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Evaluation of Corrosion Properties of Al_2O_3 and SiC Reinforced Aluminium Metal Matrix Composites Using Taguchi's Techniques

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Abstract: **We report the fabrication of alumina (Al_2O_3) and silicon carbide (SiC) particulates reinforced Al metal matrix composites (Al MMCs) by stir casting method. The effect of reinforcement and heat treatment on the corrosion properties of Al matrix and r-Al MMCs were investigated by polarization, impedance and weight loss methods. Taguchi's experimental design method was employed to set the process parameters. In order to find the optimal process parameter levels, orthogonal arrays of Taguchi, the signal-to-noise (S/N) ratio, the analysis of variance (ANOVA), and regression analyses were employed. The analysis on the effect of these parameters on the corrosion rates has also been discussed. The results revealed that the rate of corrosion decrease with increase in the content of Al_2O_3 and SiC particulates in the r-Al MMCs. A significant enhancement in the corrosion resistance was observed with heat treated of Al alloy and its composites. The morphological analysis by SEM micrographs revealed the lesser formation of cavities due to corrosion upon the increase in Al_2O_3 content which would be beneficial for corrosive application.**

Index Terms: **Al MMCs, Al_2O_3, Corrosion resistance, SiC, Taguchi technique.**

I. INTRODUCTION

Metal matrix composites (MMCs) are normally fabricated by the reinforcement of particles of one type of material into the matrix of a metal which is bound to exhibit unique properties. The reinforcement of matrix by ceramic, fibre, polymers and organic particulates into a single matrix led to the formation of hybrid composites (Alaneme KK., and Bodunrin MO., 2011; Ananda Murthy HC., 2015). The particulate reinforced MMCs are promising because of their homogenous and isotropic material properties, low cost and ability to be formed using conventional metal processing techniques. These particulate reinforced aluminium metal matrix composites having huge applications in various 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 environment (Ananda Murthy HC., and Somit Kumar Singh., 2015). 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 and superior corrosion resistance (Ananda Murthy H.C et al. 2013). The observed variation of corrosion susceptibility 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. A study conducted on aluminium alloys reinforced with TiC, TiO_2, TiB_2 and TiN (Anuj Singh Baghel et al. 2013; Balasivanandha Prabu et al. 2006; Baradeswaran A., and Elaya Perumal A., 2014; Chaudhury SK., et al. 2004) has revealed lower corrosion resistance for the composites compared to matrix alloys, owing to galvanic corrosion. These materials are expected to exhibit good corrosion resistance in the aggressive environments. The SiC has been found to poses

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excellent capability with the Al-matrix as reinforcement (Chauhan SR., et al. 2010). In general particulate reinforced Al MMCs provide a good combination of electrical and mechanical properties. Therefore, enhancement in the corrosion resistance of composite materials by reinforcing with ceramic particulates has recently gained much importance (Fakrudeen SP., et al. 2012). The reinforcement of SiC and Al_2O_3 into Al matrix resulted in superior composite properties and hence finds industrial applications. MMCs can be manufactured by various methods. The liquid metallurgy route has been the choice of the majority for the preparation of MMCs (Fakrudeen SP., et al. 2012).

Alumina (Al_2O_3) is a unique, widely used and low-cost material in the group of ceramics. It has special properties like high strength, oxidation resistance, hardness and high wear resistance. It is normally available in the purity range which lies between 95 % and 99.8 % for the most challenging high temperature applications. In this work (Khairel Rafezi Ahmad., et al. 2013) the improved corrosion resistance due to the Al_2O_3 particulate distribution of the reinforcement was observed and as well as adequate bonding between matrix and the reinforcements was obtained. The presence of Al_2O_3 particles resulted in enhanced corrosion resistance in MMCs. The Al_2O_3 particles debris in matrix alloy offer safety to the softer matrix. This reduces the defects and suppresses the penetration and the reduction of slides on the surface of the MMCs. The matrix plastic deformation can be challenged/resisted by the presence of Al_2O_3, which act as a barrier that may be more wear resistant when compared to base alloy.

