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Effect of Titanium Carbide Content on the Corrosion Behavior of Al-TiC Composites Processed by High Energy Ball Mill

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In this study, three aluminum-titanium carbide (Al-TiC) composites having the chemical composition of Al-15%TiC, Al-25%TiC, and Al-35%TiC were manufactured. The powders of the composites were ball milled in situ using a high energy ball mill and melted in a high frequency induction heat furnace before being consolidated at 1300 ºC. The effect of increasing the TiC content on the corrosion of the fabricated composites in 3.5% NaCl solutions was investigated using open-circuit potential, electrochemical impedance spectroscopy, cyclic polarization, and current-time at constant potential techniques. The surface of the corroded composites was examined using scanning electron microscopy. All results were consistent with each other confirming that the resistance of Al composites against corrosion remarkably increased with increasing the TiC content in the composite. This occurred via lowering the corrosion current, corrosion rate, and the potentiostatic current with time at -100 mV vs. Ag/AgCl, as well as enhancing the polarization resistance with increasing the weight percent of TiC in the manufactured Al composite.

Keywords: Al-TiC composites; ball milling; chloride solutions; corrosion; EIS; polarization

1. INTRODUCTION

The corrosion and corrosion protection of aluminum and its alloys have attracted the attention of numerous investigators [1-7]. This is because they have been significantly employed in industry, like in aircrafts, vehicles, furniture, containers, buildings, alkaline batteries, sacrificial anodes in cathodic protection systems, household appliances, packages, and many other applications [8-12]. All these uses come from the excellent properties of these materials, where it has good appearance and machinability, high corrosion resistance in atmosphere, high strength to weight ratio, etc [13-16]. The high resitance against corrosion for aluminum in air has been reported [5,6,12] to be due to the ability of aluminum in reacting with air to form an aluminum oxide film, which is more immune to be being attacked by most corrosive solutions than the fresh surface of the aluminum metal.

The corrosion resistance of aluminum can possibly increase by several modification methods. This can be done by modifying the surrounding environment with corrosion inhibitors, adding alloying elements to the aluminum, anodizing the surface, and/or painting the surface of aluminum with a protective coating [5,6,12,17]. Protecting aluminum against corrosion via reinforcing it with more noble alloying elements has been reported [12,17]. It has found that the addition of Cu and Ti to Al increases its corrosion resistance in near neutral chloride solutions [5,6]. According to Lin and Starke Jr. [18], the addition of small copper content (0.01 -2.1%) to Al decreases its corrosion fatigue via increasing its cyclic strain resistance and further decrement in the corrosion fatigue is recorded with increasing the content of copper. On the other hand, the addition of low graphite percentages into pure aluminum was found to increase the corrosion of aluminum in 3.5% NaCl solution due to the activation effect for graphite towards the corrosion of aluminum [19,20]. Where, the increase of graphite content increases the corrosion of aluminum via increasing the cathodic, anodic, and corrosion currents, as well as increasing the corrosion rate and decreasing the polarization resistance.

The present work aims at in situ manufacturing of three new Al-TiC composites having the following chemical compositions; 85 wt % Al + 15 wt % TiC, 75 wt % Al + 25 wt % TiC, and 65 wt % Al + 35 wt % TiC. The synthesizing process was done in situ using a high energy ball mill in order to mix the different powders uniformly. These aluminum composites were melted and sintered at 1300 °C using a high frequency induction heat sintering furnace. The aim of this work was also extended to evaluate the effect of TiC content on the corrosion behavior of these composites in 3.5 wt.% NaCl solutions. Corrosion tests included the performing of change of potential with time, impedance spectroscopy, polarization, and change of current with time measurements. The surface of the composites was investigated after being corroded using scanning electron microscopy surface analysis.

2. EXPERIMENTAL SETUP

The powders of aluminum (Al), titanium (Ti) and graphite (Gr) with micro particle sizes were used in this work as received. The powders in wt. % were mixed with steel balls at ball-to-powder ratio of 5:1 and placed in a high energy ball mill. Different percentages of powders and balls were employed in order to get the different mixtures. The chemical compositions of the fabricated composites were 85%Al+15%TiC, 75%Al+25%TiC, and 65%Al+35%TiC, respectively. Ball milling of the powders was done using Desktop 220V High Energy Vibratory Ball Mill. After the uniform mixing of powders, it was placed in 80ml steel jar for 30 min mixing time and at 2000 rpm [12]. The sintering process was done under 10 MPa pressure at 1300 ^{o}C for 4.0 min in a graphite die using a high frequency furnace

(HFIHS) [12]. The diagrammatic representation of the die and HFIHS, in which the melting and sintering processes of the fabricated aluminum composites were done, is presented in our previous work [12].

The sodium chloride (NaCl) salt with purity of 99.00% was used in this study. A three-electrode configuration conventional cell (250 cm^3 solution volume) was employed for the electrochemical measurements. The Al-TiC composite with a cylindrical shape and total area of 1 cm^2 was used as the working electrode. An Ag/AgCl electrode (in 3M KCl solution) was employed as the reference electrode. A platinum sheet as was used as the counter (auxiliary) electrode. The surface of the working electrode was ground as previously reported [5,6].

