

Full Length Research Paper

Corrosion inhibition and adsorption behavior of methionine on Mg- Al-Zn alloy

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Corrosion inhibition behavior of Mg-Al-Zn alloy was investigated by different electrochemical techniques in neutral solutions containing chloride ions at different concentrations. The corrosion rate was calculated in the absence and presence of methionine as an environmentally safe corrosion inhibitor using polarization and impedance techniques. The results reveal that methionine inhibits the corrosion reaction by adsorption onto the metallic surface. Polarization results revealed that methionine acts as an anodic inhibitor.

Key words: Mg-Al-Zn alloy, corrosion, impedance, methionine, polarization.

INTRODUCTION

Because of the inherent superior properties, such as low density, outstandingly high strength to weight ratio and excellent electromagnetic shielding characteristic, Mg alloys are now being utilized increasingly as structural materials, especially in the fields where weight reduction is critical or particular technical requirements are needed, including automotive, aeronautic, electronic and military industries, etc. (Zhang et al., 2002; Gray and Luan, 2002; Ambat et al., 2000). But the inferior corrosion resistance primarily attributed to chemistry activity of Mg has been the most severe challenge to the application of Mg alloys to wider realm, especially to aggressive environments (Ambat et al., 2000; Zhang et al., 2002).

The corrosion resistance of magnesium alloys is generally inadequate and has limited their use. Formation of conversion coating is commonly applied to magnesium alloys in order to increase the corrosion resistance. Such processes vary from the widely used chromate treatment to rare earth metal salts dip treatment (Rudd et al., 2000; Yfantis et al., 2002). In recent years, much research has been carried out to search for more environmentally acceptable processes instead of chromate treatment in surface modification process of magnesium alloys.

Natural products extracted from plant sources (Oguzie et al., 2006; Morad et al., 2002), as well as some groups

nontoxic organic compounds, which contain polar with nitrogen, oxygen, and/or sulfur in conjugated systems in their molecules (Moretti et al., 2004; Zhang et al., 2003; Oguzie et al., 2004), have been effectively used as inhibitors in many corrosion systems. The inhibiting action of such compounds is attributed, as a first stage, to the adsorption of the additives on the metallic surface. The roles of the individual functional groups in the inhibition mechanism of organic molecules have been widely reported, but the effect of having two different groups in one molecule, which may influence the corrosion process by different mechanisms, has not been properly documented and is an important consideration in this study. The test inhibitor for the present study is methionine [$\text{CH}_3\text{-S-CH}_2\text{-CH}_2\text{-CH}(\text{CO}_2\text{H})\text{NH}_2$], an amino acid containing both the ($-\text{NH}_2$) and ($-\text{S-CH}_3$) groups in its molecule. Amino acids are attractive as corrosion inhibitors because they are relatively easy to produce with high purity at low cost and are soluble in aqueous media. Ashassi-Sorkhabi et al. (2005) reported the successful application of methionine as a corrosion inhibitor for aluminum in mixed acid solution and pointed out the role of the S atom in increasing the interaction of the molecule with the metal surface. Morad et al. (2002), in their study of the inhibiting effect of methionine on mild steel corrosion in phosphoric acid, observed that adsorption of the compound onto the metal surface obeyed the Frumkin isotherm with an adsorption free energy (ΔG_{ads}^0) value of 25.4 kJ mol^{-1} .

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In the present work the effect of chloride anions on the dissolution of Mg-Al-Zn alloy was studied and methionine (MTI) as naturally occurring amino acid was used as an inhibitor for the corrosion of the alloy in aqueous 0.6 M NaCl at pH 7. The corrosion rate was calculated and the corrosion inhibition process was investigated. For these investigations conventional electrochemical techniques such as potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were used.

EXPERIMENTAL

Materials

The chemical composition of the working electrodes is Mg (91 to 93%), Al (6 to 7%) and Zn (1 to 2%). The electrode consists of massive rods, mounted into glass tubes by two-component epoxy resin leaving a surface area of 0.5 cm² to contact the solution. The cell was a three-electrode all-glass cell, with a platinum counter electrode and saturated calomel reference electrode. Before each experiment, the working electrode was polished mechanically using successive grades of emery papers up to 2000 grit, rubbed with a smooth polishing cloth, then washed with triple distilled water and transferred quickly to the electrolytic cell.

Test medium

Electrochemical measurements were carried out in buffer solutions of pH 7, the solutions were prepared from analytical grade reagents and triple distilled water. The effect of chloride ion concentration was studied in solutions containing various amounts of NaCl (0.1 to 0.6 M) at pH 7. The corrosion inhibition by methionine (Market Harborough, U K), was carried out in solutions containing 0.6 M Cl⁻. The structure of methionine is given in Figure.1.

All experiments were conducted at room temperature (25 ± 1 °C). The test electrolytes were buffer solutions of pH 7 (127.8 ml 0.1 M) COOK₆H₄COOH + (122.2 ml 0.1M) NaOH and completed to 250 ml.)

Electrochemical tests

The electrochemical impedance investigations and polarization measurements were performed using the voltalab 10 PGZ 100 "All-in-one" potentiostat /Galvanostat system. The potentials were measured against and referred to the saturated calomel electrode, SCE (E = 0.245 mV). All potentiodynamic polarization measurements were carried out using a scan rate of 10 mVs⁻¹.

RESULTS AND DISCUSSION

Potentiodynamic polarization measurements

Behavior of Mg-Al-Zn alloy in inhibitor-free aqueous solutions

The electrochemical behavior of Mg-Al-Zn alloy was investigated under polarization conditions, the linear polarization and Tafel extrapolation techniques were

used. Unless otherwise stated the polarization experiments were carried out at a scan rate of 10 mV/s. Corrosion current density (i_{corr}), corrosion potential (E_{corr}) and polarization resistance (R_p) are often used to evaluate the protective property of the films. In addition, E_{corr} and i_{corr} can be calculated from these experiments. Then, based on the approximate linear polarization at the E_{corr} , R_p values are determined by the following relationship.

$$R_p = \beta_a \beta_c / 2.3 i_{corr} (\beta_a + \beta_c) \dots\dots\dots 1$$

The potentiodynamic polarization curves of Mg-Al-Zn alloy after holding the alloy at the open-circuit potential for 90 min in naturally aerated solutions of pH 7 in the chloride free and chloride containing solutions are presented in Figure 2. The corrosion parameter that is corrosion potential, E_{corr} , corrosion current density, i_{corr} , and the corrosion rate, of the alloy in chloride-free and chloride-containing naturally aerated stagnant aqueous solutions of pH 7 were calculated and presented in Table 1. No passivity was observed in any of the chloride containing solutions (Figure 2). At low chloride ion concentration, ([Cl⁻] ≤ 0.2 M), the cathodic reaction was more affected than the anodic reaction. At higher chloride ion concentration the rates of both cathodic and anodic reaction were accelerated. The interesting point of the anodic and cathodic curve (E_{corr}) is that they did not show a significant shift with increase of chloride ion concentration up to 0.4M, but for 0.5 and 0.6 M NaCl a large negative shift (towards active side) was observed.

