Effect of pouring temperature and holding time on hardness at various locations of Al/TiB$_2$ MMC cast ingot

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Abstract: The effect of holding time and pouring temperature on the microstructure ie., size of formation of TiB$_2$, and its distribution pattern and hardness of Al/TiB$_2$ in-situ composite was studied. The pouring temperatures assigned for the casting were 750 °C, 780 °C and 810 °C and the holding times were 10 minutes, 20 minutes and 30 minutes. Microstructures have been taken using the Optical and Scanning Electron microscope (SEM) and hardness values were measured using Brinell hardness tester for the Al/TiB$_2$ MMC. ProCAST casting simulation software was utilized to find the cooling curves between time and temperature for all the different pouring temperatures of the ingot. The cooling rates alter the distribution pattern achieved. The hardness results indicate significant increase of improvement in hardness of Al/TiB$_2$ MMC in comparison with as-cast composite.

Keywords: ProCAST; Metal Matrix Composite; In-situ; TiB$_2$ particles; Al/TiB$_2$; MMC

I. INTRODUCTION

In these modern years, the need for lightweight and high-performance materials to satisfy the essential demand of aerospace and automobile sector resulted in the growth of much inventive integration of materials. Research has been concentrated on the design and compound of metal matrix composites for high-performance applications because of their outstanding strength-to-weight ratio and their physical properties [1-3]. In these composites, one of the most used matrixes is aluminum; there are many reasons for selecting aluminium matrix; it possesses a wide variety of favorable properties, such as good corrosion resistance, low density and high strength [4-5]. Amongst the many ceramic reinforcements considered for making an aluminium matrix composite, cast aluminium and TiB$_2$ were found to have excellent compatibility.

Thus, AMCs are used in sports tools, packaging for electronics, armor and automotive industries [6-8]. TiB$_2$ is a refractory compound that exhibits outstanding features such as high melting point (2790 °C), high hardness (25 GPa) and high Young's modulus (565 GPa). Its resistance to plastic deformation even at high temperatures, has shown it as a good potential reinforcing candidate in an aluminum matrix. There is lack of literature on Al/TiB$_2$ composites; reports on its synthesis processing are scarce. The unavailability of practical processing routes to overcome the poor wettability in the case of ex-situ composite and high melting temperature TiB$_2$ could have accounted for Al/TiB$_2$ MMC being preferred [9-11].

Hardness is one of the most important factors while selecting ceramics for several engineering applications, for applications like abrasive wear, etc [12-13]. Hardness increases with the volume fraction of particles and inversely with the size of particles in the MMC [14-15].

The principal objective of this current investigation is to determine how the processing parameters, such as the holding time and the pouring temperature affect the size and distribution pattern of Al/TiB$_2$ MMCs and thus hardness at various locations of an ingot.

2. EXPERIMENTAL WORK

The base alloy composition used in this work is nearly as that A356 and the volume fraction is 6% TiB$_2$. The volume fraction of the TiB$_2$ particles was calculated by means of mass fraction calculation. Chemical composition of base alloy (wt %) in the raw material:

<table>
<thead>
<tr>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.5-7.5</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
<td>0.25-0.45</td>
<td>0.1</td>
<td>0.1</td>
<td>Balance</td>
</tr>
</tbody>
</table>
The requirement for halide salts viz., $\text{K}_2\text{TiF}_6$ and $\text{KBF}_4$ were found based on stochiometric calculations and preheated at 250°C. A pre-weighted mixture of $\text{K}_2\text{TiF}_6$ and $\text{KBF}_4$ were added into the molten metal to produce TiB$_2$, and then the melt was hand stirred for 25 minutes by graphite rod before the slag was removed. Nine ingots were cast with the combinations of three different pouring temperatures of 750°C, 780°C and 810°C and three different holding times of 10 minutes, 20 minutes and 30 minutes. The geometry of the test specimen for microstructure study is 10mmx10mmx10mm. Samples were prepared for metallographic analysis by ordinary procedures: grinding, polishing and etching with a solution of 50 ml of distilled water, 4ml of HF, 2 ml of $\text{HNO}_3$ and 2 ml of HCl (using keller’s etchant). Microstructures were analyzed by using optical microscopy and scanning electron microscopy (SEM) and X-ray diffraction (XRD) study also carried out. Brinell hardness was measured with applied load of 100 kgf for 20 seconds.