Electrochemical tests were done using an Autolab Potentiostat-Galvanostat workstation. The change of potential with time (OCP) curves was collected over 20 h immersions. The impedance spectra were obtained at an OCP and the frequency was scanned between 100000 Hz and 100 mHz using an ac wave of ±5 mV peak-to-peak overlaid on a dc bias potential [6]. Cyclic polarization data were also obtained by scanning the potential from −1.80 V to 0.0 V at a scan rate of 0.001 V/s (Ag/AgCl). The potential in the back scan direction was swept from 0.0 V in the less negative direction at the same scan rate [12]. The change of current with time curves was recorded after 20 h immersion in NaCl solution before stepping the potential to a constant value of –100 mV for 1.0 h. All experiments were carried out using a fresh composite surface and new portion of the electrolytic chloride solution at room temperature.

The morphology and the elemental analysis of the surface of the aluminum composites were collected after performing the potentiostatic current time tests after 24 h immersion in NaCl solutions and applying the constant potential value for 1.0 h. This was done using a scanning electron microscope (SEM) and an energy dispersive X-ray analyzer; both were purchased from JOEL.

3. RESULTS AND DISCUSSION

3.1. Open-circuit potential (OCP) measurements

The change of OCP versus time curves obtained for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites in 3.5% NaCl solutions are displayed in Figure 1. The lowest potential vs. time was recorded for the sample with the lowest TiC content (curve 1). Where, the potential rapidly decreased in the negative direction in the first 20 min before slowly increased again towards the less negative values in about 120 min. The potential then stayed almost stable with increasing time and till the end of the experiment. The variation of potential with time nearly recorded the same behavior with increasing the TiC content (Figure 1, curve 2) but with great shift in the potential of the Al-TiC composite to the less negative values. Further shift towards the positive direction in the absolute potential was provided when the TiC content was further increased (curve 3). The high positive potential shift with increasing the TiC content may have resulted from the increased corrosion resistance in the aluminum composite with increasing its TiC percentage. This because the increase of

TiC content in the composite increases its passivity and thus decreases it corrosion in the 3.5% NaCl solutions.

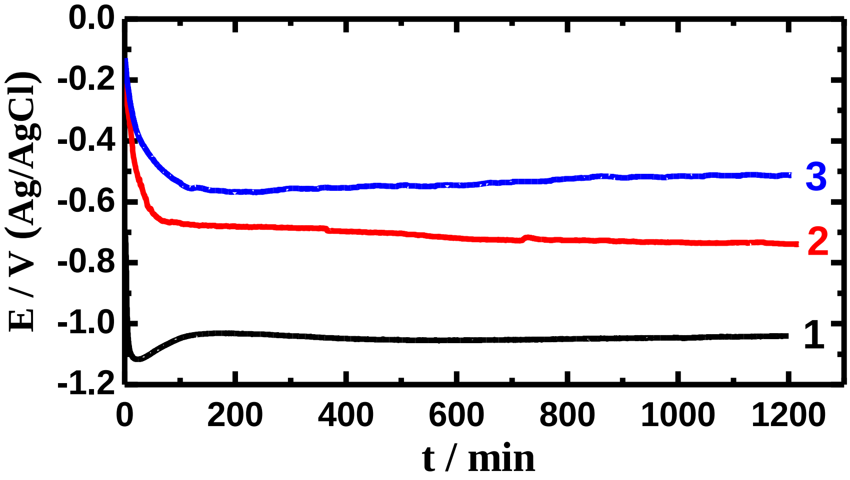

Figure 1. OCP curves obtained for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites in 3.5% NaCl solutions.

