CHAPTER 3

FABRICATION, METALLURGICAL AND MECHANICAL CHARACTERIZATION OF AA6061/AlN$_p$ COMPOSITE

3.1 INTRODUCTION

Aluminium matrix composites (AMCs) are innovative materials and they have opened up unlimited scope for modern material science and development. Desirable properties, depending on application requirements, can be imparted to the AMCs by way of suitable fabrication procedures. In recent days, this material group is being widely employed for use as constructional and functional materials. This chapter describes the fabrication of cast AA6061 alloy and AA6061 matrix composite containing 5, 10, 15 and 20 weight percentage of AlN$_p$ reinforcement fabricated by an indigenously developed modified stir casting furnace with bottom pouring attachment. The effects of weight percentage of AlN$_p$ on the metallurgical and mechanical properties of composite are also described.

3.2 FABRICATION OF AA6061/AlN$_p$ COMPOSITE

The indigenously developed modified stir casting furnace with bottom pouring arrangement (manufactured by M/s. SWAMIEQUIP, Chennai, India) was used to fabricate the composites. Extruded aluminium alloy (AA6061-T6) rods of 25 mm diameter were cleaned by acetone and placed inside the coated stainless steel crucible of the furnace. The chemical composition of AA6061-T6 alloy rods is presented in Table 3.1.
Table 3.1 Chemical Composition of AA6061–T6 Alloy Matrix

<table>
<thead>
<tr>
<th>Element</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Mn</th>
<th>Cu</th>
<th>Cr</th>
<th>Zn</th>
<th>Ni</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt.%</td>
<td>0.9</td>
<td>0.64</td>
<td>0.26</td>
<td>0.1</td>
<td>0.21</td>
<td>0.05</td>
<td>0.04</td>
<td>0.02</td>
<td>0.01</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Temperature of the electric furnace was set to 1000 °C. When the temperature of the furnace reached 650 °C, argon gas was supplied into the crucible at a constant flow rate of 2 lpm to avoid the reaction of matrix with the atmospheric air. When the temperature reached 1000 °C, 2% magnesium of total weight of the composite to be produced was added to the melt to increase the wettability of AlNₚ with AA6061 alloy (Nassaj et al 1995). A coated stainless steel stirrer coupled with electric motor was immersed into the molten aluminium alloy and stirred at a constant speed of 450 rpm to form a vortex. In order to avoid any contaminations at high temperatures, inner side of the stainless steel crucible and stainless steel stirrer were coated with WOLFRA coating. Stirring was carried out to facilitate both incorporation and uniform distribution of the reinforcement (AlNₚ) in the molten aluminium alloy (Nassaj et al 1995). Proper stirring speed is necessary to avoid excessive gas which will form due to over agitation of melts and lead to unacceptable porosity in the cast (Hashim et al 1999).

A predetermined quantity of AlNₚ of size 3 to 4 µm and purity of more than 99.6% was preheated to 750 °C in an another electric furnace. Preheating of reinforcement was carried out in order to (i) release adsorbed gases by particles, (ii) remove impurities from the particle surface and (iii) avoid drop of melt temperature (which was caused by adding non preheated particles) which would increase the viscosity resulting in difficulties in removing the entrapped gases. The preheated AlNₚ was added into the molten matrix at the side of the vortex. Figure 3.1 shows the indigenously developed modified stir casting furnace with bottom pouring attachment and the furnace used to preheat the AlNₚ reinforcement.
AlN$_p$ reinforcement was incorporated into the melt for 260 s. The mixture of molten aluminium and AlN$_p$ were further stirred for 1200 s before pouring into a preheated permanent mould at 300 °C through the bottom pouring arrangement. Figure 3.2 shows the electric heater used to preheat the permanent mould. Permanent mould was preheated in order to decrease the porosity level in the cast composite (Samuel & Samuel 1995).