Figure 3 shows the corrosion current density, i_{corr} and the polarization resistance, R_p as a function of chloride ion concentration for Mg alloy which showed a 10 fold increase in current density and decrease the polarization resistance for an increase in chloride ion concentration from 0.1 to 0.6 M. The increase in corrosion rate with increasing chloride ions concentration may be attributed to the participation of chloride ions in the dissolution reaction. Chloride ions are aggressive for both magnesium and aluminium. The adsorption of chloride ions on oxide covered magnesium surface transforms Mg(OH)₂ easily to soluble MgCl₂ (Ambat et al., 2000).

In agreement with the above observations, most of the polarization curves exhibited an increase in anodic current density with increase in chloride ion concentration (Figure 2). Cathodic curve also shifted to higher current density values with increase in chloride ion concentration. The shift of OCP to more negative values with increase in chloride ion (Table 1) concentration may be explained by the adsorption of these ions on the alloy surface at weak regions of the oxide film (Frumkin et al., 1961).

When unalloyed magnesium is exposed to the atmosphere or aqueous solutions, a grey oxide (mainly magnesium hydroxide, brucite) forms on its surface, which is stable in basic solutions (Shaw BA., 2003). Nevertheless, in presence of chloride anions, this surface

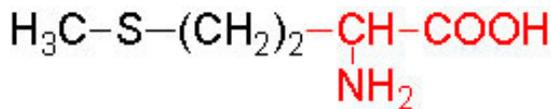


Figure 1. Structure of methionine.

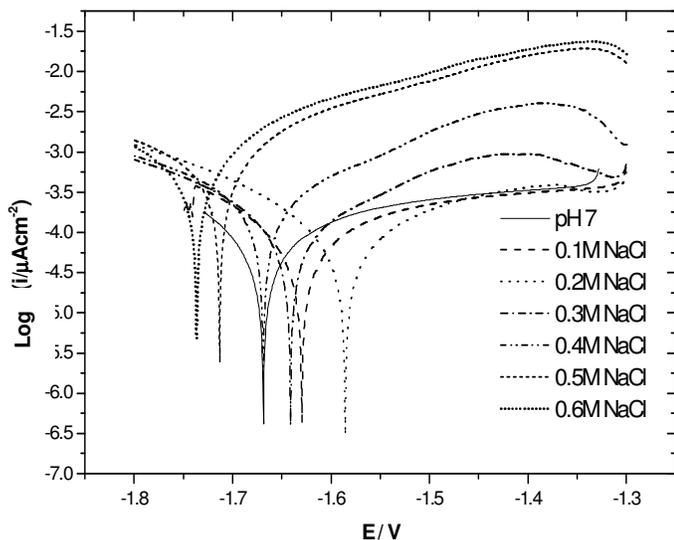
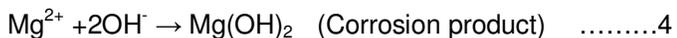


Figure 2. Potentiodynamic polarization curves of Mg-Al-Zn alloy after 90 min of electrode immersion in chloride-free and chloride-containing aqueous solutions at pH 7.

film breaks down and magnesium appears unprotected. Although corrosion mechanism of magnesium needs further investigation, it is generally reported that the following anodic and cathodic reactions take place:



Firstly, magnesium dissolves and $\text{Mg}^{2+}(\text{aq})$ cations are produced possibly through intermediate steps involving monovalent magnesium ion (Makar et al., 1990; Hoey and Cohen, 1958). Secondly, magnesium dissolution is accompanied by hydrogen evolution, since magnesium in acidic and neutral solutions is well below the region of water stability. Finally, pH raises along with the cathodic reaction due to the formation of OH^- , which favours the formation of $\text{Mg}(\text{OH})_2(\text{s})$ according to Pourbaix diagram (Van Muylder et al., 1966). Thus, the overall reaction could be expressed as



And the adsorption of chloride ions to oxide or hydroxide covered magnesium surface transforms $\text{Mg}(\text{OH})_2$ to easily soluble MgCl_2 via the following reaction (Ambat et

al., 2000)



Behavior of Mg-Al-Zn alloy in inhibitor containing aqueous solutions

Polarization experiments were undertaken specifically to separate the different effects of methionine, MTI, on the anodic and cathodic partial reactions of the corrosion process. Figure 4 illustrates typical anodic and cathodic polarization curves of Mg-Al-Zn alloy in 0.6 M NaCl with and without methionine. The corrosion parameter that is corrosion potential, E_{corr} , corrosion current density, i_{corr} , and the corrosion resistance, R_{corr} , of Mg-Al-Zn alloy electrode in methionine-free and methionine-containing naturally aerated stagnant neutral 0.6 M NaCl aqueous solutions were calculated from the potentiodynamic polarization curves and presented in Table 2. The corrosion potential, E_{corr} is shifted to the positive direction in the presence of methionine, which indicates inhibition of the anodic process of the corrosion reaction. This implies that MTI functions herein as an anodic type inhibitor.

The plots also show that MTI at the same time exerted a stimulating effect on the cathodic reaction. Also, MTI caused no pronounced change in the anodic and cathodic Tafel slopes, suggesting that the inhibitor adsorbs onto the metal surface and retards corrosion by blocking the active sites without altering the anodic and cathodic reaction mechanisms. It is clear from the potentiodynamic data that the presence of the amino acid in the solution decreases the corrosion rates, that is, the value of i_{corr} decreases. The inhibition efficiency, η , was calculated from the values of the corrosion current density without inhibitor, i_{corr} , and its value in the presence of the inhibitor, $i_{\text{corr}}(\text{inh})$, according to

$$\eta = (i_{\text{corr}} - i_{\text{corr}}(\text{inh})) / i_{\text{corr}} \times 100 \quad \dots\dots\dots 6$$

Generally with increasing inhibitor concentration, the corrosion current density and corrosion rate decrease and polarization resistance increases (Table 2) from 0.5 to 5 mM of MTI but with the increase > 5 mM of MTI concentration the inhibition efficiency decreases. In neutral solution, MTI can be protonated at the amine group, even though the presence of $\text{S}-\text{CH}_3$ decreases the stability of the positive charge. The inhibitor could interact with the corroding alloy surface via the protonated amino groups or via the S atom in the aliphatic chain which can be adsorbed at anodic sites and retard Mg electrodisolution.

