

**Influence of Particle Additives on the
Lubricated Tribology of Aluminium Metal
Matrix Composite**

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by

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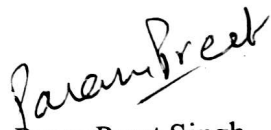
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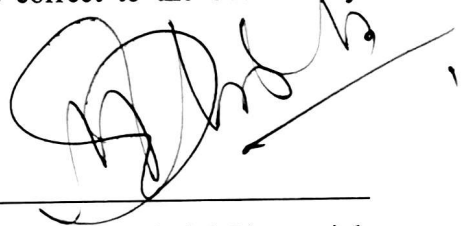
CERTIFICATE

I hereby declare that the thesis entitled “**Influence of Particle Additives on the Lubricated Tribology of Aluminium Metal Matrix Composites**” is an authentic record of my study carried out as requirements for the award of the degree of **Master of Engineering in Production Engineering** at **Thapar University, Patiala** under the supervision of **Dr. Hiralal Bhowmick, Assistant Professor, Mechanical Engineering Department, Thapar University, Patiala** during July, 2016 to July, 2017. The matter embodied in this report has not been submitted to any other university or institute for the award of any degree.

Date: 1/8/2017


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It is certified that the above statement made by the student is correct to the best of my knowledge and belief.



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Dedicated to
My parents

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ABSTRACT

Aluminium composite because of their improved mechanical properties, low density, better corrosion resistance and low production cost are very much suitable for various applications in the field of automotive, aerospace and marine industries. From the past and till the present scenario, it is evident that the research work on aluminium metal matrix composites (AMMC) is primarily focused on the fabrication and mechanical characterization. The potential tribological behaviors of these composites are mainly evaluated through dry sliding friction and wear testing. In this research work, in addition to the dry sliding test, an attempt has been made to investigate the effect of oil and oil additives on the sliding wear and friction behavior of aluminium composite. Three different types of composites were fabricated by stir casting method and their mechanical and metallurgical properties are investigated. Hardness characterization of the composites is done to evaluate the interfacial bonding between reinforcement and matrix. Metallurgical properties are studied using SEM and EDS to evaluate the distribution of particles and probabilities of defects. Then these composites are examined for their tribological characteristics under dry and lubricated contact with steel. The most tribologically favored pair is then subjected to the particle based lubrication for further enhancement of tribological response against the selected counterpart. The friction and wear property of the fabricated composites sliding against EN31 disk are investigated under dry, lubricated base oil SN 500 but without additive and lubricated with base oil in the presence of particle additives such as boric acid, MWCNT and MoS₂.

The outcome of this thesis works shows that there is a significant influence of suitably selected particle based additives on the wet tribology of composite. Starting with various types of composite-steel tribopairs and dry sliding condition, the present work, tries to design a tribo-material package for an improved tribological response under the given operating condition. From our study CNT and boric acid emerged out as potential additives for the tribological applications of Al-B₄C composites.

Key words: AMMC, Stir Casting, Additives, Wet tribology, Characterization.

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ACRONYMS

B ₄ C	= Boron Carbide
SiC	= Silicon Carbide
TiC	= Titanium Carbide
Al ₂ O ₃	= Alumina
VI	= Viscosity Index
PAO	= Polyalphaolfiens
MoS ₂	= Molybednum Disulphide
WS ₂	= Tungsten Disulphide
MWCNT	= Multi-Walled Carbon Nano Tube
API	= American Petroleum Institute
AMMC	= Aluminium Metal Matrix Composite
ASTM	= American Society for Testing and Materials
AlN	= Aluminium Nitride
μm	= Micron Meter
μ	= Coefficient of Friction
FESEM	= Field Emission Scanning Electron Microscope
SAE	= Society of Automotive Engineers
SEM	= Scanning Electron Microscope
EDS	= Energy Dispersive Spectroscopy
XRD	= X-Ray Diffraction
MMC	= Metal Matrix Composite
WC	= Tungsten Carbide
AlB ₂	= Aluminium Borate
MML	= Mechanically-Mixed Layer

Chapter 1

INTRODUCTION

1.1 COMPOSITES

Composites are the materials which are composed of two or more materials and have chemically distinct phases. The composites are heterogeneous at microscopic scale but relatively homogeneous at macroscopic scale [1]. Usually alloys do not retain their properties after mixing where as in composites materials they retain their original properties [1]. The usage of composites in various applications and industries has just become a fashion because of their superior thermal, mechanical, electrical as well as physical properties.

1.2 ALUMINIUM METAL MATRIX COMPOSITE

1.2.1 MATRIX

Aluminum alloy possesses excellent properties like high strength-to-weight ratio, corrosion resistance, good ductility, low density, high thermal conductivity, and so forth, thus making it suitable for wide range of applications in several industries like automobile, aerospace, structural applications. The uses of high strength aluminum alloys are focused on structural applications in aerospace as well as on general engineering sectors. However, aluminium alloys have not been well known for high performance tribological application because of their low hardness, poor wear resistance, and poor weldability [2].

1.2.2 REINFORCEMENT

There are various particles available which are used for reinforcement such as B_4C , SiC, TiC, B, C, and Al_2O_3 . With the reinforcement of high modulus ceramics particulates such as silicon carbide (SiC) having properties like more stability and chemical compatibility with aluminum matrix and low cost, wide range of available grade aluminum alloys are suitable candidate for wider spectrum of applications. The hard reinforcement of silicon carbide particle improves the properties like hardness and wear resistance of composite by acting as load bearing components. However, it is important that reinforcement of particles should be homogeneous to achieve the improved properties of composites [2].

1.3 FABRICATION OF COMPOSITES

Stir casting is the simplest, liquid state method and is cost effective to fabricate metal matrix composite. To avoid moisture from the particles, preheating of the reinforced particle should be done; otherwise, there is a chance of agglomeration of particulate which occurs due to moisture and gasses present in the particles. Degassing agent (hexachloroethane and magnesium) is used to reduce the gas porosities. The addition of magnesium also leads to reduced solidification shrinkage and reduced tendency towards hot tearing. Besides, it helps in the suppression of segregation, or agglomeration of reinforcements, and makes the process cycles faster. The stirrer should be immersed to about two-thirds of the depth of molten metal which is then poured into a preheated permanent mould. This is generally followed by the curing of the sample and machining of the cast samples to achieve the final surface [2].



Figure 1.1 Stir Casting Furnace [Photo courtesy: Thapar university campus, Patiala]

1.4 INTRODUCTION TO TRIBOLOGY

Tribology word came into existence from the word “tribos” which means rubbing. Tribology is that part of engineering that shares the concepts of friction, lubrication, and wear between two contacting pairs and thus knowledge of tribology helps to improve service life of interacting machine components which in turn yields substantial economic benefits. Tribology is a vast and interdisciplinary subject dealing with the underlying physics of contacting surface and adhesion to the application of advanced materials and lubricants to solve the problems of friction and wear associated with industrial application [3].

1.5 LUBRICATION

Lubrication is a process in which friction and wear rates of the contact surfaces that are in relative motion are reduced by using appropriate lubricant. The lubricant decreases adhesion component of friction as compared to abrasion component of friction. The required lubricant properties are specific to applications and also perform functions like cooling, cleaning, suspending, protection and transfer of power. Some of the examples are cylinder liners and piston rings, journal bearings, etc [3].

1.6 MODES OF LUBRICATION

When the surfaces are completely separated by the lubricant film and the influence from asperities is negligible then it is called as thick lubrication. When the lubricating film is unable to separate the sliding surfaces and asperities on surfaces begin to interfere then it is called as thin lubrication [3].

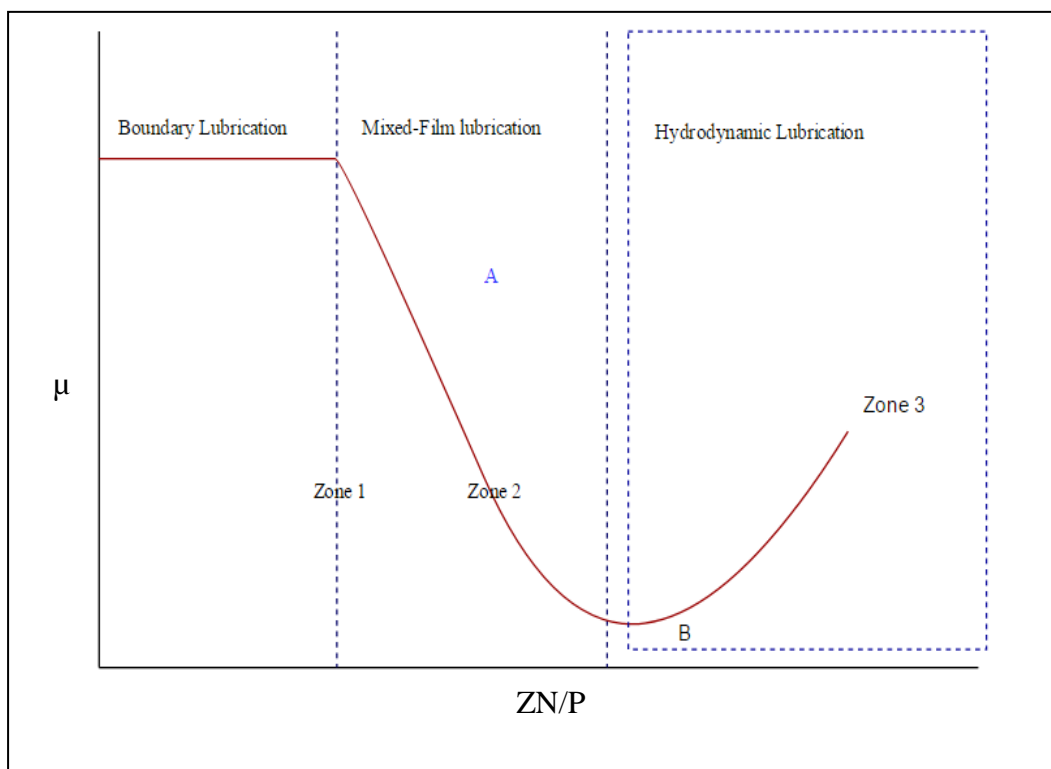


Figure 1.2 Lubrication regimes [W1]

In hydrodynamic or full film lubrication direct metal to-metal contact does not happen during the steady state working and the load carrying contacting surfaces are separated by a

relatively thick film of lubricant. In this regime, the fluid pressure is self generated by the relative motion of the contacting surfaces and wedging action developed by the surfaces moving at sufficiently high speed [W2].

In partial or mixed lubrication regime the velocity is relatively low, the load or temperature is high enough to significantly reduce lubricant viscosity which leads to the frequent contacts of the largest asperities of the mating surfaces [W2].

Boundary lubrication regime is the condition when there is a negligible fluid film thickness and there is substantial asperity contact. In boundary lubrication, the chemical and physical properties of thin surface films are of great importance whilst the properties of the bulk lubricant are not as worthy [W2].

1.7 TYPES OF LIQUID LUBRICANTS

Mineral oil or base oil: Mineral oils are used to make products including lubricating greases, motor oil and metal processing fluids and are produced in very large quantities and available in light and heavy grades. These are manufactured by the refinement of crude oil by removing high molecular weight fractions so that wax deposition is prevented while lubricating, to slow down the decrease in oil viscosity and eliminating compounds with sulphur and nitrogen to prevent corrosion of wearing surfaces. It has a density of around 0.8 g/cm³. Products are differentiated according to their compositions and properties in the oil and most important factor is the viscosity of liquid. Some of the applications of mineral oil are hydraulic fluid in hydraulic machinery and vehicles and metal working lubricant and a cutting fluid. Mineral oil is also used as brake fluid in some cars and bicycle disc brakes. Mineral oil USP can be used as an antirust agent for blades [W1].

Classification of mineral oil on the basis of their chemical form is as follows [3]

- a) Paraffinic oils: The salient features of this type of oil is good resistance to oxidation and thermal stability, low volatility, high viscosity index (VI), flash point and pour point.
- b) Naphthenic oils: The characteristics of these oils are lower viscosity index, flash points and pour points, so good for low temperature applications. When burnt soft deposits are present leading to undesirable sludge type deposits. However, abrasive wear is low.
- c) Aromatic oils: These oils have very low viscosity index because of that they have very limited use and is recommended to extract oil components during refining of

mineral oil. The characteristics of these oils are low VI, high density, high volatility, low pour point, low oxidation stability, high thermal stability, and high sulphur contents.

As there are numerous crude oils used to produce base oils and the most common type is paraffinic crude oil and naphthenic crude oil and this crude oil creates products with better solubility and good properties at low temperature [3].

According to API these oils are classified into five groups; Group I oils are produced by solvent refining of conventional petroleum base oils (Saturates < 90% and/or sulphur > 0.03%, VI of 80 to 120). Group II oils are better grade of petroleum base oil and manufactured by hydro cracking (Saturates > 90% and sulphur < 0.03%, VI of 80 to 120). Group III oils are the best grade of petroleum base and produced by hydrocracking, which make these oils purer and this group is generally described as Synthetic Technology oils (Saturates > 90%, sulphur < 0.03%, VI over 120). Group IV consists of synthetic oils made of Polyalphaolefins (PAO) and are much more stable in extreme temperatures. Group V include oils which are not included in the above, such as naphthenic, Paraffinic, and Aromatic [W1].

Synthetic oil: These oils are used in extreme operating conditions where conventional oils cannot be used and situations such as ambient temp lesser than -120°F , rotational speed more than 60,000 rpm and temperature is higher than 500°F cannot be handled by conventional oils. These oils are specifically designed via uniformly shaped molecules with shorter carbon chains to resist heat and stress. Some of the other advantages of synthetic oils include: low rate of VI with temperature and rate of oxidation is much slower. Some of common oils in this category are polyglycols, esters, silicon, etc [3].

1.8 PARTICLE BASED LUBRICATION

1.8.1 PARTICLE ADDITIVES

There are various proven solid lubricants exist which can be used as the particle additives for particle based lubrication. Amongst these is graphite, MoS_2 , WS_2 , boric acid, MWCNT, etc. Graphite has long been used, however, due to its lack of wear resistance properties, nowadays, several alternative particles are being investigated for the improvement of tribological behaviour of the contact surfaces in the presence of these particles. Molybdenum disulphide (MoS_2) is one of such type of particles which exhibit extremely low coefficient of friction in dry environments [4]. Boric acid is another potential solid lubricant due to its lamellar

molecular structure, abundance in nature. The boric acid has low friction and shear strength and the physical characteristics are that it is white, bright, and water soluble acid [5].

1.8.2 NECESSITY AND APPLICATIONS OF PARTICLE BASED LUBRICATION

The easiest and one of the prime methods to lower the weightage of total energy consumption with increased load bearing capacity, in a mechanical system is particle based lubrication. Tribologists develop quest for improved lubrication characteristics of conventional lubricants because these lubricants cannot be tailored for specific rubbing contacts. The lubricants contain various additives to enhance the existing properties. But to counteract high speed, temperature and load bearing capacity, particle based lubrication is required. Hence, in addition to the improved load bearing capacity, smooth, durable and efficient running of mechanical systems are to be possible by using particle based lubrication. Some of the potential application of particle based lubrication applications are as follows:

Gears bear with high contact pressure and experience metal to metal contact at gear teeth. In these situations extreme pressure additives dispersed in oil is required. The I.C engine parts are subjected to high temperature wherein particle additives may help in delaying oxidation, to remove the by-products of combustion from the surface. The refrigeration system where low temperature that lubricants encounter, particle additives may help to lower the pour points.

Chapter 2

LITERATURE REVIEW

2.1 INTRODUCTION

The most conventional method of fabrication of aluminium metal matrix composite (AMMC) is carried out using stir casting method in which various processing parameters like temperature, holding time etc. are varied to understand their influence on distribution of particle in matrix and mechanical properties of composites. Many authors have provided the literature for mechanical characterization as well as for dry sliding behaviour using composites of which some of the significant studies in this context will be summarized here. However, a very few studies are found in the literature which take accounts of the lubricated sliding behaviours of composite, especially using particle based lubrication. In this chapter, after reviewing the relevant literatures, an attempt will be made to find out the potential research scope and necessities of the lubricated tribological studies for composites using particle additives.

2.2 COMPOSITE FABRICATION AND THEIR CHARACTERIZATION

Sozhamannan et al. [6] pointed out that conventional stir casting has some major problems associated with the process. Uneven distribution of particles and inadequate wetting of reinforced particles by metal leads to the heterogeneous dispersion of particles. They examined the cast products by microstructure analysis. It was concluded from their microstructure analysis that the uniform distribution of particles occurred in temperature from 750°C and 800°C. Their study also revealed the fact that the holding time influences the tensile and hardness value of the composite [6]. A review on stir casting and its parameters and manufacturing of hybrid composite were performed by **Bhandari et al. [7]** using aluminium as matrix and SiC and Al₂O₃ as reinforcement. Their results show that mechanical stirrer's blade should be 4 and the angle of blade should be 45° for uniform distribution of material. They also pointed out that maintaining the temperature at 630°C for Al 6061 will provide good wettability and hence reduce the porosity and therefore preheating the mould is essential which the moisture content for entrapment [7].

Swamy et al. [8] fabricated Al6061-SiC composites (Figure 2.1) using liquid metallurgy route. SiC with varying percentages of SiC (4wt%, 6wt%, 8wt% and 10wt %) was used as reinforcement to improve the hardness, tensile strength and wear resistance of the fabricated composite as compared to the base metals. The cast composites were treated with zing solution followed by quenching in different media. The natural and artificial ageing had been was also done of quenched samples. Micro hardness, tensile strength and wear resistance of both matrix and composites had been conducted before and after heat treatment. Heat treated composites shown improved micro hardness, tensile and wear resistance when compared with Al matrix alloy [8].

