# Processing and Mechanical Behavior of TiB<sub>2</sub> and ZnSt<sub>2</sub> Reinforced Aluminium Alloy 6061 Hybrid Composite using Stir Casting

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Abstract – Aluminium Alloy 6061 is reinforced with  $TiB_2$  of average particle size 8.915 microns and Zinc Stearate (ZnSt<sub>2</sub>) of 8.9 microns. ZnSt<sub>2</sub> is a better solid lubricant and it is preheated and directly injected into the crucible during the stir casting process. Stirring of the molten melt and reinforcements are carried out using graphite stirrer within the graphite crucible. The characterization is performed through the Scanning Electron Microscope (SEM) and Energy Dispersive Spectrum (EDS). SEM analysis confirms that there is a uniform distribution of reinforcements in the hybrid composite. EDS is used to confirm the elements in hybrid composite. The experimental result confirms that the hardness increases with increasing the volume % of reinforcements and some useful conclusions are made.

Index Terms – Stir casting, Aluminium alloy, Scanning Electron Microscopy, Energy Dispersive Spectroscopy, Hardness.

## 1. INTRODUCTION

Metal Matrix Composites (MMC) consists of metal alloys Aluminium, Magnesium or Titanium as the matrix material [1]. However Aluminium matrix is the competitive material in the industries [2]. Reinforcement plays a very important role in composite because it determines mechanical properties, cost and its performance [3].

Aluminium Alloy (AA) is reinforced with ceramic material like SiC, TiB<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, B<sub>4</sub>C which result to enhance its mechanical properties wear resistance and strength to weight ratio [4]. AA 6061 is having wide number of industrial and commercial applications viz. marine fittings transport, bicycle and electrical TiB<sub>2</sub> has emerged as the outstanding material due to

its, extreme melting point and its applicability in high temperature structural applications [5]. It is also resistant to oxidation in air, HCl and HF. It is having limitations also it reacts with  $H_2SO_4$  and  $HNO_3$  also it is attacked by alkalis [6].

Low density and high specific mechanical properties of aluminum MMC make these alloys one of the most attractive material substitutes for the manufacture of lightweight parts for many types of vehicles. It's wear resistance and strength equal to cast iron, but it is having 67% lower density and three times the thermal conductivity which makes aluminum MMC alloys as exceptional materials for the manufacture of lightweight automotive and other commercial parts [7]. The behavior of TiB<sub>2</sub> particles in molten aluminium were investigated by casting experiments at different cooling rates and particle addition levels, starting with a master alloy containing in situ formed TiB<sub>2</sub> particles [8]. When there is mixing of reinforcement and matrix in molten state interaction takes place between advancing solidification part and the particles at this time solidification front pushes or entraps the particles results the particles can be found at the grain boundaries, the interdentritic regions, or within the primary grains themselves [9].

In situ composite is fabricated by the thermodynamic reaction between Potassium Hexa Fluoride and Potassium Tetrafluro Titanate ( $K_2TiF_6$ ) in the presence of AA 6061 alloy. Presence of transitional compound Al<sub>3</sub>Ti interrupts the uniform of distribution reinforcement and degrades the properties of composite [12]. Zinc Stearate (ZnSt2) is zinc soap and it is widely in industry, it is metal salt of a fatty acid. Its color is white and it repels water it is insoluble in polar solvents such as alcohol but soluble in benzene and chlorinated hydrocarbon when it is heated. It is used as a release agent in rubber, powder metallurgy [10]. Forgeablity of AA6061 produced by powder metallurgy method depends on binder (ZnSt<sub>2</sub>) [11].

Chen et al. (2016)[12]has developed TiB<sub>2</sub> composite using two step route , the step first they optimized the halide salt route to fabricate and in second step examined the effects of Al-5wt.% TiB2 composite and found that the grain refinement and interaction with the hard particles gives extra strength to the material. It is also analyzed that agglomeration during the solidification is responsible for the strength of composite.

