CHAPTER III

MATERIALS AND METHODS

3.1 OVERVIEW
In this chapter, details of the materials, processing methods, SPD processing, wear test, analysis methods and experimental procedures are introduced. The main procedures of the study are as follows: firstly, fabricate the composites secondly, process the materials by using SPD method (ECAP) with two and four passes. Thirdly, measure the mechanical properties (microhardness or tensile testing) of each sample fourth, wear testing of all the samples. Fifth, examine the wear surface and debris to confirm the wear mechanism.

Aluminum 6061 alloy was used in this work as matrix and alumina and red mud in equal quantities were used as particulate reinforcement. Four percentage reinforcement weight fractions of reinforcement particles in the range from 2.5, 5.0, 7.5 and 10.0% were used as reinforcements to develop aluminium based hybrid metal matrix composites using conventional compocasting process followed by hot extrusion. Developed composites were subjected to ECAP Processing. Both extruded and ECAP processed composites were characterized for various properties.

3.2 SELECTION OF MATERIALS
The reasons for selecting the materials used in this research are given below.

3.2.1 Matrix
Matrix used in this study is aluminium 6061 alloy whose composition is in Table 3.1.

Table 3.1: Chemical Composition of AA6061 Aluminum Alloy

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Ti</th>
<th>Cr</th>
<th>Zn</th>
<th>Mn</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>0.85</td>
<td>0.69</td>
<td>0.14</td>
<td>0.24</td>
<td>0.02</td>
<td>0.02</td>
<td>0.004</td>
<td>0.03</td>
<td>Bal</td>
</tr>
</tbody>
</table>
Aluminium 6061 is most versatile and extensively used aluminium alloys in 6000 series. Magnesium and silicon are major alloying elements in aluminium 6061 alloy. Aluminium 6061 possesses good strength, high machineability, good hardness and also light in weight. But some of properties such as low wear resistance have limited the application of this material. Aluminium 6061 alloy was selected as matrix owing to its cost and useful specific properties.

3.2.2 Reinforcement
The reinforcement materials selected for investigation are alumina (Al₂O₃) and red mud powders mixed in equal proportion having densities 3.9gm/cm³ and 3.05gm/cm³ respectively. The properties of alumina and chemical composition of red mud particles are presented in Tables 3.2 and 3.3 respectively. EDS spectra of Al₂O₃ and red mud are shown in Appendix B. Reinforcing phase consists of 2.5, 5.0, 7.5, and 10.0% of reinforcement mixture.

Table 3.2: Properties of Alumina

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Point</td>
<td>2,072°C</td>
</tr>
<tr>
<td>Density</td>
<td>3.95gm/cm³</td>
</tr>
<tr>
<td>Hardness (VH)</td>
<td>2600kg/mm/mm</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>530 GPa</td>
</tr>
<tr>
<td>Compressive Strength</td>
<td>2000-4000MPa</td>
</tr>
</tbody>
</table>

Table 3.3: Red Mud Chemical composition

<table>
<thead>
<tr>
<th>Constituents</th>
<th>%</th>
<th>Constituents</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>14.41</td>
<td>MgO</td>
<td>0.049</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>48.50</td>
<td>ZnO</td>
<td>0.027</td>
</tr>
<tr>
<td>TiO₂</td>
<td>5.42</td>
<td>CaO</td>
<td>3.96</td>
</tr>
<tr>
<td>SiO₂</td>
<td>11.53</td>
<td>V₂O₅</td>
<td>0.116</td>
</tr>
<tr>
<td>Na₂O</td>
<td>7.50</td>
<td>P₂O₅</td>
<td>0.297</td>
</tr>
<tr>
<td>MnO</td>
<td>0.17</td>
<td>Others</td>
<td>Balance</td>
</tr>
</tbody>
</table>
Reasons for selecting Al₂O₃ and red mud particles are commercial availability, low cost and high hardness value of Al₂O₃ particulates (although not as hard as SiC), and red mud is a byproduct of aluminium manufacturing by Bayers process the major constituents of red mud are Al₂O₃ and Fe₂O₃ and is available free of cost thus reducing the overall reinforcement cost.

3.3 BLENDING OF REINFORCEMENT POWDERS
The reinforcement powders Al₂O₃ and Red mud were weighed in equal quantities to produce a mix, which would result in a composite containing the equal volume or weight ratios of reinforcement to matrix. The moisture from the powders was removed by drying in an oven at 120°C before blending. The powders were then dry blended for 30 minutes. The Al₂O₃ particles were found to be less agglomerated when compared to red mud during blending.

3.4 FABRICATION OF Al₂O₃ RED MUD COMPOSITES
Conventional melt stirring technique described by Surappa and Rohatgi [179] in the recent years was used for fabricating the composites. The aluminum 6061 alloy was melted using an oil fired furnace. The furnace temperature was maintained at 720°C for 10 minutes after melting. Melt temperature was reduced to 620°C and a stainless Steel stirrer was then immersed into the melt and allowed to rotate at 450 rpm. Reinforcements Preheated to 250°C were introduced into the vortex while Stirring continued till all the reinforcement was added to the alloy in semi solid state.

