CHAPTER 3

EXPERIMENTAL AND CHARACTERIZATION DETAILS

3.1 NEED FOR THE STUDY

Literature survey confirms that TiB$_2$ and graphite reinforced into the aluminum matrix is utilized to make components requiring tensile, and great wear resistance. There was no attempt to relate the wear behavior of Al6061-10TiB$_2$-1Gr and Al6061-10 TiB$_2$-2Gr composite in micron and nano-level in the earlier research. The various characterization techniques and the different experiment techniques are discussed in this section. The most influencing parameters of tribology are identified and the relations between the tribological properties are to be discussed in the next section.

3.2 MATERIALS AND METHODS

3.2.1 Materials

The materials used for the present study were Al6061, TiB$_2$ and Gr, and the details of their compositions are given in Tables 3.1(1-4) separately. The Al and the reinforcing ingredient of the received TiB$_2$ powder has the particle size of 30–50 µm and 1–10 µm, and the graphite particle size varies from 25 to 50 µm. Figures 3.1 and 3.2 show the SEM images of the received powders Al, TiB$_2$ and Gr.
Table 3.1  Chemical composition of Al6061

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Fe</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>V</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight%</td>
<td>1.08</td>
<td>0.17</td>
<td>0.63</td>
<td>0.32</td>
<td>0.52</td>
<td>0.01</td>
<td>0.02</td>
<td>Remaining</td>
</tr>
</tbody>
</table>

Table 3.2  Chemical composition of TiB<sub>2</sub> powder

<table>
<thead>
<tr>
<th>Element</th>
<th>Ti</th>
<th>B</th>
<th>O</th>
<th>C</th>
<th>Fe</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight %</td>
<td>67.60</td>
<td>31.04</td>
<td>0.45</td>
<td>0.25</td>
<td>0.09</td>
<td>0.26</td>
</tr>
</tbody>
</table>

Table 3.3  Chemical composition of Gr powder

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight %</td>
<td>95.00</td>
<td>0.10</td>
<td>0.50</td>
</tr>
</tbody>
</table>

Table 3.4  Details of TiB<sub>2</sub> and Gr powder

<table>
<thead>
<tr>
<th>Reinforcement</th>
<th>Hardness (GPa)</th>
<th>Particle size</th>
<th>Density (g/cm&lt;sup&gt;3&lt;/sup&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiB&lt;sub&gt;2&lt;/sub&gt;</td>
<td>27</td>
<td>30-50 µm</td>
<td>4.52</td>
</tr>
<tr>
<td>Gr</td>
<td>0.25</td>
<td>25-50 µm</td>
<td>2.09-2.23</td>
</tr>
</tbody>
</table>

Figure 3.1  SEM image of the received powder- Al
51

Figure 3.2 SEM images of the received powders (b) TiB$_2$ (c) Gr

3.3 POWDER CHARACTERIZATION

The particle size and distributions of the milled powder composites were described through the SEM. The elements presented in the composite powders were identified and the particle size was measured through XRD. TEM was used to confirm the milled particle size of the powders in nano-scale. EDS is also used to find the elemental analysis.
3.3.1 Ball Milling

Figure 3.3 shows the image of a ball mill. The Gr powder was crushed into nano-scale and mixed with TiB₂ and Al6061 powders using high energy ball mill (Planetary mono mill, Fritsch, Germany-Pulverisette-6) with tungsten carbide vials using 10 mm diameter tungsten carbide balls. The ball-to-charge weight ratio was 20:1. Milling was done at 300 rpm in a wet medium, in the presence of toluene to prevent oxidation and agglomeration of the charges.

Figure 3.3 Ball mill

The milling effect in the Gr particles was characterized by using the SEM, transmission electron microscope (TEM) and particle size analyzer image. Figure 3.4 (a) shows the SEM image of the Gr powder after 10 hrs. Figure 3.4 (b) is the particle size analyzer image of the Gr powder after 10 hrs. It confirms that the average particle is 750 nm. Figure 3.4(c) shows the TEM image of the Gr powder after 20 hrs. Figure 3.4(d) is the particle size analyzer
image of the Gr powder after 20 hrs. It confirms that the average particle is 60 nm. The particle analyzer inset shows the initial wide diffraction peaks of Gr getting sharpened and reduced in intensity, when reduced in size.

