A Study of Corrosion Protection of Aluminium Alloy Al1050 On Coating with Polyaniline/phosphate Layer

By

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Abstract

Corrosion protection of aluminium alloy Al1050 on coating with polyaniline/phosphate was studied in a 0.5 M $NaClO_4$ aqueous solutions. This was performed using potentiodynamic, potentiostatic galvanostatic, cyclic, Tafel polarization and potential-time measurements. It was found that, the coating of the alloy with polyaniline/phosphate shows a remarkable corrosion potential shift to noble values. This is due to the formation of compact polyaniline/phosphate film. The corrosion rate and consequently the rate of pit nucleation decrease on the coating. This is interpreted on the basis that polyaniline in the emeraldine form (EB) stabilizes the open circuit potential in the passive region. The observed stabilization is tentatively attributed to the weak EB electrochemical activity combined with its low ion permeability providing a decrease of the hydrogen and oxygen reduction rates, and consequently improves corrosion resistance. After a period of time the substrate reaches the corrosion potential region, and the emeraldine oxidation state is converted to the leucoemeraldine (LE) form and the protection is terminated. The evidence f

EB to LE transformation is confirmed by probing the change of colour from green to

yellow-green during the corrosion.

Keywords: Aluminium alloy, polyaniline, potentiodynamic, potentiostatic,

galvanostatic, cyclic polarization, Tafel polarization, corrosion protection.

Introduction

Protecting reactive metals like aluminium by covering their surface with an organic coating is a good way of taking advantage of the mechanical properties of the metals while protecting them from corrosion. Recently, utilization of intrinsically conducting polymers as organic coating has received growing interest[1-15]. Among these polymers, polyaniline has been shown to have certain advantage over other conducting polymers including simplicity and rapidity of preparation by electrochemical methods, chemical durability against aerial oxidation and the ability to be formed in aqueous solutions[12,14]. However, application of these new coatings, in spite of their good capability on corrosion protection of metals, is subjected to some limitations. In particular, the irreversible consumption of the charge stored in the polymer coating during redox reactions which leads to a significant loss of the protective properties of the polymer coating, constitutes a major impediment. To overcoming this limitation, fabrication of conducting polymer coatings in the presence of different dopants was undertaken. It was found that doping with phosphate in the presence of phosphoric acid is excellent for corrosion protection[7,16]. Phosphate was used to produce a smooth, adhere[15] and very uniform polymer thin film[8]. While there is plenty of data on pitting corrosion of Al in different environments, especially those containing Chloride ions[17-22], relatively little information is available concerning perchlorate ions[23,24]. Cecchetto et al, have concluded that polyaniline film in the emeraldine base (EB) form have a good protective behaviour against corrosion of aluminium[12].

In view of these limitation and the high demand for facile and efficient method to protect Al we offer in this contribution a detailed study of the corrosion behaviour of a commercially available aluminum alloy (Al1050) along with the application

polyaniline/phosphate coating for its protection. Due to the lack of information concerning the passivity of Al alloy in ClO_4^- solution, the herein, study of corrosion resistance of subject aluminum alloy coated with polyaniline/phosphate layer in neutral 0.5 M $NaClO_4$ solution was investigated. This has been achieved via wide range of electrochemical methods such as potentiodynamic, potentiostatic, galvanostatic, cyclic, Tafel polarization and open circuit potential-time.

Experimental

All the chemicals used are of Analytical Reagent, AR, grade chemicals. Aniline is purified by vacuum distillation over zinc dust in order to eliminate the oxidation impurities. The purified aniline is always stored in dark. Disodium hydrogen phosphate, sodium dihydrogen phosphate, sodium hydroxide and sodium perchlorate (Merck) are used.

Aluminum alloy is commercial with 99.5% Al, specified as Al1050 according to the ASTM-Standard[25]. It is obtained from Alumisr Company, Egypt. Its chemical composition is reported in Table 1. Specimens of 10.0x50.0x1.0 mm are cut from the aluminum coupons. Specimens are masked with adhesive tape to get an effective working area of 3.3 cm^2 at one of its ends. The specimen is mechanically polished successively to a smooth surface finish, using a fine (0.5 µm) grades of SiC paper and washed with deionised water. The specimens of similar surface conditions and areas are used throughout.