3.2. Electrochemical impedance spectroscopy (EIS) measurements

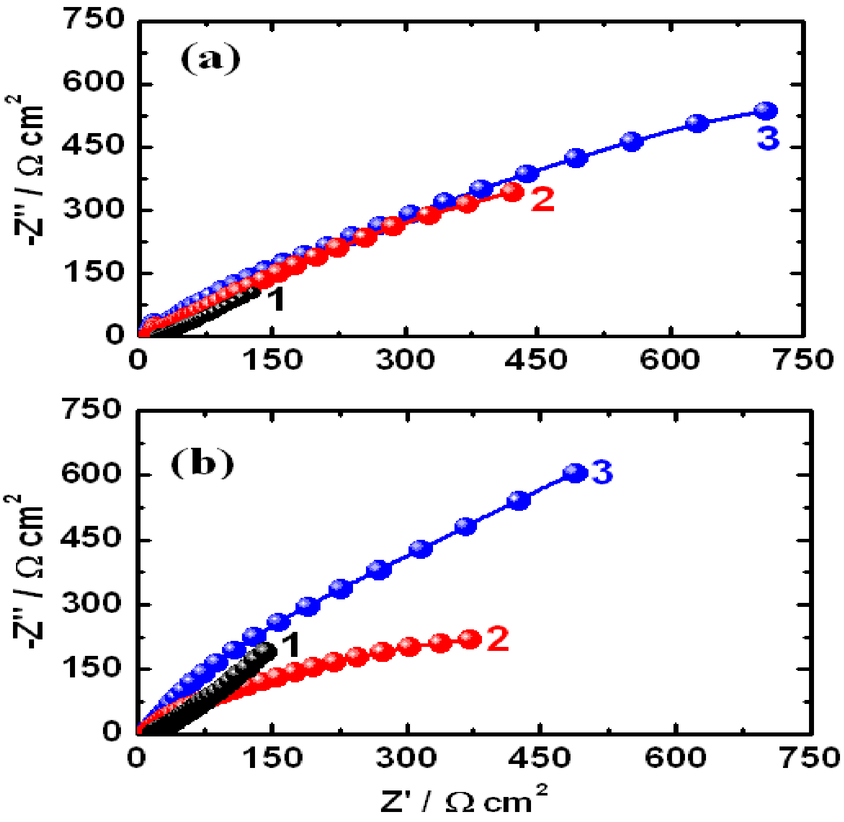

Figure 2. Nyquist plots of (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after (a) 1.0 h and (b) 24 h immersions in 3.5% NaCl solutions, respectively.

In order to report the kinetic parameters obtained from studying the effect of increasing TiC content on the corrosion of Al-TiC in 3.5% NaCl solutions, impedance experiments were carried out. Figure 2 shows the Nyquist plots obtained for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after (a) 1.0 h and (b) 24 h immersions in 3.5% NaCl solutions. The impedance spectra for the Nyquist plots shown in Figure 2 were analyzed and best fitted to the equivalent circuit model depicted in Figure 3. The values obtained from the fitting process for the elements of the circuit are recorded in Table 1. The definition of the parameters shown by the equivalent circuit is as following: R_S is the solution resistance, Q is the constant phase elements (CPEs), R_{P1} is the first polarization resistance, which refers to the chare transfer resistance between the surface of the aluminum composite and a corrosion product layer or an oxide film was formed on the surface due to its reaction with the chloride solution, C_{dl} is the double layer capacitance, and R_{P2} is another polarization resistance resulted from the interface between the formed surface film and NaCl solution [12].

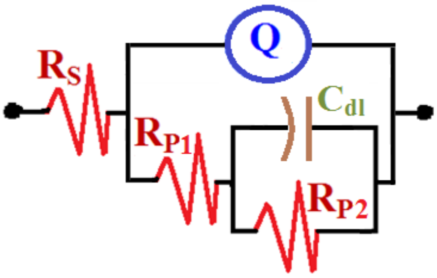

Figure 3. The equivalent circuit model that was used to fit the EIS data.

Table 1. EIS parameters obtained from the Al-TiC composites in NaCl solutions.

Sample	Impedance parameters					
	R_S/ Ω cm^2	Q		R_{P1}/ Ω cm^2	C_{dl}/ F cm^{-2}	R_{P2}/ Ω cm^2
		Y_Q/ F cm^{-2}	n			
Al-15%TiC (1 h)	15.59	0.008059	0.44	242.5	0.001380	215
Al-25%TiC (1 h)	17.79	0.003359	0.50	309.4	0.000969	281
Al-35%TiC (1 h)	18.21	0.002297	0.55	468.5	0.000702	473
Al-15%TiC (24 h)	13.55	0.003438	0.52	236.7	0.001174	214
Al-25%TiC (24 h)	14.83	0.001853	0.55	274.5	0.0003125	243
Al-35%TiC (24 h)	16.65	0.000953	0.59	296.4	0.0002224	443

It is worth to note that EIS plots shown in Figure 2 for Al-TiC composites show only one semicircle for immersion times, 1.0 h or 24 h, in the chloride solutions. The diameter of the semicircle plotted for Al-15%TiC after 1 h immersion in the chloride solution (Figure 2a, spectrum 1) recorded the smallest indicating that the corrosion resistance for this composite is the lowest. Increasing the TiC content to 25% is clearly seen to increase the diameter of the semicircle and further increment was recorded with increasing the TiC content to 35%. This reveals that the increase of the TiC content increases the corrosion resistance of Al-TiC composite. The data shown in Table 1 confirmed also that the increase of TiC content increases the values of all resistances, R_S, R_{P1} and R_{P2}. This effect also decreased the values of Y_Q (CPEs) and C_{dl}. Here, the constant phase elements, Q, represent Warburg impedance (W) because the value of the component "n" that is accompanied to Q is close to 0.5. The decrease in the values of Y_Q and C_{dl} indicates that the increase of TiC content decreases the dissolution of Al-TiC composites and thus increases the composite's corrosion resistance.

