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Fabrication and Study of Titanium Diboride Powder and Aluminium Titanium Alloy Composite

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Abstract Aluminium alloy Metal Matrix Composites (MMCs) are gaining wide spread acceptance for automobile, industrial, and aerospace applications because of their low density, high strength and good structural rigidity. In this study aluminum alloy (Al-7Si)/Titanium Diboride (60% pure) reinforced metal-matrix composites (MMCs) are fabricated by melt-stirring technique in two ways; direct cast method and cooling slope method. The Titanium Diboride (TiB_2) powder used in the fabrication of the composite was prepared by heating a mixture of Titanium dioxide (TiO_2), Boron Trioxide (B_2O_3) and charcoal in their stochiometric ratios in an extended arc thermal plasma reactor using graphite electro de in presence of flowing argon atmosphere. Microstructure and hardness of aluminium alloy (Al-7Si), direct cast composite (Al-7Si-5TiB_2) and composite (Al-7Si-5TiB_2) cast through cooling slope method were studied. It was observed that TiB_2 grains are finer and spheroid in shape in the composite casted through cooling slope method which in turn increases the hardness.

Keywords: Matrix Composite, Vicker's Hardness Test, TiB_2

I. Introduction

Advent of new technology in the field of material's engineering has always provided a possibility for development of new materials from the existing entities. The need for different mechanical properties in a single entity has called for new techniques of production in the field of metallurgical sciences. Metals have played a significant role from the time since the Iron Age. Initially, the ferrous metals were used extensively for day to day purposes. With the advent of economical processes for processing non-ferrous metals from the ores, their use has gained prominent significance in many fields. Aluminum, a non-ferrous metal thus was widely used for day to day purposes. In the recent material developments a set of new materials called composites have been widely used especially in the field of engineering. The basic idea behind their fabrication is the use of different constituent materials which can be combined to get a new material which possesses the characteristics of both of the constituent materials without losing their physical identity. Composites offer the flexibility in choosing the constituent materials as per the need and the cost involved in processing the same.

The fabrication techniques for composites have been widely investigated and previously researchers have established the techniques for fabrication. There is no hard and fast rule that these are the only techniques which can be applied for preparation of composites, as, this field is still wide open where there is a huge possibility for emergence of new techniques which can be an improvement over the existing one or which can effectively produce the composite on the economic basis. The whole idea behind this process of research is to improve the properties of the composites and also to test the suitability for mass production.

Composite material is a material composed of two or more distinct phases (matrix phase and dispersed phase) and having bulk properties significantly different from those of any of the constituents.

1) Matrix phase:

The primary phase, having a continuous character, is called matrix. Matrix is usually more ductile and less hard phase. It holds the dispersed phase and shares a load with it.

2) Dispersed (reinforcing) phase:

The second phase (or phases) is embedded in the matrix in a discontinuous form. This secondary phase is called dispersed phase. Dispersed phase is usually stronger than the matrix, therefore it is sometimes called reinforcing phase. There are two classification systems of composite materials. One of them is based on the matrix material (metal, ceramic, polymer) and the second is based on the material structure.

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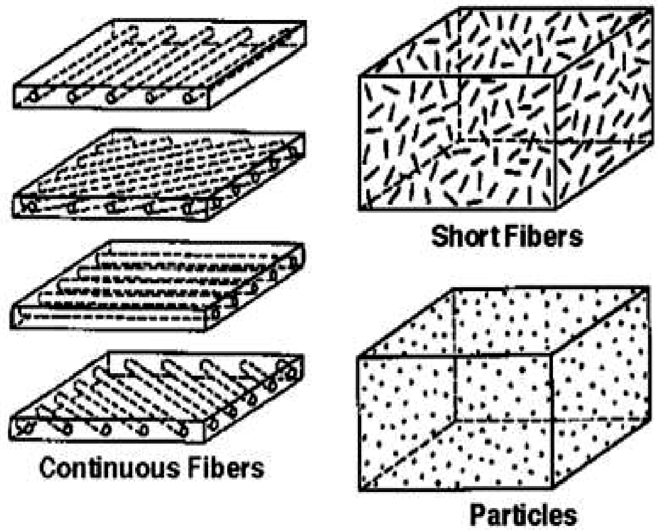

