Mechanical and Tribological Behavior of Al_2O_3, B_4C and MoS_2 Reinforced Al Composite Processed Through Powder Metallurgy

Lemi Kansu Tuge

A Thesis Submitted to the Department of Mechanical Engineering

College of Mechanical, Chemical and Material Engineering

Presented in Partial Fulfillment of the Requirement for the Degree of Master's in Manufacturing Engineering

Office of Graduate Studies

Adama Science and Technology University

June, 2025

Adama, Ethiopia

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DECLRATION

I hereby declare that this master thesis entitled ‘‘**Mechanical and Tribological Behavior of Al_2O_3, B_4C and MoS_2 Reinforced Al Composite Processed Through Powder Metallurgy**’’ is my own work. That is, it has not been submitted for the award of any academic degree, diploma or certificate in any other university. All sources of materials that are used for this thesis have been duly acknowledged through citation.

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Name of student	Lemi Kansu Tuge
Id. No.	PGR/35837/16
Department	Mechanical Engineering
College	CoMCME
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Title of work / Assignment name	Mechanical and Tribological Behavior of Al_2O_3, B_4C and MoS_2 Reinforced Al Composite Processed Through Powder Metallurgy
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ACRONYMS AND ABBREVATIONS

AMMCs	Aluminum Metal Matrix composites
ASTM	American Society for Testing and Materials
B_4C	Boron Carbide
COF	Coefficient of Friction
EDS	Energy dispersive X-ray spectroscopy
FWHM	Full width at half maximum
GRA	Grey relational analysis
HV	Vickers hardness
MMCs	Metal Matrix Composites
MMC	Metal matrix composite
MoS_2	Molybdenum disulfide
MWCNTs	Multi-Wall Carbon Nanotubes
OM	Optical microscope
Pc	Compaction pressure
PM	Powder Metallurgy
SEM	Scanning Electron Microscope
Ts	Sintering temperature
wt.%	weight percentage
XRD	X-Ray Diffraction

ABSTRACT

Nowadays, lightweight materials with better mechanical and tribological properties are highly demanded in different industries such as automotive and aerospace. Aluminum is a preferred matrix material due to its low density, but its lower hardness and strength restrict its use in structural and high-load applications in comparison to steel and titanium materials. This study focused on improving the properties of pure Al by reinforcing it with aluminum oxide (Al_2O_3), boron carbide (B_4C), and molybdenum disulfide (MoS_2) using the powder metallurgy (PM) process. The composites were fabricated by varying the weight percentages of Al_2O_3 (2,4,6 wt%), B_4C (2,4,6 wt%), and keeping MoS_2 constant at 4 wt%.The properties like hardness, compressive strength and wear resistance were investigated. The optimal composite revealed that hardness 224.8HV, 390.23MPa compressive strength and 32% improved wear resistance were obtained. By utilizing Archimedes method the porosity and density of the composite were determined. The corrosion resistance of sample was examined through electrochemical cell. The analysis of microstructure was carried out using X-ray diffraction (XRD), optical microscope (OM) and scanning electroscope (SEM). The powder metallurgy process parameters and weight percentage of reinforcement particles were optimized through Taguchi-based grey relational analysis. The enhanced mechanical and tribological properties were observed on the sample fabricated at the optimum conditions. The reinforcement's uniform distribution was demonstrated by the OM and SEM data. The existence of various phases in the composite was verified by the XRD test. The incorporation of boron carbide, aluminum oxide and molybdenum disulfide in to aluminum metal matrix exhibited the enhanced mechanical and tribological properties making it suitable for engineering applications like automotive and aerospace industries.

Keyword: Aluminum metal matrix composite, Tribological Behavior, GRA, B_4C.

CHAPTER ONE

INTRODUCTION

1.1 Background of the study

In recent years, one of the most sought-after research areas in modern technologies is composite materials. Composite materials are being more and more utilized in engineering and material science fields. Because, it has high combination of stiffness and toughness, with light weight, and better mechanical and structural properties such as high strength to weight ratio, fire resistance, corrosion, and wear resistance, and low cost production. Due to their superior properties, they are implemented in different industrial sectors, including sports, biomedical, automobile, aerospace, and construction. The term composite material means it is made up of several different materials. It was made by combining two or more component parts that have markedly various chemical or physical properties in order to generate a material with special properties higher than the component parts. As a result of such enhancement, composite material surpasses individual material in terms of properties (Rajak et al., 2019)**.**

Composite materials are of various types; three types are based on matrix. Metal matrix composites, polymer matrix composites and ceramic matrix composites are those. Because MMCs are better than PMCs and CMCs, they are used most frequently in industrial uses. MMCs have better performance than PMCs in a variety of key mechanical and physical characteristics, such as high strength and stiffness, wear resistance, stability on dimensional, and the ability to support elevated temperatures. good electrical and thermal conductivity (Sharma et al., 2020*)*.

Currently, commonly used matrix metals are Aluminum, Copper, Iron, Magnesium, Nickel and Titanium (Sharma et al., 2020). Among various types of metal alloys, aluminum alloys are extremely popular as matrix materials due to their excellent corrosion resistance, light weight, and good electrical and thermal conductivity. The need for high-strength, light-materials also prompted the development of new aluminum-based MMCs using a variety of techniques (Abebe Emiru et al., 2023). The synergy of high specific strength, stiffness, enhanced corrosion and wear properties, low density, and other properties makes aluminum-based metal matrix composites suitable for application in different engineering sectors, including the automotive, aerospace, and defense industries. Aluminum MMCs, having enhanced mechanical and tribological properties, are reinforced with a variety of ceramic particulates, including Al_2O_3, TiC, TiB_2, SiC, B_4C, etc. (Samal et al., 2022). In addition, due to its low density special strength and stiffness, excellent casting property, and abundant resources, magnesium composites are a very important lightweight matrix composite only next to aluminum matrix composites. They have better application value in automobile, aviation, and biomedicine materials. Yet, magnesium's poor ductility, poor fatigue resistance, and poor corrosion resistance prevent it from being widely applied in important technical fields (Ren et al., 2024).

Metal matrix composites can be made in a variety of ways. Solid state processes include diffusion bonding, friction stir process, high energy ball milling, vapor deposition, and powder metallurgy (PM route); process of liquid state contains stir casting, spray casting, compo casting, squeeze casting, and in situ (reactive) processing. Powder Metallurgy route (PM route) is the most effective where particulate reinforcement and matrix can be in the powder form **(**Saravanan et al., 2015). Besides, it is the method applied to manufacture magnesium or aluminum MMCs. It needs less temperature in PM; hence it is economical (Singh, & Saxena, 2020).

With pure Al as matrix and Al_2O_3, B_4C, and MoS_2 as reinforcement material, the current work was implemented on the development of Al metal matrix and examined the influence of the particles on the mechanical and tribological properties of composites. Because of its high wear resistance, chemical stability, and high hardness Al_2O_3 was chosen as reinforcement (Abyzov, 2019). Because of its better hardness and density, B_4C also been selected as a reinforcement. MoS_2 is a transition metal dichalcogenides that are known to possess outstanding physical, chemical, and electronic properties with superior strength, flexibility (Samy et al., 2021).The composite was manufactured by using powder metallurgy method.

1.2 Problem statement

Aluminum is a light weight metal and one of the most versatile and highly demanded engineering materials for various applications such as in aerospace, automotive and several others. But still the challenges are being faced by these industries as far as strength to weight ratio; wear and hardness are concerned. Because it’s limited wear resistance, strength, and hardness, pure aluminum's application in high-load applications and high-temperature conditions was restricted. Incorporating synthetic ceramics into the matrix improves certain properties, but it also reduces ductility and increases brittleness in composite materials. Thus, we must address these issues and enhance the properties further so that it can be used in a variety of applications. Study on reinforced AMMCs has been extensive; however, more study is needed to obtain better performance. Therefore, in order to enhance the properties of pure Al and address the aforementioned issues, the Al_2O_3, B_4C and MoS_2 specifically have been proposed. Al_2O_3 generally has high hardness, resistance to wear, chemical stability, and, resistance to oxidation etc. Additionally, B_4C and MoS_2 are utilized in this study to further enhance the metal matrix's properties and provide a strong wettability impact. In this study, metal matrix composite (composed of aluminum oxide, Boron Carbide and molybdenum disulfide as a reinforcing particles and pure Al as a metal matrix) had been developed and tribological and mechanical behavior had been investigated and process parameters were optimized.

1.3 Objective

1.3.1 General objective

The general objective of this research is to investigate experimentally tribological and mechanical properties of aluminum metal matrix composite reinforcing by aluminum oxide (Al_2O_3), B_4C, and MoS_2 developed through powder metallurgy process.

1.3.2 Specific objectives

The specific objectives are as follows:

- To investigate the compressive strength, and hardness of the fabricated material.
- To characterize phase, morphology and reinforcements distribution for the developed composite material.
- To Examine the wear strength, and to investigate corrosion behavior of the developed material
- To investigate the density and porosity of the processed sample.

