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# Studies on the different stirring medium for Thermal, structural Mechanical properties of Aluminum-Titanium Di Boride

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## ABSTRACT

A thorough investigation on the influence on the different stirring medium on the composite melt was carried out in an attempt to prepare In-situ Al1050-5wt.% $TiB_2$ composite. In the preparation of in-situ composites stirring medium has its own effect on the final prepared composite. The stirring of the melt slurry was made intermittently at regular intervals. The melt was stirring at regular interval was carried out using different stirring medium. The first part of experiment was carried out without stirring of the melt, in the second part the melt was stirred with motor and in the third part of the experiment the composite melt was stirred with manually and Although different stirring medium was used, the intermittent stirring of the melt was kept at constant interval time of 10 minutes.

No uniform formation of particles were found in the composite processed without stirring, when the composite melt was stirred with motor kept at 100 rpm the results showed that the particles were formed, however the particles were not only of $TiB_2$, there were also presence of other particles such as $Al_3Ti$ and $AlB_2$. However stirring of the melt manually showed excellent

results. The composite processed with manual stirring confirmed the presence of only $TiB_2$ particle in the melt which confirmed that the reaction was complete.

By choosing proper stirring medium composites with only $TiB_2$ particles can be prepared.By exactly choosing the stirring medium condition we were able to successfully produce $TiB_2$ reinforcements in the melt, there by effectively producing Al1050-5%$TiB_2$*in-situ* composite. The microstructure of the composites was examined and the mechanical properties were studied. The studies on the microstructural and mechanical properties of the composite produced using manual stirring showed superior properties.

**Key words:** Vickerhardness, XRD, Aluminium ,$TiB_2$ ,TG-DTA etc.,

## Introduction

Different types of processing techniques has been developed in conjunction with the creation of particle reinforced metal matrix composites to decrease the inconsistencies discovered among one or more processing techniques for the manufacturing of particulate reinforced metal matrix composite's with better qualities. Particulate reinforced metal matrix composites have been classified into as in-situ and ex-situ composites based on processing methodologies. Ex situ composites are composites made using processing techniques such as powder metallurgy, mechanical alloying, spray deposition and numerous additional casting production methods such as squeeze casting, compo casting, and rheo-casting. Composites which were made using all of the foregoing ex-situ processing procedures by combining previously prepared ceramic reinforcements with a matrix alloy that was either molten or powdered. The addition of reinforcements into the matrix alloy is intended to further increase the composites strength. The influences from both the alloy matrix and the particular reinforcements help to improve the total strength of the prepared composites when reinforcements are added to the alloy matrix.

The load transfer and formation of high dislocation density were revealed to be the most frequent strengthening mechanisms observed among the particle reinforced MMCs. The matrix alloy will develop a high dislocation density due to differences which arises due to the coefficient of thermal expansion (CTE) between the alloy matrix and the added reinforcements, which further improves the overall composites' strength. The applied external load is transmitted

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through the ductile alloy matrix to the harder ceramic phase during composite loading, increasing the load bearing ability of the composites. The interfacial connection between the matrix and reinforcements must be strong for the load transfer mechanism to work properly. Ex-situ treated composites have inadequate interfacial bonding strength, according to investigations [1].

Flux assisted synthesis (FAS) technology which is based on solid-liquid based technique using liquid metallurgy is proven to be one of the best and cheap feasible way in manufacturing of alloys of aluminium related particulate reinforced. The main goal of researchers in the field of MMCs was to create a lighter, stronger composite material that, when used in the defense, automobile, and aerospace industries, will consistently reduce the gas intake. The properties of lighter metal-related composite are bring into being to be superior to those of standard iron-based materials.

**Materials and Methods**

Al 1050 aluminium alloy, potassium borofluoride, potassium titanium fluoride, hexachloroethane tablets and Coverall 11 for composite fabrication were utilised in this study. Hi-Tech, Mangalore provided the Al1050 aluminium alloy, which was selected and employed as the material for the matrix. An Optical Emission Spectrometer (OES) was used to examine the alloy's composition (Model LMS 04 of spectromax, Germany). Table 4.1 shows the constituent composition of the alloy obtained. From the Madras Fluorine Pvt. Ltd company commercial grade of potassium borofluoride and potassium titanium fluoride in powder form (70 m) were purchased from., Chennai, for use in this study.

**Methodology for casting of *in-situ* Al 1050-5$TiB_2$ composite**

The studies were carried out in order to determine the best processing conditions for Al-5$TiB_2$ composites. In an electrical resistance furnace heated to the required temperature, the aluminium alloy Al was melted in a graphite crucible. Using a thermocouple which is a K-Type, the temperature of the melt was closely monitored. Protection of the melt was carried out by using Coverall 11 from contamination the melting process.

