

Development of Al-Mg Alloy for the Protection of Steel Structure in 3.5% NaCl

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ABSTRACT

Aluminium (Al) is thoroughly used as a sacrificial metal for the protection of steel in seawater. Aluminium is rarely used as a sacrificial anode without being alloyed due to its built-in properties to get passivated and form a passive layer and the continuity of its layer depends on the chemical composition and microstructure. In this paper, Aluminium-Magnesium (Al-Mg) system is considered because magnesium remarkably affects the chemical properties by changing the microstructure of Aluminium from a homogeneous solid solution to a complex structure with multiple intermetallic phases. In this research work developing Aluminium-Magnesium alloys containing 2.5 and 4.5 weight percent magnesium. The microstructures of pure aluminium and developed alloys were studied and the effect of the distribution of magnesium whether as a second phase particle, intermetallic compound or precipitates on the performance of aluminium sacrificial anode was discussed. Standard weight loss method and potentiodynamic tests are used to evaluate the performance of alloy sacrificial anode. The result shows that as the magnesium content increases corrosion rate of developed alloy increases and corrosion potential shifts in more negative directions. Corrosion potential is increased by 250 mV at 4.5% magnesium because of the formation of an intermetallic compound of aluminium & Magnesium (β phase - Al₃Mg₂) at the grain boundary. Magnesium improves the corrosion rate of developed alloy, it acts as a depassivator for the passive film of Aluminium by forming intermetallic compound and this compound has different electrochemical properties that leads to corrosion of aluminium and improves the performance of aluminium sacrificial anode.

Keywords: Cathodic protection; aluminium and its alloy; aluminium sacrificial anode; passivation and corrosion of aluminium

INTRODUCTION

The phenomenon of corrosion is as old as the history of metals, it is nothing but the degradation or oxidation of metals. Our human civilization is not possible without metals, so living with corrosion bay. Now a day it is a large challenge for the industrial world. Several techniques have been developed to prevent corrosion like inhibitors, design modification, coating and anodic-cathodic protection from which Cathodic protection (C.P.) is being deployed by major industries (Zaki Ahmad 2006; Pierre, n.d.2008).

Corrosion is initiated at the anode of the corrosion cell by generating electrons and these electrons are consumed at the cathode of the corrosion cell. This is the basic concept

of cathodic protection to convert anode into the cathode to restrict corrosion. This can be achieved by two ways: (1) Impress current method and (2) Sacrificial anode method. Sacrificial anode method is most preferable due to its ease of installation, no need for an external power source, and also suitable for localized protection. The main objective of this method is that the metal to be protected is coupled with more active metal (anodic metal) and make it cathode. Hence, all the corrosion concentrated in active metals call a sacrificial anode (Uhlig and Revie 1985; Bardalns 2013).

Nowadays, Magnesium (Mg), Zinc (Zn) and Aluminium (Al) are the most preferable sacrificial metals for cathodic protection. From which, Aluminium is acquiring appreciable properties as a sacrificial metal like

a light in weight and density, ease of availability, large electrochemical equivalent, thermal and electrical conductivity, high current capacity, and reasonable cost (Lameche-Djeghaba et al. 2014; Orozco-Cruz, Genesca, and Juarez-Islas 2007) (Wigg and Fleury, n.d., 2007).

Aluminium belongs to a group of passive metals; therefore, when used in seawater aluminium forms a passive layer of aluminium oxide which restricts further corrosion and decreases the efficiency of preventing steel structure. Hence, aluminium is alloyed with other elements to modify surface oxide and make the surface-active & shifted close circuit potential in a more electronegative direction. These alloying elements are referred to modifiers and depassivators which will break down the passive film and improve the corrosion of the aluminium anode. Generally Mg, Zn, Sn, Ga and Ti are used as an alloying elements (Vargel 2020c; Srinivas, Adapaka, and Neelakantan 2016; El-Hadad, Moussa, and Waly 2020).

Performance of aluminium sacrificial anode is based on type and amount of alloy addition and their distribution during solidification whether as a secondary phase particle or intermetallic compound. This phase have different electrochemical properties so micro galvanic cell is formed and difference in electrode potential is a driving force for the corrosion (Muazu and Yaro 2011; Umoru and Ige 2007; El-Hadad, Moussa, and Waly 2020).

