Mechanical properties enhancement and microstructure study of Al-Si-TiB₂ in situ composites

S K Sahoo*, J Majhi, A B Pattnaik, J K Sahoo and Swagat Das
Department of Metallurgical and Materials Engineering
Indira Gandhi Institute of Technology, Sarang, Odisha, INDIA-759146
*Email: sandeep.talcher@igitsarang.ac.in

Abstract. Al–Si alloy-based composite is one of the most promising MMC materials owing to its outstanding mechanical properties, wear and corrosion resistance, low cost and ability to be synthesized via conventional casting routes. Challenges in achieving clean interface between reinforced particles and matrix alloy have been overcome by means of in-situ techniques of fabrication. Present investigation is concerned with synthesizing Al-Si-TiB₂ in-situ composites through stir casting route using K₂TiF₆ and KBF₆ halide salts for exothermic salt metal reaction. X-Ray diffraction analysis revealed the existence of TiB₂ in the prepared samples. Effect of TiB₂ in-situ particles in the Al-Si base alloy has been investigated from the results obtained from optical microscopy as well as SEM study and wear analysis with a pin on disc wear testing apparatus. Improved hardness and wear properties were observed with addition of TiB₂.

1. Introduction
Al-Si Hypereutectic alloys have gained the curiosity of researchers for their capability to substitute CI counter parts in different fields of application. Al–Si hypereutectic alloys are broadly expended in aerospace industries and automotive fields in a variety of product applications demanding high strength to weight ratio, better wear and corrosion resistance, good machinability, superior castability and thermal expansion coefficient [1]. Achievement of improved mechanical properties of these alloys is feasible by concurrent grain refinement and modifying the primary as well as eutectic silicon with the reinforcement of TiB₂. Incorporation of hard TiB₂, SiO₂, SiC or Al₂O₃ particle reinforcement in the base alloy matrix seldom accounts significant ascent the density but rather guarantees appreciable enhancement in the strength to weight ratio, modulus and tribological properties [2-5].

Al-Si-TiB₂ composites reveal beneficial and exceptional features in regard to its properties and synthesis route. Being a ceramic particle TiB₂ reveals some peculiar properties like very high melting point (more than 2750°C), excellent hardness value (above 950 HV) and superior modulus (about 5.3 x 10⁵GPa). Strain fields are generally built up at the interface due to the mismatch in the coefficient of thermal expansion between Al and TiB₂ as a result of which rise in dislocation density occurs and serve as preferable sites for nucleation. Moreover, the exothermic reaction of halide salts in the Al melt accounts for a beneficial contribution for formation and reinforcement of in-situ TiB₂ particles in Al based alloys with the use of salt metal reaction. Unlike ex-situ method, incorporation of the in-situ technique for synthesis of the composite eliminates inhomogeneous distribution, poor wettability of the reinforcing phase and provides a clean interface in absence of undesirable reaction products. TiB₂ particles are accountable for grain refinement of the Al-Si alloy, pining of dislocation line, and formation of Orowan dislocation loops and also grain boundary strengthening in Al-Si-TiB₂ metal matrix composites [6-8].

In this work an attempt has been made to explore the synthesis and characterization of Al-Si eutectic and hyper-eutectic alloys with 5wt. % in-situ TiB₂ through salt metal reaction with the help of stir casting method.

2. Materials and methods
Composites of Al-Si alloy with 12.6, 15 and 18 wt.% Si reinforced with 5wt.%TiB₂ have been prepared with the use of halide salts. Physical, mechanical and tribological characterisations have been conducted to evaluate the properties of the samples.
2.1. Composite fabrication
Cp grade aluminium and master alloy of Al-50 wt% Si supplied by Minex Metallurgical Co. Ltd., have been taken in a clay-graphite crucible for melting in a pit type f/c at about 800°C to synthesise Al-x Si (x = 12.6, 15, 18) alloy. The Al-xSi-5TiB₂ composites were manufactured by adding together K₂TiF₆ and KBF₄ halide salts to molten Al-Si alloy and allowed for 30 minutes of reaction time. Titanium Diboride (TiB₂) particles are formed as a result of chemical reactions of the salts in the molten base alloy. Periodic stirring was carried out to ensure the reaction of salts to be complete and dispersion of TiB₂ particles to be uniform within molten aluminium alloy. Before casting, all the lighter dross was discharged to ensure sound casting. The desired composites specimens were casted by pouring into cast iron moulds preheated at 450°C [6].

2.2. Characterizations
2.2.1. Microstructural characterization
Samples for microstructural observations were prepared following standard procedures and etched with Keller’s reagent. The micrographs of the composites have been studied with the use of optical microscope (CARL ZEISS) at varying magnifications and with Scanning Electron Microscope (SEM) as well.

2.2.2. XRD analysis
XRD analysis of the prepared samples was carried out with Cu-Kα target to recognize presence of various constituents of in the samples by matching the obtained peaks with JCPDS data files.

2.2.3. Hardness and Density Test
Hardness values of the specimens were measured in Microvicker’s hardness tester using a square base diamond pyramid as indenter with 1kgf applied load and 15 seconds of dwelling time. Samples were tested for their density in Mettler-Toledo digital balance with density kit.

2.2.4. Wear test
A pin-on-disc wear testing machine have been in use to evaluate the wear behaviour of the cylindrical pins of the composite sample with diameter of 10mm and 30mm length. The operating test parameters comprised of varying load (10, 20, 30N) with 40mm track radius for 300 seconds at room temperature without use of any lubricating material. After every test the mass loss due to wear of each specimen was determined. The role of applied load and amount of silicon on wear characteristics of the prepared composites was studied.