Argon was supplied until the entire slurry was poured into the permanent mould. The composite was allowed to solidify in atmospheric air and then taken out from the mould. Similarly AA6061/AlN$_p$ composites containing different weight percentages of AlN$_p$ were prepared. AA6061 alloy was also cast by stir casting furnace. Mg was not added into the melt to fabricate the cast AA6061 alloy. Quantity of AA6061 alloy and AlN$_p$ reinforcement required to fabricate those composites are presented in Table 3.2. For the ease of reference
the process parameters employed to fabricate the composites are presented in Table 3.3. The fabricated AA6061 alloy and AA6061/AlN$_p$ composite castings containing different weight percentages of (ranging from 5, 10, 15 and 20) AlN$_p$ are presented in Figure 3.3.

![Figure 3.2 Preheater of Permanent Mould](image)

**Figure 3.2 Preheater of Permanent Mould**

<table>
<thead>
<tr>
<th>Material</th>
<th>AA6061 Alloy (g)</th>
<th>AlN$_p$ (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061 Alloy</td>
<td>1300</td>
<td>-</td>
</tr>
<tr>
<td>AA6061/5 wt.% AlN$_p$</td>
<td>1235</td>
<td>65</td>
</tr>
<tr>
<td>AA6061/10 wt.% AlN$_p$</td>
<td>1170</td>
<td>130</td>
</tr>
<tr>
<td>AA6061/15 wt.% AlN$_p$</td>
<td>1105</td>
<td>195</td>
</tr>
<tr>
<td>AA6061/20 wt.% AlN$_p$</td>
<td>1040</td>
<td>260</td>
</tr>
</tbody>
</table>

Table 3.2 Calculated Quantity of AA6061 matrix and AlN$_p$ Reinforcement
Table 3.3  Process Parameters Employed to Fabricate AA6061/AlN<sub>p</sub> Composites in Modified Stir Casting Process

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Process Parameters</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Flow rate of Argon gas</td>
<td>lpm</td>
<td>2</td>
</tr>
<tr>
<td>2</td>
<td>Temperature of melt</td>
<td>℃</td>
<td>1000</td>
</tr>
<tr>
<td>3</td>
<td>Preheat temperature of AlN&lt;sub&gt;p&lt;/sub&gt;</td>
<td>℃</td>
<td>750</td>
</tr>
<tr>
<td>4</td>
<td>Spindle speed of the stirrer</td>
<td>rpm</td>
<td>450</td>
</tr>
<tr>
<td>5</td>
<td>Incorporation time</td>
<td>s</td>
<td>260</td>
</tr>
<tr>
<td>6</td>
<td>Stirring time</td>
<td>s</td>
<td>1200</td>
</tr>
<tr>
<td>7</td>
<td>Preheat temperature of the mould</td>
<td>℃</td>
<td>300</td>
</tr>
</tbody>
</table>

Figure 3.3  Fabricated AA6061 Alloy and AA6061/AlN<sub>p</sub> Composite Castings
3.3 METALLURGICAL & MECHANICAL CHARACTERIZATION

3.3.1 X-ray Diffraction

Specimens of size 25 mm x 25 mm x 6 mm were cut from cast AA6061 alloy and each cast composite containing different weight percentage of AlN_p reinforcement. The specimens were cleaned with acetone. The X-ray diffraction (XRD) patterns were recorded using a Panalytical X-ray diffractometer with Cu-Kα X-ray radiation to confirm the presence of AlN_p in the composite. The same specimens were used for subsequent characterization.

3.3.2 Microstructural Analysis

Standard metallographic procedure was adopted to prepare the specimen for microstructural analysis. The specimens were polished using different emery papers of 280 grit to 1200 grit and with diamond pastes of 6 µm, 3 µm and 0.5 µm using a disc polishing machine. The polished specimens were cleaned with acetone and distilled water. The cleaned specimens were etched for 15 s with a colour etchant containing 1 g of sodium hydroxide (NaOH) and 4 g of potassium permanganate (KMnO_4) in 100 ml distilled water. The etched specimens were observed for microstructural analysis using an optical microscope (OLYMPUS-BX51M) and scanning electron microscope (SEM). In addition, Energy-dispersive X-ray Spectroscopy (EDX) analysis was carried out by using an energy dispersive spectroscope attached with the SEM (JEOL-JSM-6390).

