DEVELOPMENT OF ALUMINIUM BASED METAL MATRIX COMPOSITES

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ABSTRACT

A detailed study on the processing of Al-metal matrix composites with the reinforcement of different particulates such as SiC, TiN and TiO_2 was carried out. The Al-SiC composite powder prepared through atomisation route is expected to limit the interfacial reaction between the matrix and the ceramic particulate and help in making customer tailored components by powder metallurgy route. An attempt to avoid the interfacial reaction of matrix and ceramic is investigated by impregnating the Al matrix with TiN particulates. Similarly, Al-TiO_2 composite is investigated by new spray forming technique, where the interfacial reaction between Al and TiO_2 is expected to form intermetallic compounds and improve the bonding and mechanical properties. The results of the present studies show that the Al based composites prepared through various techniques exhibits excellent mechanical, physical and tribological properties and could emerge as promising materials for defence, aerospace and other engineering applications.

Key Words : Al-Metal matrix composites, Atomisation, Powder metallurgy, Spray forming.

INTRODUCTION

Materials that have superior physical, mechanical and tribological properties are desired for various engineering applications. The materials with less weight and high strength are considered as important materials for their potential applications and composites are no exception. In this regard, extensive research work is being carried out for the past two decades to develop wide range of metal matrix composites (MMCs) as alternatives to the conventional engineering alloys and the same has showed tremendous promise and phenomenal growth (1-4). Moreover, introducing the brittle ceramic materials as reinforcement showed improved mechanical properties of MMCs (5-7). The particulate reinforced MMCs are of more interest due to simple preparation techniques, lower cost and more isotropic properties. In addition, it is also shown that the properties of MMCs are controlled by the size and volume fraction of the reinforcement phases as well as by the nature of the matrix-reinforced interface. A fine and thermally stable ceramic particulates distributed uniformly in the matrix has led to the optimum set of mechanical properties (3,7). Among the various MMCs that are being developed, aluminium based metal matrix composites have shown a continuous improvement of properties and emerging as promising futuristic materials for various engineering applications. Most of the research on MMCs is being carried out to improve its mechanical properties by the incorporation of hard ceramic oxides/ carbides/ nitrides into the metal matrices. In this paper, the preparations of SiC, TiN and TiO_2 particulates have been incorporated to the Al matrix through air atomisation, powder metallurgy and spray deposition techniques respectively.

EXPERIMENTAL DETAILS

Al-SiC composites by air atomisation technique

Commercial grade aluminium (99.8% pure) was taken as the base material and 10 wt% of silicon was added to aluminium during melting. The addition of silicon to aluminium serves dual purposes that included the retardation of the reaction between liquid aluminium and silicon carbide at elevated temperatures (>750°C) and improvement of the flowability of liquid aluminium. It was understood that high flowability of liquid was essential for its better atomisation. In order to prepare aluminium-silicon carbide composite, commercial grade(Grindwel Norton, India) α-SiC having particle size of less than 60 mm was used. Silicon carbide powder (10wt%) was initially mixed with aluminium powder through mechanical mixing technique to give coating of Al on SiC powder for better wettability.

The required quantities of aluminium was initially melted in an induction furnace and 10 wt% silicon metal was added to the liquid aluminium. The temperature of the melt was maintained at ~900°C. The Al and SiC powder mixture was preheated at ~200⁰C and subsequently added slowly to the molten Al-Si. During addition of powder mixture the liquid was also mechanically stirred. A liquid metal temperature was maintained down to 900°C as high flowabiltiy is required for atomisation. The tundish along with the whole nozzle assembly was heated to avoid the sticking and minimise the loss of heat during pouring. The heat loss during pouring could lead to solidification and hindered the atomisation process by choking the orifice of the nozzle. A 4 mm diameter graphite nozzle was used for atomising the composite powder. The air pressure was maintained at 50 psi during atomisation. The atomised powder was collected in a water-receiving tank and major portion of water in the composite powder was removed by the centrifugal filtration. Rest (~5-7%) of water was removed during drying by a vacuum oven at ~80⁰C. The atomisation technique is schematically shown in figure 1.

