ₂ O ₃	-837	0.0378	323	-600
A413-15% Al ₂ O ₃	-904	0.0407	260	-580

Table 4 presents the electrochemical data calculated from the polarization curves. It can be seen that the A413/Al₂O₃ composites show distinct corrosion behavior, as the amount of the Al₂O₃ particles increased the corrosion current density (i_{corr}) value increased. Thus, the lower i_{corr} value was found for the unreinforced A413 alloy (0.0299 mA/cm²), whereas the highest value was for the composite with 15 wt.% Al₂O₃ (0.0407 mA/cm²), respectively. In terms of corrosion kinetics, a higher value of the corrosion current density in the composite with 15 wt.% Al₂O₃ implied a decrease in corrosion resistance, whereas a lower corrosion current density value implies better performance. Similar behavior has been reported by Karabulut et al. [27] for the corrosion of pure Al with Al₂O₃ in 3.5% NaCl where the polarization curves revealed an increment in the i_{corr} value with the addition of 10 wt.% of Al₂O₃. Thus, the addition of Al₂O₃ particles at different concentrations of 5, 10, and 15 wt.% has a negative effect in the composites by increasing the unreinforced A413 alloy corrosion rate.

3.4.2. Linear polarization resistance (LPR)

The variation in the linear polarization resistance value, R_p , with time for the unreinforced A413 aluminum alloy and A413/ Al_2O_3 composites is given in Fig. 4.

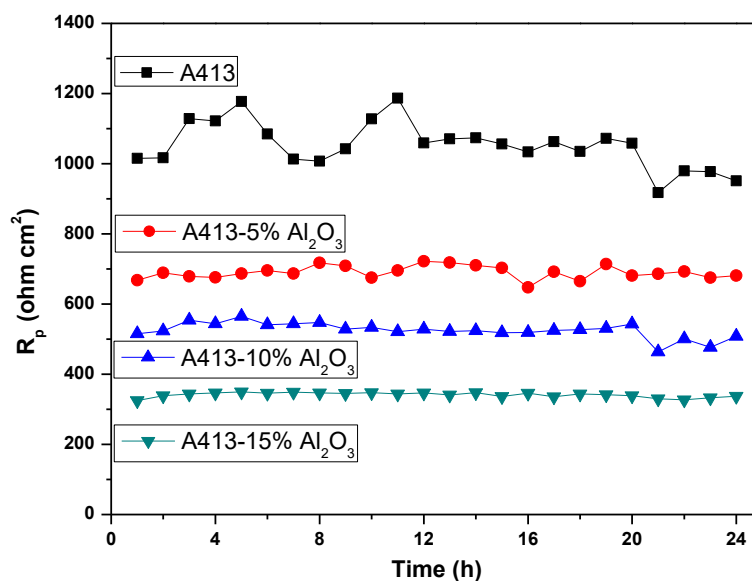


Figure 4. Change on the R_p value with time for A413 aluminum alloy and A413/ Al_2O_3 composites in 3.5% NaCl solution.

The results clearly demonstrated that the unreinforced A413 aluminum alloy has the highest R_p value, and thus the highest corrosion resistance. The initial R_p value for the unreinforced A413 aluminum alloy was 1015 ohms cm^2 , however, during the test, this value slightly decreased or increased during some periods of time due to the dissolution or breakage of the Al_2O_3 layer and its repair. Also, at the end of the test of 24 hours, the R_p value was 950 ohms cm^2 . However, when the A413 aluminum alloy was reinforced with Al_2O_3 particles, the R_p value of the composites decreased to values between 324 and 667 ohm cm^2 during the first hour of the test, decreasing its corrosion resistance and increasing its corrosion rate. The lowest R_p value, and thus the higher susceptibility corrosion, corresponds to the composite with 15 wt. % Al_2O_3 , with a value of 336 ohm cm^2 . Therefore, the LPR results support the polarization curves results above that indicate that the unreinforced A413 aluminum alloy has less susceptibility to corrosion in a 3.5 wt.% NaCl a solution, and the corrosion resistance of composites decreases with an increase of the Al_2O_3 reinforced particles.

3.4.3. Electrochemical impedance spectroscopy (EIS)

EIS data in both Nyquist and Bode diagrams for the A413/ Al_2O_3 composites at different concentrations (5, 10, and 15 wt.%) of Al_2O_3 in an electrolytic medium of 3.5 wt.% of NaCl during an immersion period of 24 hours are shown in Fig. 5. In the Nyquist diagrams, Fig. 5a, the presence of two capacitive, depressed semicircles, one at high and intermediate frequencies and a second one at lower

frequency values is shown; the response of the A413/ Al_2O_3 system at high and intermediate frequencies is attributed to the electrochemical reactions processes resulting from the creation of an oxide film on the surface of the metal substrate, whereas the low frequency semicircle is due to the charge transfer process that occurs from the metal to the solution through the double electrochemical layer. Since the shape of the Nyquist diagrams did not change with the addition of the reinforcement, the corrosion mechanism remained unaltered.

