CHAPTER 3

FABRICATION AND CHARACTERIZATION
OF AA 6061-B\textsubscript{4}C MMC

3.1 INTRODUCTION

Metal matrix composites (MMCs) are competent materials to be employed in various applications in industry because of their improved properties. A vast range of MMC materials has been planed and studied to combine the desirable attributes of metals and ceramics. But the suitable fabrication procedure with excellent mechanical properties of these materials and the relatively low production cost make them very attractive for a variety of applications in automotive and aerospace industries.

This chapter describes the fabrication process and the characterization of AA6061 based MMC reinforced with B\textsubscript{4}C as a particulate material. Also, the microstructures and mechanical properties such as tensile strength and hardness of produced MMCs are detailed in this chapter.

3.2 FABRICATION PROCESS OF Al-B\textsubscript{4}C MMC

The proposed Al- B\textsubscript{4}C MMC was produced by modified stir casting process. AA 6061 alloy was used as a matrix; B\textsubscript{4}C with a size of 10 \(\mu\) was used as a reinforcement and K\textsubscript{2}TiF\textsubscript{6} (Potassium fluotitanate) flux was used for enhancing wettability of B\textsubscript{4}C with Al melt during stir casting process. The
chemical composition and the mechanical properties of AA6061 is given Tables 3.1 and 3.2 respectively.

**Table 3.1  Chemical Composition of Aluminum Alloy (6061-T6)**

<table>
<thead>
<tr>
<th>Elements</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>% by weight</td>
<td>0.95</td>
<td>0.54</td>
<td>0.22</td>
<td>0.17</td>
<td>0.13</td>
<td>0.09</td>
<td>0.08</td>
<td>0.01</td>
<td>Balance</td>
</tr>
</tbody>
</table>

**Table 3.2  Mechanical Properties of Matrix and Reinforcement Materials**

<table>
<thead>
<tr>
<th>Property</th>
<th>Al</th>
<th>B₄C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm³)</td>
<td>2.70</td>
<td>2.52</td>
</tr>
<tr>
<td>Melting point (°C)</td>
<td>652</td>
<td>2445</td>
</tr>
<tr>
<td>Coefficient of thermal expansion(10⁻⁶/°C)</td>
<td>23.5</td>
<td>5.0</td>
</tr>
<tr>
<td>Hardness (VHN)</td>
<td>107</td>
<td>2900</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>72</td>
<td>450</td>
</tr>
</tbody>
</table>

A batch of 1250 g of aluminum alloy was melted in stainless steel crucible at 920°C using an electrical resistance furnace shown in Figure 3.1. The melt was agitated with the help of a mechanical stirrer to form a fine vortex (Hashim et al 1999, Sevik et al 2006). The mixtures of preheated B₄C particles with an equivalent amount of K₂TiF₆ flux were added at a constant feed rate into the vortex. The process parameters employed are given in Table 3.3. Argon gas was supplied into the melt during the operation to provide an inert atmosphere. Modified stir casting equipment having the provision for transferring the melt into a permanent mold with a bottom pouring arrangement attached to the furnace is shown in Figure 3.2. After stirring the molten mixture, it was poured down into the preheated permanent mold shown in Figure 3.3. The AMCs having different weight percentages (4, 6, 8,
10 and 12) of B₄C were fabricated by the same procedure. The maximum amount of B₄C was limited up to 12% due to the formation of more slag on the melt surface which caused the inclusions in the trail castings. The manufactured MMCs are shown in Figures 3.4 and 3.5.

Figure 3.1  Electrical Resistance Furnace
Figure 3.2  Bottom Pouring Attachment Provided in Electrical Resistance Furnace

Figure 3.3  Preheated Permanent Mold
Table 3.3  Process Parameters of Modified Stir Casting Process

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Units</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stir speed</td>
<td>RPM</td>
<td>300</td>
</tr>
<tr>
<td>Stirring Time</td>
<td>Minutes</td>
<td>5</td>
</tr>
<tr>
<td>Temperature of melt</td>
<td>°C</td>
<td>920</td>
</tr>
<tr>
<td>Preheated temperature of $B_4C$</td>
<td>°C</td>
<td>400</td>
</tr>
<tr>
<td>Preheated temperature of mold</td>
<td>°C</td>
<td>250</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>g/sec</td>
<td>0.8-1.2</td>
</tr>
</tbody>
</table>

