The Effect of Electroless Ni Coating of SiC Particles on the Corrosion Behavior of A356 Based Squeeze Cast Composite

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The corrosion behavior of electroless Ni coated SiC particle reinforced squeeze cast aluminum based composite was investigated by potentiodynamic scanning (PDS) and electrochemical impedance spectroscopy (EIS) techniques in aerated and deaerated chloride solutions. Microstructural and interfacial characterization of the composite was carried out by using an optical microscope, scanning electron microscope (SEM), energy dispersion spectroscopy (EDS) and X-ray diffractometer (XRD). It has been observed that electroless Ni coating of SiC particles is not an effective method to improve its corrosion resistance in structural applications containing halide solution.

0 INTRODUCTION

Properties of metal matrix composites (MMCs) strongly depend on the interfacial phenomena between the metal matrix and ceramic reinforcement [1] to [4]. The wettability of reinforcement by liquid metal is the key factor to achieve high interface bonding strength [5] to [7]. Unfortunately, the wettability of SiC particle reinforcement with molten aluminum matrix is generally poor. However, several methods including the modification of the matrix composition, coating of the reinforcement and control of process parameters have been widely used to improve the interfacial properties [8] to [11]. Among these methods, electroless nickel (EN) coating of the reinforcement, which is a simple, low-cost and an easy to use process, has been successfully applied to prevent undesired interfacial reactions and promote the wettability through increasing the overall surface energy of the reinforcement [12] to [14].

The corrosion properties of aluminum based metal matrix composites reinforced with coated ceramic particles is still in the investigation stage. In addition, the published literature on the corrosion behavior of aluminum based composites is rather limited and often contradictory [15] and [16]. The reason is generally related to the quantity of alloying elements in the Al matrix, reinforcement shape, size and volume fraction and composite production method [17] and [18]. Also, many researchers reported that galvanic actions between the uncoated SiC particle and matrix alloy is not effective in the corrosion behavior of the composite due to a semiconductor structure of SiC phase. In other words, SiC is cathodic to aluminum and does not polarize easily [19] to [21]. However, the corrosion susceptibility of the composite increases due to the presence of Al_4C_3 phase, which forms at the SiC/Al matrix interface [17], [22] and [23]. Al_4C_3 reduces not only the corrosion resistance but also the ductility and strength of the composite [24] and [25]. Similar results have been observed in our previous studies [26] and [27]. Coating of the reinforcement changes the physical and mechanical properties of the composites, as well as the corrosion behavior [16] and [18]. Also, surface treatment of SiC reinforcement may improve the wetting behavior with Al matrix. However, the protective film on the matrix surface can be easily broken due to a mismatch in coefficient of thermal expansion (CTE) between different phases. In addition, corrosion can decrease the interfacial strength between SiC and Al matrix [20], [28] and [29]. Recently, a considerable attention has been paid especially to the use of these composites in marine applications [30]. However, several drawbacks of these materials, such as low temperature ductility and poor corrosion resistance hinder their wide range of application in alkaline environments [8].

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In this study, corrosion behavior of Al–7%Si–0.7%Mg (A356) based metal matrix composite reinforced with electroless nickel (EN) coated SiC particles produced by squeeze casting technique has been investigated. Another purpose of this study was to increase wettability between matrix/reinforcing phase and preventing undesirable interface reactions. The coating morphology, microstructural and interfacial properties of the composite, and the effect of EN coating of SiC reinforcement on the corrosion properties has been examined and discussed in detail.