Prolonging the time of immersion, 24 h before performing the impedance measurements, is seen to slightly decrease the diameter of the obtained semicircles, Figure 2b, at all TiC contents. This indicates that the increase of immersion time lowers the corrosion resistance via increasing the dissolution of aluminum from the aluminum composites. Furthermore, the increase of TiC content was also found to significantly decrease the corrosion of the composite by increasing its corrosion resistance. The EIS parameters listed in Table 1 for the composites immersed for 24 h in 3.5% NaCl solutions confirmed also that. Where, the values of R_S, R_{P1} and R_{P2} that were recorded after 24 h immersion are lower than the values of the same parameters obtained for the composites after only 1 h immersion time. At the same time, the values of Y_Q and C_{dl} are slightly higher for the composites immersed for 24 h compared to the values of the same parameters obtained after only 1 h immersion in the chloride solutions. Like in the case of 1 h immersion, the impedance parameter, Y_Q, for the composites immersed for 24 h represents Warburg impedance, W, as indicated from the values of the component "n" listed in Table 1. In all cases, increasing the content of TiC within the aluminum composites was found to increase the resistance against corrosion. On the other hand, prolonging time of immersion decreases the corrosion resistance because of the chloride ions attack towards the surface of Al-TiC composites.

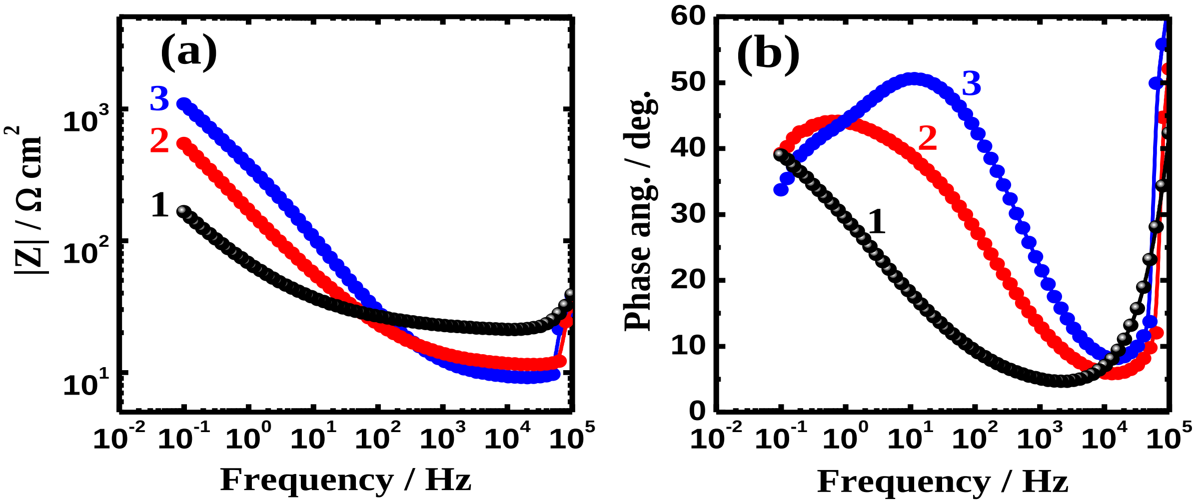

Figure 4. Bode (a) impedance and (b) phase angle plots of (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after 1.0 h immersion in 3.5% NaCl solutions

Figure 4 shows the Bode (a) impedance of the interface (|Z|) and (b) phase angle (Φ) plots obtained for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after 1.0 h immersion in 3.5% NaCl solutions. The Bode plots were obtained for the three aluminum composites after their immersions for 24 h in the chloride solutions and the plots are shown in Figure 5a and Figure 5b, respectively. It is seen from Figure 4a and Figure 5a that the increaser of TiC content increases the values of |Z|, particularly at the low frequency regions. According to Mansfeld et al. [22,23], the increase of |Z| values at low frequency region indicates on the higher of the passivation of the surface. This is turn confirms that the increase of TiC percentage increases the corrosion resistance of the aluminum. It is also seen from Figure 4a and Figure 4b that the maximum values of Φ increased with the increase of TiC content in the aluminum composites. This gives another prove that the aluminum composite gets more protected against corrosion with the increase of TiC concentration. Although, the increase of immersion time from 1.0 h to 24 h increased the corrosion of the aluminum composites, the increase of TiC content still increased the corrosion resistance in 3.5% NaCl solutions.

