



FABRICATION AND CHARACTERIZATION OF AL LM25/TiB₂ IN-SITU COMPOSITES

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ABSTRACT

The Al LM25/TiB₂ (10 wt %) composite is fabricated using in-situ process by stir casting method. LM25 alloy is melted in a graphite crucible at 800°C using electric furnace in argon gas atmosphere and a mixture of K₂TiF₆ and KBF₄ is added to the melt gradually through the hopper attached at the top of the furnace. The melt is stirred with the aid of mechanical stirrer which rotates at 200 rpm for 40 minutes intermittently. The reaction between K₂TiF₆ and KBF₄ in those conditions results in the formation of TiB₂. This melt is poured in the stainless steel mould which is preheated at 200°C and the obtained casting have dimension 100 mm length and 20 mm diameter. The composite specimens are then machined according to the specification requirement of the experiments. All the specimens are polished using emery sheets of grade 1/0 and 2/0 followed by velvet polisher. The specimen used for microstructure analysis is further etched with Keller's reagent. Spectroscopy of LM25 alloy is carried out to observe its elemental composition. X-ray diffraction is used to ensure the formation of TiB₂ during casting process. Inverted metallurgical microscope and Vickers hardness tester are used to study the microstructure and microhardness of the fabricated composite respectively. The X-ray diffraction results revealed the formation of TiB₂ particles in the fabricated composite. Microstructure analysis revealed uniform distribution of TiB₂ in the aluminium matrix and Microhardness test shows an increase in hardness of the composite (91HV) as compared to the unreinforced alloy (82HV) by 10%.

Keywords: in-situ, stir casting, LM25, TiB₂, microstructure, micro hardness.

1. INTRODUCTION

Aluminium Matrix Composites (AMCs) have started becoming a good substitute to aluminium alloys in many applications due to their increased resistance to wear and improved strength per unit mass. Initially, only specific ceramic particles were used because of restrictions in bonding with the matrix. But with advancement in technology, many new ceramics like silicon dioxide, titanium dioxide, titanium carbide and aluminium nitride are available for addition into the aluminium matrix to prepare composites.

There are many methods for fabricating aluminium matrix composites with ceramic as reinforcement. The main methods are powder metallurgy [1, 2] and stir casting [3, 4]. Both these two methods have their own drawbacks and advantages. Because of this, a lot of importance is given for process selection. The simplest of the two methods is Liquid state processing i.e. stir casting because it is simple, easy to adapt and can be applied for fabrication in large quantity. Liquid state processing is divided into two types. They are ex-situ method and in-situ method. If the ceramic particles are added to the melt externally, it is called ex-situ method. If the particles are synthesized within the melt, then it is called in-situ method. [5]. The benefits of in-situ method are finer particle size, uniform distribution and economic way of processing [6, 7]. Wang *et al.* [8] produced Al/Titanium diboride (TiB₂) composite which involves in-situ reaction of Al-4B alloy and electrolytic low titanium aluminium. The results revealed the improved grain refinement of TiB₂ particles. Narayana *et al.* [9] fabricated Al5083/Boron Carbide (B₄C) surface composite using friction stir processing and studied its tribological behaviour. Jebeen *et al.* [10] fabricated AA6061/Silicon

Carbide (SiC) by ex-situ method. The results revealed intra granular distribution of SiC particles and because of the increase in quantity of SiC, the material is more brittle. Gurcan *et al.* [11] performed wear analysis of AA6061 aluminium alloy and its composites which revealed an increase in wear resistance of the composite. Sajjadi *et al.* [12] heat treated micro and nano Alumina (Al₂O₃) particles and injected them into molten Al356 alloy to form composites. The results showed that heat treatment improved wet ability and distribution of nano particles. Chaudhury *et al.* [13] developed a new processing methodology of fabrication of Titanium dioxide (TiO₂) composites known as spray deposition method and compared its mechanical properties with those of Al-2Mg-5TiO₂.

