CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

The aluminium matrix composites are produced with single and binary reinforcements by compocasting process. The composites are subjected to mechanical testing to analyse its mechanical behaviour. The wear rates of composites are computed by experimentation based on Design of Experiments (DOE) developed by Response Surface Methodology (RSM). The statistical models are developed to predict and optimize the experiments by Response Surface Methodology (RSM). All the above procedures are discussed in detail in this chapter.

3.2 MATERIALS USED

LM13 aluminium alloy is used as base matrix, silicon carbide and graphite particles are used as reinforcements for this research work. The brief details about the materials are discussed in the following sections.

3.2.1 Matrix

Aluminium alloy based matrix is widely used in developing composites owing to the following properties as low density, chemical compatibility, thermal
stability, high compression strength, tensile strength and economic. Aluminium alloy is classified into wrought and cast alloys. Pistons are widely manufactured with LM13 aluminium alloy. LM13 is a eutectic (silicon -12%) cast aluminium alloy with density 2700 kg/m$^3$. The presence of silicon content around 12% enhances the properties of LM13 aluminium alloy with high resistance to wear, high load bearing capacity and low coefficient of thermal expansion. Thus LM13 is selected as metal matrix for this study. The chemical composition of LM13 aluminium alloy is shown in Table 3.1

### Table 3.1 Chemical Composition of LM13 Aluminium Alloy

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content %</td>
<td>83.42</td>
<td>13.02</td>
<td>0.310</td>
<td>1.047</td>
<td>0.252</td>
<td>0.865</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Ga</th>
<th>Zn</th>
<th>Ti</th>
<th>Pb</th>
<th>Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content %</td>
<td>0.938</td>
<td>0.014</td>
<td>0.0068</td>
<td>0.068</td>
<td>&lt;0.0050</td>
<td>&lt;0.0049</td>
</tr>
</tbody>
</table>

#### 3.2.2 Reinforcements

Reinforcements are added to improve various properties of material. In aluminium matrix composites, hard reinforcement such as Al$_2$O$_3$, SiC strengthens the matrix both extrinsically, through improved load transfer intrinsically by increasing the dislocation density. Soft reinforcements such as graphite and molybdenum disulphide (MoS$_2$) reduces wear rate, friction and increases seizure resistance.

In the present study, both hard and soft reinforcement has been used to produce composites. SiC is used as reinforcement to produce composite. The chemical reaction of SiC is low with aluminium alloy. It is found to be relatively cheap. A significant enhancement in composite properties, such as stiffness,
strength and fracture toughness, low reactivity and low cost makes SiC as an ideal reinforcement for manufacturing composites.

Graphite is a solid lubricant used as second reinforcement in this research work. Graphite exhibits superior lubrication, high load bearing capacity and surface speed performance compared to other solid lubricants. The mechanical properties of the reinforcements are shown in Table 3.2

<table>
<thead>
<tr>
<th>Reinforcements</th>
<th>Hardness (GPa)</th>
<th>Grain size (µm)</th>
<th>Density (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>24.5-29</td>
<td>25</td>
<td>3210</td>
</tr>
<tr>
<td>Gr</td>
<td>0.25</td>
<td>37</td>
<td>2267</td>
</tr>
</tbody>
</table>

### 3.3 PRODUCTION OF COMPOSITES

The LM13-SiC and LM13-SiC-Gr composites are fabricated by double stir casting or compo casting process using stir casting furnace setup as shown in Figure 3.1. In the first step, based on the experimental design a measured quantity of aluminium alloy was placed inside graphite crucible of stir casting furnace. The temperature of furnace was maintained at 800°C and the alloy was melt in graphite crucible. Meanwhile based on the experimental design a measured quantity of reinforcements, silicon and graphite was pre-heated at 400°C for about 30 min in a pre-heating furnace as shown in Figure 3.1. The main aim of preheating is to remove surface impurities if any and to reduce oxide formation during the process. Once the metal attains liquid state it was stirred continuously at 600-800 rpm to remove any unwanted gases formed during melt and in order to enhance the degassing process hexochloroethane (C₂Cl₆) tablet was added to the melt and was stirred continuously. After degassing the melt was stirred continuously to create a vortex, and the preheated SiC and Gr particles were added slowly and continuously.
into the vortex of the molten metal. In order to enhance wetting of reinforcements with aluminium alloy, 1wt% of Mg metal powder was added to the molten metal. The melt was then stirred continuously and the stirrer was moved up and down frequently within the mixture for about 10 min to ensure uniform distribution of the added particles. After this the furnace was set to 550°C and the composite mixture was allowed to attain a semi-solid state in the crucible. In the second step the slurry mixture was reheated to a 750°C. Once the slurry attains the molten state it was stirred again at 300 rpm for 2 min. Finally it was poured into a 100 mm x 100 mm x 10 mm pre-heated mild steel mould. The melt was allowed to solidify in atmospheric air and it was later removed from the mould. The composites with single and binary reinforcements with different wt. % were produced by the same procedure.