Al-TiN composite prepared through powder metallurgy route

Ti sponge (>98% pure) was nitrided at 1550°C for 45 min using nitrogen gas and the lumpy mass is ground. The process of nitridation and grinding were repeated for 3 times to obtain single phase TiN powder. Finally the TiN powder was pulverised in a cup mill using agate jar to reduce the particle sizes and again milled with copper powder (-300 mesh and 99.5% pure), which is added to increase the wettability of Al and TiN. The atomised Al powder (99% pure) having a particle size of less than 53 μm was mixed with TiN and Cu. The powder mixture was ball milled for 12h and subsequently compacted in the form of cylindrical pellets (2-cm diameter and 1- cm height) by applying a load of 250 MPa. The pellets were sintered at 450°C for 1h and subsequently hot pressed at 450°C for 15 min. The details of the experimental conditions are given in Table-1.

Table: 1 : Composition and Heat-treatment condition for the fabrication of Al-TiN composites

Sample	Composition (Wt %)			Processing Condition	
	Al	TiN	Cu	Sintering	Hot pressing
A	86	10	4	723 K for 30 ins. in N_2 atmosphere	723 K for 15 min. under a load of 250·MPa
B	66	30	4	723 K for 30 min. in N_2 atmosphere	723 K for 15 min. under a load of 250 MPa

Preparation of Al-TiO_2 composite by spray forming technique

The schematic diagram of the spray forming technique employed is shown in figure 2. The procedure for melting was similar to the atomisation technique. The atomisation unit consisted of two metallic tubes being placed concentrically inside the nozzle. The outer tube was fitted to the atomiser, while the inner tube was suspended by fixing it on the steel frame with a screw and jack arrangement, so that the tube could be concentrically moved to the desired position. Mild steel and inconel tubes were used as outer and inner tubes respectively. Metallic tubes were selected due to their high thermal shock resistance as compared to the commonly used ceramic nozzles. Further, the same set of tubes could be reused. Care was taken to prevent the reaction of melt with the tubes, by providing boron nitride coating on the surfaces of the tubes. It was found that this coating not only helped in preventing any reaction between the melt and the tubes, but also erosion has not been taken place during the preparation of the alloy. This was due to poor wettability between Al melt and the boron nitride coating. The end of the tubes was narrowed in the form of cone at the orifice. Two sets of such concentric tubes were made with varying outer and inner diameter, so that the flow rates of the melt and particles could be varied. This in turn helped in varying the volume fraction of the material passing through the tube. A funnel type arrangement was made on the inner tube so that the ceramic particles (rutile in the present case) could be easily pass. The deposition was carried out on a copper substrate, which was fixed at 30cm below the nozzle. The substrate was rotated at different speed and the speeds of rotation were optimised by measuring the density of the composite preforms for one set of tube.

The average size of the rutile particles was 113μm with a standard deviation of 38μm. About 1.5 Kg of aluminium was melted in a resistance furnace and degassed by adding hexachloroethane. Prior to atomisation Mg (2wt%) was added to the melt to increase its flowability and the wettability of the melt with the ceramic particles (TiO_2). The melt and the preheated (200°C) rutile particles were atomised simultaneous through outer and inner tubes respectively. The melt was atomised by using argon gas, so that fine droplets and the rutile particles were dispersed uniformly. The resultant composite mixture (mushy droplet and rutile particles) was subsequently deposited on a rotating copper substrate, placed vertically below the atomiser. The whole atomising assembly (graphite crucible, atomiser and nozzle) was preheated to 600°C to avoid choking of melt at the orifice during atomisation.