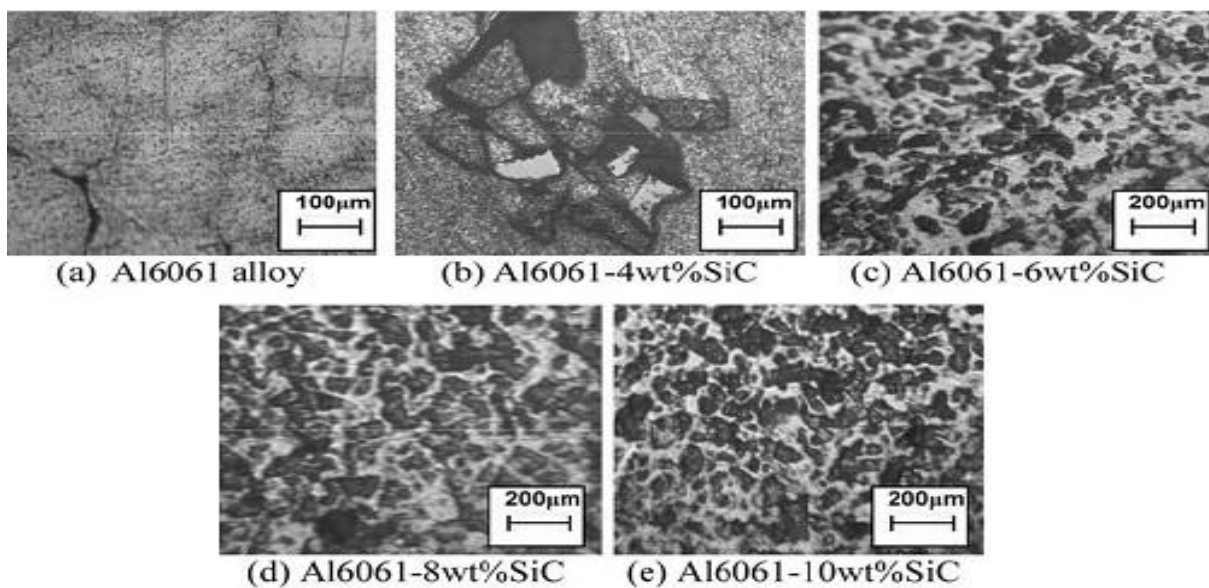


Figure 2.1 Optical microphotographs of base Al6061 and Al6061–SiC composites at 4, 6, 8 and 10 wt% SiC [8]

Kumar et al. [9] studied on Al6061-SiC and Al7075-Al₂O₃ composites and characterized for tensile strength, As per ASTM standard the sample was prepared from all the composites for hardness, tensile strength, wear and microstructural tests. The increasing percentage of reinforcement content in composites revealed that hardness and density of their composites increases. Optical microscope revealed that uniform distribution of reinforcement into the composites. According to rule of mixture for composites the experimental density value agreed with the theoretical density value of composites. It has been shown that Al6061-SiC reflects superior tensile strength than Al7075-Al₂O₃ composite [9]. **Prabu et al. [10]** have carried out their study aluminium reinforced with silicon carbide. The effect of stirring

speed and stirring times was examined to integrate 10% SiC in aluminium matrix. Further the study of micro structure and distribution of reinforcement is done by using the leica optical microscope and scanning electron microscope. The hardness was carried out on composites by Brinell hardness machine and results showed that hardness of composite are guided by the stirring speed and time. Microstructure explained that clustering or agglomeration of particle was more at lower stirring time with lower stirring speed. Therefore in rise in stirring speed and time lead towards the better distribution of particles and though the hardness of composite's affected by stirring speed and time. At 600 rpm and 10 minute stirring time the hardness of composites revealed is uniform. However, after certain stir speed the properties of composites degraded again [10].

Wahab et al. [11] have investigated the mechanical characterization of aluminium composites reinforced with aluminium nitride. Two types of mould used to cast the composites are graphite crucible and stainless steel permanent mould. Optical microscope was taken to judge the distribution of reinforcement and morphology of composites. The hardness of pure aluminium was recorded as 44 HV and further increased to 89 HV for the composite reinforced with 5 wt. % AlN powder. Therefore Hardness was increased with the increasing percentage of AlN particles into the matrix [11]. **Surendran et al. [12]** have used aluminium LM25 reinforced with alumina and prepared the composite from Die casting method. The compositions used are 0%, 1%, 1.5%, 2.5% and 5%. The tensile test, wear test, hardness test, impact test were carried out. The LM25 Al alloy with 5% of alumina showed the better characteristics as compared to the other composites [12].

2.3 DRY SLIDING WEAR BEHAVIOR OF AMMC

Ahmad et al. [13] investigated dry sliding wear behaviour of alumina particles reinforced with Aluminium. Wear rate was taken under dry sliding condition at room temperature with help of pin on disc type wear testing machine. The load of 25, 50, 75, 100 N were taken along with sliding speed of 250, 500, 750, 1000 rpm to calculate the wear rate. Thus, observed that percentage difference in weight loss of composite was approximately 52%. At lower load, for both the materials, coefficient of friction was almost same but at higher load percentage difference in weight loss decreased [13]. **Behera et al. [14]** focused on wear behaviour of three different composites reinforced with alumina, titanium dioxide and copper. The composites were fabricated with the help of Powder metallurgy technique by using increase in furnace temperature from 100°C to 700°C at rate of 3°C/min. It showed that

wear rates decreases with increase in sliding velocity for all composites. Alumina had shown the high wear as compared to other composites [14]. The wear test were carried out on Pin on disk wear testing apparatus by Surendran et al. [12] and results showed that composites with increase in percentage of reinforcement showed that there is decrease in wear rate. It was observed that composites with higher percentage of reinforcement showed higher hardness [12]. SiC contributes in improving the wear resistance of Al6061-SiC composites, which was reflected by the low wear rate of composites [8]. **Padmavati and Ramakrishnan [15]** focused on wear and friction behaviour of Al6061 reinforcement with various percentage of MWCNT and SiC reinforcement are prepared by stir casting and die casting. Pin on disc apparatus was used to perform wear test and seen that for all values of load there is an increase in coefficient of friction with percentage of MWCNT and decrease in specific wear rate. As hybrid ratio increases there is increase in hardness of composites [15].

Table 2.1 Coefficient of friction value for 3 samples[15]

COF Load (Kg)	Al-15%SiC	Al-15%SiC- 0.5%CNT	Al-15%SiC- 1%CNT
0.5	0.532	0.404	0.263
1	0.569	0.487	0.382
1.5	0.589	0.535	0.486

Prabhakar et al. [16] have casted aluminium-boron carbide composite to study the tribological behaviour for these composites. The aluminium reinforced with boron carbide 5wt% with particle size of 33µm and fabricated through stir casting. Wear testing was done on pin-on-disc tribometer. The process parameters taken in consideration such as load (10N, 20N and 30N), velocities (1m/s, 2m/s and 3m/s), and sliding distances (1000m, 1500m and 2000m). In context with the results the wear rate and coefficient of friction increases with increase in load and sliding distance and velocity showed inverse relation as compared to load [16]. **Monikandan et al. [17]** they have studied the hybrid composites that are fabricated with stir casting process and AA6061-10 wt. % B₄C–MoS₂ hybrid composites reinforced with 2.5, 5 and 7.5 wt. %. The mechanical and tribological properties were evaluated and microstructural of the composites showed the uniform distribution of reinforcement (B₄C and MoS₂) in the composite. Hardness and fracture toughness of the hybrid composites are decreased with an increase in the addition of MoS₂. Dry sliding studies

were performed on pin-on-disk revealed the existence of MoS₂-lubricated tribolayer on the worn pin surface which significantly influence the tribological properties. The involvement of MoS₂ particles decreased the friction coefficient and wear rate of the composite [17].

2.4 LUBRICATED SLIDING WEAR BEHAVIOR OF AMMC

A lot of research has been done on lubricated sliding for various materials. However, lubricated tribological behaviour of AMMC is studied in a very limited scope and yet to be explored for potential tribological applications.

Singh et al. [2] have studied the friction and wear behaviour of Al 7075 reinforced with SiC under sliding speed and load for both dry and lubricated condition. Fabrication of composite took place with the help of stir casting process and the experiments were performed on pin on disk tribometer. Composite have showed lower coefficient of friction and wear rates as compared with pure aluminium under dry and lubricated condition. During dry sliding condition coefficient of friction of pure aluminium and the composite decreases with increase in load, whereas it increases with increase in sliding speeds. On the other hand wear rates of both pure aluminium and the composites increase with increase in load as well as with sliding speeds. Wear mechanisms were explored by the FESEM images of worn surfaces. The value of COF was found to be minimum in case of lubricated condition in composite. The values of coefficient of friction and wear rates were minimum in case of both pure aluminium and composite under lubricated condition compared to dry condition. They also concluded that thin lubricating film formation between surface of specimen and rotating disk surface as it decreases the ploughing action and leads to decrease in breakage of small particles from aluminium alloy and composite, which in turn, lowered the wear rates in lubricated condition as compared with dry condition, as shown in Figure 2.2 [2].

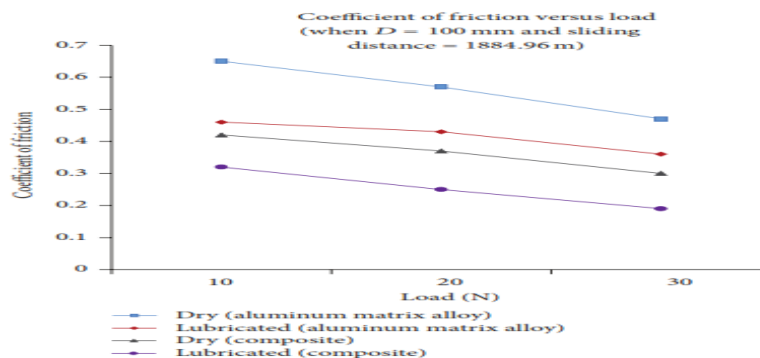


Figure 2.2 Graph of COF [2]

Martinez et al. [18] have studied the aluminium 6061 and reinforced with intermetallic particles such as Ti_3Al , TiB_2 and $TiAl$ and the comparison had been made between intermetallic particles. The composites were fabricated by P/M route and tribological properties were analyzed in dry sliding and lubrication under SAE 5W40. They analyzed the effect of speed of sliding, pressure, regime of lubrication and content of reinforcement in the composites on the coefficient of friction. The apparatus used was pin-on-ring for carrying out experimental results and these mechanisms were analyzed by SEM. Results showed that the reinforcement content affects wear behaviour and loss decreases with volume fraction of reinforcement on the composite. AMMCs reinforced with TiB_2 particles showed the better wear resistance in dry as well as in lubricated conditions. For the AMMCs reinforced with $TiAl$ showed the best wear resistance [18]. **Walker et al. [19]** have found out that the beneficial effect of Al-SiC composite can be limited by the abrasive nature of the SiC, leading to increased counter face wear rates. Using pin-on-ring tribometer and a commercial synthetic oil, they investigated the lubricated sliding response of two aluminium alloys i.e. 2124 and 5056 and AMMC reinforced with a number of particulate intermetallics, Ni_3Al , Cr_3Si , $MoSi_2$ and SiC. $NiAl_3$ had shown better tribological behaviour under dry sliding conditions. The incorporation of a particulate reinforcement reduced the lubricated sliding wear rate of both alloys. However, use of the SiC as reinforcement lead to severe abrasion of both contacting surfaces and wear mechanisms was understood by using SEM [19]. **Babic et al. [20]** have used A356 as a base matrix alloy and oil with viscosity grade VG46 (ISO3848) for the tribological study of A356/10SiC/1Gr hybrid composites. Hybrid composite specimen was obtained by compocasting procedure. Tribological tests were done on tribometer with block-on-disc for various values of sliding speed, normal load, sliding distances. Wear mechanisms were investigated by SEM. It was revealed that with increase in sliding speed, wear rate of the hybrid composite and the base alloy decreases. Also, the composite showed their superior performance as compared to the base alloy A356 [20].

Dixit and Khan [21] have studied the aluminium alloy and the partial lubricated sliding wear behaviour of SiC reinforced aluminium composites produced by Vortex method with the help of a pin on disc tribometer. Wear rate, frictional heating and friction coefficient were studied and wear tests were carried out with help of SAE 20W-40 oil, SAE 20W- 40 oil and graphite particles. The sliding velocities of 2.1 and 8.4 m/sec and applied load of 10 to 200 N in different lubricated condition influenced graphite dispersed in the oil and the wear behaviour of the samples has been studied. The (Aluminium-based) matrix alloy was characterized to examine the influence of the dispersoid (SiC) phase on the wear. The

composite showed the higher frictional heating and friction coefficient than pure alloy in all the cases. The wear rate and frictional heating increased with load and speed, while friction coefficient was affected in reverse way [21].

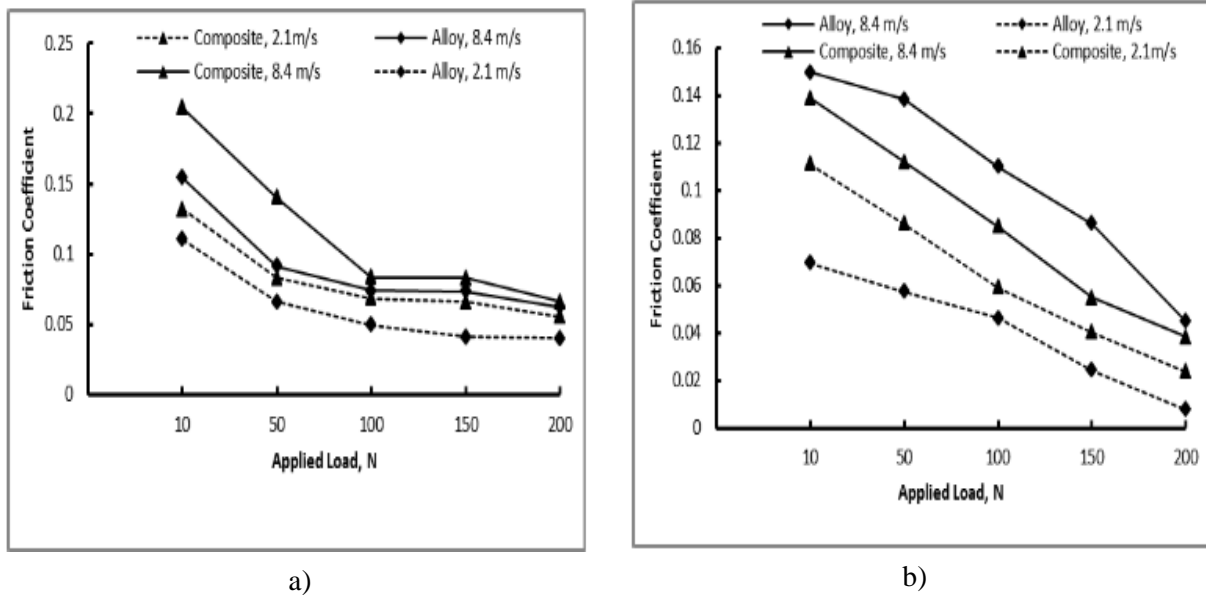


Figure 2.3 (a) Friction Coefficient in Oil Lubricated Environment (b) Friction Coefficient in Oil + (7-10µm Graphite) Lubricated Environment [21]

2.5 LUBRICATED SLIDING USING PARTICLE ADDITIVES

A lot of research also has been carried out on the potential particle additives that can be used for the wet lubrication studies of tribo-pairs. However, they are not yet explored for the AMMC tribology. Some of the significant studies involving the potential particle additives for our purpose are briefly summarized here.

Ilie & Tita [22] have studied the tribological properties of MoS₂ nanoparticles under different friction conditions. These particles were mixed in a lubricating oil using a four-ball tribometer and wear test were carried out in a block-on-ring tribometer [22]. **Rosentsveig et al. [23]** have carried out tribological testing using nanoparticles in two synthetic PAO oils and results were compared to bulk (2H platelets) MoS₂ and IF-WS₂. Tribological performance study had unveiled that the IF nanoparticles strongly depends on their crystalline order and size [23]. **Hu et al. [4]** have studied the tribological properties of paraffinic oil containing MoS₂ additives of various morphology and sizes to improve these properties of paraffin. The MoS₂ nano balls and slices show better friction reduction as compared to microparticles. The tests were investigated at 50C, rotating speed of 1450 rpm and constant

load of 300N and steel with hardness of 61-63 HRC [4]. **Düzcükoğlu and Acaroğlu [5]** had studied the wear performances using commercial mineral oil and vegetable oils and a combination of vegetable oil and boric acid on a pin-on-disc test apparatus [5]. **Greenberg et al. [24]** studied the tribological properties of IF-WS₂ nanoparticles dispersed in API grade I base oils of three different viscosities under different regimes of lubrication and proposed transfer film formation mechanism decreased friction to almost half of its original value in case of mixed lubrication regime [24]. **Kalin and Kogovšek [25]** have tried to compare tribological behaviour of MoS₂ and WS₂ as fullerene-like particles, platelets and nanotubes. The authors come with the fact as described that particle morphology did not significantly affect the coefficient of friction [25].

2.6 RESEARCH GAPS

After going through the literature reported by different authors following research gaps are identified.

- Majority of the works found in the literature are primarily directed towards the development and improvement of the mechanical properties of composites, whereas a little attention has been given on the lubricant formulation for the use of these composites in real world scenario where they might be subjected to the lubricated sliding.
- Although, the considerable research has been undertaken on the dry sliding wear and friction behaviour of AMMC with various particulate reinforcements under dry sliding conditions, however, a huge scope of study still exists for the lubricated sliding behaviour of the same, which has huge potential applications.
- A huge gap still exists to optimize the material design for the lubricated sliding applications of these composites, such as in automobile engines.
- The use of solid lubricant in conventional liquid lubricant is yet to explore for the moving composite material contacts.
- There are limited literatures available on the wet tribology and the influence of particle additives in case of tribocontacts made of composite.

Chapter 3

OBJECTIVE AND METHODOLOGY

3.1 INTRODUCTION

A lot of research has been done to reduce friction and wear resistance between tribo contacts. In many cases it is found to enhance the friction characteristics of the composite to some extent, by making it self-lubricating in nature. However, self lubricating and hybrid composites are not self sufficient to provide an enhanced tribology with high load bearing capacity, that is desired in lots application such as automotive applications. However, the emerging research on composites along with the potential benefit of using particle additive in lubricated contact motivate us to formulate a well designed tribo system to reduce friction and wear in various applications so that we can optimize our inputs. Lastly, there is the requirement for extensive research to harness the benefits of wet tribology of AMMC for the minimizing the friction and wear in the potential applications such as in automotive sectors.

3.2 OBJECTIVES OF THESIS

- Selection of the various reinforcements for the composite to be fabricated for tribological study
- To fabricate a metal-matrix composites with available infrastructure.
- To carry out the mechanical and metallurgical characterization of fabricated composites.
- Selection of particle additives for lubricated sliding between tribo-pair of AMMC-steel contact.
- To study the wear and friction behaviour of composites under dry, lubricated sliding and particle aided lubricated sliding using pin-on disc tribometer.
- Investigate the friction and wear mechanism by analyzing the worn surfaces using SEM, EDS and XRD.