The melt was degassed by plunging some hexachloroethane by using a graphite plunger as soon as the aluminium melt reached the fixed holding temperature. The halide salt which were packed in the aluminium foil were dried salt and added in batches to the aluminium alloy melt as soon as the melt reaches the set temperature again, and the melt has been intermittently agitated with a zirconia which has been coated on mild steel rod for uniform mixing of the melt.

The whole melt was kept at 850°C temperature for the period of one hour after the exactly weighed stoichiometry of the salts were added to guarantee complete the halide salts and the melt.Thealuminium alloy melt has been stirred intermittently at regular intervals to achieve uniform dispersion of the precipitated particles and to spark the interaction between the salts and melt. $TiB_2$ particles were studied in situ.

The slag has been decanted from the aluminium alloy composite melt as soon as the reaction completes. The meltwas transferred into a 30 mm dia and 170 mm height mild steel mold that had been warmed to around 250°C with the removal of the slag.

**Analysis of the processed composite using Acid dissolution technique**

Acid dissolution is one of best and easiest method for the calculating the weight percent of Titanium di-boride particles in the prepared composite. In this method the weight percentage can be calculated accurately. As the ceramic particles were created inside the melt it is difficult to estimate the amount of particles that were formed during the in-situ exothermic reaction. From the earlier results and from equation 3.1 we know that not only titanium di boride particles are formed inside the melt during the exothermic reaction, there is also a high chance for the formation of $AlB_2$ and $Al_3Ti$ particles. Calculating the weight percentage of the particles formed in the composite along with these intermediate particles will give wrong results. However the composite that we prepared were having some intermediate particles the error in total weight percentage of particles in the composite was carefully eliminated using proper acids.

The prepared composites were sliced at different parts of the casting and ten samples were taken for the analysis. The procured samples were dried and washed, then an ultrasonic treatment was given to the samples and all the samples were washed with acetone. Once the samples were

dried after washing it with acetone, further they were weighed in a sensitive weighing balance which had an resolution of 0.1µm. Once the samples were accurately weighed, they were dissolved individually by using sodium hydroxide solution with was kept. As the dissolution of matrix happened in the solution of sodium hydroxide leaving behind the in situ formed ceramic particles, the particles were taken out separately using a Whatman Ashless filter paper bearing number 42. Then the particles were carefully removed, dried, and weights for the estimation of exact weigh percentage value. The particles were then weighed, and by comparing the weight before and after acid dissolution the overall weight percentage of the composite was estimated.

Metal matrix composites were developed in response to the increasing mandate for weightless and tougher materials. When compared to monolithic alloys, MMCs were created by adding the beneficial properties of certain metals and specific ceramics reinforcements, resulting in higher strength-to-cost ratios and strength-to-weight. MMCs are unusual in that the desired qualities for a specific application may be modified by choosing the right matrix, reinforcements, and manufacturing processes. MMCs are classified as continuous fiber reinforced MMCs or discontinuously reinforced MMCs based depending on the type of reinforcements incorporated into the matrix.

**Results and Discussion**

**XRD analysis on the composite processed with different intermittent stirring**

The X-ray diffraction studies which were performed during the processing of Al1050-5%$TiB_2$ composites processed without stirring with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.4.6. The K2TiF6 and KBF4 salts were added according to the stoichiometric calculations. The XRD pattern obtain from the composites processed from the above conditions shows the peaks of Al, $TiB_2$, $Al_3Ti$ and $AlB_2$ phases which indicate that at the above mentioned conditions there is no complete transformation of the fluoride salts to the $TiB_2$ phase. The presence of intermediate phases which further indicates that the transformation is not complete at 5 minutes of intermittent stirring. Moreover the high intensity of $Al_3Ti$ peaks in the Fig. 4.6 further confirm that there is more formation of $Al_3Ti$ particles that the $AlB_2$ particles. Along with the $Al_3Ti$ and $AlB_2$ peaks the slag $AlF_3$, $K_2TiF_6$ and

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$KAlF_4$ were also found. The high intensity of $Al_3Ti$ peaks in the XRD analysis also indicated that the boron in the melt has been considerably reduced; hence there is formation of more $Al_3Ti$ particles than $AlB_2$ particulates. The presence of other peaks shows that there is meager amount of $TiB_2$ particles have formed during the reaction process.