1 EXPERIMENTAL PROCEDURE

1.1 Electroless Nickel (EN) Coating

Electroless nickel coating of SiC particles was carried out as follows: cleaning of SiC particles in acetone for 15 min. After cleaning, particles were sensitized for another 15 minutes in a solution containing 10 g/L $SnCl_2$ and 30 mL HCl. The sensitized particles were immersed in a solution containing 0.25 g/L $PdCl_2$ and 3 ml/L HCl for 15 min in order to activate the nonmetal SiC particle surface. Rinsed and dried SiC particles were gently dispersed in an electroless nickel bath containing 45 g $NiCl_2$, 8 g NaH_2PO_2.H_20, 100 g $Na_3C_6H_5O_7$ and 50 g NH_4Cl dissolved in 1 L distilled water. Deposition was carried out at 80 to 90 °C and 8 to 9 pH.

1.2 Composite Manufacturing

A composite reinforced with 20 vol. % electroless nickel coated SiC particles with an average size of 15 μm was produced by a squeeze casting technique. Al–7%Si–0.7%Mg matrix alloy was melted in a ceramic crucible and then SiC particles were added by stirring continuously. The mixture was superheated up to 750 °C and then poured into a preheated (400 °C) die and finally solidified under a pressure of 100 MPa.

1.3 Characterization

Characterization studies were carried out by using optical microscope, scanning electron microscope (SEM), energy dispersion spectrometer (EDS) and X-ray diffractometer (XRD).

1.4 Corrosion Tests

Electrochemical investigation of the composite was performed with potentiodynamic polarization (PDS) and electrochemical impedance spectroscopy (EIS). All experiments were carried out with a computer-controlled potentiostat (PCI4/750, GAMRY Instruments, Inc., Warminster, PA) in aerated and deaerated 3.5% NaCl solution at room temperature. Ag/AgCl and platinum (Pt) electrodes were used as a reference and auxiliary electrode, respectively. The solution was deaerated in order to remove the oxygen with nitrogen (N_2) gas. Deaerated process was started 60 minutes prior the measurement and continued until the end of the experiment. Specimens were immersed into the solution until obtaining a steady open circuit potential (OCP). After reaching equilibrium, polarization test started at the cathodic overpotential and the scan was stopped when the specimens reached the anodic corrosion current density of 10 mA.cm^{-2}. All potentiodynamic scanning (PDS) tests were carried out according to the ASTM standard [31]. EIS measurements were carried out using AC signals of amplitude 10 mV at the open circuit potential (OCP) in the frequency range of 100000 to 0.01 Hz. The exposed area of the test specimens was about 5 x 5 mm (±0.01), and all data have been normalized according to the surface area. Impedance parameters were calculated by fitting the experimental results to an equivalent circuit model by using the Echem Analyst software.

2 RESULTS

2.1 Characterization

Surface and elemental Ni mapping of electroless nickel coated SiC particles are given in Figs. 1a and b, respectively. It has been observed that the EN coating is homogenous, uniform and the thickness is about 1 μm. A typical microstructure of the composite produced by squeeze casting is shown in Fig. 2. In our previous study [32], it was demonstrated that the microstructure consists mainly from aluminum

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dendrites and eutectic silicon with no evidence of macro-pores. The amount of micro-pores was measured as 0.54% (in vol.).
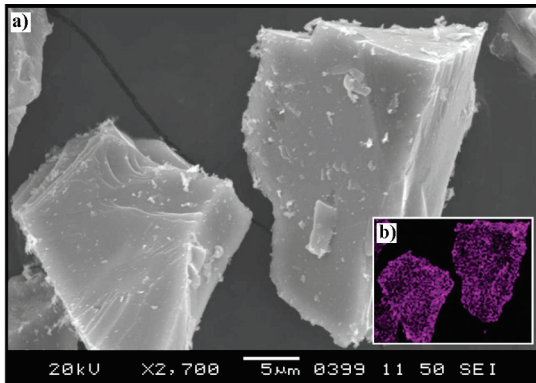