A stagnant and naturally aerated aqueous solution with a volume of about 200 ml of the solution is used for each electrochemical experiment. All electrochemical experiments are performed at room temperature in a one-compartment polypropylene cell containing three electrodes. Pretreated Al1050 alloy as working electrode, two platinum foils as counter electrodes and saturated calomel electrode (*SCE*)

reference electrode are used. The working and counter electrodes are arranged parallel to one another to ensure uniform current distribution.

Aqueous 0.50 M phosphate buffer (pH 5.0) solution is prepared by dissolving the appropriate amount of disodium hydrogen phosphate and sodium dihydrogen phosphate in deionised water. A 0.20 M solution of aniline is freshly prepared in the aqueous phosphate buffer solution. A 0.50 M solution of sodium perchlorate, $NaClO_4$, (the corrosion agent) is freshly prepared by dissolving the appropriate amount in deionised water.

A Potentiostat/Galvanostat (EG&G model 263A) and a Personal Computer are used. M352 Corrosion and M270 Electrochemical software (EG&G Princeton Applied Research) are used for all electrochemical experiments.

Commercial aluminum alloy Al1050 is coated with polyaniline/phosphate layer potentiostatically at a constant applied potential of 1.55 V versus *SCE* in a used three-electrode cell from the prepared aqueous solution of aniline/phosphate for a period of 30 minutes at room temperature and pH 5.0. After the aluminum polyaniline/phosphate modified electrode is formed, the building solution is removed from the cell and the modified electrode is rinsed several times with deionised water.

Corrosion Studies

1-Potentiodynamic anodic polarization studies

The potentiodynamic anodic polarization curves are recorded on scanning the electrode potential, including compensation for the ohmic drop potential of the solution, automatically from −1.15 (more negative than the corrosion potential) up to 0.20 volt versus *SCE* with scan rate of 1.0 mVs^{-1}. The anodic polarization curves are recorded as log current density (log *j*) against potential (*E*), after allowing t

electrode to reach the corrosion potential (E_{corr}) for a period of 1 h immersed in a 0.5 M NaClO$_4$ solution at room temperature (298 K).

2-Potentiostatic (current/time transient) studies

The potentiostatic measurements are carried out at a given step anodic potential, $E_{s,a}$, of (-0.05 V > (E_{pit} = -0.31 V). The current/transients are recorded in 0.50 M sodium perchlorate solution.

3-Galvanostatic (potential/time transient) studies

In galvanostatic potential/time transients, a constant anodic current density of 10 μA cm^{-2} is applied and the variation in potential is recorded as a function of time in 0.50 M sodium perchlorate solution.

4-Cyclic polarization studies

Cyclic polarization measurements are carried out by sweeping the potential of -1000 mV from the corrosion potential, E_{corr} towards the positive direction until the potential is approximately +200 mV from E_{corr} at a given scan rate of 1.0 mVs^{-1}, then it is reversed till the starting potential, giving one complete cycle in 0.50 M NaClO$_4$ solution.

5-Tafel polarization studies

A Tafel plot is performed on polarizing the potential about 300 mV anodically and cathodically from the corrosion potential, E_{corr}, at scan rate of 1.0 mVs^{-1} in 0.50 M sodium perchlorate solution. The resulting current is plotted on a logarithmic scale.

6-Open circuit potentials as functions of time Studies

Open circuit potentials as functions of time (from the moment of immersion until their steady state values) are followed to examine the characteristics of the potential-time curves under open circuit conditions.