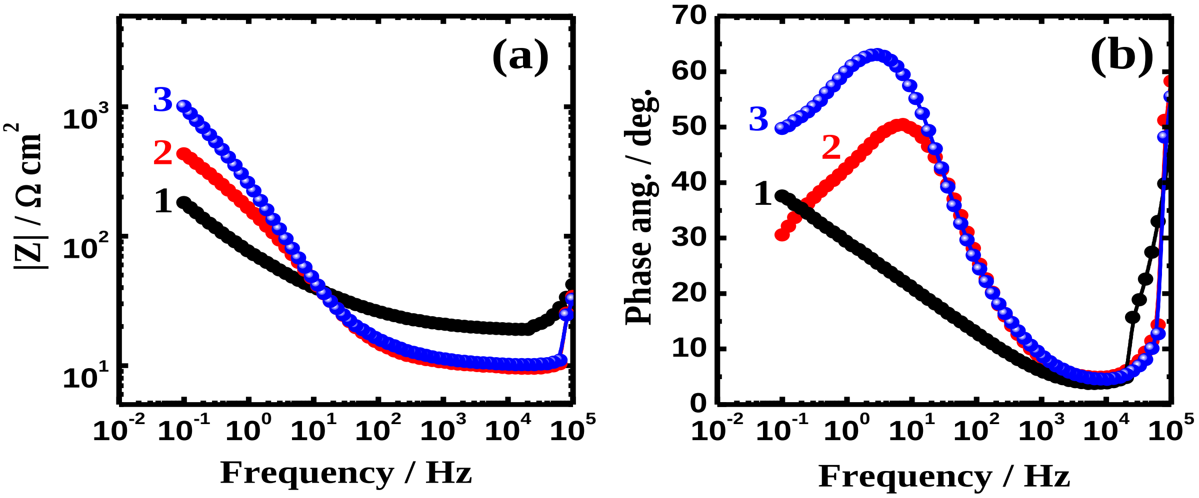

Figure 5. Bode (a) impedance and (b) phase angle plots of (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after 24 h immersion in 3.5% NaCl solutions.

3.3. Cyclic potentiodynamic polarization (CPP)

CPP technique is one of the strongest methods that have been successfully employed to report the mechanism of corrosion and corrosion mitigation for metals and alloys in corrosive media. We have been used the CPP technique in understanding the uniform and pitting corrosion as well as corrosion inhibition phenomena for different metallic materials in different aggressive solutions [12,13,19]. Figure 6 shows CPP curves obtained for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after (a) 1 h and (b) 24 h immersions in 3.5% NaCl solutions at room temperature. The values of corrosion parameters such as cathodic Tafel slope (βc), anodic Tafel slope (βa), corrosion current density (j_{Corr}), corrosion potential (E_{Corr}), polarization resistance (Rp), and corrosion rate (R_{Corr}) obtained from CPP curves for the different Al-TiC composites after 1 h and 24 h immersion in the test solution are depicted in Table 2. The values βc, βa, j_{Corr}, and E_{Corr} were calculated from the intersection of the extrapolated anodic and cathodic Tafel lines located next to the linearized current regions as reported earlier [23,24]. The R_P values for Al-TiC composites were calculated from the parameters listed in Table 2 using the Stern–Geary equation as has previously reported [23,24]. Moreover, the values of were obtained R_{Corr} according the following equation [25];

$$R_{Corr} = j_{Corr}\left(\frac{k E_W}{d A}\right) \quad (1)$$

Where, *k* is a constant that defines the units for the corrosion rate (*k* = 3272 milli inches per year, mpy)), E_W the calculated equivalent weight in grams/equivalent of Al-15%TiC (=8.55), Al-25%TiC (=7.95), and Al-35%TiC (=7.35), *d* the density in gcm^{-3}, and *A* the area of electrode in cm^2.

The polarization curve 1 that is depicted in Figure 6a provides that Al-15%TiC composite showed an active-passive behavior. Where, the anodic current rapidly increased to reach a region with very little change in its value with the increase of potential (passive region) before rapidly increased again till the end of the direct scan of potential. The increase of current at the beginning of the anodic polarization branch was due to the dissolution of Al metal (Al^0) to cationic aluminum (Al^{3+}) [19,20];