3.3 EXPERIMENTAL PROCEDURE

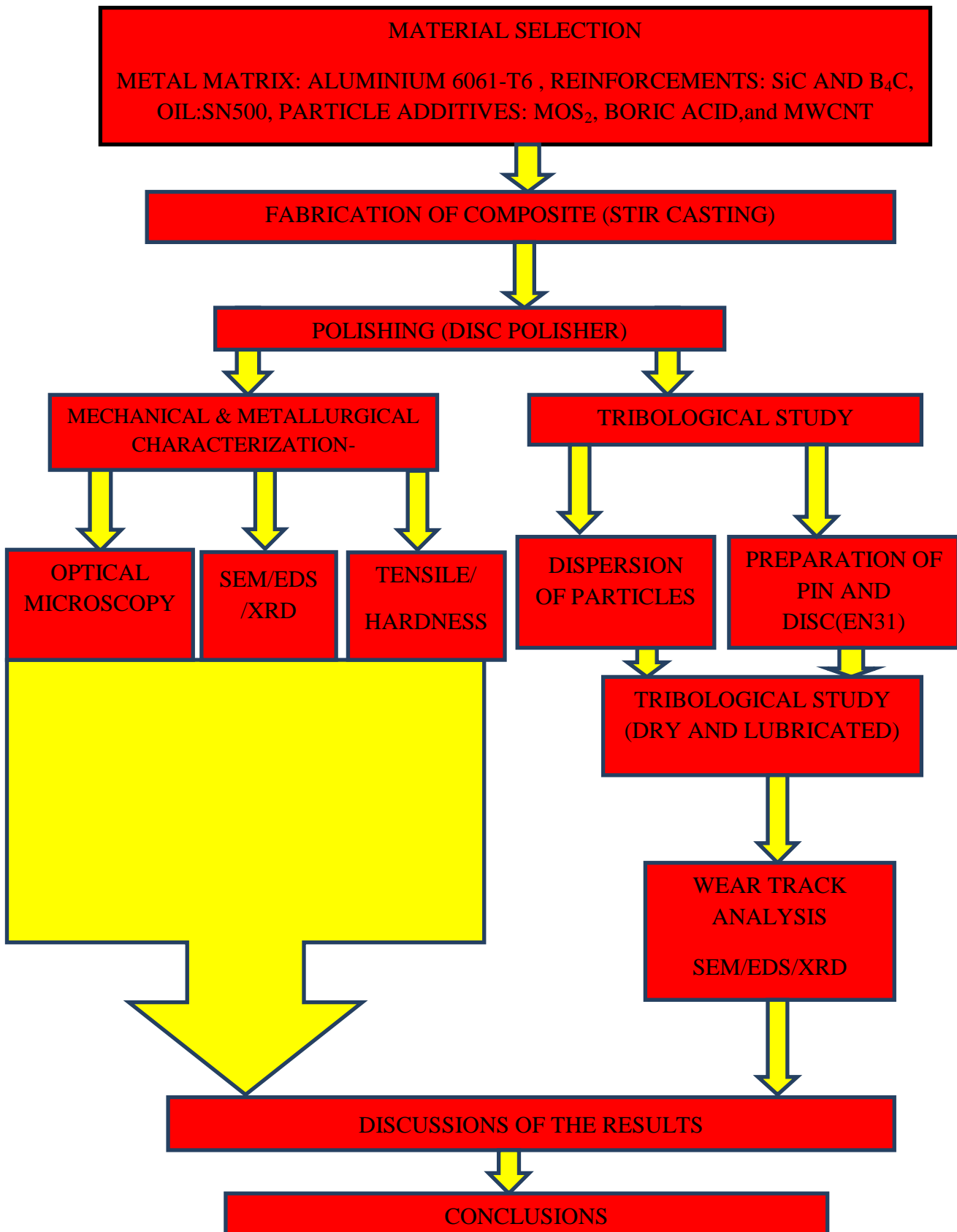


Figure 3.1 Flow chart of work plan

3.4 MATERIAL SELECTION

Aluminium 6061 is selected as base material for the matrix to form a composite because of its inherent qualities such as its abundance, low cost, soft, durable, lightweight, ductile, malleable metal, high corrosion resistance, excellent heat conductivity, etc. The selected aluminium with alloyed content (Al6061) has the following composition:

Table 3.1 Composition of Al 6061 [26]

Component	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zin	Al
Wt. %	0.04- 0.35	0.15- 0.4	Max. 0.7	0.8- 0.12	Max 0.15	0.4- 0.8	Max 0.15	Max 0.25	Balance

SiC is considered as particulate reinforcement for the composite because of its high strength, hardness and elastic modulus, besides its excellent thermal conductivity and thermal shock resistance. SiC also has superior chemical inertness. Properties of the selected silicon carbide particles are shown below.

Table 3.2 Properties of SiC [W3]

Density	3.21 gm/cc ³
Hardness	2800 kg/mm ²
Fracture toughness	4.6 MPa
Thermal conductivity	120 W/m.K
Specific Heat	750 J/kg.K
Size	37 µm

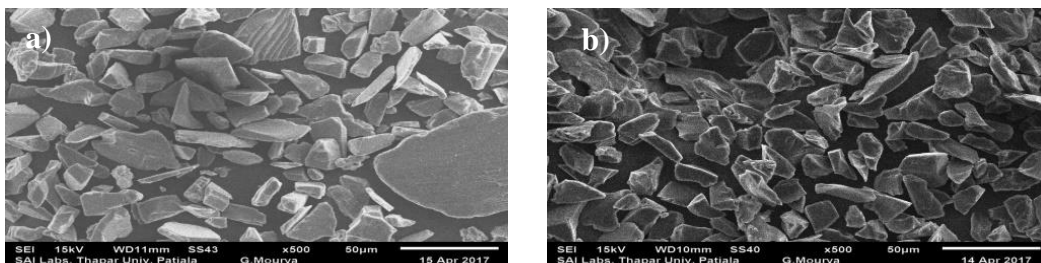


Figure 3.2 SEM micrographs of (a) SiC (b) B₄C

Boron carbide is believed to be the third hardest metal behind cubic boron nitride and diamond. It has been considered as the reinforcement because of its extreme hardness, good

chemical resistance, good nuclear properties, low density, and excellent chemical and thermal stability. Properties of selected boron carbide are shown below.

Table 3.3 Properties of B₄C [W4]

Density	2.52g/cm ³
Hardness	2900-3580 kg/mm ²
Fracture toughness	2.9 - 3.7 MPa
Thermal conductivity	30-42 W/m.K
Specific Heat	840 J/kg.K
Size	25-28 μm

3.5 FABRICATION OF COMPOSITES

The composites were fabricated by Stir Casting method. The process starts with taking Al 6061 alloy in the form of 23.8 mm diameter rods are cut into 150 mm length and were placed in a graphite crucible. Then the aluminium was melted in a resistance heated muffle furnace at the desired temperature of 750⁰C for 2-3 hours. Then in the mean time, reinforcement particles were heated separately in a Baking Oven to a temperature of 200⁰C to remove moisture from the powders taken. Then the magnesium ribbon was put into the molten metal for the wettability and after some time the boron and/or silicon carbide were mixed into the molten metal. The mixture was stirred by using mechanical stirrer for about 10 minutes at an impeller speed of 400 rpm. The melt temperature was maintained at 750⁰C during addition of the particles. The molten metal was then poured into the die to cast plates of 150mmx75mmx50mm size. This same procedure was followed for all the composites prepared. The processing parameters are summarized in the table below.

Table 3.4 Processing parameters for stir casting

S.No	Parameters	Value
1	Furnace temperature	750 ⁰ C
2	Pre heated temperature of particles	200 ⁰ C
3	Spindle speed	400 rpm
4	Stirring time	10 mins



Figure 3.3 Cast composites (a) SiC 5% B₄C 5%, (b) B₄C 10%, (c) SiC 10% [Photo courtesy: Thapar university, Patiala]

A brief overview on the equipments and components which are used in stir casting process are given below.

Casting furnace (Figure 3.4a) used for the composite fabrication has a maximum temperature range of 1100^o C and desired temperature can be obtained via electrical resistance heating elements. The stirrer (Figure 3.4b) is designed to get homogeneous mixture of metal in liquid and liquid-solid form with reinforcement. This stirrer is connected with electric motor (range 22-480) with help of Stainless steel rod. The stirrer is positioned near the bottom of a ceramic crucible (Figure 3.4c) while mixing.

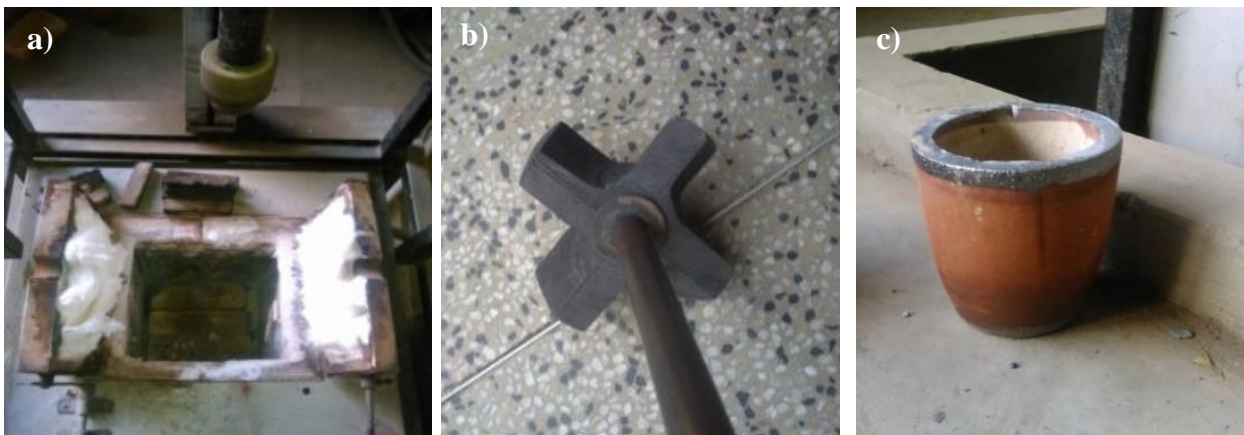


Figure 3.4 (a) Muffle Furnace, (b) Designed Graphite stirrer, (c) Ceramic crucible [Photo Courtesy: Thapar university campus, Patiala]

The liquid-solid metal is poured into mould having a pattern shape (Figure 3.5a) to cast the MMCs. A power hacksaw is used for cutting the cast materials into small piece so it to keep metal into crucible. Also, reinforcement is preheated in oven to get moisture-less (dry). Because dry reinforcement (powder) mix properly in liquid metal. The range of baking oven is 500°C.



Figure 3.5 (a) Cast iron mould , (b) Power Hacksaw[Photo Courtesy: Thapar university campus, Patiala]

3.6 PREPARATION OF PARTICLE BASED LUBRICANTS

The particles were dispersed in base oil (SN 500) and the dispersions of particles were done with the help of ultrasonicator probe as shown in Fig. [Make: Oscar Ultrasonic converter, India] for 1 hr. The concentration used for dispersion is 0.1% (w/v) and it was kept same for all additives dispersed in oil. The additives used are boric acid, MoS₂, MWCNT.



Figure 3.6 Ultrasonicator [Photo Courtesy: Thapar university campus, Patiala]

The properties of base oil are given in Table 3.5 and that of particle additives in Table 3.6.

Table 3.5 Properties of Base oil

S.No	Properties	SN 500
1	Kinematic viscosity@40 ⁰ C	84 cST
2	Kinematic viscosity@100 ⁰ C	14 cST
3	Density @15 ⁰ C	826 kg/m ³

Table 3.6 Properties of particle additives

S.No	Oil additive	Size (μm)	Density (g/cm^3)
1	MoS ₂	1-15	4.80
2	MWCNT	length:3-8 μm ,dia.:10-20nm	2.60
3	Boric acid	10-20	1.44

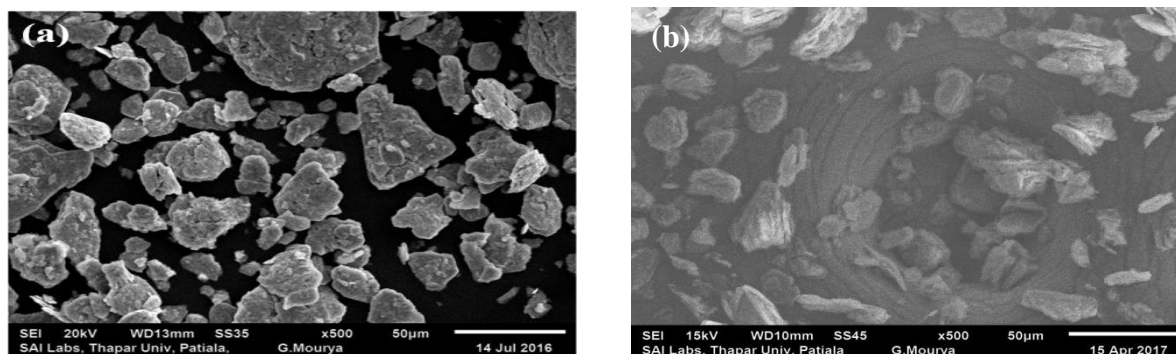


Figure 3.7 SEM micrographs of (a) MoS₂, (b) boric acid

3.6 METALLURGICAL CHARACTERIZATION

X-ray diffraction (XRD) was used to establish the structure of composites and wear tracks. X-rays are produced with high speed electrons from a hot tungsten (W) filament. The anode is a water-cooled block of copper (Cu) containing desired target.



Figure 3.8 X-ray diffractometer [Photo courtesy: SAI Labs, Thapar University Campus, Patiala]

In SEM high energy electron focus beam used for generate different variety signals to get information about sample morphology, chemical composition, and crystalline structure.

The SEM used for this study is a highly accurate and precise instrument (Make: JSM-6510LV, JEOL Ltd, Tokyo, Japan) for fast characterization and imaging of fine structures and has a magnification range from 5–300,000 X (printed as a 128mm x 96 mm micrograph). It was used to study the worn surfaces and investigate the wear mechanism. This facility is available at SAI Labs, Thapar Technology Campus, Patiala.



Figure 3.9 Scanning electron microscope [Photo courtesy: SAI Labs, Thapar University Campus, Patiala]

Optical microscope (Figure. 3.10) is used to observe the internal structure of the metals at different scale and provide a qualitative and quantitative description. In a present study metallurgical microscope is used to analyses the shape, size and dispersion of the reinforcement particles in the matrix alloy. Before examining the structure of the composite, samples are well polished with the help of fine grade emery paper and then etched with etching solution for 20 to 40 seconds.



Figure 3.10 Metallurgical Microscope [Courtesy: Advanced metallurgical lab, Thapar University, Patiala]

3.7 TRIBOLOGY STUDY

To perform the experiments for our research work the pin-on-disk apparatus (Model: TR-201LE, DUCOM make), shown in Figure 3.11 (Make: Mettler-Toledo, Greifensee, Switzerland) was used to examine the dry sliding and lubricated sliding wear characteristics and the coefficient of friction of the composites. Cast samples were machined to form tribometer pins for testing. The pins of diameter 5.98 ± 0.02 were measured by vernier calliper having least count 0.02. Test pins were polished with emery papers from 600 to 2000 grit size. EN31 was the disc used as counter surface having hardness 58-62 HRC, supplied by Ducom, Bangalore, India.

Tribometer runs were carried out as per ASTM G-99 standard. In the present study test samples (prepared tribometer pin) are tested under following operating conditions as shown in table. For each test run, disc and pin was cleaned with acetone or hexane. The friction forces were directly obtained from the automatic digital reading and then calculated coefficient of friction. Samples were weighed before and after the test using a digital weighing balance as shown in Figure 3.12

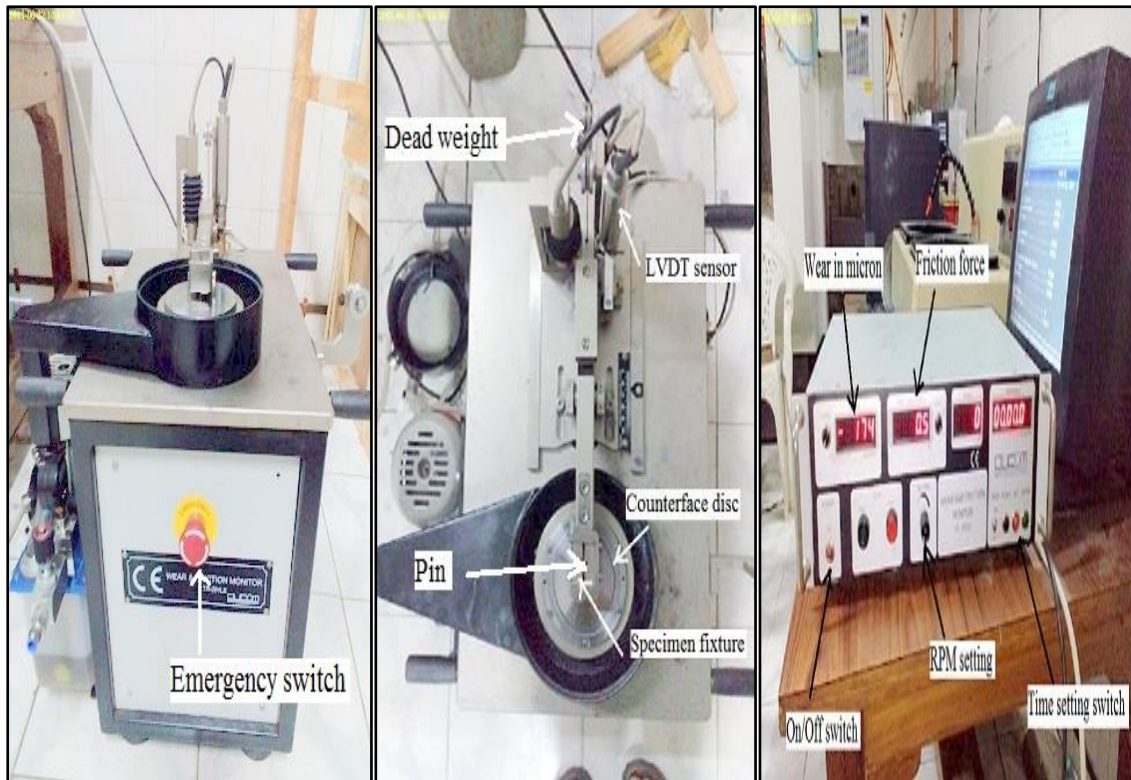


Figure 3.11 Components of pin-on-disk test apparatus [Photo Courtesy: Thapar university campus, Patiala]



Figure 3.12 Weighing machine [Photo Courtesy: Thapar university campus, Patiala]

Table 3.7 Operating Parameters and sliding conditions for sliding wear tests.

Parameters	Description
Test setup	Pin-on-disc
Wear test standard	ASTM-G99
Wear pin	Material: Al6061, Al6061/SiC/B ₄ C Geometry & size: Hemispherical tip ; ϕ 6 × 3.5 mm Initial roughness: 0.12 μ m
Counter disc	Material: EN31 Geometry & size: Flat ; ϕ 100 × 8 mm Initial roughness: 0.2 μ m
Normal load	9.8 N
Sliding velocity	Constant (0.5 m/s)
Time duration	1800 sec
Sliding distance	900 m
Lubrication condition	Dry sliding & Wet lubricated
Temperature	25±2°C

In this study, profile projector (Figure 3.13) is used for the profile study of the worn out and freshly made pin ends. The main function of profile projector is to project the image of an area to be selected on to the screen using diascopic illumination. It magnifies the profile of object and display on to built-in-projection screen. On the screen there is grid which rotates the image at 360° in X-Y directions. The screen display profile of object and magnifies it for ease of calculation of linear measurements. The object is opaque then light will not pass through it but it will form a profile of object.