3.3.3 Hardness Test

The specimens prepared for microstructural analysis were used for microhardness and macrohardness tests. The microhardness of the etched specimens was measured using a microhardness tester (MITUTOYO-MVK-H1) at a load of 500 g applied for 15 s at twenty different locations on all samples. The macrohardness was measured using a Brinell Hardness Tester (7KB3000) at
a load of 500 kg applied for 15 s at eight different locations on all samples. The average values of microhardness and macrohardness were recorded.

3.3.4 Tensile Test

Three tensile specimens were prepared from cast AA6061 alloy and each cast composite (containing 5, 10, 15 and 20 wt.% of AlN_p reinforcement) as per the American Society of Testing and Materials (ASTM: E8/E8 M-11) standard. The dimensions of the specimen as per the ASTM standard are presented in Figure 3.4. The yield strength (YS), ultimate tensile strength (UTS) and percent elongation (PE) were determined at ambient temperature using a Computerized Universal Testing Machine (HITECH TUE-C-1000) and its average values were recorded. The fabricated tensile specimens as per ASTM standard are shown in Figure 3.5.

![Figure 3.4 Dimensions of the Tensile Specimen as per ASTM E8/E8 M-11 Standard](image)

3.4 RESULTS AND DISCUSSIONS

3.4.1 Metallurgical Characterization

3.4.1.1 X-ray diffraction analysis

The XRD patterns obtained from AA6061 cast alloy and each AA6061/AlN_p (containing 5 to 20 wt.% of AlN_p) composite were superimposed and presented in Figure 3.6. The XRD patterns reveal the presence of AlN_p
Figure 3.5  Fabricated Tensile Specimens

Figure 3.6  XRD Patterns of Fabricated AA6061 Alloy and AA6061/AlN_p Composites
reinforcement in the composite. The peaks of AlN are distinctly clear and they increase with the increase of AlN_p content while the peaks of AA6061 decrease. It is also interesting to note that the peaks of AA6061 in the composite are slightly shifted to lower 2θ when compared to that of AA6061 alloy. The XRD patterns also indicate that there is no formation of intermetallic compounds resulting from the reaction of AlN_p with AA6061 alloy matrix during the casting process. From the XRD patterns, it is obvious that AlN_p are thermodynamically stable at a fabrication temperature of 1000 °C.

3.4.1.2 Microstructural analysis

Optical photomicrographs of fabricated cast AA6061 alloy and AA6061/AlN_p composite containing different weight percentages of AlN_p reinforcement are presented in Figures 3.7-3.12. Microstructure of cast AA6061 alloy presented in Figure 3.7 reveals the formation of α-aluminium dendritic network structure which has formed due to the super-cooling of alloy during solidification. Precipitation of Mg_2Si is also visible in the microstructure.

Figures 3.8-3.11 show the microstructure of AA6061/AlN_p composites containing 5, 10, 15 and 20 wt.% of reinforcement of AlN_p reinforcement respectively. Microstructures of the composites presented in figures clearly reveal the homogeneous distribution of the AlN_p reinforcement in the AA6061 alloy matrix and there is no evidence of porosity and cracks in the castings. This might be related to proper process parameters employed for the production of castings. During solidification of AA6061/AlN_p composite, AlN_p are rejected in the direction of refined α-Al grains. Refinement of α-Al grains can be attributed to the following two causes. (i) AlN_p offer resistance to the growing α-Al phase during the solidification process and (ii) AlN_p themselves act as a nucleus on which α-Al grains solidify (Han et al 2002). Precipitation of Mg_2Si in the matrix is indicated in Figure 3.9. The sources for the formation of Mg_2Si are addition of
Mg in molten Al alloy matrix and Mg and Si are already present in the AA6061 alloy as major constituent.