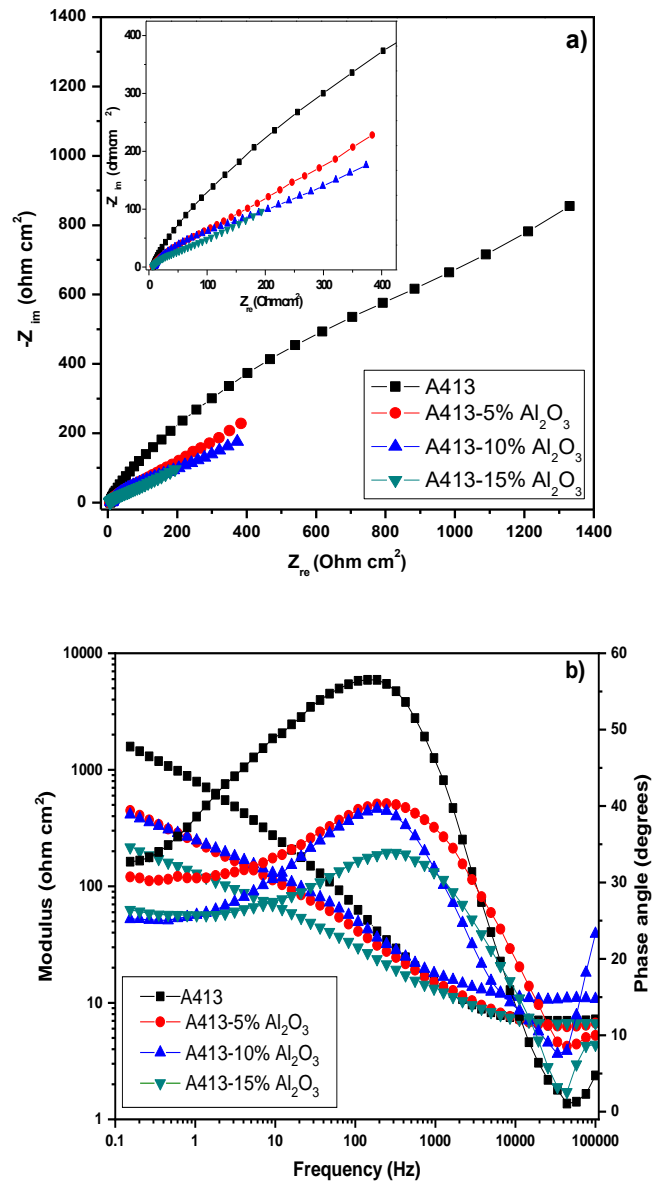


Figure 5. a) Nyquist and b) Bode diagrams for A413 aluminum alloy and A413/ Al_2O_3 composites in 3.5% NaCl solution.

The semicircles diameter of the unreinforced A413 alloy is larger than that for the composites and there was an appreciable decrease in the semicircle diameter with the addition of Al_2O_3 particles, which indicates a change in the corrosion reactions on the electrode surface. The larger semicircle

exhibits the presence of passive film on the surface of the sample which turns out to be the main reason for the decreased corrosion rate, whereas, the smaller semicircle display low polarization resistance, thus, an increase in the corrosion rate.

To carry out a more detailed analysis of the information obtained by the EIS technique, it was decided to represent the results of the A413/Al₂O₃ system using Bode diagrams (Fig. 5b), which represent the variation of the total impedance as a function of frequency. Bode diagrams show that the highest impedance was for the unreinforced A413 aluminum alloy. In the A413/Al₂O₃ system, the total impedance tends to decrease as the weight percentage of Al₂O₃ increased (Table 5), which represents lower protection against corrosion, coinciding with the data obtained in the polarization resistance technique (Fig. 3).

Table 5. Electrochemical parameters used to simulate the EIS data.