The silicon carbide (SiC) is a vital non-oxide ceramic material. It has special properties like high hardness, chemical and thermal stability, high erosion resistance and high melting point. All these qualities make SiC highly suitable for applications in a variety of industrial and automobile products because they possess high strength and are suitable for elevated temperature working conditions. Generally, SiC is used as an abrasive material and used in a wide band gap semiconductor application for high temperature and high power electronic components. Better hardness of this material led to its use in machine tools and in other structural applications which require desired properties such as, load carrying and transfer of strength to matrix (Khalid Abd El-Aziz., et al. 2015; Mahendra KV., and Radhakrishna K., 2010; Mehdi Saeidi., et al. 2015). Thus, Al_2O_3 and SiC particulates have been chosen for Al reinforcement.

In the present work, authors have made an effort to fabricate Al/SiC-Al_2O_3 metal matrix composites with various weight percentages of particulates subjected to heat treatment and study the corrosion behavior in 3 % NaCl medium by the electrochemical impedance method and weight loss method in sea water. The morphological analysis was conducted by using scanning electron microscope (SEM). In addition, a plan order for performing the experiments was generated by Taguchi method using orthogonal arrays and analysis of parameters was conducted by using ANOVA technique.

II. Materials and Methods

A. Preparation of Al/Al_2O_3/SiC composites

The liquid metallurgy method was used to fabricate composites with discontinuous fibers and particulates. Care was taken to maintain an optimum casting parameter of stirring speed (150 rpm), stirring time (2 min) and pouring temperature (700°C). The reinforcements were preheated prior to their addition in the aluminium alloy melt. Degassing agent was used to reduce gas porosities. The molten metal was then poured into a permanent cast iron mould of diameter 25 mm and length 180 mm. The die was released after 2 minutes and the cast specimens were taken out. Experiments were carried out based on the L27 OA.

In the present work, three process parameters, each at three levels, were selected to evaluate the properties. After defining the process parameters, the number of levels for each parameter was decided. The choice of number of levels depends on the trend in which the parameters affect the response result. Table I displays the designated values for the corresponding levels of all process parameters. Based on Taguchi technique, the L27 OA was constructed. The cause for using L27 OA is to assess the importance of interaction terms. Interaction means the impact of a functional variable on the effects of other variable.

Table I. Parameters and their levels in the experimental design

Process Parameters	**Level - 1**	**Level - 2**	**Level - 3**
Al_2O_3(wt. %)	2	4	6
SiC (wt. %)	3	6	9
Aging Temperature(°C)	140	160	180

B. Corrosion Testing and Analysis

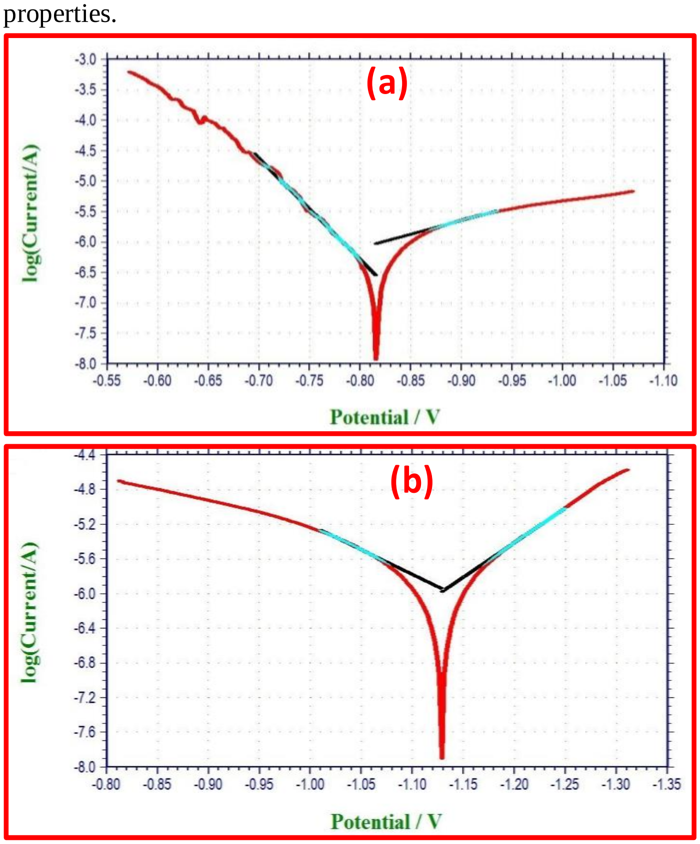
Electrochemical impedance methods were used to examine the corrosion behavior by applying Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization technique. All aspects were studied with respect to saturated calomel electrode (SCE). In addition, corrosion rate measurements were also done by weight loss methods. In the present research, corrosion examination was carried out at room temperature (27 °C) by conventional weight loss technique. The test samples were machined into standard sizes of 20mm diameter and 20mm thickness. Corrosion studies were carried out for 365 days (01 year) by weight loss method by immersing the as cast and composite specimens in sea water and the moisture content was monitored continuously. The corroding medium used for the test was sea water collected from Pondicherry beach (near Thiruvannamalai), Tamil Nadu. All 27 weighed specimens were dipped in sea water. Specimens of matrix and composites were immersed in corrodent for 365 days. ANOVA analysis of corrosion results has also been conducted and presented.