1.4 Significance of the study

This research focused on development of aluminum metal matrix composite reinforced with aluminum oxide (Al_2O_3), B_4C, and MoS_2. It was also examined the mechanical and tribological behavior of the composites. Thus, it plays a significant role for stakeholders at the national and international levels both practically and academically. In practical terms, it will be applied by industries for developing metal matrix composites made of aluminum. It will advance our theoretical and practical understanding of the fields of research and the industries that require these composites. To enable the creation of advanced materials for a variety of applications, it is essential to investigate this metal matrix composites capabilities and special qualities. Furthermore, it will be used by researchers as a reference in the future.

1.5 Expected outcome of the study

In this research, two major outcomes will be expected such as:

- Developed light weight, hard and wear resistant aluminum metal matrix composite reinforced with aluminum oxide (Al_2O_3), MoS_2 and B_4C and
- Improved corrosion resistant, compressive strength and uniform particle distribution of composite material suitable for engineering applications.

1.6 Scope of the study

The primary focus of the study was upon using powder metallurgy to develop aluminum metal matrix reinforced with B_4C, MoS_2, and aluminum oxide. For this study, a range of experiments had been conducted, such as processing $Al/Al_2O_3/B_4C/MoS_2$ by varying Al_2O_3, B_4C, weight percent and taking constant weight percent of MoS_2. Furthermore, the produced composites compressive strength, wear resistant, corrosion resistant and hardness were examined. Distribution of the phase and reinforcement particles had been determined. In addition to this the process parameters of powder metallurgy were optimized.

1.7 Motivation of Research

The need for advanced lightweight materials with outstanding properties are now days becoming more and more important in engineering applications. Aluminums low density and good corrosion resistance make them very desired for applications including, automotive, aerospace and biomedical engineering. But pure Al was hindered to widely use, as a result of lack of sufficient strength and resistance to wear. The objective of this study is to address these problems through fabricating Al composites with improved mechanical properties, wear resistance, and corrosion resistance, by reinforcing pure Al by aluminum oxide (Al_2O_3), boron carbide (B_4C), and molybdenum disulfide (MoS_2). High-quality composites can be produced in a manageable and efficient method by employing powder metallurgy, which provides uniform reinforcement distribution, controlled microstructure, and decreased porosity.

1.8 Limitations of the Study

Minimum pressure was used in compressing the samples. This can result in poor interaction of particles, which is a requirement for successful sintering. High porosity and low density can be produced as a result of low compaction pressure. Since the sintering process was performed in an uncontrolled environment; it might have caused thermal fluctuation or non-uniform heating and cooling rates which are the reasons for porosity. The uncontrollable state may also cause the development of new phases that may alter the composite properties. The small sample size may limit the generalizability of the influence of the proportion of reinforcing weight and powder metallurgy process parameter. Furthermore, a small sample size may provide an unclear result. This study was utilized powder metallurgy for the fabrication of the composite. It could be challenging to determine the impact of different processing techniques on the produced composite. Because different processing techniques can produce different microstructural and properties of mechanical, the observation of this study work might not be valid for other processing techniques. Also, the effect of powder metallurgy on aluminum metal matrix composites properties cannot be compared with other processing techniques.

1.9 Thesis Organization

This study was organized by five chapters and every chapters of this thesis was targeted to the experimental evaluation of the Al- Al_2O_3 -B_4C -MoS_2 composites fabricated through powder metallurgy technique. In chapter one problem statement, background of the study, significance of the study, objective of the study, expected outcome of the study, the study's scope, and motivation of research were described. Under chapter two research papers which are done on composites of aluminum metal matrix, and fabrication methods of composites of metal matrix were reviewed. Also literature summary was discussed, and literature gap was identified. In chapter three the experimental methods and the materials utilized was proposed. Additionally, the characterization and testing methods, also the experimental analysis method of fabricated sample were discussed under this chapter. In chapter four the results obtained were discussed briefly. Chapter five presents the conclusion, future work and recommendations.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This section elaborated on a wide range of published work done on aluminum and its alloys by different sources. It encompasses an overview of the influence of reinforcements on aluminum metal matrix composites. Moreover, the fabrication process was elaborated and research gap was determined.

2.2 Composites

By combining two or more distinct materials, a composite material was created for the purpose of improving their physical, mechanical, thermal, and electrical properties. In other words, it consists of materials that significantly vary in chemical or physical properties and when brought together, a material with particular properties is formed. Composites typically includes single continuous phase, referred to as a matrix, and one or more discontinuous phases, called by reinforcements. Thus reinforcing and matrix phases form up composite materials. Based on matrix it is classified into metal, polymer, and ceramic matrix. Fibers and particles are utilized in reinforcing phase (Aherwar et al., 2020), (Nagavally, 2016).

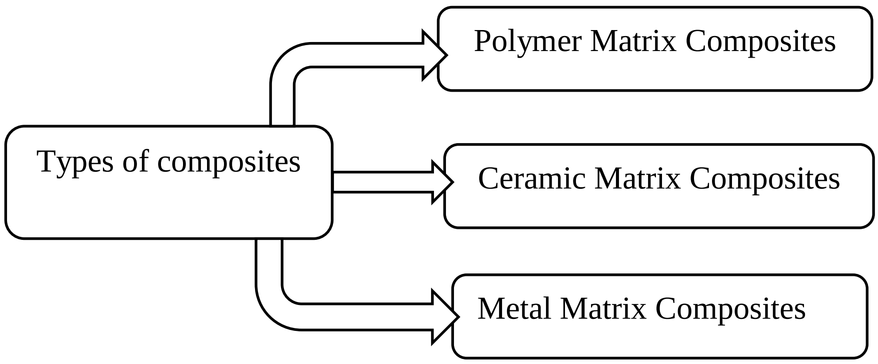

Figure 2.1Classification of composites depends on their matrix.

2.2.1 Polymer Matrix Composites

Polymer Matrix Composites (PMCs) have various applications because of their useful properties and cost-effective processing methods. Ceramic fibers are used for matrix strengthening to impart high strength properties of PMCs. Thermoplastic or thermosetting plastics used as matrix and carbon, glass, steel, and natural fibers as reinforcement (Aherwar et al., 2020). It possesses low weight, high strength, good impact and compression resistance. Owing to their good mechanical properties and simplicity in manufacturing, they are considered to be advanced composite materials. They also possess design flexibility and comparatively higher fatigue and corrosion resistance than many other materials (Kangishwar et al., 2023*)*.

2.2.2 Ceramic Matrix Composites

These materials consist of another ceramic matrix with ceramic particles, fibers, and whiskers. Glass, metals, polymers, and ceramics can all be used to strengthen the ceramic matrix. They possess high melting points, good compressive strength, excellent corrosion resistance, and high-temperature stability (Aherwar et al., 2020). It is highly demanded in high-temperature applications. Ceramic matrices possess a greater melting point, greater hardening properties, lower coefficients of thermal expansion compared to metal and polymer matrices. The major function of CMCs is to improve toughness as monolithic ceramics are brittle with high levels of stiffness and strength. Al_2O_3, B_4C, SiC and ZrO_2 are commonly used ceramic materials. These composite materials are used to develop a range of aviation engines; aircraft brake disks, high-temperature gas turbine components, and sliding bearing components (Dhanasekar et al., 2022).

2.2.3 Metal Matrix Composites

MMCs are generally acknowledged as sophisticated materials. Compared to traditional materials, MMCs have superior mechanical and thermal properties, high strength-to-weight ratio, improved fatigue and creep behaviors, better wear resistance (Chawla, 2016). Nowadays, Al, Cu, Mg, Fe Ni, and Ti are common matrix metals. The automotive and aerospace sectors use aluminum alloys due to its high strength, resistance to corrosion, low density, and adequate forming and joining capabilities. Ceramic particles are used to strengthen aluminum. As a result of its excellent resistance to corrosion and tolerable strength at high temperatures, titanium alloys are frequently utilized in aeronautical components. Magnesium also widely utilized in electronic devices due to the fact that it is the lightest of various non-ferrous metals and today increasingly in greater demand in the automotive, aerospace, and other structural markets (Anand & Senthil Kumaran, 2020). Copper is easy to shape and cast and is applied in electronics as parts and electrical contacts for systems. They can be utilized in aerospace, automotive and other ground transport (Aherwar et al., 2020).

2.3 Fabrication Methods of Metal Matrix Composites

2.3.1 Powder Metallurgy

The most important methods for processing MMC is powder metallurgy because of its ease of use and versatility (Baliarsingh et al., 2023*)*. A method which is a solid state utilized for the manufacture of magnesium or aluminum MMCs. Process variables are; compressing pressure, sintering time and sintering temperature (Sultan et al., 2024). PM is cost-effective method for the processing of complex parts with high strength, dimensional accuracy, lower scrap loss, and lower temperature (D. K. Sharma, Mahant, et al., 2019). This process enables simple production of multiple geometrical parts and mass production at economical cost. Steps involved in the process are; powder mixing, compaction of mixed powder and sintering process to achieve consolidation (Singh et al., 2020b). This method offers accurate shapes, satisfactory mechanical properties, high homogeneity and controlled porosity of components. Powder materials and tools are, however, expensive; besides, components are ductile and low-strength.