Figure 3.1 MMC - Stir Casting Setup
3.4 MICROSTRUCTURAL ANALYSIS OF COMPOSITES

Characterization of composite was done to analyse surface morphology, microstructures, surface contaminations, spatial variations, elemental distribution and crystalline structures. Square specimen with side 10 mm is used to carry out characterization study on the composites. The specimens are prepared by standard metallographic procedures followed by etching. The SEM and EDS images of alloy and composites are obtained from ZEISS-Field Emission Scanning Electron Microscope (FE-SEM) shown in Figure 3.2.
3.5 MECHANICAL BEHAVIOUR OF COMPOSITES

The mechanical properties of composites and matrix alloy were tested at room temperature with 55% relative humidity. The specimens are obtained by machining the cast alloys and composites using a wire cut EDM machine with a dimensional tolerance ± 0.01mm. The equipments and procedures for conducting tests are described in the following sections.

3.5.1 Micro Hardness (HV)

The micro hardness (HV) of alloys and composites was measured using Vickers hardness or micro hardness tester (MITUTOYO-MVK-H1) shown in Figure 3.3. The tests were conducted according to ASTM A-370 standard on polished specimens at 5 different locations with an applied load of 500 g for duration of 15 s.
3.5.2 Macro Hardness (HRC)

The macro hardness (HRC) of alloys and composites was measured using Rockwell hardness tester (RAB-250) shown in Figure 3.4. The tests were conducted according to ASTM E-18 standard on polished specimens at 5 different locations with an applied load of 100 kgs for duration of 15 s
3.5.3 Ultimate Tensile Strength (UTS)

The UTS of alloys and composites was measured using computerized Universal Testing Machine (UTM) (HITECH TUE-C-1000) shown in Figure 3.5. The tensile tests were conducted with specimens prepared according to ASTM E8M-04 standard shown in Figure 3.6.

Figure 3.5 Computerized Universal Testing Machine
3.6 DRY SLIDING WEAR TEST OF COMPOSITES

The wear experiments are conducted with specimens obtained from cast alloy and composites. The specimens are prepared with dimensions 10 mm x 10 mm x 50 mm respectively. The end surface of the composite pins are cleaned and polished with 600 grade followed by 1000 grade abrasive paper. The dry sliding wear experiments were conducted at room temperature according to ASTM G9905 standard using pin-on-disc machine (DUCOM TR20-LE) shown in Figure 3.7. The sliding disc is made up of grey cast iron with micro hardness 261 (HV) and surface roughness 0.0001microns. A computer aided data acquisition records the height loss. The experiments are conducted based on design matrix and the wear rate is computed for 31trial runs using Equation (3.1).

\[
\text{Wear Rate} = \frac{V_L}{D} \quad (3.1)
\]

Where,

\(V_L\) - Volume loss in \(m^3\)

\(D\) - Sliding distance in m
3.7 STATISTICAL ANALYSIS BY RESPONSE SURFACE METHODOLOGY (RSM)

RSM is as a statistical tool establishes relation between input factors and responses with quantitative data from experimentation. It is used to form multivariate regression equations to develop empirical models for predicting and optimising the input parameters. RSM is used to design experiments, statistically build empirical models with experimental results and optimize input parameters to achieve the desired results with the given conditions. The procedure to develop RSM model is explained below.

