Processing and characterization of \textit{in-situ} Al-Si-2TiB\textsubscript{2} Composites

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**Abstract**—There is always an increasing demand for advanced materials with enhanced properties through economic processing routes with reference to their field of applications. Tailoring the mechanical properties of light weight Al-Si alloys through stir casting route by virtue of incorporating TiB\textsubscript{2} reinforcement is much simple and convincing with the use of salt metal reaction. High strength to weight ratio, good wear and corrosion resistance, superior castability, machinability and thermal expansion coefficient of Al-Si alloys make them suitable for aerospace and automobile applications. One of the most effective routes of achieving these attributes is by grain refinement and modifying the eutectic silicon accompanied by the reinforcement of TiB\textsubscript{2}. In the present work, the synthesis and characterization of Al-Si eutectic and hypo-eutectic alloys with 2wt. % in-situ TiB\textsubscript{2} through salt metal reaction with the help of stir casting method have been studied. XRD analysis confirmed the formation of in-situ TiB\textsubscript{2} while microstructural changes due to the presence of TiB\textsubscript{2} were observed on both optical as well as SEM micrographs. Enhanced mechanical properties and wear behaviour of these composites were observed with increasing Si content. Besides, worn surface analysis has also been carried out using SEM to study the tribological mechanisms supporting the wear test results.

**Index Terms**—Al-Si alloys, in-situ, microstructure, salt-metal reaction, TiB\textsubscript{2}, wear.

1 **INTRODUCTION**

The Al-Si-TiB\textsubscript{2} composites proved themselves to be an emerging class of MMCs for various engineering applications like automobile industries, aerospace, naval vessels and many more. The properties of these composites can be tailored by altering the matrix composition, amount of reinforcement, wettability of the dispersed phase at interface along with the diameter of these ceramic particles [1]. Reviews on Al-Ti-B ternary system underlined that a little amounts of Ti (0.005 wt.%) and B (0.001 wt.%) in molten aluminium or its alloys have a outstanding tendency of grain refinement [2]. Most of the investigators revealed that reinforcement of TiB\textsubscript{2} enhances tensile strength at cost of a small decrease in ductility. This substantial improvement in properties can be accounted for the homogenous distribution of TiB\textsubscript{2} particles in the matrix [3]. During in situ processing, ultrafine particles of TiB\textsubscript{2} are synthesised within the melt as a result of exothermic reaction among the added constituents with molten aluminium alloy. Fine and uniform distribution of thermodynamically stable reinforcement with clean and clear interface can be achieved by virtue of in-situ synthesis as compared to conventional ex-situ techniques [4].

Amongst different in-situ processing techniques salt-metal reaction using K\textsubscript{2}TiF\textsubscript{6} and KBF\textsubscript{4} halide salts, is simple and fit for bulk production with less contamination [5-8]. The reinforcement of in-situ TiB\textsubscript{2} particles in the matrix offers clean interface of matrix–particle, dispersion strengthening with mismatch in thermal expansion resulting in higher strength and reduced wear rates.[9].

In this study, salt metal route has been adopted for the synthesis of TiB\textsubscript{2} reinforced Al-Si alloys during stir casting. The present study aims at synthesis and physical, mechanical as well as microstructural characterisation of Al-Si-TiB\textsubscript{2} composites.

2 **EXPERIMENTAL DETAILS**

In the present investigation, in-situ Al-Si-2TiB\textsubscript{2} Composites were synthesized following the procedures for stir casting method. And with the help of prior charge calculations, three different compositions were prepared by varying the Si content of the composite, viz.: Al-7Si-2TiB\textsubscript{2}, Al-9Si-2TiB\textsubscript{2} and Al-12.6Si-2TiB\textsubscript{2}. Material characterization of all the samples was carried out by optical microscopy and SEM analysis, which was followed by mechanical testings for obtaining hardness and wear properties.