Figure 3.4  Typical Stir Cast AA6061-$B_4C$ Composite
3.3 EVALUATION OF METALLURGICAL AND MECHANICAL PROPERTIES OF MMCs

3.3.1 Evaluation of Microstructure

Metallurgical characterization of fabricated MMCs was carried out using an optical microscope, scanning electron microscope with EDAX and X-ray Diffraction analysis. The specimens prepared from the cast MMCs were polished and etched as per the standard metallographic procedure. The colour etchant used to reveal the microstructure in this study consisted of 2-3 g sodium molybdate, 5 ml HCl (35%) and 1-2 g ammonium bifluoride in 100 ml distilled water. The specimens were immersed in the etchant at room temperature until the surface was colored and dried in hot air later.
3.3.1.1 Optical Microscopy

The microstructures of the color etched specimens were observed using a metallurgical microscope (Olympus Microscope - BX51M). Photomicrographs were taken on the top surface of all cast MMC specimens.

3.3.1.2 X-Ray Diffraction Analysis

X-ray diffraction analysis was carried out to find the presence of different elements in the MMC. It was carried out by using a Panalytical X-ray diffractometer and diffraction patterns were obtained for MMCs containing different amounts of B₄C.

3.3.1.3 Scanning Electron Microscopy with EDS Analysis

The microstructures of color etched specimens as well as the fractured surfaces of tensile specimens were observed using a scanning electron microscope attached with an energy dispersive spectroscopy (JEOL JSM-6390). The EDAX were performed to determine the chemical composition of the matrix and reinforcement formed.

3.3.2 Evaluation of Mechanical Properties

3.3.2.1 Hardness Test

To evaluate the hardness of the composites, the Vicker hardness and Brinell hardness tests were conducted. The microhardness of polished samples was measured at different locations using the Vicker hardness tester (Mitutoyo MVK-H1) at a load of 300 g for 10 sec. The macrohardness was measured using Brinell Hardness Tester (model 7KB3000) at a load of 500 kg for a period of 15 sec.
3.3.2.2 Tensile Test

The tensile tests were used to assess the mechanical behaviors of the cast composites and the matrix alloy. The tensile specimens were prepared from the cast MMCs as per ASTM E08 standard. The dimensions of the specimens are shown in Figure 3.6. The ultimate tensile strength (UTS) was estimated using a computerized universal testing machine (TUE-C-1000). Three specimens prepared from each MMCs and base alloy as shown in Figure 3.7 were tested and the average value of tensile strength was estimated.

![The Dimensions of Tensile Specimen](image)

**Figure 3.6** The Dimensions of Tensile Specimen

![Tensile Specimens of AA6061–B₄C Composites](image)

**Figure 3.7** Tensile Specimens of AA6061–B₄C Composites: (a) Before Fracture and (b) After Fracture
3.4 RESULTS AND DISCUSSION

Aluminum reinforced with B$_4$C particulate composites are successfully fabricated by a modified stir casting process. The addition of K$_2$TiF$_6$ flux has improved the wettability of B$_4$C particles with molten aluminum and facilitated the incorporation of B$_4$C particle in the Al matrix. The flux reacts with the melted surface of B$_4$C particle and produces Ti layers around the surface of B$_4$C particles. This reaction is exothermic in nature and heat is evolved in the vicinity of B$_4$C particle-melt interface which enhances the incorporation of particles into the melt (Kennedy and Brampton 2001).

