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# A study on microstructure and mechanical properties of Al 6061–TiB<sub>2</sub> in-situ composites

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#### ABSTRACT

In situ reinforced aluminium based metal matrix composites (AMMCs) are emerging as one of the most promising alternatives for eliminating the inherent defects associated with ex situ reinforced AMMCs. Researchers in recent past have attempted various processing techniques for the development of in-situ composites, of which liquid metallurgy is the most widely adopted technique. Development of in-situ composites via liquid metallurgy route using master alloys is a relatively new processing technique. Very little information is available providing the usage value of these reinforcing materials.

The present study is an attempt to explore the processing and characterization of in situ AMMCs using master alloys as reinforcement materials. Al  $6061-TiB_2$  in-situ composites were fabricated by liquid metallurgy route using Al 6061 as the matrix material and Al-10%Ti and Al-3%B as reactive reinforcements. Tests carried out on the fabricated composites include XRD, metallographic studies, EDAX analysis, microhardness, grain size analysis and tensile strength tests. The developed composites exhibited superior structural properties when compared with base alloy.

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#### 1. Introduction

Aluminium based metal matrix composites are the most extensively used class of structural materials owing to their inherent ability to enhance or govern the performance parameters associated with industrial processes. AMMCs are used as potential material for various applications in automobile, chemical, mineral processing, aerospace and transportation industries due to their excellent mechanical properties, tribological behaviour and low thermal expansion co-efficient [1].

Al 6061 alloy is the most widely used 6xxx series aluminium alloy offering a range of good mechanical properties, good surface finish, excellent corrosion resistance and high workability [2]. In recent past researchers have used wide variety of ceramic dispersoids such as SiC [3,4],  $B_4C$  [5],  $Si_3N_4$  [6,7],  $Al_2O_3$  [8] and  $TiO_2$  [9] as reinforcement material in Al6061 based MMCs. However compared to these dispersoids  $TiB_2$  stands out as an outstanding reinforcement, owing to the fact that it possesses high hardness, high strength to density ratio and refractory nature to avoid formation of interfacial reaction products with aluminium. Furthermore, in contrast to most ceramics,  $TiB_2$  exhibits higher electrical and thermal conductivity [10–13].

Aluminium based composites are mainly fabricated by liquid metallurgy route via ex situ technique. This approach involves addition of externally synthesized reinforcement into the molten matrix. But this conventional ex situ technique pose various problems like poor bonding, uneven distribution, poor isometric properties, formation of interfacial reaction products, inherent casting defects due to incomplete adhesion of reinforcement to matrix and thermo dynamic instability of dispersoids with matrix [14–18]. To overcome the above deficiencies, presently researchers are focusing on in situ technique of fabricating aluminium based MMCs. The conventional powder metallurgy and casting methods are suitably redefined to obtain in situ phases by controlling various thermodynamic and kinetic reactions. Past researchers have adopted various fabrication techniques like lanxide<sup>TM</sup> process [19], martin marietta's XD<sup>TM</sup> synthesis [20,21], self propagating high temperature synthesis (SHS) [22], reactive gas injection process [23], plasma deposition [24,25], laser composite surfacing [26], liquid metallurgy using salts [27-29] and liquid metallurgy using master alloys [30,31] to develop in situ MMCs.

Since the formation and growth of reinforcement takes place within the matrix during fabrication, in situ composite have several inherent advantages over ex-situ composites. The major advantages include grain refinement, uniform distribution of particles, excellent bond and improved thermodynamic compatibility between reinforcement and matrix and economy of processing [1,10,13]. Researchers have used numerous starting materials of Ti and B such as titanium and boron powders,  $K_2TiF_6$  and  $KBF_4$  salts,  $TiO_2$  and  $HBO_3$  salts, and  $TiO_2$  and  $B_2O_3$  salts for developing  $TiB_2$  reinforced Al matrix in situ composites. The use of master alloys as reactive reinforcement does exhibit several advantages

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over commonly used powders and salts. The presence of AlB<sub>2</sub> and Al<sub>3</sub>Ti in master alloys readily triggers reactions leading to the formation of TiB<sub>2</sub>. While in salt method reactive reinforcements are not fully utilized due to the formation of slag at high temperatures. Further the overall reaction is complicated and leads to the formation of other unwanted byproducts. Master alloy method facilitates finer reinforcement formation when compared to other methods. The reaction time associated with master alloy method is very less in comparison with salt method in particular. In-situ composites prepared by using Ti and B powders involve reaction temperature of 1000 °C and above, whereas master alloy method is limited to 810 °C. Further, while using Ti and B powders, purity of powders is a big constraint which is not encountered in master alloy method.

However very few researchers [30,31] have used master alloys for the development of TiB<sub>2</sub> reinforced in-situ composites. Meager information is available regarding the processing and characterization of in-situ composites via master alloy method. The present work focuses on the development of TiB<sub>2</sub> reinforced Al 6061 in-situ composite choosing Al 6061 as matrix material and Al–10%Ti and Al–3%B master alloys as reactive reinforcements.

#### 2. Experimental details

Al 6061 alloy and Al–10%Ti and Al–3%B master alloys were procured from Fenfee Metallurgicals, Bangalore in the form of ingots. The chemical compositions of the procured alloys are given in Table 1.