2.3.2 Stir Casting

The traditional method for creating discontinuous particle reinforced MMCs is stir casting, which is a liquid phase technique. It is a liquid phase process in which mechanical stirring is employed for injecting or mixing the reinforcement particles into the molten metal matrix melted above its melting point (D. K. Sharma, Mahant, et al., 2019). Process parameters regulating and determining the distribution of reinforcements within the matrix are: melting temperature, time for holding, time and speed of stirrer, stirrer position and geometry in the melt, solidification rate, and feed rate (Baliarsingh et al., 2023). Powders are the most often utilized reinforcing materials in this technique. This process is Simple, flexible, low cost, and appropriate for mass production, good interface for matrix reinforcement. However, achieving high porosity and homogeneity is challenging, and high temperatures can lead to poor wetting conditions and the potential for a reaction between the reinforcement and matrix (Singh et al., 2020b).

2.3.3 Squeeze Casting

Squeeze casting is the outcome of combining the forging and casting processes. When molten metal is compressed, it solidifies. Reinforcement combined with or without pressure in the molten metal matrix. The operation to be executed in this process is initially the metal selected is preheated, then pouring molten metal, followed by pressure application to molten metal, and ultimately it is mixed and taken out from die and punch.(Baliarsingh et al., 2023) It reduces material waste as scrap as it generates the products almost with net-shape and doesn't require common feeding devices like runners, gates, risers. The process parameters like; Die and pouring temperature, mould pressure and temperature, filling rate and pressurizing melt's time delay are used to apply on squeeze casting quality. Additionally it is elastic, convenient, economical and used for mass production. But very hard to achieve homogeneity, and reinforcement and matrix are reacted (Singh et al., 2020b).

2.3.4 Spray-up

The spray pistol can be used to spray reinforcement and matrix simultaneously. Either room temperature or high temperature can be used for the curing procedure. Upon completion of the curing process, the mold is opened and the material is removed for further processing. The big, resinous products produced by the spray-up process have poor mechanical properties. It's simple, and the material is inexpensive, rapid fiber and resin deposition(Singh et al., 2020b).

2.3.5 Ultrasonic- Assisted Casting (UAC)

The UAC technique effectively disrupts nano-composite agglomerations. By the application of ultrasonic vibrations of high energy to the melt, the root cause of particle agglomeration during MMC processing can be minimized. There are two stages of composite processing in which ultrasonic vibration can be incorporated: reinforcing mixing, and solidification (Baliarsingh et al., 2023). Material obtained through this process has fine mechanical properties and uniformity of attributes and can be suitable for fabricating composites in bulk. But tooling required, makes it extremely costly and frequency sound is extremely high (Singh et al., 2020b).

2.3.6 Friction Stir Processing (FSP)

To change a material's properties, a process known as friction stir processing (FSP) employs local plastic deformation. FSP solves the liquid phase processing interfacial reaction problems through operation below the melting point of the substrate (D. K. Sharma, Mahant, et al., 2019). FSP homogenizes and refines microstructure by sliding and placing a non-consumable tool, improving sheet metal properties. Grain structures are recrystallized by this solid-state process, improving formability, particularly at high temperatures. FSP in casting of magnesium improves local mechanical properties and repairs casting defects and aluminum alloys that has exhibited substantial improvements in mechanical properties fatigue life, ductility, and strength (Singh et al., 2020).

2.4 Effect of Reinforcements on Properties of AMMCs

2.4.1 Effects of reinforcements on mechanical properties

Dorasila & Madhusudhan. (2024) successfully synthesized AA6061 based composites by reinforcing through graphene and SiC via powder metallurgy. They conducted the study by varying weight percentage of graphene and SiC with (AA 6061), (1 wt% SiC and 1.5 wt% Graphene), (2 wt% SiC and 2.5 wt% Graphene) and (3 wt% SiC and 3.5 wt% Graphene). It was observed that better hardness and densification were obtained for composites with 2.5 wt% of graphene. Yet, poor hardness of composites with 1.5% graphene was observed. Thus, 2 wt% SiC and 2.5 wt% graphene is the ideal material to be used to produce highly hard and dense AA6061 hybrid composites. Generally, the result indicated the mechanical properties of AA606/SiC/graphene were better.

Arvind Sankhla et al. (2024) carried out work on the impact of laser surface treatment on wear behavior and hardness of Al-MMC processed via powder metallurgy. Al-MMC's surface goes through metallurgical transformation because of treatment of laser, which may also improve the reinforcement particles binding with the matrix. Al-MMC is subjected to laser treatment after its fabrication. The research verifies that as compared to untreated Al-MMC, as SiC wt% increased the hardness also improved. Even the wear resistance was enhanced. As a new layer had been developed over the treated surface, which did not allow the release of SiC particles, laser-treated samples reflected less wear than untreated ones.

El-sayed et al. (2021) investigated the tribological and mechanical properties of an Al6061 alloy that has been hybridized via a powder metallurgy technique with multiwall carbon nanotubes. Various wt% reinforcing values of 0, 0.5, 1, and 1.5 are used in processing the material. Microstructure of MWCNT-hybridized Al6061 alloy exhibited a good interfacial adhesion bonding and uniform distribution of MWCNTs. Due to increase in wt% of MWCNTs, the Vickers microhardness number of the matrix alloy improved and also wear rate enhances at 1.5wt%. Moreover, at 1.5wt% MWCNTs and 700 MPa compaction pressure, the composites showed a well-defined improvement in relative density, microhardness, and wear rate. Mechanical and tribological performance was found to be enhanced.

Venkatesh & Deoghare. (2020) have been produced successfully the Al-Kaoline composite by powder metallurgy technique. 10%, 15%, 20%, and 5% wt% reinforcement were used while processing composites. The outcome demonstrated that when 20% reinforcement was added to unreinforced aluminum, the hardness value of Al-Kaoline composites increased from 77 to 187 VHN. The study found that incorporating high-hardness ceramic particles like Al_2O_3 and SiO_2 of 0% to 20% in Kaoline reinforcement, tensile strength and compression strength of composites improved as reinforcements were added. Additionally, the study concluded that the material studied was appropriate in automobile industry for producing car body and pistons, which must be strong and light weighted.

Nishanth et al. (2020) have been synthesized successfully AA7075 MMC with SiC reinforcement through powder metallurgy method. Mechanical properties were improved by increasing the SiC reinforcement from 0% to 10% SiC. Hardness, ultimate compressive strength and Density were enhanced as the percentage of SiC was rose from 0% to 10% and porosity reduces. As the created composite was defect-free, it found to be applicable in the aviation and aerospace fields.

(Mahesh Kumar & Venkatesh, 2018) they had stir casted aluminum metal matrix composites with alumina. According to the paper, introduction of Al_2O_3 reinforcement into aluminum matrix composites (AMMCs) tremendously improves their mechanical properties. The addition of 4-10% of alumina improves wear resistance, compressive strength, and hardness. The test confirmed noteworthy improvement in Brinell hardness of as much as 172% at 10% Al_2O_3. At this point, wear resistance was highest, and testing indicated least volume loss. Microstructural analysis verified that alumina was suitably dispersed in the matrix.

2.4.2 Effects of reinforcements on tribological properties

(Mahan et al., 2023) they had investigated the influence of TiO_2on dry sliding wear behavior of aluminum AA2024 composite. The study examined how different TiO_2 nanoparticle contents reinforce AA2024 aluminum composites. The composites exhibited 22% improvement in wear resistance and 68% improvement in hardness after heat treatment with 5 weight percent TiO_2. The mechanical enhancements were made possible by beneficial phases that were created by TiO_2. Owing to the evenly distributed small precipitates, wear testing revealed improved dry sliding behavior.

Kumar et al. (2023) report that self-lubricating Al-Mg-Si/MoS_2 composite powders were produced by powder metallurgy with the aim of enhancing tribological and corrosion behavior through modification of the concentration of xMoS_2.Tribometer wear characteristics were evaluated at varying load applied and sliding distances using the Taguchi model (L9).As the study reported that, adding MoS2 to Al-Mg-Si improved resistance to corrosion and reduction of COF and wear. As the distance rise up to 250 m, the COF varied; however, as the distance increased up to 3000 m, it remained stable. The paper explained that the developed composite is effective in auto parts where liquid lubricants are not recommended.

(Ahmadifard et al., 2022) the study was reported that, the addition of Ti_3AlC_2 MAX phase and Al_2O_3 nanoparticles to Al7075-T6 alloy through friction stir processing has a great enhancement in its tribological and mechanical properties. Maximum tensile strength and hardness of Al-100% Al_2O_3 composites were attained by grain refinement, while MAX phase enhances lubrication by minimizing wear and friction. Hard particles result in a change of the wear processes from adhesive to a combination of adhesive and abrasive wear. Better wear resistance was displayed by FSP samples with higher MAX phase, which have shallower wear tracks according to SEM analysis.