Magnesium is one of the two most soluble elements in aluminium (the other being zinc) in the solid-state, 14.9 wt.% at 451°C and 1.7 wt.% at room temperature and forms a eutectic type of system. The excess magnesium beyond the solubility limit precipitated as Al₃Mg₂ (β -phase). This β -phase has mainly formed at the grain boundary and is incoherent with the matrix. The open-circuit potential of this β -phase is -1150 mV SCE which is very anodic compared to the Aluminium matrix (-760 mV SCE) leading to selective dissolution and improving the corrosion of Al-Mg alloy and if Mg exceeds 3.5 weight per cent than the mass loss is observed due to sensitization (Choi et al. 2018; Vargel 2020a; Thesis 2017; Vargel 2020b).

The idea of this paper is to develop an Al-Mg sacrificial anode in which Mg act as a depassivator and improve performance of Aluminium sacrificial anode for the protection of steel in seawater.

EXPERIMENTAL DETAILS

MATERIALS

99.8% Pure Aluminium wire and 99.9% Magnesium Ribbon used for this study are obtained from Honest metal cast and Chemdyes corporation respectively. As received

chemical composition given in Table 1.

CASTING AND CHARGE CALCULATION

Total two compositions are developed, one is aluminium containing 2.5 weight % Mg and another is aluminium containing 4.5 weight % Mg through liquid metallurgy route. Melting Practice is carried out in Electric resistance furnace around 720 °C temperature using metallic die having 5 rod, each rod have 2.5 cm diameter and 15 cm length.

Volume of each rod (v) = $\pi r^2 h = 73.60 \text{ cm}^3$
 Mass of one rod (m) = ρ (density). $v = 199 \text{ gram}$
 (density of pure Aluminium is 2.7 gm/cc)
 Total Mass required for melting is 1.4 Kg.
 (Including Runner & Riser)

For Design of alloy taking total mass 1.5 kg considering melting. Charge material for furnace is given in Table 2.

TABLE 1. Chemical Composition of raw material

Material	%Al	%Mg	%Zn
Aluminium Wire*	99.78	0.001	0.010
Magnesium Ribbon	--	99.5	0.005

*Aluminium wire containing impurities like Cr, Si, Fe, Cu and Mn less than 0.1%.

In Furnace, Aluminium was charged and after getting a processing temperature of around 720 °C, the addition of Magnesium was carried out in a bath of liquid aluminium. Stirring was done with the help of stainless steel rod to get uniform molten metal. Drossing and Degassing were carried out for better melting practice than the molten mixture was poured into a permanent metallic die made-up of mild steel. After cooling & solidification casting was removed than sampling was accomplished for various characterization.

TABLE 2. Charge calculation for furnace

No	Alloy	Aluminium (grams)	Magnesium (grams)	Total (grams)
A	Al+2.5%Mg	1462	38	1500
B	Al+4.5%Mg	1432	68	1500

CHEMICAL ANALYSIS

Chemical analysis of developed alloy was carried out with Bruker Q4 TASMAN advanced CCD based optical emission spectroscopy according to ASTM E1251:2017.

The chemical composition of the developed alloy was listed in Table 3.

MICROSTRUCTURE AND PHASE ANALYSIS

Test specimens were prepared by conventional metallography practice according to ASTM E407-07(2015) including rough and fine grinding up to 1200 number Sic emery paper using kerosene as a coolant. The ground surface was polished with the alumina paste on velvet cloth to obtain a surface having mirror polish. The polished surface was etched by 0.5% HF solution for 10 seconds. The etched sample was carefully handled and washed with water and air dry for 30 seconds, after that image was observed in optical microscope and JEOL 5610 LV Scanning Electron Microscope (SEM) with Energy Dispersive Spectroscopy (EDS) at different magnifications. X-Ray Diffraction (XRD) with Pan Analytical X'pert Pro Machine was carried out using Cu as the anode material with a k-alpha wavelength of 1.54060 Å for phase identification.