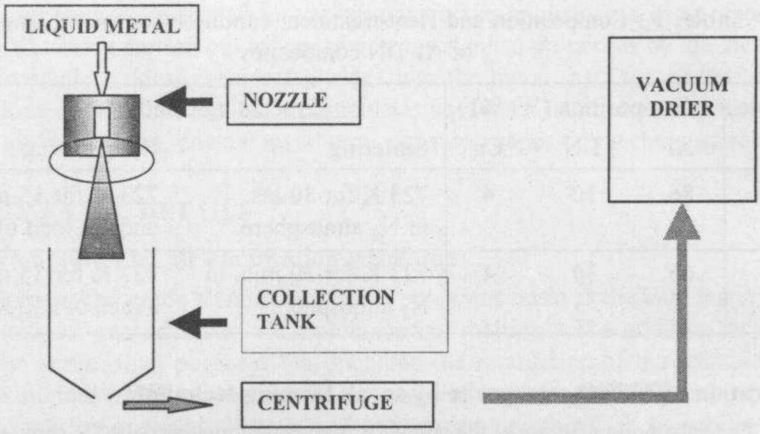


Fig. 1 : Schematic diagram of air atomisation process

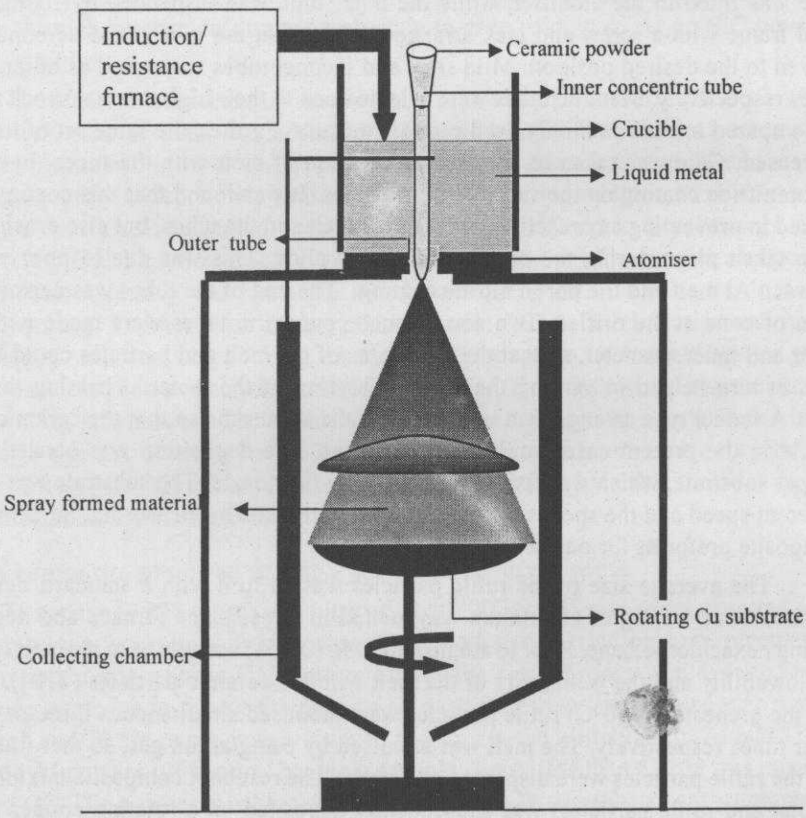


Fig. 2: Schematic diagram of spray forming set-up for preparation of Al-TiO_2 composites

RESULTS AND DISCUSSION

Al-SiC composites

The particle size analyses of the mechanically mixed SiC and aluminium (50:50) powder (before addition to molten aluminium) was found to be in the range of 30 to 50 μm. The XRD results confirmed the presence of SiC and aluminium in the mixed powder. Since SiC was coated with aluminium, the apparent intensity of SiC peaks were lower than expected (Fig. 3).

The atomised powders (composite mixture of aluminium and silicon carbide) as well as cold compacted sample were examined by using scanning electron microscope and EDX. During SEM studies, it was observed that most of the silicon carbide particles were uniformly distributed. The presence of Si was detected by the EDX analysis, whereas, X-ray diffraction analyses indicated the presence of SiC in the powder (Fig. 3). The EDX observation in conjunction with the X-ray phase analyses confirmed the dispersed black particles as SiC. The average particle size of the atomised powder was observed to be around 20-60 micrometer.