Results and Discussion

Potentiodynamic anodic polarization studies

Potentiodynamic anodic polarization curve for aluminum alloy, Al1050, coated with polyaniline/phosphate film is recorded in 0.50 M $NaClO_4$ electrolyte at 298 K, as depicted in Fig. 1. Potential sweep is started from the corrosion potential towards the anodic direction in order to avoid the reduction of perchlorate ions to Cl^- ions[26]. It is revealed that, the electrode moves into the active region on increasing potential to more positive values as shown in Fig. 1. In this region, corrosion of aluminum is the dominant reaction taken place. As the potential is further increased, the manifestation of passivity persists at a potential of -0.69 V vs. *SCE*, known as the passivation potential, E_p. In the passive region, current is insensitive to potential increase. Once the potential reaches a sufficiently positive value, E_{pit} = -0.12 V vs. *SCE* (the pitting potential), a rapid increase in the current density is observed. The sudden increase in current is ascribed to pitting (localized breakdown of passivity). The polarization behaviour of the coated electrode shows a remarkable potential shift to the noble values than that for the uncoated one. Therefore, the polyaniline/phosphate coating seems to be successfully protects the aluminum alloy from pitting corrosion in $NaClO_4$ solution.

Potentiostatic (current/time transient) studies

Potentiostatic (current/time) transient measurements for uncoated and polyaniline/phosphate coated aluminum alloy electrodes are carried out in 0.50 M sodium perchlorate solution at step potential, $E_{s,a}$, of -0.05 V vs. *SCE*, more noble than the pitting potentials of the two subject electrodes, -0.31 and -0.12 V vs. *SCE*, respectively. At this potential the two electrodes are exposed to the pitting corrosion.

From these data, the current density initially decreases to a minimum value yielding a corresponding characteristic pitting time parameter, namely the incubation time (t_i). After incubation time is attained, the current starts to increase. The increase in current suggests that, the pit growth is the dominant process and a number of well-developed pits could be observed following this active period, i.e., the incubation time.

Relevant information about the pitting growth of subject electrodes can be derived from the analysis of the rising part of the current transients. On plotting the pit growth current density, j_{pit}, against the square root of time, straight regression lines are obtained, c.f. Fig. 2. Thus, the growth current density depends linearly on the square root of time. This observation can be taken as an indication that, the pit growth is an instantaneous three-dimensional growth under diffusion control[27]. These results agree well with Hills mode[28], which can be ascribed by equation 1:

$$j_{pit} = A t^{1/2} \qquad (1)$$

The slope A of the j_{pit} versus $t^{1/2}$ relationship can be taken as a measure of the rate of pit growth. The incubation time, t_i, before pit growth to occur can be calculating from the intercept of equation (1). Moreover, the rate of pit nucleation (i.e. the number of events per unit time) is defined by $(1/t_i)$[27].

Comparing the estimated values for the coated electrode with that for the uncoated one, reveals that, the coating with polyaniline/phosphate film decreases the rate of pit nucleation than that for the uncoated one, $(3.4\pm0.1)\ 10^{-2}$ and $(4.2\pm0.1)\ 10^{-2}$ s^{-1}, for coated and uncoated electrodes, respectively. On the other hand, the incubation time for the coated electrode is higher than that for the uncoated one, 29.2 ± 0.9 versus 23.6 ± 0.8 seconds for the two electrodes, respectively. Thus, it is concluded that, the coating decreases the rate of pit nucleation indicating low active sites available for pit nucleation.

Galvanostatic (potential/time transient) studies

To study the growth kinetics of the protecting film formed on the polyaniline/phosphate coated aluminum alloy Al1050 and its breakdown, the electrode is anodized galvanostatically in 0.50 M $NaClO_4$ solution at constant anodic current density of $j_a = 10\ \mu A\ cm^{-2}$. The results obtained are shown in Fig. 3. On looking at these curves, it is seen that, immediately after switching the anodic current density, the anodic potential rises sharply and almost linearly reaching a maximum at a certain critical pitting (or breakdown) potential, E_{pit}, and at a characteristic pitting time parameter, namely the incubation time t_i[29]. They are estimated to be -50.0 ± 1.3 mV vs. *SCE* and 26.0 ± 0.8 seconds, respectively. The sharp rise of the anodic potential with time is related to thickening and growth of the protective layer on the electrode surface. The slope of the linear rise in potential $(dE/dt)_{ja}$ represents the rate of passive layer growth[30]. It is calculating to be 11.0 ± 0.3 mVs^{-1}. After incubation time, t_i, the potential decreases with time as a result of pit growth. The rate of pit growth is calculated from the linear decrease in potential with time. It is found to be 8.0 ± 0.3. Finally, after a period of time of ≈ 35 seconds, the potential reaches a steady state value at almost -0.52 V vs. *SCE*, it corresponding to the repassivation potential, E

Under a galvanostatic conditions, E_{rp} is the only potential at which the active area remains constant and independent of time. The steady-state potential is attributed to the stabilization of the repassivation process.

