

# CORROSION STUDIES OF SILICON CARBIDE (SiC) REINFORCED ALUMINIUM 7075 METAL MATRIX COMPOSITES (MMCS) IN ACIDIC AND ALKALINE MEDIA

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**Abstract:** The corrosion behavior of Aluminium 7075 reinforced with SiC metal matrix composites was investigated in different concentrations of phosphoric acid medium and potassium hydroxide medium at 25°C. The study was done by electrochemical method using Tafel polarization techniques and electrochemical impedance spectroscopy (EIS) technique. The surface morphology was investigated using scanning electron microscope (SEM). The results showed that the Aluminium 7075 matrix undergoes intense corrosion in sodium hydroxide medium than in phosphoric acid medium. The corrosion rate of SiC reinforced Aluminium 7075 composites increases with an increase in the concentration of acid as well as alkali. The corrosion rate decrease with increase in the percentage of SiC particulate in Aluminium 7075. Desirable reaction mechanism was proposed for the corrosion of Aluminium 7075 in phosphoric acid medium and potassium hydroxide medium. The results obtained by Tafel polarization and electrochemical impedance spectroscopy (EIS) technique were in good agreement with each other.

**Keywords:** Corrosion; Aluminium 7075; MMCs; Phosphoric acid; Potassium hydroxide; Tafel polarization; EIS

## 1. INTRODUCTION

Composites have extended the horizon of engineering materials beyond the realm of natural combinations to man-made combinations. The conventional divisor of materials into metals, ceramics and polymers have been bridged by human ingenuity, developing metal/ceramic, ceramic/polymer and metal/polymer composites possessing a combination of properties, which cannot be obtained in one of the constituents alone (1). Modern composites constitute an important class of design and weight efficient structural materials that are encroaching every sphere of engineering applications. Superior directional properties, high specific strength and stiffness properties, design tailoring, ease of manufacturing complex shapes as well as scores of other attributes make these materials ideally, “Designers choice”.

Metal matrix composites (MMCs) possess significantly improved properties including high specific strength; specific modulus, damping capacity and good wear resistance compared to unreinforced alloys (2). A metal matrix composite (MMC) is composite material with at least two constituent parts, one being a metal. The other material may be a different metal or another material, such as a ceramic or organic compound. When at least three materials are present, it is called a hybrid composite.

MMCs are made by dispersing a reinforcing material into a metal matrix. The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together (3). The matrix is usually a lighter metal such as aluminum, magnesium, or titanium, and provides a compliant support for the reinforcement. The reinforcement material is embedded into the matrix. It is used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity.

Corrosion, which is a severe problem faced by almost all industries can be considered as one of the worst technical calamities of our time. Corrosion is a serious problem it definitely contributes to the depletion of our natural resources. Now-a-days corrosion control has been improved, due to increasing use of metals in all field of technology (4).

Corrosion study of matrix and composites have received considerable attention by researchers because of their wide range of industrial applications and economic considerations. (Christian Vargel, 2004; costing and Heins, 1931; Paul and Sigwalt Juniere, 1964). MMCs are made by dispersing a reinforcing material into a metal matrix. The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. The matrix is usually a lighter metal such as aluminum, magnesium, or titanium, and provides a compliant support for the reinforcement. The reinforcement material is embedded into the matrix. It is used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity (5). The need for housing, energy generation and transportation in the developing countries is driving the development of metal matrix composites into the developed world. There is also a great increase in the metal matrix composite research in some developing countries like China, India, Egypt and Argentina. These efforts recognize the fact that the ease of production, economical costs and the strength and durability of metal matrix composites make them the ideal raw material for low cost long lasting products (6).

Due to their wide applications, they frequently come in contact with acids or alkali during de-scaling, pickling, electrochemical etching and extensively used in many chemical industries. Most of the reported studies were conducted on corrosion of various metals and alloys in HCl and H<sub>2</sub>SO<sub>4</sub> media (Paul and Sigwalt Juniere, 1964; Ating et al., 2010; Umoren et al., 2009; Obi-Egbedi et al., 2012; Nnanna et al., 2011) (7).

Phosphoric acid medium is widely used for acid cleaning and electro polishing of aluminium (Christian Vargel, 2004). Potassium hydroxide is usually used for degreasing purpose. According to the available literature, not much study has been done regarding the corrosion behaviour of Aluminium 7075 reinforced composites in phosphoric acid medium as well as with sodium hydroxide medium. As a part of our studies with corrosion behavior of Aluminium 7075 matrix and SiC reinforced with Aluminium 7075 in phosphoric acid medium and Potassium hydroxide medium were studied and we report herein the results of corrosion behavior of Aluminiummatrix and reinforced with SiC particulates in phosphoric acid and Potassium hydroxide medium of different concentrations at 25°C (8).

## 2. METHODS

### — Materials

The experiment were performed with specimens of Al 7075 matrix and Al 7075 containing 2, 4, 6 percentage of SiC particulates of size 60-80µm. The composition of Aluminium 7075 is given in Table 1. Cylindrical test specimens were sealed with Teflon resin material in such a way that the area exposed to the medium was 1.0 cm<sup>2</sup>. It was polished with 280, 600, 1000 and 2000 grade emery papers. Further polishing done with disc polisher using levigated alumina to get mirror surface (9). It was then dried and stored in desiccators to avoid moisture before using it for corrosion studies.