TABLE 3. Chemical Analysis of Developed Alloy

(%) Elements	Al + 2.5% Mg	Al + 4.5% Mg
Mg	2.461	4.537
Al	96.87	94.74

CORROSION ANALYSIS

The standard weight-loss method was carried out to find out corrosion rate according to ASTM standard G1-03 (2017). Test samples having 1.5±0.5 cm height and 2.54 cm diameter were given the exposure of 3.5 weight % NaCl solution. Corrosion solution was produced by dissolving 3.5±0.5 grams of NaCl powder into 100 ml demineralised water. Samples were finely ground with the help of 1000 grit emery paper. The four-digit accurate weight pan balance has been used to measure the initial weight of samples. Take three samples for each alloy and consider the average of that value. All the alloy samples were immersed in 3.5% NaCl solution for 3 week and 6 weeks. After 3rd and 6th week, a test sample is taken out from the corrosion media and rinsed with water, cleaned with acetone and air-dried after that final weight was measured and corrosion rate was calculated using the MPY formula.

$$\text{Corrosion Rate (MPY)} = 534/\text{DAT}(1)$$

where, W = Weight loss (gm), D = Density (gm/cc), A = Area (inch²), T = Time (Hrs)

A potentiodynamic study was carried out to find corrosion potential and corrosion current of developed alloy using potentiostatgamry reference 600 according to

ASTM G-5 standard. Test sample having the size of 5 cm height and 1-inch dia. This study is carried out using three-electrode systems in which the working electrode is our test sample (develop alloy), mild steel is a counter electrode and a standard calomel electrode is used as a reference electrode. Environment is 3.5% NaCl for testing and test sample were giving exposure of Environment on 1*1 cm window.

RESULT & DISCUSSION

MICROSTRUCTURE ANALYSIS

The typical Microstructure of pure aluminium and aluminium-containing 2.5 weight per cent Magnesium, 4.5 weight per cent Magnesium are shown in Figure 1(a), Figure 1(b) and Figure 1(C) respectively which was taken by optical microscope and Fig 6, Fig 7 and Figure 2(c) respectively which was taken by scanning electron microscope (SEM) along with Energy Dispersive Spectroscopy (EDS) Analysis.

According to binary phase diagram of Aluminium Magnesium system, in the microstructure two phases will presence one is a solid solution of Aluminium i.e. α aluminium and another is an intermetallic compound of aluminium and magnesium i.e. β phase (Al_3Mg_2) which is form by eutectic reaction. Figure 1(a) shows Microstructure of pure aluminium without the addition of magnesium containing only an aluminium matrix and some impurities like Fe and Si. In Figure 1(b) it is observed α aluminium matrix and presence of Magnesium in a small dark circle and also at the grain boundary which is unreacted Magnesium will remain in only solid solution. In Figure 1(c) presence of unreacted Mg in a small dark circle and also reacted Magnesium presence at grain boundary were observed. As the amount of Magnesium increases the clustering and gathering of Mg and the formation of Al-Mg Intermetallics increases at the grain boundary and microstructure changes from coarse grain to fine grain.

SEM images in backscattered mode at 100X also indicate the presence of magnesium and α - aluminium matrix. EDS analysis confirms the presence of magnesium. The dark black portion shows the presence of magnesium and the grey portion is α - aluminium matrix. Figure 2(a) noted only aluminium matrix and some impurities present in raw material. Figure 2(b) shows the solid solution of aluminium containing magnesium in the form of a dark black circle. Magnesium has negligible solubility at room temperature and below 100°C only 3 wt%, in order that in the case of Al +2.5 wt% Mg alloy free form of Magnesium is observed. As the Magnesium content increases clustering and gathering of an intermetallic compound of Aluminium

and Magnesium are observed at the grain boundary as in the case of alloy Al + 4.5 wt% Mg as shown in Figure 2(c). Morphology of grain boundary changes as the amount of Mg is increases, this was also confirmed from the optical micrograph (Panchal and Rao 2019).