From these data it is revealed that, the pitting potential (E_{pit}), and the repassivation potential for the polyaniline/phosphate coated subject electrode has a more noble value than that of the uncoated one, -50 and -250 mV vs. *SCE*. On the other hand, the incubation time for the coated electrode is higher than that for the uncoated one, 26.0 ± 0.8 and 15.0 ± 0.4 seconds for the two electrodes, respectively. Moreover, the pit nucleation growth rate for the polyaniline/phosphate coated electrode is lower than that for the uncoated one, $(3.70\pm0.05)\ 10^{-2}$ and $(4.4\pm0.1)\ 10^{-2}$ s^{-1}, respectively. This confirms that, passivation is elevated on coating.

Cyclic polarization studies

Cyclic potentiodynamic anodic polarization scans are generated for uncoated and polyaniline/phosphate coated aluminum alloy Al1050 in 0.50 M NaClO$_4$ electrolyte. The obtained graphs are recorded in Fig. 4. Tracing the current hysteresis loopes for the subject electrodes indicates that, at -1.05 V vs. *SCE*, on anodic potential scanning, the uncoated electrode receives pitting showing a rapid increase in the current density till it reaches the passivation region. The electrode retains its passivity up to the pitting potential, E_{pit}, of -0.26 V vs. *SCE*, then the current rises on further sweep. These donate pit initiation and continue to rapid increase. On reversing the scanning direction, the current density remains constant, then decreases and reaches a plateau at repassivation potential value of E_{pro} = -0.71 V vs. *SCE*.

On coating the surface of the subject electrode with polyaniline/phosphate film, the anodic cyclic polarization graph is shifted to noble values. Table 2 shows the current and potential data for the uncoated and coated electrodes. It is clear that, t

coated electrode has nobler pitting (E_{pit}) and repassivation potentials (E_{rp}) and has smaller repassivation hysteresis loop than the uncoated one. This suggested that the electrodes have different pit morphologies, populations, and size and/or pit passivation rates. Accordingly, the pit corrosion resistance of the coated alloy appears to be notably higher than of the uncoated one.

Tafel polarization studies

Potentiodynamic polarization responses, Tafel curves, for the uncoated and the polyaniline/phosphate coated aluminum alloy Al1050 electrodes are recorded in 0.50 M NaClO$_4$ solution. Fig. 5 shows the obtained Tafel responses. From these data, it is revealed that, the corrosion potential of the coated substrate, E_{corr} = -0.683 V vs. *SCE*, is shifted 367 mV more noble to that of the uncoated one, E_{corr} = -1.05 V vs. *SCE*. Moreover, the corrosion current for the uncoated subject electrode, j_{corr}, of 430 mμA cm^{-2}, decreases on coating with polyaniline/phosphate, 260 mμA cm^{-2}. The potentiodynamogram for the coated substrate shows very low values of anodic and cathodic currents compared to that of the uncoated one. Both a significant anodic shift of the corrosion potential and a decrease of the corrosion current are due to formation of protective layers. It can be suggested that the protective layer proceeds through passivating reaction of polyaniline with a metal surface where it acts as a redox catalyst and as a noble metal with respect to a metal[31].

Table 3 comprises the corrosion Tafel data for the two subject electrodes. Analysis of these data shows that, the corrosion rate of the aluminum alloy is significantly reduced as a result of the reduction in the I_{corr}. It is important to note that the cathodic current density of the polyaniline/phosphate coated aluminum is significantly lower than that of the uncoated one. This observation suggests that the polyaniline coating is strongly adhering and it provides nearly perfect coverage of t

aluminium alloy surface. The measured cathodic current density may be due to the reduction of the polyaniline and thus, it is contributed by the polyaniline itself. Consequently, the coating restricts the anodic and cathodic reactions in aggressive media; this decreases the susceptibility to the pitting corrosion[32].