Table 1: Composition (wt%) of Aluminium 7075 alloy

ELEMENT	Ti	Cr	Mn	Fe	Si	Cu	Mg	Zn	Al
PERCENTAGE	0.15	0.2	0.4	0.5	0.5	1.8	1.9	3.25	Bal

### —Preparation of composites by liquid melt metallurgy tecnique

The composites were prepared by liquid melt metallurgy technique through the process of Vortex method. Figure 1 is the furnace used for the preparation of the composite is basically an electrically heated 3-phase resistance furnace of 12KW capacity fitted with 3 pairs of 14 guage kanthal A1 grade heating coils. The furnace is fitted with an alumina crucible at its center with an opening at bottom and melt can be introduced directly into the mould. The furnace can be tilted at 90 degrees on its horizontal axis.



Figure 1: Bottom pour furnace

A muffle furnace was used to preheat the SiC particulates to an optimum temperature of about 400°C and maintained at that temperature till it was introduced into the molten alloy melt. The preheating of the reinforcement is necessary in order to reduce the temperature gradient between the molten metal and the reinforcement to achieve better bonding by reducing difference in the surface energy (10). AL-7075 alloy has a melting point range of 580°-660°C. The melt was superheated to a temperature of 630°C and maintained at that temperature. A stainless steel impeller / stirrer coated with aluminite coating in order to prevent the migration of ferrous ions from the stirrer material to the melt is introduced in to the melt.

The impeller used for stirring was of centrifugal type with 3 blades welded at 45° inclination and 120° apart. The stirrer was rotated at a speed of 500rpm and a vortex was created in the melt. The depth of stirring, and degassing parameters like gas flow rate, pressure and time of flow were standardized. This was done as basic study by using water module studies, and base metal was used to optimize the other parameters for better soundness and mechanical properties. After the addition of the particulates, the melt was degassed by using pure nitrogen for about 3-4 minutes in to melt and pour directly in to the preheated mould. Following this procedure, AL-7075/SiC particulate composites of 0%, 2%, 4% and 6% by weight were prepared. The prepared composites are subjected to evaluation of important weight loss test, open circuit potential test, potentiodynamic studies, stress corrosion test, mechanical behavior in both as cast and heat treated condition and micro structural studies (11).

— **Medium**

A stock solution of phosphoric acid medium was prepared using analytical grade phosphoric acid medium (85%) and double distilled water. Phosphoric acid medium concentrations 0.5M, 0.75M and 1.0M was prepared by appropriate dilution. A stock solution of Potassium hydroxide was prepared by dissolving analytical grade potassium hydroxide pellets in double distilled water and standardized by volumetric method using phenolphthalein indicator (12). Solutions of required strengths 0.5M, 0.75M and 1.0M were prepared by appropriate dilutions as and when required. Experiments were carried out at 25°C.

— **Electrochemical measurements**

Electrochemical measurements were carried out by using an electrochemical work station, CH608E-series, Instrument (USA) with CH instrument beta software shown in Figure 2. Polarization measurements were carried out using conventional three electrode assembly having Pyrex glass cell with platinum counter electrode and saturated calomel electrode (SCE) as reference electrode. The working electrode was made up of Al7075 matrix and composites. All the values of potential values were measured with reference to the saturated calomel electrode (13). The

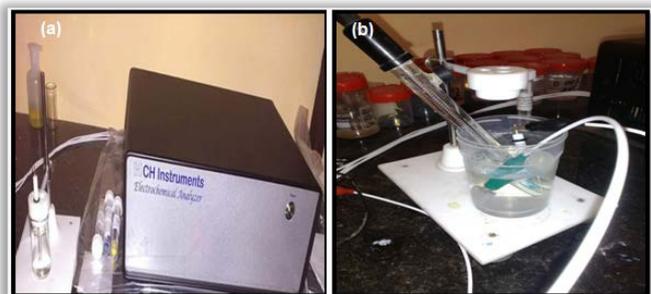


Figure 2. (a) The Electrochemical work station and (b) Experimental setup

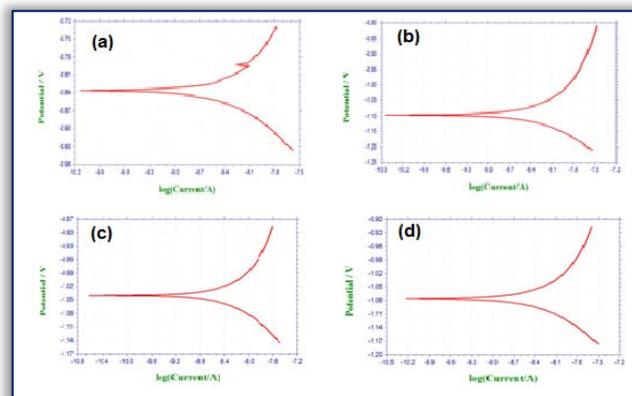


Figure 3. Tafel polarization curve for Al7075 in 0.5N H<sub>3</sub>PO<sub>4</sub> containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