C. Characterization

The Electrochemical experiments were conducted on an electrochemical analyzer CHI608E potentiostat and in a tri-electrode system, consisting of r-Al MMC electrode, platinum wire and Ag/AgCl as working, counter and reference electrodes, respectively in 0.1M KC at 10 mVs^{-1} scan rate, with a potential variation of −0.6 to 1.0 V (vs Ag/AgCl electrode) and frequency range of 1 Hz – 1 MHz at 5 mV amplitude. Microscopic analysis has been conducted by scanning electron microscopy with energy-dispersive X-ray spectroscopy (VEGA3 TESCAN with a magnification of x67.6K (Accelerating voltage 25 kV).

III. Results and Discussion

The Potentiodynamic polarization experiments were conducted in an open air and de-oxygenated conditions. The polarization curves of Al matrix and $Al/Al_2O_3/SiC$ composites obtained in 3.5% NaCl were presented in Fig. 1a to 1d. The corrosion current density (I_{corr}) and corrosion rate were calculated using Tafel extrapolation method and are as shown in Table 2. It is observed that the corrosion current density (I_{corr}) and corrosion rate values have decreased with increase in wt. % of reinforcement (Al_2O_3 and SiC) particles and aging temperature. Adding hard ceramic particulates into the Al alloy matrix increases the corrosion resistance due to its physical properties.



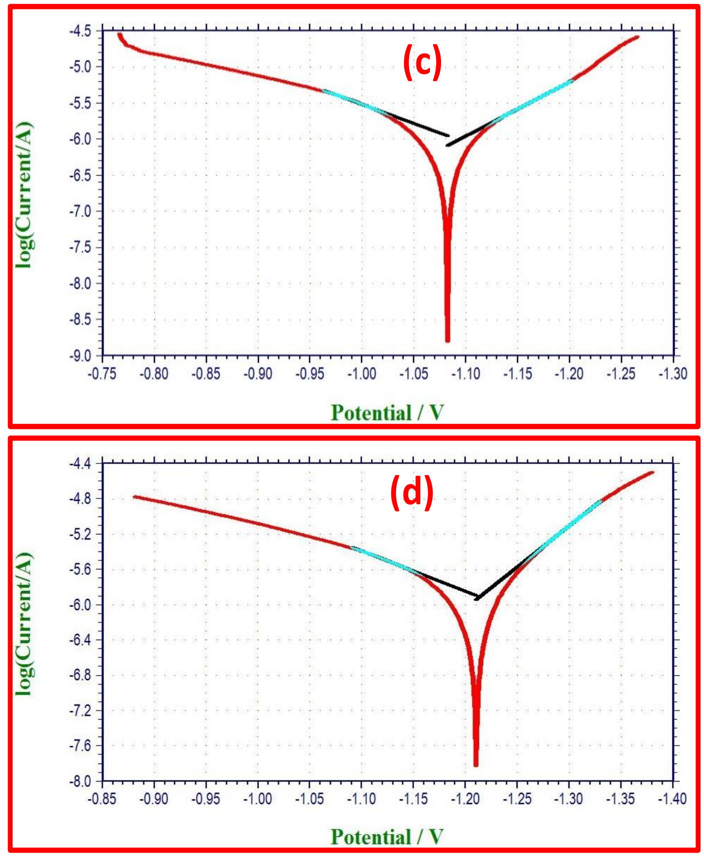


Fig. 1. Polarization curves of (a) As cast (b) 2% Al_2O_3+9% SiC+180 ºC aging temperature, (c) 4% Al_2O_3+9% SiC+180 ºC aging temperature, (d) 6% Al_2O_3+9% SiC+180 ºC aging temperature.