The protection efficiency (PE) for polyaniline/phosphate is calculated by using the following expression:

$$PE\% = [(R_{pc} - R_p)/R_{pc}] \text{ x } 100 \qquad (2)$$

where R_p and R_{pc} denote the polarization resistance of uncoated aluminum alloy and polyaniline/phosphate coated one, respectively. The *PE* calculated from potentiodynamic polarization data is found to be ~99.2%. Thus, these results reveal the capability of polyaniline to act as a corrosion protective layer on aluminum alloy.

Potential-Time Studies

The corrosion potential E_{corr} values measured for the uncoated and polyaniline/phosphate coated aluminum Al1050 alloy on varying immersion time are plotted against time (min) in 0.50 M NaClO$_4$ solution. From the obtained data, the corrosion potential, E_{corr}, of the uncoated alloy decreases sharply attaining a steady state at -1.05 V vs. *SCE*. On the other hand, for the coated electrode, E_{corr} decreases steadily to the steady state at -0.683 V vs. *SCE*, nobler than that for the uncoated one. Furthermore, the coated electrode takes longer time than the uncoated one, $\approx$ 80 and 8 minutes, respectively to attain the steady state. Thus, the polyaniline/phosphate layer offers protection, i.e. E_{corr} is stabilized in the passive region. This could only be explained via the passivation of the subject substrate on coating.

On the whole, our data and the literature allow us to suggest that, for the conditions under investigation, the stabilization of the open circuit potential for the subject coated aluminum alloy is due to the barrier effect of emeraldine base (EB),

semi-oxidised state, the most stable, and the only conductive form of polyaniline/phosphate in neutral environment[33-37]. The weak EB electrochemical activity combined with its low ion permeability provides a decrease of the hydrogen and oxygen reduction rates, and improves corrosion resistance. After a period of time the substrate reaches the corrosion potential region, and the emeraldine oxidation state is converted to the leucoemeraldine (LE) form and the protection is terminated. The evidence for EB to LE transformation is confirmed from the change of colour from green to yellow-green observed during the corrosion potential measurement.

Conclusion

Aluminum alloy Al1050 was efficiently protected from corrosion on potentiostatically coating with adhesive layer of polyaniline/phosphate. This is revealed from the remarkable corrosion potential shift of the coated aluminum to noble values relative to the uncoated one. Furthermore, the corrosion rate and consequently the rate of pit nucleation decrease on coating. This is tentatively attributed to stabilization of the open circuit potential in the passive region by polyaniline in the emeraldine form (EB). The weak EB electrochemical activity combined with its low ion permeability providing a decrease of the hydrogen and oxygen reduction rates, and consequently improves corrosion resistance.

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Legend of Figures:

Fig. 1: Experimental potentiodynamic anodic polarization curves of the polyaniline/phosphate coated and the uncoated aluminum alloy Al1050 samples in a 0.50 M NaClO$_4$ at 298 K.

Fig. 2: Dependence of the pit growth current density, j_{pit}, on $t^{1/2}$ for uncoated and coated aluminum alloy in 0.50 M NaClO$_4$ solution at 298 K and at step potential of $E_{s,a}$ = -0.05 V vs. *SCE*.

Fig. 3: Galvanostatic potential/time transients recorded for uncoated and coated aluminum alloy Al1050 in 0.5 M NaClO$_4$ solution at constant current density of j_a = 10 µA cm^{-2}.

Fig. 4: Cyclic potentiodynamic polarization for uncoated and modified aluminum alloy Al1050 in 0.50 M NaClO$_4$ solution at scan rate of 1.0 mVs^{-1}.