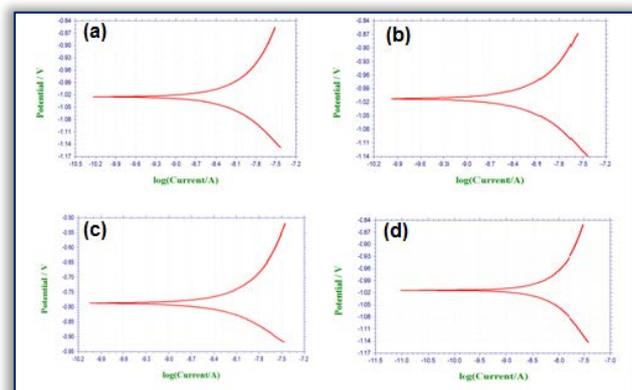


Figure 4. Tafel polarization curve for Al7075 in 0.75N H<sub>3</sub>PO<sub>4</sub> containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

polarization curves were recorded immediately after the EIS studies on the same electrode continuously one after the other.

≡ **Tafel polarization studies**

Finely polished Al 7075 specimens of 1.0 cm<sup>2</sup> surface area were exposed to corrosion medium of different concentrations of phosphoric acid (0.5N, 0.75N and 1.0N) potassium hydroxide (0.5N, 0.75N and 1.0N) separately at 25°C. The potentiodynamic current- potential curves were recorded by polarizing the specimen to -250 mV cathodically and +250 mV anodically with respect to open circuit potential (OCP) at scan rate of 5 mV/s (14).

≡ **Electrochemical impedance spectroscopy (EIS) studies**

Electrochemical impedance spectroscopy (EIS), which gives early information about the electrochemical processes, at the metal solution interface, has been used in many reports on the corrosion studies (15). The corrosion behavior of the Al 7075 matrix and composites were also obtained from EIS technique using electrochemical workstation, CH608E- Instrument (USA). In EIS technique a small amplitude ac signal of 10 mV and frequency spectrum from 100 kHz to 0.01Hz was impressed at the OCP and impedance data were analyzed using Nyquist plots. The charge transfer resistance,  $R_t$  was extracted from the diameter of the semicircle in Nyquist plot. In all the above measurements, at least three similar results were considered and their average values are reported (16).

— **Scanning electron microscopy (SEM)**

The scanning electron microscope images were recorded to establish the interaction of acid medium and alkaline medium with the metal surface using Analytical scanning electron microscope. The surface morphology of Aluminium 7075 matrix and composites were immersed in phosphoric acid medium and potassium hydroxide medium was compared with that of polished metal sample (17).

**3. RESULTS AND DISCUSSION**

— **Tafel polarization measurements**

The effect of phosphoric acid medium and potassium hydroxide medium on the corrosion rate of 7075 aluminium matrix and composites containing 2,4 and 6 percentage of SiC sample was studied using Tafel polarization technique (18).

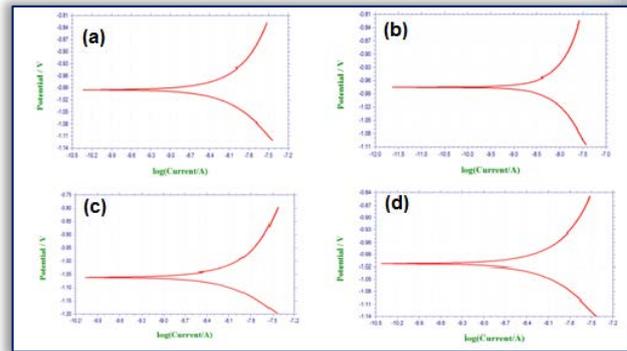


Figure 5. Tafel polarization curve for Al7075 in 1.0N H<sub>3</sub>PO<sub>4</sub> containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

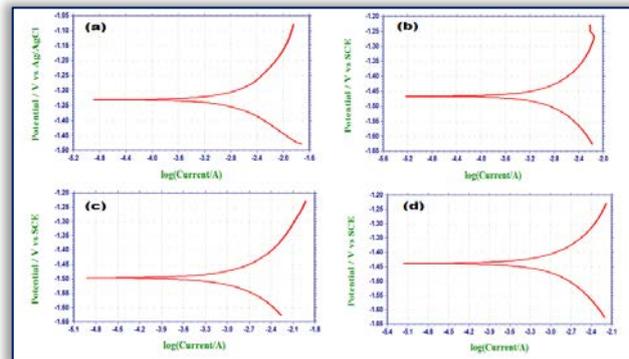


Figure 6. Tafel polarization curve for Al7075 in 0.5N KOH containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

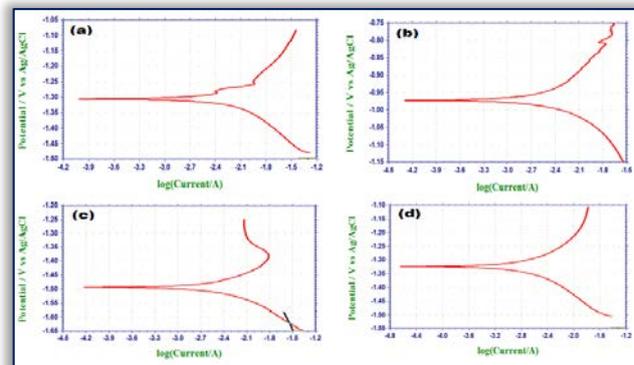


Figure 7. Tafel polarization curve for Al7075 in 0.75N KOH containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

