CHAPTER 4

CHARACTERISATION OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF PARTICULATE ALUMINIUM / SILICON CARBIDE COMPOSITES

4.1 INTRODUCTION

Extensive studies on dry sliding wear of aluminium silicon alloy based composites have been carried out. However, limited tribological studies have been reported on Al-Si10Mg alloy composites like stir cast Al-Si10Mg/Al₂O₃ and Al-Si10Mg/Al₂O₃/MoS₂p (Hybrid) composites (Dharmalingam et al., 2010) and A360 cast Al/17SiC and Al/17SiC/17B₄C composites prepared by pressure infiltration method (Soy et al. (2011), Soy et al. 2012) are limited. In addition, dry sliding wear of Al-Si10Mg/SiCₚ composite produced by stir casting method has not been investigated in detail. In this Chapter, the mechanical and the tribological behaviour of Al-Si10Mg/10SiCₚ and Al-Si10Mg/20SiCₚ is presented and compared with that of Al-Si10Mg alloy.

4.2 MICROSTRUCTURES

Specimens prepared using a stir casting technique, as discussed in section 3.3 were used for the investigation. Optical micrographs of unreinforced aluminium alloy as well as those of composites (Figure 4.1a-c) show as cast (dendritic) structure consisting of silicon particles in a eutectic matrix. The microstructure of the composites (Al-Si10Mg/10SiCₚ and
Al-Si10Mg/20SiCₚ shows the increasingly fine grain structure, due to heterogeneous nucleation caused by the addition of SiCₚ. Al-Si10Mg/20SiCₚ exhibits the finest microstructure due to the higher fraction of SiCₚ added.

Figures 4.1a, 4.1b, and 4.1c show the microstructures of Al-Si10Mg, Al-Si10Mg/10SiCₚ, and Al-Si10Mg/20SiCₚ, respectively. Figures 4.1b and 4.1c also confirm the uniform distribution of SiCₚ in the matrix. Stirring of melt using impeller as well as the presence of magnesium in the alloy improved the wetability, which resulted in a better particle distribution. Other features of the microstructure are relatively lower porosity in the casting, an important aspect in cast metal matrix composites.
4.3 PROPERTY EVALUATION OF ALUMINIUM / SILICON CARBIDE COMPOSITES

Enhancement in physical and mechanical properties due to the addition of SiC to the matrix alloy in varying percentages was investigated in detail. The following sections deal with density measurement as well as evaluation of ultimate tensile strength, percentage elongation and hardness of the composites. The results of these tests were compared with those of unreinforced alloy.

4.3.1 Density

SiC has a density 3200 kg/m$^3$, higher than that of aluminium alloy (2650 kg/m$^3$) and hence an increase in SiC content will increase the density of the composite. Density of Al-Si10Mg/10SiC$_p$ and Al-Si10Mg/20SiC$_p$ was marginally higher than that of aluminium alloy by 0.78 % and 1.33% respectively (Figure 4.2). This is in agreement with the theoretical density predicted by the Rule of Mixtures using the Equation (4.1).

$$\rho_c = \frac{\rho_{SiC} \rho_{Al}}{\rho_{Al} M_f^{SiC} + \rho_{SiC} M_f^{Al}} \text{ kg/m}^3 \quad (4.1)$$

where $M_f^{SiC}$ & $\rho_{SiC}$ are the mass fraction and density of SiC$_p$ respectively.

$M_f^{Al}$ & $\rho_{Al}$ are the mass fraction and density of Aluminium alloy respectively.

A similar increase in density composites was found with the addition of SiC$_p$ to Al-Si alloy (Aigbodion and Hassan 2007 and Sahin 2005) as well as with addition of Al$_2$O$_3$ to aluminium alloys (Kok 2005).
4.3.2 Hardness

Hardness of composite depends on the hardness of the reinforcement and the matrix. In the present investigation it was observed that the hardness of the composites was higher than that of the unreinforced alloy. The presence of hard SiC<sub>p</sub> increases hardness of the composites. Hardness increased by 17% in the case of Al-Si10Mg/10SiC<sub>p</sub> while in the case of Al-Si10Mg/20SiC<sub>p</sub> it was 27% (Figure 4.3).
Ultimate Tensile Strength