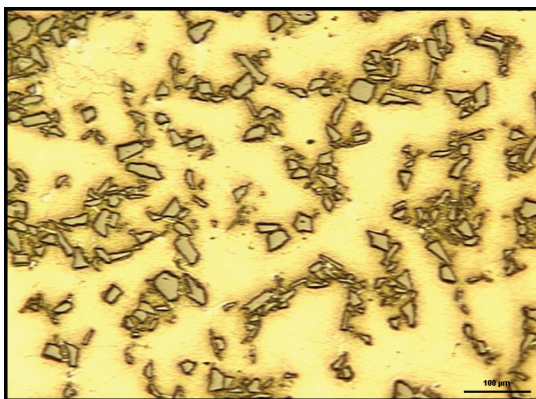Fig. 1. *a) SEM image and b) X-ray map of Ni on the SiC particle surface*

Fig. 2. *Optical micrograph of squeeze cast composite*
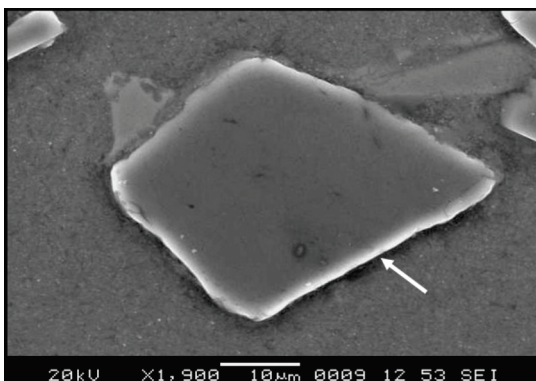

Fig. 3. *SEM micrograph showing the SiC/matrix interface*

Higher magnification micrograph of the composite demonstrates a good SiC/matrix interface in terms of porosity (Fig. 3). In addition, XRD pattern of the composite indicates that Al4C3 reaction product does not occur (Fig. 4).
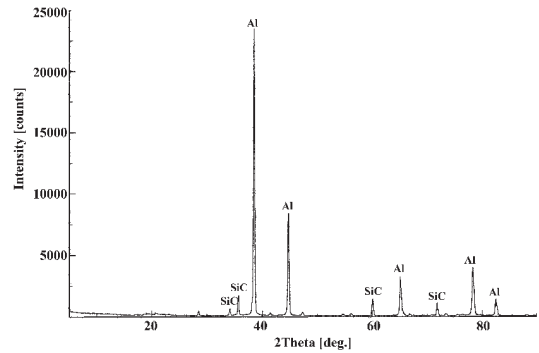

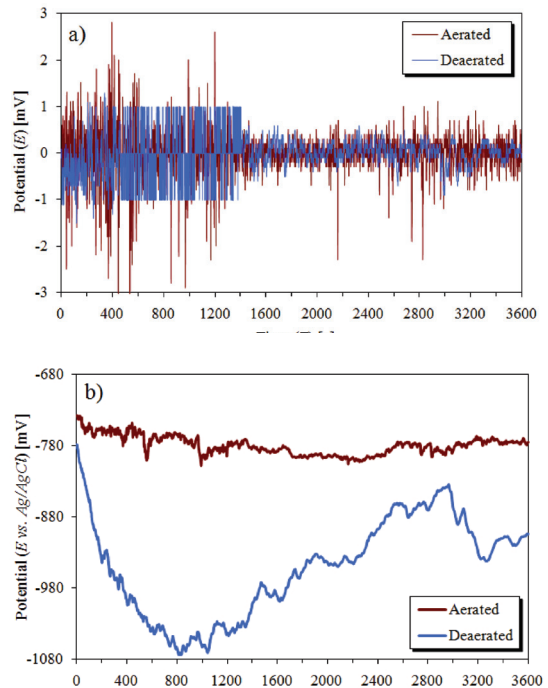Fig. 4. *XRD pattern of squeeze cast composite*

2.2 Corrosion Tests

Fig. 5. *a) open circuit potential–time (E_{ocp}–T) curves, b) electrochemical potential noise measurements in aerated and deaerated 3.5% NaCl solutions*

Open circuit potential (OCP) and electrochemical noise measurements of the composite in aerated and deaerated 3.5% NaCl solutions measured against time are given in Fig. 5. Noise values were calculated from the variation between two potential values obtained from OCP.