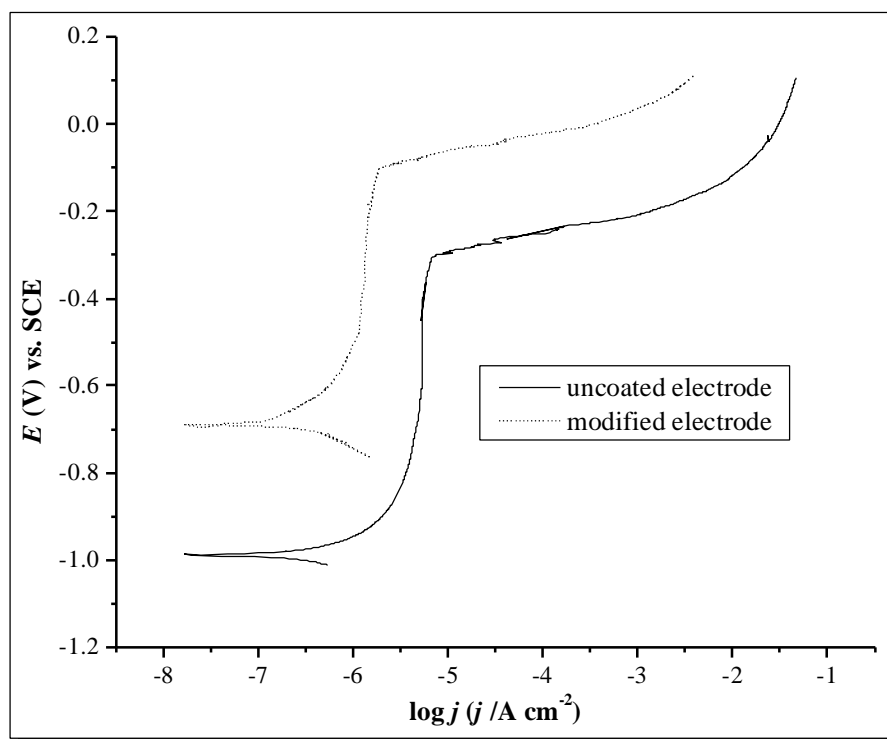**Fig. 5:** Tafel curves obtained for uncoated aluminum alloy Al1050 and modified electrodes in 0.50 M NaClO$_4$ solution at 298 K.