2.1 **Synthesis of in-situ Al- xSi-2TiB\textsubscript{2} composites**

Synthesis of the composites was carried out via stir casting route taking C\textsubscript{r}-Al (99.9\% purity) and Al-50 wt\% Si master alloy as starting materials and melting in a graphite crucible in a non-ferrous melting furnace at 800\degree C. The Al-xSi- 2TiB\textsubscript{2} (x = 7, 9, 12.6) composites were prepared based on the exothermic reaction between K\textsubscript{2}TiF\textsubscript{6} and KBF\textsubscript{4} halide salts along with molten Al-Si alloy. This was accomplished by addition of calculated amounts of both these salts and allowing a reaction time of 30 minutes. As a result of chemical reactions of the salts, Titanium Diboride (TiB\textsubscript{2}) particles are formed in the molten base alloy. Intermittent stirring was performed for complete reaction of salts and homogenized distribution of TiB\textsubscript{2} particles within molten matrix phase. For ensuring sound casting, all the floating drosses were decanted properly before pouring the melt into the preheated (at 450\degree C) mould [10].

2.2 **Microstructural characterization**

Samples for microstructural characterization were prepared by following the standard metallographic polishing procedures and etching the samples with keller’s reagent. The micrographs of the samples were analysed under computerized...
optical microscope (CARL ZEISS) with differing magnifications and Scanning Electron Microscope (SEM) as well.

2.3 XRD analysis
The X-ray diffraction analysis of the prepared samples (of dimensions 10mm x 10 mm x 2mm) was conducted using Cu-Kα radiation in order to identify various phases present in the samples. The resultant peaks were matched with the standard ones from the JCPDS database.

2.4 Hardness and density
Hardness test was carried out using MicroVicker’s hardness tester using a load of 1 kgf and a dwell time of 15 secs. A minimum of five indentations were made on each test sample and the average hardness value was reported.

The density values of all the samples were measured by using METTLER TOLEDO density kit.

2.5 Wear test
A pin-on-disc wear testing machine was used to study the dry sliding wear behavior of each composite sample. Based on the ASTM G99 standard, cylindrical samples of 30 mm height and 10 mm diameter were machined from each cast composite, which was then followed by conducting the test at room temperature for 5 minutes without using any lubricant and maintaining a track radius of 40 mm. For studying the effect of load (N) and sliding speed of the disc (in rpm) on wear behavior, the applied load was varied as 10N, 20N and 30N and the rpm was varied as 300, 400 and 500 rpm.

3 RESULTS AND DISCUSSIONS

3.1 Microstructural analysis
The optical micrographs of Al-7Si-2TiB₂, Al-9Si-2TiB₂ and Al-12.6Si-2TiB₂ in-situ composites are respectively shown in Fig. 3.1. The composite with eutectic composition of the Al-Si binary alloy indicates needle form of Silicon uniformly scattered throughout the matrix. This can be clearly observed in the corresponding SEM micrograph as shown in Fig. 3.2. TiB₂ particles act as nucleating sites and enhances the nucleation rate than the growth rate, thereby resulting fine eutectic structure.

3.2 XRD analysis
The largest peaks in the obtained XRD results represent Aluminium peaks and smaller peaks signify the existence of Silicon in the synthesized composites. Very small peaks confirm formation of in-situ TiB₂ particles in the base alloy. The Si peak intensity was found to increase marginally with increasing Si content in the composite.
3.3 Density and hardness results
The density and VHN histograms (as illustrated in Fig. 3.4 (a) and (b)) explain that with increasing Si content of AMC, the density value was found to decrease while the hardness was observed to increase. The increasing nature of hardness is attributed to the increasing amount of hard eutectic phase in the matrix.

3.4 Wear analysis
Fig. 3.5 shows wear rate vs Si content plots at different applied load and rpm. In all the curves, the wear rate was found to decrease with increasing Si content because of increasing volume fraction of harder and well dispersed eutectic phase in the matrix as a whole. But the wear rate was brought up with higher applied load and found to be maximum at 30N load. However, wear rate was found to vary inversely with the sliding velocity (rpm), which was attributed to lower residence time.

4 CONCLUSIONS
Present investigation leads to the conclusions as follows:
1) Al-Si-TiB2 in-situ composites were successfully synthesized by salt metal reaction using K2TiF6 and KBF4 salts through stir casting method.
2) Microstructure study showed the uniform distribution of eutectic phase due to presence of TiB2.
3) The XRD analysis showed the presence of TiB₂ in the Al-Si matrix.

4) Density test observations and hardness values were in confirmation with presence of different constituents of the prepared composite samples.

5) Wear rate was found to decrease at all the operating conditions with increasing Si content and sliding velocity, but load enhances wear rate.

REFERENCES