From the SEM micrographs presented in Figures 3.14-3.17 and optical photomicrograph of composite captured at higher magnification focused on AlN_p presented in Figure 3.12, it is clear that AlN_p are clustered and are distributed homogeneously in the matrix. Hence, appearance of single particles in the optical photomicrographs contain cluster of AlN_p. It is also evident from the photomicrographs that cluster of AlN_p in the matrix are of spherical shape.

Wetting of small individual reinforcement particle is difficult due to increase in the surface energy required for the matrix surface to deform to a small radius as the particle begins to penetrate in it. The smaller particles are also more difficult to be dispersed due to its inherently greater surface area. Hence, these smaller particles have a tendency to clump together resulting in wetting of particles with molten matrix (Hashim et al 2001). An addition of alloying element such as magnesium reacts with aluminium and yields magnesium aluminate (spinel) MgAl_2O_4 at the surface of AlN_p which has enhanced the wettability with aluminium melt. The spinel is formed at the interface during the process according to the following reaction (Nassaj et al 1995).

\[
3\text{Mg} + 4\text{Al}_2\text{O}_3 \rightarrow 3\text{MgAl}_2\text{O}_4 + 2\text{Al} \quad (3.1)
\]

Formation of magnesium aluminate is confirmed from the EDX analysis which is presented in Figure 3.13. Source of oxygen for the above reaction is the oxide layer formed over the molten aluminium alloy matrix. Since an inert atmosphere was maintained at the top of the molten matrix, constant motorized stirring action was carried out during the incorporation of AlN_p in the matrix and molten metal was poured through bottom of the crucible, there was no formation of intermetallic compounds and absence of oxide layer inclusions in cast composites.
Figure 3.7  Optical photomicrograph of As-Cast AA6061 Alloy

Figure 3.8  Optical Photomicrograph of AA6061/5 wt.% AlN_p Composite
Figure 3.9  Optical Photomicrograph of AA6061/10 wt.% AlN$_p$ Composite

Figure 3.10  Optical Photomicrograph of AA6061/15 wt.% AlN$_p$ Composite
Figure 3.11 Optical Photomicrograph of AA6061/20 wt.% AlN_p Composite

Figure 3.12 Optical Photomicrograph of AA6061/5 wt.% AlN_p Composite
Focused on AlN_p Reinforcement
Figure 3.13  EDX Analysis of AA6061/20 wt.% AlN$_p$ Composite

SEM micrographs of fabricated AA6061/AlN$_p$ composites containing 5, 10, 15 and 20 weight percentage of AlN$_p$ reinforcement are presented in Figures 3.14-3.17 respectively. SEM micrographs show the uniform distribution of AlN$_p$ reinforcement in the AA6061 alloy matrix. Figure 3.18 reveals a good bonding between AA6061 alloy matrix and AlN particle. The addition of magnesium enhances the wettability between AlN$_p$ and AA6061 alloy matrix by the formation of spinel at the interface which results in good bonding between AlN$_p$ and AA6061 alloy matrix.

Figure 3.14  SEM Micrograph of AA6061/5 wt.% AlN$_p$ Composite
Figure 3.15  SEM Micrograph of AA6061/10 wt.% AlN_p Composite

Figure 3.16  SEM Micrograph of AA6061/15 wt.% AlN_p Composite
Figure 3.17  SEM Micrograph of AA6061/20 wt.% AlN_p Composite

Figure 3.18  SEM Micrograph of AA6061/5 wt.% AlN_p Composite Showing the Bonding Between the Matrix and AlN_p
3.4.2 Mechanical Characterization