Figure 3.4  (a) SEM image of the Gr powder after 10 hrs milling. (b) Particle size analyzer image of the Gr powder after 10 hrs. (c) TEM image of the Gr powder after 20 hrs. (d) Particle size analyzer image of Gr powder after 20 hrs

3.4 MIXING

The Al6061 and hybrid composites of Al6061 were prepared by the P/M method by adding 10 wt% of TiB₂ and 1 and 2 wt% of nano-Gr after mechanical alloying. The mechanical alloying was performed for 2 hr. During the mixing Al, TiB₂ and nano-Gr were mixed in the same bowl. Figure 3.5(a) is the SEM image of the Al6061– 10TiB₂–1Gr nano-powder composite after
mechanical alloying at 2 hr and Figure. 3.5 (b) is the SEM image of the Al6061–10TiB₂–2Gr nano powder composite after mechanical alloying at 2 hr.

Figure 3.5  (a) SEM image of the Al6061–10TiB₂–1Gr nano-powder composite. (b) SEM image of the Al6061–10TiB₂–2Gr nano-powder composite after mechanical alloying

3.5 X-RAY DIFFRACTION ANALYSIS

X-ray diffraction (XRD) investigations of the prepared powder composites were carried out using (PANalytical, Model: XPERTPRO). The XRD results of the prepared Al6061, Al6061–10TiB₂–1Gr hybrid nano-composite and Al6061–10TiB₂–2Gr hybrid nano-composite are shown in Figure. 3.6 (a)–(c). Peak values were collected over the 2θ range of 10–100° with a step size of 0.0170° and step time of 20.3142 sec. All the samples showed wide diffraction peaks, which could be indexed to the structure of Al6061, TiB₂ and Gr.
They revealed that the characteristic peaks in the XRD patterns were consistent with JCPDS files no. 89-2837, 07-0275 and 89-8487 for Al6061, TiB₂ and Gr respectively.

(a) XRD pattern of the Al6061 powder
Figure 3.6 (b) shows the XRD pattern of the Al6061–10TiB$_2$–1Gr hybrid powder composite and Figure 3.6 (c) is the XRD of the Al6061–10TiB$_2$–2Gr nano-powder composite.

Figure 3.6 (b) and (c) reveal that the intensity of TiB$_2$ was greater in the (101) plane ($2\theta=44.6714^\circ$, JCPDS 07-0275). In addition, the intensity of TiO$_2$ and CuO was observed at different peaks and confirmed through JCPDS software, notably at $2\theta=65.0960^\circ$, JCPDS 89-4746 (TiO$_2$ 110), $2\theta=38.3029^\circ$, JCPDS 89-2531 (CuO 111) and $2\theta=44.5010^\circ$, JCPDS 39-1483 (AlB$_2$ 101).
Figure 3.6 (a) XRD pattern of the Al6061 powder, (b) XRD paten of the Al6061–10TiB$_2$–1Gr hybrid powder composite (c) XRD paten of the Al6061–10TiB$_2$–2Gr hybrid powder composite.

Figure 3.6 (b) and (c) show the presence of aluminum (in the largest peaks), notably at 2θ=38.7618°, 65.3376°, 78.4779°, 82.7099° of (110), (200), (311), (222) of the diffraction peak of Al6061, corresponding to the file name (JCPDS 89-2837). The presence of TiB$_2$ particles (indicated by minor peaks) was found corresponding to 2θ angle 45.0223°, 61.3770°, 78.3867°, 88.6000° of (101), (110), (201), (112) of the diffraction peaks of TiB$_2$ corresponding to the file no. (JCPDS 07-0275) and carbon (indicated by minor peaks), which were identified through the 2θ angle 26.9314°, 61.4688°, 68.5539° of (200), (220), (113), JCPDS software (89-8487), (89-8498), (89-8493) respectively.
A clearly visible carbon peak can be observed in the hybrid composites. In addition, the intensity of TiO$_2$ and Ti was observed at different peaks and confirmed through JCPDS software, notably at Ti, $\theta=55.2646^0$, $82.8910^0$ of (200), (201), JCPDS (89-4913, 89-4893) and TiO$_2$, $\theta=65.3376^0$ of (125), JCPDS (89-4746) respectively. A gradual marginal shift of the Al peaks to higher angles, with an increase in the wt% of Gr content in the hybrid composite is also evident from Figure 3.6 (b) and (c).