Whenish et al. (2020) carried out the effect of SiCp reinforcement on Al6061composite via powder metallurgy for the fabrication of gear blank, which requires enhanced wear resistance and self-lubrication. The gear blanks were fabricated by adding 50 μm-sized SiC particles as reinforcement at various wt% (3%, 6%, and 9%) and binding the Al6061 with zinc stearate. In this study, the compacted green portion was carried out at 500°C sintering process. The study confirmed that increased hardness was a result of dispersion of SiCp.

Ashrafi et al. (2020) have been able to fabricate Fe_3O_4-SiC reinforcing aluminum matrix hybrid nano filler composite through powder metallurgy. The current work demonstrated how the wt% of the reinforcements affect the mechanical, microstructure, corrosion and tribology properties of the materials with reference to SiC-Fe_3O_4 nanoparticles. Al-Fe_3O_4-SiC reinforcement is added into the composite, and the result had a good corrosion resistance (lower corrosion rate). The research indicated that by raising the weight fraction of SiC-Fe_3O_4, tribology, corrosion resistance, and mechanical properties were improved.

Norul Amierah et al. (2019) were directed to the investigation of powder metallurgy processed aluminum–graphite composite mechanical and tribological behavior. In this work the material was synthesized by using 3,5, and 7 wt% graphite. The result showed that the incorporation of Gr led to a decline in mechanical properties. But the mechanical properties of the 3 wt% Gr composite were superior to others. Upon addition of Gr, wear rate and COF also went down to their lowest at 3 weight percent Gr. The surface of wear of 3 wt%Gr reinforced composite exhibited a smooth layer of graphite.

(Sudha et al., 2019) investigated the tribological and mechanical characteristics of graphite and tungsten carbide reinforced aluminum alloy. The study investigated how powder metallurgy can improve aluminum 6061 composites by adding graphite (Gr) and tungsten carbide (WC) reinforcements. It discovered that the content of Gr increases hardness (according to BHN) and wear resistance. While SEM analysis indicates lower friction through Gr and WC-formed surfaces, processing assures uniform reinforcement distribution. These reinforcements have exhibited improvements in composite materials by enforcing considerable control over tribological, mechanical properties.

2.4.3 Effects of reinforcements on microstructure

(Monteiro & Simões, 2024) They investigated the influence of multiwalled carbon nantubes on Al6061 nanocomposites. The study showed how the microstructure of Al6061 was greatly enhanced by the addition of MWCNTs, resulting in smaller, uniform grains. Hardness is increased and properties are optimally balanced by ideal sintering at 580°C; density and matrix reactions are influenced by high temperatures. Mechanical properties are influenced by secondary phase development, as shown by EDS analysis. Focusing at three points, carbon nanotubes (CNTs) are important for strengthening behavior and structure.

(Melik et al., 2022) the study investigated the sintering temperature influence on mechanical and microstructural behavior on Al6061 via powder metallurgy. The study documented that Al6061 alloy composites reinforcing by SiC nanoparticles fabricated using powder metallurgy were tested. Density and hardness were maximized by sintering at 600°C (32.2 Hv). SEM and EDS analysis validated the bonding and distribution of SiC nanoparticles. The material integrity was verified by measuring density and argon gas milling to promote consistent dispersion of reinforcements. The findings indicate the contribution of SiC to improved mechanical properties.

Islam (2021) have been synthesized successfully AMMCs reinforcing through TiO_2 using powder metallurgy methods with varying contents of TiO_2 ranging from 4 to12 wt% with an aim to aerospace, defense, and automobile industries applications. According to the study, Al-TiO_2 composites' porosity, hardness, and compressive strength all improved as the TiO_2 content rose from 4 to 12 weight percent. Compressive strength enhanced from 67.90 MPa to 77.53 MPa, porosity enhanced from 1.80 to 2.01, and hardness increased from 19.51 MPa to 26.08. The work established that the composites are well dispersed in the microstructure. Additionally, as wt% was added, the interface bond between the grains got better and air spaces among the grains reduced.

Akbarpour & Alipour. (2018) performed microstructure and tribological properties of SiC nanoparticle reinforced nanostructured aluminum developed by powder metallurgy method. The study yielded composite material with wear resistance and low weight. By the incorporation of 4 vol% SiC nanoparticles into Al matrix, grain size decreased and nanoparticle dispersion became uniform. The wear resistance and friction coefficient lowered with an increased applied load. Abrasion and delamination of wear mechanisms were observed, according to SEM observation.

2.4.4 Effects of processing methods

Akgümüş Gök et al. (2024) was emphasized the use of powder metallurgy in the production and characterization of composites made of aluminum reinforced with B_4C, SiC, MgO, and Al_2O_3 powders. Studies showed that the major properties like material density, resistance to wear and hardness can be influenced by altering sintering time, temperature, and powder composition. In general, the paper stated that time of sintering and temperature of sintering has influence on increasing the density and reducing porosity of composite materials. Further, higher compaction pressure leads to a more homogeneous structure via enhanced density. SiC- and B4C-reinforced composites are suitable in high-performance applications such as the cutting tool, automotive, aerospace, and defense sectors due to their improved qualities.

Abd El Aal et al. (2022) investigated the properties of Al-MMC, which is made via high pressure torsion and powder metallurgy. The investigation put that high pressure torsion at 10GPa and powder metallurgy are used to fabricate Al-Al_2O_3 and SiC with varied volumes up to 20%. But the HPT process results in fully dense products. Fragmentation of reinforcement and grain size of Al matrix was reduced due to HPT. The Al and Al MMCs' tensile strength and microhardness were improved by the manufacture of ultrafine grains.

(Xue et al., 2022) had synthesized Cr3C2 reinforced aluminum through ultrasonic assisted casting technique. The study reported that tensile and yield strength improved over 100% when the Cr_3C_2 percentage had attained its best 3.0%. To the extent that grain refinement as well as CTE mismatch was driven to reinforce, Vickers hardness and density improved with particle additions. At more than 3.0 weight percent of Cr_3C_2, fracture changes to plastic, signifying the stress resistance of the composite. The effectiveness of ultrasonic treatment was confirmed by improved mechanical and distribution characteristics.

(D. K. Sharma, Sharma, et al., 2019) was examined the influence of boron carbide in aluminum matrix composites. The investigation revealed that AA7075/B4C and other hybrid composites fabricated by Friction Stir Processing exhibit better hardness, elongation, and wear resistance than AA6061/B_4C composites, even though this latter exhibited more homogeneous particle distribution. Higher B4C content increases mechanical properties like hardness, strength and wear resistance at the cost of decreasing electrical conductivity, density, and thermal expansion. Nevertheless, there were problems such as poor wettability, non-uniform particle dispersion, and unwanted interfacial reactions, particularly when employing liquid metallurgical methods. Nano-AMCs have the potential with improved microstructures with uniform reinforcement distribution, improved mechanical properties, and retained ductility when processed by K2TiF6 flux or two-step stirring. Al/B4C composites display superior interfacial bonding and particle dispersion compared to Al/SiC or Al/Al_2O_3 for common reinforcements; smaller B_4C particles yield better mechanical properties.

(Manochehrian et al., 2019) had developed the surface reinforcing of A356 aluminum alloy by nanolayered Ti3AlC2 (2.5, 5, and 7.5vol %) MAX phase through friction stir processing. The study revealed that incorporation of Ti_3AlC_2 MAX phase particles enhanced the tribological and mechanical behavior, which made them applicable for high-abrasive applications. FSP minimized porosity and greatly refined the microstructure. It led to the improvement of microhardness, tensile strength, and resistance to wear by the addition of Ti_3AlC_2 particles and also lowered the friction coefficient.

(Sudha et al., 2019) had reported the mechanical and wear behavior of aluminum processed by powder metallurgy. The paper illustrates the excellent improvement of AMMC characteristics that powder metallurgy (PM), in particular sparks plasma sintering (SPS), provides. High hardness (280 HV), low porosity, and better reinforcement distribution were achieved by SPS. Fivefold the hardness of pure aluminum is shown by AMMCs containing 15 weight percent Al2O3-AlB12. Apart from creating a lubricating mechanically mixed layer (MML), wear resistance was enhanced with growing reinforcement. The microstructural analysis revealed, smaller powders homogeneity is improved by preventing clustering, which degrades properties. These developments indicate the superiority of PM over traditional processes in the fabrication of high-performance AMMCs.

2.5 Applications of Metal Matrix Composites

The large fields of technical uses of MMCs are accountable for their increasing popularity. MMCs known as high strength-to-weight ratio materials have features such as wear resistance, reduced thermal expansion, improved raised temperature performance, high specific strength, and improved electrical performance. They are used in electronics, packaging, sports, recreation, infrastructure, space exploration, defense, cars, rail transportation, maritime transportation, and aerospace. Common matrices include Al, Mg, Ni, Ti, Cu, and Fe, whereas reinforcements include SiC, AlN, TiC, Si_3N_4, Al_2O_3, B_4C, Y_2O_3, TiB_2, and ZrO_2. Aluminum MMCs are especially appreciated in the automotive, defense and aerospace industries owing to their enhanced tribological performance, corrosion resistance, and strength-to-weight (D. K. Sharma, Mahant, et al., 2019). Magnesium metal matrix composites, being lightweight, bioactive, and biodegradable, minimize the need for a second surgery, promote proper degradation in the human body, and improve recovery rates. They are in a position to replace titanium and aluminum better because of their elastic modulus; this is comparable to the human body's elastic modulus. A high specific strength and an improved corrosion-resistant non-ferrous metal is titanium.