2.1 Finite Element Analysis

The permanent mold with 60mmx70mmx180mm dimensions was modeled by using Unigraphics (NX-4) modeling software. Then the permanent model was exported to the proCAST casting simulation software as shown in figure (1) for thermal analysis. Further, the permanent mold model was fine meshed using mesh tool as shown in figure (2). After meshing, the necessary boundary conditions for thermal analysis were assigned and 24 locations of the model as shown in figure (3) were identified by node and the cooling curves were arrived at. Finally the meshed model was introduced for thermal analysis to find out the cooling curves viz., temperature vs time at three different pouring temperatures of 750°C, 780°C and 810°C for three different holding times of 10 minutes, 20 minutes and 30 minutes.

Figure (1): Permanent mold solid model was created by using Unigraphics and imported from proCAST simulation software.

Figure (2): Mesh diagram for the permanent mold.
2.2 Brinell Hardness Tester
The hardness values of Al/TiB₂ MMC were experimentally determined for the three different pouring temperatures with three different holding times. The test conditions are, load = 100 kgf, ball diameter = 1/16 inch and scale = B. Three replications were taken for these six locations in each cast ingot.

3. RESULT AND DISCUSSION

3.1 Microstructural analysis
The microstructure of cast alloy as matrix with 6 wt% TiB₂ reinforcement content was studied. In general, the TiB₂ is not uniformly distributed, but tends to be collected at inter-dendritic boundaries. Fig (4a) to (4c) shows the microstructure of A356 alloy 6 wt% TiB₂ composite in the as-cast condition. The XRD image confirms the presence of TiB₂ particles, in figure 4d.

Figure (4a): SEM image for pouring temperature 780 °C and holding time 30 minutes at location 23
Figure (4b): SEM image for pouring temperature 780 °C and holding time 30 minutes at location 21

Figure (4c): SEM image for pouring temperature 780 °C and holding time 30 minutes at location 19

Figure (4d): XRD image for pouring temperature 780 °C and holding time 30 minutes at location 21.
It can be seen that the TiB₂ reinforcements is not uniformly distributed and most of TiB₂ accumulated at the grain boundaries due to the low stirring speed.

3.2 Influences of cooling curves:

For all 24 different locations temperature vs time curves were obtained using ESI proCAST simulation casting software. Out of the 24 locations six locations (fig. 3) were short listed and at these six locations the local conditions of cooling rate, turbulence and fluidity were found to drastically vary. At these selected six different locations temperature vs time curves are found to be as shown in figures below figure 5(a) to figure 5(f).

Figure (5a): Temperature – time curve for 780 °C pouring temperature at location – 1.

Figure (5b): Temperature – time curve for 780 °C pouring temperature at location – 4.

Figure (5c): Temperature – time curve for 780 °C pouring temperature at location – 7.
Figure (5d): Temperature – time curve for 780 °C pouring temperature at location – 19

Figure (5e): Temperature – time curve for 780 °C pouring temperature at location – 21.

Figure (5f): Temperature – time curve for 780 °C pouring temperature at location – 23
At location – 4, the fall through height is lesser and at the same circulation of molten metal will not take place much as the cooling rate is low and these results in the size of TiB$_2$ particles found in this location to be more.

Whereas, at location – 23, the cooling rate is so high, and hence more circulation molten metal occurs which results in the TiB$_2$ particles fragmented near the surface of the ingot and more number of smaller TiB$_2$ particles could be observed.

Thus in Figure (10) the size of TiB$_2$ particles in location – 23 can be found to be lower than the size of TiB$_2$ particles in location – 4.