### 3.6 COMPOSITE PREPARATION

#### 3.6.1 Compaction

The cylindrical compacts were prepared using Al, TiB$_2$ and nano-Gr powders.

![Schematic representation of compaction using UTM](image)
Figure 3.7 demonstrates that schematic representation of compaction using UTM. In these machines, the process of the powders were blended for 120 minutes. The utilization compacted with appropriate punch and die set assembly on a Universal Testing Machine having 1 MN limit. Metal Powder Industries Federation (MPIF) standard 42 was utilized for compaction.

3.6.2 Sintering

The sintering was completed in a inert gas which was circulated in electric muffle furnace at 560°C (833K) for a holding time of 60 minutes. When the sintering schedule was over, the sintered performs were cooled in the heater inside to the room temperature. Figure 3.8 demonstrates the schematic diagram of sintering furnace. To forestall oxidization, the green compacts were at first secured with dormant argon air in the furnace. After the culmination of sintering, the performs were cleaned by a fine wire brush. The theoretical density of the composite was measured by utilizing the Archimedes principle as depicted in ASTM B32 standard.

![Sintering furnace setup](image)
3.6.3 Extrusion

The sintered preforms were extruded by the extrusion die set assembly so that the pore size is decreased and the relative density is expanded. Amid this procedure, the preforms were warmed to 300°C by utilizing an inert gas circled electric muffle furnace. The diameter of the specimens was reduced in different steps to achieve the preform density of 92% from 85% for micro-level composite and the nano-level the density increased from 87% to 94%. During the extrusion, the diameter was reduced by 12 mm. Further, the diameter of the preforms was reduced from 12 to 8 mm by turning operation. The diameter of the specimen was reduced in different steps to get the density of extruded specimen as 99%. The turned preforms were polished using 600 grit silicon carbide papers. Figure 3.9 shows the detail view of the hot extrusion setup.

![Figure 3.9 Exploded view of hot extrusion set up](image_url)
(a) OM image of the Al6061−10TiB₂−1Gr hybrid composite

(b) SEM of the Al6061−10TiB₂−1Gr hybrid composite
(c) OM image of the Al6061–10TiB₂–2Gr hybrid composite

(d) SEM image of the Al6061–10TiB₂–2Gr hybrid composite
Figure 3.10 (a) OM image of the Al6061–10TiB$_2$–1Gr hybrid composite (b) SEM of the Al6061–10TiB$_2$–1Gr hybrid composite (c) OM image of the Al6061–10TiB$_2$–2Gr hybrid composite (d) SEM image of the Al6061–10TiB$_2$–2Gr hybrid composite (e) EDS pattern of the Al6061–10TiB$_2$–2Gr hybrid composite

Figure 3.10(a) shows the optical microscope (OM) image of the extruded Al6061–10TiB$_2$–1Gr hybrid composite, and it confirms that all the grains are uniform in size. Figure 3.10 (b) is the SEM image corresponding to the Al6061–10TiB$_2$–1Gr hybrid composite. It confirms that there is no pore. Figure 3.10 (c) shows the OM image of the Al6061–10TiB$_2$–2Gr hybrid composite. It confirms the presence of the Al matrix and reinforcements also shows how Al completely binds the primary reinforcements TiB$_2$ and Gr particles and their alloying elements. Figure 3.10 (d) confirms that the bonding between the Al matrix and the reinforcements is good. Energy dispersive spectrum (EDS) is used for the elemental analysis of the hybrid composite sample. Figure 3.10 (e) shows the EDS pattern of Al6061-10TiB$_2$–2Gr hybrid composite after extrusion with peaks of Al, titanium, boron and Gr.
3.7 SUMMARY

In this present research work, Al6061-based composites and hybrid composites were prepared using P/M, and the characterization was performed with XRD, EDS, particle size analyzer, TEM and SEM. Based on the present experimental and simulation work, the following conclusions were drawn:

XRD pattern confirms the crystalline plane through the different peak intensity using JCPDS software and EDS results confirms the presence of the functional elements in the Al6061–10TiB₂–2Gr hybrid composite.