Not much research has been done in LM25 aluminium alloy and TiB₂. Majority of fabrication has been done through ex-situ process. So in this paper, LM25 is reinforced with TiB₂ particles which are formed because of the reactions between Potassium hexafluorotitanate (K₂TiF₆)/Potassium tetra fluoro borate (KBF₄) salts with aluminium and study the effect on microstructure and micro hardness of LM25/TiB₂ composite.

2. MATERIAL SELECTION

In the present work, LM25 alloy is preferred and its elemental composition is given in Table-1. LM25 is one of commonly used age-hardenable cast alloy for various applications. The alloy can be cast in permanent or sand mould and possesses excellent cast ability, good corrosion resistance, good wear resistance, pressure tightness, and better machining and welding characteristics. As Aluminium alloy LM25 has high



strength to weight ratio, it is more preferred for industrial use.

Table-1. Elemental composition of LM25.

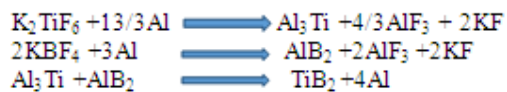
Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
7.33	0.44	0.10	0.09	0.37	0.07	0.06	Remaining

TiB₂ is the most preferred among other ceramics due to their several desirable properties. TiB₂ can withstand high temperature as it has high melting point, high hardness and high modulus characteristics. The embodiment of reinforcements in aluminium matrix improves wear resistance, thermal, and mechanical properties. At temperature above 600°C TiB₂ reacts with oxygen present in the air and forms Boron oxide (B₂O₃) which is a strong and hard material. Hence the formation of TiB₂ is preferred in the aluminium matrix during casting process using in-situ method.

3. EXPERIMENTAL PROCEDURE

3.1 Fabrication of metal matrix composites

For producing Al LM25/TiB₂ (10 wt%) composite, initially 614g of LM25 alloy is cut. 214g of KBF₄ and 200g of K₂TiF₆ are mixed separately so that no lumps of salts are formed. The LM25 alloy is melted in a graphite crucible at 800°C using electric furnace in argon gas atmosphere. After the metal gets melted, the melt is stirred using a mechanical stirrer at a constant speed of 200 rpm, due to this vortex is formed at the center of the melt. The mixed salt is added into the vortex using the hopper provided at the top of the furnace. The temperature of the molten mixture is maintained at 850°C and periodic stirring was done for 40 min with time period of 10 minutes. The reactions that occurs during the casting process are as shown below. K₂TiF₆ reacts with Al and produces Titanium tri-aluminide (Al₃Ti). Aluminium diboride (AlB₂) is obtained during the reaction between KBF₄ and Al. The products Al₃Ti and AlB₂ reacts with each other to form TiB₂.



After 40 min, the crucible is taken out from the furnace and molten metal is poured into a mild steel mould which is preheated at 200°C. The metal is allowed to cool. After cooling, the cast is removed from the mould and the obtained composite specimens have length 100 mm and diameter 20 mm. The setup diagram is shown in Figure-1 and the cast samples are shown in Figure-2.

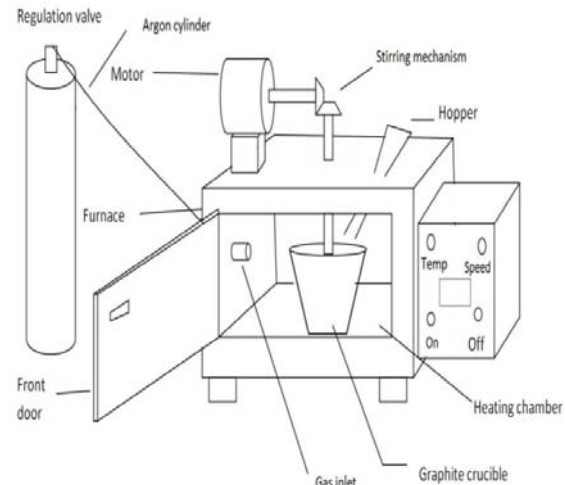


Figure-1. Experimental setup of electric furnace.



Figure-2. Composite specimens.