Figure 3.13 Profile projector [Photo Courtesy: Thapar university campus, Patiala]

3.8 MECHANICAL CHARACTERIZATION

Hardness: Hardness is probably known as the resistance of a material to localized deformation or simply referred as resistance to indentation or scratch. The deformation can be in the lot of forms like indentation, cutting, scratching, elongation, bending. In case of ceramics, metals and polymers deformation is considered as plastic deformation of the surface. Hardness can be measured through various techniques such as Rockwell's, Brinell's and Vicker's hardness test are the most significant and important. Composition of mixture plays very important role in approximation of hardness. Volume and reinforcement of individual constituent sets the hardness value which lies between the higher and lower value of constituent. The reinforcement with Silicon Carbide (SiC), Alumina (Al_2O_3) and aluminized preferred for imparting higher hardness. MMC with particle in it has higher hardness than other reinforcements such as SiC, B_4C TiC, Al_2O_3 , and WC are good

reinforcements for higher hardness [27]. The size, shape as well as the concentration of the particle influences the hardness. With increase in the filler content, hardness as well as the load bearing capacity of composites increases. The micro hardness tester used for calculating the hardness is shown in Figure 3.14.



Figure 3.14 Micro-hardness tester [Photo Courtesy: Thapar university campus, Patiala]

Ultimate Tensile Strength: The mechanical properties of MMC depend upon the properties and structure of reinforcements. Various researchers found out that tensile strength of reinforced composites (SiC) is more than the unreinforced Aluminium. Ramakrishnan [28] concludes with the help of mathematical formulation models concludes that addition of SiC in 6061 aluminium matrices helps to acquire 10-70% additional strength depends on volume fraction and particle size. Due to Al matrix, dislocation density close to matrix reinforcement interface increases and causes strengthening effect [29]. Tensile strength of MMC depends upon the stress transfer from metal matrix to reinforcement. Increase in reinforcement helps to bear more loads on strong SiC reinforcement. In composites, crack needs to propagate along matrix and reinforcement both due to strong matrix interface. Strengthening

mechanism is described by Reddy [30] where he describes two possibilities for increase in strength, increase in reinforcement with smaller particle and other is formation of cluster or precipitates at the particle/ matrix interface. Small particle sizes have large surface area for transferring stress from matrix and nucleated voids are not blend easily.

Chapter 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter discusses about the results of mechanical and metallurgical properties of the aluminium metal matrix composites, particle characterization and tribological behavior of fabricated composite under different contact conditions.

4.2 MICRO HARDNESS

The micro hardness results of Al6061 and their composites with different percentage of reinforcement of silicon carbide and boron carbide are shown in the Table 4.1.

Table 4.1 Micro-hardness values for aluminium alloy and composites

S.No	Sample Name	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Mean
1	Al 6061	77	83	83.5	83.56	74	80.212
2	AMMC SiC 10%	97	96	102	101	104	100.2
3	AMMC B ₄ C 10%	165	156	148	149	140	151.6
4	AMMC B ₄ C 5% SiC 5%	144	145	140	145	148	144.4

Images in Figure 4.1 show the indent mark on the samples after microhardness test.

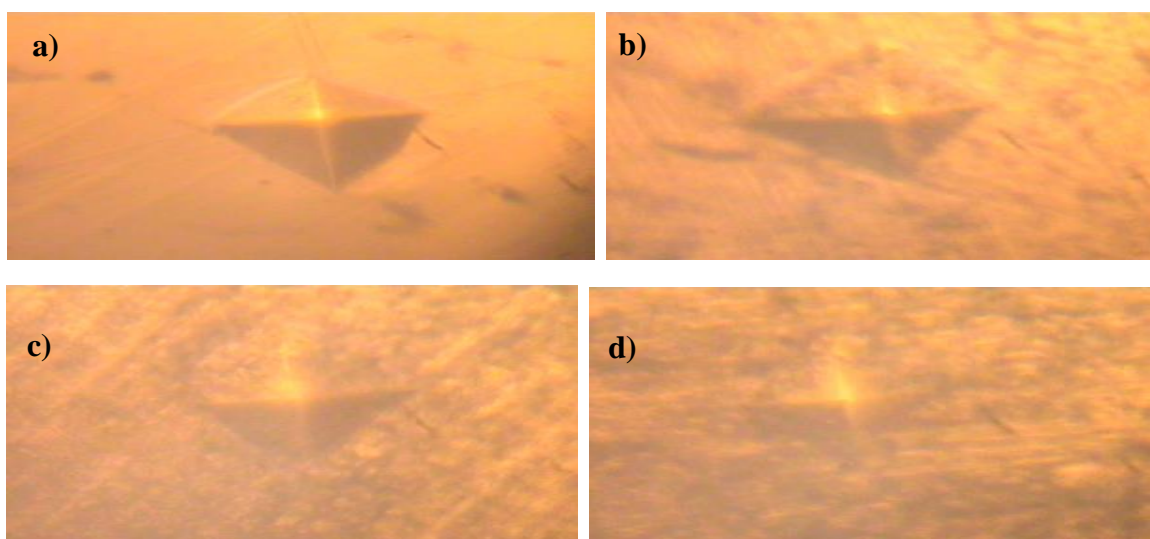


Figure 4.1 Microhardness tests of (a) Al 6061 (b) AMMC SiC 10% (c) AMMC B₄C 10% (d) AMMC B₄C 5% SiC 5%

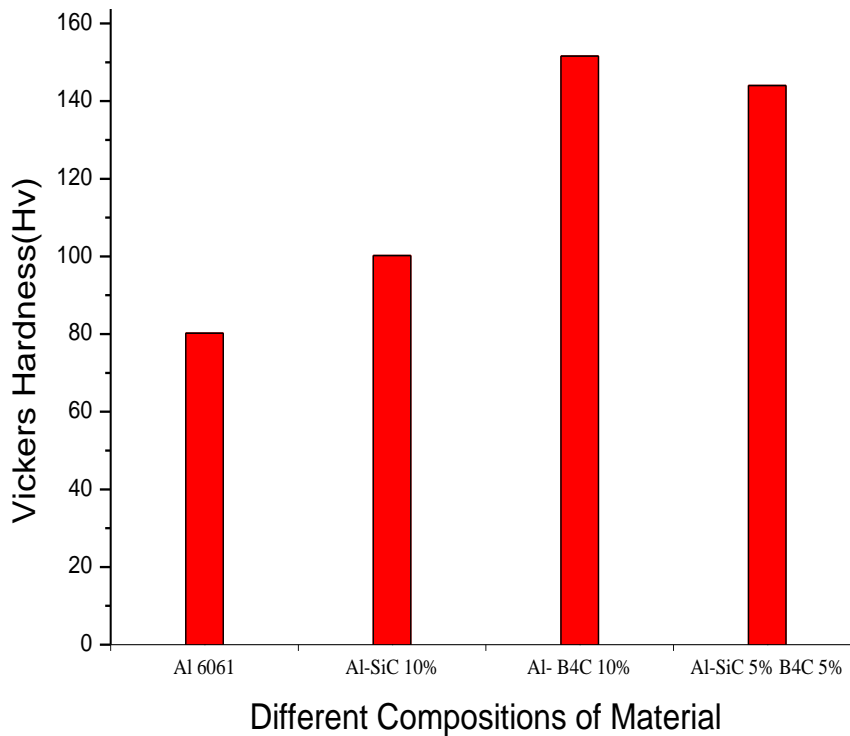


Figure 4.2 Vicker's hardness for different composition

We can see that the hardness value got by us comes out to be as the reported value as in case for the base metal. The Figure 4.2 shows the hardness for the base alloy and the fabricated composites. The mean hardness of the composites are greater than the base alloy due to obvious reasons as these composites are reinforced with hard SiC and B₄C particles. From the Table 4.1 the hardness results show that the hardness of aluminium composites increases with the use of harder material from SiC to B₄C and hybrid composites respectively. This is because of the reason of uniform distribution of SiC and B₄C particles in aluminium matrix (as we can see from the SEM images of particle characterization). With the increase in wt% of these particles overall hardness of the composite increases. The hardness of B₄C reinforced composite is found to be largest whereas for the hybrid composite it is found to be comparable to the single B₄C reinforced composite. It may be due to the two hard reinforcement interactions. Thus, it is clear that the single reinforced composite with higher content reinforcement can also be replaced by the hybrid composite with lesser amount of individual reinforcement provided the other properties are also improved.

4.3 METALLURGICAL CHARACTERIZATION OF COMPOSITES

4.3.1 SEM AND OPTICAL MICROGRAPHS

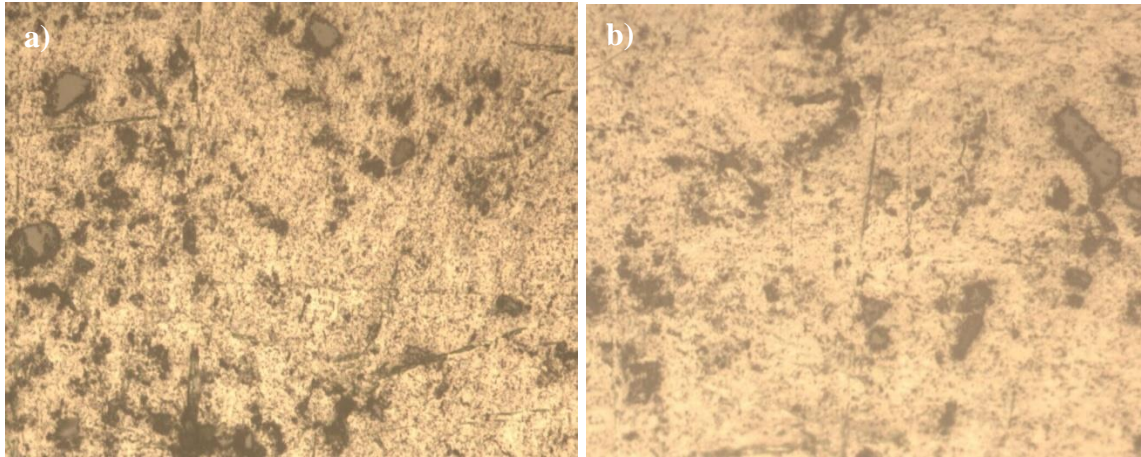


Figure 4.3 Optical images for Al-SiC composite (a) 10x (b) 20x

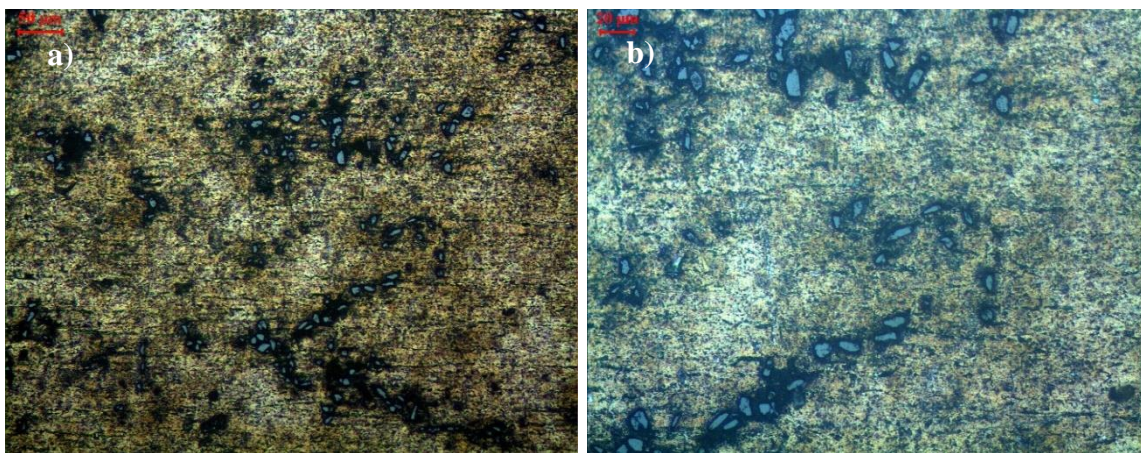


Figure 4.4 Optical images for Al-B₄C composite (a) 10x (b) 20x

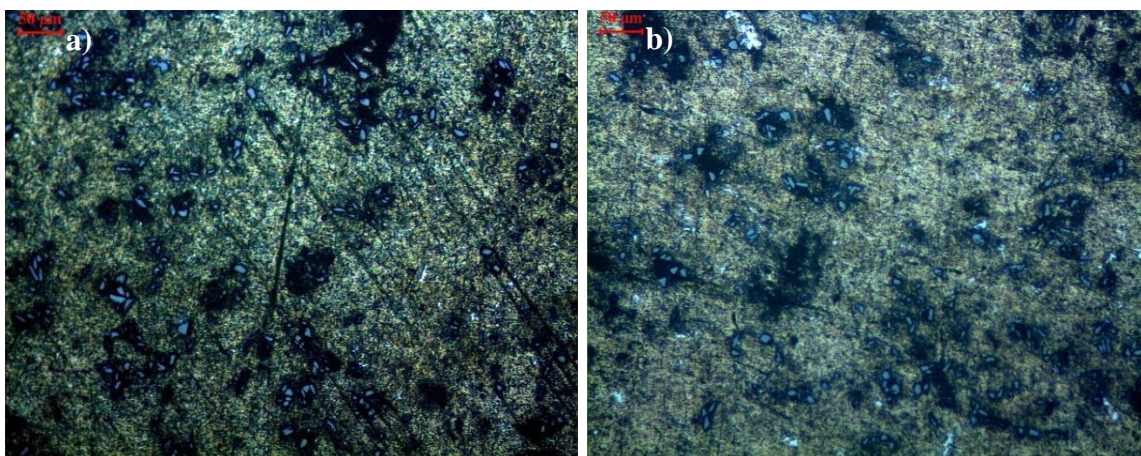


Figure 4.5 Optical images for Al-SiC-B₄C composite (a) 10x (b) 20x

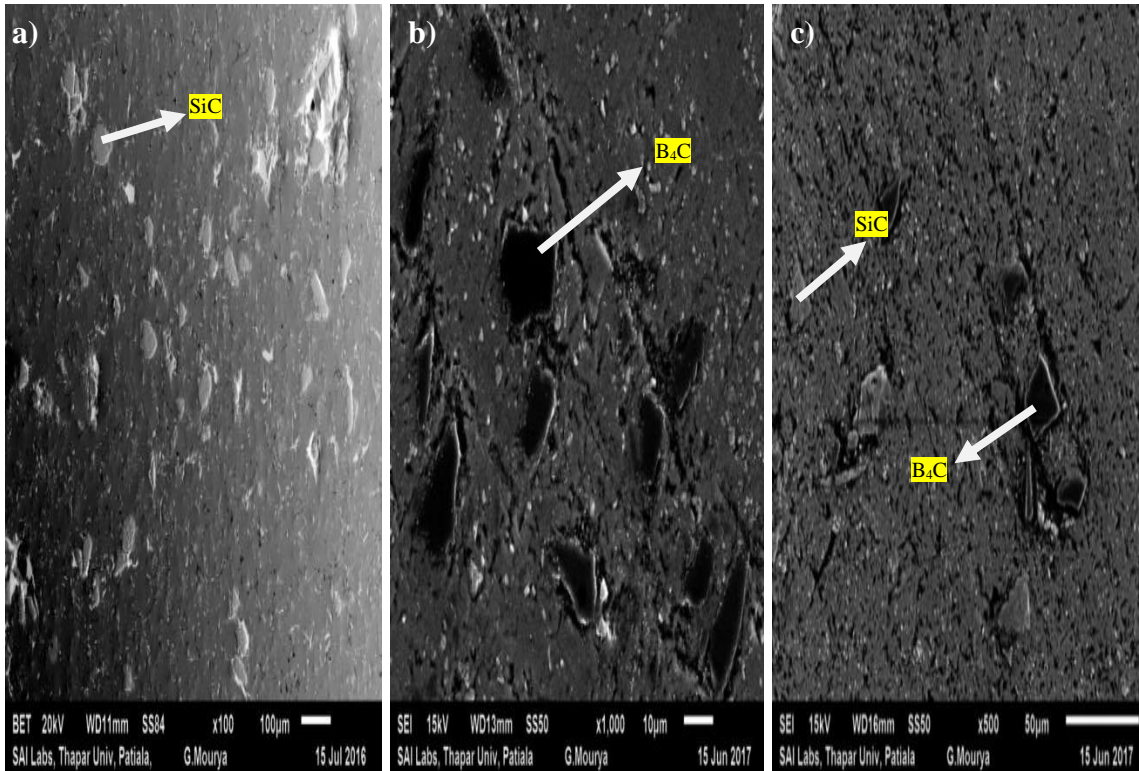


Figure 4.6 SEM Images (a) SiC (b) B₄C (c) SiC-B₄C

Figures 4.3-4.5 show the optical images of the fabricated composites at different magnification. Figure 4.6 shows the SEM images of the same. Both optical and SEM images show the uniform distribution of SiC, B₄C and hybrid composites in matrix. The mechanical properties of the composites improve because of this uniform distribution. The process parameters of stir casting like stirring speed and time, etc. were suitably selected for this purpose.

4.3.2 COMPOSITION ANALYSIS OF THE FABRICATED COMPOSITES

The Figure 4.7 shows the EDS spectra and Table 4.2 shows the elemental compositions on the selected surfaces of these composites. From the figures it is evident that the reinforcing agents are clearly distinguishable in the matrix.