3.4.2.1 Hardness analysis

Figure 3.19 shows the average microhardness and macrohardness of cast AA6061 alloy and AA6061/AlN\(_p\) composites. Error bars superimposed on the graph indicate the standard deviation obtained in averaging of results. Macrohardness of cast AA6061 alloy is 38 BHN. The macrohardness of AA6061/20 wt.% AlN\(_p\) composite is 79 BHN which is 107.89% greater than that of cast AA6061 alloy. Similarly microhardness of cast AA6061 alloy is 44 VHN. Microhardness of AA6061/20 wt.% AlN\(_p\) composite is 91 VHN which is 106.81% greater than that of cast AA6061 alloy. Both macrohardness and microhardness of AA6061/AlN\(_p\) composite linearly increase with the addition of AlN\(_p\) due to the presence of hard AlN\(_p\) in AMCs.

![Figure 3.19 Effect of Weight Percentage of AlN\(_p\) on Microhardness and Macrohardness of Al/AlN\(_p\) Composite](image-url)
3.4.2.2 Analysis of tensile behavior

Figure 3.20 reveals the typical engineering stress strain diagrams of cast AA6061 alloy and AA6061/AlN\textsubscript{p} composites containing various weight percentage of reinforcement obtained by tensile test conducted at room temperature. The average YS measured at 0.2\% offset, UTS and PE of AA6061/AlN\textsubscript{p} composites with various weight percentage of AlN\textsubscript{p} are presented with error bars in Figures 3.21 to 3.23 respectively. Both YS and UTS are increased by the addition of AlN\textsubscript{p}. But PE of the AMC decreases when the amount of AlN\textsubscript{p} increases. YS of AA6061/20 wt.\% AlN\textsubscript{p} composite is 158 MPa which is 95.12\% greater than that of AA6061 alloy. UTS of the AA6061/20 wt.\% AlN\textsubscript{p} composite is 241 MPa which is 46.95\% greater than that of AA6061 alloy.

![Graph showing stress-strain curves for different weight percentages of AlN\textsubscript{p}.]
Figure 3.21  Effect of Weight Percentage of AlN\textsubscript{p} on YS of AA6061/AlN\textsubscript{p} Composite

Figure 3.22  Effect of Weight Percentage of AlN\textsubscript{p} on UTS of AA6061/AlN\textsubscript{p} Composite
An increase in volume fraction of reinforcement of low coefficient of thermal expansion material in the matrix having higher coefficient of thermal expansion changes the microstructural characteristics of the metal matrix with a concomitant contribution to increasing strength. As such, an increase in the volume fraction of the reinforcing ceramic particulate results in an increase in dislocation density around the reinforcement particles during solidification (Dinaharan et al 2011a), reduction in grain size, and an overall reduction in the substructure. The above microstructural changes tend to increase the resistance offered to the motion of both microscopic and macroscopic level dislocations under the influence of a far-field stress (Gupta & Srivatsan 2001). Accordingly, an increase of weight percentage of hard AlNₚ reinforcement in the AMCs increases the magnitude of resistance offered to the motion of both microscopic and macroscopic level dislocations. As a result macrohardness, microhardness and UTS increase with the increase in weight percentage of AlNₚ reinforcement. Reinforcement particles clustered as spherical shape (depicted in Figure 3.12) also contribute to higher UTS by reducing notch effect. Good bonding between the matrix and AlNₚ reinforcement and uniform distribution of AlNₚ reinforcement increase the load bearing capacity of the composite (Tjong & Ma 2000).

The enhancement in UTS is, however, correlated with a reduction in ductility, which reduces to about 4.1% for the AA6061/ 20 wt.% AlNₚ composite, as apparently shown in Figure 3.23. The ductility of aluminium matrix is reduced due to (i) increasing weight percentage of hard AlNₚ reinforcement in the matrix which reduced the flowability of matrix, (ii) more grain boundaries per unit area (due to the refinement of grain size as a result incorporation of reinforcement particles) reduces the slipping of matrix and (iii) increased AlNₚ in the composite reduces the volume fraction of ductile aluminium alloy matrix. All those effects contribute to the decreasing of percent elongation of the composite.
The mechanical properties of AA6061 alloy and AA6061 matrix composites reinforced with different weight percentage of AlN<sub>p</sub> are summarized in Table 3.4. It is evident that the incorporation of AlN<sub>p</sub> into AA6061 alloy matrix significantly improves the strength of the AA6061/AlN<sub>p</sub> composites.