Fei Chen et al. (2015) [13] developed the composite with the mechanical stirring of salts develop TiB<sub>2</sub> particulates and to fabricate composite based on situ. Processing parameters of stirring salts/aluminum interface mechanical have extraordinary effects on microstructure and mechanical properties of the final composites. It is concluded that proper stirring intensity and duration plays an important role to overcome the formation of severe agglomeration, to prevent inclusion entrapping and melt oxidation. The results have shown that during the first and last 15 min of 60 min holding time is important for the distribution of particles in the composite while stirring at 180rpm between these intervals. The fabricated composite shows improvement in the mechanical properties

Suresh et al. (2014)[14] have manufactured composite using high energy stir casting technique with Al6061 and using TiB<sub>2</sub> powder using various proportions (0,2,4,6,8 and 10%) Also investigated structural characterization through XRD, EDS and SEM. Mechanical properties like hardness, tensile and wear. Mechanical properties such as tensile strength, wear resistance and hardness increased by the percentage of TiB<sub>2</sub> present in the samples when compared with base aluminium alloy

Selvaganesan & Suresh (2013)[15] concentrates on production of Al-TiB<sub>2</sub> MMC using the stir casting method with various compositions of  $TiB_2$  (0, 3, 6, 9 and 12). The tensile strength increased of 9% TiB<sub>2</sub> is 219MPa while with 0% TiB<sub>2</sub> it is 178 MPa. The hardness value with 12% TiB<sub>2</sub> is 89VHN but with 0% TiB<sub>2</sub> it is 40.44VHN.It is revealed that the composite developed shows uniform distribution of the particle in the matrix and good interface bonding between the ceramic phase and the metallic matrix.

Lakshmi et al. (1998) [16] investigated Al-TiB<sub>2</sub> composite fabricated by exothermic reaction between K<sub>2</sub>TiF<sub>6</sub> and KBF<sub>4</sub> salts at 850°C Period of reaction was varied between 10 min to 40 min and investigated that there is decreasing trend in grain size of the composite up to a reaction time of 30 min and it increases slightly for a reaction time of 40 min. They used X- ray diffractometer to confirm the presence of TiB<sub>2</sub> and to estimate the weight percentage of TiB<sub>2</sub> particles in the composite various reaction holding times.

Gaurav Mahajan et al. (2015)[17] fabricated hybrid composite using SiC and TiB2 with various composition of reinforcement as 10% SiC with various percentage of titanium boride 2.5, 5 and 10 TiB<sub>2</sub> and examined mechanical tribological properties. Microstructure of 10% SiC shows that it is present in inter dendritic structure and is evenly scattered in the Aluminium matrix. While composite with 10% SiC with 2.5 and 5 TiB<sub>2</sub> reinforcement sample. TiB<sub>2</sub> is present in hexagonal crystal form. These crystals are enclosed by inter dendritic structure of SiC uniformly distributed in the AL matrix. Hardness value increases on addition of SiC and TiB<sub>2</sub> to the AA6061 matrix by considerable amount. Hardness increases by 38% in the case of Al/10 SiCp while in the case of Al/10SiCp/5TiB<sub>2</sub> it is 35.7% .With the addition of SiC and TiB<sub>2</sub> to the matrix material wear rate and coefficient of friction decreases.

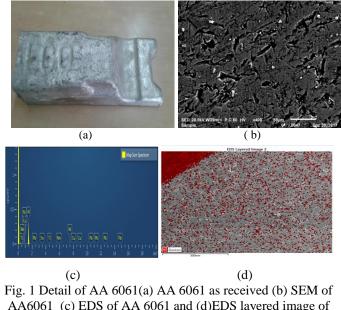
Literature confirms that there is no work has been carried out with the addition of TiB<sub>2</sub> and ZnSt<sub>2</sub> in AA6061 alloy. Present work focuses on improvement of particle distribution of TiB<sub>2</sub> by injecting ZnSt<sub>2</sub> during metal matrix casting by stir casting method.

### 2. MATERIALS AND METHODS

2.1 Material

AA 6061 was used as matrix material and TiB<sub>2</sub> and Zinc stearate are the reinforcement materials.

2.1.1 Matrix material



AA6061 (c) EDS of AA 6061 and (d)EDS layered image of AA6061

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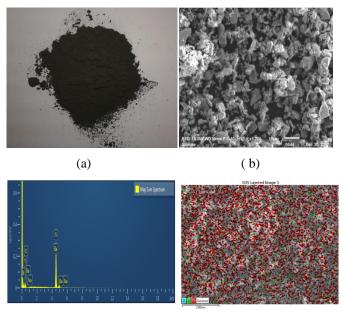
AA 6061 was used as the matrix material and it is in the form of ingots. The Fig. 1(a) shows the received ingot from Hindalco, Renukoot in Uttar Pradesh. Fig 1(b) shows the SEM image of AA 6061 at X400 and Fig 1(c) shows the EDS of AA 6061 it confirms the purchased material was Al 6061 is aluminum with alloy elements of Cu,Si,Mn,Fe,Ni,Zn,Ti,Pb,Sn.