Fig 1: Types of particulate Reinforcement

i. Classification of composites I
(Based on matrix material)
Metal Matrix Composites (MMC)
Metal Matrix Composites are composed of a metallic matrix (aluminum, magnesium, iron, cobalt, copper) and a dispersed ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) phase.
Ceramic Matrix Composites (CMC)
Ceramic Matrix Composites are composed of a ceramic matrix and embedded fibers of other ceramic material (dispersed phase).
Polymer Matrix Composites (PMC)
Polymer Matrix Composites are composed of a matrix from thermoset (Unsaturated Polyester (UP), Epoxiy (EP)) or thermoplastic (Polycarbonate (PC), Polyvinylchloride, Nylon, Polysterene) and embedded glass, carbon, steel or Kevlar fibers (dispersed phase).

ii. Classification of composite materials II
(Based on reinforcing material structure)
Particulate Composites
Particulate Composites consist of a matrix reinforced by a dispersed phase in form of particles.
Fibrous Composites
Short-fiber reinforced composites.
Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length<100*diameter).

Long-fiber reinforced composites. Long-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of continuous fibers.

3) Laminate Composites

When a fiber reinforced composite consists of several layers with different fiber orientations, it is called multilayer (angle-ply) composite.

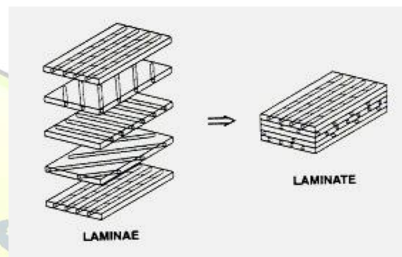

Fig 2: Types of laminar Reinforcement

A. METAL MATRIX COMPOSITES
Metal Matrix Composite (MMC) is a material consisting of a metallic matrix combined with a ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) dispersed space.
Aluminium Matrix Composites (AMC)
This is the widest group of Metal Matrix Composites.
Matrices of Aluminium Matrix Composites are usually based on aluminum-silicon (Al-Si) alloys and on the alloys of 2xxx and 6xxx series. Aluminium Matrix Composite (AMC) is reinforced by:

- Alumina ($\mathbf{Al_2O_3}$) or silicon carbide (**SiC**) particles (particulate Composites) in amounts 15-70 vol%;
- Continuous fibers of alumina, silicon carbide, Graphite (long-fiber reinforced composites);
- Discontinuous fibers of alumina (short-fiber reinforced composites);

The following properties are typical for Aluminium Matrix Composites:

- High strength even at elevated temperatures
- High stiffness (modulus of elasticity)
- Low density
- High thermal conductivity
- Excellent abrasion resistance.

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Aluminium Matrix Composites (AMC) are used for manufacturing automotive parts (pistons, pushrods, brake components), brake rotors for high speed trains, bicycles, golf clubs, electronic substrates, cores for high voltage electrical cables.

A. COOLING SLOPE

The method involves pouring of the melt from top, down an oblique and channel shaped plate cooled from bottom by counter flowing water. The melt, while flowing down, partially solidifies and forms columnar dendrites on plate wall. These dendrites are broken into equiaxed grains and are washed away with melt. The melt, together with the equiaxed grains, forms semisolid slurry collected at the slope exit and cast into billets having non-dendritic microstructure. The final microstructure depends on several process parameters such as slope angle, slope length, pouring superheat, and cooling rate.