The uniform dispersal of ceramic particulates in an Al alloy matrix shows improved corrosion resistance. The increase in wt. % of the reinforcing particulates causes an increase of the corrosion resistance in composites (Murthy HC., et al. 2010). Al_2O_3 is believed to form stable inner oxide layer which act as a shield to prevent corrosion (Niveen J., et al. 2015; Nunes PCR., and Ramanathan LV., 1995). The impedance spectra recorded the Nyquist plots as shown in the Fig. 2 (a-d). The semicircle of the Nyquist plots indicates the dispersal of capacitance (charge) due to inhomogeneity related to the electrode surface. The irregular semicircles show a non-ideal electrochemical performance on the electrode surface, which is due to frequency distribution, roughness of metal surface and inhomogeneity. Nyquist plots show capacitive loop which is associated to the behavior of double layer capacitance and as well as the charge transfer method between electrolyte and metal surface. The diameter of the semicircle reduces with increase in acid concentration indicating an increase in the rate of corrosion. Increase in diameters of Nyquist plots signifies increased protective nature of the inhibitor against damage of material in the corrosive solution (Rambabu P., et al. 2017; Ravikumar M., et al. 2018).

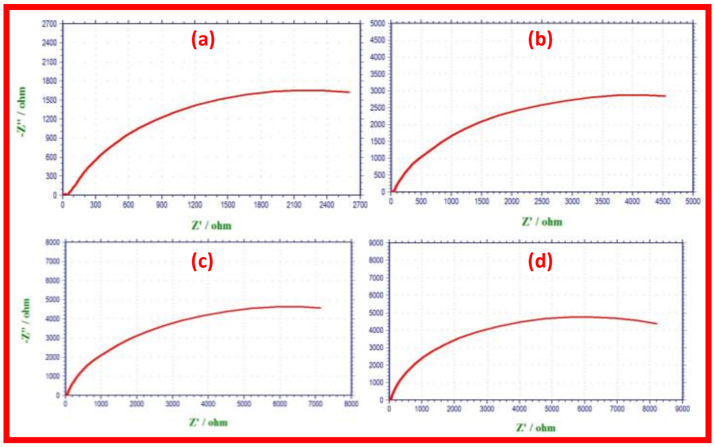


Fig. 2. Nyquist plots of (a) As cast (b) 2% Al_2O_3+9% SiC+180 ºC aging temperature, (c) 4% Al_2O_3+9% SiC+180 ºC aging temperature, (d) 6% Al_2O_3+9% SiC+180 ºC aging temperature.

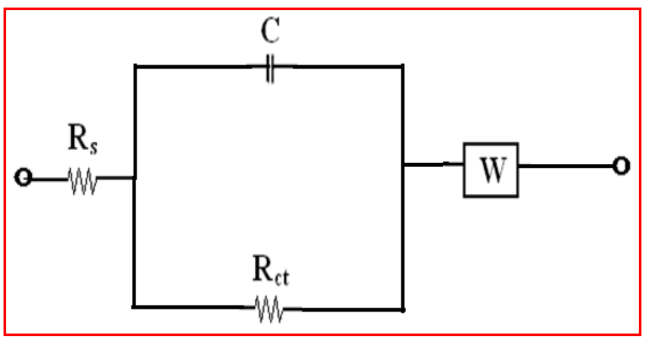


Fig. 3. Proposed equivalent circuit of Nyquist plot.

Table II. EIS data for As cast, 2% Al_2O_3+9% SiC+180 ºC aging temperature, 4% Al_2O_3+9% SiC+180 ºC aging temperature and (d) 6% Al_2O_3+9% SiC+180 ºC aging temperature.

Name of the Sample	R_s (Ω)	R_{ct} (Ω)	C_{dl} (F)	W
Ascast	12.23	26.92	3.181×10^{-6}	0.1543×10^{-3}
2%Al_2O_3+9%SiC+180ºC aging temperature	11.74	46.52	2.867×10^{-7}	0.8678×10^{-4}
4%Al_2O_3+9%SiC+180ºC aging temperature	11.89	64.75	9.694×10^{-7}	0.1452×10^{-5}
6%Al_2O_3+9%SiC+180ºC aging temperature	11.76	85.28	2.126×10^{-8}	0.9476×10^{-5}

Fig. 3 shows an equivalent circuit obtained by fitting the EIS data, in the circuit Warburg part *W* represents the within the lower-frequency range of impedance curve Q_1 represents the constant it is parallel to charge-transfer resistance (R_{ct}) and also the low frequency capacitance (Q_2), and parallel to the outflow resistance (R_l) (Ravikumar CR., et al. 2017; Ravikumar CR., et al. 2017; Girish KM., et al. 2018). From the Table II, the order of charge transfer resistance (R_{ct}) and corresponding Capacitance (F) for samples are as given below. 6%Al_2O_3+9%SiC+180ºC aging temperature > 4%Al_2O_3+9%SiC+180ºC aging temperature > 2%Al_2O_3+9%SiC+180ºC aging temperature > Ascast.

