Comparison of Potentiodynamic Polarization and Weight Loss Measurement Techniques in the Study of Corrosion Behavior of 6061 Al/SiC Composite in 3.5 M NaCl Solution

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ABSTRACT - 6061 Al/SiC composite is one of the widely used composite for structural applications in marine industry. Corrosion is the major concern in marine applications. The corrosion behavior of the aforesaid composite is determined using two techniques namely potentiodynamic polarization and weight loss measurements. It is observed the results obtained by both techniques showed similar trend. The peak aged composite show lower corrosion resistance in 3.5 M NaCl solution at room temperature compare to other groups of aged composite. The aluminum as corrosion resistive coating reduces the corrosion rate of the peak aged composite to a considerable extent.

1. INTRODUCTION

One of the main draw backs of aluminum matrix composites is the decrease in corrosion resistance compared to the base alloy used as matrix. The Base alloy has the tendency to develop a protective oxide surface film which give rise to corrosion resistance; The introduction of reinforcing phase leads to in homogeneities and can cause discontinuities in the surface film, increasing the number of sites where corrosion can be initiated and making the composite more vulnerable to corrosion attack [1-3]. Several researchers have been working on the corrosion behavior Aluminum metal matrix composite in aques solutions and the results have proved that that the major corrosion mechanism is found to be pitting and galvanic. According to the literature, the protective oxide film breakdown due to the introduction of reinforcement and the presence of precipitates of alloying elements. The study on the effect of aging on the corrosion behavior of the composite is inevitable since the corrosion behavior varies with respect to processing types, heat treatment, size and volume percentage of reinforcement, type of alloying elements and environmental conditions [4&5].

The common techniques used to determine the corrosion behavior of metals and alloys are potentiodynamic polarization technique and weight loss measurements. The electrochemical reactions are utilized to brief the corrosion phenomena of metals and alloys. Measurements of current-potential relations under predetermined conditions can give the information on corrosion rates, coatings and films, passivity, pitting tendencies and other important data. The corrosion current potential relationship is obtained in potentiodynamic polarization technique. The electrode potential is scanned carefully with respect to corrosion current in this technique. These measurements are used to determine corrosion characteristics of metals and alloy specimens in aqueous environments. The current-potential profile of a specimen measured in a short duration. Hence this technique is commonly used in the investigations such as corrosion rates, effect of protective coatings and corrosion inhibition efficiency. By this technique, the corrosion characteristics of different metals and alloys can be compared with respect to different corrosive environment which will provide the information on long term performance of different engineering components. The weight loss measurement is one the simple methods of observing corrosion of metal and alloys. The technique measures the weight loss of the specimen due to corrosion phenomena when immersed in corrosion media. When the procedures of weight loss measurements are followed carefully, accurate results can be obtained using this technique [6-8].

The present work aims at comparing the corrosion behavior analysis of 6061Al/SiC composite in 3.5 M NaCl solution when both techniques namely potentiodynamic polarization and weight loss measurements are considered.

2. EXPERIMENTAL WORK

2.1 Material

In the present work, 6061Al-15 volume percentage SiC composite material is under consideration. The composite is made of 6061Al alloy reinforced with particulate SiC (99.9% purity) and $28\mu m$ size. It was prepared by stir casting technique.

Element	Cu	Si	Mg	Ti	Mn	Fe	Cr	Zn	Al	Others total
Composition (Wt. %)	0.15- 0.40	0.40- 0.80	0.80- 1.20	0.15 max	0.15	0.70 max	0.04- 0.35	0.25 max	Bal	0.15 max

Table.1 Standard composition of Al 6061 alloy

The composition of the base metal Al 6061 alloy is given in Table.1.

2.2 Electrochemical testing

For electrochemical testing, solutionised, under aged, over aged and peak aged specimens of 3 cm length and 1.1 cm diameter are mounted in epoxy material. The specimens were polished with various grades of SiC paper, up to 1000 grade, wet polished to 3 μ m grit with diamond paste and degreased just prior to immersion in 3.5 M NaCl solution room temperatures namely 303K during electrochemical testing.