Because of its greater strength and negligible weight, it finds extensive application in the chemical, pharmaceutical, and aeronautical sectors (Arora et al., 2022).

2.6 Multi-response optimization

Multi-response optimization is a technique that is used to optimize more than one response or result in experiments or processes at a time. Optimization methods are utilized to find the "best" operating circumstances for a system to give it the optimum performance achievable under the limitations available at the moment. In fields like product development, manufacturing, and engineering, where several performance traits are to be optimized simultaneously rather than separately, this technique is utilized. Engineers can determine solutions using this technique that maximize all the responses without making any one response suboptimal. Simultaneous maximization of more than one response or outcome in a process or an experiment is what multi-response optimization techniques aim for. By trading off compromises between competing goals, these techniques seek the optimal combination of controllable variables to produce the desired results for every response (Ganesan et al., 2018). These multi-response optimization methods are Data Envelopment Analysis (DEA), Grey Relational Analysis (GRA), and Taguchi Method (Abdullahu et al., 2024).

GRA is a technique that can be applied in combination with the Taguchi method to address multi-response optimization issues, utilizing the advantages of both approaches (Zaman et al., 2020). It seeks to quantify the proximity or closeness of a set of response profiles to one another and determine which input variable setting achieves the maximum overall closeness or likeness to the target response. This is a more appropriate method, and it becomes easy to forecast how various parameters will affect the levels under study. The Grey Relational Grade (GRA) approach optimizes characteristics of multiple performances by converting the parameters derived from the Taguchi approach into a single performance characteristic. Grey relational analysis is especially beneficial for situations with unpredictable environments, complex systems, or limited data.

2.7 Summary of Literature

From the above discussed reviewed studies in AMMCs the study showed outstanding improvements, even though there were some issues that must be overcome. Those issues involved failure to realize a uniform reinforcement distribution, which affects mechanical properties and their performance; even though the AMMCs displayed some enhanced mechanical propperties, divergence in performance across various operating conditions had occurred. And also there is inconsistency in testing methods and lack of long term performance. The aforementioned above mentioned challenges with respect to AMMCs shall be addressed by using Al metal martix reinforced with Al_2O_3, B_4C, and MoS_2. The aluminum oxide has ability to enhance the mechanical properties of Al by strengthening it. B_4C, which is well-known for its hardness and low density, can help in developing a fine microstructure and enhance its mechanical properties by carefully arranging reinforcement. B_4C provides superior resistance to wear, and MoS_2 is used as a solid lubricant. When combined, they can greatly enhance wear performance and enhance component life in frictional situations. In addition to this they improve the durability of the Al composites by reducing the need for frequent replacements and minimizing waste. So, an effective method of these challenges related to AMMCs is using Al matrix reinforced with B_4C, MoS_2, and aluminum oxide processed through powder metallurgy and optimizing its process parameters.

2.8 Research Gap

It has been revealed from the literature review that most researches have been implemented on synthesis, characterization and testing of aluminum metal matrix composites using ceramics applicable to various applications. However, limited work has been existed on the use of Al as a matrix with B_4C, MoS2, and aluminum oxide as reinforcement materials. Also in a few literatures boron carbide, aluminum oxide, and molybdenum disulfide were used as reinforcement materials individually or in combination with other materials in the reinforcing of aluminum. But there is insufficient of comprehensive studies on the synergistic effects of combining B_4C, MoS_2, and Al_2O_3 as reinforcements in Al matrices, and also there is insufficient optimization of processing parameters in powder metallurgy particularly regarding their effect on mechanical, corrosion, metallurgical and tribological properties. To address this research gap, this research work aims to fabricate Al metal matrix composites reinforced by aluminum oxide, molybdenum disulphide and boron carbide and evaluate their mechanical, metallurgical, and tribological properties.

CHAPTER THREE

MATERIALS AND METHODOLGY

3.1 Introduction

This chapter presents an overview of the materials and methodology employed in the fabrication, testing and characterization of aluminum composites. The experimental procedures, specimen preparation methods, test setups, and material selection employed in composite making were discussed.

3.2 Materials

Effective knowledge of composite property is required for material selection based on the application desired. Knowledge and understanding of the properties of materials required makes it easier when selecting materials for reinforcement and matrix. Aluminum oxide, Molybdenum disulfide and boron carbide were selected as materials reinforcement and aluminum (Al) as matrix metal.

3.2.1 Aluminum (Al)

Pure aluminum (Al) is light weight and silvery-white metal. Its particle size was 74µm that has 99.5% purity.

Table 3.1 Properties of pure Al (Stockist, 2005)

Properties	Units
Density	2.7g/cm^3
Melting point (^{0}C)	660.2
Color	Silvery-white

Table 3.2 Chemical composition of pure Al as per company

As	Pb	Fe	Al
0.0005	0.03	0.5	Rest

3.2.2 Aluminum oxide (Al_2O_3)

Alumina, often known as Al_2O_3, is a very extensively used chemical in science and engineering due to its superior properties. Because of its high hardness, chemical inertness, and great wear resistance, it can be utilized in refractories, composites, and ceramics. Alumina which has particle size of 74µm and 99.5% purity was utilized in this work.

Table 3.3 properties of aluminum oxide (Abyzov, 2019)

Properties	Aluminum oxide
Color	White
Vickers Hardness HV (Gpa)	20.6 – 29.4
Density (g/cm^3)	3.99
Melting point (°C)	2054
Compressive strength (Mpa)	2550 - 3100

3.2.3 Molybdenum disulfide (MoS_2)

Molybdenum disulfide is an inorganic compound containing molybdenum and sulfide. Its chemical formula is MoS_2. It possesses excellent mechanical characteristics especially hardness. The size of the powder particle was 89µm.

Table 3.4 Mechanical properties of MoS2 Rahmati et al. (2014)

Properties	Molybdenum disulfide
Color	Lead gray to black
Hardness (Mohs scale) basal plane	1–1.5
Density (g/cm^3)	4.8–5.0
Melting point (°C)	1185
Cristal structure	Hexagonal-layered

3.2.4 Boron Carbide (B_4C)

Boron carbide is the third hardest substance. Because of its unique set of properties, boron carbide was selected for several engineering purposes. Boron carbide finds application in refractory applications as a result of its high melting point and stability to heat. It is also beneficial in abrasion resistance powder and coating owing to its improved abrasion resistance (Domnich et al., 2011). It possesses very good performance in ballistics due to its low density and high hardness. Last but not least, boron carbide is utilized vastly in nuclear applications since it has the ability to absorb neutron radiation. Besides this, boron carbide is a semiconductor with resistance at high temperatures. The average size of particle used in this current study was 200 µm.

Table 3.5 Typical properties of B4C (Sudha et al., 2019).

Property	Value
Density ($g.cm^{-3}$)	2.52
Melting Point (°C)	2445
Hardness (Knoop 100 g) ($kg.mm^{-2}$)	2900 – 3580
Young's Modulus (GPa)	450 – 470

Table 3.6 Chemical composition of boron carbide Mohd Adnan (2020)

B	C	B_2O_3	Fe	Si	Others
79	20	0.5	0.2	0.1	Balance

a) b) c) d)

Figure 3.1 Powders materials a) Al b) MoS_2 c) Al_2O_3 d) B_4C

3.3 Methodology

The sample fabrication, testing and characterization processes were addressed in Figure 3.2. The samples were processed by the powder metallurgy method. This powder metallurgy process consists of: mixing, compacting, and sintering. The flow chart is shown in the diagram that represents the complete flow chart for the current research work. Raw materials bought from commercial sources initially and then the matrix powder was mixed with the reinforcements. Different characterization was done for Al-Al_2O_3-B_4C-MoS_2 composites with the help of OM, SEM and XRD to examine microstructure, particle size and phase and particle distribution of reinforcement over matrix. The pin-on-disc tester machine was used to evaluate the samples' resistance to wear. The samples corrosion resistance of was examined using the electrochemical technique. Furthermore, the Vickers hardness test and universal testing machine were used to determine the samples' microhardness and compressive strength correspondingly.