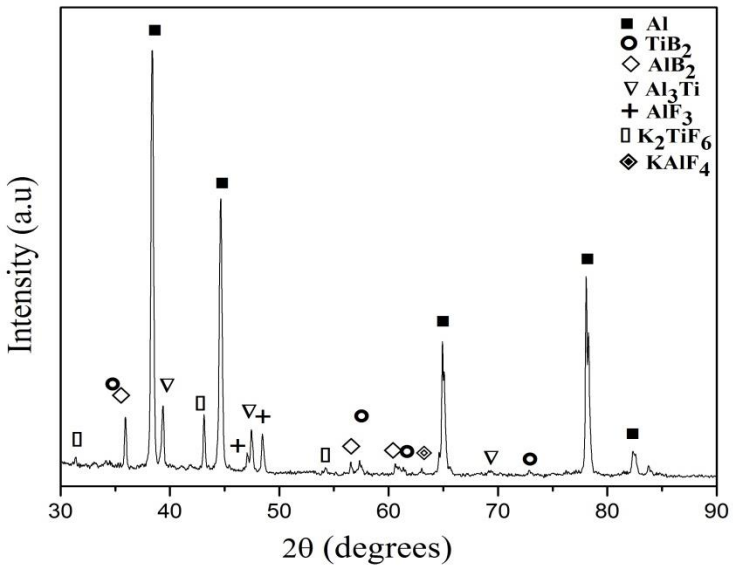


Fig. 1XRD pattern of *in-situ* composite prepared without stirring

The X-ray diffraction studies which were performed during the processing of Al1050-5%$TiB_2$ composites with motor stirring with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.1. In this condition the preparation of $K_2TiF_6$ and $KBF_4$ salts an excess of 10% $KBF_4$ salts is added. Under these processing conditions the X-ray diffraction image shows the only $TiB_2$peaks which indicate that even at 850°C of melt temperature there is complete transformation of the fluoride salts to the $TiB_2$ phase. The presence of intermediate phases further indicates that the transformation is incomplete with the stirring of the melt with motor. However the absence of other intermediate peaks in the Fig. 1 has reduced indicating that the transformation is proceeding in the right direction.

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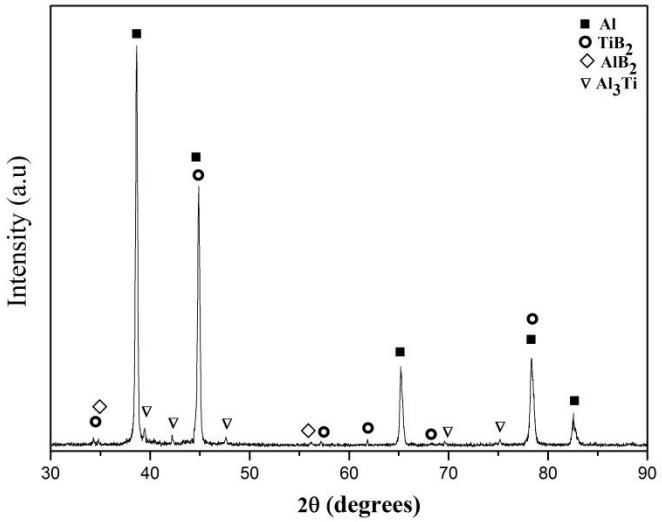


Fig.2 XRD pattern of *in-situ* composite prepared with motor stirring

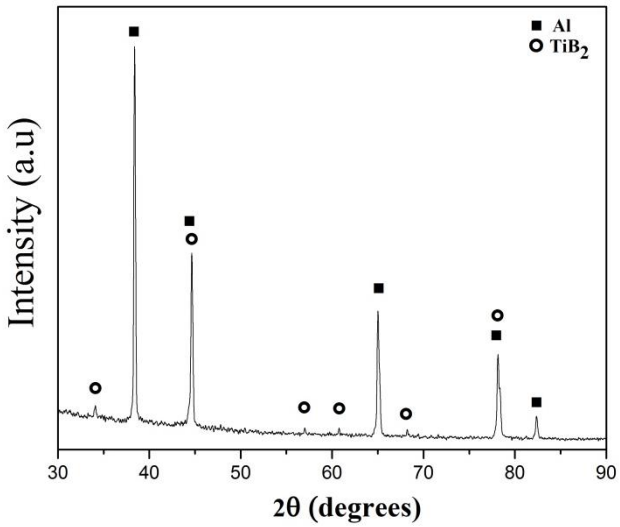


Fig. 3 XRD pattern of *in-situ* composite prepared with manual stirring

The X-ray diffraction studies which were performed during the processing of Al1050-5%$TiB_2$ composites with manual stirring of composite with a processing temperature of 850°C for a constant time of 60 minutes is shown in Fig.2 &3 . In the above conditions during the preparation of composite with manual stirring. Under these processing conditions the X-ray

diffraction image shows peaks of only $TiB_2$ particles. The XRD on the prepared composite confirm that with manual stirring of the melt.

**DTA-TGA analysis**

Fig.4 shows the curve obtained from the TG and DTA analysis. By using this analysis we can find the decomposition behaviour of the added salts to the aluminium melt. The curve was obtained for the temperature range of 30 to 900 ºC.