Ultimate Tensile Strength (UTS) of the Al-Si10Mg unreinforced alloy is 218 MPa. It has been reported in previous researches that the addition of Al₂O₃ to AA6061 and AA7005 led to an increase in tensile strength (Ceschini et al. 2006). Similar results were reported in the case of Al-alloy/SiC_p composites by Cöcen and Önel (2002) as well as in the case of Al-Al₂O₃, Al-illite (K(Na,Ca)Al1.3Fe0.4Mn0.2Si3.4Al0.6O10(OH)₂) and Al/SiC_p composites (Surappa and Rohatgi 1981). However, studies on addition of Al₂O₃ to 2024 Al –alloy have shown a decrease in UTS (Abdel-Azim et al. 1995) as well as in the case of aluminium-graphite composite (Lin et al. 1998). UTS of the composites decreased significantly with the addition of 10 and 20% by weight of SiC_p. The decrease in UTS was about 6% in the case of Al-Si10Mg/10SiC_p whereas it was 10% in the case of Al-Si10Mg/20SiC_p. The observed decrease in UTS is probably due to particle pull out and crack propagation during testing, which initiate at SiC_p matrix interface.
Figure 4.4 Variation in Ultimate Tensile Strength of composite with increase in Weight Percentage of SiC$_p$

4.3.4 Percentage Elongation

Percentage elongation of the composites was found to be lower than that of the unreinforced alloy, confirming that the addition of SiC$_p$ lowers the ductility of the composite. The addition of 10 and 20 Wt% SiC$_p$ resulted in a decrease in % elongation by 25% and 65% respectively (Figure 4.5). As the SiC$_p$ percentage increases the composites, become increasingly brittle. At higher loads, debonding between the SiC$_p$ and the aluminium matrix occurs which results in particle pullout (Figure 4.6) resulting in a decrease in elongation. A similar result was observed in case of SiC$_p$ to Al 2124, 7075 alloys as well as monolithic aluminium(Srivatsan et al. 1991 and Doel and Bowen 1996). This may be because failure appears to occur by the accumulation of internal damage to particles either by particle fracture or by interfacial failure. Such damage introduces voids, which grow and lead to reduced ductility in these composites. (Doel and Bowen 1996 and Hall et al. 1994).
4.3.5 Investigation on fracture behaviour of composites

SEM micrograph of the fractured surface of Al-Si10Mg (Figure 4.6.a), shows transcrystalline ductile fracture while Figure 4.6.b showing fracture surface of Al-Si10Mg/10SiC_p, reveal particle pull out and transcrystalline ductile fracture indicating a mixed mode of fracture. Mechanism of tensile fracture in metal matrix composite was due to interfacial debonding, fractures of reinforcement as well as void nucleation and growth.

Figure 4.5 Variation in % elongation of composite with increase in weight percentage of SiC_p

Figure 4.6 SEM micrographs showing a) transcrystalline ductile fracture of Al-Si10Mg b) particle pullout and brittle fracture of Al-Si10Mg/10SiC_p
4.4 TRIBOLOGICAL CHARACTERISATION OF ALUMINIUM / SILICON CARBIDE COMPOSITES

Studies on dry wear behaviour of Al-Si10Mg/SiC composites were conducted using a Pin on Disk apparatus (discussed in section 3.3). A RSM - GA approach was employed for modelling, analysis and optimisation of the wear behaviour of Al-Si10Mg/SiC\(_p\) composites. Modelling of wear rate using RSM requires the experiments to be conducted as per DoE, as discussed in the following section.

4.4.1 Experimental design

A three level second order Box Behnken design was adapted to study the linear, quadratic and two-factor interaction effects shown in Table 4.1. The upper limit of a process variable was coded as +1 and the lower limit as −1. The coded value of any variable for any intermediate value was determined using the relation

\[
x_i = \frac{2X - (X_{\text{max}} + X_{\text{min}})}{X_{\text{max}} - X_{\text{min}}}\quad (4.2)
\]

where \(X_i\) is the coded value of a variable \(X\) with any value between \(X_{\text{min}}\) and \(X_{\text{max}}\), the lower and upper limits of the variable. In the present study, three parameters, namely sliding speed, applied load and weight percentage of SiC\(_p\) were chosen as the variables.