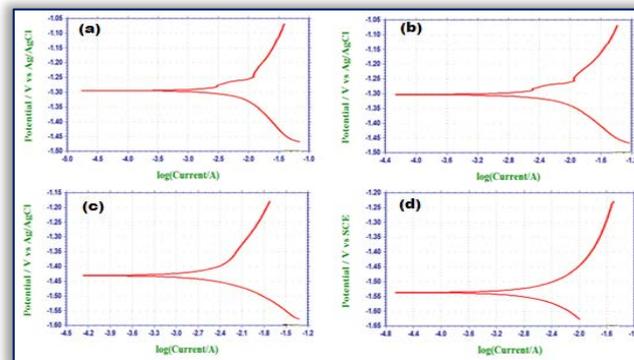


Figure 8. Tafel polarization curve for Al7075 in 1.0N KOH containing (a) 0% SiC, (b) 2% SiC, (c) 4% SiC, (d) 6% SiC particulates at 25°C

Figure 3-8 represents the potentiodynamic polarization curves of aluminium 7075 matrix and composites containing 2, 4 and 6 percentage of SiC in different concentration of phosphoric acid and potassium hydroxide medium at 25°C. Corrosion parameters such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $I_{corr}$ ), anodic slope ( $b_a$ ), cathodic slope ( $b_c$ ) are obtained from the tafel polarization curves (23). Results are tabulated in tables 2 and 3. The obtained results indicate that the corrosion rate ( $v_{corr}$ ) of Al7075 increases with increase in concentration of phosphoric acid and potassium hydroxide medium and corrosion rate considerably decreases with increase in percentage of SiC particulate. The positive shift in the corrosion potential ( $E_{corr}$ ) with the increases in the concentration of phosphoric acid medium and sodium hydroxide medium indicates that the anodic process is much more affected than the cathodic process. (EI-Syed, 1997). This observation is in accordance with Muralidharan (Muralidharan and Rajag opalan, 1979), who proposed dependence of  $E_{corr}$  and  $I_{corr}$  on solution parameters. Tafel slopes remained on most unchanged indicating that acid strength does not change the mechanism of the corrosion process. It is evident from data of tables 2 and 3 that corrosion rate is more in case of potassium hydroxide medium than in phosphoric acid medium (19).

Table 2: Polarization and Impedance parameters for the Al7075 matrix and composite in different concentrations of  $H_3PO_4$

Tafel polarization							EIS
Conc. Of $H_3PO_4$ (N)	% of SiC	$I_{corr}$ ( $Acm^{-2}$ )	CR( $v_{corr}$ ) ( $mmy^{-1}$ )	$b_a$ ( $V dec^{-1}$ )	$-b_c$ ( $V dec^{-1}$ )	$E_{corr}$ (V vs SCE)	$R_{ct}$ ( $ohmcm^2$ )
0.5	0	7.078	2.903	5.464	7.457	-0.93	29.3
	2	6.219	2.561	5.014	7.159	-0.81	33.3
	4	5.959	2.454	5.063	7.482	-0.74	38.2
	6	3.118	1.284	5.063	7.913	-0.77	63.5
0.75	0	7.932	3.267	4.752	6.187	-0.75	33.1
	2	7.952	3.275	4.604	5.958	-0.74	36.9
	4	7.807	3.215	4.325	6.118	-0.91	65.2
	6	7.722	3.180	4.604	5.810	-0.74	67.4
1.0	0	9.386	3.865	4.466	5.862	-0.72	31.9
	2	9.408	3.874	4.390	5.699	-0.70	36.4
	4	8.262	3.403	4.335	5.946	-0.79	67.2
	6	8.102	3.337	4.622	5.944	-0.74	70.2

Table 3: Polarization and Impedance parameters for the Al7075 matrix and composite in different concentrations of KOH

Tafel polarization							EIS
Conc. Of KOH (N)	% of SiC	$I_{corr}$ ( $Acm^{-2}$ )	CR( $v_{corr}$ ) ( $my^{-1}$ )	$b_a$ ( $V dec^{-1}$ )	$-b_c$ ( $V dec^{-1}$ )	$E_{corr}$ (V vs SCE)	$R_{ct}$ ( $ohmcm^2$ )
0.5	0	$2.410 \times 10^{-3}$	$9.93 \times 10^2$	5.136	6.666	-1.34	37
	2	$1.717 \times 10^{-3}$	$7.10 \times 10^2$	2.900	6.032	-1.46	41
	4	$1.836 \times 10^{-3}$	$7.56 \times 10^2$	5.033	4.885	-1.49	51
	6	$1.330 \times 10^{-3}$	$5.48 \times 10^2$	5.212	4.890	-1.44	69
0.75	0	$1.382 \times 10^{-2}$	$5.69 \times 10^3$	3.654	4.655	-1.32	39
	2	$7.917 \times 10^{-3}$	$3.26 \times 10^3$	2.977	4.902	-0.99	42
	4	$6.789 \times 10^{-3}$	$2.79 \times 10^3$	4.537	5.901	-1.49	69
	6	$3.025 \times 10^{-3}$	$1.24 \times 10^3$	5.925	6.096	-1.34	70
1.0	0	$1.492 \times 10^{-2}$	$6.14 \times 10^3$	3.560	4.716	-1.29	57
	2	$1.381 \times 10^{-3}$	$5.62 \times 10^3$	3.792	4.925	-1.31	69
	4	$5.920 \times 10^{-3}$	$2.43 \times 10^3$	3.027	8.399	-1.44	70
	6	$4.214 \times 10^{-3}$	$1.73 \times 10^3$	5.751	5.946	-1.54	94