3.2 Macrostructural evaluation

Inverted metallurgical microscope (ZEISS AXIOVERT 25CA) is used to perform microstructure evaluation using two samples that are machined from the cast specimen to the dimensions of 10mm length and 8 mm diameter. These specimens are grinded using a bench grinder to remove the burr and it is polished on a linisher polisher. Then the specimens are polished using emery paper of grade 1/0 and is further polished by changing the direction using emery paper of grade 2/0. It is followed by Velvet polishing where alumina (liquid) is poured over the velvet covered disc. Then it is washed in water and allowed to dry. The specimen used for microstructure analysis is finally etched with Keller's reagent.

3.3 X-ray diffraction evaluation

X-Ray Diffraction (XRD) of the Al LM25/TiB₂ composite is carried out to ensure the formation of TiB₂



during the stir casting process. For this experiment, a semi-circular specimen of length 3mm and diameter 6 mm is cut from the cast. The specimen is grinded and polished prior to the analysis. PCPDF software and diffraction data from Joint committee on powder diffraction standards are used to get the variable slit intensity vs 2θ graph for Al and TiB_2 . The results of these graphs are interpreted into the XRD graph.

3.4 Micro hardness evaluation

Vickers Hardness tester is used to study the micro hardness of the fabricated composite. The hardness of the samples is determined using diamond indenter with a load of 100N applied for 15s and the value reported is average of five readings taken at different position on same sample.

4. RESULTS AND DISCUSSION

Microstructure, X-Ray Diffraction and Microhardness evaluation results are discussed in detail in the following sub sections.

4.1 Energy dispersive X-ray analysis

Energy Dispersive X-Ray analysis (EDX) is used to check the presence of Titanium and Boron. Figure-3

shows peaks of different elements present in the composite with counts as Y axis and ionization energy as X axis. EDX shows that TiB_2 (10 wt%) is formed in the composite which can be identified from the overall composition given in Table-2.

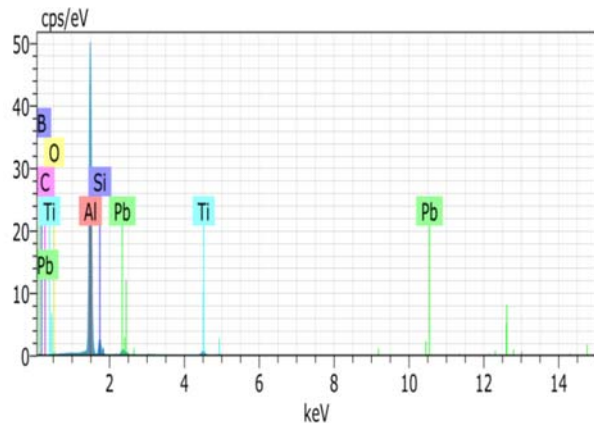


Figure-3. EDAX analysis of composite specimen.

Table-2. EDX Results.

Element	Atomic No.	Series	Unn. C (wt%)	Norm. C wt%	Atom. C at %	Error 1 sigma wt%
Al	13	K	63.70	81.14	67.24	2.98
B	5	K	11.74	14.95	30.93	4.40
Ti	22	K	3.07	3.91	1.82	0.14
		Total	78.50	100.0	100.0	

4.2 Microstructure of LM25/ TiB_2 AMC

Figure-4 shows the microstructure of the composite specimen which indicates the presence of TiB_2 in the aluminium matrix. The aluminium grains solidify surrounding the TiB_2 particles, where TiB_2 acts as a nucleus. Uniform distribution of TiB_2 particles is seen and this could be probably due to intermittent stirring action. While stirring, vortex is formed at the centre of the crucible which cause the particles to spread uniformly in the matrix.

Titanium and boron atoms are obtained from Al_3Ti and AlB_2 respectively which are the intermediate products formed due to reaction between K_2TiF_6 and KBF_4 added to the molten aluminium. Boron atoms are pulled towards Al_3Ti particles. Reaction occurs between titanium and boron atoms in the interval between stirring, resulting in TiB_2 formation. As the boron atoms are small, they get easily diffused through TiB_2 particles. The emptying of Al_3Ti particles occurs due to natural cracking and breaking down of Al_3Ti particles. This increases the formation of TiB_2 in the aluminium matrix.