It is well known that a corrosion reaction may be masked by the heavily reduction reactions in aerated solution. Thus, especially aluminum alloys, pitting onset, passive areas and subtle polarization effects can not be seen completely. Therefore, the solution was deaerated to remove the oxygen by introducing nitrogen into the system. Fig. 6 shows a typical potentiodynamic polarization curve of the composite obtained in aerated and deaerated 3.5% NaCl solutions. Corrosion parameters obtained from these polarization curves are collected in Table 1.
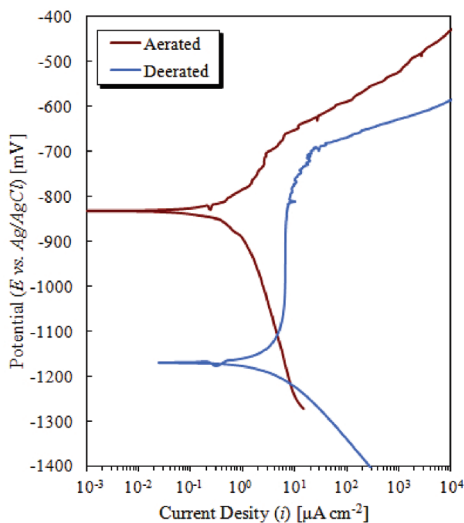

Fig. 7. *Nyquist diagram of the sample exposed to aerated 3.5% NaCl*

Fig. 6. *Potentiodynamic polarization curves in aerated and deaerated 3.5% NaCl solutions*
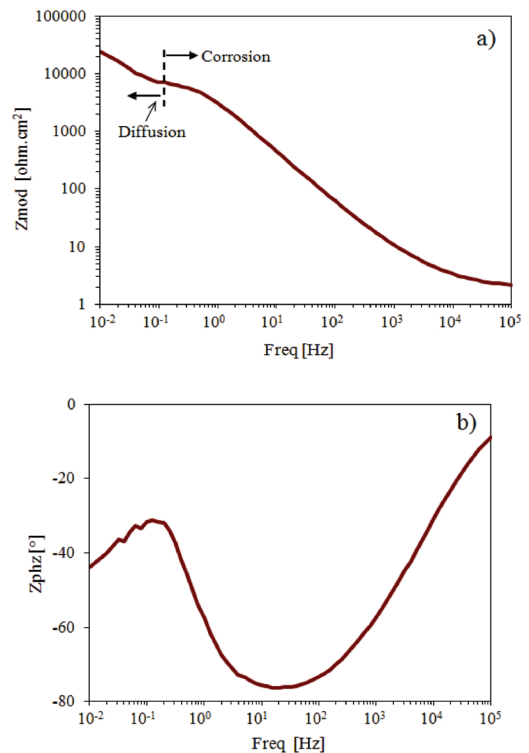
Fig. 8. *Bode plots of the sample exposed to aerated 3.5% NaCl: a) magnitude, b) phase angle*

Fig. 7 shows the impedance response of the composite in a Nyquist representation. In addition, the corrosion behavior of the composite is further illustrated by Bode plots in Fig. 8.

In order to obtain accurate results, complex plane plots were analyzed by fitting the experimental results to the equivalent circuit as given in Fig. 9. It is obvious from Fig. 7 that fitted results present a similar shape with experimental results. Also, impedance parameters derived from complex plane plots are given in Table 2.
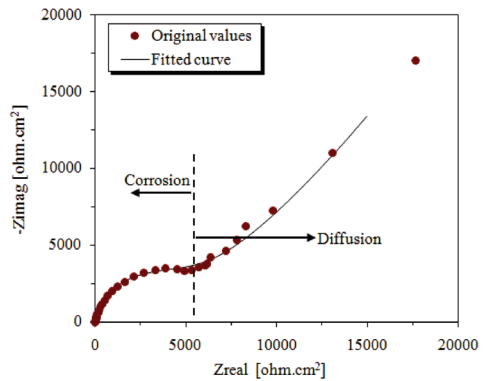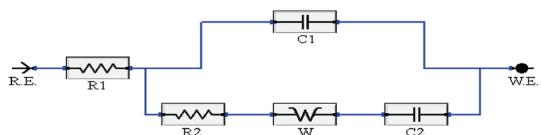