$$Al = Al^{3+} + 3e^- \quad (2)$$

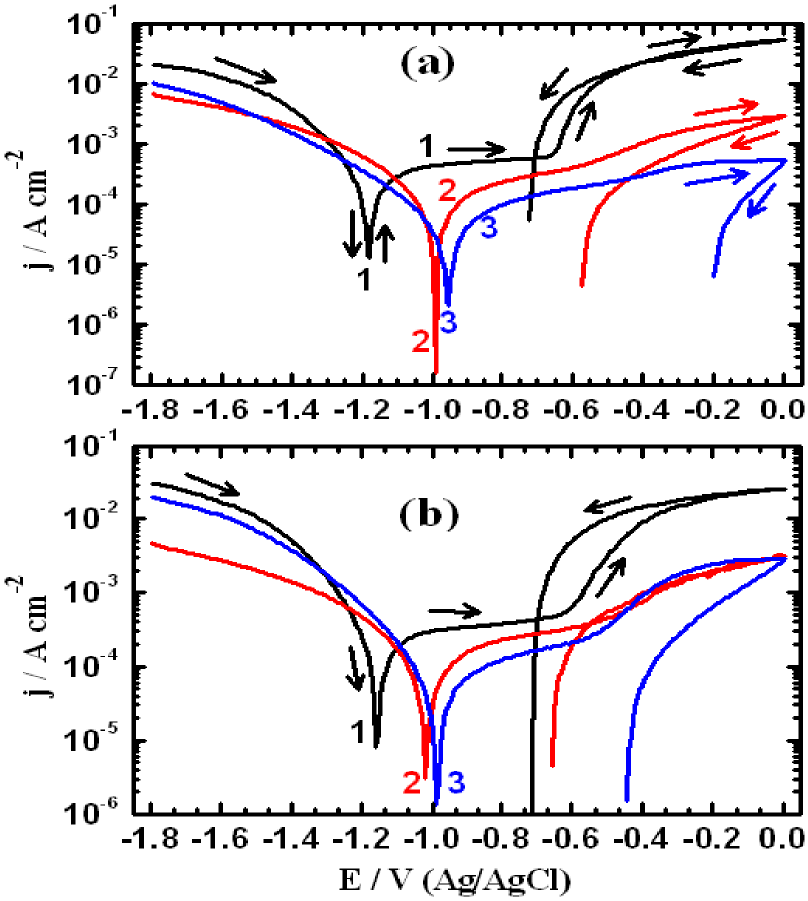

Figure 6. CPP curves of (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after (a) 1 h and (b) 24 h immersions in 3.5% NaCl solutions.

The produced electrons from this reaction were consumed by the cathodic reaction, which has been reported to the reduction of oxygen [10]. On the other side, the passive region was formed due to the formation of aluminum oxide film on the surface of the composite leading to stabilizing the current values over a wide range of the applied potential [12,18];

$$2Al + 6OH^- = Al_2O_3.3H_2O + 3e^- \quad (3)$$

Under the increase of the applied potential and the chloride ions attack, the oxide layer gets destroyed and broken-down leading to the increase of current values. Revising the potential in the backward direction shows that the reversed current was higher than its values in the forward direction and led to the appearance of a hysteresis loop. This potential-current behavior means that the pitting corrosion occurs, where the chloride ions attack the flawed areas of the aluminum oxide film and aluminum chloride and oxychloride complexes are formed then dissolved in the solution [6];

$$Al^{3+} + 4Cl^- = AlCl_4^- \quad (4)$$

$$Al^{3+} + 2Cl^- + 2OH^- = 4Al(OH)_2Cl_2^- \quad (5)$$

Table 2. Polarization parameters obtained for Al-TiC composites in 3.5% NaCl solutions.

Sample	**Corrosion parameter**					
	β_c / Vdec^{-1}	**E_{Corr} / V**	**β_a / Vdec^{-1}**	**j_{Corr} / µA cm^{-2}**	**R_P / Ω cm^2**	**R_{Corr} / mpy**
Al-15%TiC (1 h)	0.170	–1.165	0.242	180	241.2	1.6785
Al-25%TiC (1 h)	0.180	–0.985	0.240	70	638.9	0.6070
Al-35%TiC (1 h)	0.190	–0.958	0.235	27	1692	0.2164
Al-15%TiC (24 h)	0.165	–1.145	0.245	150	285.8	1.3988
Al-25%TiC (24 h)	0.175	–1.015	0.240	68	647.1	0.5896
Al-35%TiC (24 h)	0.185	–0.960	0.210	37	1155.8	0.2966

The increase of Al-TiC content within the Al-TiC composite after 1.0 h immersion in the chloride solutions highly decreased the cathodic and anodic currents as well as the value of j_{Corr} and shifted the values of E_{Corr} to the less negative direction. The increased corrosion resistance of the composite with increasing the concentration of TiC was further confirmed by the parameters of Table 2. Where, the increase of TiC content greatly decreased the values of j_{Corr} and R_{Corr} and significantly increased the values of R_P. Moreover, the increase of TiC content eliminated the occurrence of pitting corrosion, which was found to take place only for the Al-15%TiC composite as indicated by the appearance of hysteresis loop on the CPP curve 1. At higher TiC contents, 25% and 35%, the disappearance of the hysteresis loop as seen from Figure 6a (curve 2 and curve 3) revealed that the pitting corrosion did not occur.

Prolonging the exposure period of time for 24 h in the chloride test solution before measurements (Figure 6b) increased the corrosion parameters of the aluminum composites. Where, the values of j_{Corr} and R_{Corr} recorded higher ones and the values of R_P were lower in compared to those values obtained from the CPP curves shown in Figure 6a for Al-TiC composites immersed for only 1 h. This is confirmed not only by the polarization curves but also by the data listed in Table 2. The decreased corrosion resistance and the increased corrosion rate after 24 h immersion in the chloride solution are most probably due to the increased dissolution of aluminum resulted from the corrosive and continuous attack of the chloride ions, which increased with increasing the time of immersion in NaCl solutions. The CPP curves thus prove that the increase of TiC content increases the corrosion resistance of Al-TiC composite and this effect decreases with the increase of immersion time.

3.4. Change of current with time data

Chronoamperometric current time (CCT) measurements were carried out to shed more light on the effect of increasing the TiC content on the uniform and pitting corrosion of the aluminum composite at an active anodic potential (−100 mV *vs.* Ag/AgCl). Figure 7 shows the potentiostatic current-time curves obtained at -100 mV for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after 24 h immersion in 3.5% NaCl solutions. It is seen from Figure 7 that the initial current of Al-15%TiC (curve 1) abruptly increased in the first few minutes due to the dissolution of either an oxide film or a corrosion product layer was formed on the aluminum surface during its 24 h immersion in the solution. The current rapidly decreased again before slightly decreasing with increasing time up to the end of the run.