3. Results and discussions
3.1. Microstructural characterization
Figure 1 and figure 2 reveals that the composite with eutectic composition of the alloy exhibits needle form of silicon uniformly scattered all over the Al-matrix with little amount of primary silicon due to non equilibrium cooling. But in the Al-Si alloy with hyper eutectic compositions more primary Si particles are evident in micrograph. In presence of TiB₂ particles, those act as the preferred sites for nucleation during solidification, refine the structure and almost rounded shaped primary Si of smaller size were formed unlike cast base alloys.

Figure 1. Optical Micrographs of (a) Al-12.6Si-5TiB₂ (b) Al-15Si-5TiB₂ (c) Al-18Si-5TiB₂.
Figure 2. SEM Micrographs of (a) Al-12.6Si-5TiB$_2$ (b) Al-15Si-5TiB$_2$ (c) Al-18Si-5TiB$_2$

3.2. XRD analysis

The largest peaks in the obtained XRD results describe the presence of aluminium. Minor peaks signify the existence of silicon and TiB$_2$ particles. It is also noticed in figure 3 that the intensity of the peaks of silicon increases with its increase in the weight percentage in the base alloy. Tiny peaks of TiB$_2$ in the XRD pattern corroborate the occurrence of TiB$_2$ in the selected alloy matrix.

Figure 3. XRD analysis of composites

3.3. Hardness and density

Figure 4 (a) Hardness as a function of amount of silicon (b) Density as a function of amount of silicon of the composites

The VHN and density histograms of figure 4 illustrate that AMC with 18 % Si possesses utmost hardness with minimum density value. Rise in the quantity of the hard primary silicon as well as eutectic Al-Si
phases as a result of increasing Si content in the base alloy accounts for enhanced resistance to indentation and provides high hardness values for the composites.

3.4. Wear test

Figure 5. Wear rate with respect to amount of Si and applied load at (a) 300 rpm, (b) 400 rpm, (c) 500 rpm and Wear rate with respect to amount of Si and rpm at (d) 10N, (e) 20N, (f) 30N.
Figure 6 Sp. wear rate w.r.t. amount of Si and load at (a) 300 rpm, (b) 400 rpm, (c) 500 rpm and Sp. wear rate w.r.t. amount of Si and rpm at (d) 10N, (e) 20N, (f) 30N.

Figure 5 shows wear rate of prepared samples with three varying wt% of Si, at different applied load and rpm. The wear rate found to decrease with increasing amount of Si because of enhancement in hardness of the composite, refining of primary as well as eutectic silicon and their modification but the wear rate was brought up with higher applied load and found to be maximum at a load of 30N. Figure 6 shows specific wear rate of the composite and a similar trend was observed for varying amount of Si and
minimum specific wear rate was observed in composites with increased amount of silicon in all conditions. Uniform dispersion of the TiB$_2$ particles as a result of intermittent stirring and achievement of size reduction of blocky primary silicon as a result of presence of TiB$_2$ particles contributes to lower sp. wear rate in Al-18Si-5TiB$_2$ composites.

Generally, with increasing applied load sp. wear rate decreases for a fixed sliding distance due to the work hardening of the sliding surface. Modification in morphology from coarse eutectic Si plates to fine needles and refinement of the primary Si leads to high wear resistance of Al–Si alloys. Wear rate as well as sp. wear rate found to be inversely proportional to sliding speed due to more resident time for intimate contact of the mating surfaces and more material flow required maintain the relative motion. This corroborates the findings of S.A. Kori, T.M. Chandrashekharaiah [9].

4. Conclusions
Present analysis leads to the conclusions as follows:
1) In-situ Composites of Al-Si alloys reinforced with 5wt%TiB$_2$ have been successfully manufactured through exothermic reaction of halide salts via stir casting route.
2) Occurrence of TiB$_2$ in the prepared composites was ascertained by XRD analysis.
3) Higher hardness and lower density values were perceived in composites with higher amount of silicon.
4) Wear rate as well as specific wear rate found to decrease at all the working conditions with higher silicon content.
5) Microstructure study disclosed the role of TiB$_2$ in alteration in morphology and distribution of primary and eutectic silicon in Al-Si base alloy.

References
[1] Gupta M and Lavernia E J 2001 Journal of materials processing technology 54 1-4 261-270.
[2] Mandal A, Maiti R, Chakraborty M and Murty B S 2004 Materials Science and Engineering A 386 1-2 296-300.
[3] Sahoo S K, Majhi J, Patnaik S C, Behera A, Sahoo J K and Sahoo B P 2017 Elixir Materials Science 113 49066-69.
[4] Mandal A, Murty B S and M. Chakraborty 2009 Materials Science and Engineering A 506 1-2 27-33.
[5] Mandal A and Makhlouf M M 2009 Transactions of the Indian Institute of Metals 62 4-5 357-360.
[6] Mallikarjuna C, Shashidhara S M, Mallik U S and Parashivamurthy K I 2011 Materials & Design 326 3554-59.
[7] Rajan H B M, Ramabalan S, Dinaharan I and Vijay S J 2014 Archives of civil and mechanical engineering 14 1 72-79.
[8] Sahoo J K, Sahoo S K, Sutar H and Sarangi B 2017 IOP Conference Series: Materials Science and Engineering 178 1 (IOP Publishing, 2017) pp1-10.
[9] Kori S A and Chandrashekharaiah T M 2007 Wear 263 1-6 745-755.