Wear parameters used in experimentation and their levels are given in Table 4.1. The range of the parameters for experimentation as well as optimizations were selected based on preliminary trials.
Table 4.1 Experimental factors and their levels based on DoE

<table>
<thead>
<tr>
<th>Factor</th>
<th>Notation</th>
<th>Unit</th>
<th>Factor Level</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Natural</td>
<td>Coded</td>
<td>-1</td>
</tr>
<tr>
<td>Sliding speed</td>
<td>Speed</td>
<td>X₁</td>
<td>m/s</td>
</tr>
<tr>
<td>Applied load</td>
<td>Load</td>
<td>X₂</td>
<td>N</td>
</tr>
<tr>
<td>Weight percentage of SiC</td>
<td>%SiC</td>
<td>X₃</td>
<td>%</td>
</tr>
</tbody>
</table>

A second order mathematical model based on the first order equation, interaction between the terms and the squares of the terms was selected (Equation 4.3). Polynomial equation for three factors considered in the present case are given as

\[ Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{2} \sum_{j=i+1}^{3} b_{ij} X_i X_j + \sum_{i=1}^{3} b_{ii} X_i^2 \]  

(4.3)

where \( Y \) is the response, i.e. wear rate;

\( X_i \) is the coded values for speed, load and SiC\(_p\) as shown in Table 4.1.

\( b_0, b_i, b_{ij} \) and \( b_{ii} \), are the regression coefficients of the polynomial equation to be determined.
### Table 4.2 Box Behnken Design of Experiment matrix in Natural form

<table>
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<tr>
<th>Exp No</th>
<th>Coded</th>
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<th></th>
<th></th>
<th>Experimental wear (X 10^{-9} mm^3/m)</th>
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<td>X₁</td>
<td>X₂</td>
<td>X₃</td>
<td>Speed</td>
<td>Load</td>
<td>%SiC</td>
</tr>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>3</td>
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<td>1</td>
<td>30</td>
<td>20</td>
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</table>

#### 4.4.2 Development of Response Surface Model

Dry sliding wear tests were conducted for 15 different combinations of sliding speed, applied load and weight percentage of SiCₚ as shown in Box Behnken Design (Table 4.2.). Final mathematical model developed in coded forms to predict wear rate of Al-Si10Mg and its SiCₚ composites was
Wear rate = 1.94437 - 1.06826 X₁ + 1.00493 X₂ - 0.45214 X₃ + 0.440837  
X₁*X₁ - 0.413647 X₁*X₂ x 10⁻⁹ mm³/m  
(4.4)

Converting the above equation into natural forms using Equation (4.5) gives

Wear rate = 2.552 - 0.885* Speed + 0.813*Load - 0.045*%SiC +  
0.11*Speed*Speed - 0.103 Speed*Load x10⁻⁹ mm³/m (4.5)

where speed is the sliding speed (m/s), load is the applied load (N) and %SiC is the weight percentage of SiC.

The residual of experimental wear and predicted wear by the RSM is shown in Figure 4.7.

Figure 4.7 Residual of experimental wear and predicted wear by the RSM for Al-Si10Mg/SiCₚ composites

4.4.3 Analysis of the developed model

Analysis of Variance (ANOVA) was employed to confirm the fitness of the mathematical model. The associated P value for the model was
lower than 0.05 (i.e., $P=0.05$, or 95% confidence) confirms that the model can be considered to be statistically significant. The value of $R^2$ is 95.64 %, which means the regression model provides an excellent explanation of the relationship between the factors and the response (wear rate). Figure 4.7 shows the residual of experimental wear and predicted wear by the RSM showing good fit of the model. The ANOVA table for the quadratic model for wear rate is shown in Table 4.3.