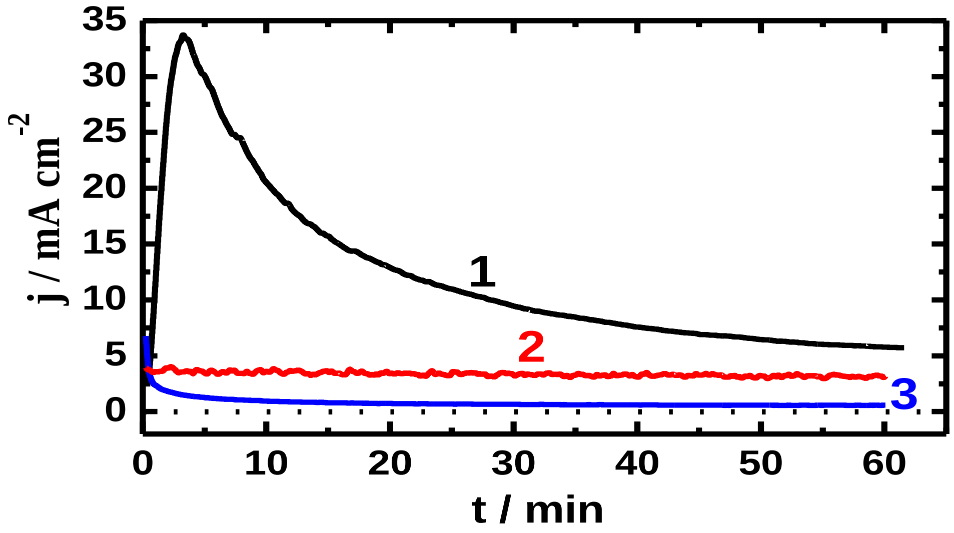

Figure 7. CCT curves recorded at –100 mV/AgCl for (1) Al-15%TiC, (2) Al-25%TiC and (3) Al-35%TiC composites after 24 h immersion in 3.5% NaCl solutions.

This is attributed to the formation of corrosion products that enable the surface to resist the corrosive attack of chloride ions at the applied active potential. Increasing the TiC content to 25%

lowered the obtained current across the whole time of the experiment. Further increasing the content of TiC, 35%, provided further decreases in the current of the aluminum composite recording the lowest values. These lower absolute values of currents were recorded due to the increased passivity of the surface of aluminum composites a result of oxide film formation. The formed oxide film is most probably an aluminum oxide (Al_2O_3, see equation (3)) either alone or with the corporation of titanium oxide. The current-time measurements thus confirm the results obtained by polarization and EIS experiments and proved that the increase of TiC content decreases the corrosion of aluminum composite via increasing its corrosion resistance towards both uniform and pitting corrosion.