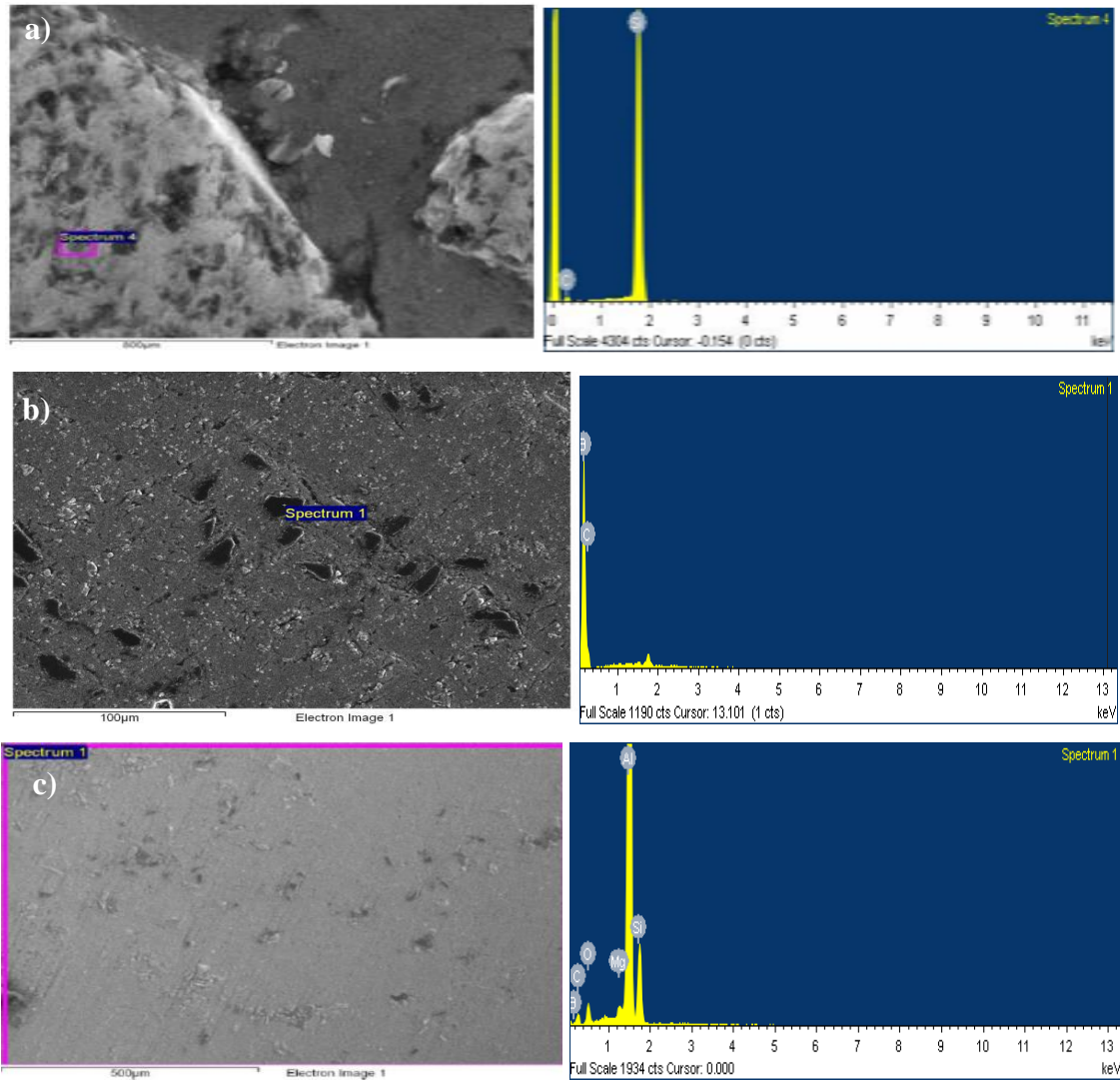


Figure 4.7 EDS on various composites (a) Al-SiC, (b) Al-B₄C (c) Al-SiC-B₄C

Table 4.2 Chemical compositions from EDS on reinforced particle of various composite

Element	Al-B ₄ C	Al-SiC-B ₄ C	Al-SiC
B K	66.54	4.21	
C K	33.46	15.66	35.62
O K		7.17	
Mg K		0.68	
Al K		61.56	
Si K		10.73	64.38
Totals		100	

The Figure 4.7 and Table 4.2 clearly show the composition of the respective composite. The elements detected through EDS are as expected for the various samples. The spectra for Al-B₄C and Al-SiC samples were focussed on the reinforced particles, so the base metal is relatively not detected. In some situations, however, some amount of oxides can also be seen, due to the effect of high temperature processing of the composites, such as in Al-SiC-B₄C composite as shown in the Table 4.2. The presence of oxides further helps in hardening and strengthening of the matrix, which may be quite a beneficial for the improvement of the mechanical and wears resistance properties of the composite.

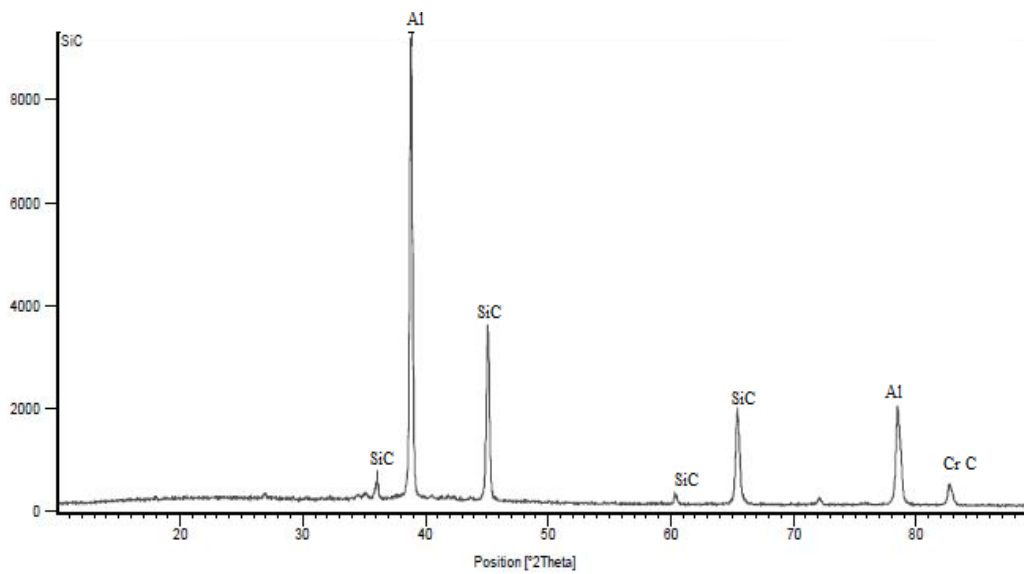


Figure 4.8 XRD for Al-SiC composite

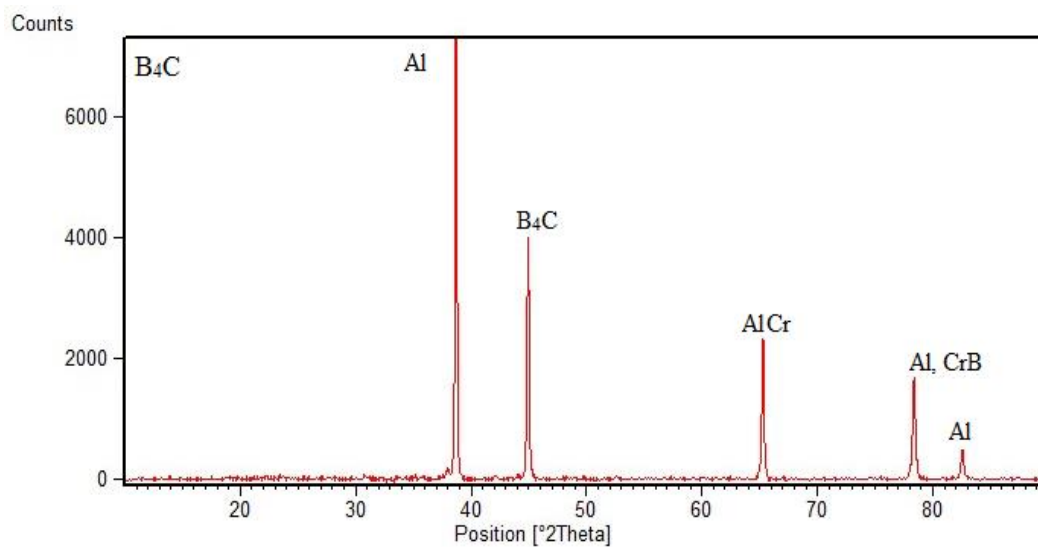


Figure 4.9 XRD for Al-B₄C composite

Further investigation on the chemical composition of the fabricated composites is carried out by XRD. Figure 4.8 shows the X-ray diffraction patterns of the Al-SiC composite. From the XRD peaks observed between 30 to 90 degrees it is clear that the most significant peaks consist of SiC coming from the reinforcement and Al from the base metal, along with some negligible amount of CrC, etc formed during casting. The similar trend can also be observed for B₄C composite, as shown in Figure 4.9 CrB formed in this case serves as a catalyst for processing the hydrocarbons. On the other hand, XRD patterns for the hybrid composites (Figure 4.10) shows AlSiC, AlB₂ peaks besides the presences of SiC and B₄C. This, however, may compromise the hardness and wear resistance of the resulting composite.

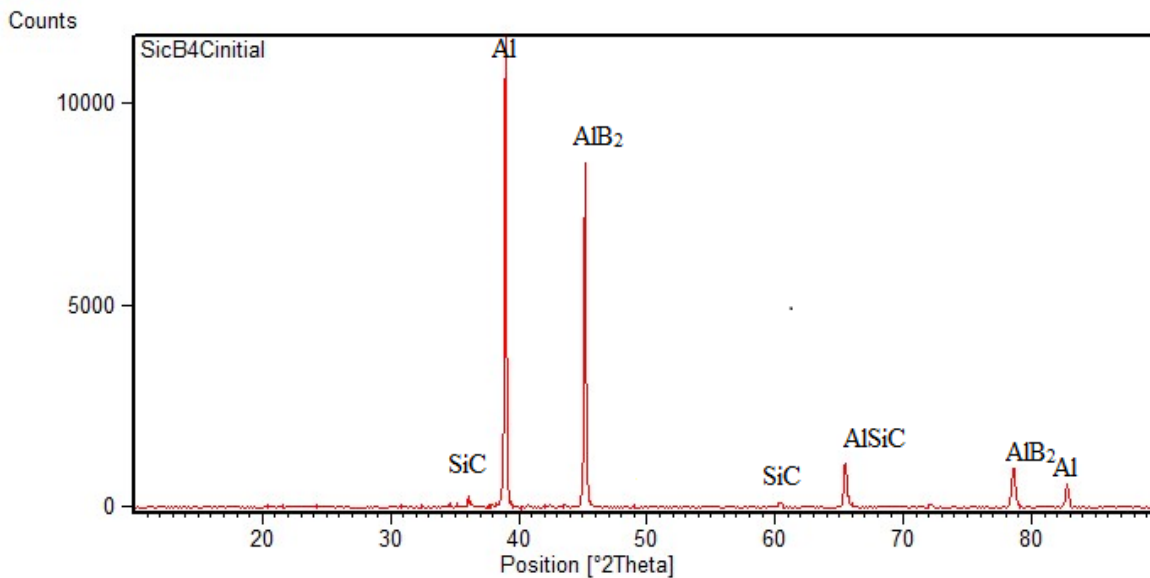


Figure 4.10 XRD for Al-SiC-B₄C composite

4.4 TRIBOLOGY STUDY AND WORN SURFACE ANALYSIS

Sliding wear tests were performed for the unreinforced aluminium alloy, single particle reinforced composite as well as for hybrid composites using a pin-on-disc apparatus at normal load of 10 N and sliding speed of 0.5 m/s with a constant sliding distance of 900 m. The tests were carried out in three different lubrication condition viz, dry, lubricated with base oil and lubricated with base oil containing particle additives. The wear rate was

calculated by weighing the specimens before and after the test and formula used to calculate the wear rate and cof is

$$\text{Wear rate} = \text{Volume loss/sliding distance (mm}^3/\text{m)}$$

$$\text{Cof} = \text{Frictional Force/Normal reaction}$$

4.4.1 DRY SLIDING WEAR AND FRICTION

Coefficient of friction and wear rate obtained from the dry sliding experiments are shown in Table 4.3. Table 4.3 shows there is a significant decrease in the value of the cof and wear rate for the B₄C composite as compared to the other composite at dry sliding condition. This may be attributed to the low density, high hardness and thermal stability of boron carbide as compared to the SiC. Also, the composite with B₄C reinforcement shows relatively stable and lowest friction throughout the complete duration of the friction test, as depicted by Figure 4.11

Table 4.3 Coefficient of friction and wear rate for composites under dry sliding test

S.No.	Sample Name	Condition	$\mu \pm \sigma$	Wear rate (mm ³ /m)
1	B ₄ C 10%	Dry	0.115± 0.0105	1.38x10 ⁻⁰⁴
2	SiC 10%	Dry	0.217±0.0561	4.98x10 ⁻⁰⁴
3	SiC5%-B ₄ C5%	Dry	0.260±0.0482	7.28x10 ⁻⁰⁴

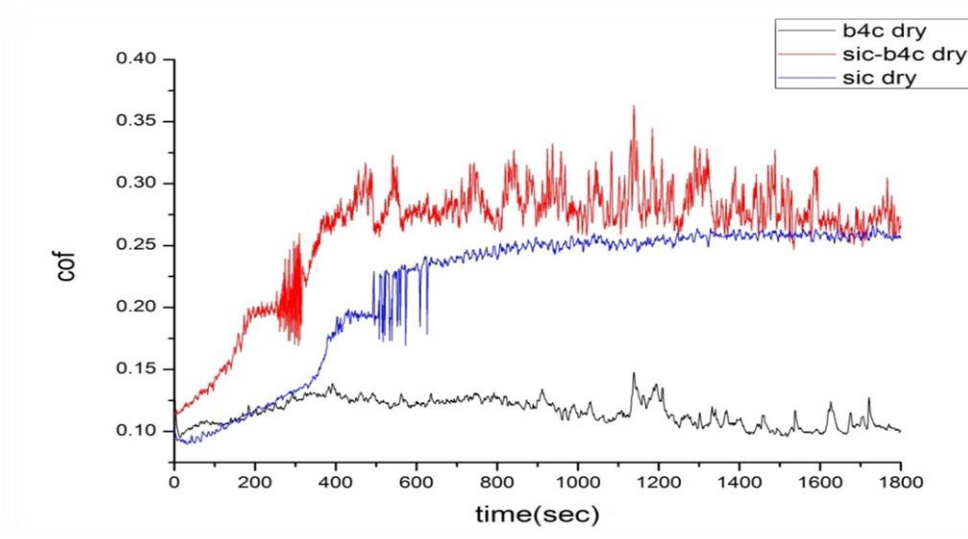


Figure 4.11 Variation of COF for various composites in dry conditions

To understand the underlying wear mechanism, further studies of worn surface analysis are carried out using SEM, EDS and XRD of wear tracks. Figure 4.12 shows the wear track for composites sliding in dry condition. It can be observed from the figure that the surface of the Al-B₄C composite pin has small size wear grooves than the other two types of composites; Al-SiC composite is having the largest size. This indicates that Al-B₄C composite is adequately hardened and strengthened by the reinforcement than that of its other counterparts. Moreover, during running in period mechanically mixed layer (MML) prevents the severe wear due to abrasion particle present under the contact. Thus, it is evident from the SEM micrographs in Figure 4.12 that the B₄C reinforcement decreases the sizes of wear grooves due to an improvement in the hardness and load-carrying capacity of the composites, enhancing the abrasion resistance of the composites. This in turn reduces the wear loss as compared to the other two composites.

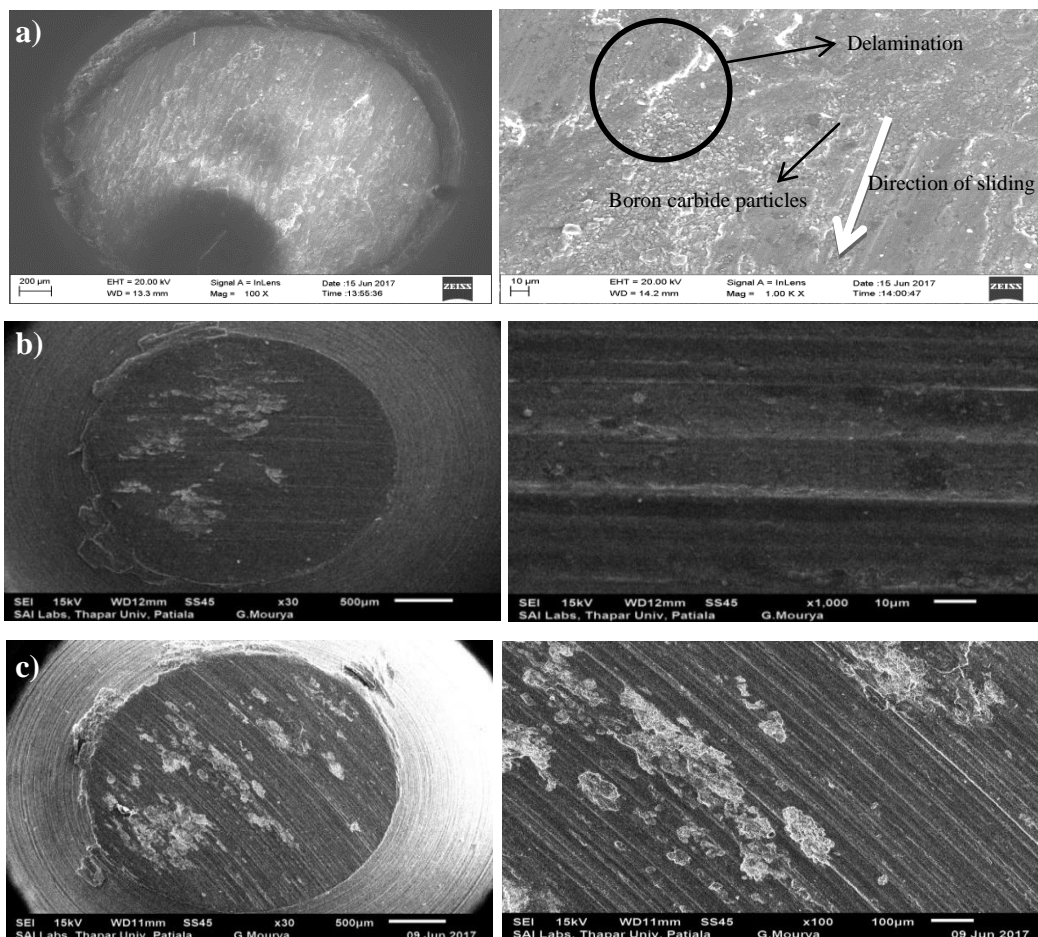


Figure 4.12 SEM images on the wear tracks of composite pins under dry sliding wear test (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

On the other hand there is a decreased wear resistance for Al-SiC and Al-SiC-B₄C reinforced hybrid composite as compared to the Al-B₄C composite. This is due to the fact that the reinforced particles are not capable of withstanding the high pressure offered by the hard asperities of the counter surface or the reinforced particles are pulled out from the soft matrix during the sliding. Either of the above again implies that there is a weak bonding of the reinforcements into the aluminium matrix. In case of hybrid composite there may be again the problem of segregation of particles of different nature which may lead to the clusters of particles into the matrix, as a result of which the mechanical properties of the fabricated composite is not enhanced up to the expected level.

Besides the above possibilities for variable wear and friction behaviour of these composites, these wear tracks are further investigated by EDS and XRD to gain more insight into the wear mechanism. Figure 4.13 shows the EDS spectra carried out in the selected regions of the dry sliding wear tracks from the three composites. Table 4.4 shows the elemental composites obtained from these spectra. The wear tracks generated on the Al-B₄C and Al-SiC-B₄C composites shows significant presence of boron and carbon. This indicates that the B₄C reinforcements are intact even during or after the sliding of pins against the hard counter surface made of EN31.