Figure 3.1: Cast Ingots of Fabricated Composites
The semi solid composite melt was cast into ingots in preheated metal moulds. Temperature at the time of pouring was kept higher than the stirring temperature to increase fluidity. The cast ingots are shown in the Figure 3.1.

### 3.5 SECONDARY PROCESSING (EXTRUSION)

The cast composites machined were hot extruded with a ratio 12:1 using a 50T capacity hydraulic press. The extrusion temperature was 540°C. The composite work pieces were heated to 540°C for 30 min in an oven prior to extrusion. Molybdenum grease served as a lubricant. Rods of diameter 20mm as shown in Figure 3.2 were obtained which were used for further processing and characterization.

![Figure 3.2: Extruded rods of Composites](image)

### 3.6 ECAP PROCESSING

Extruded composites samples 120 mm long and 20 mm diameter were ECAP processed on a hydraulic press. All the samples were pressed using route C. Molybdenum grease was applied on the work piece as lubricant to minimize the friction during processing. All the composites were processed for two and four ECAP
passes at a processing temperature of 250°C. A specially designed die used in this work is shown in Figure 3.3, with an internal angle $\phi = 120^0$.

The angle of arc of curvature $\psi = 0^0$. The two channels had equal cross-sections. It was noted that the composites could sustain up to four passes beyond which the samples cracked. Figure 3.4 shows the ECAP processed samples.

**Figure 3.3: ECAP Die and Processed Piece**

**Figure 3.4: ECAP Processed Samples**
3.7 MICROSTRUCTURAL CHARACTERIZATION
Aluminium 6061 and composite was polished with different grades of sand papers. Final polishing was carried out on polishing cloth using diamond paste of 6 and 1µm. Keller’s reagent was used for etching to reveal the microstructure. Microscopic examinations were performed on the composites and matrix alloy using an optical and scanning electron microscope.

3.8 DENSITY MEASUREMENT PROCEDURE
The densities of the composites were measured by Archimedes principle [180]. The densities were calculated by the following Equation 3.1.

\[
\rho = \frac{m_1 \rho_1}{m_2}
\]

\( \rho \) = composite density
\( \rho_1 \) = density of liquid (water)
\( m_1 \) = mass of composite measured in air
\( m_2 \) = mass of liquid displaced by test piece

Rule of mixtures was used to calculate theoretical densities [181]. Using Equation 3.2 below, the theoretical densities of the composites can be deduced:

\[
\rho_c = V_m \rho_m + V_r \rho_r
\]

\( \rho_c \) = composite density
\( \rho_m \) = matrix density
\( \rho_r \) = reinforcement density
\( V_m \) = matrix volume fraction
\( V_r \) = reinforcement volume fraction
By dividing Equation 3.1 by Equation 3.2 and multiplying by 100 the percentage of theoretical density obtained in the actual composite samples produced can be obtained from Equation 3.3 below:

\[
\left(\frac{\rho_1 \rho_m}{\rho_r \rho_m + V_r \rho_r}\right) \times 100
\]

Equation 3.3 expresses the measured density as a percentage of the ideal density. This percentage density will be used in the forthcoming results section to determine how close to ideal density the processed composites are.

### 3.9 HARDNESS

The Rockwell hardness of matrix and composites were ascertained using Rockwell hardness tester. Prior to testing the samples were polished to a 1\(\mu\)m finish. The values reported are average of five measurements.

### 3.10 TENSILE TESTS

The tensile tests of extruded and ECAP processed hybrid composite samples were carried out using UTE 40 make universal testing machine. Cylindrical tensile tests specimens as shown in Figure 3.5 were prepared as per ASTM E-8 standards.

![Figure 3.5: Tensile Test Specimens after Tensile Test](image-url)
3.11 Fractography
Fractography was carried out using scanning electron microscope (Model: Hitachi S 3700N) with the objective of establishing the failure modes. Tensile fracture surfaces of alloy and composites were characterized to study the fracture behaviour.

3.12 TRIBOLOGICAL (WEAR) MEASUREMENTS
The dry sliding wear performance of processed composites was determined by the weight loss from the composite. Experiments to assess the wear performance of the composites were conducted using pin on disc tribometer (Magnum Make) as shown in Figure 3.6 connected to a computer with data acquisition system. Wear tests were carried out according to ASTM G-99 at room temperature of 28-35°C. Wear tests were performed at BITS Pilani Hyderabad Campus.

Cylindrical pins of composites as shown in Figure 3.7 having 6mm diameter and 25mm long were utilized as wear test samples. Counter body consisted of a disc 8mm thick and 170mm in diameter made out of EN24 material and hardened to HRC 56. To calculate mass loss sample pins were weighed before and after each test to an accuracy of 0.01mg. Data acquisition system connected to a computer was used to monitor the values of frictional force during the test, thus facilitating calculation of friction coefficient.

Wear tests were undertaken under nominal loads 10, 20 and 30N at 200 and 300RPM and fixed sliding distance of 500m. Weight loss due to wear was measured by finding the weight of Pins before and after each test. Study and characterization of Wear surfaces was carried out using scanning electron microscope.
Figure 3.6: Pin on Disc Tribometer Used for the Wear Tests

Figure 3.7: Composite Pins for Wear Tests