Cylindrical coupons of length 1cm and 1.1 cm diameter were cut from peak aged composite rod and were coated by aluminum using DC magnetron sputtering technique. A magnetron sputter coating system was used to deposit Aluminum layer of thickness \sim 4 Microns (Determined by Gravimetric method) on the composite substrate at room temperature so that the coating procedure did not affect the temperature history of the specimen. A high-purity Al target (99.99%) with a diameter of 33 cm was mounted on the cathode, and argon was used as the bombardment gas. Prior to deposition the sputtering module was pumped down to a base pressure of 4 x 10 $^{-5}$ Pa. The sputtering pressure was set at 0.8 Pa. The target to substrate distance was 7.5 cm and the substrate was 10 x 10 x 3 mm 6061 Al/SiC composite. The power used for Al sputtering was set at 130 W. The sputtering was performed at room temperature for 180 minutes.

Similar experiments were carried out for the composite samples coated with aluminum. Thickness of the coating is around 3.4 microns (Gravimetric determination). For the electrochemical testing, the geometric area of the specimens exposed was 1 cm². All solutions were prepared with distilled water and reagent grade chemicals. A solution volume of 500 mL was used for all tests. Electrochemical measurements were performed with a potentiostat under software control (CH Instrument, USA Model 604 A).

Polarization scans were initiated from -1100 mV versus SCE and scanned to approximately -200 mV, all potentials being relative to the open-circuit corrosion potential. Polarization curves recorded over the range -1100 to -200 to mV, from which corrosion rates were estimated. All potentiodynamic scan rates were 0.33 mV/s. Electrochemical cell was composed of a three-neck glass flask, a large area platinum counter electrode, and saturated calomel electrode (SCE) as a reference electrode. All potentials are referenced to the SCE. Polarization resistance measurements were conducted via potentiodynamic polarization measurements. Cyclic polarization curves were recorded in order to find out pitting potential (E_{pit}) and protection potential (E_{prot}).

The corrosion rate is obtained from the following expression [9]

$$CR(mpy) = \frac{0.129x \ I_{CORR} \ xEq.Wt}{D} - - - (1)$$

Where,

 I_{corr} = Corrosion current density in μ A/cm².

Eq.Wt = Equivalent weight of the corroding specimen in gm.

D = Density of the corroding species in g/cm²

 I_{corr} is obtained by dividing the corrosion current (i_{corr}) value by the area of specimen exposed to the electrolyte.

$$I_{CORR} = \frac{1}{2.3R} \left(\frac{\beta_a \beta_c}{\beta_a + \beta_c} \right) - - - (2)$$

Where,

R- Polarisation resistance ($k\Omega/cm^2$)

 β_a – Anodic slope (Volts/Decade); β_c – Cathodic slope (Volts/Decade)

2.3 Weight loss measurements

Rectangular immersion specimens of dimension 4 cm x 4 cm x 0.5 cm were ground to 1000 grit before immersion into an 3.5 M NaCl aqueous solution. The quantity of solution was as per ASTM G31 standards; [10] i.e. 40 mL of solution per 1 cm^2 of exposed specimens' area. Weight loss measurements were made every 5 days. Solution loss (due to evaporation) compensation was being carried out every two or three days by addition of double distilled water up to the initial solution level. The specimens were first water and mechanically cleaned using a brush made of plastic strings and then they were ultrasonically cleaned for 15 min. Weight loss measurements followed with an accuracy of 0.1 mg. The total immersion duration was 40 days. After the specimens' final withdrawal they were chemically cleaned in 2 wt % CrO_3 and 5 wt % H_3PO_4 aqueous solution heated at 353 K for 6-7 s according to ASTM G1. The corroded surfaces were analyzed by SEM.

The corrosion rate is calculated as [9],

$$CR (mpy) = 534 \left(\frac{W}{DxAxT}\right) - - - (3)$$

Where, W - Weight loss, mg.

D - Density of specimen, g/cm³.

A - Area of the specimen, sq inches and

T - Time of exposure in hours.