Fig. 9. *Equivalent circuit model represents the metal/solution interface*

Table 1. *Corrosion parameters obtained from polarization curves of the composite in aerated and deaerated 3.5% NaCl solutions (E vs. Ag/AgCl)*

Test condition	E_{ocp} [mV]	E_{corr} [mV]	E_{pit} [mV]	E_{pass} [mV]	I_{pass} [μA/cm²]	I_{corr} [μA/cm²]
Aerated solution	-776	-833	-696	-	-	1.25
Deaerated solution	-904	-1169	-718	-1135	6.15	2.50

Table 2. *Impedance parameters derived from complex plane plot in aerated 3.5% NaCl solution*

$R1$ [Ω.cm²]	$R2$ [Ω.cm²]	$C1$(x10⁻⁶) [F.cm⁻²]	$C2$(x10⁻³) [F.cm⁻²]	W(x10⁻⁶) [S.s^½.cm⁻²]
10.65	4850	30.33	6.862	258.6

2.3 Corrosion Morphology

Fig. 10 is a typical SEM micrograph of a squeeze cast composite exposed to 3.5% NaCl solution, where the presence of preferentially localized corrosion can be seen at SiC/matrix interface.
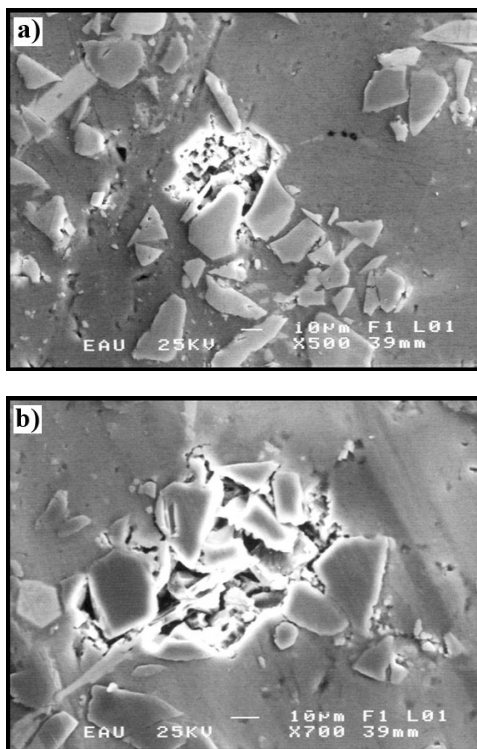

Fig. 10. *SEM micrographs showing the corroded surfaces of the sample*

Fig. 11 shows the corrosion progress paths on the corroded surface of squeeze cast composite at the initial and intensive corrosion stages. At extensive corrosion levels, the presence of pits at the SiC/matrix interface can be considered as regions where SiC particles are partially or completely detached (Fig. 12).