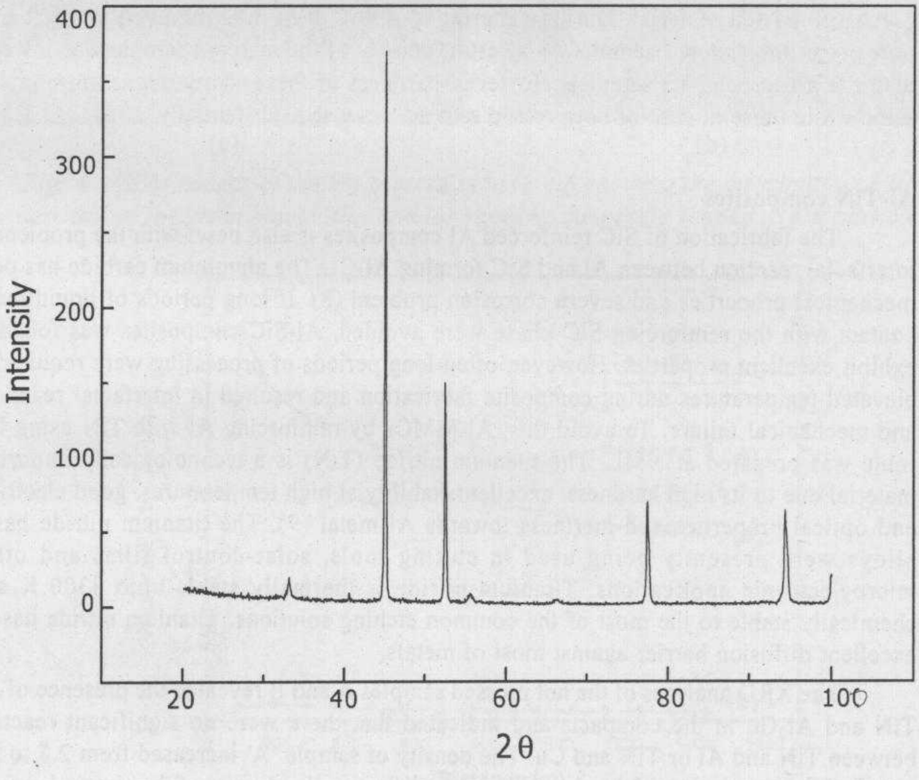


Fig. 3 : XRD pattern of Al-SiC Composite powder

The microstructural examination of the sintered composite samples showed that the SiC particles were well distributed in the matrix. It was also been observed that the bonding between the aluminium particles was not proper in the samples sintered at 450⁰C, whereas the silicon carbide particles were well bonded with the aluminium particles. To improve the bonding among the aluminium particles, the samples were hot pressed at 450°C. This leads to improvement of the hardness and density values (Table 2).

Table 2 : Density and hardness values of various stages of Al-SiC composite.

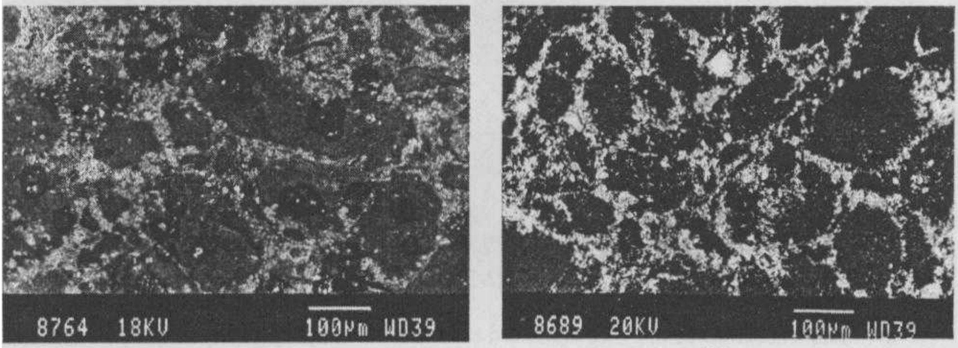
Item	Green compact	Sintered (450⁰C)	Hot Pressed (450⁰C, 260 MPa)
Density	2.4 gm/cc	2.3 gm/cc	2.7 gm/cc
Hardness	48 VPN	50 VPN	55 VPN

It was observed from the results that the density of sintered sample was apparently lower compared to the green compact sample. This could be probably due to the presence of binding material e.g. PVA, which might have covered the open porosity in the case of green compacted material. During sintering at 450⁰C PVA was removed and left high porosity in the sintered sample. As a result, density of the sintered sample was lowered than the green compact samples. However, hardness of these samples was more or less same while those in case of hot pressed sample increased substantially.

Al-TiN composites

The fabrication of SiC reinforced Al composites is also beset with the problem of interfacial reaction between Al and SiC forming Al_4C_3. The aluminium carbide has poor mechanical properties and severe corrosion problem (8). If long periods of liquid metal contact with the reinforcing SiC phase were avoided, Al-SiC composites was found to exhibit excellent properties. However, often-long periods of processing were required at elevated temperatures during composite fabrication and resulted in interfacial reactions and mechanical failure. To avoid this, Al-MMCs by reinforcing Al with TiN using PM route was prepared at NML. The titanium nitride (TiN) is a technologically important material due to its high hardness, excellent stability at high temperatures, good electrical and optical properties and inertness towards Al metal (9). The titanium nitride based alloys were presently being used in cutting tools, solar-control films and other microelectronic applications. Titanium nitride is thermally stable upto 3300 K and chemically stable to the most of the common etching solutions. Titanium nitride has an excellent diffusion barrier against most of metals.