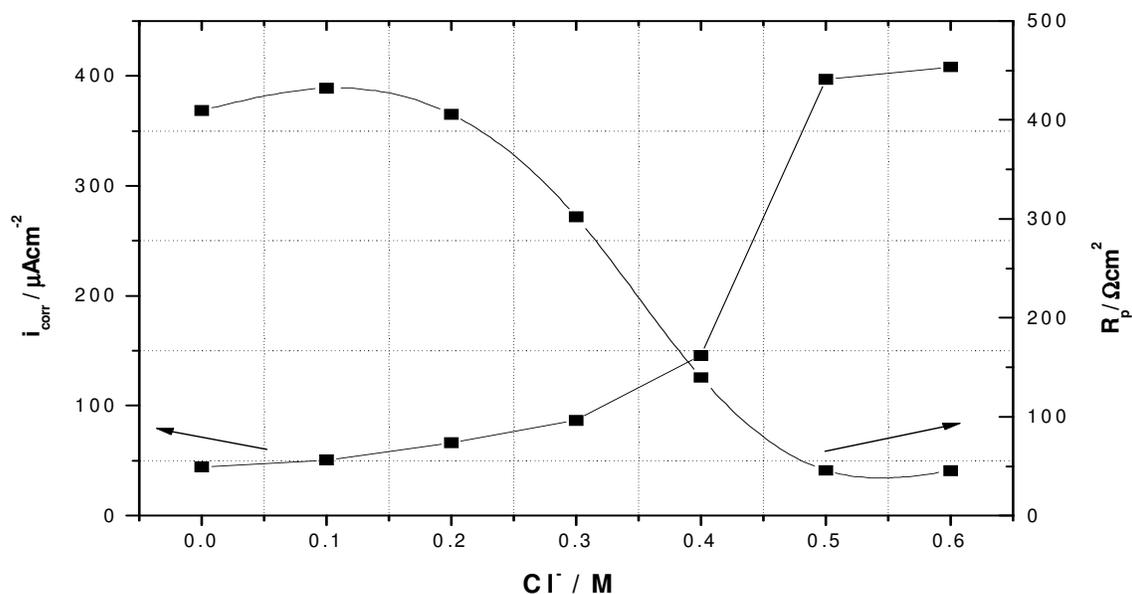
The sulfur containing amino acids can be adsorbed as bidentate ligands in which surface coordination is taking place through both the amino group (or carboxylic group) and the $-\text{S}-$ moiety. The compound thus has the ability to influence both the anodic partial reactions, giving rise

Table 1. The corrosion parameter of Mg-Al-Zn alloy in buffer solution containing different concentration of chlorides.

Cl ⁻ / M	E _{corr} / mV	i _{corr} / μAcm ⁻²	R _p / Ωcm ²	B _a / mV	B _c / mV	Corrosion rate/mmy ⁻¹
0.0	-1668	44	410	149	-71	1.0
0.1	-1629	62	401	195	-130	1.4
0.2	-1585	72	400	203	-140	1.6
0.3	-1640	76	288	156	-119	1.7
0.4	-1668	153	137	131	-138	2.5
0.5	-1712	496	46	112	-191	9.2
0.6	-1737	516	46	116	-173	11.8

Table 2. The corrosion parameter of Mg-Al-Zn alloy in buffer of pH 7 and 0.6 M NaCl solution containing different concentration of methonine (MTI).

MTI concentration / mM L ⁻¹	E _{corr} / mV	i _{corr} / μAcm ⁻²	R _p / Ωcm ²	η %
0.0	-1737	516	46
0.5	-1716	450	40	13
1.0	-1700	155	80	70
5.0	-1697	124	93	75
10.0	-1708	370	55	28

**Figure 3.** Variation of corrosion current density i_{corr} and polarization resistance of Mg-Al-Zn alloy in stagnant naturally aerated buffer solution (pH 7.0) containing different concentration of chloride at 25°C.

to the recorded anodic inhibition mechanism. The decreasing of the inhibition at high concentration (10 mM) may be due to some steric interaction from coordination of sulfur atom that the substituted methyl group may exert.

The electrochemical impedance measurements:

The results of the potentiodynamic polarization experiments were confirmed electrochemical impedance spectroscopy (EIS), where EIS is a powerful technique in