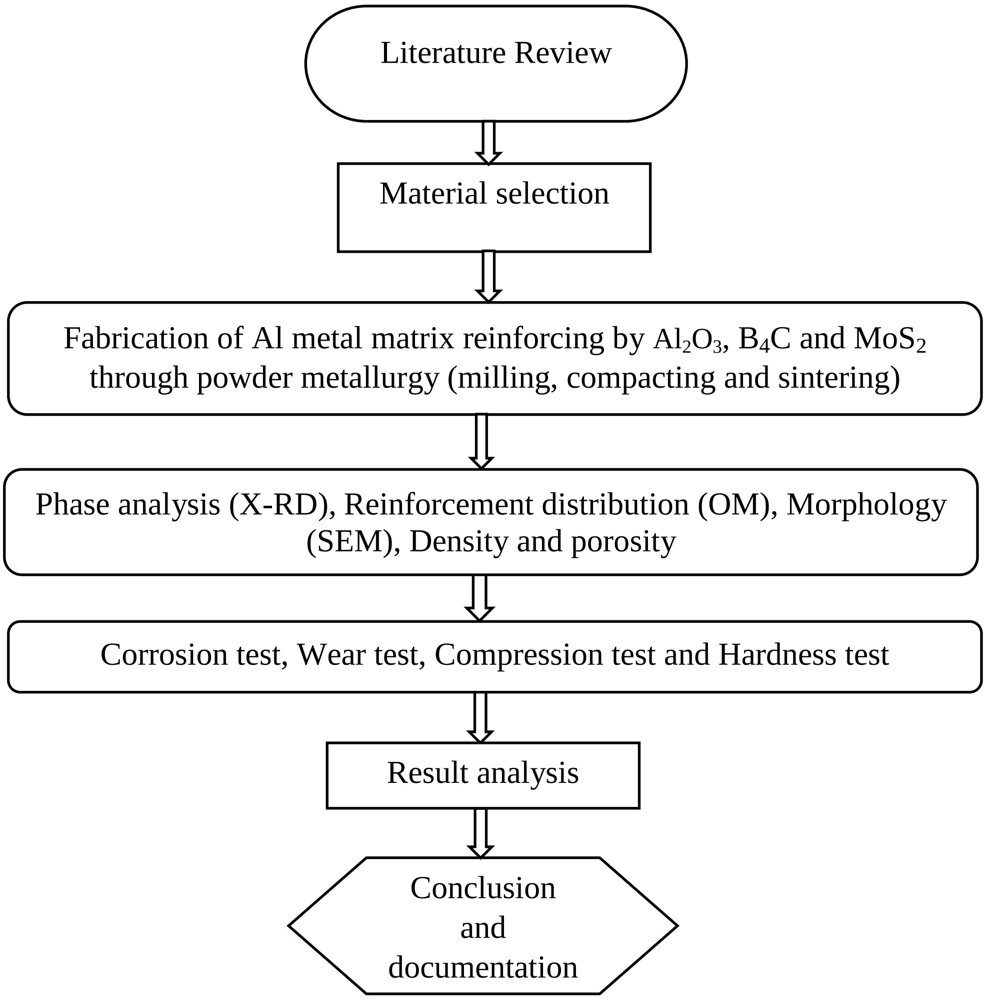

Figure 3.2 Flow chart of overall process

3.3.1 Powder metallurgy for processing of MMC

For the processing of MMCs, the powder metallurgy (PM) process had the potential to be an industrially feasible, energetically efficient, and economically viable method. Metal matrix composites based on powder metallurgy are being chosen and used for component development in various industries, including electronics, automobile, and aviation Nayak et al. (2022). The samples were fabricated and tested through different test procedures. Powder metallurgy process parameters that can influence processing performance are weight percentage, milling time, sintering temperature, compaction pressure, and particle size. In this current study work, the influence of wt% reinforcement and PM process variables both (sintering temperature and compaction pressure) at three levels has been specified and compression, corrosion, wear, hardness and density of the composite made were examined.

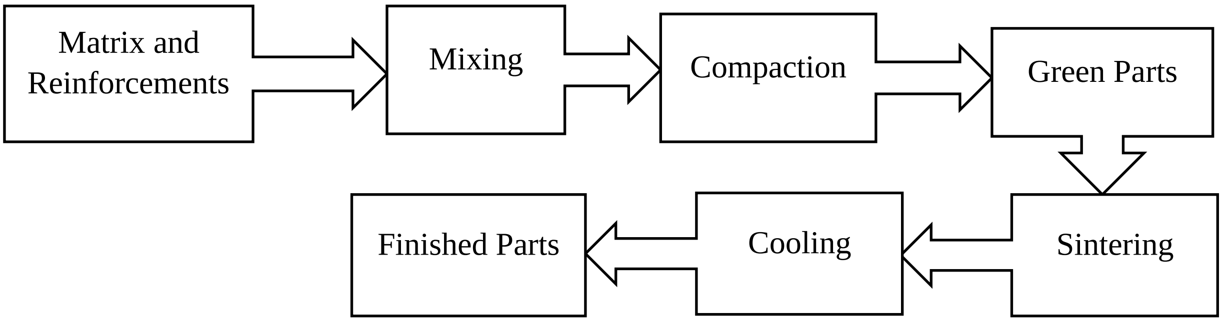

Figure 3.3 Schematic diagram of powder metallurgy process

3.4 Sample Fabrication process

The sample preparation was implemented for different compositions of reinforcements B_4C (2 - 6 wt. %), Al_2O_3 (2 – 6 wt. %), MoS_2 (4 wt. %) and Al (84 – 92 wt. %). And their weights were weighed using weighing balance (analytical precision balance) which has a capacity 252g/0.1mg. Each sample has a total weight of four grams. The total weight of the reinforcements and matrix of every sample was computed from the selected weight percentage of matrix and reinforcements from Table 3.7.The matrix weight percentage of every sample was determined from the reinforcements' weight percentage. Table 3.7 shows the fabricated samples by powder metallurgy.

Table 3.7 The fabricated Composite samples via powder metallurgy.

Samples	Reinforcements			Matrix
	B_4C (wt%)	Al_2O_3 (wt%)	MoS_2 (wt%)	Al (wt%)
S1	2	2	4	92
S2	2	4	4	90
S3	2	6	4	88
S4	4	2	4	90
S5	4	4	4	88
S6	4	6	4	86
S7	6	2	4	88
S8	6	4	4	86
S9	6	6	4	84

3.4.1 Blending of Powder

The matrix powder was mixed with reinforcement particles Al_2O_3, B_4C and MoS_2 that have different weight percentages for 1hr(Sudha et al., 2019) using high energy mixer/milling machine. The agglomeration of powder particles was decreased when matrix and ceramic powders are properly blended. The balls utilized were zirconia balls as shown in the figure 3.5b. The ball to powder weight ratio has been managed at 10:1(S. K. Sharma et al., 2025). At speed of 400rpm all the powders were allowed for uniformly mixing and blending (Neha, 2023).

3.4.2 Compaction of Powder

The powders were poured in to die once it had been blended for the proper amount of time. Using a hydraulic pressing machine for 5min of time (Mansoor, 2025), the powder was compressed by applying load and (50,55,60)Mpa pressure (Sudha et al., 2019) at the same speed for every composition of powders. The aim of compaction is to create a green compact that is sufficiently strong. The hydraulic press machine (lab - press) along with the punch was given in the figure (3.5 a).

3.4.3 Sintering Process

Sintering process used for bonding and densification of the particles. This sintering process involves three phases (heating, sintering and cooling). In this heating the sintering was started at room temperature (20°C) and heated the compacted green body at a steady rate of 10°C/min (Arockiasamy et al., 2010)(Zhou et al., 2022) to minimize oxidation and thermal shock. But during the sintering the samples were heated at sintering temperature(580, 600,620)°C (Monteiro & Simões, 2024);(Melik et al., 2022) for 1hr(Victor et al., 2020); (S. K. Sharma et al., 2025). Additionally, during cooling the samples were cooled at constant cooling rate of 10°C/min(Zhou et al., 2022) to avoid cracking, thermal stress and maintain the microstructure durability. To avoid oxidation, sintering is usually performed in a controlled atmosphere, e.g., vacuum, nitrogen or argon.

(a) (b) (c)

Figure 3.4 a) High energy mill/mixer machine b) Weighing equipment c) Measuring powders

(a) (b)

Figure 3.5 a) Hydraulic press machine b) Zirconia ball

a) b)

Figure 3.6 Fabricated samples a) green compact (S1, S6 and S8) and sintered samples b) high temperature box furnace

3.5 Preparation of Specimen for Characterization and Testing

Once the composite sample was properly formed and sintered, it was now ready to be tested and characterized using various techniques. In the current study the samples were made in accordance with ASTM E3 standards and were lathed to the required size and shape. Then the sample was ground with various grades of silicon carbide sand paper (120, 220, 240 and 360) in order to erase any marks obtained by cutting and make it a level, mirror-like surface free from unnecessary distortion. And specimens were polished gradually.