XRD ANALYSIS

The XRD was carried out using Cu as the anode material with a k-alpha wavelength of 1.54060 Å to characterize various phases present in the alloy. The scan range was 10° to 110°. Phase analysis of pure aluminium, Aluminium with 2.5% Magnesium and Aluminium with 4.5% Magnesium shown in Figure 3. The ICDD standard value for aluminium is 04-012-7848. The peaks corresponding to aluminium with describe planes are represented in Table 4. In the case of Pure Aluminium XRD Analysis shows the presence of only the Aluminium phase as described in Figure 3 and in case of Aluminium with 2.5% Magnesium and Aluminium with 4.5% Magnesium also shows the presence of pure aluminium. Here, in both cases, no separate peak for magnesium or aluminium-magnesium intermetallic was not observed. As discussed earlier in the introduction part of this paper, magnesium has maximum solid solubility in aluminium is 14.9 wt% at 451°C.

TABLE 4. Values of 2θ and d-spacing for pure aluminium and aluminum containing 2.5 wight percent Magnesium, 4. °weight percent Magnesium, 6.5 weight percent Magnesium and 8.5 weight percent Magnesium.

Plane	Pure Al		Al+ 2.5% Mg		Al+ 4.5% Mg	
	2θ	d	2θ	d	2θ	D
(1 1 1)	38.4460	2.33959	38.2897	2.34877	38.2499	2.35113
(2 0 0)	44.6903	2.02612	44.5261	2.03321	44.4432	2.03681
(2 2 0)	65.0656	1.43236	64.8209	1.43718	64.7691	1.43820
(3 1 1)	78.1835	1.22160	77.9498	1.22468	77.7216	1.22770

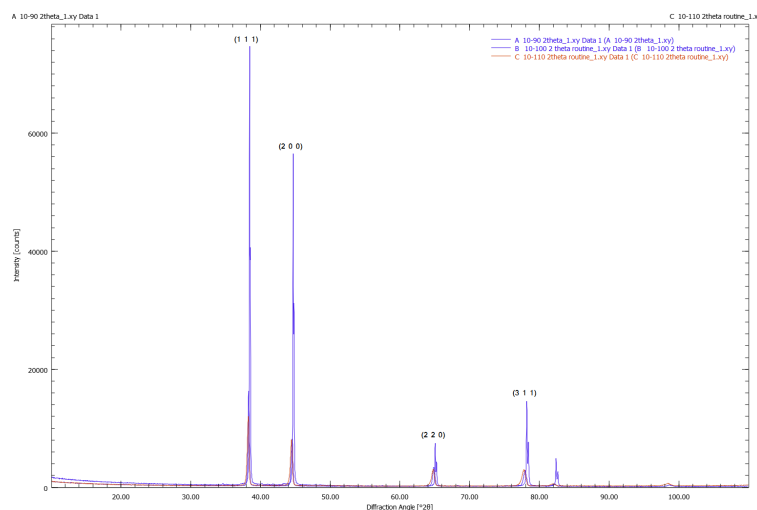


FIGURE 3. Pattern of (A) Pure Aluminium (B) Aluminium with 2.5% Magnesium (C) Aluminium with 4.5% Magnesium

Magnesium is soluble in aluminium so when we are going to add magnesium, it goes into the solid solution of aluminium. That's why we don't observe a peak for magnesium due to the low amount of addition. The peak of all planes shifted towards the left as the Mg content increases and also the area and height of the peak are changed as compared to the pure aluminium pattern which confirms the presence of Mg(Kawasaki et al. 2016; Goel et al. 2014).

CORROSION RATE MEASUREMENT BY THE STANDARD WEIGHT-LOSS METHOD

The standard weight-loss method is carried out to find out corrosion rate according to ASTM standard G1-03 (2017). Samples of each 3 chemistry are given exposure to a 3.5% NaCl solution. Each sample is taken out from the exposure media at the intervals of 3 weeks and 6 weeks and the corrosion rate is measured in MPY (Miles per year). SEM images were also taken after 3rd week and 6th week of immersion to have an idea about Microstructural changes during immersion. The corrosion rate of pure aluminium, Aluminium with 2.5% Magnesium, and Aluminium with 4.5% Magnesium are plotted in Figure 4.