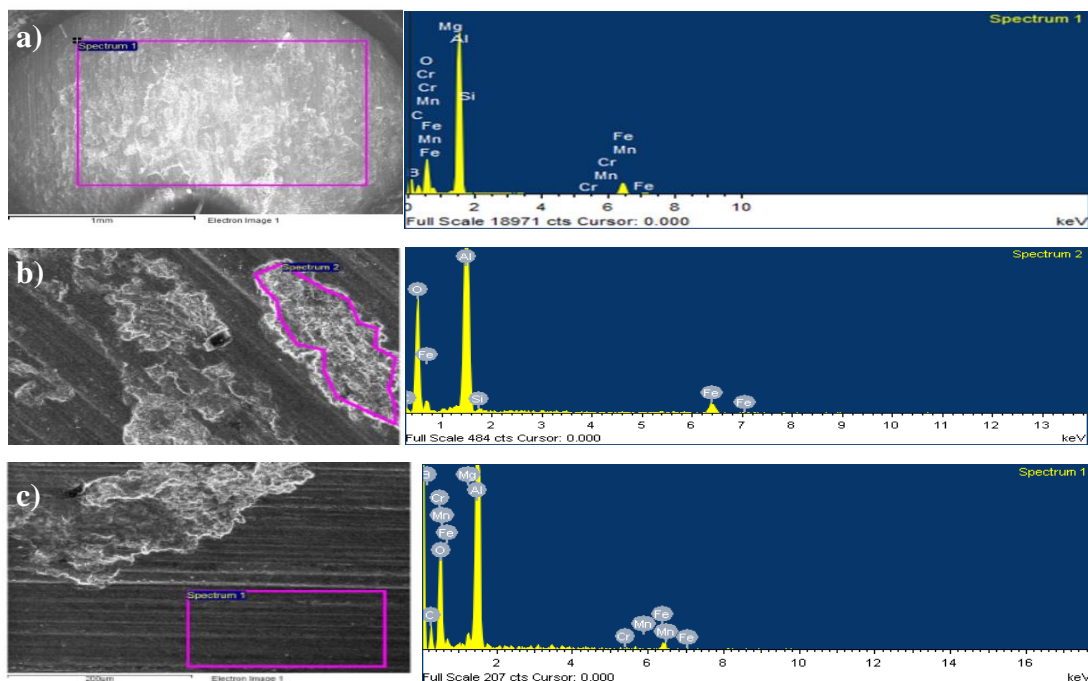


Figure 4.13 EDS on the wear tracks of composite pins under dry sliding wear test (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

However, in case of Al-SiC composite the negligible presence of Si makes it clear about the flushing out of the reinforced SiC from the matrix. As a result, grooves are generated on the surface of the matrix. Further contact of EN31 with the soft aluminium matrix, accelerates both the abrasive and adhesive action to the soft matrix, causing more wear on the pin surface by chemical or mechanical mixing as shown in the SEM images. The significant presence of Fe on the pin surface of the Al-SiC composite confirms the proposed fact. However, there is the formation of discrete or patchy oxide layers on the surface of the wear tracks in all the cases which is responsible for reduced wear rate and coefficient of friction.

Table 4.4 Elemental compositions (wt %) of the surface of the pin wear track of various composites under dry sliding test

Element	Al- B₄C	Al- SiC	Al-SiC- B₄C
B K	20.20		37.97
C K	23.84	9.12	23.98
O K	26.87	39.47	18.12
Mg K	0.15		0.47
Al K	21.11	40.30	17.29
Si K	0.15	0.58	
Cr K	0.10		0.15
Mn K	0.12		0.01
Fe K	7.47	10.52	2.02
Totals	100		

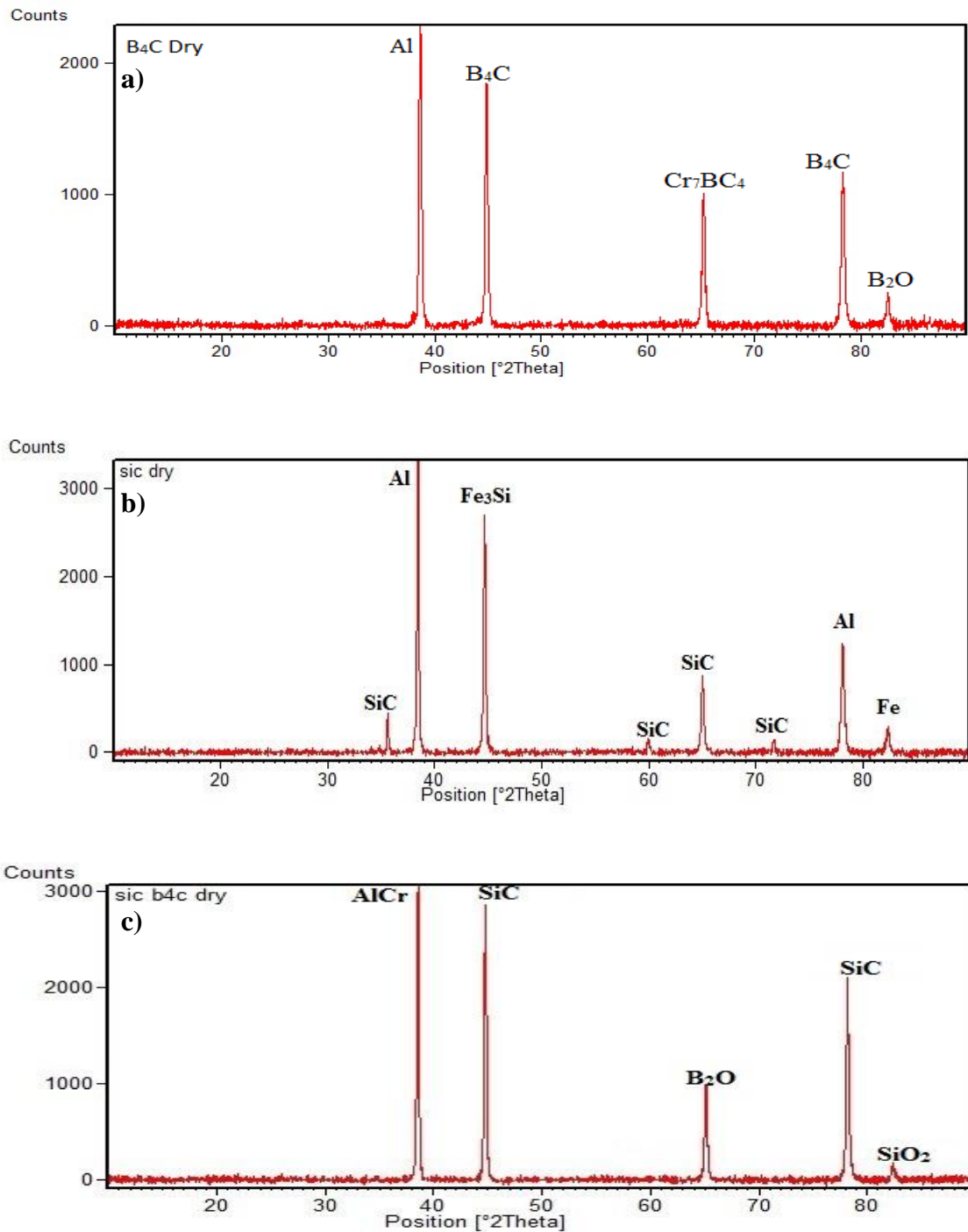


Figure 4.14 XRD for wear tracks on (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

4.4.2 LUBRICATED SLIDING WEAR AND FRICTION

After the dry sliding test evaluation of the fabricated composites, they are subjected to the lubricated sliding condition. Table 4.5 shows there is significant decrease in the value of the *cof* for all the composite from dry conditions to wet conditions, Al- B₄C being the best. The

cause of such low friction and wear rate is the formation of tribolayer and presence of low interfacial strength film between the tribo contacts.

Table 4.5 Coefficient of friction and wear rate for composites under lubricated sliding test

S.No.	Sample Name	Condition	$\mu \pm \sigma$	Wear rate (mm^3/m)
4	B ₄ C 10%	Oil	0.054±0.0015	2.3×10 ⁻⁰⁵
5	SiC 10%	Oil	0.075±0.0088	6.08×10 ⁻⁰⁵
6	SiC5%-B ₄ C5%	Oil	0.056±0.0065	5.75×10 ⁻⁰⁵

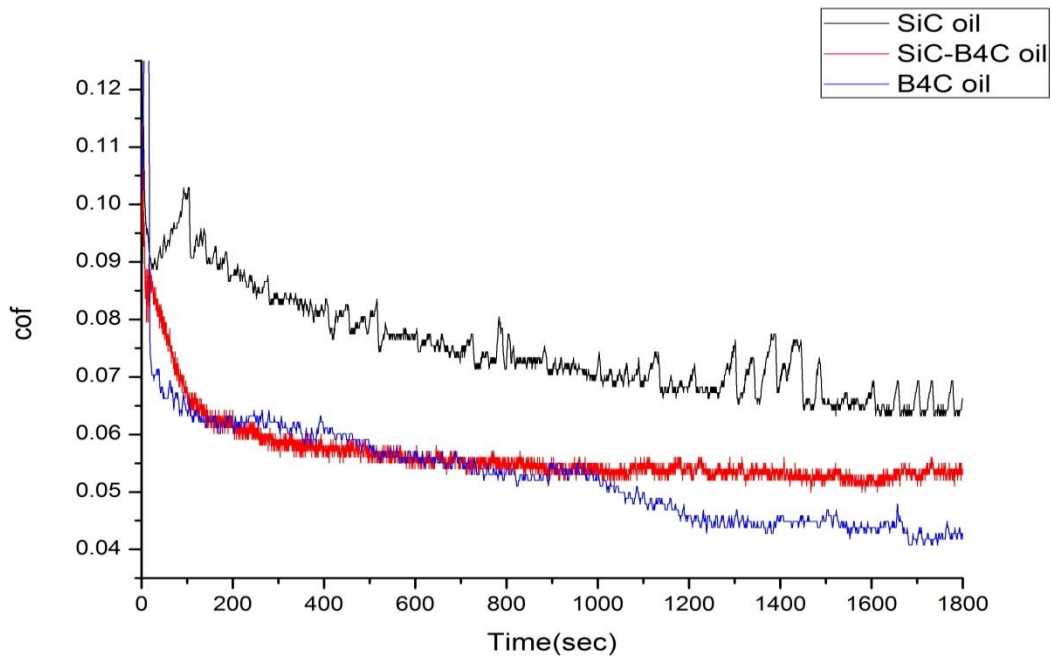


Figure 4.15 COF comparison of All Composites in lubricated conditions

This is clearly depicted in the friction plot (Figure 4.15). In lubricated sliding the smooth surface of pin shows the less wear out as compared to dry due to the fact that in this case metal to metal contact decreases due to oil film present in the asperities and transformation of severe wear regime to mild wear regime. Moreover, generated heat is dissipated in oil, temperature of the junction is decreased, thereby lead to the formation of weldments, thereby less adhesive and in case of oil mode the boundary lubrication regime changes to mixed and hydrodynamic regime and oxidation is reduced due to formation of lubricating film on the surface. In all the cases, especially in Al-B₄C and Al-SiC-B₄C

composite there is stable tribofilm formation after which the friction force attains the steady state constant condition. The layer is more stable in case of Al-B₄C composite. The smoothing effect of wear tracks as observed by the SEM micrographs in Figure 4.16 also suggests the same. Figure 4.16 shows the less wear out of the pin and smoothing of the surfaces as compared to dry sliding test surfaces. However, in case of Al-SiC composite due to the poor bonding of SiC in matrix and resulting third body abrasion the wear is still large as depicted by the large fractured surfaces on the worn surfaces of the pins (Figure 4.16b).

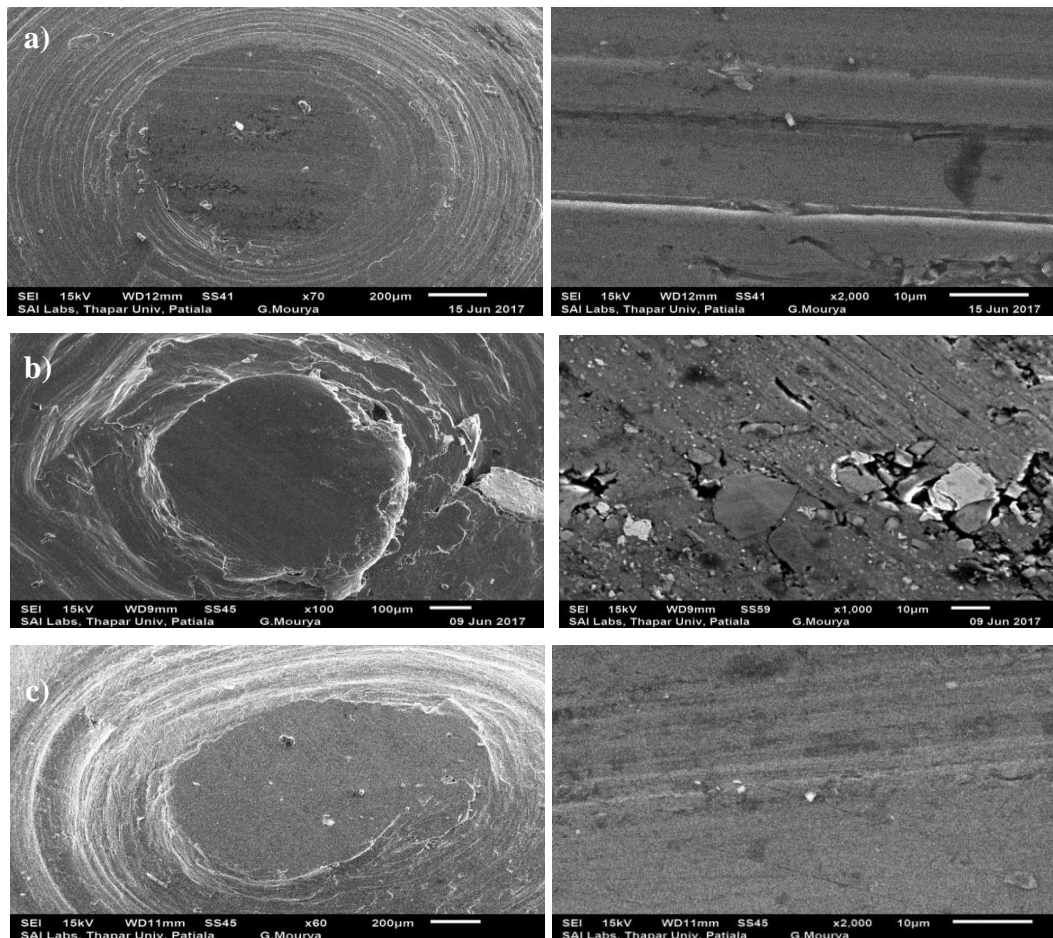


Figure 4.16 SEM images on the wear tracks of composite pins under lubricated sliding wear test (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

Figure 4.17 shows the EDS on the worn surfaces of the different composites. The elemental compositions show the existence of boron in substantial amount in case of Al-B₄C and Al-SiC-B₄C composites. On the other hand, Al-SiC can neither preserve the reinforced SiC intact nor it can sustain under rubbing to form permanent layer between the contacts, so that it can prevent the wearing out of the bare matrix. The large presence of bare aluminium

in Table 4.6 clearly indicates the possibility of the above fact. This is further verified following the XRD results obtained from the wear tracks of the composites.

Figure 4.18 shows the XRD spectra taken on the wear track of pins for different composites. As proposed earlier we can observe boron rich Al-B phase along with the oxide layers on the wear tracks of the Al-B₄C composite. The tribological response in case of Al-B₄C composite is enhanced by the formation of boron-rich Al-B phase at the grain boundaries films or layers with a reduction in the interfacial strength.