**Table 4.3. Analysis of Variance for wear rate $X \times 10^{-9}$ mm$^3$/m**

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>SeqSS</th>
<th>AdjSS</th>
<th>AdjMS</th>
<th>F</th>
<th>P</th>
<th>% Contribution</th>
</tr>
</thead>
<tbody>
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<td>Speed</td>
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<td>1.0115</td>
<td>1.0115</td>
<td>9.8657</td>
<td>0.0119</td>
<td>43.1</td>
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<tr>
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<td>3.8428</td>
<td>3.8428</td>
<td>37.4796</td>
<td>0.0002</td>
<td>38.2</td>
</tr>
<tr>
<td>%SiC</td>
<td>1</td>
<td>1.6354</td>
<td>1.6354</td>
<td>1.6354</td>
<td>15.9508</td>
<td>0.0031</td>
<td>7.7</td>
</tr>
<tr>
<td>Speed*Load</td>
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<td>0.6844</td>
<td>0.6844</td>
<td>6.6753</td>
<td>0.0295</td>
<td>3.2</td>
</tr>
<tr>
<td>Speed*Speed</td>
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<td>0.7255</td>
<td>0.7255</td>
<td>0.7255</td>
<td>7.0762</td>
<td>0.0260</td>
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<tr>
<td>Error</td>
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<td>0.9228</td>
<td>0.9228</td>
<td>0.1025</td>
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<td>0.9228</td>
<td>0.9228</td>
<td>0.1318</td>
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<td>Pure Error</td>
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</table>

The contribution of various variable and their interactions is shown in Figure 4.8. Of the three variables, speed contributes to the maximum effect of 45% to wear as linear and another 4% as Squares, followed by Load with 40%. %SiC contributes to 8% of the total effect on wear. The interaction between speed and load contributes to another 3%.
4.4.4 Effect of variables on wear rate

Main effect plot of mean wear rate (Figure 4.8) was used to study the effect of individual parameters namely, speed, load and %SiC. The progress of wear was followed using a Scanning Electron Microscopy (JEOL 6360 JSM model), to confirm the results of RSM.
4.4.4.1 Effect of sliding speed

Increase in speed from 1 m/s to 5 m/s resulted in a decrease in wear rate by about 55-70% for under different combinations of load and %SiC. SEM micrograph of wear surface of the Al-Si10Mg/10SiCp composite under a constant load of 50N at 1 m/s (Figure 4.10a) showed deep grooves caused by ploughing and delamination in a few areas indicating the removal of the material and very little amount getting retransferred to the surface as delamination. In case of Al-Si10Mg/10SiCp composite under a constant load of 50N at 5 m/s (Figure 4.10b), the SEM micrograph of wear surface shows extensive surface delamination with furrows indicating the surface had become strain hardened. This is in agreement with results of Subramanian (1991) in near eutectic Al-Si alloys where as an increase in speed resulted in a decrease in wear rate. An increase in the sliding speed was found to increase the strain rate that in turn, increases the hardness or flow strength. This increase in the flow strength will reduce the true area of contact and thus result in a lower wear rate. Archard’s wear law states that the amount of wear is inversely proportional to the hardness the material. Thus, wear rate decreases with an increase in sliding speed.

The influence of speed on wear behaviour is linked to the inherent property of the Aluminium alloy matrix. It has been observed that the wear rate of AlSi12Mg decreased as sliding speed increased until it reached a minimum and thereafter it increased (C. Subramanian, 1991). At low speeds, deep grooves were formed because of ploughing of the soft matrix by hard reinforcements. In previous studies on wear behaviour of Al/SiCp composites (Kwok and S.C. Lim, 1999), it was observed that at speeds less than 3 m/s, the wear mechanism observed was abrasion and delamination, which later changed to a combination of abrasion, delamination, adhesion and melting. This may be due to the formation of transfer layer at higher speed, which in
turn prevented the direct metallic contact, thus reduces the wear rate. A similar behaviour was reported during the wear behaviour of AA6061 alloy and composites. (Rosenberger et al., 2005). SEM micrograph (Figure 4.10 b) further shows that the furrows replaced delamination mechanism observed in the Al-Si10Mg/10SiC_p at higher speeds.