Al 6061 alloy was melted using an electrical resistance furnace in a batch of 3 kg. The molten alloy was degassed using commercially available hexachloroethane tablets. Further, scum powder was used to remove the possible inclusion of traces of unwanted materials during composite preparation. In the present work, a stoichiometric Ti:B ratio of 1:2 was adopted. The percentage weight of Al–10%Ti was varied from 5% to 10% in steps of 2.5% while the weight of Al–3%B was varied from 10% to 20% in steps of 5%. The master alloys were added to the molten Al–6061 alloy in the above said stoichiometric ratios. The composite melt was maintained at a temperature of 810 °C and an in situ reaction time of 30 min was allowed. It was then agitated using a mechanical stirrer for proper mixing of the in situ products and poured into the pre heated metal-lic moulds.

The composites were machined suitably to obtain samples for carrying out SEM, XRD, grain size analysis, hardness and tensile tests. SEM study was carried out using JEOL scanning electron microscope. X-ray diffraction patterns were recorded using Philips X-ray diffractometer.

Microstructure studies on metallographically polished specimens of alloy and the composite were carried out using MEIJI optical microscope. Grain size analysis was performed on polished samples of alloy and the composites using ASTM E 112-96 standard procedure.

Microhardness tests were conducted on thoroughly polished samples of both alloy and developed composites by adopting standard testing procedures. All the samples were applied with a load of 100 g for a period of 10 s using Vickers microhardness tester. The test was carried out at 5 different locations to avoid the possibility of indenter resting on the hard reinforcement particles. The average of these 5 readings was taken as hardness of the samples. Tensile tests were carried out using instron universal testing machine, as per ASTM-A370 standard.

Further, to find out the exact weight percentage of  ${\rm TiB_2}$  formed, known weights of composite samples were dissolved in hydrochloric acid. The matrix was dissolved in acid leaving behind the in situ reinforcements. The extracted particles were thoroughly cleaned, dried and weighed. The difference in the weight of composite mate-

rial used for analysis and the weight of extracted particles was taken as the measure of amount of  $TiB_2$  and  $Al_3Ti$ . The amount of  $TiB_2$  in the extracted powders was estimated by using the ratios of peak intensities of  $TiB_2$  and  $Al_3Ti$  obtained from XRD studies.

#### 3. Results and discussion

#### 3.1. Extracted particle analysis

Table 2 gives the weight percentage of  $TiB_2$  and  $Al_3Ti$  particles obtained from developed composites. It is evident that maximum and minimum  $TiB_2$  formation has been observed in sample-Y and sample-X respectively. However, a notable observation is formation of  $Al_3Ti$  in all the composites with an increasing trend.

Fig. 1 shows the SEM of extracted particles of sample-Y. The micrographs clearly reveal blocky  $Al_3Ti$  along with fine  $TiB_2$  particles. The extracted particles comprising of  $TiB_2$  and  $Al_3Ti$  was confirmed by EDAX analysis shown in Fig. 2. Further the XRD pattern shown in Fig. 3 reconfirms the constituents of extracted particles.

#### 3.2. Microstructure

Fig. 4 shows optical microphotographs of Al 6061 alloy and the developed composites. All developed composites show uniformly distributed TiB<sub>2</sub> particles along with traces of flake like Al<sub>3</sub>Ti. The micrographs clearly reveal maximum TiB<sub>2</sub> formation in sample-Y and minimum in sample-X. Further, the Al<sub>3</sub>Ti intermetallic formation is enhanced with increase in percentage addition of master alloys. The micrograph of sample-Z shows maximum formation of Al<sub>3</sub>Ti, justifying the above fact.

The in situ reaction starts with the initiation of cracks and voids on external surfaces of Al<sub>3</sub>Ti, with diffusion of free boron atoms into these sites. The general sequence of formation of TiB<sub>2</sub> is summarized elsewhere [30]. Further, one cannot omit the possibility of reaction between Al<sub>3</sub>Ti and AlB<sub>2</sub> particles which are predominantly found in master alloys, to form TiB<sub>2</sub> particles. This possibility is justified because of the unstable nature of Al<sub>3</sub>Ti and AlB<sub>2</sub> in a reaction mixture containing Ti/B ratio> 1/2. However, after reaching a certain limit, Al<sub>3</sub>Ti formed is retained in the melt due to the lack of availability of boron atoms. The chemical reactions taking place inside the melt are summarized below:

$$Ti + 3Al \rightarrow Al_3Ti$$
 (1)

$$Al_3Ti + 2B \rightarrow TiB_2 + 3Al$$
 (2)

$$AlB_2 + Al_3Ti \rightarrow TiB_2 + 4Al \tag{3}$$

Fig. 5 shows SEM micrographs of both base alloy and developed in-situ composite. They reveal homogeneously distributed  ${\rm TiB_2}$  particles that are either cubical or hexagonal in shape. As mentioned earlier sample-Y shows better  ${\rm TiB_2}$  formation in comparison with other composites. The average size of the  ${\rm TiB_2}$  particles formed is in the range of 0.5–5  $\mu$ m. In general, the micrographs show no common casting defects such as porosity or shrinkages, thus showcasing the quality of castings.

The X-ray diffraction analysis of the developed composites is shown in Fig. 6. The X-ray profile of the composite clearly shows only aluminium,  $TiB_2$  and  $Al_3Ti$  peaks. The relative fractions of the three phases are calculated based on intensity of diffraction peaks. From the XRD pattern, it is observed that the relative fractions of  $Al:TiB_2:Al_3Ti$  is of the order of 5.97:2.25:1, 5.84:3.37:1 and 4.83:1.91:1 for sample X,Y and Z respectively.

Fig. 7 shows a clean and clear interface between matrix and reinforcement that is attributable to the in situ method of reinforcement formation and dispersion. Since TiB<sub>2</sub> formation takes place

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