![Graph showing the effect of weight percentage of AlN<sub>p</sub> on percent elongation of AA6061/AlN<sub>p</sub> composite.](image)

**Figure 3.23** Effect of Weight Percentage of AlN<sub>p</sub> on Percent Elongation of AA6061/AlN<sub>p</sub> Composite

Figure 3.24 reveals the fracture surface of AA6061 alloy. It shows a net work of large size dimples which indicate large amount of plastic flow occurred prior to failure. Figures 3.25 and 3.26 reveal the fracture surface of AA6061 matrix composite containing 10 and 20 wt.% of AlN<sub>p</sub> respectively. It shows a net work of dimples whose size is smaller compared to AA6061 alloy. The AlN<sub>p</sub> refined the grain size of matrix alloy and reduced the ductility which resulted in smaller size dimples.
Table 3.4  Mechanical Properties of AA6061 Alloy and AA6061/AlN<sub>p</sub> Composites

<table>
<thead>
<tr>
<th>S. No</th>
<th>Material</th>
<th>0.2% YS (MPa)</th>
<th>UTS (MPa)</th>
<th>Microhardness (VHN)</th>
<th>Macrohardness (BHN)</th>
<th>PE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AA6061 Alloy</td>
<td>82</td>
<td>164</td>
<td>1.53</td>
<td>44</td>
<td>2.15</td>
</tr>
<tr>
<td>2</td>
<td>AA6061/ 5 wt.% AlN&lt;sub&gt;p&lt;/sub&gt;</td>
<td>98</td>
<td>181</td>
<td>2.52</td>
<td>55</td>
<td>2.16</td>
</tr>
<tr>
<td>3</td>
<td>AA6061/10 wt.% AlN&lt;sub&gt;p&lt;/sub&gt;</td>
<td>136</td>
<td>202</td>
<td>1.53</td>
<td>68</td>
<td>2.27</td>
</tr>
<tr>
<td>4</td>
<td>AA6061/15 wt.% AlN&lt;sub&gt;p&lt;/sub&gt;</td>
<td>146</td>
<td>225</td>
<td>2.08</td>
<td>76</td>
<td>2.22</td>
</tr>
<tr>
<td>5</td>
<td>AA6061/20 wt.% AlN&lt;sub&gt;p&lt;/sub&gt;</td>
<td>158</td>
<td>241</td>
<td>3.46</td>
<td>91</td>
<td>2.20</td>
</tr>
</tbody>
</table>

<sup>a</sup>average value,  <sup>b</sup>standard deviation
Figure 3.24  SEM Micrograph of Fracture Surface of Tensile Specimen of AA6061 Alloy

Figure 3.25  SEM micrograph of Fracture Surface of Tensile Specimen of AA6061/10 wt.% AlN$_p$ composite
3.5 SUMMARY

- The cast AA6061 alloy and AA6061/AlN_p composite containing different weight percentage of reinforcement viz., 5, 10, 15 and 20 were successfully produced by stir casting method.
- The homogeneous dispersion of AlN_p in AA6061 alloy matrix and spherical in shape were found.
- The good bonding between the AlN_p reinforcement and the aluminium alloy matrix was obtained.
- XRD patterns ensured the presence of AlN_p in the composites.
- UTS of the AA6061/AlN_p composite increased linearly with increase in percentage of AlN_p reinforcement.
- Higher the percentage of AlN_p reinforcement in the matrix, higher was the macrohardness and microhardness of the composite but lower was the PE of the composite.