3.5.1 Microstructural Study

Microstructural examinations were done on the samples using optical microscopy (HUVIZT), XRD, EDS and scanning electron microscopy. Phase analyses of the specimen were examined by an X-ray diffracton (XRD-7000 X-RAY DIFFRACTOMETER). It was able to work in a 2θ measurement range (10–90°), 40 kV tube voltage, 40 mA tube current, and a 3°/min scan speed. Origin Pro software was used to determine phases and quantitatively analyze phases. Different magnifications were used in examining the distribution of reinforcements in the Al matrix. This distribution of the reinforced particle had already been studied using optical microscopy. The elemental composition were identified by using EDS. Additionally, SEM microstructural tests were performed in an attempt to examine interfacial integrity and distribution of reinforcement between matrix and reinforcement.

a b

Figure 3.7 Equipment used for characterization a) OM b) SEM

3.5.2 Density Measurements

One way to describe a material's density is as its mass per unit volume. The density of the sample that was manufactured was measured after sintering. Density is a very important property of materials, especially metal matrix composites, since it affects their performance, weight, and strength in structural engineering, automotive, and aerospace industries. In this result, density was calculated by employing Archimedes principle based on ASTM B962-23. The sample mass in air and the immersion fluid of our choice can be found using this procedure. After measuring mass of sample in air, mass of the sample submerged in fluid was measured. Lastly, the density was determined using equation (3.2).

$$\rho\text{sample} = \frac{M\text{air}}{M\text{air} - M\text{water}} * \rho\text{water} \tag{3.2}$$

Where: ρ_{sample} = sample density,

m_{air} = mass of the sample measured in air,

m_{water} = mass of the sample measured when submerged in water,

ρ_{water} = density of water

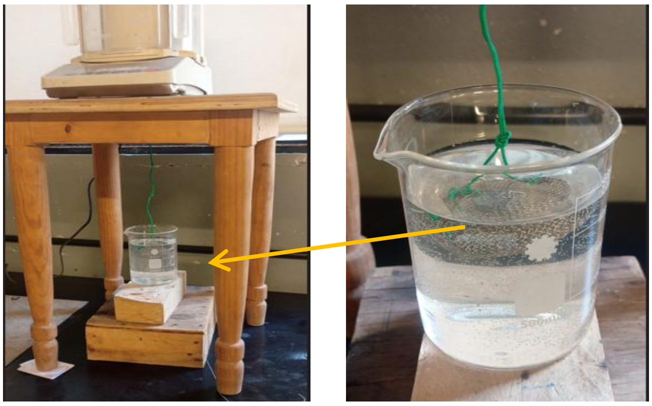

Figure 3.8 Density measurement using Archimedes technique

The density of water is typically 1 g/cm^3 at room temperature. And the density of distilled water at 20°C= 0.9982 g/cm^3 (Sulaiman et al., 2012).

3.5.3 Porosity measurements

The term porosity describes the existence of pores or voids in the composite material or a percentage by volume of pores or voids in a material and can influence its density, performance, and mechanical properties. The porosity is denoted by the symbol (ϕ). The fractional value of porosity is between 0 and 1 or 0% and 100%. Archimedes technique was utilized in this study to determine the porosity of the sample as per ASTM B962-23. The steps to be followed in this technique are: Calculation of the theoretical density of the composites manufactured depends on weight percent of matrix and reinforcements.

$$\rho_t = (W_m*\rho_m)+(W_r*\rho_r) \quad (3.3)$$

Where: ρ_t = theoretical density

ρ_m = density of the matrix material

ρ_r = density of reinforcement materials

Wm = weight fraction of the matrix material

W_r = weight fraction of the reinforcement material

The volume of the samples was calculated by measuring buoyant force when the sample is submerged in a liquid (typically water).

$$\text{Buoyant force} = W_{air} - W_{fluid} \quad (3.4)$$

Where, W_{air} = weight of the sample in air

W_{fluid} = weight of the sample when submerged in the fluid

$$\text{Volume of the sample } (V_{\text{sample}}) = \frac{\text{buoyant force}}{\text{density of the fluid}} = \frac{\text{Wair} - \text{Wfluid}}{\rho\text{fluid}} \tag{3.5}$$

Then the apparent density of the sample was calculated by using weight in air and volume of the sample.

$$\text{Apparent density } (\rho_A) = \frac{\text{Wair}}{V\text{sample}} \tag{3.6}$$

At the last the porosity was determined:

$$\text{Porosity } (\phi) = \frac{\text{Theoretical density} - \text{Apparent density}}{\text{Theoretical density}} * 100$$

$$\Phi = \frac{\rho t - \rho A}{\rho t} * 100 \tag{3.7}$$

3.5.4 Hardness Test

The ability of a material to withstand scratches, abrasions, and indentations is called hardness. Brinell, Rockwell, or Vickers tests are most used for its testing. Vickers hardness tester (HVS-50) apparatus has been used within this research to test the hardness of the composite materials, which have been prepared under ASTM B933-20. In Vickers testing, the impressions on the surface material are formed by means of a diamond indenter as loads, preferably in the range 1kgf to 120kgf, are utilized (SarithNaidu & Venkatasubbaiah, 2024). Then 10kgf load was applied with the holding time of 10s. The diameter of the indentations is then used to determine the hardness values. Three impressions were made to reduce uncertainties. The angle between the opposite faces of the pyramid is 136°. The hardness (HV) was determined by applying the following equation.

$$\text{HV} = \frac{Constant * Test\ force}{\text{Surface area of indentation}} = \frac{0.102 * 2F(\sin\frac{136°}{2})}{d^2} \tag{3.8}$$

Where, HV = Vickers hardness number

F = applied load in kilogram force,

d = average diagonal length of the indentations in millimeters

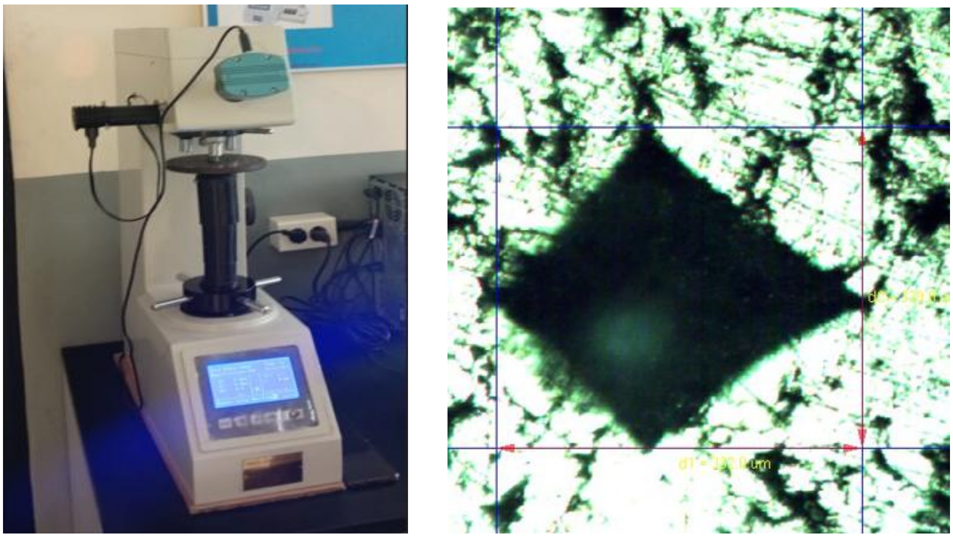

Figure 3.9 Vickers Hardness tester and test result

3.5.5 Compression Test

Compression test was utilized to examine compressive strength of the material. The ability of a material to withstand compressive loading without failure is known as compressive strength. The sample was prepared as a required size and form. In this research a specimen manufactured at optimum level was compressed by applying load gradually until it failed using a universal testing machine (model:WDW-100S,capacity 100KN) to carried out a compression test, and the results are evaluated in accordance with ASTM B925-15 standards.

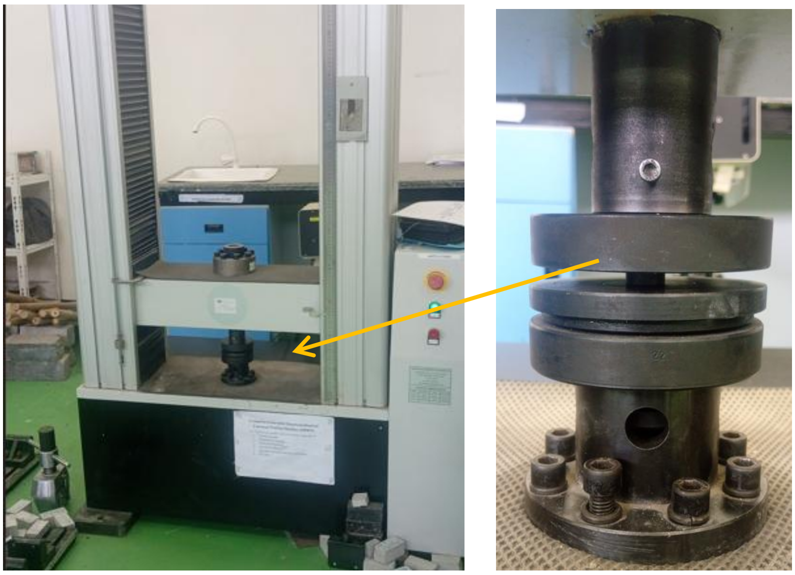

Figure 3.10 Computer controlled electromechanical UTM for compression test

3.5.6 Corrosion Test

Corrosion is a natural phenomenon that occurs when metals and other materials chemically react with their surroundings and lead to their gradual deterioration. To study the corrosion of the made composites, the following process can be carried out. The specimen was cut into desired dimensions and polished into smooth surface. The specimen is submerged in an electrochemical cell with an electrolyte solution of 3.5% NaCl solution for 30 minutes (Turhan et al., 2011). Potentiodynamic polarization test was utilized for the evaluation of corrosion behavior. Through this potentiodynamic polarization, the potential of the specimen was swept through the anodic and cathodic ranges and the resulting current was recorded.