The XRD analyses of the hot pressed samples A and B revealed the presence of Al, TiN and Al_2Cu in the compacts and indicated that there were no significant reaction between TiN and Al or TiN and Cu. The density of sample 'A' increased from 2.7 to 2.9 gcm^{-3} after hot pressing of the sintered pellets. Whereas the density of the sintered sample B increased from 2.8 to 3.0 gcm^{-3} after hot pressing. The maximum densities attained by the sample A and B after hot pressing are 98% and 91% respectively. The observed low densities in the sample B was possible due to the presence of excess amount of secondary phases in the matrix, which was not participating during sintering and also hindered the mass transport processes during sintering. The microstructural analyses of the samples exhibited that the TiN particles were uniformly distributed along the grain boundaries in the hot pressed Al-10TiN samples (Fig. 4a). Needle shaped precipitations were observed in the Al-30TiN samples (Fig. 4b) and that the composition of these grains were detected to be Al_2Cu from the EDX analyses. The fabricated composites also exhibited excellent mechanical properties (10). The higher volume fraction of Al-30TiN exhibited better wear properties as compared to Al-10TiN. Similarly, both the composites showed better wear response than pure Al (Fig. 5).



(a) (b)

Fig. 4 : SEM images of the hot pressed sample; (a) showing the distribution of TiN particles at the grain boundaries and (b) showing the needle shaped Al_2Cu particles

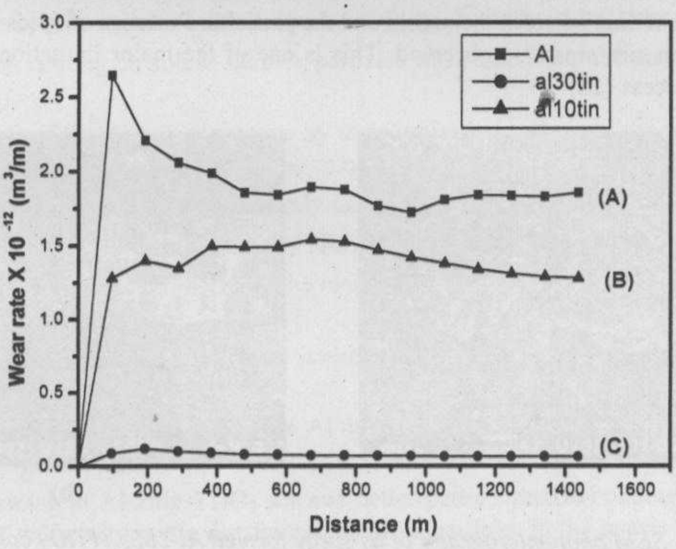
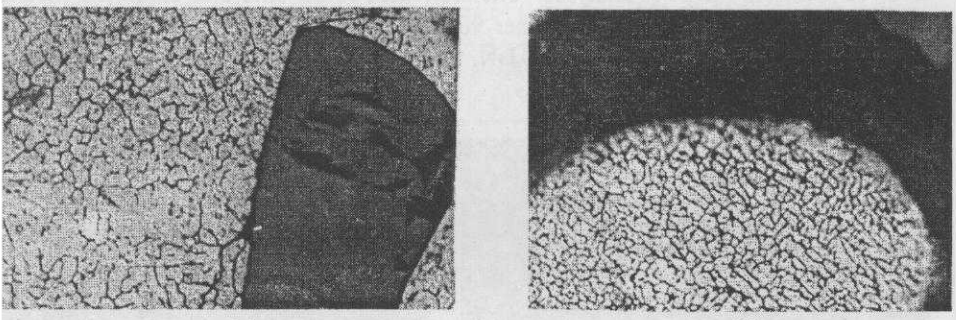