Sample	Modulus (ohm cm ²)	R _s (ohm cm ²)	R _{cp} (ohm cm ²)	C _{cp} (F/cm ²)	R _{ct} (ohm cm ²)	C _{dl} (F/cm ²)
A413	1945	7.3	412.7	9.6x10 ⁻⁵	1525	1.5x10 ⁻³
A413-5% Al ₂ O ₃	568	6.7	145.3	2.7x10 ⁻⁴	416	5.4x10 ⁻³
A413-10% Al ₂ O ₃	504	10.8	156.2	2.5x10 ⁻⁴	337	6.7x10 ⁻³
A413-15% Al ₂ O ₃	264	6.7	81.3	4.8x10 ⁻⁴	176	1.3x10 ⁻²

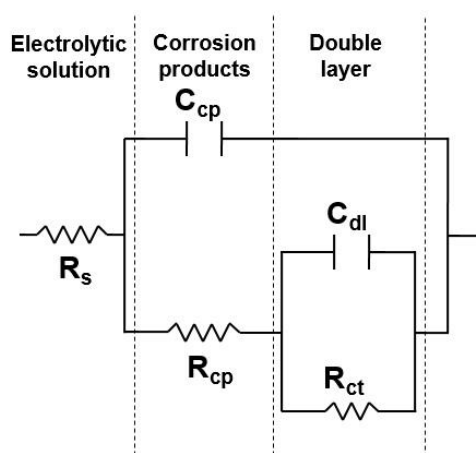


Figure 6. The equivalent electric circuit used to simulate the EIS data.

The diagram representation of the phase angle (Fig. 5b) and the broadening of its curve represent the presence of two time constants, the first corresponding to the formation of an oxide layer and the second to a charge transfer process associated with the effect of the capacitance of the double layer. It is also seen that the phase angle decreased with the increase of Al₂O₃ content. This means that the composites with increasing concentrations of Al₂O₃ are more susceptible to corrosion. Unreinforced

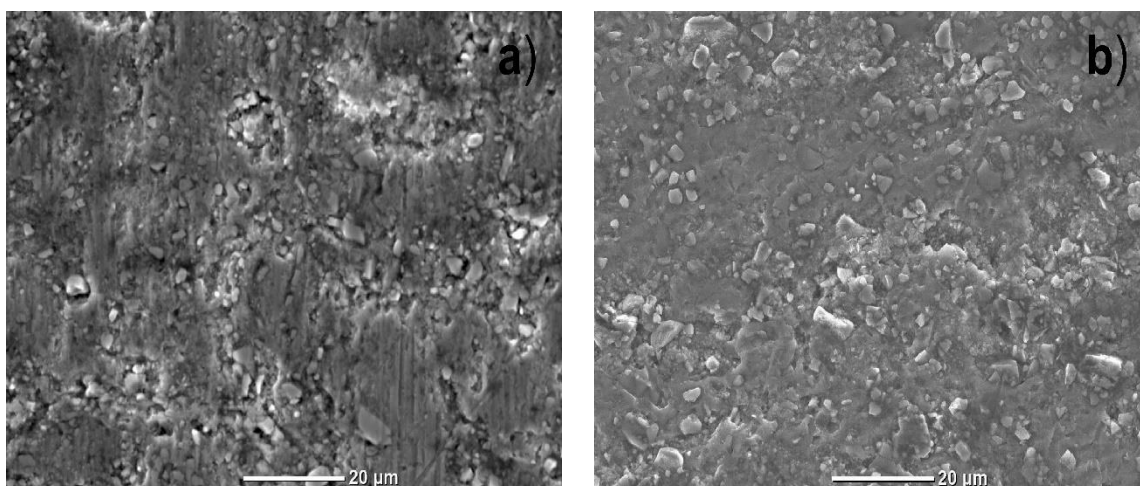
A413 alloy obtained the maximum value of phase angle (56°) in the intermediate frequency region, thus, exhibiting the best corrosion resistance as compared to the composites.

The equivalent circuit for the A413/ Al_2O_3 system is shown in Fig. 6. In this circuit, R_s corresponds to the resistance of the electrolyte or solution, followed by the corrosion products resistance (R_{cp}) and capacitance (C_{cp}) respectively, as well as a charge transfer resistance (R_{ct}) and the capacitance of the double layer (C_{dl}).

The data presented in Table 5 were extracted from the different Bode and Nyquist diagrams obtained from the EIS evaluation, these values were used for the simulation and determination of the capacitance of the A413/ Al_2O_3 composites at different concentrations of Al_2O_3 . Table 5 exhibited that the values of charge transfer resistance (R_{ct}) and corrosion products resistance (R_{cp}) decreased as the amount of Al_2O_3 increased in the A413 matrix alloy. Therefore, this behavior of the composites indicates a lower corrosion resistance than that of A413 alloy without reinforcement. However, the capacitance of the corrosion products and double layer capacitance values, C_{cp} and C_{dl} respectively, increased with the addition of Al_2O_3 particles. The lowest C_{dl} value was for the unreinforced A413 alloy, thus, this alloy has obtained a better passive film than the composites.