Fig. 1

Fig. 2.
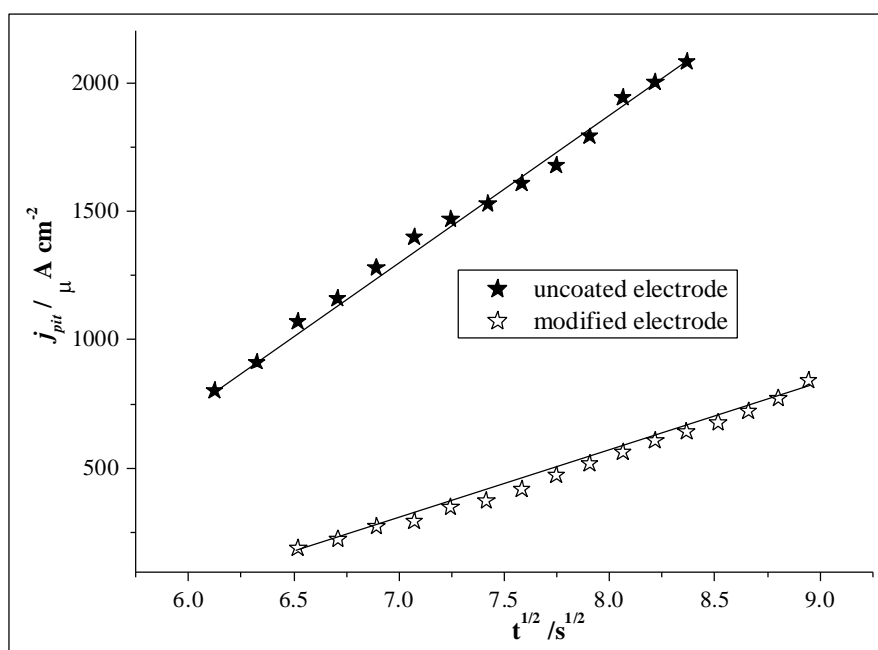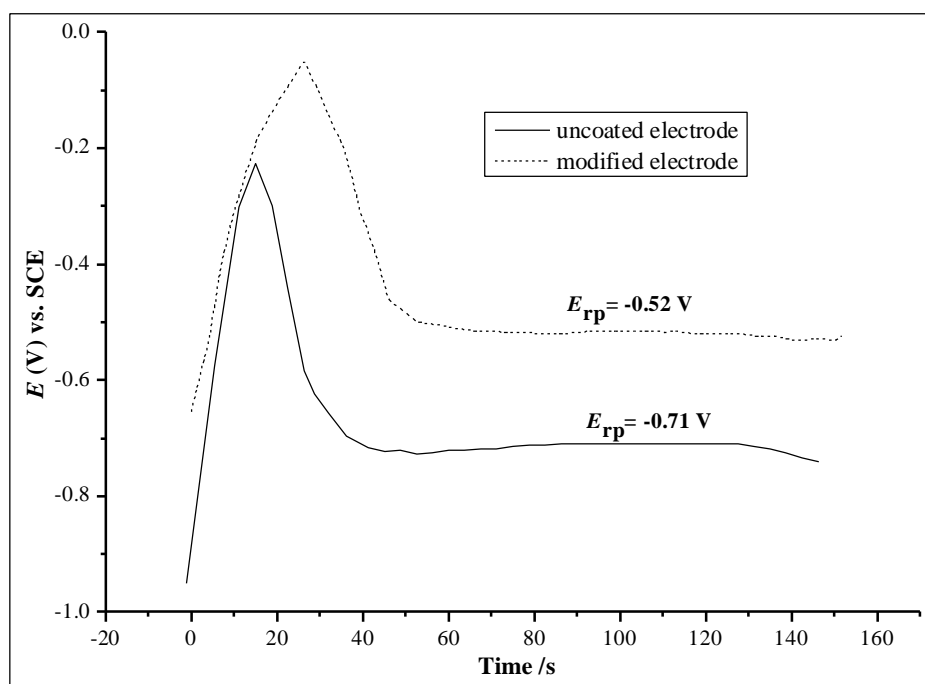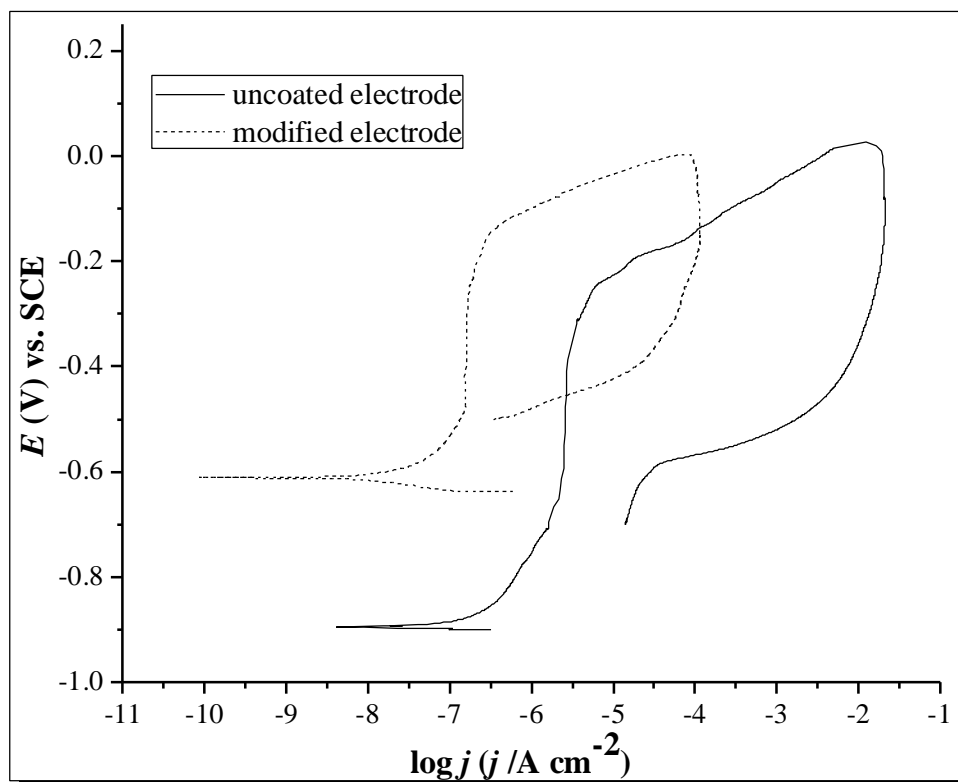
Fig. 3.