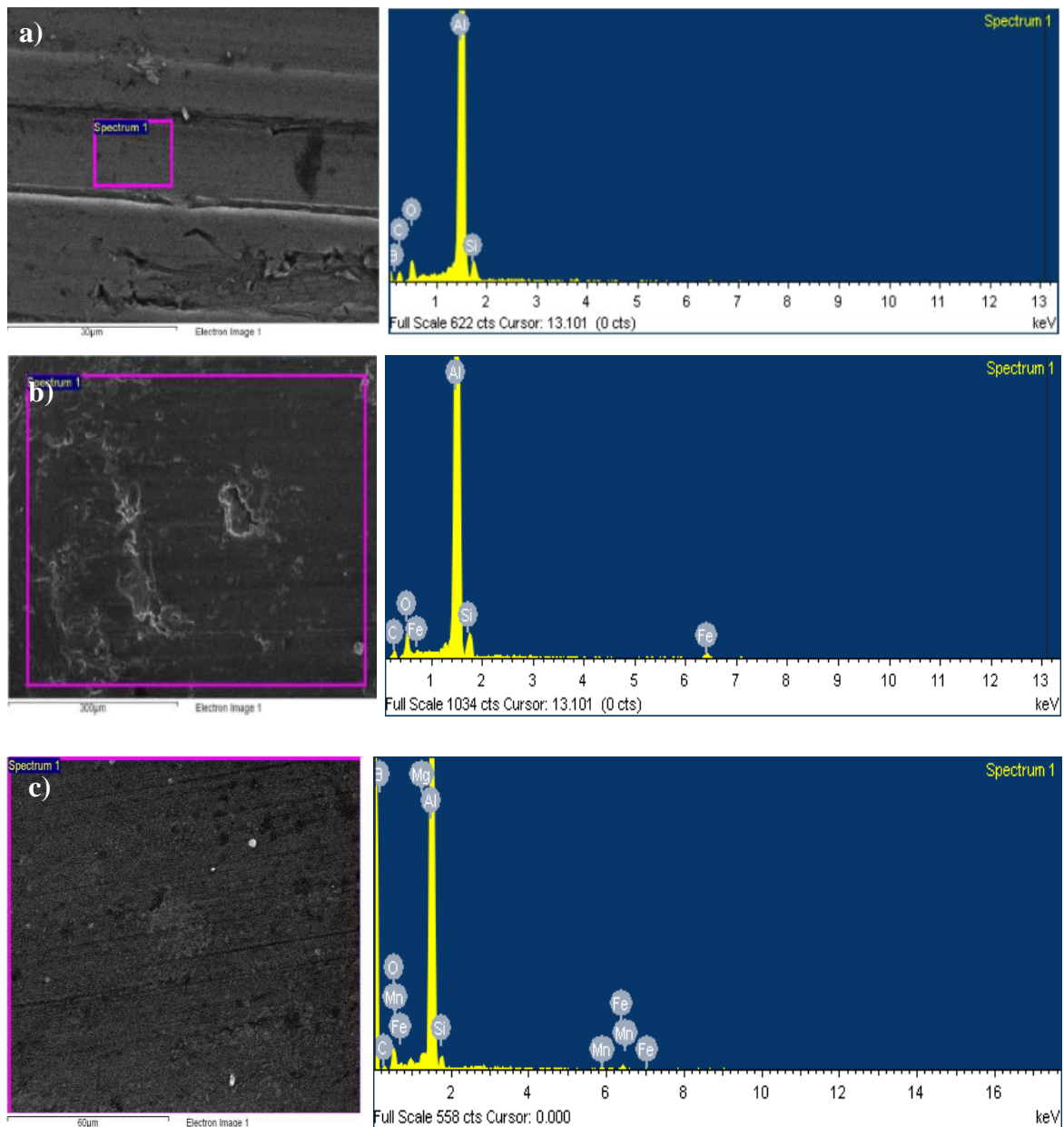


Figure 4.17 EDS on the wear tracks of composite pins under lubricated sliding wear test (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

Table 4.6 Elemental compositions (wt %) of the surface of the pin wear track of various composites under lubricated sliding test

Element	Al-B ₄ C in oil	Al-SiC in oil	Al-SiC-B ₄ C in oil
B K	46.18		33.62
C K	16.90	11.69	12.23
O K	6.26	9.79	4.35
Mg K			0.16
Al K	28.89	70.04	34.81
Si K	1.33	4.57	0.72
Mn K			0.20
Fe K		3.91	1.34
Totals	100.00		

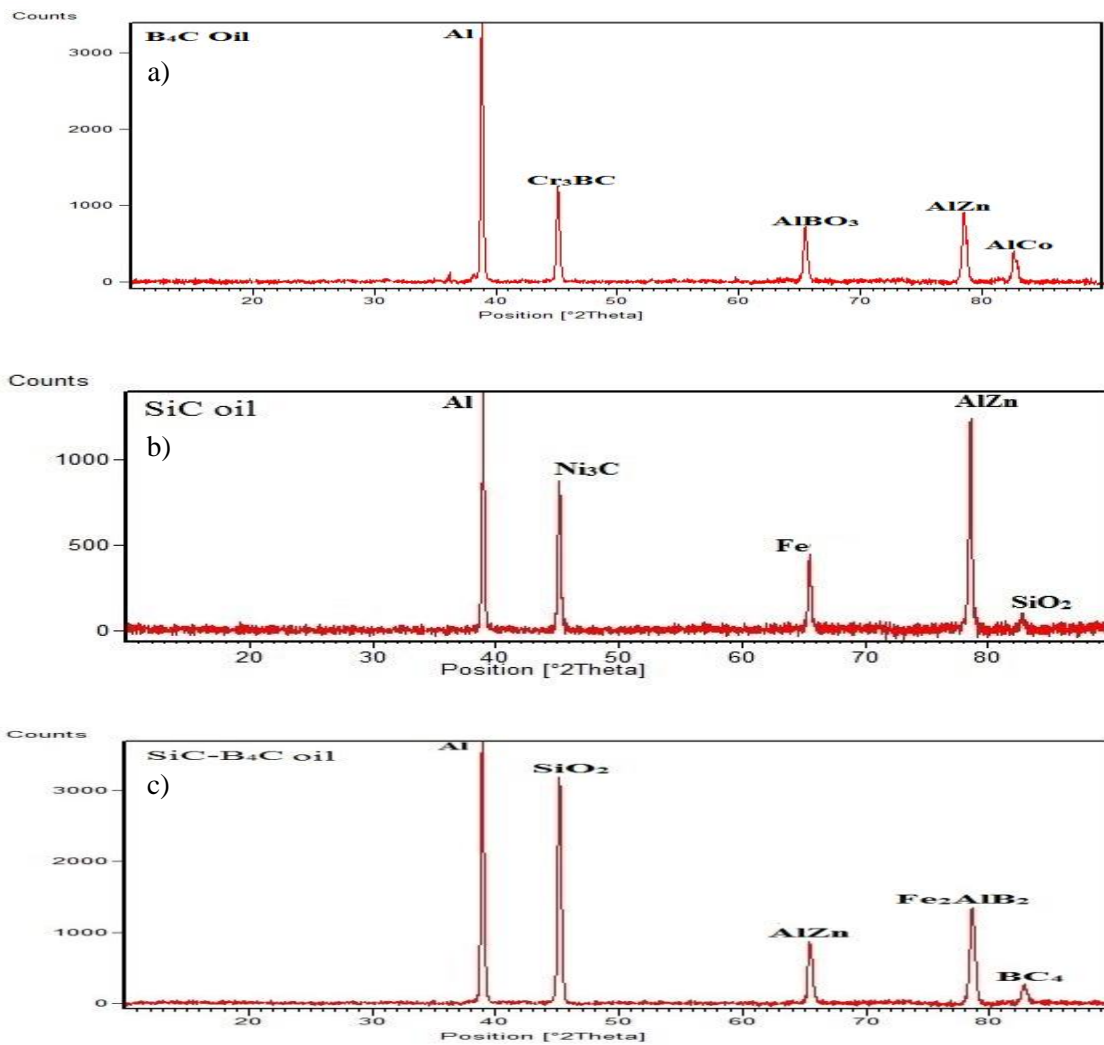


Figure 4.18 XRD for wear tracks on (a) Al-B₄C (b) Al-SiC (c) Al-SiC-B₄C

Formation of the surface coatings of oxidized and carbide layer further enhances the tribological characteristics of the composite pins. As a result both the wear rate and the coefficient of friction are reduced. The outcome of these layers is correctly illustrated by the experimental response of low and stable friction values as shown in the Figure 4.15

4.4.3 LUBRICATED SLIDING TEST WITH PARTICLE ADDITIVES

Following the most favorable results obtained from the dry and lubricated sliding test, this section focuses on the further formulation of lubricant by adding particle additives. Depending upon the previous results, Al-B₄C composites are used for further investigation involving various particle additives. Three different particle additives are selected for the study viz, Boric acid, MoS₂ and MWCNT. These particles can act as the solid lubricant when they are used in dry sliding.

Table 4.7 shows the coefficient of friction and wear rate for composites under lubricated sliding test in the presence of particle additives. As shown in the table 4.7, both cof and wear rate of composites is significantly reduced as compared to the dry sliding test or wet sliding test without any additives. The lamellar structure of boric acid and MoS₂ helps in reduction of the interfacial strength leading to the low coefficient of friction.

Table 4.7 Coefficient of friction and wear rate for composites under lubricated sliding test

S.No.	Sample Name	Condition	$\mu \pm \sigma$	Wear rate(mm ³ /m)
7	B ₄ C 10%	Oil+Boric acid	0.039± 0.0083	1.96x10 ⁻⁰⁵
8	B ₄ C 10%	Oil+MoS ₂	0.046±0.0048	3.44x10 ⁻⁰⁵
9	B ₄ C 10%	Oil+MWCNT	0.029±0.0070	1.27x10 ⁻⁰⁵

Besides, under loading these particles gets fragmented and forms continuous or discontinuous mechanical layer which further helps in decreasing the wear rate of the composite surface, by preventing them from the hard counter surface. On the other hand, MWCNT due to its structural stability and load bearing capability is found to be most effective in enhancing the tribological behavior of the composite under investigation.

The worn surfaces shown in the Figure 4.19(a) clearly shows that the grooves are much smaller than the previous tests without additives. In case of runs with boric acid we can see formation of a protective film layer of boron compounds. These layers of boron compounds increases the dynamic stability and thermal resistance of sliding metal by

preventing rupture it leads to decreased wear as these layers formed on the surface of metal slides against each other and prevents wear. Moreover, lamellae of boric acid align themselves parallel to direction of relative motion and slide over each other easily. Hence, with boric acid and oil combinations the surfaces were smooth.

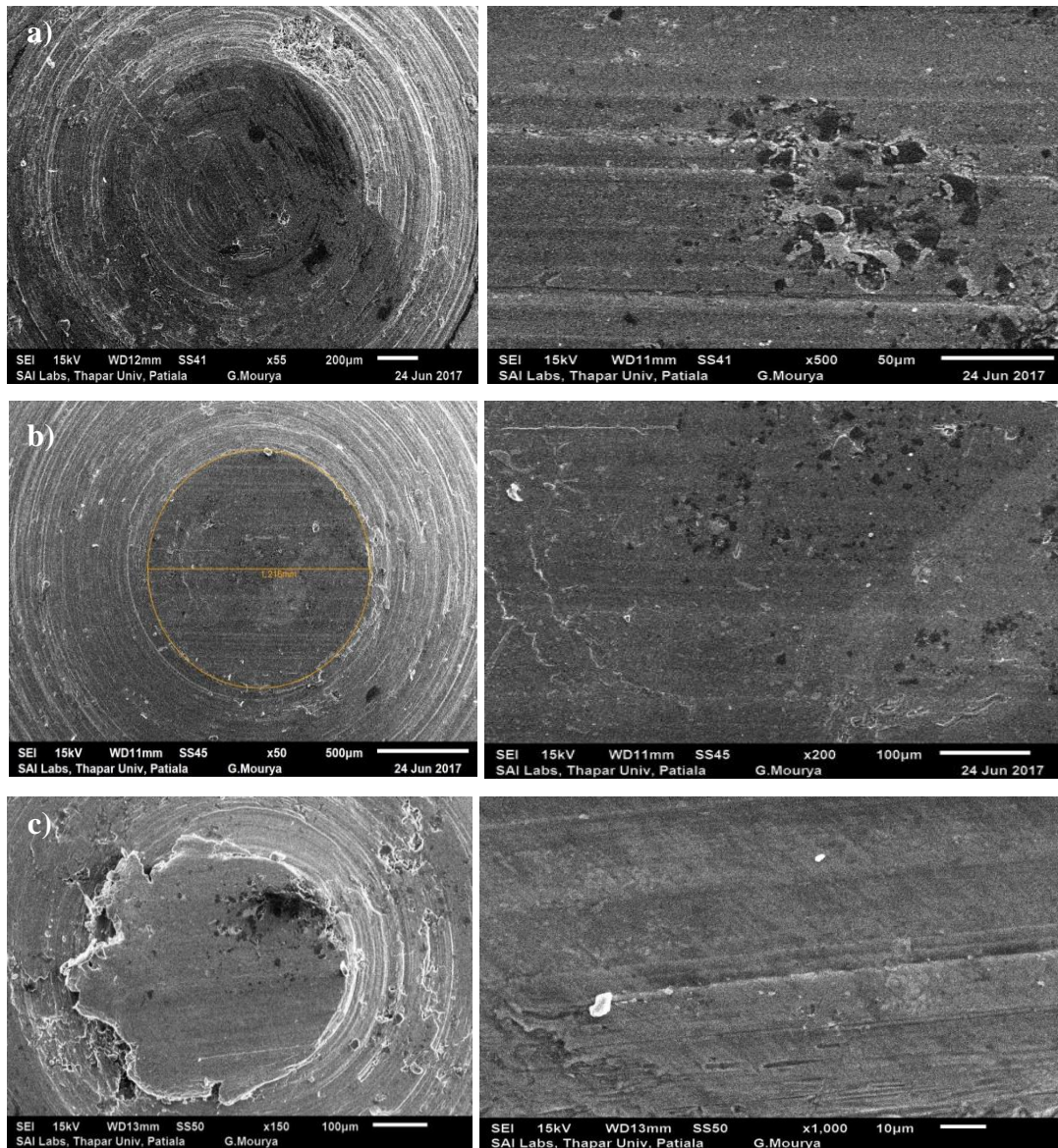


Figure 4.19 SEM images on the wear tracks of composite pins under particle aided lubricated sliding wear test (a) Boric Acid (b) MWCNT (c) MoS₂

Figure 4.19(b) shows the wear tracks for B₄C composite tribotested under lubricated sliding with MWCNT. MWCNT has high aspect ratio and high mechanical strength. CNT's can bridge crack and lock propagation of cracks and reduce the wear to some appreciable amount. Dislodgement of individual graphene layers and this layering of CNT will provide

lubricious effect and chain mobility of MWCNT's supports less fractured surfaces and in case of MWCNT dispersed in oil, CNT can restrain metal/ceramic particles and make material denser. This further enhanced the contact strength of composite and reduces wear than in dry condition. MoS₂ is found to be least effective as shown in Figure 4.19(c) may be due to its size and morphology, which ranges from submicron to the micron size, whereas the selected boric acid and MWCNT are nanosized.

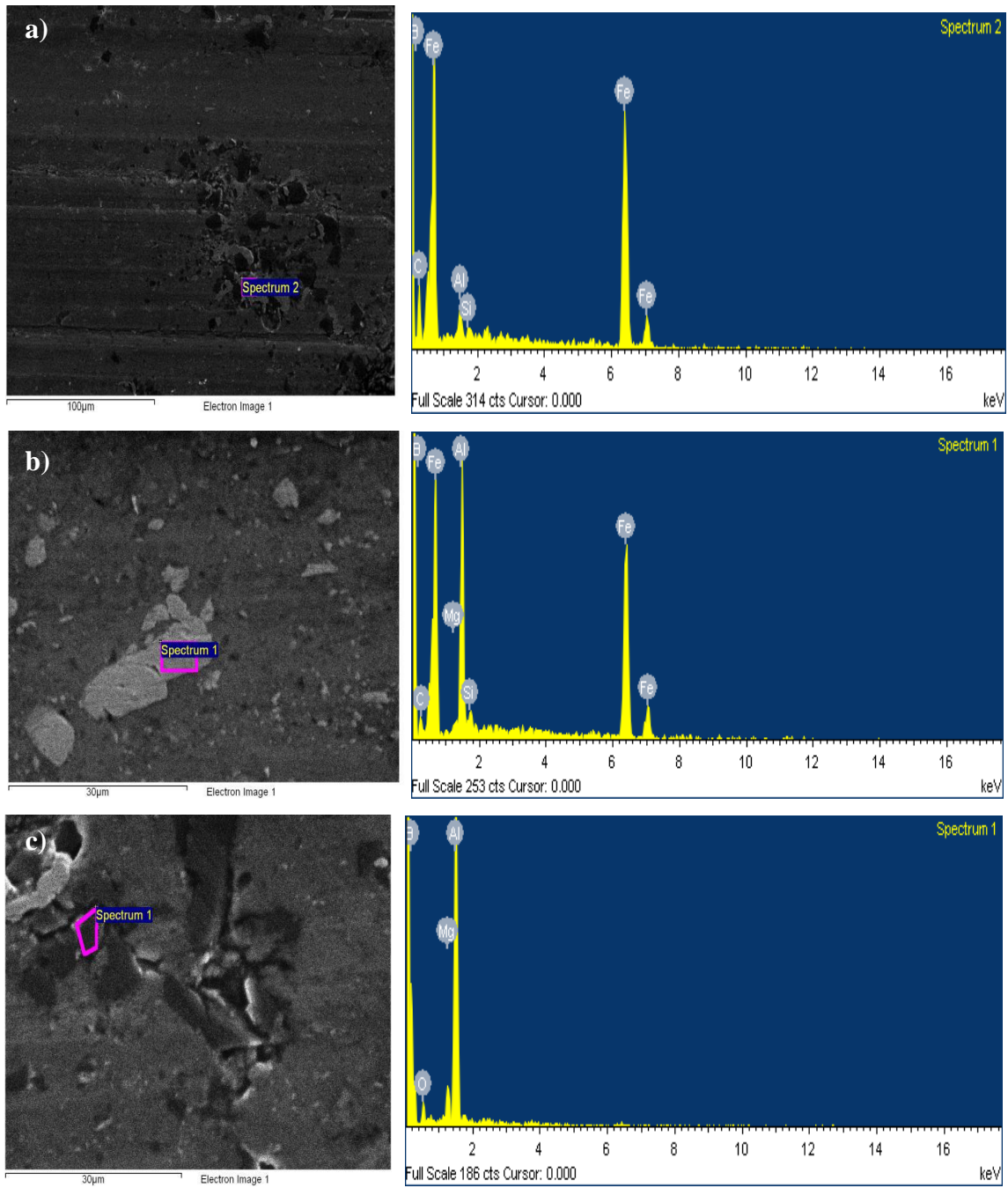


Figure 4.20 EDS on the wear tracks of Al-B₄C composite pins under particle aided lubricated sliding wear test (a) Boric Acid (b) MWCNT (c) MoS₂

Table 4.8 Elemental composition (wt %) of the surface of the pin wear track of B₄C composite under particle based lubricated sliding test

Element	Al-B ₄ C in oil+Boric	Al- B ₄ C in oil+MWCNT	Al- B ₄ C in oil+MoS ₂
B K	28.81	29.83	89.98
C K	24.28	13.77	
O K			2.90
Mg K		0.02	0.80
Al K	0.73	9.28	10.32
Si K	0.24	0.50	
Fe K	45.94	46.60	
Totals			

Fig. 4.20 shows the EDS results taken on the selected regions of the wear tracks of the worn out pins. The spectra show the presence of huge presence of boron in all the samples. In case of runs with boric acid and MWCNT the significant presence of Fe can also be observed which indicates the possible chemical or mechanical interaction of the surfaces and particles and Table 4.8 shows the elemental composites obtained from these spectra. Figure 4.21 shows the XRD patterns for wear tracks of Al-B₄C composites of worn out surfaces. In case of runs with boric acid enough percentage of boron leads to formation of boron compounds like boron oxide hydroxide (a form of boric acid), aluminium iron and aluminium hydroxide. The XRD peaks on the wear track of the samples ran with boric acid shows the significant presence of boric acid and AlO(OH) which are lamellar in nature. Also due to the interaction of aluminum and iron from the contacting surface the tribochemical action results in a compound called AlFe which is having very low shear modulus. This indicates that the tribo layers formed with these chemical are good enough to prevent the wearing out of the composite surface during sliding against hard EN31.