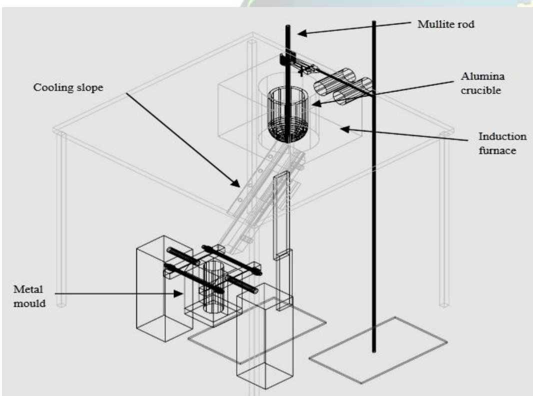

Fig. 3: Schematic Diagram of casting using Cooling Slope

B. *MICROHARDNESS*

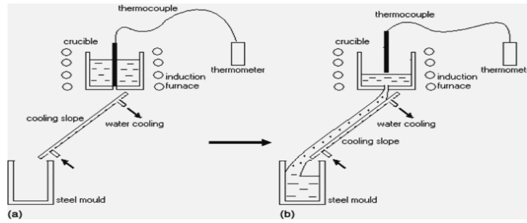

Fig. 4: Cooling slope arrangement in a casting process

Microhardness testing of metals, ceramics, and composites is useful for a variety of applications for which 'macro' hardness measurements are unsuitable: testing very thin materials like foils, measuring individual microstructures within a larger matrix, or measuring the hardness gradients of a part along the cross section. Microindentation hardness testing (or microhardness testing) is a method for measuring the hardness of a material on a microscopic scale. Microhardness testing per ASTM E-384 gives an allowable range of loads for testing with a diamond indenter; the resulting indentation is measured and converted to a hardness value. The actual indenters used are Vickers (more common; a square base diamond pyramid with an apical angle of 136° as shown in Fig.5) or Knoop (a narrow rhombus shaped indenter). The result for either Vickers or Knoop microhardness is reported in kg/cm^2 and is proportional to the load divided by the square of the diagonal of the indentation measured from the test. Vickers hardness is also sometimes called Diamond Pyramid Hardness (DPH) owing to the shape of the indenter. The test samples should have a smooth surface and be held perpendicular to the indenter. All things being equal, a lighter indenter load will require a smoother surface for a satisfactory test. Samples are usually mounted in plastic to fix them during preparation and testing. The hardness values obtained are useful indicators of a material's properties and expected service behavior.

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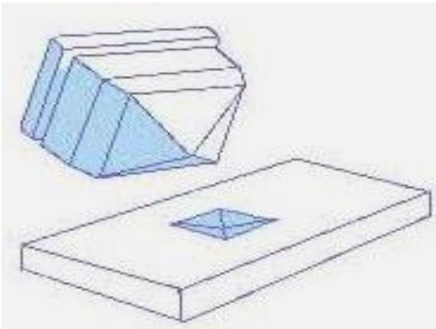

Fig 5: Schematic diagram of the square base diamond pyramid Vickers hardness indenter and sample indentation.

C. WEAR

Wear is progressive damage to a surface caused by relative motion with respect to another substance. One key point is that wear is damage and it is not limited to loss of material from a surface. However, loss of material is definitely one way in which a part can experience wear. Surfaces may look smooth, but on a microscopic scale they are rough. When two surfaces are pressed together, contact is made at the peaks of the roughness or asperities as shown in . The real area of contact can be much less than the apparent or nominal area. At the points of intimate contact, adhesion, or even local welding, can take place. If we want to slide one surface over the other then we have to apply a force to break those junctions.

The definition of wear includes contact situations involving sliding, rolling, and impact between solid bodies, as well as contact situations between a solid surface and a moving fluid or a stream of liquid or solid particles. The wear in these latter situations is normally referred to as some form of erosion, such as capitation, slurry, or solid particle erosion.

If one surface is slid over another then the asperities come into contact and there is a possibility that wear can occur. The breaking of all these little junctions can cause material removal (adhesive wear), or the asperities of a hard surface can plough grooves in a soft surface (abrasive wear). Wear is usually unwelcome; it leads to increased clearances between moving components, increased mechanical loading and maybe even fatigue. But in grinding and polishing processes the generation of high wear rates is desirable. As well as adhesive and abrasive wear, there are other mechanisms whereby material can be removed from a surface.

Erosive wear occurs when particles (or even water droplets) strike a surface and break off a bit of the material. Hard particles can become trapped in contacts and cause material to be removed from one or both of the surfaces.

When a ductile material is abraded by a blunt grit, then cutting is unlikely and the worn surface is repeatedly deformed . In this case wear debris is the result of metal fatigue.

II. MATERIALS AND METHODS

A.WHY A356 ?

In the present fabrication process of composite we have selected A356 as the matrix element which has the following thermophysical properties and composition as shown in Table1.

Alloy A356 has greater elongation, higher strength and considerably higher ductility than the base metal. This alloy has good resistance to most forms of corrosion.

Typically this alloy is used in castings for aircraft parts, pump housings, impellers, high velocity blowers and structural castings where high strength is required. It can also be used as a substitute for aluminum alloy 6061.