Fig. 4.

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Fig. 5.

Table1 : Chemical composition (wt %) of aluminum alloy Al1050
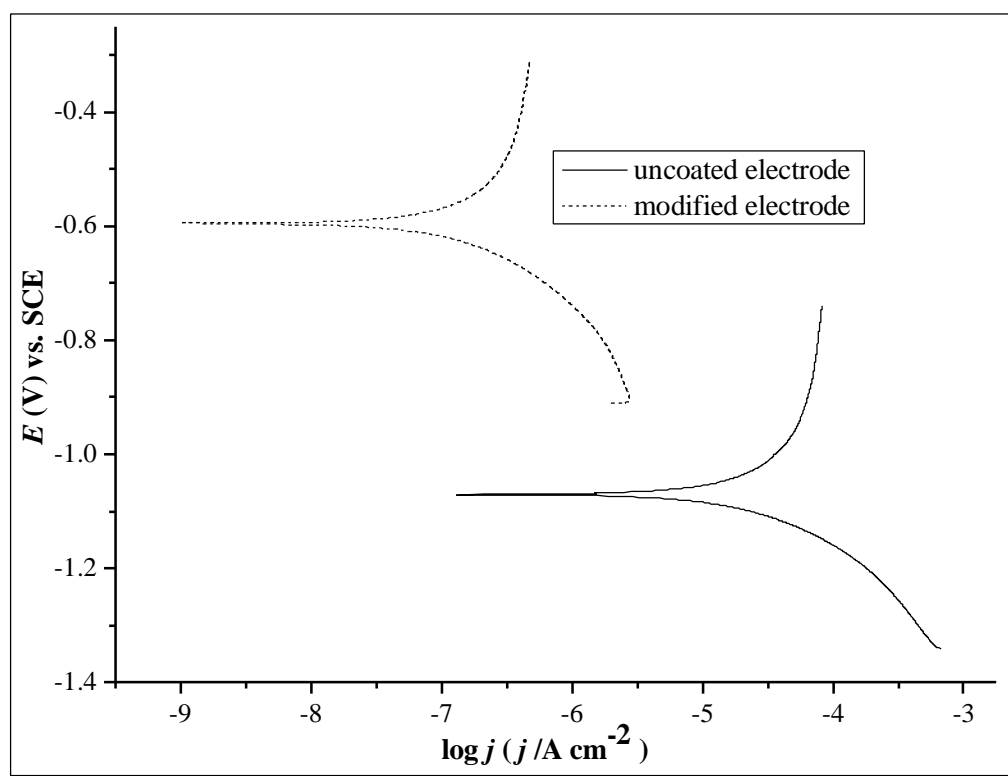
Al1050	Si	Fe	Mg	Mn	Cr	Cu	Ni	Ti	V	Zn	Others	Al
composition (wt.%)	*0.25	*0.40	*0.05	*0.05	–	*0.05	–	*0.03	*0.05	*0.05	0.03	Min 99.50

*Max

Table 2. Cyclic potentiodynamic polarization data for uncoated and modified aluminum alloy

Al1050 in 0.50 M $NaClO_4$ solution at scan rate of 1.0 mVs^{-1}.

Parameters	uncoated electrode	modified electrode
E_{corr} (V vs. SCE)	-1.05	-0.69
E_{pit} (V vs. SCE)	-0.26	-0.11
j_{pass} (A cm^{-2})	2.5×10^{-6}	1.6×10^{-7}
j_{max} (A cm^{-2})	0.016	3.16×10^{-5}
E_{pro} (V vs. SCE)	-0.71	-0.51

Table 3. Corrosion Tafel data obtained for uncoated and coated aluminum alloy Al1050 in

0.5M $NaClO_4$ solution.

	uncoated electrode	coateed electrode
$-E_{corr}$, (V vs. SCE)	1.05	0.683
I_{corr}, (μA cm^{-2})	43.00	0.240
Corrosion rate, (μm year^{-1})	477.52	2.79
b_a, (V/decade)	0.70	0.380
b_c, (V/decade)	0.10	0.090