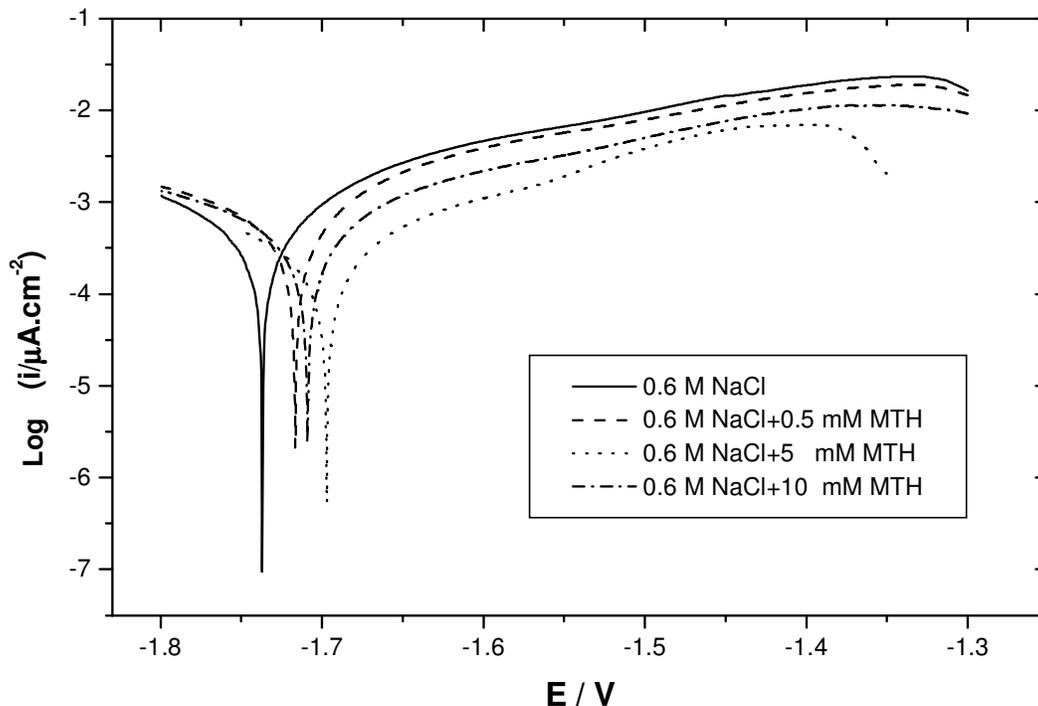


Figure 4. Potentiodynamic polarization curves of Mg-Al-Zn alloy after 90 min of electrode immersion in methonine free and methonine containing 0.6 M NaCl aqueous solutions at pH 7.

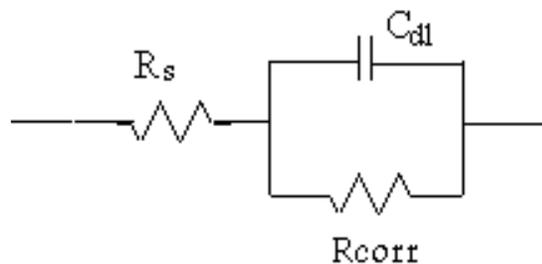


Figure 5. Equivalent circuit used in fitting impedance data.

studying corrosion mechanisms and adsorption phenomena (Macdonald et al., 1987). The experimental impedance results are simulated to pure electronic models that can verify the mechanistic models and enables the calculation of numerical values corresponding to the physical and/or chemical properties of the electrochemical system under investigation (Badawy et al., 1999; Hladky et al., 1980). The impedance data were analyzed using software provided with the electrochemical workstation, where the dispersion formula was used. A simple equivalent circuit model consisting of a parallel combination of a capacitance, C_{dl} , and resistor, R_{corr} , representing the corrosion resistance of the electrode, in series to a resistor, R_s , representing the solution resistance, is shown in Figure 5.

The electrode impedance, Z , for such a circuit is given by:

$$Z = R_s + [R_{corr} / 1 + (2\pi f R_{corr} C_{dl})^2]^{-1/2} \quad \dots\dots\dots 7$$

The effect of chloride ions concentration on the corrosion behavior of Mg-Al-Zn alloy in aqueous neutral solutions at pH 7 was investigated. The Bode plots for Mg-Al-Zn alloy in chloride-free and chloride-containing aqueous solutions of pH 7 are presented in Figure 6a. The diagrams show resistive regions at high and low frequencies and capacitive contribution at intermediate frequencies. The low frequency loop was related to the charge transfer resistance and the double-layer capacitance of the electrode, while the high frequency one was attributed to the presence of a partially protective surface layer of MgO or Mg(OH)₂. This feature can be observed in the Nyquist representation and the phase angle (Figure 6b).

As shown in Figure 6a, the impedance ($|Z|$) of Mg alloy is dependant on the concentration of the chloride ions. The increase in the concentration of Cl⁻ in the solution decreases the $|Z|$ value, while the phase shift (Θ)_{max} increases with a concomitant shift to higher frequencies (Figure 6b). This trend is most likely due to an increase in the surface film capacitance with an increase in the adsorbed amount of the Cl⁻ ions on the electrode surface. The impedance of Mg alloy show two well capacitive loops at low concentration (0.1 to 0.3 M NaCl) while at high concentration (>0.3 M) the second loop is depressed

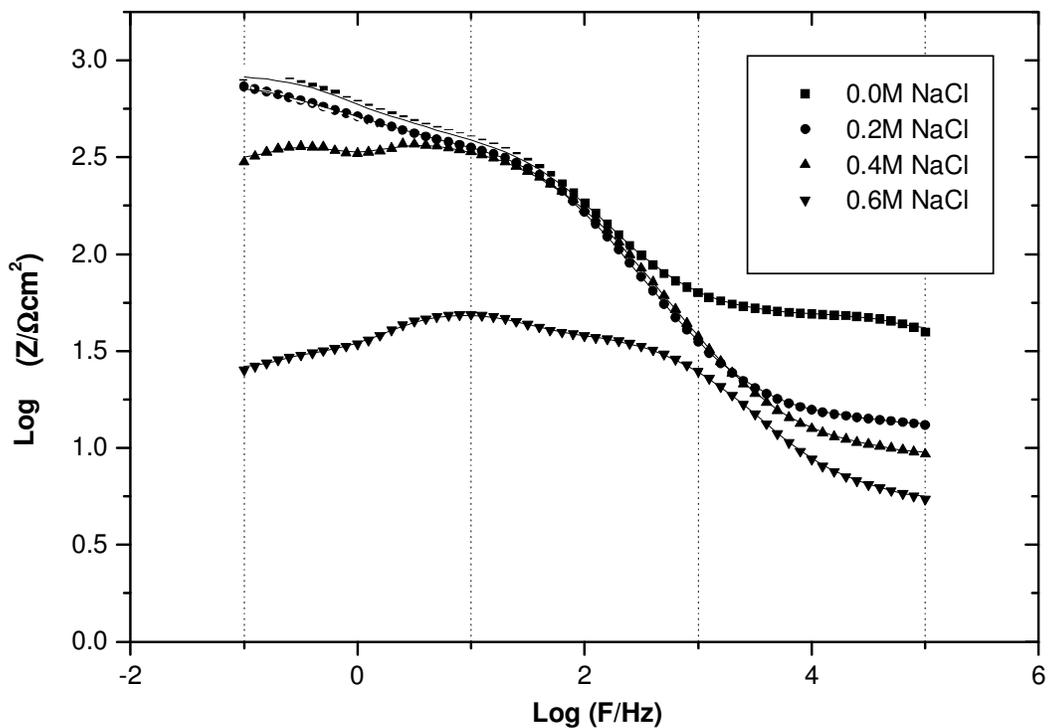


Figure 6a. Bode plots for Mg-Al-Zn alloy after 90 min of the electrode immersion in neutral aqueous solutions containing different concentration of chloride at 25°C.