TABLE 1.
THERMOPHYSICAL PROPERTIES AND COMPOSITION OF A356

A356 aluminum alloy	
Solidus Temperature	831 K
Liquidus Temperature	876 K
Latent Heat	429 J/g
Density at melting point	2.362 g/cm^3
Composition	Si = 6.9% Fe=0.08% Mg=0.34% Ti=0.013% B=0% Sr=0%

B. Why TITANIUM DIBORIDE ?

Titanium diboride is a fully dense, hot pressed, electrically conductive ceramic with exceptional hardness. Properties of TiB_2 reveal that the compound is a good choice as reinforcement. Titanium diboride has high wear resistance and high strength at elevated temperatures. The high density, combined with the high elastic modulus and high compressive strength, have lead to its use in armour components. It is resistant to most chemical reagents, and has excellent wettability and stability in liquid metals such as Aluminium and Zinc. This, combined with its high electric conductivity, has lead to its use in Hall- Héroult cells

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for Aluminium production. It has found uses as crucibles for molten metal. It is an attractive material for the aluminium industry, used as an inoculant to refine the grain size when casting aluminium alloys, because of its good wettability and low solubility in molten aluminium and good electrical conductivity.

Table 2
Properties of Titanium diboride

Property	
Density (g.cm^{-3})	4.52
Melting Point (°C)	2970
Modulus of Rupture (MPa)	410-448
Hardness (Knoop)	1800
Elastic modulus (GPa)	510 -575
Poisson's Ratio	0.1 - 0.15
Volume resistivity (ohm.cm) at 20°C	15x10^{-6}
Thermal conductivity (W/m.K)	25

i. Preparation of TiB_2

Preparation was carried out using a mixture of Titanium dioxide (TiO_2), Boron Trioxide (B_2O_3) and charcoal in their stochiometric ratios. Heating of the mixture was done in extended arc thermal plasma reactor using graphite electrode in presence of flowing Argon atmosphere.

ii. FABRICATION OF Al-7Si-TiB_2

A356 (Alloy of Aluminium) was selected as the matrix element. TiB_2 (60% Pure) was selected as the reinforcement component.

The following steps elucidate the process of fabrication of the composite:

Step I: The Titanium diboride was prepared by carbothermal reduction using thermal plasma technique.

Step II: The solid mass of TiB_2 was powdered to the required particle size using the Ball Mill as shown in Fig 6.

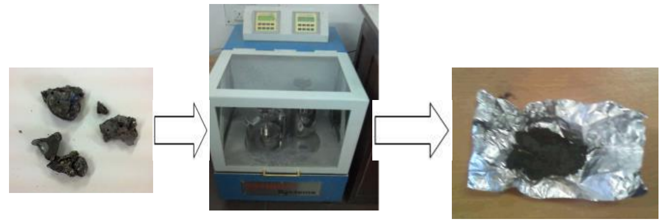

Fig.6:Powdered form of TiB_2 after Ball Milling

Step III: The **charge calculations** were made as follows:

For 95gm of Al-7Si we will add 5gm of TiB_2 powder Such that, the weight percentage of the constituents will be,

Weight Percent of TiB_2 = 5%
Weight Percent of A356 = 95%

Accordingly, we took 247gms of Al-7Si alloy and added 12.5gms of TiB_2 powder to it for fabricating the composite.

Step IV:

The cover flux is a mixture of
NaCl = 45%
KCl= 45%
NaF= 10%

It was sprinkled inside the graphite crucible before placing the alloy into it. The cover flux provides a physical barrier to oxidation of the melt.

Step V:

The temperature of the furnace [THERELEK] was set up to 760˚C as shown in Fig. 7. According to the charge calculations the alloy (A356) was placed in the graphite crucible and the graphite crucible was placed inside the furnace chamber.

Fig. 7: Furnace [THERELEK]

When the temperature of the furnace reached the mark, the graphite crucible was taken out and Degasser (C_2Cl_6) was added.

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Then, the crucible was again placed inside the furnace for heating. When the temperature of the furnace came up to the mark, the crucible was taken out and the TiB_2 powder was added to the melt. Simultaneously, stirring was done using a Zirconia coated graphite rod. The stirring continued for a minute and the mixture was placed back in the furnace.
When the temperature reached up to the mark, the melt was taken out and Grain Refiner (Al-5Ti-1B) and successively Grain Modifier (Al-10Sr) in were added and the melt was stirred for few seconds and was kept back in the furnace.
Step VI:
At this point the hot melt was casted using the following two methods:
i. Direct Pouring of the Melt into the Mould.