— Electron impedance spectroscopy (EIS) measurements

The corrosion behavior of 7075 Aluminium matrix and composites were also investigated by EIS techniques in various concentrations of phosphoric acid medium and potassium hydroxide medium (20). The impedance spectra were recorded and displayed as Nyquist plots as a function of acid and alkali strength. Nyquist plots for different concentration of phosphoric acid medium and potassium hydroxide medium at 25°C are shown in Figures 9-11 and 12-14 respectively. The EIS results are reported in tables 2 and 3.

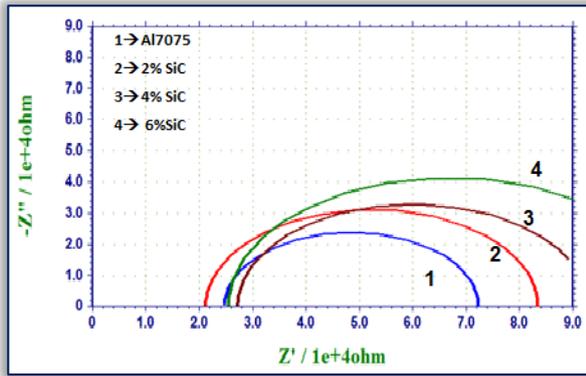


Figure 9. Nyquist plots for Al7075 in 0.5N H<sub>3</sub>PO<sub>4</sub> containing different percentage of SiC at 25°C.

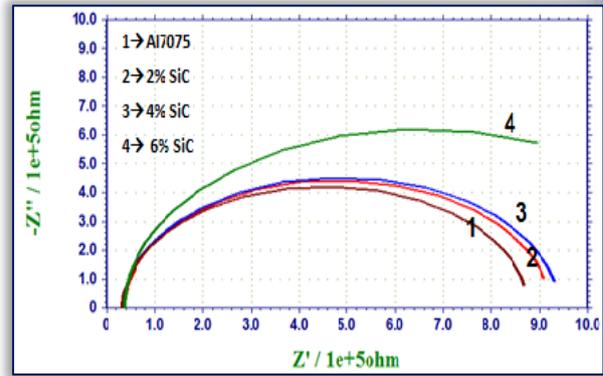


Figure 10. Nyquist plots for Al7075 in 0.75N H<sub>3</sub>PO<sub>4</sub> containing different percentage of SiC at 25°C.

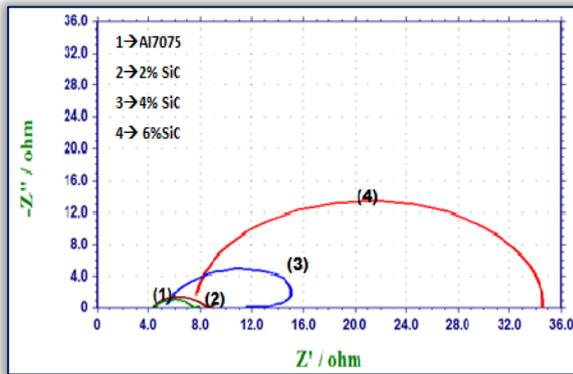


Figure 11. Nyquist plots for Al7075 in 1.0 N H<sub>3</sub>PO<sub>4</sub> containing different percentage of SiC at 25°C.

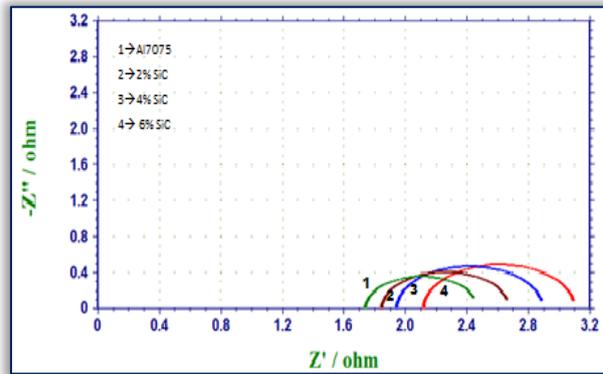


Figure 12. Nyquist plots for Al7075 in 0.5N KOH containing different percentage of SiC at 25°C.

