Effect of TiN particulate reinforcement on corrosive behaviour of aluminium 6061 composites in chloride medium

H C ANANDA MURTHY*, V BHEEMA RAJU[†] and C SHIVAKUMARA[‡]

Department of Chemistry, R N Shetty Institute of Technology, Rajarajeshwari Nagar, Bangalore 560 098, India

[†]Department of Chemistry, Dr. Ambedkar Institute of Technology, Bangalore 560 056, India

[‡]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, India

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Abstract. In the present investigation, the corrosive behaviour of Al 6061–TiN particulate composites prepared by liquid metallurgy has been studied in chloride medium using electroanalytical techniques such as Tafel, cyclic polarization and electrochemical impedance spectroscopy (EIS). Surface morphology of the sample electrodes was examined using scanning electron micrography and energy dispersive X-ray methods. X-ray diffraction technique was used to confirm inclusion of TiN particulates in the matrix alloy and identify the alloying elements and intermetallic compounds in the Al 6061 composites. Polarization studies indicate an increase in the corrosion resistance in composites compared to the matrix alloy. EIS study reveals that the polarization resistance (R_p) increases with increase in TiN content in composites, thus confirming improved corrosion resistance in composites. The observed decrease in corrosion rate in the case of composites is due to decoupling between TiN particles and Al 6061 alloy. It is understood that after the initiation of corrosion, interfacial corrosion products may have decoupled the conducting ceramic TiN from Al 6061 matrix alloy thus eliminating the galvanic effect between them.

Keywords. Composites; corrosion; SEM; EDX; EIS

1. Introduction

Particulate-reinforced aluminium metal matrix composites (Al MMCs) and other aluminium-based materials having a wide range of applications in diverse fields such as light weight automotive structures, forgings for suspension, chassis, as well as advanced automotive components are exposed to a wide variety of corroding environments. Alloys of Al reinforced with ceramic oxides, carbides, nitrides and mineral silicate particulates possess attractive characteristics such as high specific modulus, high specific strength, low thermal expansion coefficient, light weight and low cost (Berghezan 1966; Kelly 1967; Chawla and Chawla 2006) and superior corrosion resistance.

Studies on aluminium alloys reinforced with SiC (Clyne and Withers 2001), B (Agarwal and Broutman 1980), Al₂O₃ (Chawla and Chen 2001), TiC (Kainer 2006) and ZrB₂ (Froyen and Verlinden 1994) report lower corrosion resistance for the composites compared to matrix alloys, owing to galvanic corrosion. On the other hand, Al composites reinforced with garnet (Trumper 1987), albite (Johansen 2010), quartz (Nair *et al* 1985) and glass fibre (Pohlman 1978) exhibited higher corrosion resistance compared to their matrix alloys. Recent studies on the corrosion resistance of TiB₂ particulate-reinforced A356 alloy show a marked

decrease with increase in TiB₂ content as reported by Sun *et al* (Paciej and Agarwala 1986). In the case of TiC–Al 2024 composites, the TiC reinforcement is reported to decrease the anodic current density as well as amount and size of the pit (Trowsdale *et al* 1996). AlN particles which are highly insulating are reported to increase the susceptibility to pitting attack attributable to microgalvanic coupling between the matrix and reinforcement and hydrolysis of AlN particles (Pfeifer 1977). The corrosion resistance of Al–Mg and Al–Cu composites is found to be higher than that of composites reinforced with mica particles (Trzaskoma *et al* 1983). The conflicting results can possibly be explained by differences in fabrication methods and composition which yield dramatically different electrochemical behaviours (Griffiths and Turnbull 1994).

The observed variation of corrosion resistance in Al MMCs is attributed to chemical or mechanical factors such as composition of the matrix alloy, nature of reinforcing particles, fabrication methods, chemical or mechanical factors such as alloying, segregation, interfacial reactions, oxidized layers, residual stress around reinforced particles in the matrix and galvanic coupling between matrix and reinforcement.