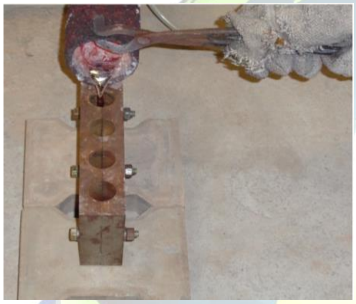

Fig.8: Direct pouring of melt

ii. Melt poured through the Mild Steel Cooling Slope into the Mould.

Fig. 9: Mild Steel Cooling Slope

The cooling slope as shown in Fig. 9 was placed at an angle of 45˚ with the horizontal. The total slope length was subdivided over entire length starting from the bottom point of the slope. For the present experimental purpose, the melt was poured at the mark of 300.

Step VII:
The solidified casting (composite) as shown in Fig.10 is taken out from the mild steel mould.

Fig. 10: Al-7Si-5TiB_2 as cast

The cast products were further used for material characterization and for mechanical testing purposes.

IV. MATERIAL CHARACTERIZATION

The microstructure evaluation of any material forms the basic step before evaluating its properties. It is a scenario in which we are able to see the phase composition of the constituent elements. It also indicates the structure of the grain which is crucial for evaluation of the properties.
For evaluation of the microstructure, the composite and alloy specimen were non-conventionally machined (Using WEDM) to the required size of
Diameter=12mm
Height =15mm

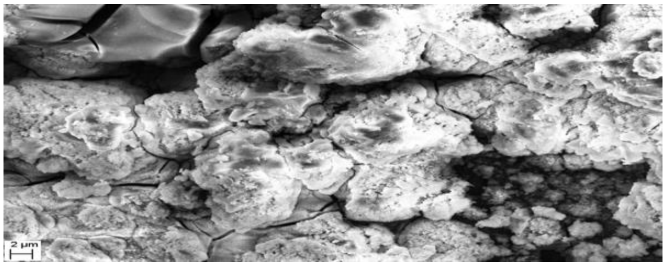

Fig. 11

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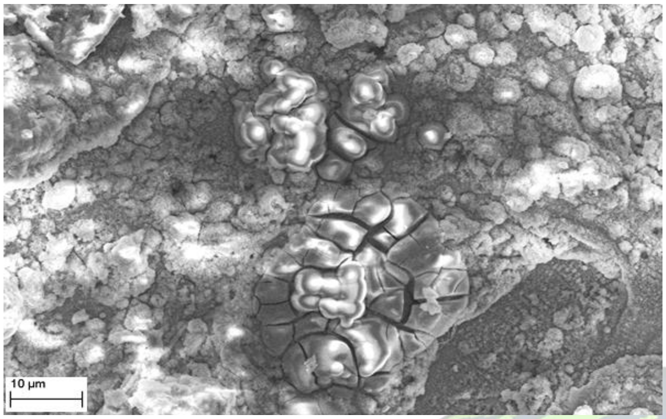

Fig. 12: Al-7Si-5TiB_2 directly cast [3250X] (Without Etching)

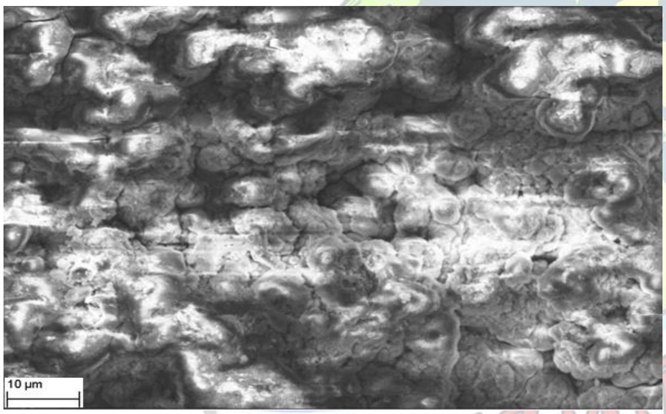

Fig. 13: Al-7Si-5TiB_2 cast through Cooling Slope [3250X] (Without Etching)

The specimens were then etched using Keller's Reagent.