3.4.1 Evaluation of Microstructure

3.4.1.1 Optical Micrograph

The optical photomicrographs of the cast AA6061 alloy and the fabricated MMCs are shown in Figures 3.8 to 3.13. Figure 3.8 shows the microstructure of cast AA6061 alloy exhibiting a typical dendritic structure. In most cast aluminium alloys, solidification begins with the development of a dendritic network of primary aluminum. The secondary dendritic arm spacing is essentially determined by alloy composition, cooling rate, local solidification time and temperature gradient. Microstructure indicating the intermetallic phase Mg$_2$Si is formed during casting around the dendrites. The mechanical properties of the cast alloys are determined primarily by the secondary dendritic arm spacing and the morphology of interdendritic phases in their microstructure (Mazlee 2007).
**Figure 3.8  Photomicrograph of Cast AA6061 Alloy**

It is observed from Figures 3.9 to 3.13 that B₄C particles are dispersed uniformly in the aluminum matrix at all weight percentages. It may be due to the almost equal value of density of matrix and reinforcement material causing the particle neither float nor decent in the mixture. The sizes of the B₄C particles appear to be homogeneous throughout the aluminum matrix. This can be attributed to the effective stirring action and the use of appropriate process parameters. Homogeneous distribution of particles is a prerequisite to enhance the mechanical properties of the matrix alloy. During the optical microscopic examination, when an attempt was made to focus on the B₄C particulates, the rest of the matrix got defocused and vice-versa. Such an effect is demonstrated through some typical photo micrographs presented in Figures 3.10 and 3.12. This could be due to the harder particulates were protruding out from the top surface of the specimen when compared with the soft Al matrix after polishing them.
Figure 3.9 Photomicrograph of AA6061/4 wt. % $B_4C$ MMC

Figure 3.10 Photomicrograph of AA6061/6 wt.% $B_4C$ MMC
Figure 3.11  Photomicrograph of AA6061/8 wt. % $B_4C$ MMC

Figure 3.12  Photomicrograph of AA6061/10 wt. % $B_4C$ MMC
3.4.1.2 X-Ray Diffraction Analysis of MMCs

The XRD analysis presented in Figure 3.14 confirms the presence of \( \text{B}_4\text{C} \) reinforcement within the matrix. The peak of \( \text{B}_4\text{C} \) is increasing with increased \( \text{B}_4\text{C} \) content while the peak of Al is decreasing. It is also evident from the XRD pattern that the \( \text{B}_4\text{C} \) particles do not react with Al matrix and produce any other compounds. The \( \text{B}_4\text{C} \) particles are thermodynamically stable at the synthesizing temperature used in this work. This may be due to the formation of Ti layer around \( \text{B}_4\text{C} \) particles which tend to act as a reaction barrier and prevents the interfacial reactions between the \( \text{B}_4\text{C} \) and aluminum matrix.
3.4.1.3 Scanning Electron Microscopy

Figures 3.15 to 3.19 show scanning electron micrograph of fabricated MMCs. The SEM images reveal that the homogeneous dispersion of B₄C particles in the matrix. However, in some region in Figure 3.18 the weak Ti layer is observed. The presence of weak Ti is also evident from EDAX analysis and is depicted in Figure 3.20. During the solidification of composites the aluminum dendrites solidify first and the B₄C particles are rejected by the solid-liquid interface. Hence, the particles are segregated at the inter dendrite region (Kok 2005). Ti reaction layer was formed when adding K₂TiF₆ flux into the melt where K and F contributed for removing the oxide film from the Al surface (Toptan et al 2010). The weak reaction layer in the
region was not so clear in the SEM image due to the removal of interfacial reaction layer during polishing.

Figure 3.15  SEM Photomicrograph of AA6061-4 wt. % B₄C MMC

Figure 3.16  SEM Photomicrograph of AA6061-6 wt. % B₄C MMC
Figure 3.17  SEM Photomicrograph of AA6061-8 wt. % $\text{B}_4\text{C}$ MMC

Figure 3.18  SEM Photomicrograph of AA6061-10 wt. % $\text{B}_4\text{C}$ MMC
Figure 3.19  SEM Photomicrograph of AA6061-12 wt. % B₄C MMC

Figure 3.20  EDAX analysis of AA6061-10% B₄C Composite
3.4.2. Evaluation of Mechanical Properties

The estimated mechanical properties of AMMCs are presented in Table 3.4.