As observed in the plot of corrosion rate versus time is linearly communicated that the corrosion rate of pure aluminium and its alloy with different amounts of magnesium is increased with time of exposure and also, opines that magnesium improves corrosion of aluminium. Magnesium damaged the oxide layer that forms on the surface of the aluminium. Observation confirms as the amount of magnesium increases the corrosion rate is increases. In the case of pure aluminium, the corrosion rate is almost at a steady value range because of the formation of the passive layer. In the case of Aluminium- 4.5% Magnesium corrosion rate is increased as exposure time is increased. This is because of the presence of magnesium. Here, magnesium goes into the solid solution of aluminium and decreases the possibility of passivation and corrosion is observed with time. In the case of Aluminium - 4.5% Magnesium, the corrosion rate is almost thrice compared to pure aluminium. Here, in this case, some magnesium will remain in a solid solution of aluminium and some magnesium react with aluminium and form an intermetallic compound of aluminium and magnesium which was moving towards the grain boundary and near the grain

boundary area, as confirmed from the microstructure. The grain boundary is electrochemically different compared to the alloy microstructure. This causes an electrochemical potential set up between the two and an exchange of electrons takes place. This situation leads to intergranular corrosion. This phase has an electrode potential of -1,150 mV ESC and the aluminium matrix has an electrode potential of -780 mV ESC. While comparing these values Al-Mg intermetallics act as an anode and the Al matrix act as a cathode in a galvanic cell. These lead to localized corrosion or dealloying. Under chemical attack, this anodic phase is dissolved leaving behind cavities or pits. This phase is concentrated at the grain boundaries which restrict the formation of a passive oxide layer. This condition was observed at higher magnesium content because the volume fraction of the intermetallics is higher. This phase acts as an initiation site for corrosion and at this point breaking of the oxide layer is happened. As suggested by several theories, at the breaking point where no oxide layer is present from that area pitting corrosion is started in presence of chlorides which leads to aluminium dissolution (Scully 2003; Bruzzone, Barbuccih, and Cerisolah, n.d.; Barbucci et al., n.d 2000.; Nişancioğlu 2007).

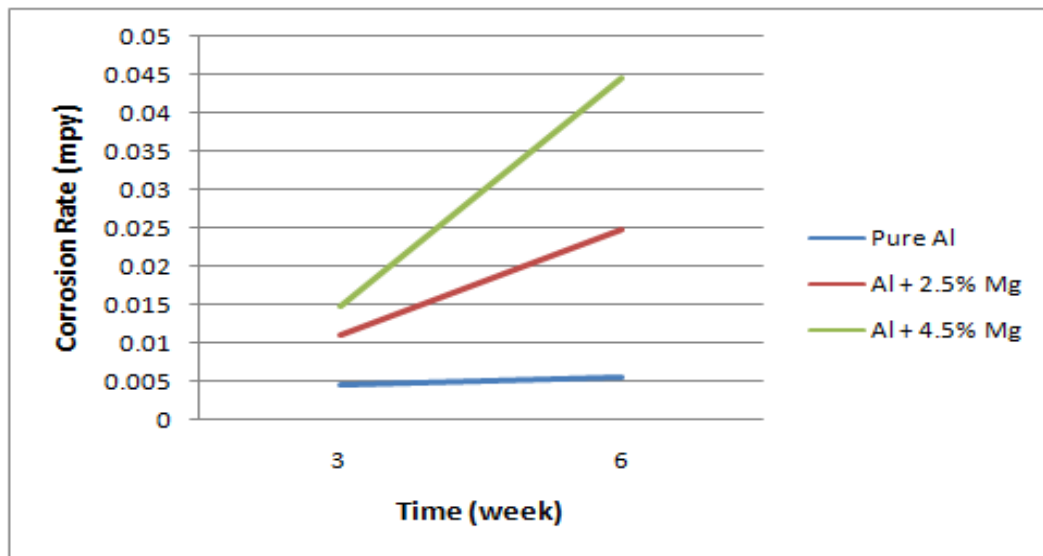


FIGURE 4. Corrosion rate of PureAluminium, Aluminium with 2.5% Magnesium, Aluminium with 4.5% Magnesium