In the present research, corrosion tests were carried out at room temperature (27 C) by conventional weight loss technique. In each case, the corrosion rate of the MMCs was found to decrease with increase in wt. % of reinforcement and aging temperature.

Table III depicts the ANOVA analysis of corrosion results obtained from the weight loss study for the MMCs. It is seen that Al_2O_3 has maximum significance due to highest percentage of contribution (83.97%) compared to other process parameters. Similar results were observed by other researchers (Balasivanandha Prabu., et al. 2006; Ravikumar M., et al. 2018), who revealed increased corrosion resistance in MMCs due to the presence ofAl_2O_3 particles.

Table III. ANOVA analysis of Corrosion loss (wt. loss in gms)

Source	DOF	Seq SS	AdjSS	Adj MS	F	P	Cont. (%)	Remarks
Al_2O_3	1	30.3	0.97	0.97	52.5	0.000	83.97	Significant
SiC	1	4.1	0.17	0.17	9.6	0.005	11.33	Significant
Aging temperature	1	0.4	0.11	0.11	6.1	0.022	1.18	Significant
Al_2O_3 * SiC	1	0.8	0.84	0.84	45.8	0.000	2.34	Significant
SiC * Aging temperature	1	0.0	0.00	0.00	0.0	0.994	0.01	Insignificant
Aging temperature * Al_2O_3	1	0.05	0.05	0.05	2.8	0.108	0.14	Insignificant
Error	20	0.36	0.36	0.01			1.02	
Total	26	36.1					100	

Summary of Model: R-Sq=98.98% R-Sq(adj) = 98.67%

DOF; Degrees of Freedom; SeqSS.: Sequent sum Square; AdjSS.: Adjacent Sum Square; F: Fisher value; P: Probability; Con.%: Percentage of Contribution.

Based on results of corrosion behavior by weight loss method, Table IV shows ranking of all the factors as per their delta values. For corrosion loss, Al_2O_3 has maximum influence since delta of means ranked it as 1 as shown in Table IV.

Fig. 4 presents the main effects plot of corrosion loss and "smaller is better" criteria is applied for the present study. The plot clearly depicts that the corrosion rate gradually decreased with increasing level of process parameters. level-3 of Al_2O_3, level-3 of SiC and level-3 of aging temperature are the optimum points in corrosion loss of composites in this study.

Table IV. Response Table for corrosion loss (wt. loss in gms)

Level	Al_2O_3	SiC	Aging Temperature
1	3.856	3.006	2.644
2	2.371	2.427	2.507
3	1.260	2.053	2.336
Delta	2.591	0.954	0.308
Rank	1	2	3

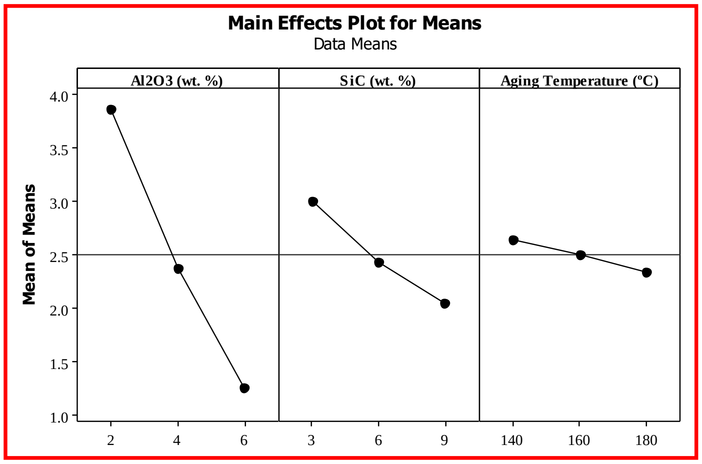


Fig. 4. Main effects graph for corrosion loss

Fig. 5 displays the interaction among parameters on the responses of corrosion loss. The corrosion loss shows different values owing to strong effect of interaction among the parameters. The corrosion behavior by weight loss of the composites is also affected by increasing the wt. % of particles as well as the aging temperature (Siva Prasad D., and Rama Krishna A., 2011). The general regression equation for corrosion loss by weight loss method is as shown in the Eq. (1).