Table 3.4 Mechanical Properties of Produced AA6061-B₄C Composites

<table>
<thead>
<tr>
<th>S. No</th>
<th>% of B₄C in Al matrix</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Microhardness (VHN)</th>
<th>Macrohardness (BHN)</th>
<th>% of Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>160</td>
<td>45.02</td>
<td>30.00</td>
<td>4.5</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>185</td>
<td>51.37</td>
<td>34.43</td>
<td>4.0</td>
</tr>
<tr>
<td>3</td>
<td>6</td>
<td>191</td>
<td>58.52</td>
<td>38.91</td>
<td>3.5</td>
</tr>
<tr>
<td>4</td>
<td>8</td>
<td>199</td>
<td>69.61</td>
<td>43.64</td>
<td>3.0</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>206</td>
<td>75.58</td>
<td>50.32</td>
<td>3.0</td>
</tr>
<tr>
<td>6</td>
<td>12</td>
<td>215</td>
<td>80.82</td>
<td>58.61</td>
<td>2.5</td>
</tr>
</tbody>
</table>

3.4.2.1 Hardness Test

The mechanical properties of matrix alloy AA6061 is improved upon B₄C incorporation. Figure 3.21 shows the effect of weight percentage of B₄C reinforcement particulates addition on the hardness of fabricated MMCs. It is observed that the microhardness and macrohardness of MMCs linearly increase when the amount of reinforcement particulates increases. Addition of reinforcement particles in the matrix increases the surface area of the reinforcement. The presence of such hard surface area of particles offers more resistance to plastic deformation which leads to increase in the hardness of composites. It is reported (Ramesh et al 2009) that the presence of hard ceramic phase in the soft ductile matrix reduces the ductility of composites due to reduction of ductile metal content which significantly increases the hardness value.
Figure 3.21  Effect of B₄C Particulates on the Hardness of AA6061-B₄C MMCs

3.4.2.2  Tensile Test

Figure 3.22 shows the effect of the weight percentage of B₄C particulates on UTS and % E of composites. It can be inferred that B₄C particles are very effective in improving the tensile strength of composites from 185 MPa to 215 MPa. It may be due to the strengthening mechanism of the reinforcement (Feng et al 2009). The addition of B₄C particles in the matrix induces much strength to matrix alloy by offering more resistance to tensile stresses. It is well known that the thermal expansion coefficient of B₄C particle is 5x10⁻⁶/°C and for aluminum alloy is 23x10⁻⁶/°C. The thermal mismatch between the matrix and the reinforcement causes higher dislocation density in the matrix and load bearing capacity of the hard particles which subsequently increase the composite strength (Toptan et al 2010). The percentage elongation of the composite is decreased with the addition of B₄C content in the matrix.
Figure 3.22  Effect of B₄C Particulates on UTS and % of Elongation of AA6061-B₄C MMCs

Figures 3.23 and 3.24 show the fracture surfaces of 4% and 10% B₄C composite specimens respectively. The fracture surface of 4% B₄C MMC reveals nonuniform dimples indicating a ductile mode of fracture whereas, fractured surface of 10% of B₄C composite material reveals a mixed mode of fracture behavior. The fracture surfaces of the materials show the intergranular fracture which is fractured through grain boundaries. The shear ductile fracture does not take place, but it is not a totally brittle fracture.

3.5  SUMMARY

Fabrication of Metal Matrix Composites with B₄C particles by casting process is usually difficult because of the poor wetting between Al and B₄C and agglomeration phenomena which result in weak mechanical properties. In the present work, AA6061 aluminum alloy matrix composites reinforced with B₄C particles were produced by modified stir casting route.
Figure 3.23  Fracture surface of AA6061-4wt. % B₄C MMC

Figure 3.24  Fracture surface of AA6061-10wt. % B₄C MMC
Titanium containing flux (K₂TiF₆) was used to increase the wetting between Al and B₄C and facilitate the incorporation of B₄C particles into molten aluminum. The MMCs containing different weight % (4, 6, 8, 10 and 12) of B₄C was manufactured. The microstructure and mechanical properties of the fabricated MMCs were analyzed. The good dispersion of B₄C particles in the matrix was observed from the OM and SEM images. However, the little amount of segregation of the particles was revealed by the SEM image. The XRD pattern and EDAX analysis indicated the presence of B₄C particles and the formation of the Ti layer during the process respectively. The mechanical properties like hardness and tensile strength were improved with the increase in weight percentage of B₄C particulates in the Aluminum matrix.