3.5. Scanning electron microscope (SEM) and energy dispersive X-ray (EDX) investigations

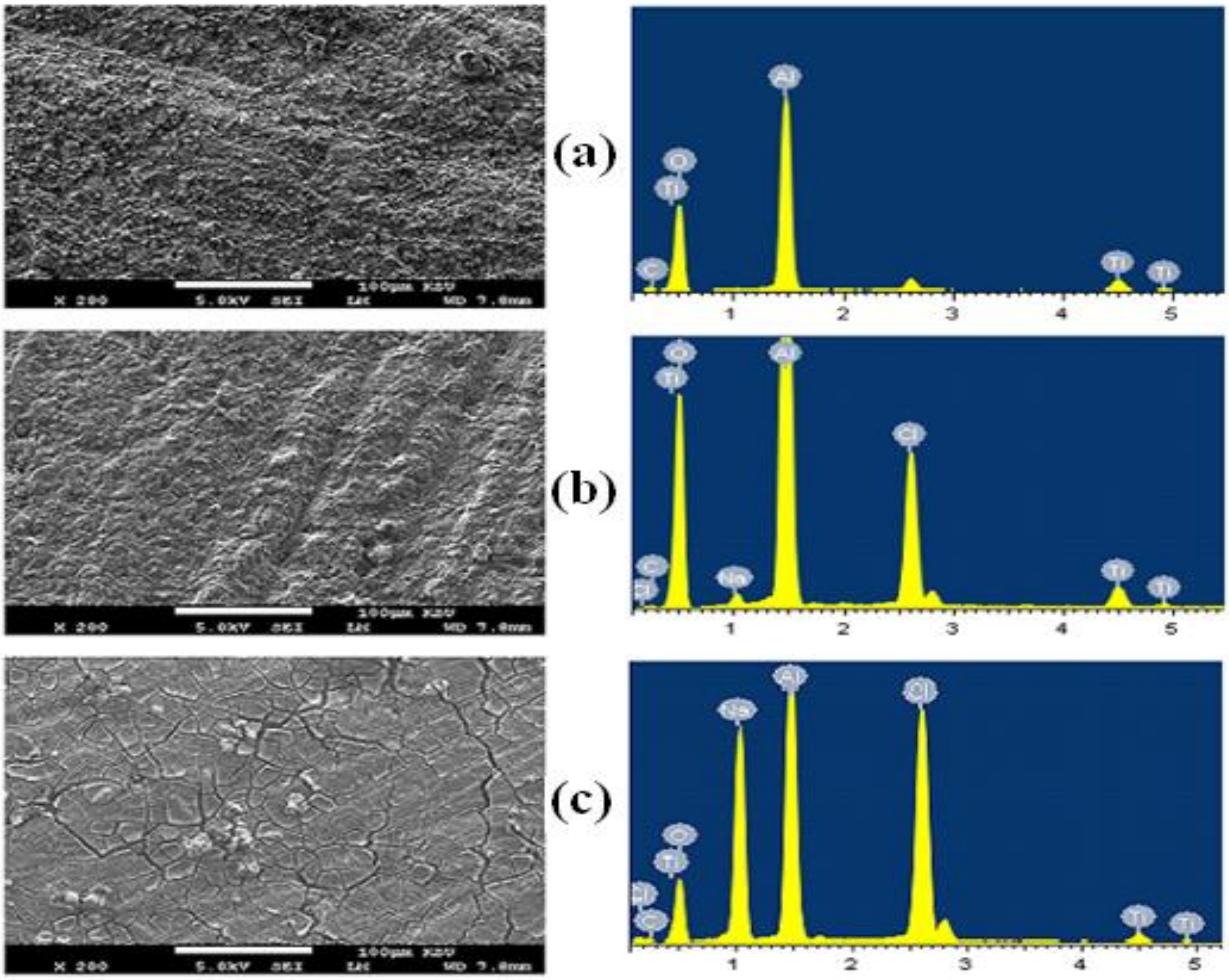

Figure 8. SEM images and EDX profile analyses obtained after 24 h immersion in 3.5% NaCl solutions for the surface of (a) Al-15%TiC, (b) Al-25%TiC and (c) Al-35%TiC composites, respectively before stepping their potential to –100 mV(Ag/AgCl) for 1.0 h.

Figure 8 shows the SEM images and EDX profile analyses obtained for the surface of (a) Al-15%TiC, (b) Al-25%TiC and (c) Al-35%TiC composites that were sintered at 1300 ºC after their immersions for 24 h in 3.5% NaCl solutions followed by applying–100 mV as a constant potential for 1.0 h. The SEM micrograph obtained from the surface of the aluminum composite that had only 15%TiC (Figure 8a) shows a severe corrosion, which is mostly uniform. The corrosion of the aluminum composite led to the formation of some corrosion products cover the surface. The EDX profile analysis for the surface shown in Figure 8a indicated that the weight percent of oxygen was 51.59 in addition to the presence of 10.37% C, 4.62% Ti, and 33.42% Al. Also, the SEM image shown in Figure 8b for the composite that contained 25% TiC showed little deterioration and the EDX profile indicated on the presence of 30.28% O, 21.03% C, 4.78% Ti, 1.9% Cl, 1.1% Na, and the rest was Al metal.

Furthermore, the SEM image of Figure 8c shows that the surface of the aluminum composite is fully covered with a thick layer of corrosion products. Analyzing the surface using EDX profile analysis confirmed that the weight percents of the elements found on the surface were as following; 24.19% O, 28.44% C, 4.63% Ti, in addition to the aluminum. SEM/EDX investigations thus revealed that the increase of TiC decreased the corrosion the aluminum composite via decreasing its dissolution and increasing its resistance towards both uniform and pitting corrosion. Where, the increase of TiC content increases the passivity of the surface and that decreases the aggressiveness action of the chloride ions attack.

4. CONCLUSIONS

The effect of TiC additions on the corrosion behavior in 3.5% NaCl solutions of newly fabricated Al-TiC composite was reported. The composites were in situ synthesized using high energy ball mill (HEBM) and HFIHF techniques before being sintered at 1300 ºC. OCP measurements indicated that the increase of TiC content remarkably shifts the potential of Al composite to the less negative values. EIS Nyquist plots revealed that the diameter of the obtained semicircle, the solution resistance and polarization resistances of the composite significantly increase with increasing the percent of TiC. The EIS Bode plots proved that the values of the impedance of the interface (|Z|) and the maximum phase angle (Φ) increase with increasing TiC percent, which means that the surface of the composite gets more passivated and corrosion resistant. This was also confirmed by CPP curves, where the increase of TiC content was found to highly decrease the cathodic current, anodic current and the values of j_{Corr} and R_{Corr} as well as increase the values of Rp of the Al composite. The potentiostatic current-time curves obtained at –100 mV vs. Ag/AgCl indicated that the corrosion of the composite decreases through decreasing the absolute currents with the increase of TiC percentage within the Al composite. SEM/EDX investigations showed that the surface of the composite gets more protected and passivated with TiC content by forming an oxide film and/or a corrosion product layer that decreases the aggressiveness action of the chloride ions attack.

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