Visual inspection of immersing sample should be carried out after 3 weeks and 6 weeks. As observed in visual inspection in the case of pure aluminium sample surface was dull as exposure time increased. Observation gives an idea about the occurrence of general surface corrosion as shown in Figure 5. In the case of Al+2.5% Mg same things are observed, surface becomes duller and duller as

compared to the as-received sample as shown in Figure 6. In the case of Al+4.5% Mg surface dullness is observed and white scale formation is observed as shown in Figure 7. SEM Analysis should also be carried out after 3 weeks and 6 weeks of immersion which is shown in Figures 8 and 9 respectively. After 3 weeks of immersion, in the case of pure aluminium Figure 8(a), general surface corrosion

is observed and an oxide film is observed which is confirmed by EDS analysis as shown in Figure 10 and also in the case of 2.5% Mg content alloy Figure 8(b) general surface corrosion is observed as the magnesium content increases in alloy, more corrosion is observed in case of 4.5% Magnesium as shown in Figure 8(c). After

6 weeks of immersion, in the case of pure aluminium surface corrosion is observed as shown in Figure 9(a) and in the case of Al+2.5% Magnesium as shown in Figure 9(b) but in the case of Al+2.5% Mg and in Al+4.5% Mg, as shown in Figure 9(c) breaking down or pilling out of oxide film, is observed.



FIGURE 5. Visual Inspection for Pure Aluminium after exposure

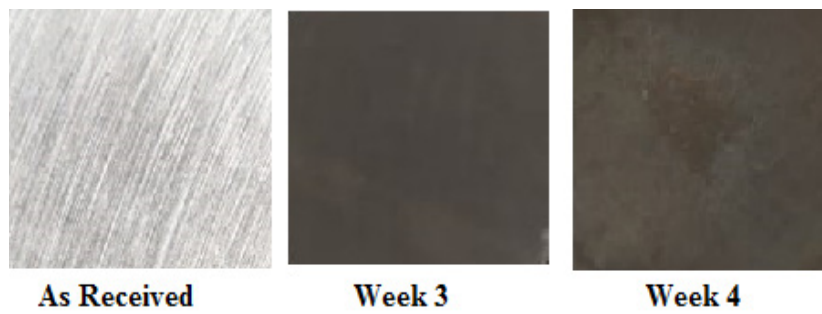


FIGURE 6. Visual Inspection for Al+2.5 %Mg after exposure

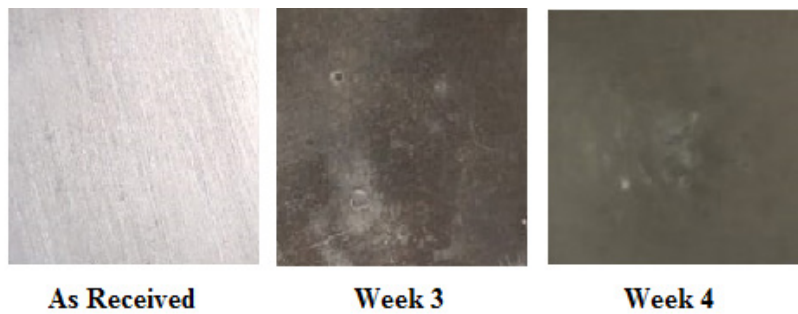


FIGURE 7. Visual Inspection for Al+2.5 %Mg after exposure

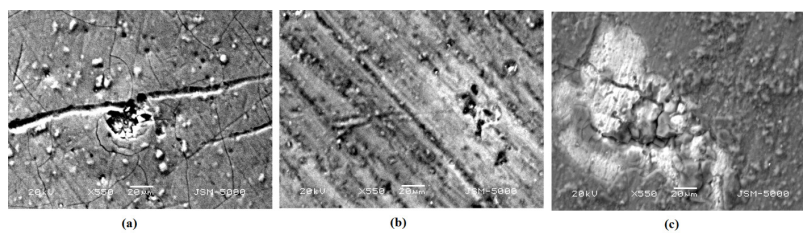


FIGURE 8. SEM images of (a) Pure Aluminium (b) Al+2.5% Mg (c) Al + 4.5% Mg at 550X after 3 weeks of immersion in 3.5% NaCl