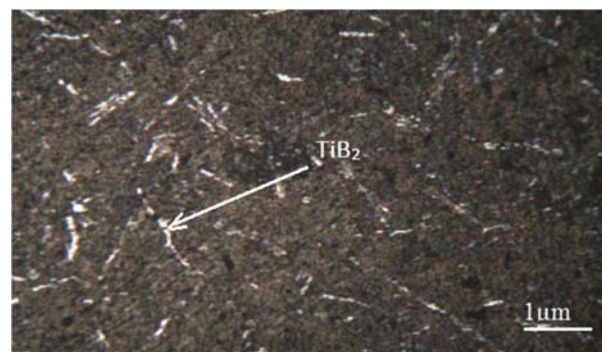


Figure-4. Microstructure of Al LM25/ TiB_2 composite specimen.

4.3 X-ray diffraction analysis of LM25/ TiB_2 AMCs

After synthesizing LM25/ TiB_2 composite by the in-situ reaction, the XRD patterns of the prepared composites are obtained as shown in Figure-5. The diffraction peaks of TiB_2 particles are clearly seen. It is also observed in the figure that the aluminium in the composite peaks have higher 2θ value with respect to



aluminium because of the formation of TiB_2 in the aluminium matrix.

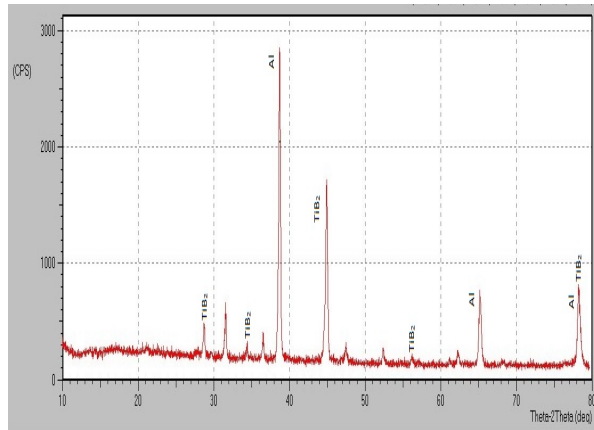


Figure-5. XRD peaks of the Al LM25/ TiB_2 composite.

4.4 Hardness:

A notable rise in hardness of the alloy matrix can be seen with addition of TiB_2 particles. Hardness test reveals a higher hardness indicating the existence of TiB_2 particulates in the matrix.

Hardness of LM25 alloy is measured to be 82HV and by addition of TiB_2 , hardness of Al LM25/ TiB_2 composite increased to 91HV. The grain boundary of aluminium alloy is refined by presence of TiB_2 . The homogeneous distribution of TiB_2 particles is caused due to intermediate stirring. The TiB_2 improves the strength as it deposits in the dislocations. This phenomenon is called Orowan strengthening [13]. TiB_2 particles improve bonding and form a clear border which slows down removing of the particles from the aluminium matrix and thus hardness is increased.

5. CONCLUSIONS

LM25/ TiB_2 AMCs were successfully synthesized by the in-situ reaction of inorganic salts such as K_2TiF_6 and KBF_4 to molten aluminium and TiB_2 particles are formed. Other intermediate compounds form a negligible part of the composite and has less significant effect on the properties of the composite. The microstructures of the developed AMCs showed uniform distribution of TiB_2 particles inside the matrix having proper boundary lines between them and improved intermetallic bond. Because of the presence of TiB_2 particles, there is an improvement in the mechanical properties of the composite. Thus Titanium di boride reinforced LM25 composite can be used for manufacturing cylinder heads, liners, pistons, brake rotors and calipers in automobile industry due to its improved properties.

ACKNOWLEDGEMENT

The authors are grateful to the Department of Mechanical Engineering, Amrita School of Engineering, Coimbatore Institute of Technology and PSG College of Engineering and Technology, for providing the facilities.

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