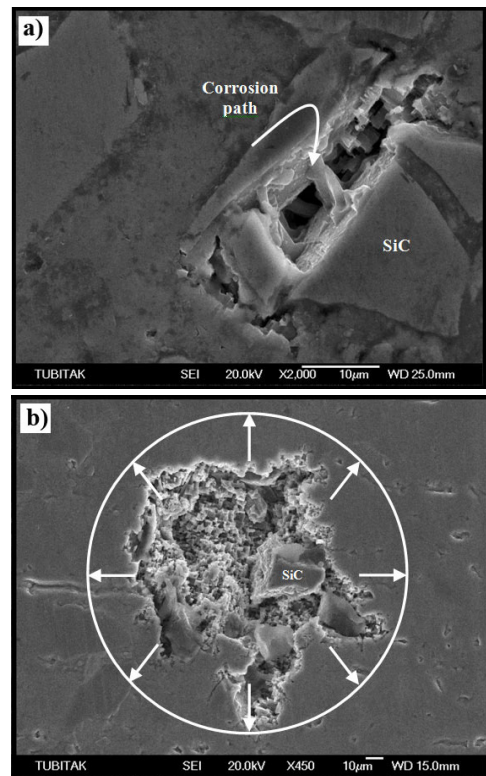Fig. 11. *Initial and intensive stages of corrosion: a) fresh pit, b) grown pit (arrow directions present corrosion paths)*

Fig. 13 reveals the preferential removal of the matrix material around EN coated SiC particles. This figure illustrates that SiC particles are not greatly affected by the corrosion process. The corrosion progress on aluminum rich matrix, causes an intense porous structure, without any

corrosion product on SiC particle surface. In addition, it was observed that corrosion did not cause any damage in the EN coating, as illustrated by small, light spheres in Fig. 13.
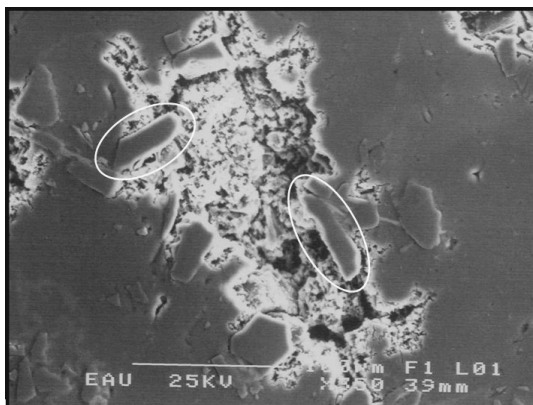

Fig. 12. *Detachment of SiC particles from the structure due to intensive matrix corrosion*
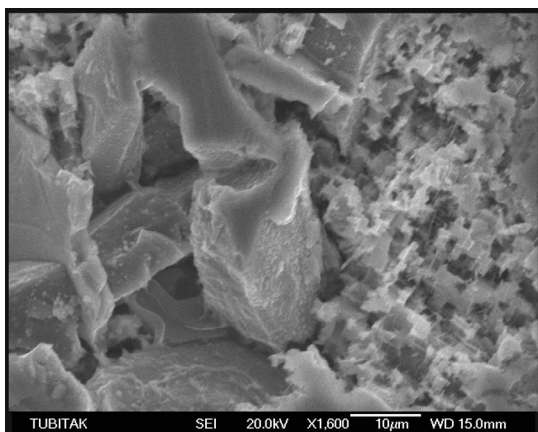

Fig. 13. *High magnification SEM micrograph of EN coated SiC particles after the corrosion test*

3 DISCUSSION

In the literature, it has been attributed that the following parameters significantly affect the corrosion behavior of metal matrix composites in macro scale; (i) reinforcement volume fraction [26] and [33], (ii) composition of the matrix alloy [28] and [34], (iii) reinforcement coating [15] and [18], (iv) composite manufacturing method [33], and (v) thermal or thermo-mechanical post treatment [33] and [35]. In addition, the corrosion principle of aluminum alloys under passive

conditions establishes the pitting [19]. Therefore, for these alloys, the pitting potential value is directly related to the corrosion susceptibility, and it can provide more realistic results related to the electrochemical behavior of the composites. In spite of using the same matrix alloy and reinforcement volume fraction in this study, the corrosion and pitting results are relatively more noble (Table 1) compared to our previous study [18] and [26]. Also, since no post treatment was applied in this study, the difference in potential values cannot be explained by parameters (i), (ii) and (v) given before. Therefore, the main reason of the difference can be attributed to the manufacturing method (iv) and/or reinforcement coating (iii).