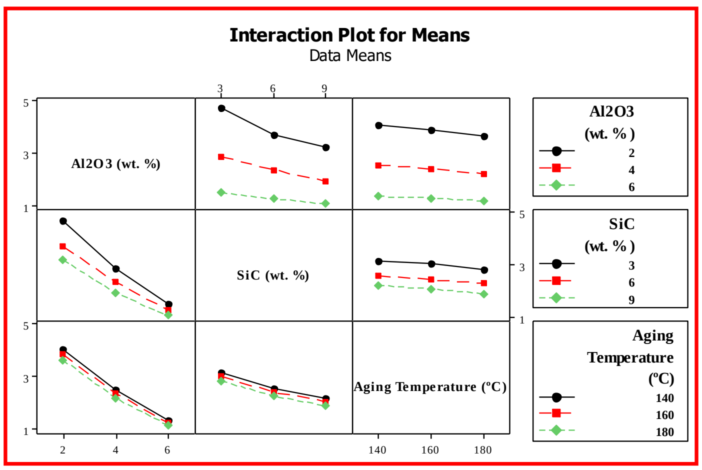


Fig. 5. Interaction graph for corrosion loss

$$\text{Corrosion loss} = 9.39938 - 1.17844\ Al_2O_3 - 0.33668\ SiC - 0.014325\ \text{Aging Temperature} + 0.0442542\ Al_2O_3 * SiC + 4.44444e\text{-}006\ SiC * \text{Aging Temperature} + 0.00164938\ \text{Aging Temperature} * Al_2O_3 \quad (1)$$

A plot of experimental values against predicted values has been drawn for all three levels as depicted in Fig. 6. Conformation of accuracy of such forecasting of trials has been made and the comparison of experimental values verses predicted values is shown in the graph (Fig.6).

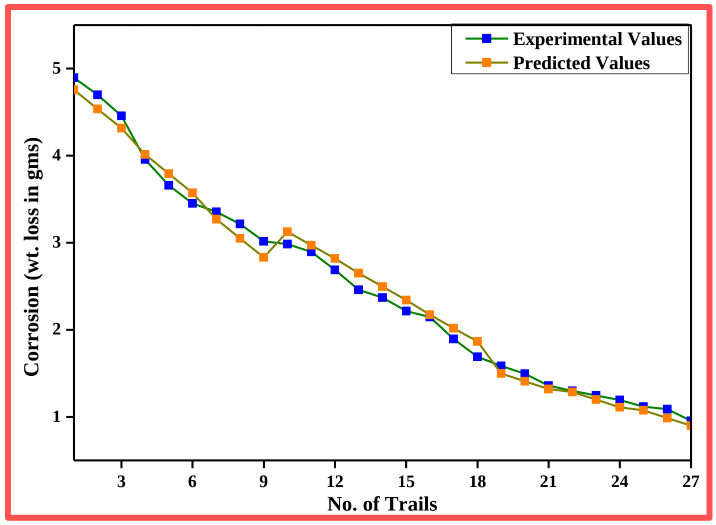


Fig. 6. Comparative plot between experimental values and predicted values of corrosion loss (by wt. loss method)

Fig. 7 depicts the 3D surface plot of corrosion loss with the combination of Al_2O_3, SiC and aging temperature. From Fig. 7 (a), it is noted that corrosion loss of the composites was observed to decrease with increase in the wt. % of reinforcement Al_2O_3 and SiC particles at constant aging temperature. In Fig. 7 (b), it is seen that corrosion loss of the composites slightly decreased by increase in the aging temperature and wt. % of SiC while keeping wt. % of Al_2O_3 constant. From Fig. 7 (c), it is inferred that the corrosion loss of the composites decreased more by increase in wt. % of Al_2O_3 and has slight variations due to increase in aging temperature while keeping wt. % of SiC constant. From the observation outcome it is seen that the effect of Al_2O_3 content on control of corrosion rate is highly significant and also the corrosion is slightly affected by addition SiC particles as well as the aging temperature (Siva Prasad D., and Rama Krishna A., 2011).