The corrosive behaviour of reinforced Al composites investigated in acidic, neutral, alkaline and various salt media reveal that Al composites suffer greater localized pitting corrosion in chloride ion environment compared to other media. Thus since Al MMCs lose their mechanical properties due to corrosion and lead to failure during service, research on

*Author for correspondence (anandkps7@yahoo.com)

the corrosive behaviour is equally important as studies on fabrication and mechanical behaviour of Al MMCs.

The present investigation involves the study of corrosive behaviour of unreinforced Al-6061 matrix and its TiN particulate composites in NaCl medium. Titanium nitride is a hard, dense and refractory material with low electrical resistivity ($\sim 10^{-7}$ ohm cm) and is isomorphous with TiC. TiN is a good reinforcement for aluminium matrix, as it is known to enhance corrosion resistance of the matrix. The main objective of the present study is to establish the role and effectiveness of TiN on the corrosive behaviour of Al 6061 composites.

2. Experimental: Material selection

2.1 Aluminium alloy 6061 matrix

Aluminium alloy 6061 had the composition: Si – 0.6%, Fe – 0.1%, Cu – 0.3%, Mn – 0.02%, Mg – 0.8%, Zn, Cr, Ti – 0.01% each and remaining Al.

2.2 Reinforcement

TiC of A.R. grade was obtained from Sigma Aldrich and used as reinforcement in the form of particulates.

2.3 Composite preparation

The liquid metallurgy route using vortex technique was employed to prepare the composites. Addition of reinforcement material TiO_2 into the molten Al-6061 alloy melt was carried out by creating a vortex in the melt using a mechanical stainless steel stirrer coated with alumina (to prevent migration of ferrous ions from the stirrer material to the alloy). The stirrer was rotated at a speed of 450 rpm in order to create the necessary vortex. TiO_2 particles were pre-heated to 400 °C and added into the vortex of liquid melt at a rate of 120 g/min. TiO_2 particulates were in the size range of 50–80 μm . The weight percentage of TiO_2 used was 2–6 in steps of 2%. The composite melt was thoroughly stirred and subsequently degasifiers were added. Castings were produced in permanent moulds in the form of cylindrical rods (diameter, 30 mm and length, 150 mm).

2.4 Specimen preparation

Cast material was cut into 20 × 20 mm cylindrical pieces using an abrasive cutting wheel. The matrix alloy was also cast under identical conditions as composites, for comparison. The samples were successively ground using 240, 320, 400, 600, 800, 1000, 1500 and 2000 grade emery papers and polished according to standard metallographic techniques and degreased in acetone and dried. The samples were weighed up to fourth decimal place using electronic balance and the exact specimen dimensions were noted down. For polarization studies, composites and pure alloy matrix were

cut into rectangular specimens of 2 cm length, 1 cm width and 1 mm thickness and prepared as described above.

3. Results and discussion

3.1 Polarization measurements

Titanium nitride is a hard, dense and refractory material with low electrical resistivity ($\sim 10^{-7}$ ohm cm) and is isomorphous with TiC. Also, TiN particles have poor wettability with liquid aluminium. The open circuit potentials (OCP) were recorded for Al6061 matrix alloy and its TiN composites (2, 4 and 6 wt%) in three different concentrations of neutral NaCl media, viz. 0.1 N, 0.5 N and 1.0 N solutions. The plots for the matrix alloy and the composites in decinormal NaCl medium is shown in figure 1 and the evaluated open circuit potentials are given in table 1. It is observed that the OCP values increase in positive direction with increase in TiN content, from -0.8062 V for Al 6061 matrix to -0.7487 V for reinforced composites (6 wt% TiN) in 0.1 N NaCl medium. Similar increase in OCP values in the positive direction with increase in TiN content is observed in 1 N NaCl medium, from -0.8987 V for Al 6061 matrix to -0.7761 V for reinforced composites (TiN – 6 wt%).

OCP values shift considerably towards negative direction for Al 6061 matrix alloy and its TiN composites, as shown in figures 2 and 3. The increase in OCP value in the cathodic direction is about 90 mV as the concentration of NaCl is increased by one order of magnitude, for the matrix alloy as well as the composites. However, this increase in OCP value of 90 mV for TiN reinforcement is lower when compared to the corresponding increase of 140 mV for Al– TiO_2 composites.