![SEM micrograph of wear surface of Al-Si10 Mg/ 10SiC_p composite under a load 50N a) sliding speed of 1 m/s showing extensive grooves b) sliding speed of 5 m/s showing extensive delamination (Furrows)]

**Figure 4.10** SEM micrograph of wear surface of Al-Si10 Mg/10SiC_p composite under a load 50N a) sliding speed of 1 m/s showing extensive grooves b) sliding speed of 5 m/s showing extensive delamination (Furrows)

### 4.4.4.2 Effect of applied load

As the applied load increased from 10 N to 50 N, the wear rate increased (Figure 4.9) by about 60-70%. A higher degree of delamination was observed at lower applied loads, which reduces to minor delamination at higher applied loads. This may be due to greater compaction of subsurface leading to formation of dense laminated layers at higher applied loads. Dwivedi (2006) has also reported that in hypereutectic Al–Si alloy, the wear rate gradually increases with increasing applied load from 10 to 50 N.
SEM micrographs at low applied load and speed of 10 N and 1 m/s respectively in Al/10SiC composites (Figure 4.11.a) showed removal of bulk material as ribbons. Hard counter face of steel abraded the bulk material of the pin, leading to excessive plastic ploughing of the materials followed by mild delamination. Similar results have been reported during studies on wear behaviour of Al-Si12 alloy (Ramachandra and Radhakrishna, 2007). An increase in applied load increases the pressure on the pin resulting in an increase in the interfacial temperature, leading to the softening of the material and an increase in the plastic flow. SEM micrographs at low applied load and speed of 10 N and 1 m/s respectively in Al/10SiC composites (Figure 4.11.b) showed showing lesser grooves and more delamination. This result is in agreement with those obtained by Lim and others during dry sliding wear of Mg Alloy (C.Y.. Lim et al., 2003).

![Figure 4.11 SEM micrograph of wear surface of Al-Si10Mg/10SiC\textsubscript{p} composite under a sliding speed of 1 m/s and a) under a load 10N showing ploughing and delamination b) under a load 50N showing lesser grooves and more delamination](image)
4.4.4.3 Effect of reinforcement

Wear rate of both composites decreased with increase in SiC<sub>p</sub> content as shown in (Figure 4.7.). Wear rate decreased in the range of 15-50% for an increase in SiC<sub>p</sub> from 0 to 20% under varying conditions. This observed decrease may be attributed to the increase in hardness of SiC<sub>p</sub> composites compared to the unreinforced alloy. Similar results have been reported in wear of AA7009, AA7010 and A2024 (Rao and Das, 2010) and during wear studies on AlSi17 alloy (Soy et al., 2011). It may be seen that the wear mechanisms of Al-Si10Mg alloy shows severe ploughing (Figure 4.12a). However, as the % SiC<sub>p</sub> increased, the wear mechanism changed to delamination and severe abrasion in Al-Si10Mg/SiC<sub>p</sub> composites (Figure 4.12b) and delamination with mild abrasion (Figure 4.12c).

![Figure 4.12 SEM micrograph of wear surface under a load 50N and sliding speed of 1 m/s showing extensive ploughing for a) Al-Si10Mg alloy b) Al-Si10Mg/10SiC<sub>p</sub> composite and c) Al-Si10Mg/20SiC<sub>p</sub> composite showing a combination of grooves and mild delamination](image-url)
4.4.5 Response surface analysis of dry sliding wear

The main objective of the present investigation was to minimize wear rate. In addition the influence of wear rate due to the remaining process parameters, namely sliding speed, applied load and weight percentage of SiC\textsubscript{p} were of major interest. RSM model for wear rate was used to analyze the two-factor interaction effects on wear rate by plotting 3D-response surface plots. The interaction effects of (Speed- Load), (Speed - % SiC) and (Load - % SiC) on wear rate were analyzed keeping the other parameters at a constant level using RSM models.
Figure 4.13 Interaction effect of rate a) sliding speed and Wt% SiC<sub>p</sub> on WR (at constant load =30N) b) applied load and Wt% SiC<sub>p</sub> on WR (at constant speed =3m/s) c) sliding speed and applied load on WR (at constant SiC<sub>p</sub> % =10)
From Figures 4.13 a) and b) it may be concluded that at lower speed and absence of SiC$_p$, a higher wear rate is observed. With an increase in both speed and % SiC$_p$, the wear resistance is gradually improved. This is due to the combined effect of strain hardening at higher speed as well as the presence of SiC$_p$, which increases the hardness of the material. From the interaction of speed and applied load, it may be concluded that the wear rate gradually increased with increase in applied load while it showed a gradual decrease with an increase in speed (Figure 4.13 c). Wear rate is lower at higher speed and lower loads.