Table 3: Composition of Keller's Reagent

Compound	Quantity [ml]
Water	95
Nitric Acid [HNO_3]	2.5
Hydrochloric Acid [HCl]	1.5
Hydrofluoric Acid [HF]	1

After etching the specimen, the specimen was lightly cleansed with water. Then the specimen was observed through the microscope of the Micro-hardness test machine. The obtained figures are as shown in Fig. 14, Fig.15 and Fig. 16. From the figures it is inferred that the grain size [Fig. 14 and Fig. 15] of the alloy/composite cast directly is bigger than the one casted using Cooling Slope [Fig. 16]. Also, the grains are packed closely as shown in Fig. 16 which was cast using Cooling Slope.

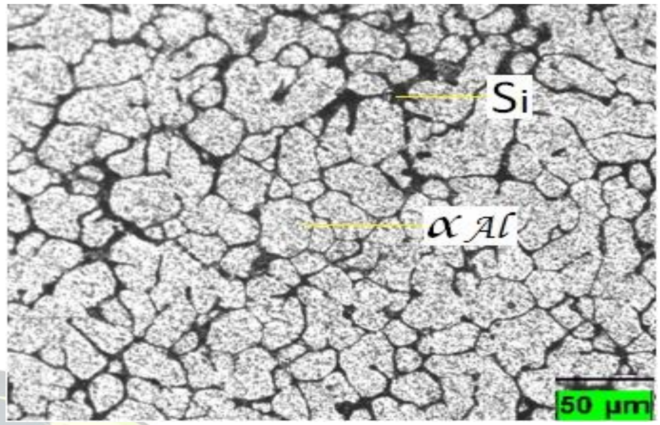

Fig. 14: Al-7Si poured directly into mould (With Etching)

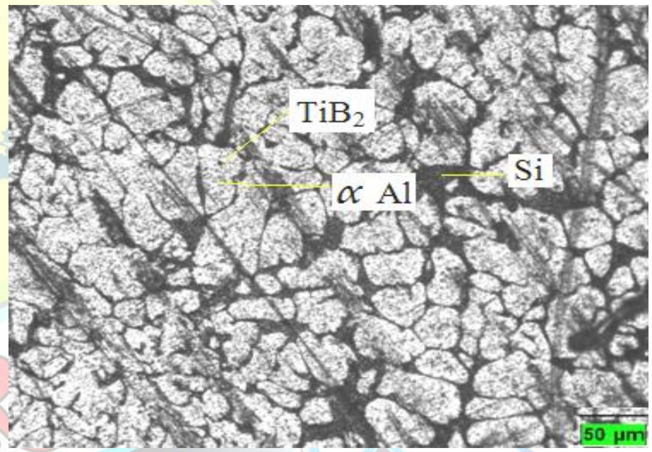

Fig. 15: Al-7Si-5TiB2 poured directly into mould (With Etching)

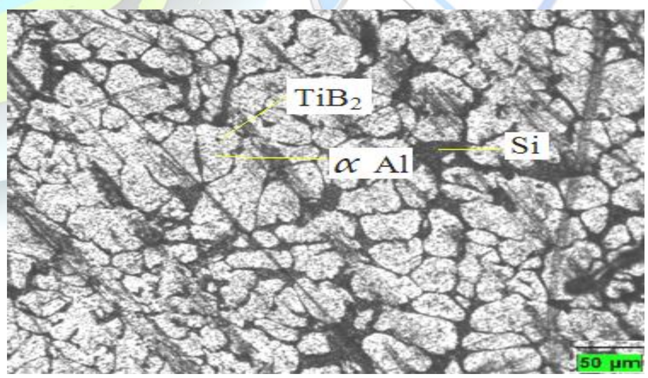

Fig 16: Al-7Si-5TiB_2 cast through Cooling Slope (With Etching)

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V. VICKERS HARDNESS TEST

The Vickers hardness test was performed to determine the micro-hardness of the specimen on the micro scale.
The following steps were adopted for the same:
Step 1:The as cast composite block and the Alloy block was machined to get the required shape and size of the specimen for performing the hardness test.
Step 2:The machined specimen having the Ø12mm and a length of 15mm were thoroughly polished for getting a flat surface.
Step 3: Then the specimen was placed on the slot and was scanned at 10X for a clear image of the surface as shown in