Typical polarization curves for Al 6061 matrix alloy and composites containing 2, 4 and 6% by weight of TiN particulates in different concentrations of NaCl are shown in figures 4–6. The evaluated electrochemical corrosion parameters for the matrix alloy and the composites are

Figure 1. Open circuit potentials for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 0.1 N NaCl medium.

Table 1. Open circuit potentials of Al 6061 matrix alloy and its TiN composites in various concentrations of NaCl media.

TiN content in composites	OCP (E_{corr}) values (in volts) in various NaCl media		
	0.1 N	0.5 N	1.0 N
0%	-0.8062	-0.8594	-0.8987
2%	-0.7678	-0.8398	-0.8531
4%	-0.7562	-0.7568	-0.8007
6%	-0.7487	-0.7503	-0.7761

Figure 2. Open circuit potentials for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 0.5 NaCl medium. **Figure 3.** Open circuit potentials for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 1.0 NaCl medium.

given in tables 2 and 3. The corrosion parameters, corrosion current density (I_{corr}) and corrosion rate were obtained from the Tafel polarization measurements. It can be observed from the Tafel plots and the tables that corrosion current values and corrosion rates decrease with increase in TiN content in the composites, in each of the NaCl concentrations. Cyclic polarization curves for Al 6061 matrix alloy and composites containing 2, 4 and 6% by weight of TiN particulates, in 0.5 N NaCl solution are shown in figure 7. Cyclic polarization curves of the reinforced and unreinforced Al alloys

Figure 4. Tafel polarization plots for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 0.1 NaCl medium. **Figure 5.** Tafel polarization plots for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 0.5 NaCl medium.

were found to be similar in overall shape. Cyclic polarization measurements show that the pitting potential increases towards positive direction with increase in TiN content confirming the results from other measurements that composites of TiN are more corrosion resistant than the matrix alloy.

Open circuit potential measurements revealed that the corrosion susceptibility of the matrix alloy decreases with increase in TiN content in the composites. TiN reinforcement particles thus impart good corrosion resistance to the Al-TiN composites with an increase in the concentration of the chloride medium from 0.1 N to 0.5 N to 1.0 N NaCl. Our results suggest the presence of lower percentage of TiN particulates

Figure 6. Tafel polarization plots for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 1.0 NaCl medium.

Figure 7. Cyclic polarization plots for Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites in 1.0 NaCl medium.

Table 2. Corrosion current densities (I_{corr}) of Al 6061 matrix alloy and its TiN composites in various concentrations of NaCl media.

TiN content in composites	Corrosion current density, I_{corr} (Acm^{-2})		
	0.1 N NaCl	0.5 N NaCl	1.0 N NaCl
0%	6.626×10^{-7}	7.653×10^{-7}	1.006×10^{-6}
2%	4.290×10^{-7}	5.626×10^{-7}	6.790×10^{-7}
4%	1.597×10^{-7}	2.605×10^{-7}	3.643×10^{-7}
6%	1.564×10^{-7}	1.596×10^{-7}	2.067×10^{-7}

Table 3. Corrosion rates of Al 6061 matrix alloy and its TiN composites in various NaCl media.

TiN content in composites	Corrosion rate (milli inches per year)		
	0.1 N NaCl	0.5 N NaCl	1.0 N NaCl
0%	0.2842	0.3283	0.4300
2%	0.1840	0.2410	0.2960
4%	0.0841	0.1110	0.1560
6%	0.0670	0.0684	0.0886

in composites which could be attributed to its poor wettability as compared to TiO_2 particles (Rhee 1970). The conducting TiN particulate possibly forms microgalvanic couple with Al 6061 matrix alloy and causes pitting corrosion. The observed decrease in corrosion rate in the case of composites is due to decoupling between TiN particles and Al 6061. It seems that after the initiation of corrosion, interfacial corrosion products may have decoupled the conducting ceramic TiN from Al 6061 matrix alloy, thus eliminating the galvanic effect between them (Srinivasan 2005).