From the point of manufacturing method, in both liquid infiltration and squeeze casting methods, a composite material solidifies under pressure, therefore, its effect on microstructure, in terms of porosity and interfacial properties, is insignificant. As reported in our previous studies, the porosity ratio of 20% volume fraction SiC reinforced composite manufactured by liquid infiltration [18], stir casting and extrusion [20] and squeeze casting [32] is the same and approximately 1%. Porosity, which acts as preferential sites for corrosion, is one of the key parameters in the corrosion behavior of composite materials. Therefore, it can be concluded that, the most effective parameter that differentiates the corrosion results obtained in each study is the type of reinforcement coating (iii).

As it is known, if the gradient of corrosion rate with time is neglible, it can be said that the material undergoes general corrosion. In such a case, it is easier to determine the corrosion rate and the lifetime of the material. However, as observed in this study, if the corrosion behavior changes significantly with time (Fig. 5), this indicates that the material undergoes pitting corrosion. In this case, the test carried out in aerated solution to determine the corrosion rate and the obtained data lose its validity and reliability, respectively [23] and [36]. In such circumstances, using the electrochemical noise technique and performing the tests in deareated solution gains importance. As seen from Fig. 5a, the open circuit potential (OCP) values of the composite measured under aerated condition are more positive than in

deaerated condition. This can be explained by the affinity of the present and dissolved oxygen in aerated condition. The reason is that in deaerated condition, the oxygen reduction reaction given by reaction (1) will not occur, thus the growth of the surface oxide layer will be prevented. Consequently, dissolution of the metal became more dominant and the protective oxide layer became gradually thinner.

$$\tfrac{1}{2}O_2 + H_2O + 2e^- \rightarrow 2OH^- \qquad (1)$$
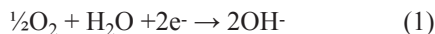
In order to evaluate localized interactions, the electrochemical potential noise magnitudes of each sample were compared (Fig. 5b). 25 minutes after the sample was immersed into the electrolyte it was observed that localized corrosion is hindered due to passivation, and this inhibition is relatively stronger in deaerated solution. This can be easily seen in the magnitued graph of the noise amplitude (Fig. 5b). This result verifies that the difference in potential values observed in both studies arise from the composite structure rather than the oxidation in dilute solution.

In our previous study [18], corrosion was preferentially started and continued at the interface between the Al matrix and alloying elements rather than reinforcement. Also, since SiC particles were coated with SiO_2 and TiO_2, no detrimental galvanic corrosion was observed between SiC particles and matrix alloy. In addition, up to date, no galvanic effect has been reported between Al and SiC [28] and [33]. However, in this study, SiC particles were coated with nickel which is a relatively good conductive metal, and a metallic bond is expected to occur where Al and Ni are in direct contact. An interface with a metallic bond is more ductile than other bonds, and is desirable in metal matrix composites [8]. However, as a metallic bond has a relatively high electrical conductivity, electrons in the matrix alloy can be easily drained through the conductive EN coating in a corrosive environment,. Also, it has been demonstrated that Ni can react with Al matrix and intermetallic phases such as $NiAl_3$ and Al_3Ni_2 can occur preferentially at the Al/SiC interface [14]. These intermetallic phases frequently have solution potentials differing from that of the matrix alloy in which they occur, therefore, localized galvanic cells may be formed between these phases and the matrix. Thus, these phases are cathodic to the Al matrix and decrease the corrosion resistance in aqueous saline media [19] and [28].