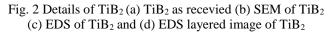
## 2.1.2 Reinforcement material

 $TiB_2$  and  $ZnSt_2$  are used as the reinforcement material. It is purchased from Sigma Aldrich, Chandigarh, India. The purity of the powders is 99.7 and 98.3 % respectively.

# 2.1.2.1 Titanium diboride

The fine particles of TiB<sub>2</sub> are used as a one of the reinforcement having size of less than 10  $\mu$ m. The hardness of TiB<sub>2</sub> is 1800 knoop [6]. The volume % of TiB<sub>2</sub> varied during the fabrication. Fig. 2(a) shows the purchased TiB<sub>2</sub> in the form of powder. Fig 2(b) shows the SEM of TiB<sub>2</sub> powder at X1200. Fig 2(c) shows the EDS of TiB<sub>2</sub>.The result of EDS confirms that the purchased was TiB<sub>2</sub>. The elements are identified as Ti,B,O.





(d)

#### 2.1.2.2 Zinc stearate

(c)

Particle size of zinc Stearate is 8.9 microns. It is the most powerful mold release agent among all metal soaps its chemical formula  $C_{36}H_{70}O_4Zn$ . The volume % of Zinc stearate varied during the fabrication process. Fig 3(a) shows the purchased ZnSt<sub>2</sub> in the form of powder, Fig 3(b) shows the SEM of ZnSt<sub>2</sub> at X1200 and Fig.3(c) shows the EDS of ZnSt<sub>2</sub>. The result shows the purchased product is  $ZnSt_2$ . The elements are identified as Zn,C,O,Na.

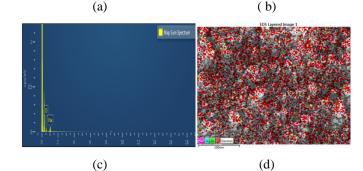


Fig. 3 Detail of ZnSt<sub>2</sub>(a) ZnSt<sub>2</sub> as received,(b) SEM of ZnSt<sub>2</sub> and (c) EDS of ZnSt<sub>2</sub> (d) EDS layered image of ZnSt<sub>2</sub>.

#### 2.2 Stir casting

AA 6061 was used as matrix material and TiB<sub>2</sub> and Zinc sterate is used as reinforcement material with different volume % in order to produce composite. The fabrication was done at IIT Ropar. It is shown in Fig. 4(a). The AA 6061 was charged to the graphite crucible. The matrix material heated up to melting point by setting furnace temperature of 730  $\,^{0}C$  as shown in the Fig. 4(b). The TiB<sub>2</sub> was separately placed in muffle furnace for preheating at 200  $\,^{0}C$  for 10 mins. The stirring was done at 450 rpm with the continuous addition of TiB<sub>2</sub> through the pipe arrangement and stirring was continued for 10 mins. Mix was poured to the preheated and lubricated mould; ZnSt<sub>2</sub> was injected to the crucible at the time of pouring and provided the stirring only in the 30 Seconds. The volume % of reinforcement in different samples is shown in the Table 1.The fabricated samples are shown in Fig 4(d)





(b)



Fig. 4 Details of stir casting (a) stir casting machine (b) prehaeting of reinforcements in muffle furnace (c) mould (d) casted Samples

Table 1 Volume % of different reinforcement

Sample	%AA6061	%TiB <sub>2</sub>	%ZnSt <sub>2</sub>
1	100	0.0	0.0
2	95	2.5	7.5
3	95	5.0	5.0

## 2.3 Characterization

2.3.1 Scanning electron microscope with EDS

The SEM and EDS characterization of specimens were examined directly by SEM with EDS attachment (Model JEOL JSM-IT 100). SEM was used to observe the surfaces and observe the proper dispersion of reinforcement in the composite. Two samples were cut from the casted piece using wire cut EDM. The surface of the samples were clean by emery paper of different grit size varies from 100 to 330 to give good surface finish. Finally specimens were dipped in ethanol in order to get the clear surface and the dipped samples were dried in normal atmospheric conditions.

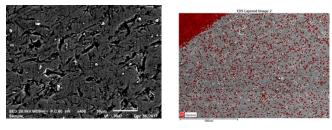
#### 2.4 Hardness testing

Specimen hardness was checked using the Mitutoyo HM 100 series with a test force range 0.1 to 1 kgf as per the ASTM E-384 standard. The diamond type indenter was used to produce indentation on the material. The specimens for hardness tests were prepared from casted rod usi.ng wire cut EDM and followed by diamond polishing.