Increase in the corrosion susceptibility of both Al 6061 matrix alloy and composites at higher concentrations of aggressive chloride ions is probably due to the breakdown of stable oxide film on Al 6061 matrix as a result of increased adsorption of chloride ions. This indicates the lowest corrosion tendency for the TiN (6 wt%)-reinforced composites as compared to matrix alloy. The results show that the

increase in Cl^- ion concentrations yields more cathodic OCP values confirming enhanced adsorptivity of Cl^- ions in both matrix alloy and its TiN composites. This corroborates the report by earlier researchers suggesting greater degree of penetration ability of chloride ions into the passive hydrated oxides of aluminium as well as reduction in the kinetics of oxide film repair.

Tafel polarization results show that TiN-reinforced composites have lower corrosion rate as compared to matrix alloy. Observations also show that both the I_{corr} values and corrosion rate decrease with increase in content of TiN and increase with an increase in concentration of NaCl medium. This observation contradicts the composite theory of corrosion according to which reinforcement particles cause cracks and discontinuity on the surface of metal/alloy (Trowsdale *et al* 1996). It is observed that the interface between Al 6061 matrix and TiN reinforcement is stronger as reported

(Sharma 2001) in the study of corrosive behaviour of Al 6061/albite composite. In the unreinforced Al alloy, the possible cathodic sites could be eutectic silicon and/or intermetallic precipitates; however, in the composites, cathodic sites may be reinforcements, second-phase precipitates or interfacial reaction products (Nunes and Ramanathan 1995).

The presence of a more conductive phase at the interface provides an easier path for the electron exchange necessary for oxygen reduction and drives the anodic reaction at a higher rate in MMCs than aluminium alloy. Improved corrosion resistance has been reported (Wlodarczyk-Fligier *et al* 2010) in Al composites reinforced up to 5% Ti(C, N) particulates when compared with the matrix alloy in 3% NaCl medium. This corroborates the prediction that presence of a small amount of TiN reinforcement in composites fails to rupture the oxide layer of Al. It is believed that the nature of the passive film formed on the TiN composites is sufficiently stable to reduce the corrosion rate after few min

of immersion (Alaneme 2011). It has been reported that in the case of TiN-reinforced Al MMCs fabricated by powder metallurgical route (Ray *et al* 2002) that the presence of TiN-particles at grain boundaries played a significant role in its densification and improved mechanical as well as wear resistance properties. The improved corrosion resistance in TiN-reinforced Al 6061 composites compared to matrix alloy can be attributed to good interface bonding between the ceramic TiN and Al matrix alloy (Das 2004). TiN particle reinforced composites exhibit lesser surface irregularities which is due to refined microstructure as observed during the study of electrochemical behaviour of haematite particulates reinforced Al alloys (Jagadeesh *et al* 2005).

A probable explanation for the observed trend in pitting corrosion could be that the pitting is initiated at random sites of imperfections on the protective oxide layer of the matrix alloy, while in the composites, pits around the reinforcement particles coalesce to form a crevice. The active nature of the crevices would cathodically protect the remainder of the matrix and restrict pit formation and propagation (Garrard 1994). It is found that E_{RP} values increase in the noble direction with increasing TiN content in composites. This points to the fact that pit propagation in the composites is retarded more significantly than in the Al 6061 matrix alloy. The higher repassivation potential exhibited by the TiN-reinforced composites is attributable to the reduction in the pit dissolution kinetics by the oxides of metals. However, the repassivation potentials are more negative to the pitting potentials for each of the composites as well as the Al 6061 matrix alloy indicating a tendency to undergo pitting. ΔE_1 is the direct measure of pitting susceptibility and is used to assess the repassivation behaviour of propagating pits, the ease with which local active sites can be eliminated.

Figure 8. SEM micrograph of Al 6061 matrix alloy.

Figure 9. SEM micrograph of TiN particles.

Figure 10. SEM micrograph of corroded Al 6061 matrix alloy.

Figure 11. SEM micrograph of corroded Al 6061-TiN (2 wt%) composite.

The pitting loop given by ΔE_1 is observed to decrease from the matrix alloy to the composites and thus with increasing TiN content, a decreasing tendency to pitting in the TiN-reinforced composites is observed. However, the magnitude of change in pitting potential was found to be small (59 mV) varying from -0.7007 V in the matrix alloy to -0.6597 V in the composite with highest content of TiN reinforcement (Murthy *et al* 2010).