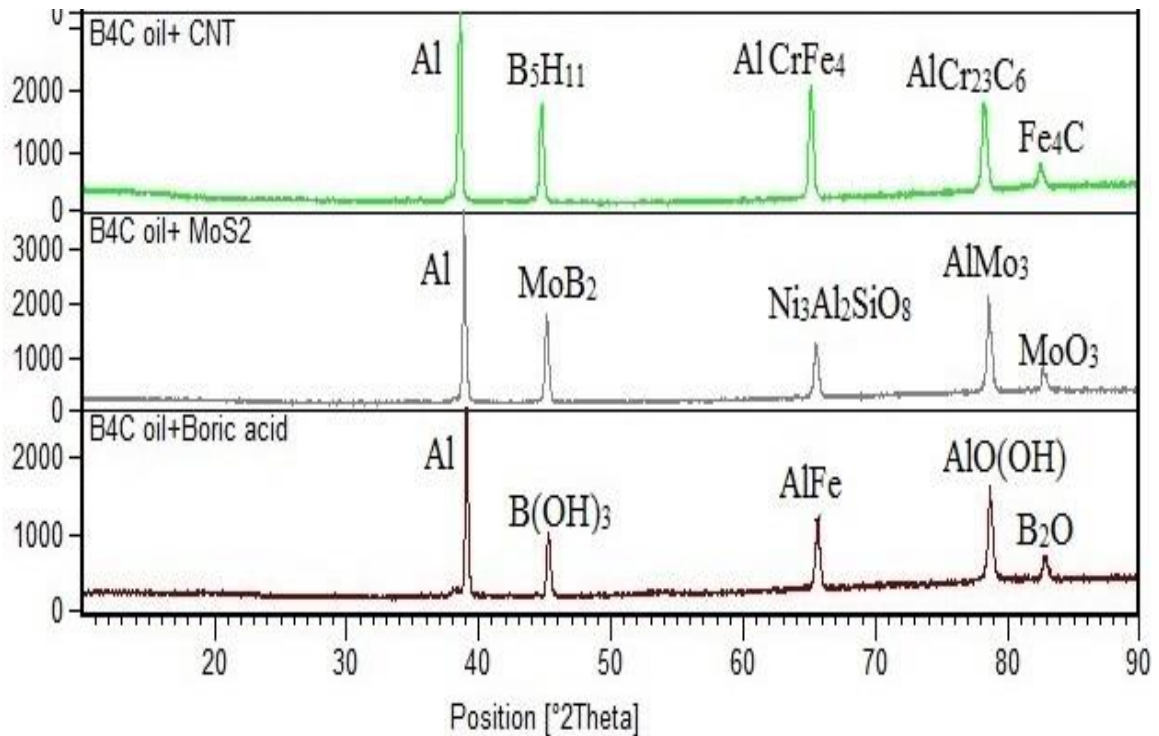


Figure 4.21 XRD on the wear track of Al-B₄C composite slides under particle-aided lubricants

MoS₂ has the properties like structure of MoS₂ is close packed hexagonal laminar structure. Due to weak Vander walls forces in lamina, it provides easy shearing during sliding; thereby reduce wear as well as friction but these features cannot be implemented because of the reason that no presence of MoS₂. In case of MoS₂ additive, Mo compounds like molybdenum boride, molybdenum oxide and aluminium molybdenum are formed and due to presence of nickel in EN 31 disc nickel compound is formed known as nickel aluminium silicon oxide. Thus, when MoS₂ is used as additive the tracks are exposed to the hexagonal structured MoB₂ and MoO₃, besides the other intermetallics. This helps in the reduction of interfacial strength which in turn reduces the friction and the coating prevents the wearing out of underneath substance. In case of MWCNT additives the enough percentage of carbon leads to the formation of carbon compounds such as aluminium chromium carbide, iron carbide and new form of boron compound is formed. However, in case of the MWCNT although the chemical peaks does not clearly indicates the mechanism of the reduction of friction and wear, however, considering its microstructure and superior mechanical properties, its superior tribological properties can be easily predicted and can be regarded as the potential additive for composite lubrication.

4.4.4 WORN SURFACE ANALYSIS OF COUNTER DISC FOR THE SELECTED TEST RUN

To validate our theory proposed during the investigation of wear mechanism in the presence of additives further investigation is carried out on the wear tracks of counter disc made of EN31.

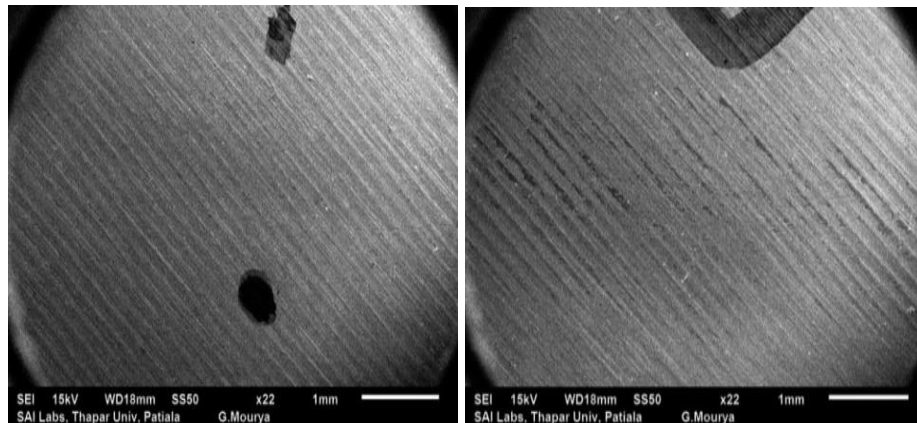


Figure 4.22 SEM images on the wear tracks of EN 31 Disc (a) Boric Acid (b) MWCNT

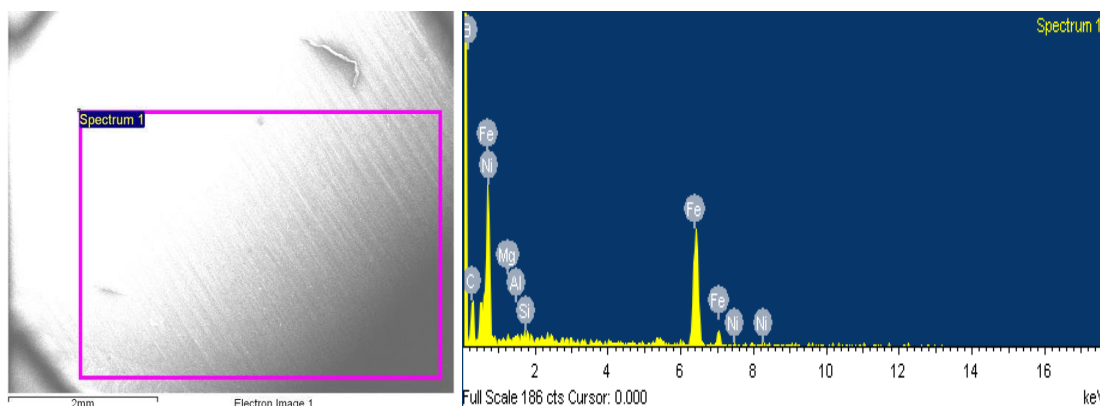


Figure 4.23 EDS on the wear tracks of EN 31 Disc (in the presence of oil and MWCNT)

Figure 4.22 shows the SEM micrographs of wear tracks on the disc which depicts that there was very less wear out in case of disc as this steel is very hard as compared to the composite. As seen in the micrographs no significant abrasion or adhesive wear can be found on the counter surface. This is probably due to the fact that hard abrasives from the pin rarely comes in direct contact with the disc surface due to the presence of tribofilm formed in-between.

Table 4.9 EDS Composition of the wear tracks on En 31

Element	Boric Acid additive	MWCNT additive
B K	36.06	50.51
C K	32.30	40.73
Mg K	0.25	0.16
Al K	0.18	0.10
Si K	0.18	0.10
Fe K	30.80	8.35
Ni K	0.23	0.06
Totals	100.00	

Figure 4.23 and Table 4.9 shows the results of EDS taken on the selected region of wear tracks on disc. The abundant presence of both boron and carbon along with the relatively low amount of Fe on the track clearly indicates whether the transfer of B_4C from the reinforcement of composites to the disc surface or the formation of transfer layer on the disc surface. As it is already discussed and illustrated with evidence that there is the formation of tribolayers of various boron and carbon compounds on the pin surfaces which not only prevented the wearing out of pin surface but also the wearing out of counter disc, hence the high possibility of tribofilm transfer on the surface of disc is clearly depicted.

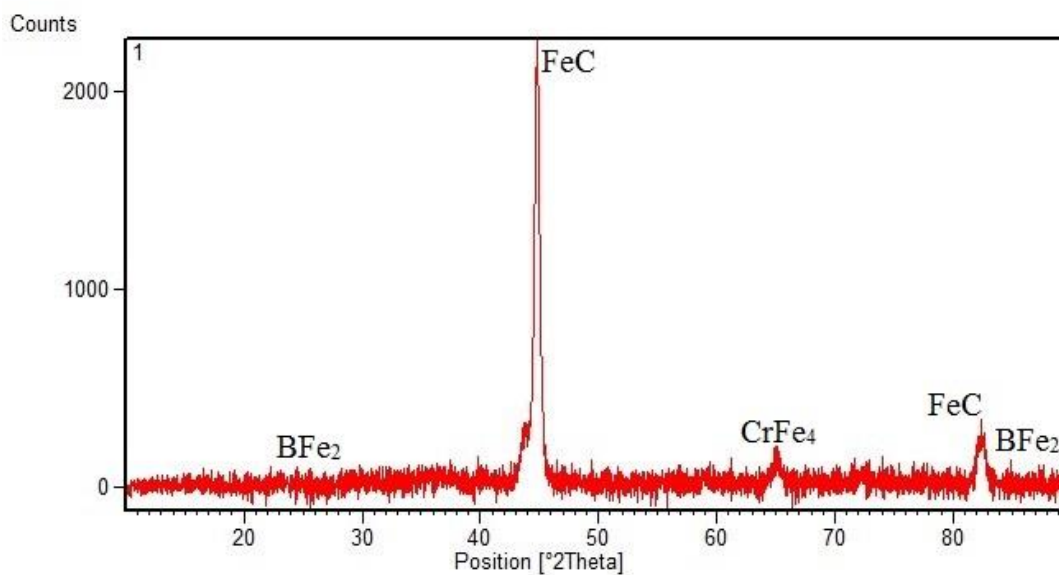


Figure 4.24 XRD for EN 31 Disc on sliding conditions of various composites

4.4.5 COMPARISON OF FRICTION AND WEAR BEHAVIOUR OF AL-B₄C COMPOSITE UNDER DIFFERENT CONDITIONS

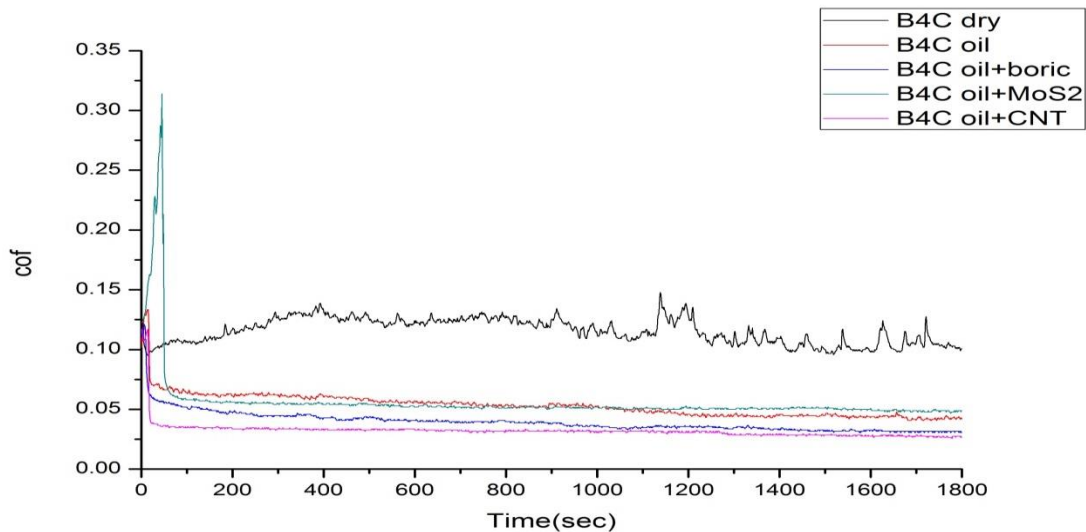


Figure 4.25 COF comparison of B₄C in different conditions

The variation of coefficient of friction for B₄C composites under dry and wet conditions with particle additives with constant parameters of 1kg load and 0.5m/s sliding speed are shown in Figure 4.25. It shows there is significant decrease in the value of the cof for the B₄C composite from dry conditions to wet conditions in the presence of particle additives. The possible causes of friction reduction are discussed in the previous sections. In case of dry condition the cof is more as compared to the lubricated and particle based lubrication because under lubricated condition the thin film of lubrication or tribofilm is formed between the tribo-pair of Al-steel. In case of runs with MoS₂ as additive the value of cof is nearly equal or slightly greater than the oil conditions due to the formation of other less tribo-favorable Mo compounds. As these films carries particles they are fragmented into layers, reorient themselves along sliding plane or forms chemical compound with low interplanar bonding which leads to lower cof. In case of base oil with boric acid dispersion it was noticed that, initially boric acid gets evenly distributed in oil after a run-in time, which subsequently is able to provide a continuous tribolayer with low shear resistance in the contact interface acting as an excellent lubricant. The boric oxide film continuously replenished boric acid to sliding contact under ambient conditions. In the case of CNT

dispersion in oil, the nanodimensions of the particle and structural stability influences the friction as well as wear.

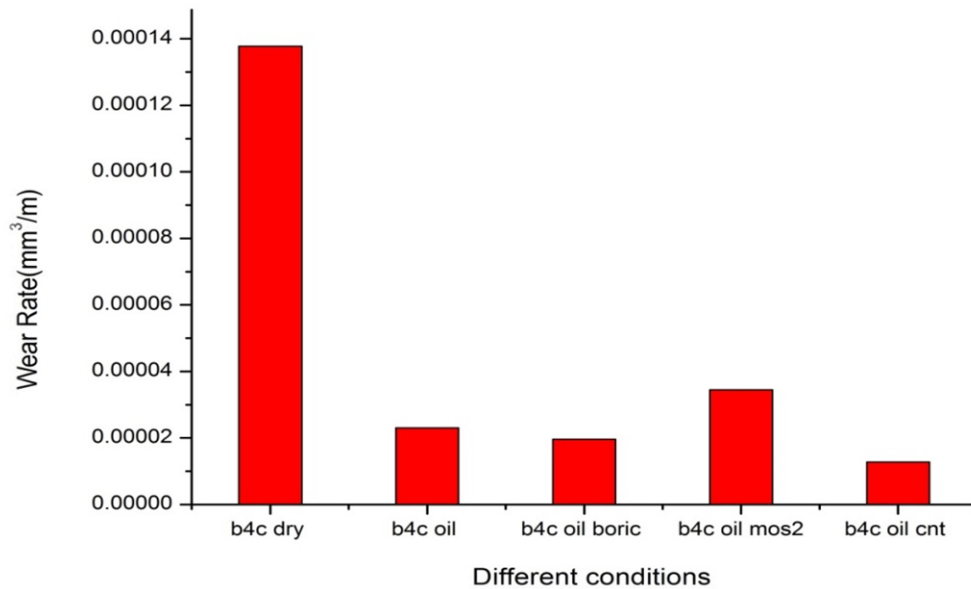


Figure 4.26 Wear rate for B₄C in different conditions

Figure 4.26 shows the wear rate for B₄C composites in dry sliding, lubricated sliding without additive and lubricated sliding in the presence of various particle additives. It is observed from the above figure as well as from our previous discussion that in lubricated conditions wear rates are lower than dry condition for the composite. Further reduction of wear can be made by suitably selecting the additives. Due to the increased load carrying capacity and thermal and structural stability nano/micro particle additive can be used. From our study CNT and boric acid emerged out as potential additives for the tribological applications of Al-B₄C composites too.

Chapter 5

CONCLUSIONS

5.1 CONCLUSIONS

This chapter summarizes the work done during the present research and key findings and outcome achieved from it. Three different types of composites were fabricated by stir casting method and their mechanical and metallurgical properties are investigated. Then these composites are examined for their tribological characteristics under dry and lubricated contact with steel. The most tribologically favored pair is then subjected to the particle based lubrication for further enhancement of tribological response against the selected counterpart. For the particle based lubrication three different types of particles (Boric acid, MoS₂ and MWCNT) is selected. Boric acid as additive has been identified as the potential solid lubricant due to its lamellar molecular structure and its abundance and with no harmful effects to human body.

After going through the results and discussion in the previous chapter several conclusions can be drawn which are as follows:

- The Al composite reinforced with B₄C showed the superior hardness than Al-SiC and Al-SiC-B₄C. However, the hardness of hybrid composite is found to be comparable to Al- B₄C with same amount of reinforcement.
- The uniform distribution in these composites leads to improved hardness. SEM and optical micrographs favor the fact of uniform distribution of particles in the matrix.
- In general, it is observed that wear rate reduces with increase in hardness of the composite. However, an exception is found in case of SiC-B₄C composite since this composite showed more wear out than Al-SiC composite under dry sliding. This is attributed to the fact that there is more clustering and interparticle interaction which leads to the weak bonding between the reinforcement and matrix.
- Friction studies showed that the presence of intermetallics, oxide layers and transfer film formation under various conditions which decreases the coefficient of friction as well as wear rates. The XRD and EDS analysis confirms the presence of various phases of compounds present in the wear tracks.

- Besides the compound formed due to tribochemical action, the layered structure of boric acid and the mechanical and structural stability of the CNTs are believed to be the primary cause of friction and wear rates.
- Dry sliding wear test against EN31 suggests that we can reduce the friction by 47% and wear by 72.2% if we select B₄C as the reinforcement in AMMC instead of SiC. However, the hybridization does not give any potential benefit for dry sliding behavior.
- Lubricated sliding reduces the coefficient of friction from corresponding dry sliding by 53%, 65.4% and 78.5% for Al-B₄C, Al-SiC and Al-SiC-B₄C composites, respectively.
- Lubricated sliding reduces the wear rate from corresponding dry sliding by 83%, 87.8% and 92% for Al-B₄C, Al-SiC and Al-SiC-B₄C composites, respectively.
- The effect of additives dispersed in oil helps in reducing the μ and wear rate of Al-B₄C composite significantly.
- From the present investigation, CNT and boric acid emerged out as potential additives for the tribological applications of Al-B₄C composites, whereas MoS₂ showed no prominent effect to be used as potential additive for wet tribology involving AMMC.
- Particle aided lubricated sliding of Al-B₄C reduces the coefficient of friction from corresponding lubricated sliding by 27%, 46% and 14% when Boric acid, MWCNT and MoS₂ is used, respectively.
- Particle aided lubricated sliding of Al-B₄C reduces the wear rate from corresponding lubricated sliding by 14.7%, and 44% when Boric acid and MWCNT respectively, but in case of MoS₂ it was increased to 33.13%.

5.2 SCOPE OF FUTURE WORK

- Further investigation on the tribological behavior can be made using different reinforcement such as cubic boron nitride and other carbides under lubricated sliding.
- The influence of the size, shape and surface functionalized particles on the composite tribology can be investigated, so that to achieve very low friction and wear rate we can focus on lubricant design for any given economically designed composite.
- Further study on the tribology of these composite for high temperature application can be carried out.

- Multiscale modeling on the mechanical and wear behavior can be put into investigation for liquid lubrication of composite.

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LIST OF PUBLICATIONS

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