The main effect of EN coating on the corrosion behavior of composite might be morphologic. The high pressure applied in squeeze casting might have an indirect effect on the corrosion resistance of the composite. During the solidification of composite under high pressure, some residual stresses might appear and cause an increase in the overall internal stresses in the composite. However, it has been widely accepted that these residual stresses have no or little effect on the corrosion behavior of the composite [23] and [28]. However, dislocations accumulated around Al dentrites and/or Al/SiC interface can indirectly cause a degradation of the surface oxide layer, and might be effective in the change of the corrosion morphology of the composite [37] and [38]. At the advanced stage of the corrosion, if SiC coating is a nonconductive material, the corrosion will continue around these particles since SiC is a noble material and will not undergo corrosion. Thus, localized corrosion in Al matrix alloy will progress quickly and in depth, and causes less material loss. However, if the SiC coating is a conductive material, this increases the ratio of local cathode area around the SiC particle where low resistance areas exist and electrons leave easily from these areas, therefore, corrosion will progress around the SiC particles where the pits extend at the surface not in depth. As a result, this kind of constitutions affect the normal pitting corrosion behavior of the composite. This implies that the matrix/reinforcement interfaces are active sites for corrosion and the corrosion morphology clearly demonstrates that it progresses as pitting corrosion. The initial and intensive stages of the corrosion based on Fig. 11 where the pit morphology differs at each stage. Also, as seen from Fig. 11, corrosion starts in-depth at the early stages of corrosion, and later, as a result of excessive polarization, it expanses to the surface. This can be attributed to the eutectic silicon phase, which segregates to Al dendrites during solidification [32] since Si decreases the solution potential of Al to more noble values [28]. Due to this fact, Al grain boundaries close to the Al/SiC interface, where there are relatively active sites as a result of EN coating, will become more

active. Consequently, these regions will become localized regions where the oxide film layer has a relatively high solubility and surface conductivity. Therefore, after corrosion propagates in-dept for a while, it will interact with these localized regions and will propagate around these regions, and will consequently spread out (Fig. 11). This progress will occur in a very short time and will continue until the reinforcement detach from the matrix (Figs. 12 and 13). This approach is well in agreement with the observation of Modi et al. [30]. As a result, this kind of constitutions may affect the normal corrosion behavior of the material.

The corrosion potential and polarization resistance during the immersion of a system in a corrosive medium can provide information regarding the ongoing corrosion reaction process. This information, as well as additional information about the solution/protection layer/composite surface interfaces can be obtained from EIS. Figs. 7 and 8 demonstrate the Nyquist and bode plots of the composite after the EIS scanning. At low frequency, the region of the Nyquist plot shows an approximately straight line with a slope of 45° (Fig. 7). In addition, the phase angle approaches 45° at low frequency (Fig. 8b). In this case, it can be said that the corrosion rate is controlled by the diffusion of electrochemical active species (EAS) to the composite surface and is referred as Warburg diffusion [39]. The result confirms the Nyquist plot because Warburg Impedance appears as a straight line with a slope of 45° in a Nyquist plot. Diffusion is observed at relatively low frequencies and corrosion is observed at higher frequencies (Fig. 8). This behavior demonstrates that the electron exchange between the composite and EAS is very fast. This can be attributed to a relatively high surface electrical conductivity of the Al/SiC interface because of EN coating, which supports the above discussions. As a result, the EN surface coating method significantly affects the corrosion characteristics and the morphology of the composite.

4 CONCLUSIONS

In this study, it has been observed that Al/SiC interfaces are active sites where corrosion preferentially starts in-depth at the early stages, and later, as a result of excessive polarization,

expanses to the surface, which indicates that the corrosion type is 'pitting'. Such corrosion behavior has been attributed to the conductivity of the coating material, eutectic silicon phase and dislocations accumulated around Al dendrites, by forming preferential sites for corrosion. In addition, the high pressure applied in squeeze casting might have an indirect effect on the corrosion resistance of the composite. It can be concluded that despite improving the wettability, electroless nickel coating is not an effective method to improve the corrosion resistance in structural applications containing halide solution.

ACKNOWLEDGEMENTS

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