Fig.5 : Wear rate of (a) Al, (b) Al-10TiN and (c) Al-30TiN

Al-TiO_2 Composites

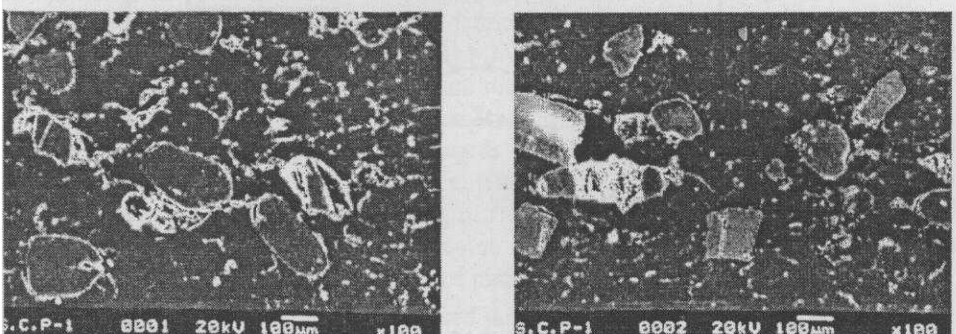
The microstructural investigations of over sprayed powder showed that some of rutile particles were engulfed by the liquid aluminium during atomisation and resulted in the formation of a composite powder. One of such composite powder is shown in figure 6a. Both the over sprayed powder Al-2Mg powder (Fig. 6b) and composite powder have revealed dentritic structure. This was in contrast with the as spray formed composite, which revealed fine equiaxed structure observed during macrostructural observations (11).



(a) (b)

Fig.6 : Optical micrographs of over sprayed powder (a) Al-rutile and (b) Al-2Mg alloy X500

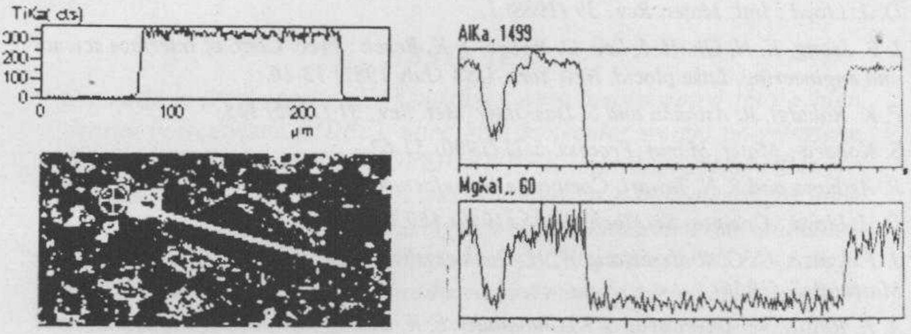
The evolution of the fine equiaxed structure in as-sprayed deposited perform could be attributed to the generation of large number of nucleation sites owing to the turbulent fluid flow condition during deposition of the mushy droplets (12). The microstructure of as spray formed composite at two different locations (center and periphery of the preform respectively) is shown in fgure 7a and 7b. This shows the uniform distribution of dispersoid with good bonding between the matrix and the particles. Presence of voids in the range of 40-70 µm in size was also observed. This is one of the major limitations of the spray forming process (12).



(a) (b)

Fig.7 : SEM photomicrograph of as spray formed Al-2Mg-11TiO_2 composite at locations a) centre and b) at periphery

Back scattered elctron image and lines of Al, Ti and Mg of spray formed composite are shown in figure 8 a,b. At the particle/matrix interface, Al_2O_3 phase was formed in the form of a thin layer. This was probably due to the ex-situ reaction between the Al melt and TiO_2 particles. Further distribution of Mg was observed to be uniform through out the matrix. Higher cooling rates involved in the spray forming process might be prevented the preferential segregation of Mg at the interface. In all the three line profiles, deflection in the concentration of the constituents was noted at some places due to the presence of porosity.



(a) (b)

Fig. 8 : (a) Back scattered image of Al-2Mg-7TiO₂ spray formed and line profile of Ti and (b) line profile of Al (top) and Mg (bottom)

CONCLUSIONS

1. Al-SiC composite powder has been successfully prepared by atomisation technique.
2. Aluminium coating on silicon carbide particles was responsible for the better wettability.
3. Preparation of the Al-SiC composite by the atomisation technique can eliminate number processing steps as compared to other methods.
4. The SiC particles are almost uniformly distributed through out the matrix.
5. Al-TiN composites were successfully fabricated by powder metallurgical route
6. No significant interaction between the matrix and reinforcement is observed during sintering and hot pressing
7. Fabricated Al-TiN composites show improved densification only after hot pressing, which also increase the hardness
8. Incorporation of TiN particles in the Al matrix improves the mechanical properties and wear resistance
9. Spray forming of Al-2Mg-7TiO_2 showed better particle/matrix bonding and higher degree of uniformity in the distribution of rutile particles in the matrix
10. cold rolling of spray formed Al-TiO_2 composites exhibited improved hardness values
11. The ultimate strngth of as-sprayed composite increased considerably on cold rolling.

ACKNOWLEDGEMENTS

The authors would like to thank Director, National Metallurgical Laboratory, Jamshedpur, for his kind permission to publish this paper.

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