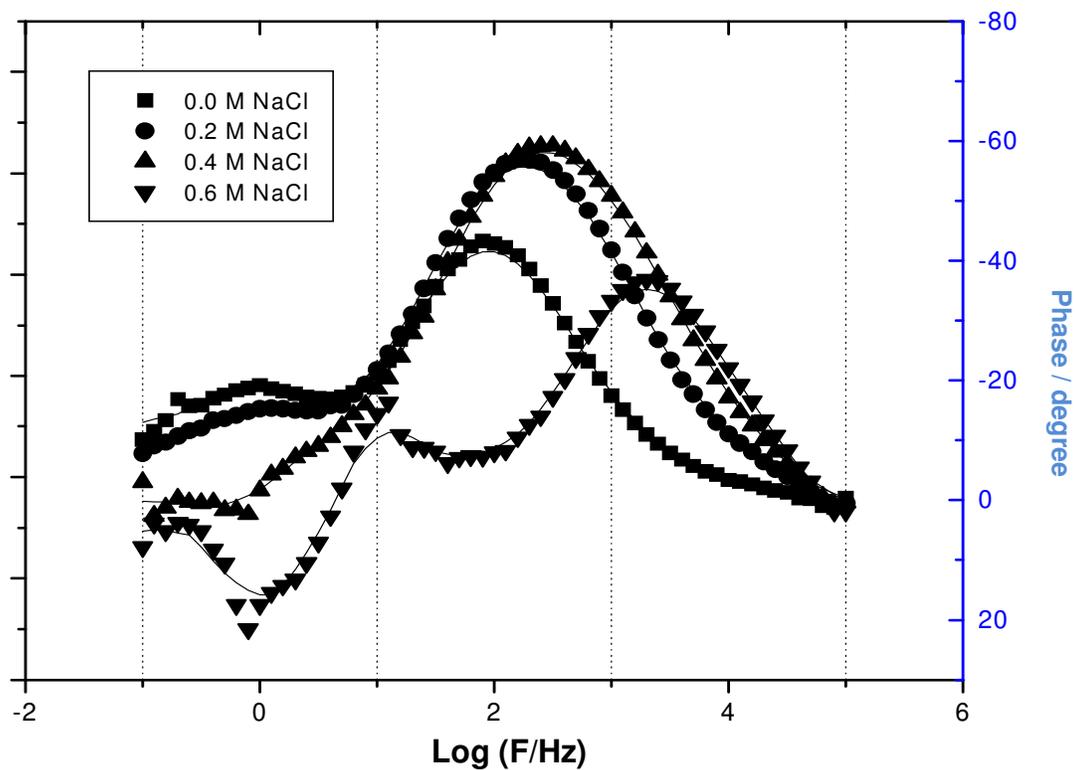


Figure 6b. Phase plots for Mg-Al-Zn alloy after 90 min of the electrode immersion in aqueous solutions containing different concentration of chloride at 25°C of pH 7.

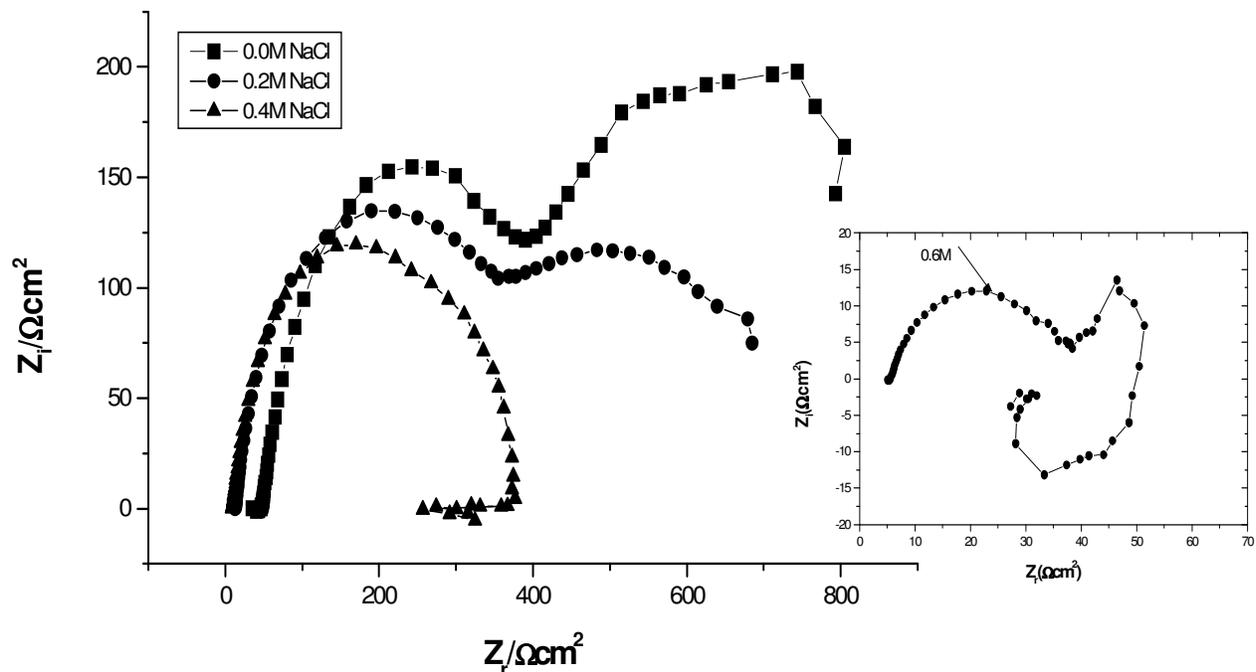


Figure 6c. Nyquist plots for Mg-Al-Zn alloy after 90 min of the electrode immersion in aqueous solutions containing different concentrations chloride at 25°C of pH 7.