3. RESULTS AND DISCUSSIONS

3.1 Corrosion behavior of 6061Al/SiC composite

Typical polarization curves for artificially aged 6061Al-based MMC containing 15 vol% SiC_p with and without aluminum coating in 3.5 M NaCl solution at 303K are shown in Fig 1. The corrosion characteristics are presented in table 2. The results of the polarization experiments carried out in 3.5 M NaCl solution at room temperatures, indicate that the polarization curves of solutionised, over aged and under aged composite samples shift towards the left with higher zero current potential (ZCP) and lower current densities (I_{corr}) compare to the polarization curve of peak aged samples. This shows that there is an increase in corrosion rate for peak aged composite material. This was in agreement with the results obtained by other authors for aluminum alloy particulate reinforced composites [11]. Aluminum is more reactive by nature, but it has a good resistance to corrosion because of the formation of a passive oxide film. But aluminum is prone to galvanic corrosion when alloyed to more noble elements; this in turn leads to promotion of galvanic corrosion of the aluminum matrix in aluminum MMCs. The degree of galvanic corrosion depends on 1) the electrolyte to which it is exposed and 2) the reinforcement volume percentage. During fabrication of the composite there is a chance for microstructural change due to reinforcement/matrix reactions forming new inter metallic phase. Intermetallics can contribute to corrosion [12].

If the newly formed intermetallics are cathodic to the matrix, then severe corrosion will take place with respect to the matrix. Also the difference in coefficients of thermal expansion between the matrix and reinforcement generates higher dislocation density which in turn increases the corrosion rate. [13].

Table 2 Corrosion characteristics of the artificially aged composite with and without aluminum coating in 3.5 M NaCl solution (aerated) at 303 K.

Material	Corrosion potential (E corr)	Polarization resistance (R _p)	$\begin{array}{c} \text{Corrosion} \\ \text{current density} \\ \text{(I}_{\text{corr}}) \end{array}$	Corrosion rate	
	mV Vs SCE	$(k\Omega/cm^2)$	$(\mu A/cm^2)$	(Mils/year)	
Solutionized	-705	8.5	5.99	2.77	
Over aged	-729	6.05	6.31	2.91	
Under aged	-756	5.28	6.7	3.1	
Peak aged	-804	3.99	7.91	3.65	
Aluminum coated peak aged	-667	12.86	4.05	1.87	

Peak aging of the composite is achieved when GP zone formation of the secondary phase alloying elements in the matrix is uniform and fine in nature [11]. The alloying elements differ in galvanic potential with aluminum matrix and

the severity of the problem depends on to what extent they differ galvanically. The uniform distribution and fineness of the precipitates in peak aged composites might be the cause for the more number of pits observed, compared to other group of aged composites. The SEM analysis (Fig 2 to 5) shows that the peak aged composite after 40 days of immersion at room temperature in 3.5 M NaCl solution (aerated), has more pits randomly distributed in the matrix. It is very clear that the interface between the matrix and the reinforcement gets corroded in all cases regardless of the aging temperature and time history.

Since the peak aged composites are more prone to corrosion in NaCl solution, aluminum coating of thickness 3.4 microns is given on the peak aged composite and the corrosion resistance is determined using polarization technique. In 3.5 M NaCl solution at room temperature aluminum coated peak aged samples show better corrosion resistance. This is clearly visible from the Fig 2& 5. The aluminum coating reduces the corrosion rate to half of the original value of peak aged composites in 3.5M NaCl solution at room temperature.

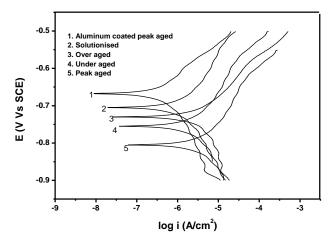


Fig 1 Potentiodynamic polarization curves of the artificially aged composite with and without aluminum coating in 3.5 M NaCl solution (aerated) at 303 K.

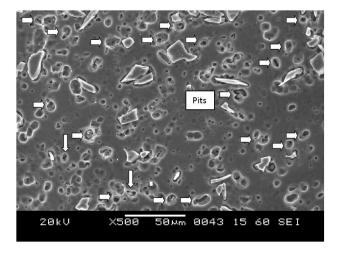


Fig 2 SEM microstructure of the peak aged composite material immersed in 3.5 M NaCl solution (aerated) at room temperature for 40 days.

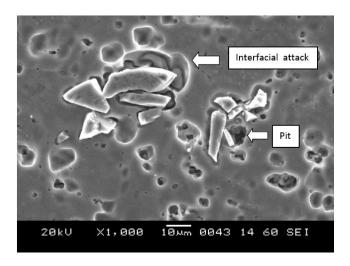


Fig 3 SEM microstructure of the peak aged composite material immersed in 3.5 M NaCl solution(aerated) at room temperature for 40 days showing interfacial attack and pits.