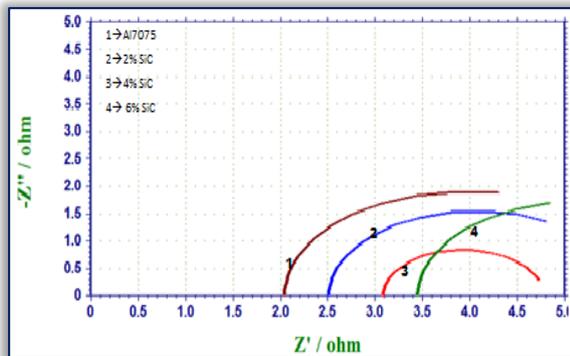


Figure 13. Nyquist plots for Al7075 in 0.75N KOH containing different percentage of SiC at 25°C.

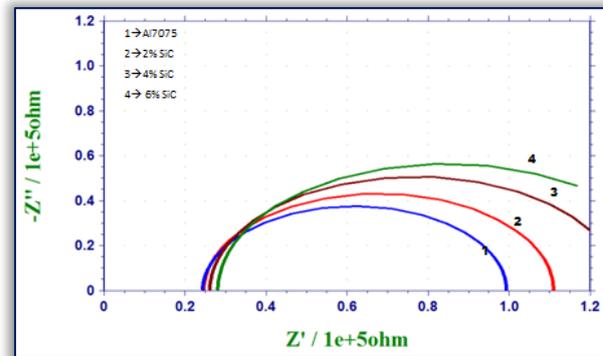


Figure 14. Nyquist plots for Al7075 in 1.0 N KOH containing different percentage of SiC at 25°C.

Nyquist plots for Aluminium 7075 reinforced with different percentage of SiC in various concentrations of phosphoric acid and potassium hydroxide are shown in Figures 7-12, the impedance diagrams show semicircles, indicating that the corrosion process is mainly charge transfer controlled (Wit and Lenderink, 1996; Lenderink et al., 1993) (21). The depressed semicircles of the Nyquist plots suggest the distribution of capacitance due to inhomogeneous surface associated with the metal surface (Morad, 1999). It was evident from these plots that the impedance response of Aluminium 7075 matrix in different concentration of phosphoric acid and potassium hydroxide has significantly altered due to the presence of SiC particulates. The plots were similar in all the composites. Semicircles are obtained which cut the real axis at higher and lower frequencies (22). At higher frequency end, the intercept corresponds to  $R_s$  and at lower frequency end the intercept corresponds to  $R_s + R_t$ . The difference between these two values gives  $R_t$ . The results show that  $R_t$  values increases with increase of percentage of SiC particulates. The electrochemical impedance parameters derived from the Nyquist plots are shown in Table 2 and 3. The results obtained from electrochemical impedance spectroscopy (EIS) method can be interpreted in terms of the equivalent circuit of the electrical double layer shown in Figure 15 which has been used previously to model the Aluminium / acid and base interface. The Nyquist plots were similar

for the Aluminium/SiC composites. The semicircle in all cases corresponds to a capacitive loop (23). The semicircle radii depend on the percentage of SiC particulates. The diameter of the capacitive loop depends increased with increase of SiC percentage. The diameter of the capacitive loop increased with increase of SiC particulates, the increase is significant in 6% SiC. Nyquist plots obtained in the real system represent a general behavior where the double layer on the interface of metal/solution does not behave as a real capacitor.

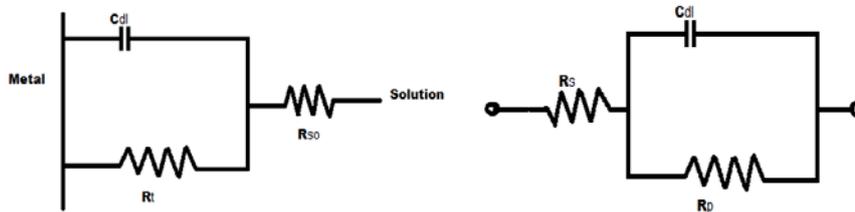


Figure 15: Equivalent circuit for the impedance measurements

On the metal side electrons control the charge distribution whereas on the solution side it is controlled by ions (24). As ions are much larger in size than the electrons, the equivalent ions to the charge on the alloy will occupy quite a large volume on the solution side of the double layer. From the table 2 and 3, it was clear that charge transfer resistance values were increased with increasing percentage of SiC.

— Scanning electron microscopic study

The SEM images of freshly polished surface of Al7075 are given in Figure16 which shows polished surface with few scratches due to polishing (25). The surface morphology of the Al 7075 alloy sample was examined by SEM immediately after corrosion tests in 1.0M phosphoric acid medium and 1.0M potassium hydroxide medium. The SEM image of corroded sample given in Figure 17 and 18 shows the degradation of alloy, with more or less uniform attack in phosphoric acid and potassium hydroxide medium (26).