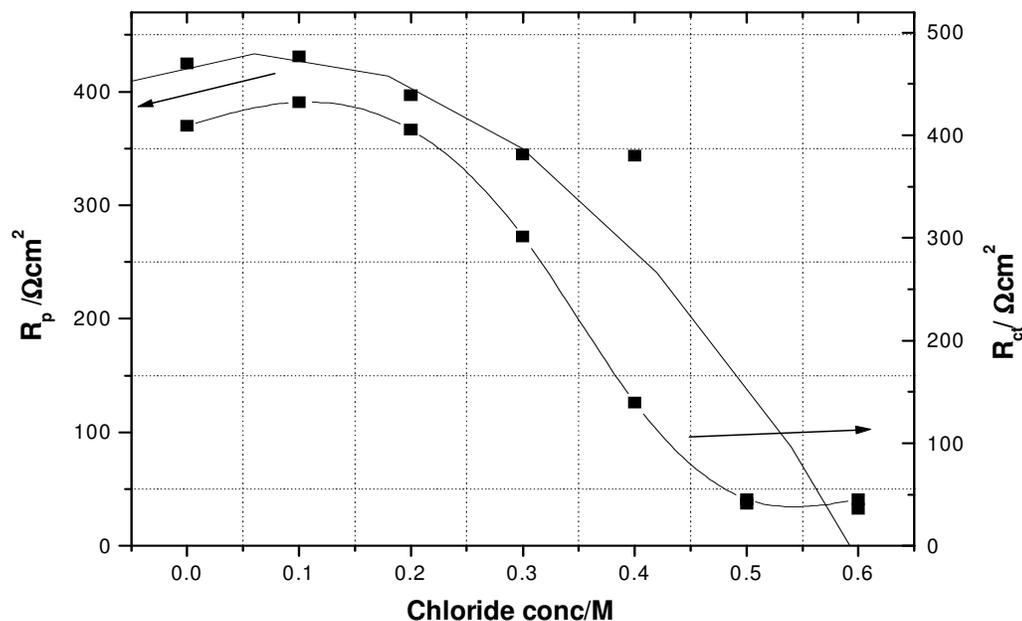


Figure 7. Variation of charge transfer resistance and polarization resistance of Mg-Al-Zn alloy in stagnant naturally aerated buffer solution (pH 7.0), containing different concentration of chloride at 25°C.

and shows a loop at higher frequency. This feature is associated to the presence of an inductor in an electrical equivalent circuit. This inductor is due to adsorption and desorption phenomena occurring on the surface of the sample (Kim and Kim, 2000; Greenblatt JH., 1956). The

Bode representation of the impedance does not show a clear sharp maximum (Figure 6a), therefore, the presence of two time constants (associated to two capacitors) was assumed.

Figure 7 shows the calculated values for the charge

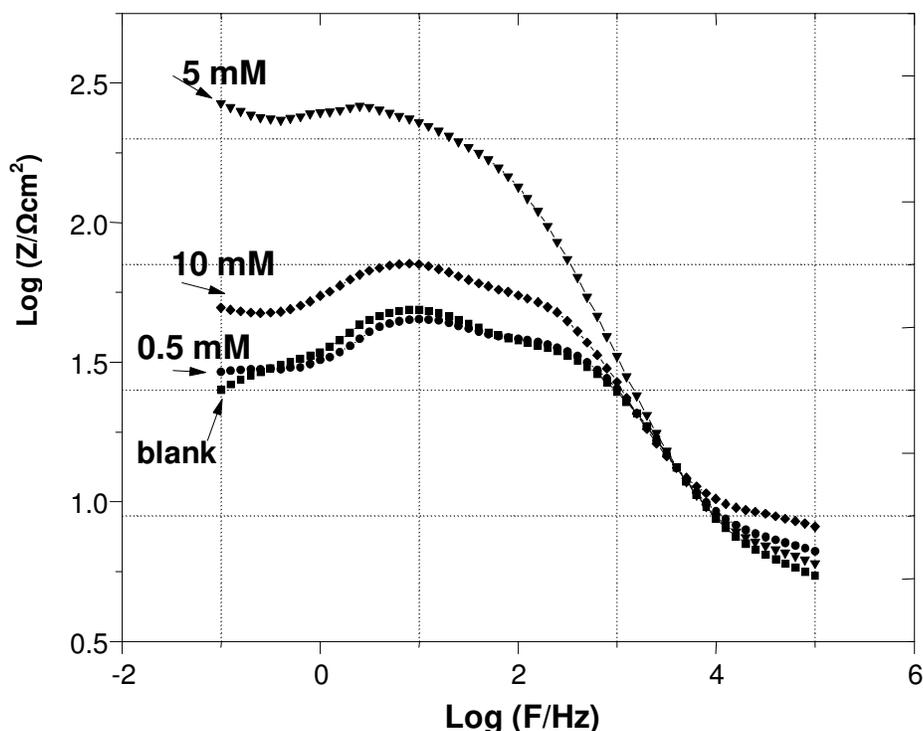


Figure 8a. Bode impedance plots of Mg-Al-Zn alloy after 90 min of electrode immersion in methionine free and methionine containing 0.6 M NaCl aqueous solutions at pH 7.

transfer resistance (R_{ct}) obtained from the impedance measurements and polarization resistance (R_p) which were obtained from polarization measurements with different concentration of chloride ions. The figure indicates that the resistance of Mg alloy to corrosion decreases with the increase of chloride ion concentration and the impedance measurements are inline with the polarization measurement.

Measurements were undertaken to assess the impedance parameters of the alloy surface/electrolyte interface in the presence of different concentrations of MTI. Representative examples of the impedance spectra are given in Figure 8, panels a, and b, which exemplify, respectively, the Bode, and phase angle plots obtained for Mg-Al-Zn alloy in 0.6 M NaCl solution in the absence and presence of 0.5 to 10 mM MTI. The spectra obtained with inhibitor consist of two depressed capacitive loops corresponding to two time constants. The high-frequency part of the impedance and phase angle describe the behavior of an inhomogeneous surface layer, whereas the low-frequency component depicts the kinetic response for the charge transfer reaction (Khaled and Hackerman, 2003). It is observed that increasing the concentration of MTI results in an increase in the impedance of the interface in (Figure 8a), and in the maximum phase angle (Figure 8b), which indicates inhibition of the corrosion process.

The values of the electrochemical impedance parameters derived from the Nyquist plots are given in Table 3. It is observed that introduction of MTI into the chloride containing solution leads to an increase in the charge transfer resistances, which become more pronounced as the MTI concentration is increased. This implies that an increase in MTI concentration, correspondingly, reduces the corrosion rate of the Mg-Al-Zn alloy by virtue of adsorption onto the metal/electrolyte interface, thereby protecting the alloy from the attack of the corrodent.