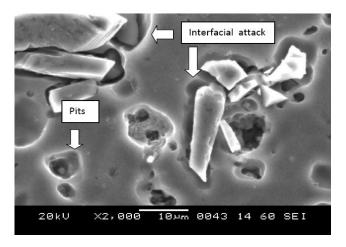


Fig 4. SEM microstructure in higher magnification of the peak aged composite material immersed in 3.5 M NaCl solution(aerated) at room temperature for 40 days showing interfacial attack and pits.

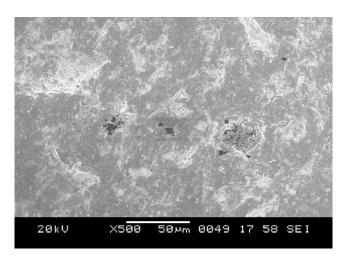


Fig 5 SEM microstructure of the aluminum coated peak aged composite material immersed in 3.5 M NaCl solution(aerated) at room temperature for 40 days.

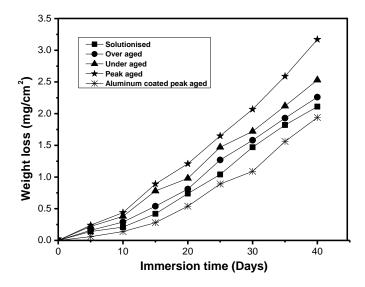


Fig 6. Weight loss versus immersion time for the composites in 3.5 M NaCl solution (aerated).

Aluminum as a monolithic coating effectively covers the interfacial regions and the precipitates of alloying elements from the corrosion media. Hence the galvanic corrosion is minimized to a large extent. This is clearly visible from the SEM picture of the aluminum coated peak aged composite specimen immersed in 3.5 M NaCl solution for 40 days (Fig 5). Hence it can be stated that the aluminum coating of thickness 3.4 microns, improves the corrosion resistance of peak aged composite.

Immersion	Weight loss in mg/cm ²					
time					Aluminum	
(Days)					coated peak	
	Solutionised	Over aged	Under aged	Peak aged	aged	
5	0.14	0.16	0.22	0.24	0.06	
10	0.21	0.29	0.38	0.44	0.14	
15	0.42	0.54	0.78	0.89	0.28	
20	0.74	0.81	0.98	1.21	0.54	
25	1.04	1.27	1.47	1.65	0.89	
30	1.47	1.58	1.72	2.07	1.09	
35	1.82	1.93	2.12	2.59	1.56	
40	2.11	2.26	2.53	3.17	1.94	

Table 3. Weight loss measurements in 3.5 M NaCl solution (aerated).

Table 4. Comparison of corrosion rates by different techniques

Material	Corrosion rate in 3.5 M NaCl solution (aerated) in mpy				
	Polarization technique	Weight loss measurement			
Solutionised	2.77	2.74			
Over aged	2.91	2.93			
Under aged	3.1	3.28			
Peak aged	3.65	4.12			
Aluminum coated peak aged	1.87	2.52			

The results of weight loss measurements (Fig 6, table 3&4) are almost in agreement with the results obtained by polarization technique. Table 4 shows the corrosion rates of aged composites and aluminum coated peak aged composite calculated in mpy, by potentiodynamic polarization method and weight loss measurements. Weight loss measurements also indicate that the peak aged composite has more corrosion rate when compared to that of other groups of aged

composites. When aluminum coating is applied on the composite, the galvanic corrosion is reduced since the coating covers the interface regions and the alloying elements precipitates on the surface of the matrix. Hence the corrosion prone regions are unexposed to the electrolyte leading to lower corrosion rate. The corrosion rates obtained by weight loss measurement technique for peak aged composite and the aluminum coated composite are falling slightly higher when compared to that of potentiodynamic polarization technique. This may be due to more exposing time of the composite specimen in the electrolyte in the case of weight loss measurement. But the results of the later mentioned technique backs the results obtained in polarization method in all respect.

4. CONCLUSIONS

- 1. Potentiodynamic polarization technique and weight loss measurement show that the peak aged composite is more prone to corrosion in 3.5M NaCl solution at room temperature when compared to other groups of aged composites.
- 2. Both techniques show that the aluminum coating can be effectively used as corrosion resistive coating to improve the corrosion resistance of 6061 Al/SiC in 3.5 M NaCl solution at room temperature.
- 3. Slight variation is observed in the corrosion rates obtained by both techniques due to more of exposure time of the composite to electrolyte in the case of weight loss measurements.

5. REFERENCES

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