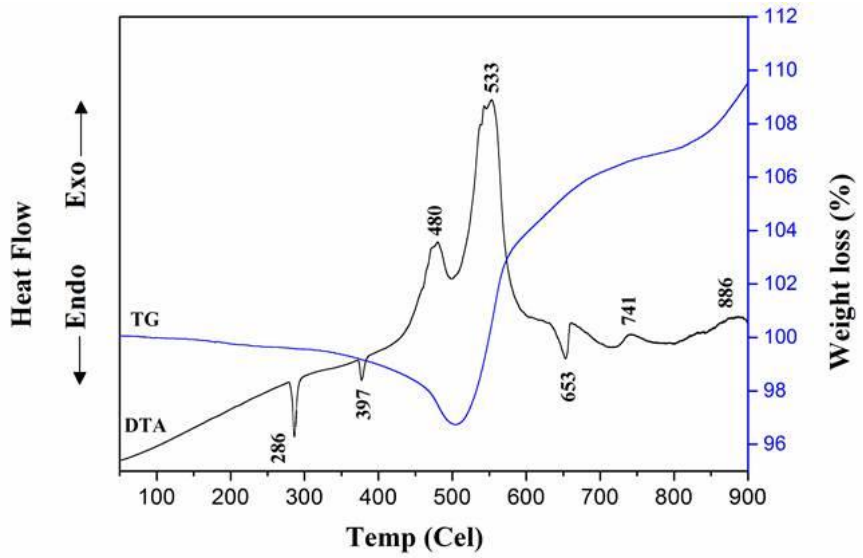


Fig. 4 TG and DTA graph showing $K_2TiF_6$ and $KBF_4$ salts with aluminium melt

As the temperature kept on increasing both endothermic and exothermic reaction was evident. The endothermic and exothermic reaction was due to the decomposition reaction. In the DTA curve the first endothermic peak at 286ºC. This peak occurred du the transformation of potassium tetrafluoro borates polymorphic transformation. The second peak at 397ºC which is endothermic is caused by the moisture removal from the fluoride salts. The sample had showed a weight loss of 1.2 percent till the second endothermic reaction. The loss of moisture in the sample had caused the weight loss. Two exothermic peaks at 480 ºC and 533ºC was seen when the sample was further heated.

The peak which is seen at 480ºC was due to the exothermic reaction of potassium tetra fluoroborate with Al. Similarly the peak at 533ºC which is exothermic was due to reaction of potassium hexafluoro borate with Al.

We can see in the TG curve that a weight loss of 2.5% occurred. When the salts started to react with Al by an exothermic reaction some gases such as $TiF_4$ and $BF_3$ evolved and which would be the cause for the reduction in weight of the salts. However as the exothermic reaction started the increase in weight of the sample was due to the formation of $AlB_2$ and $Al_3Ti$ particles. Furthermore, the increase in weight was due to the formation of slag.

**Mechanical properties**

Stirring medium has an important impact not only in the mixing of the salts it also plays an important role in deciding the mechanical properties of the composite. The hardness value obtained for composite and unreinforced alloy is shown in table 4.3. When the composite was processed at motor stirring conditions of 850°C for a holding time of 60 minutes, the hardness of the composite was found to be 65 VHN. The increase in hardness of the composite compared to unreinforced alloy was found to be 33%. The $Al_3Ti$ and $TiB_2$ particles had contributed to the increase in hardness of the composite compared to unreinforced alloy.

| Intermittent stirring time | Hardness ($HV_5$) | (0.2%) Y.S (MPa) | U.T.S (MPa) | % Elong. |
|---|---|---|---|---|
| AA6082 | 22 | 128 | 178 | 8 |
| Without Stirring | 43 | - | - | - |
| Motor stirring | 65 | 160 | 192 | 10 |
| Manual stiring | 68 | 161 | 219 | 11 |

Table 1 Al 1050-5$TiB_2$ composites Hardness and tensile properties prepared at different intermittent stirring.

## CONCLUSIONS

Overall, the prepared composites at different stirring medium had greater impact on the Thermal and mechanical properties than the unreinforced alloy, according to the findings. Several causes can be attributed to the properties of the composites:

(i) Manual stirring of the melt with salts was found to be desirable condition for production Al1050-5%$TiB_2$ composite.

(ii) Composite processed with motorized stirring of melt with mixture of $K_2TiF_6$ and.

(iii) Motorized stirring of the melt led to the presence of unwanted slag and salts in the melt which in turn increased the viscosity of the melt which led to the difficulty in pouring of the composite melt

The composites' characterization shows that without stirring the slag in the matrix alloy also increased due to the uneven mixing of salts with the melt. As a result, the above-mentioned causes could be to blame for the composites' decreased qualities. Based on the knowledge gathered from this opinion, it was decided stirring the melt manually was desirable condition for obtaining the best results.The overall observations also confirmed that the Stirring medium also had an important role in deciding the properties of the prepared composite.

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