3.4.4. SEM micrographs

SEM micrographs acquired from the surface of the corroded composite samples after the LPR measurements are shown in Fig. 7(a-d). The SEM micrograph of the unreinforced A413 alloy (Fig. 7a) showed a type of localized corrosion such as crevice with lower deterioration. As can be noticed, the crevice corrosion increased and was deeper as the percent of Al_2O_3 increased in composites. Thus, the composite with 15 wt.% of Al_2O_3 exhibited a higher surface deterioration, therefore, a lower corrosion resistance (Fig. 7d). All the samples evidence a localized crevice type of corrosion commonly reported by researchers in different AMMCs [28-30]. The corrosion process of the samples was given by the formation of the passive layer which forms and covers the surface, however, this was interrupted at the A413 matrix/ Al_2O_3 interface, inducing a localized type of corrosion.



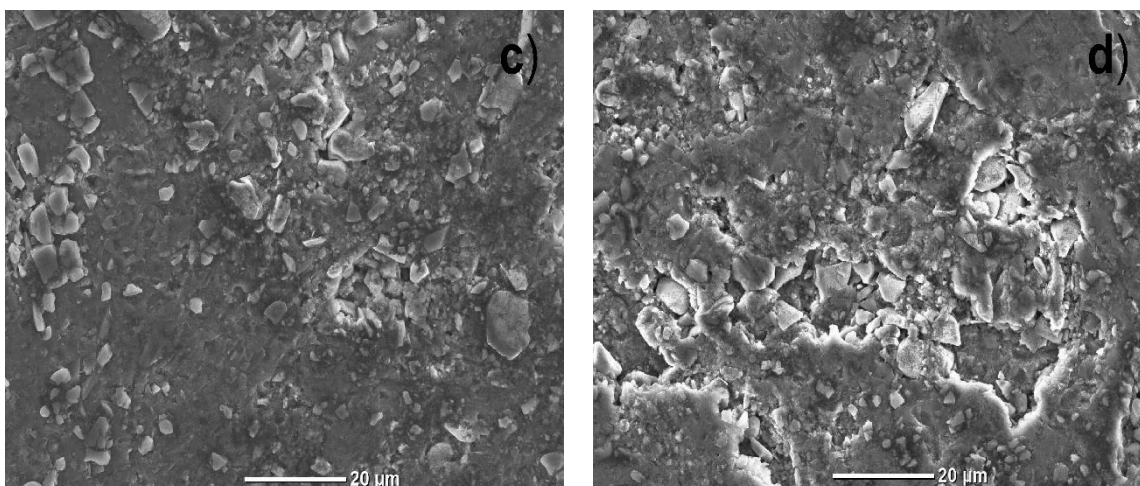


Figure 7. SEM micrographs of A413/Al₂O₃ composites containing (a) 0, (b) 5, (c) 10, and (d) 15 wt.% Al₂O₃ corroded in 3.5% NaCl solution.

This might be explained in terms of the galvanic effect, due to the Al₂O₃ particles acted as cathode, whereas the surface of the A413 aluminum alloy acted as anode due to its different electrochemical potential that causes the presence of microgalvanic cells. The SEM analysis was in accordance with the results of the electrochemical techniques, where the unreinforced alloy exhibited higher corrosion resistance as compared to the composites.

4. CONCLUSIONS

The microstructure, microhardness, and corrosion behavior of A413/Al₂O₃ composites were investigated and the following conclusions are made: The microstructure analysis confirms a fairly uniform distribution of Al₂O₃ particles into the composites. The hardness results confirm that the Al₂O₃ reinforcement had a positive effect on increasing the microhardness of the composites. The composite A413-15 wt.% Al₂O₃ exhibited the highest microhardness value of 104.5 HV, an improvement of 57.3 % in comparison to the A413 alloy without reinforcement. Polarization curves showed that the corrosion current density and corrosion rate in composites increases with increasing the percent of Al₂O₃ particles. Thus, the unreinforced A413 alloy has the higher corrosion resistance. EIS results revealed that the semicircle diameter, impedance, and phase angle tends to decrease as the weight percentage of Al₂O₃ increases, which indicated that the composites are more susceptible to corrosion as compared to unreinforced A413 alloy. Corrosion process was charge-transfer controlled and it was unaffected by the reinforcement. The SEM micrographs after corrosion tests present a type of localized corrosion, where the crevice corrosion increases by increasing the content of the Al₂O₃ particles in composites. It can be concluded that the reinforcement material had a negative effect decreasing the corrosion resistance of the composites.

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