### 3.3 Hardness:

**(a). Effect of pouring temperature on hardness:**

Three specimens were cut at location-23 from the ingots cast with pouring temperatures of 750 °C, 780 °C and 810 °C and 30 minutes holding time for the hardness test using Brinell hardness tester. The hardness values are tabulated in the table 1. The hardness values of MMCs were found to be more for higher pouring temperatures and the trends are shown in the graph (figure 6). At higher pouring temperatures the formation of TiB$_2$ will be more resulting in higher hardness.

During processing, TiB$_2$ molecules form and between the TiB$_2$ molecules the fluron gas formed as a byproduct will try to escape out causing low pressure between the TiB$_2$ molecules. The surrounding aluminium liquid will eventually consolidate the TiB$_2$ molecules together and a bigger TiB$_2$ particle will result slowly. Also, with time the surrounding TiB$_2$ molecules formed will attach themselves to the TiB$_2$ particle already formed and growth in size may thus take place. This growth in size will cease when the reaction is complete. Due to the prevailing local turbulence is more as the fall through height is more while pouring from ladle at location – 23, the bigger TiB$_2$ particles get fragmented into smaller ones.

<table>
<thead>
<tr>
<th>Location</th>
<th>Pouring Temperature in °C</th>
<th>Hardness measured 30 minute</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>750</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>780</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>810</td>
<td>61</td>
</tr>
<tr>
<td>23</td>
<td>750</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>780</td>
<td>66</td>
</tr>
<tr>
<td></td>
<td>810</td>
<td>68</td>
</tr>
</tbody>
</table>

Table (1): Hardness values for three pouring temperature with 30 minutes holding time

![Figure 6: Hardness values at location – 23](image-url)
Figure 7(a): SEM image for pouring temperature of 750°C with 30 minutes holding time at location 23

It can be observed (Figure 7a to 7c) that the presence of TiB₂ particles are more as the pouring temperature increases.

Figure 7(b): SEM image for pouring temperature of 780°C with 30 minutes holding time at location 23

Figure 7(c): SEM image for pouring temperature of 810°C with 30 minutes holding time at location 23
(b). Effect of holding time on hardness:

Three specimens were cut at location - 4 from the MMC ingot with the pouring temperature of 780 °C and 10 minutes, 20 minutes and 30 minutes holding times for hardness test using Brinell hardness tester. The hardness values are tabulated in the table 3. The hardness values of MMCs increase with the holding times and it is shown in the graph (figure 8).

<table>
<thead>
<tr>
<th>Location</th>
<th>Pouring Temperature in °C</th>
<th>Hardness measured</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10 minute</td>
<td>20 minute</td>
</tr>
<tr>
<td>4</td>
<td>750</td>
<td>57</td>
</tr>
<tr>
<td></td>
<td>780</td>
<td>58</td>
</tr>
<tr>
<td></td>
<td>810</td>
<td>59</td>
</tr>
<tr>
<td>23</td>
<td>750</td>
<td>62</td>
</tr>
<tr>
<td></td>
<td>780</td>
<td>63</td>
</tr>
<tr>
<td></td>
<td>810</td>
<td>63</td>
</tr>
</tbody>
</table>

Table (3): Hardness values 780 °C pouring temperature with three holding times

![Figure 8: Hardness values at location – 4](image)

![Figure (9a): SEM image for pouring temperature of 780 °C with 10 minutes holding time at location – 4](image)
With increase in holding time it can be observed (Figure 9a to 9c) that the presence of TiB$_2$ particles to be more.

For 750 °C with 30 minutes at location - 4

For 750 °C with 30 minutes at location - 23
CONCLUSION

The number of TiB$_2$ particles increase due to the fragmentation and thus at location-23, the hardness increases. Also as the pouring temperature increases the TiB$_2$ formation will be more resulting in higher hardness.

At location - 4, the number of TiB$_2$ particles and TiB$_2$ particle size and hardness increases when the holding time increases.

REFERENCES