SEM techniques

Figures 9a, b and c present the macroscopic surface appearance of the corroded samples after 30 min immersion period in stagnant naturally aerated solution of pH 7 with and without chloride and methionine. There was no obvious corrosion for the samples exposed to 0 M NaCl pH 7 solution indicating that the formed film provided resistance to corrosion initiation. The whole surface was homogeneously covered by a layer of corrosion products, indicating that the corrosion was uniform. The amount of visible corrosion qualitatively increased in the presence of chloride ion at pH 7 (Figure 9b). Figure 9c shows the Mg alloy surface after immersion in 0.6M NaCl in the presence of methionine (5 mM). The addition of

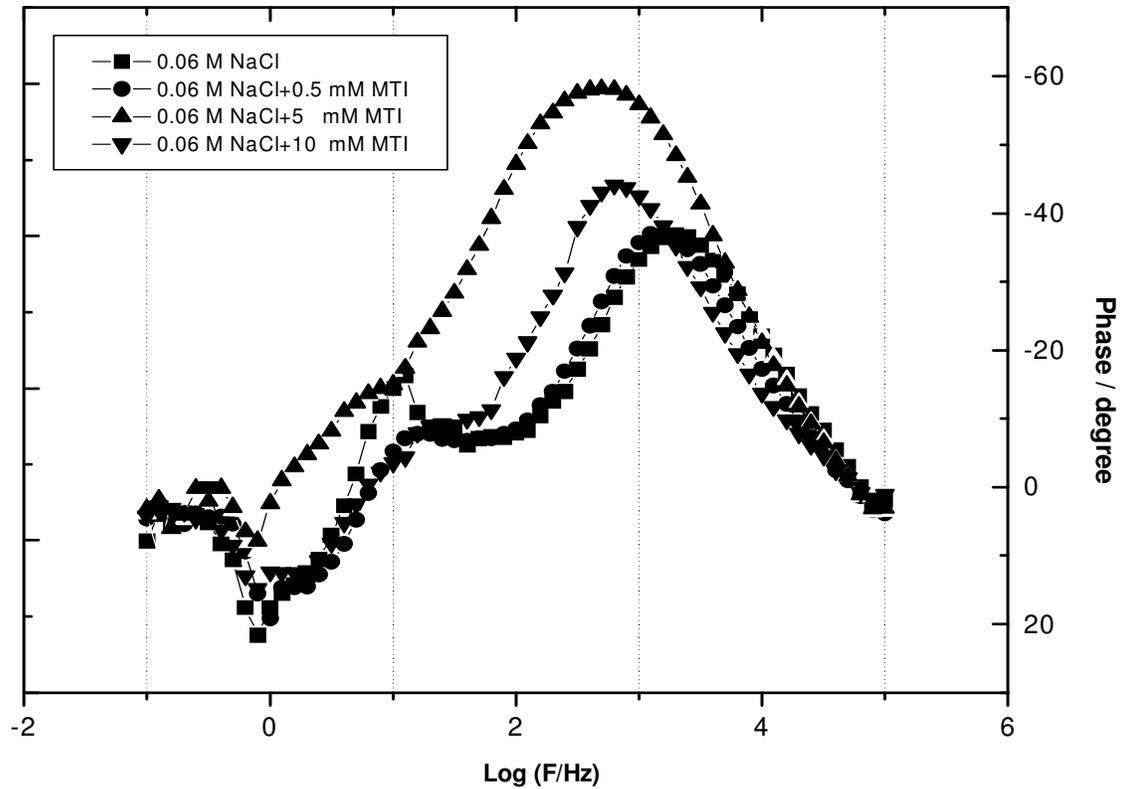
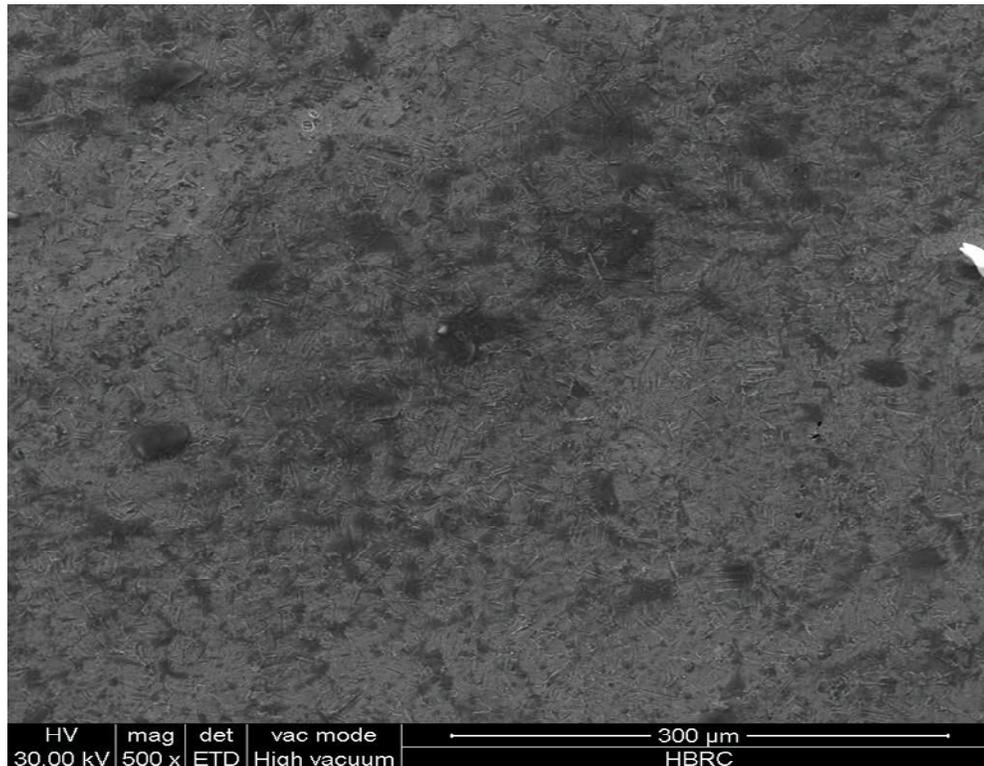


Figure 8b. Phase plots for Mg-Al-Zn alloy after 90 min of electrode immersion in 0.6 M NaCl solution containing different concentrations of methionine.