## 3. RESULTS AND DISCUSSIONS

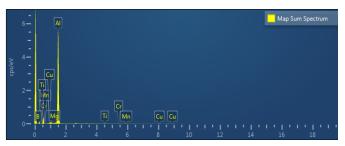
# 3.1 Characterization of casted samples

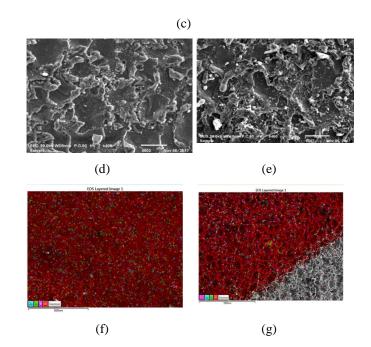
SEM images of hybrid composite confirm that there is a proper distribution of reinforcement material. Fig. 5(a) is the SEM image of pure AA6061 which shows less porosity and less oxide formation. Fig. 5(b) shows the EDS image of pure AA6061 which confirm the major alloying elements Cu, Si, Mn, Fe, Ni, Zn, Ti, Pb, Sn. Fig 5(c) shows the SEM image of Al6061/5%TiB<sub>2</sub>/5%ZnSt<sub>2</sub> Fig 5 (d) shows the EDS image of Al6061/5%TiB<sub>2</sub>/5%ZnSt<sub>2</sub> which confirms the various elements in composite like Mn,Cu,MgTi,Cr and B. The presence of boron confirms the dispersion of B in the composite. Fig 5(e) shows the SEM image of Al6061/2.5%TiB<sub>2</sub>/7.5%ZnSt<sub>2</sub>. Fig. 5(h) is the EDS analysis of AA6061/2.5%TiB<sub>2</sub>/7.5%ZnSt<sub>2</sub>. Fig. 5(h) is the elements like B, Mn, Mg, Ti, and Cr.



(a)

(b)





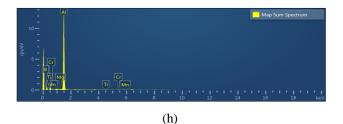


Fig. 5 (a) SEM of pure Al6061(b) EDS of pure Al6061(c) EDS image with 5 TiB<sub>2</sub> and 5 ZnSt<sub>2</sub>(d) SEM image with 5% TiB<sub>2</sub> and 5% ZnSt<sub>2</sub>(e) SEM image with 2.5% TiB<sub>2</sub> and 7.5% ZnSt<sub>2</sub>(f) Layered EDS image with 5% TiB<sub>2</sub> and 5 ZnSt<sub>2</sub>(g) Layered EDS image with 2.5% TiB<sub>2</sub> and 7.5% ZnSt<sub>2</sub> (h) EDS image with 2.5 TiB<sub>2</sub> and 7.5 ZnSt<sub>2</sub>.

#### 3.2 Effect on hardness

Hardness of the composite was measured by using Mitutoyo HM series as per ASTM E-384. The test was carried out with a load of 0.2 kg with 15 sec dwell time at normal atmospheric conditions. The test was carried out at three different locations. The results of hardness test conducted in AA 6061 and the hybrid composite containing different vol. % of TiB2 and ZnSt2 reveals that there is significant improvement in the hardness with the addition of TiB2 reinforcement. Hardness of composite increases due to the addition of TiB<sub>2</sub> particles. Also TiB<sub>2</sub> is acting as an obstacle to the moment of dislocation causing increase in hardness [18].Increase in value of hardness of composite can be due to poor heat expansion factor of ceramic particles while comparing with AA6061alloy by producing a lot of mismatches at the boundary of reinforcement and matrix [19].Hamid Abdulhagg et al. also claimed that addition of rigid ceramic particles increases the bulk hardness of AA [20].

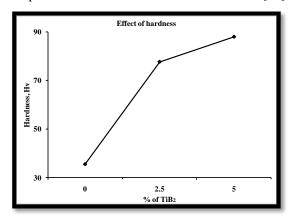


Fig. 6 Effect of hardness

#### 4. CONCLUSIONS

• The AA 6061/TiB<sub>2</sub> with ZnSt<sub>2</sub> was successfully faricated by liquid mettalurgy stir casting route.

- The SEM and EDS results confirmed that there is proper mixing of matrix material and reinforcement material.
- Hardness of the hybrid composite is higher than the base material. This is due to the addition of hard reinforcement.
- Hardness increases with the increasing the vol% of reinforcements.
- EDS result confirm the presence of various elements in the hybrid composite.

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