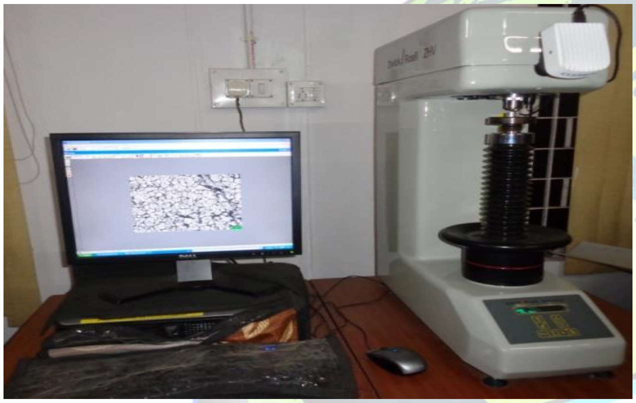


Fig. 17: Vickers Hardness Tester [ZHV30, Zwick Roell]

The indentation was done at the required spot for both alloy and composite.Once indentation was complete the indented region was selected and the hardness value was generated by the computer as shown in Fig 18.

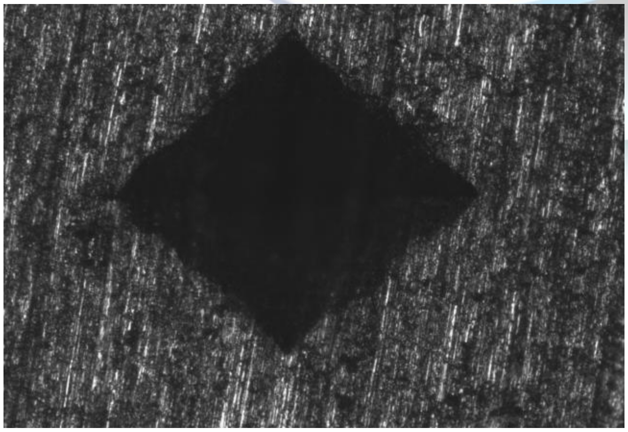
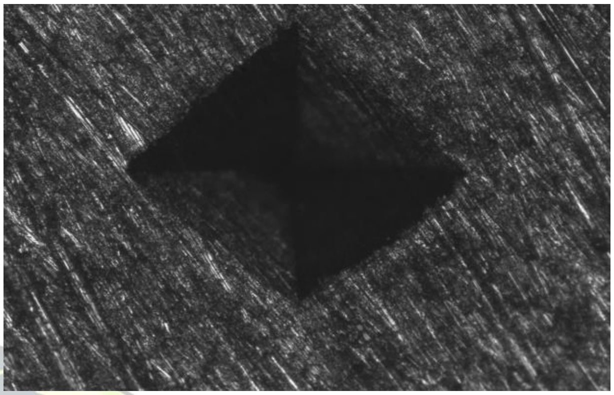


Fig. 18: a) Indentation on A356 surface (Without Etching)



b) Indentation on surface of composite (Without Etching)

VI. RESULTS AND DISCUSSION

The results of the Vickers hardness values which were obtained for both alloy and composite at different regions over the surface.
The Load applied by the indenter for each Indentation = 5Kgf.
There is an increase in micro-hardness value for the composite than the alloy which is attributed to the addition of TiB_2 particles. Further, the hardness value for the composite casted using the cooling slope method showed much higher hardness value than the composite which was casted directly. This is because the cooling slope decreases the time for solidification as it basically provides a uniform cooling surface for the melt when poured on to it. Hence, the

VII. CONCLUSION

The present experimental work has been successfully carried out. The basic objective of fabricating a material (Composite) possessing good mechanical (Hardness) properties than the base material has been demonstrated with results.
It can be concluded as :
A. The preparation of Al-7Si/5TiB_2 by the casting process using manual stirring gave a good end product.
B. The addition of TiB_2 particles to the alloy A356 gave rise to better wear resisting characteristics.
C. The hardness of the composite got improved due to the presence of TiB_2 particles in the matrix.
D. The cooling slope method of casting gives an improvisation in the material characterization to that of the

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conventional method of direct pouring. For semi-solid processing this method is aptly suitable.

E. The preparation of TiB_2 by carbothermal reduction using thermal plasma technique infuses the idea of fabrication of the same from an economical point of view.

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