a



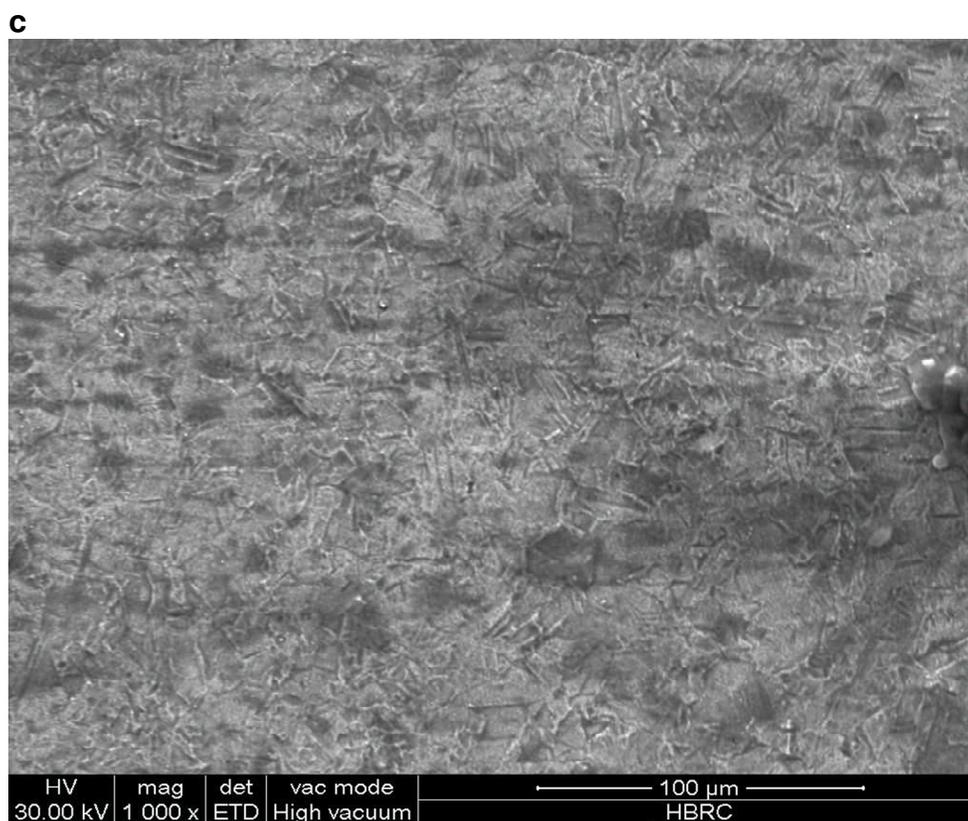
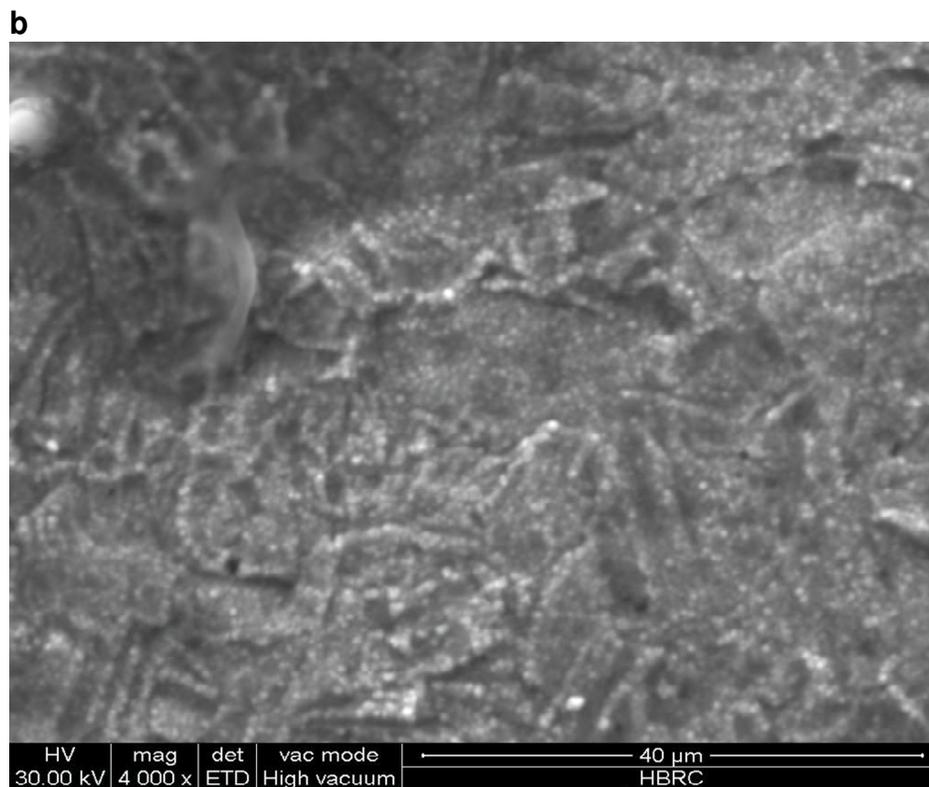


Figure 9. Surface of the corroded samples after immersion in pH 7 buffer solution containing. (a) No NaCl; (b) 0.6 M NaCl; (c) 0.6 M NaCl and 5 mM methionine.

Table 3. The electrochemical impedance parameters of Mg-Al-Zn alloy in buffer of pH 7 and 0.6 M NaCl solution containing different concentration of methionine (MTI).

MTI concentration / mM L ⁻¹	R _s / Ωcm ²	R _{corr} / Ωcm ²	C _{dl} / μFcm ²
0.0	6	33	7.5
0.5	7	34	7.4
1.0	8	214	14.8
5.0	5	255	12.4
10.0	9	54	11.7

methionine increase the covered surface and decreases dissolution of magnesium.

Conclusion

The corrosion of Mg-Al-Zn alloy in solutions depends on the chloride ion concentration. The corrosion behavior is consistent with the current understanding that the corrosion behaviour of magnesium alloys is governed by a partially protective surface film, with the corrosion reactions occurring predominantly at the imperfections of the partially protective film. The implication is that the fraction of film-free surface increases with increasing chloride ion concentration. This is consistent with the known tendency of chloride ions to cause film breakdown.

Methionine functions as an inhibitor of Mg-Al-Zn alloy corrosion in chloride-containing aqueous solutions. An anodic inhibition mechanism is proposed. The corrosion inhibition efficiency increases with the concentration of the inhibitor and is related to the concentration and chemical structure of the amino acid.

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