4.4.6 Optimization of wear conditions

Response surface analysis models may have a non-linear relationship between the wear rate and the process parameters. It is necessary to find the optimal process parameters to minimize the wear rate, even though the fact that there are three process parameters and finding the global optimum using the Response Surface Method becomes difficult. GA can be used as global search to find the global optimum of wear rate.

The objective of wear optimization was to find optimal values of Speed ($X_1$), Load($X_2$) and %SiC ($X_3$) to minimize wear rate, subject to constraints in coded form

$$-1 \leq X_1 \leq 1; \quad -1 \leq X_2 \leq 1; \quad -1 \leq X_3 \leq 1$$

Selection of the chromosomes to produce successive generations is important in Genetic Algorithm. Roulette wheel selection, a selection step involves the spin of a wheel that in the long run tends to eliminate the least fit population members. Number of mutation Chromosome in the next generation is termed as Mutation Count. Uniform Mutation and roulette wheel selection algorithm, suitable for minimization problems was adopted in the present study.
Genetic Algorithm methodology used for the study is discussed in section 3.8. The GA search starts with a population of chromosomes randomly generated within a prescribed search space. The population size of each generation was set as 50. Roulette wheel selection function was used to select the chromosomes. One point crossover and Uniform mutation with a probability of crossover and mutation as 0.8 and 0.01 was chosen respectively with an Elite Count of five. Stopping criterion of maximum 100 generations was set to limit the running of GA. The optimal result was obtained after 59th generation as shown in Figure 4.14. The optimized wear rate was obtained as \(0.5233 \times 10^{-9} \text{ mm}^3/\text{m}\) for the combination of values in coded form for sliding speed, applied load and weight % SiC as 0.589, -0.998 and 0.83 respectively. Converting these values in natural form using the Equation 4.2, a sliding speed = 4.132 (approximately 4 m/s), applied load =10.036 (approximately 10 N) and weight percentage of SiC = 18.31 (approximately 20) were obtained.

Figure 4.14 The Best fitness plots in each generation of Al-Si10Mg/SiC composites
4.4.7 Confirmation Test

A confirmation wear test was performed with sliding speed, applied load and weight percentage of SiC\textsubscript{p} obtained from GA (4 m/s, 10 N and 20\% SiC\textsubscript{p}). SEM micrograph of wear surface in Al-Si10Mg/20SiC\textsubscript{p} under a applied load of 10 N and sliding speed of 4 m/s (minimum wear rate condition) shows mild grooves with mild delamination (Figure 15) confirming a low wear rate. It was found that the experimental wear rate at this condition as $0.554 \times 10^{-9}$ mm$^3$/m with a prediction error rate as 5.6\%. Thus, it may be concluded that GA can be effectively used to predict the optimum wear characteristics of Al-Si10Mg/SiC\textsubscript{p} composites.

![SEM micrograph of wear surface](image)

Figure 4.15 SEM micrograph of wear surface of Al-Si10Mg/20SiC under a load of 35N and sliding speed of 5 m/s showing mild delamination.
4.5 SUMMARY

The application of RSM - GA optimization for wear rate of Al-Si10Mg alloy and its composites is presented in this Chapter. Mathematical model was developed based on RSM using experimental results as per Box Behnken design considering sliding speed, applied load and weight % SiC\textsubscript{p} as process parameters. Consistency of the developed model was additionally checked using supplementary random experiments. The minimization of wear rate was carried out by RSM-GA approach. Major conclusions drawn from the present investigation are

i. Wear rate increased with an increase in applied load. However, wear rate decreased with increase in sliding speed as well as %SiC\textsubscript{p}.

ii. GA-RSM method could predict the minimum wear rate condition with the accuracy of 94%.

iii. Minimum wear rate occurred in Al-Si10Mg/20SiC\textsubscript{p} under an applied load of 10N and sliding speed of 4 m/s.

iv. SEM investigations has been used to study the influence of process parameters on wear rate of composites.

v. SEM investigation has been used to confirm the results of RSM-GA models.