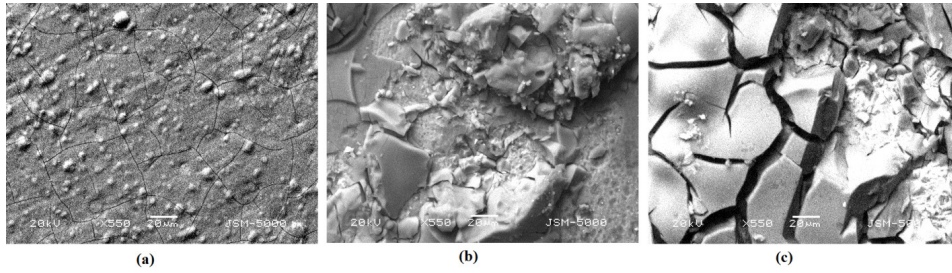


FIGURE 9. SEM images of (a) Pure Aluminium (b) Al+2.5% Mg (c) Al + 4.5% Mg at 550X after 6 weeks of immersion in 3.5% NaCl

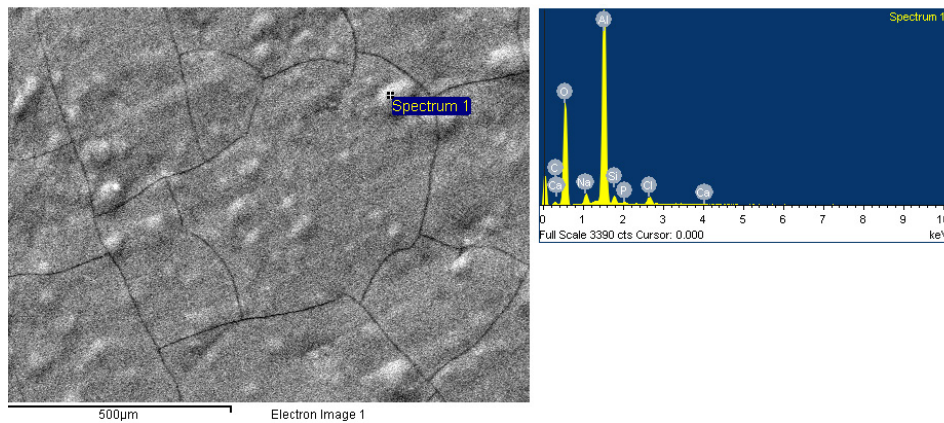


FIGURE 10.EDS analysis of pure aluminium after 6 week of immersion in 3.5% NaCl

POTENTIODYNAMIC STUDY

This test is carried out to find out corrosion potential and corrosion current of pure aluminium, Aluminium with 2.5% Magnesium, and Aluminium with 4.5% Magnesium using potentiostat/gammry reference 600 according to ASTM G-5 standard. Polarization curve of pure aluminium, Al+2.5%Mg and Al+4.5%Mg are recorded in Figure 17 and electrochemical properties of pure aluminium, Al+2.5%Mg and Al+4.5%Mg are listed in Table 5.

A comparison of corrosion behaviour of pure aluminium Al+2.5%Mg and Al+4.5%Mg was done. Polarization curves give the idea about dissolution reaction and reduction reaction. The addition alloying elements having more negative potential compared to pure aluminium typically increases the corrosion potential to

more noble values. Figure 11 shows a potentiodynamic scan of all 3 samples. All the curves show active potential. The corrosion potential of pure Aluminium is -920mV which becomes more negative after the addition of magnesium and the corrosion current of aluminium 14.60 μA is also increased by magnesium addition. As more magnesium is added which is anodic to aluminum passivation curve shifts towards a more negative direction. As discussed earlier above the 3 wt% Magnesium containing aluminium alloy corrosion susceptibility is increased. It was communicated from corrosion potential value above 3 wt% Mg that is aluminium containing 4.5% Mg have -1170 mV. Based on Microstructural examination, it is clearly shows that dissolve or un dissolve magnesium remain present at the grain boundary which is leading to dissolution of aluminium matrix and corrosion rate is improved in case of higher magnesium content.