Step 1: Determination of the influencing factors and its levels affecting the experiments are to be examined.
Step 2: Generation of a response surface design (Central Composite or Box-Behnken Design)

a. Central Composite Design (CCD): It is mostly recommended when sequential experimentation has to be performed. CCD handles information through a planned factorial experiment. This design can effectively handle experiments with two or more factors and its various levels.

b. Box-Behnken Design: It is used when experiments are performed is non-sequential and only once. This design helps to estimate the first and second-order coefficients. Since this have limited design points it requires only less time to generate design of experiments compared to central composite design. This design ensures all factors are not set their highest levels simultaneously. A comparison of CCD and Box Behnken design are shown schematically in Figure.3.8.

![Figure 3.8 Response Surface Design for Three Factors](image)

- a) Central Composite Design
- b) Box-Behnken Design

Step 3: Experimentation and collection of response data.

Step 4: Development of a Response Surface model with experimental data.

The response surface model is built with the experimental results. The statistical significance of a model is found using Analysis of Variance (ANOVA) and regression model is developed to predict and optimize the experimentation. Response surface and contour plots gives an insight about effect of input parameters
on responses and it is also used to optimize the input parameters affecting the responses. RSM is being frequently useful in modelling and optimisation of processes. If \( X_1, X_2... X_n \) denotes independent variables and \( Y \) is a dependant variable on independent variables, then \( Y \) is called the response. Thus \( Y \) is written as function of many independent variables as \( Y = f(X_1, X_2... X_n) + E \), where \( E \) is a random error constituent. By plotting desired response \( Y \) with independent variables \( X_1, X_2... X_n \), the surface obtained is called response surface. The form \( f(X_1, X_2... X_n) \) is unknown and complicated to be solved. Thus, RSM simplifies form \( f(X_1, X_2...X_n) \) to a lower-ordered polynomial in some regions of independent process variables. Thus RSM is not only used to examine the response over the full space of the factors, but also helps in region of importance to obtain optimal value.

The best response can be obtained by examining the combination of factors in a response surface model. The values of linear regression coefficients, square and interaction terms of RSM models can be determined using Equation (3.2).

\[
B = (X^TX)^{-1}XTY
\]

Where,

\( B \) - Estimate of factor matrix
\( X^T \) - Transpose matrix
\( X \) and \( Y \) - Response matrix

The coefficient \( R^2 \) is used to test the goodness-of-fit of a model and it also provides a variability measure of response with input factors. The error between the predicted model and experimental is calculated with Equation (3.3).

\[
Average\ Error\ % = \frac{100}{n} \sum_{1}^{n} i \left( \frac{Y(Exp)_{i} - Y(Pred)_{i}}{Y(Exp)_{i}} \right)
\]

Where,

\( Y(Exp)_{i} \) - Measured response corresponding to \( i^{th} \) trial
\( Y(Pred)_{i} \) - Predicted response corresponding to \( i^{th} \) trial
\( n \) - Number of data sets
Step 5: Analysis of Variance (ANOVA)

ANOVA was developed by Sir Ronald Fisher a British statistician. It is a method of partitioning the sources of variation and degrees of freedom in an experiment. The F-test is simply a ratio of sample variances. Comparing the F-ratio of a source with the tabulated F-ratio is called the F-test. By performing ANOVA on a set of data the respective sum of squares and corrected sum of square are obtained. The contribution of each factor is obtained by comparing corrected sum of squares and total sum of squares. The error denotes the adequacy of model. Since ‘error’ is an unknown effect it cannot be controlled. If error is less than 15%, it can be assumed that no important factors have been omitted from the experiment.

Step 6: Use of Contour and Surface plots

It is used to analyse the effect of interaction on response. It is used in establishing desired response with the operating conditions. A contour plot gives a two-dimensional view, where all the points with same response are connected together forms contour lines. A surface plot displays response surface in three-dimensional view which gives a vivid picture of interactions and response.

3.8 SUMMARY

Wear in a material is affected by various parameters and it is highly important to analyse its wear behaviour. Selection of best operating conditions is always a challenge in traditional method. It requires many experiments to be conducted to achieve a satisfactory result. In any process, the desired solutions are obtained either by experience or from reference sources. However it does not provide any optimal solution for a problem. An approach based on DOE technique is proved to be the best possible solution with minimum number of experiments. RSM is one such versatile technique used to obtain optimal solution of any process.