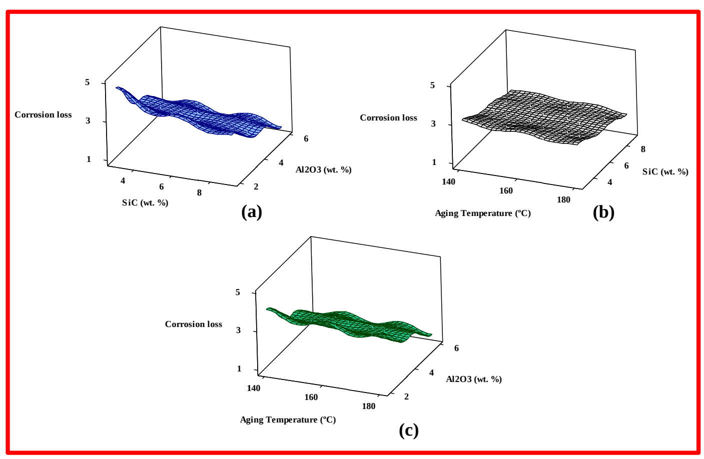


Fig. 7. Effect of corrosion loss with the interaction of Input Parameters

A SEM image depicted in Fig. 7 of the corroded surface was observed to study the behavior of corrosion. The present investigation was made to produce a corrosion resistant Al MMCs for application in marine field since aluminium is more vulnerable to corrosion effect.

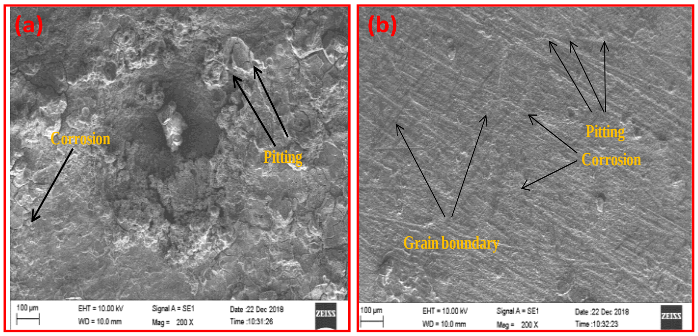


Fig. 8 (a) SEM micrograph of Monolithic. (b) SEM micrograph of Al - 6% Al_2O_3 - 9% SiC composite.

Fig. 8(a) presents the SEM micrographs of the corroded surface of unreinforced as cast specimen. In addition, Fig. 8(b) shows the SEM micrographs of un-corroded surface with metallurgic ally strong reinforcement bonding of Al_2O_3 and SiC with the matrix. The micrographs clearly confirm the fact that the material loss due to corrosion was lowered by the presence of Al_2O_3. This is due to the reduction in salt deposition owing to increase in the percentage reinforcement (Sorana D., et al. 2007). The surface of Al/Al_2O_3/SiC composites has been less damaged compared with matrix material. Increase in the amount of Al_2O_3/SiC particulates resulted in lesser surface degradation (compare Figure 8a, b). In addition, grain boundary corrosion and pitting corrosion were observed on the pure Al matrix as well as on the Al/Al_2O_3/SiC composites. The combined influence of reinforcement inclusions and structural flaws/ defects produced at the time of MMC fabrication decrease the susceptibility of Al/Al_2O_3/SiC composites to pitting by reducing the required driving force.

Conclusion

In the present investigation, the Al/Al_2O_3/SiC hybrid MMCs were successfully fabricated using the stir casting method. Taguchi's design of experiment method was used to analyze the coefficient of friction and the corrosion rate of the composites was found to be lower than that of the corresponding matrix alloy. The corrosion by weight loss of the composite decreased with increase in the weight percentage of the Al_2O_3 and SiC content. The corrosion rate was found strongly dependent on the heat treatment conditions. The optimum parameters for minimization of corrosion obtained were at 6 wt. % of Al_2O_3, 9 wt. % of SiC and 180 °C of aging temperature. The rate of corrosion was found to decrease with increase in the content of Al_2O_3 and SiC particulates in the Al MMCs. A significant enhancement in the corrosion resistance was observed with heat treated Al alloy and its composites. The morphological analysis by SEM micrographs revealed the lesser damage for Al MMCs than for the Al matrix. Finally, it can be concluded that the particles of Al_2O_3 and SiC have enhanced the corrosion resistance of the reinforced Al MMCs which can be utilized for future applications in various sectors.

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