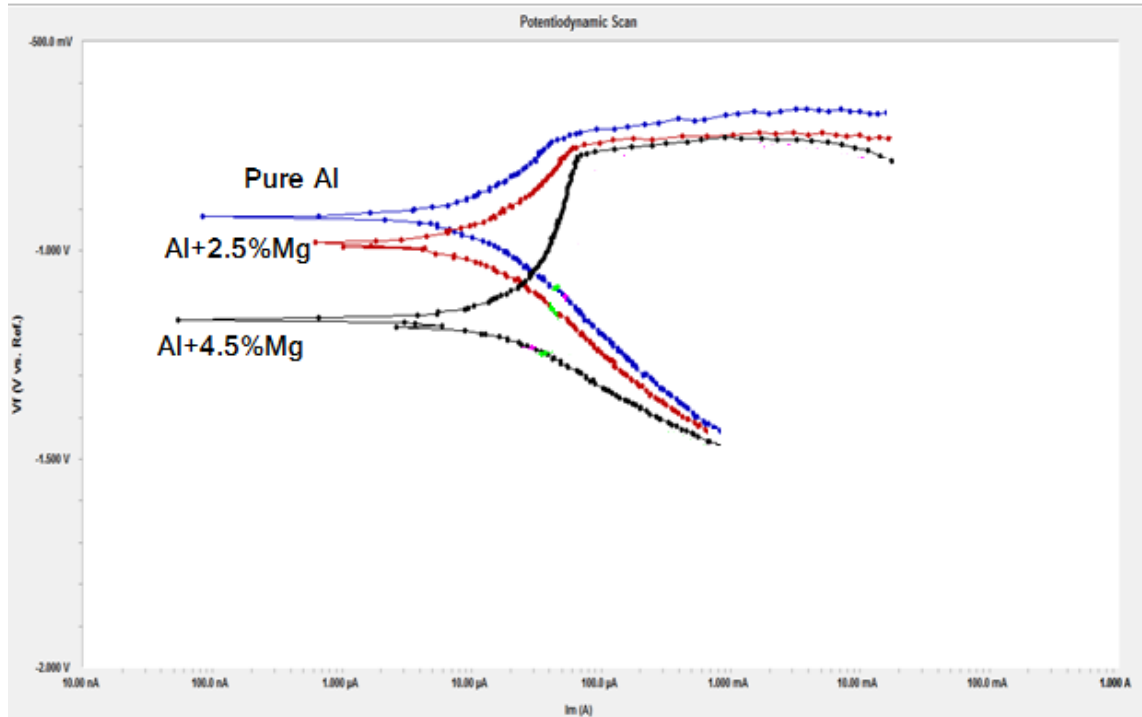


FIGURE 11. Polarization curve of (a) Pure Aluminium (b) Aluminium with 2.5% Magnesium (c) Aluminium with 4.5% Magnesium

TABLE 5. Corrosion properties of Pure Al, Al+2.5%Mg and Al+4.5%Mg

Alloy	E corr (mV)	I corr (μ A)	Corrosion rate (mpy)
Pure Aluminium	-920.00	14.60	25.05
Aluminium + 2.5% Magnesium	-985.00	19.30	33.10
Aluminium + 4.5% Magnesium	-1170	28.20	48.42

CONCLUSION

THE EXPERIMENTAL WORK MADE THE FOLLOWING CONCLUSIONS.

In the presence of a chloride-containing corrosive environment, the protective oxide layer of aluminium loses anti-corrosive properties.

The addition of magnesium modifies microstructure with fine grain and ensure the uniform structure of developed alloy also improve self corrosion.

Magnesium damages the passive layer of aluminium by the formation of the Al-Mg intermetallics which

prevents the formation of a homogeneous passive layer on the surface of the aluminium.

The addition of magnesium reduced corrosion potential by -250mV and shifted to the more active region.

The corrosion current is increased almost two folds by the addition of magnesium and the corrosion rate is almost double compared to pure aluminium.

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DECLARATION OF COMPETING INTEREST

None

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