Pitting corrosion resistance of the TiN particle reinforced composites has been observed to increase with increasing

Figure 12. SEM micrograph of corroded Al 6061-TiN (4 wt%) composite.

Figure 13. SEM micrograph of corroded Al 6061-TiN (6 wt%) composite.

TiN content as reported by others (Kiourtsidis and Skolianos 2007). The ΔE_2 value of 93.6 mV in Al matrix alloy is found to increase to 159.5 mV in Al-6 wt% TiN composite. It can be seen that the addition of TiN to Al matrix significantly increases the passive range between the unreinforced matrix alloy and the reinforced composites.

3.2 Scanning electron microscopy

Scanning electron microscopy (SEM) images of the Al 6061 matrix alloy before corrosion test and reinforcement TiN particles are shown in figures 8 and 9, respectively. The

Figure 14. Back-scattered electron SEM of Al 6061–TiN (6 wt%) composite showing regions of Al–Fe–Si-rich phase and TiN particles.

Figure 15. EDX spectrum of interface between TiN and Al 6061 matrix.

particle size of reinforcement TiN particulates is about 2–10 μm as observed in the SEM image. SEM image of Al 6061 matrix alloy and its TiN (2, 4 and 6 wt%) composites taken after polarization studies in decinormal chloride medium, after usual pretreatment, are presented in figures 10–13. A comparison of the SEM images of the samples before and after the polarization studies clearly indicates severe surface deterioration due to pitting corrosion in all the samples. Pitting susceptibility for aluminium alloys is mainly affected by the microstructural heterogeneity of these commercial alloys. The decrease in corrosion deterioration of composites can be attributed to matrix-reinforcement interfaces and formation of micro/sub-micro local cells at the metal surface.

Figure 16. XRD pattern of TiN particulates.

A rapid dissolution of aluminium within the pit tends to produce an excess of positive charge in this area. The migration of chloride ions towards the pit increases the concentration of AlCl_3 in the pit. Since the solubility of oxygen is virtually zero in concentrated solution, no reduction of oxygen occurs within the pit. Cathodic oxygen reduction on surfaces adjacent to pits tends to suppress the corrosion thereby protecting the matrix metal adjacent to the pit (Ramachandra and Radhakrishna 2006).

3.3 Energy dispersive X-ray analysis

Energy dispersive X-ray (EDX) analysis spectra shows the presence of various elements in the TiN-reinforced Al 6061

Figure 17. XRD pattern of TiN phase in Al 6061-TiN (6 wt%) composite.

Figure 18. Nyquist plots of Al matrix alloy and its TiN composites in 0.5 N NaCl medium.

surface. The back-scattered SEM image of TiN (6 wt%)-reinforced Al 6061 sample is shown in figure 14. The presence of less intense peaks for the elements Ti and N in the EDX spectrum of TiN (6 wt%)-reinforced Al 6061 composite confirms the incorporation of TiN particles into Al matrix, but only to a small extent as it exhibits low wettability.

Even though galvanic coupling is assumed to be established between conductive TiN and Al matrix alloy, the extent of corrosion is insignificant (Adler *et al* 2008) even at 6 wt% of TiN particles in composites. It is observed that the matrix alloy and composites contain Al-Fe-Si-rich and Mg_2Si intermetallic phases.

The elements in these intermetallic phases undergo oxidation to yield their respective oxides such as SiO_2 , FeO and MgO, which reduce the corrosion rate in composites by decreasing the microgalvanic coupling between conducting intermetallic phases and matrix alloy. The presence of SiO_2 , FeO and MgO at the interface between TiN and Al 6061 is confirmed by EDX spectrum which showed intense peaks for Si, Fe, Mg and O as shown in figure 15. Corrosion resistance is more pronounced in TiN-reinforced composites as they contain higher amount of intermetallic phases due to the possible increased reactivity between the reinforcement particle and other alloying elements.