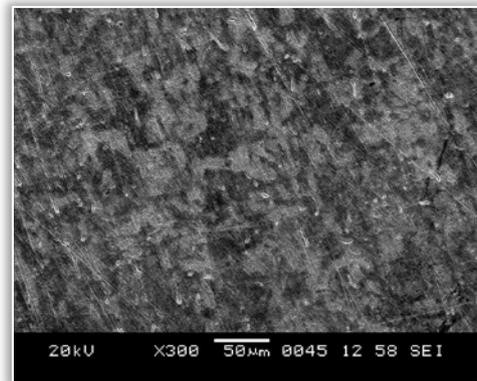


Figure 16. SEM image of polished surface of unreinforced Al 7075 before corrosion

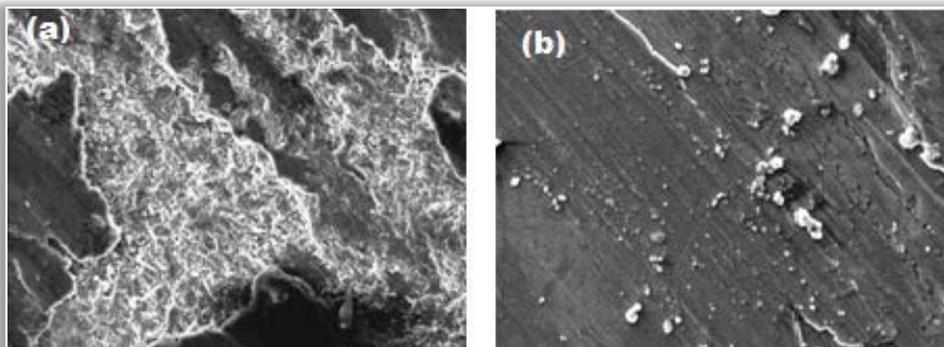


Figure 17. SEM images of Al 7075 after corrosion in 1M phosphoric acid containing (a) 4% SiC, (b) 6% SiC particulates.

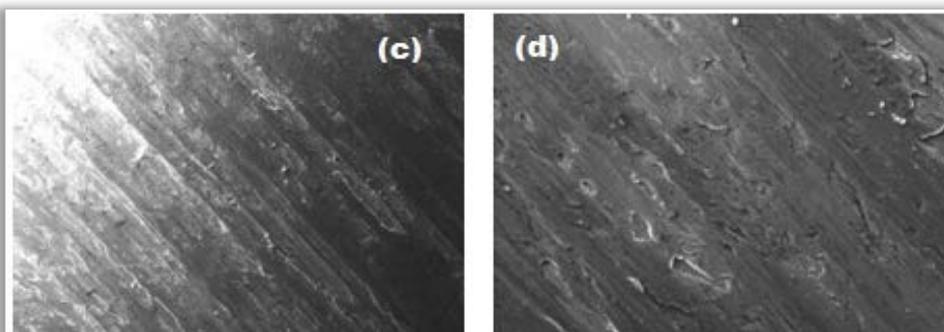


Figure 18. SEM images of Al 7075 after corrosion in 1M potassium hydroxide containing (c) 4% SiC, (d) 6% SiC particulates.

### — X-Ray Diffraction Analysis

The X-ray diffraction technique is an important method used to study phase analysis in MMCs to indicate the reaction between reinforcement and matrix and to determine composite composition. XRD pattern of unreinforced Al7075 and Al 7075/6 wt% SiC is given in Figure 19 and 20. The XRD pattern shows the presence of Al7075 matrix and SiC particulates in the composite. From Figure 11 it can be observed that a uniform structure is formed in Al 7075 matrix. From Figure 20 it can be observed that, mainly there are peaks of Al matrix and SiC reinforcement. The XRD patterns confirmed the better distribution of SiC in Al7075 matrix, important technique in determining the resultant composite properties (27).