3.4 X-ray diffraction studies

X-ray diffraction (XRD) pattern of the composite (6 wt% TiO_2 -reinforced) and that of TiN particulates is given in figures 16 and 17. XRD spectra of the composite clearly shows major 2θ peaks of TiN (36.87° , 42.78° and 61.98°) in the TiN (6%)-reinforced Al 6061 composite indicating the incorporation of reinforcement particulates in the matrix alloy.

3.5 EIS studies

EIS study of the matrix alloy and its composites is carried out at OCP in order to assess the contribution of oxide film on Al to the corrosion resistance of the composites. Nyquist plots of the Al 6061 matrix alloy and its TiN composites in 0.5 N NaCl medium is shown in figure 18. It can be observed from the Nyquist plots that radius of the capacitive loops above the real axis increased with increase in TiN content for composites.

Electrochemical parameters obtained in EIS studies of Al 6061 matrix alloy and its TiN composites are given in table 4.

Table 4. Electrochemical parameters for Al 6061 matrix alloy and its TiN composites obtained from EIS studies.

TiN content in composites	R_s (ohm)	R_p (ohm)	CPE (F)
0%	2.843	13208	9.42×10^{-6}
2%	2.981	15680	1.59×10^{-6}
4%	3.873	23768	7.83×10^{-7}
6%	4.432	29831	5.94×10^{-7}

Based on the analysis of the impedance spectra, an equivalent circuit model $R[QR(LR)]$ was proposed for both the matrix alloy (figure 19) and composites (figure 20), simulating their electrochemical behaviour.

It can be found that the impedance spectra of all the samples are dominated by capacitance of the oxide film, but the diameters of the capacitance arcs increase with increase in TiN content, which means that the resistance of the surface oxide film on the composites increases with increase in TiN reinforcement.

This can be attributed to the fact that the hydroxide formed by the oxides of Si, Mg and Al plays an inhibitive role around conducting TiN and intermetallic phases. The equivalent circuit consists of a constant phase element (CPE), Q , in parallel with the parallel resistors, R_t (charge transfer resistance) and R_L (inductance resistance) and the latter is in series with the inductor L . In this case, the polarization resistance can be calculated using the formula as given below:

$$R_p = \frac{R_L \times R_t}{R_L + R_t}$$

Figure 19. Equivalent circuit model used to fit the experimental data of Al 6061 matrix alloy in 0.5 N NaCl medium.

Figure 20. Equivalent circuit model used to fit the experimental data of Al 6061-TiN (6 wt%) composite in 0.5 N NaCl medium.

Impedance spectroscopic studies show decreasing trend of polarization resistance, R_p , on increase of TiN reinforcement.

The measured value of polarization resistance, R_p , increases while the CPE value decreases with increase in TiN content. This shows that the corrosion rate decreases with increase in the content of TiN reinforcement. It is also observed that the R_p value increases with increase in TiN content confirming improved corrosion resistance in composites. These results complement polarization tests and are similar to the TiO_2 composites system.

4. Conclusions

The corrosion behaviour of unreinforced Al 6061 matrix alloy and its composites reinforced with TiN (2, 4 and 6 wt%) were studied in 0.1 N, 0.5 N and 1 N NaCl media. Corrosion rates of the matrix alloy and the composites from the polarization curves, show a decrease with increase in the content of TiN reinforcement. Increased corrosion resistance in composites is believed to be due to reinforcement particulates modifying the microstructure of matrix and also acting as physical barrier to the initiation and development of pitting corrosion. Increase in the corrosion susceptibility of both Al 6061 matrix alloy and the composites at higher concentrations of aggressive chloride ions is probably due to the breakdown of stable oxide film on Al 6061 matrix as a result of increased adsorption of chloride ions. XRD and EDX analysis of all the composites confirm positive inclusion of the reinforcement particulates in the matrix alloy and also the presence of intermetallic phases such as Al-Fe-Si-rich and Mg_2Si formed at matrix-reinforcement interface. SEM pictures reveal severe deterioration of the surface of both matrix alloy and its composites of all the reinforcements. The measured value of polarization resistance, R_p , increases while the CPE value decreases with increase in content of reinforcement in all the cases.

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