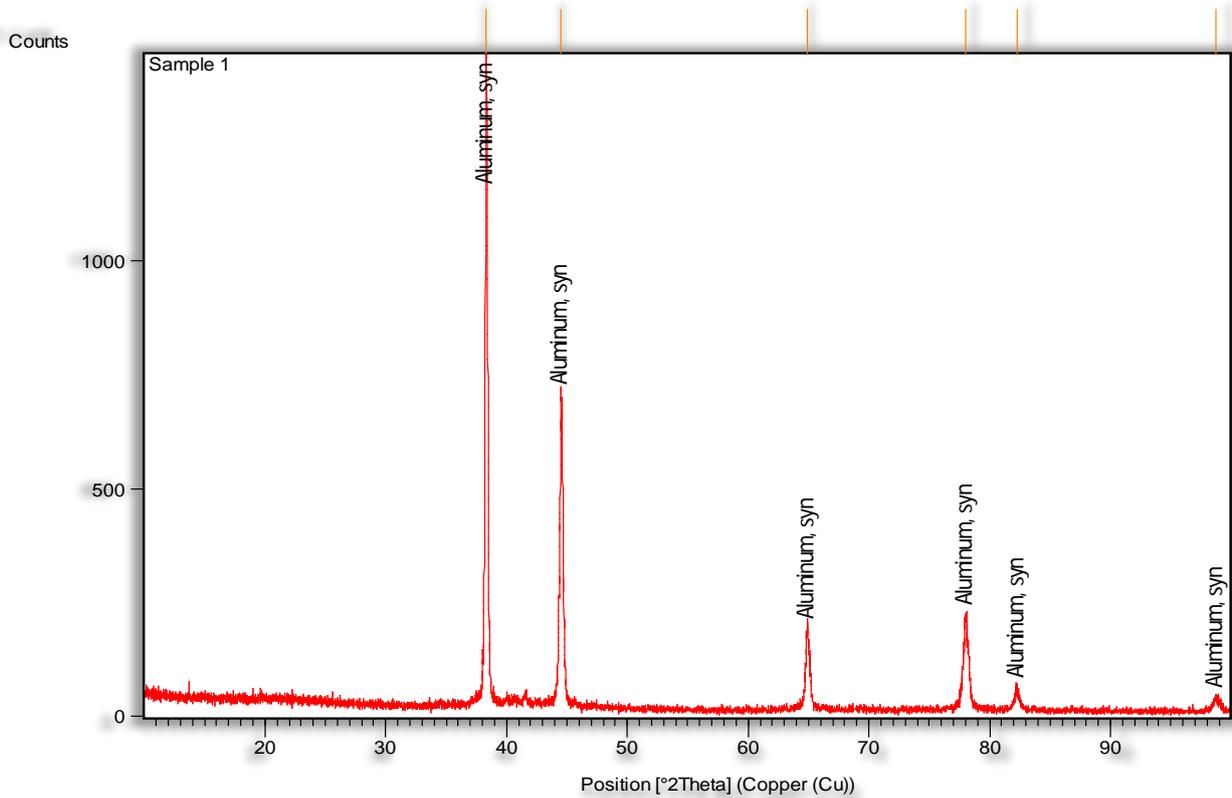


Figure 19: X-Ray diffraction analysis patterns of the unreinforced Al7075.

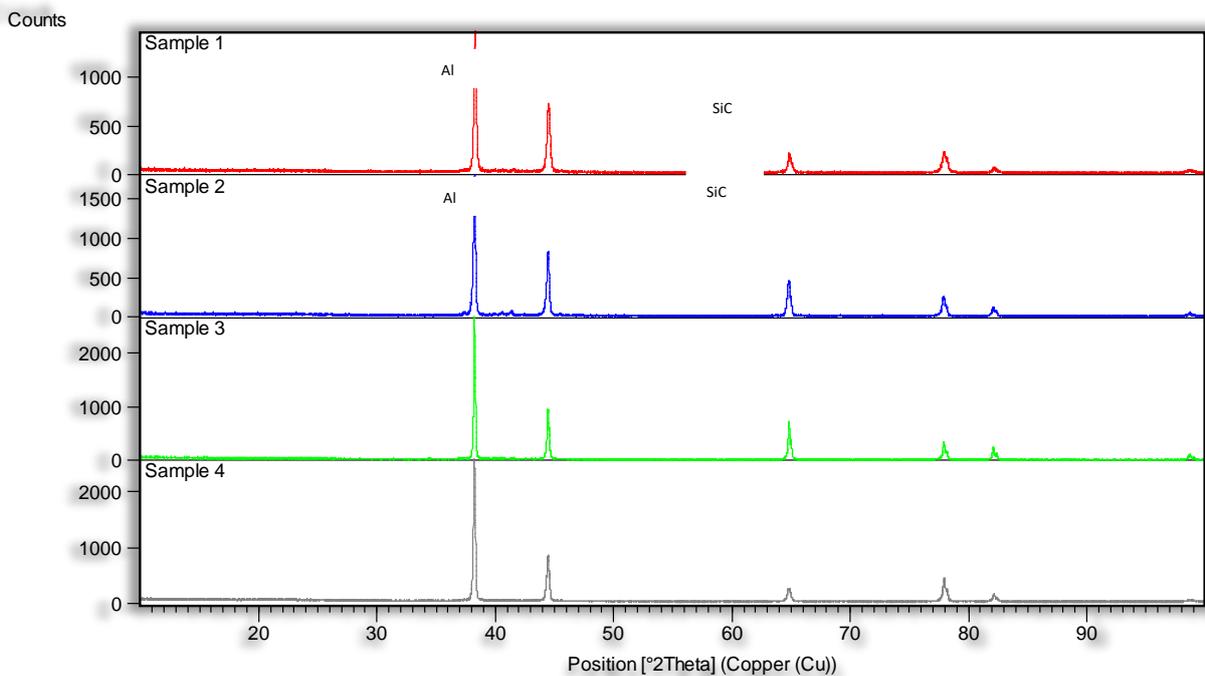


Figure 20: X-Ray diffraction analysis patterns of Al7075 containing 6% SiC particulates.

#### 4. CONCLUSIONS

- Al7075 alloy undergoes corrosion both in phosphoric acid medium and potassium hydroxide medium;
- The corrosion rate increases with an increase in the concentration of phosphoric acid medium as well as potassium hydroxide medium;
- The corrosion rate decreases with increase in percentage of SiC particulate in Al7075;
- The corrosion of Al7075 alloy is more severe in potassium hydroxide medium than in phosphoric acid medium.

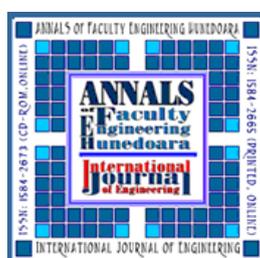
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