The Tafel curve, which was extrapolated from the Tafel equation, described the result of this test. Tafel curves may be employed to explain the correspondence between current density and potential in electrochemical reactions. It supplies the information on corrosion potential (Ecorr) and corrosion current (Icorr) that might be employed to examine corrosion behavior. The potential for anodic and cathodic reactions to balance on the metal's surface is known as the corrosion potential, i.e., where there is zero net flow of current. Corrosion potential is used to forecast corrosion behavior because the higher the corrosion potential, the lower are the chances of corrosion. Corrosion current is a measure that describes the rate of electrochemical reactions taking place during corrosion. Lower corrosion current density shows a slower corrosion rate. The corrosion rate of composite was measured by applying Stern-Geary equation (Sakthi et al., 2023).

$$\text{Corrosion rate (mmpy)} = 0.00327 * I_{corr} * \frac{\text{Ew}}{d} \tag{3.10}$$

Where, I_{corr} = corrosion current

Ew = Equivalent weight (g)

d = density (g/cm^3)

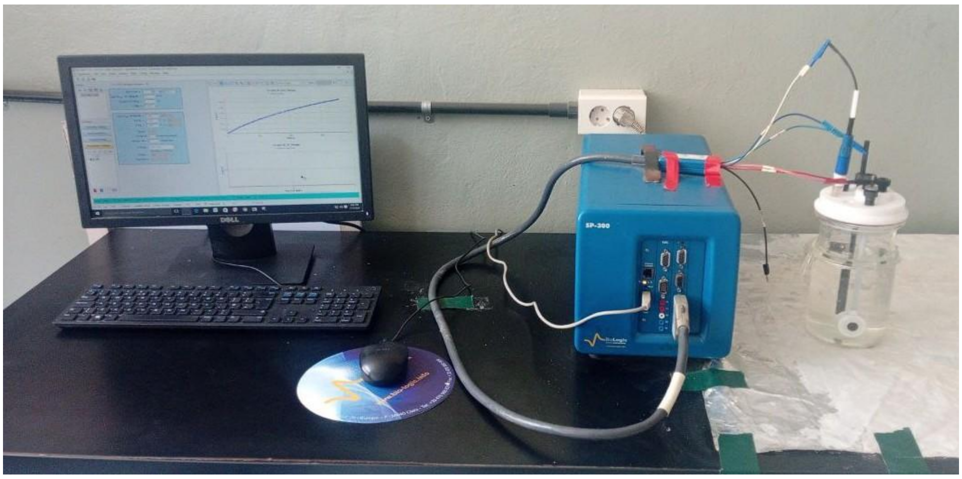

Figure 3.11 Potentiostat (SP-300) used for corrosion test

3.5.7 Wear Test

The gradual loss or degradation of material from a solid body's surface brought on by mechanical contact with another surface or environment is known as wear. In this current work the wear test was performed by utilizing pin-on-disc machine (TR-20-MICRO) as per ASTM G99-23 standard. The sample size was prepared with 12mm length and 6 mm diameter. The wear test process parameters were identified according to literature survey. The normal standard force of 30N (Sabry et al., 2020) was applied on the 30mm diameter of wear truck (Ambadekar & Choudhari, 2020) with 10 min (Alshalal et al., 2023) and operated at sliding speed 250 rpm (Gopal Krishna et al., 2019) at room temperature without lubrication. The device was powered on and the pin slid against the rotating disk for the test duration. Then, the sample was weighed before and after to observe how much weight was lost. The difference in weight can be used to calculate the wear. Knowing the wear mechanisms and the rate of wear was calculated by comparing the wear data. Weight loss, friction coefficient, wear rate, and volumetric wear rate, were determined using the following equations.

$$\Delta W = Wi - Wf,\ Sd = \pi DNT \tag{3.11}$$

$$Wr = \frac{\Delta W}{Sd} \tag{3.12}$$

$$Vloss = \frac{\Delta W}{\rho sample} \tag{3.13}$$

$$Wv = \frac{Vloss}{Sd} \tag{3.14}$$

$$\mu = \frac{Ff}{P} \tag{3.15}$$

Where, ΔW = weight loss of the sample (g),

S_d = sliding distance (m)

W_i = weight of sample before wear (g)

μ = friction coefficient

W_f = weght of sample after wear (g)

F_f = friction force (N)

W_r = wear rate (g/m)

P = applied load (N)

W_v = volumetric wear rate (mm^3/m)

N = sliding speed (rpm)

V_{loss} = the sample volume loss (mm^3)

T = duration time (s)

ρ_{sample} = density of the sample (g/ mm^3)

D = wear truck diameter (mm)

Figure 3.12 Micro pin on disc tribometer used for wear testing (located in AASTU)

3.6 Experimental design for fabrication of the composites

This experiment work was conducted on the impact of powder metallurgy process variables (sintering temperature and compaction pressure) and wt% of reinforcement (Al_2O_3 and B_4C). As the aim of this research is to increase properties of pure aluminum the sample processing were done by varying wt% of Al_2O_3 and B_4C.The wt% of reinforcement was established based on literature survey. According to Abebe Emiru et al.(2023) the hardness and strength of the matrix were improved by maintaining constant 4 wt% of MoS_2 and varying wt% of other reinforcements. MoS_2 was kept constant as wt% of MoS_2 was varied from 0wt% to 4wt% the mechanical properties were improved, above 4wt% the mechanical properties were degraded due to particle agglomeration and porosity improvement(Rao et al., 2021). And also in accordance of(T. S. K. Kumar & Kandavel, 2023) the hardness and corrosion resistant were improved by increasing wt% of B_4C (2,4,6), in the matrix. (Mahesh Kumar & Venkatesh, 2018) from this research that the hardness, compressive strength and wear resistance were improved as wt% of Al_2O_3 (4, 6, 8, 10) were increased. Thus as per previous researches the wt% of strengthening particles were thought to have improved hardness, compressive strength and density.

Table 3.8 Process parameters of PM and their levels

Levels	Factors			
	B_4C (wt%)	Al_2O_3 (wt%)	Compaction pressure (Mpa)	Sintering temperature(°C)
1	2	2	50	580
2	4	4	55	600
3	6	6	60	620

Table 3.9 Experimental design depends on L9 orthogonal array.

Samples	Reinforcements		Compaction	Sintering
	B_4C (A)	Al_2O_3 (B)	pressure (Pc)	temperature (Ts)
S1	2	2	50	580
S2	2	4	55	600
S3	2	6	60	620
S4	4	2	55	620
S5	4	4	60	580
S6	4	6	50	600
S7	6	2	60	600
S8	6	4	50	620
S9	6	6	55	580

3.7 Optimization Method

To determine the value at which wt% of reinforcement, sintering temperature, and compaction pressure is best and analyzing data is very important. GRA and ANOVA were utilized to optimize and analysis the wt% of reinforcement and both powder metallurgy parameters based on the result obtained from experimental testing. Grey relational analysis was applied to convert multi-responses from experiments carried out by design of experiments of taguchi method (table 3.8). It is a robust decision-making and optimization tool, particularly with complex systems involving incomplete or imprecise information. Following steps were followed to apply the grey relational analysis.

Step1: Convert the obtained response data in to S/N ratio (*Xi*) through the following formulas.

$$\eta = -10\log[\frac{1}{n}\sum_{i=1}^{n}(Xi^2)] \quad (3.16)$$

Equation (3.16) can be used when smaller is better.

$$\eta = -10\log[\frac{1}{n}\sum_{i=1}^{n}\left(\frac{1}{Xi^2}\right)] \quad (3.17)$$

Equation (3.17) can be used when larger is better.

Where: η = signal to noise ratio

Xi = the experimental result per trial

n = number of replications

i = number of trials

The result of porosity and density was converted to signal to noise ratio using equation (3.16) since its smaller result was needed. But the experimental result of hardness was converted to signal to noise ratio by equation (3.17) since the higher result was needed.

Step 2: Normalize the result data to minimize uncertainty and eliminate the impact of using different units. One of the inputs is normalized, a process that scales the data into an appropriate range for additional analysis and distributes it uniformly. The range of obtained value is between 0 and 1.

$$Zij = \frac{max(Xij) - Xij}{max(Xij) - min(Xij)} \tag{3.18}$$

Equation (3.18) was used for normalizing the S/N ratio of density and porosity.

$$Zij = \frac{Xij - \min(Xij)}{max(Xij) - min(Xij)} \tag{3.19}$$

Equation (3.19) was used for normalizing the S/N ratio of hardness.

Where: *Zij* = the *i*th normalized value of *j*th response

Xij = signal to noise ratio value for *i*th trial of *j*th response

i = trial number (*i* = 1, 2, ..., n)

j = response number (j = 1, 2, …, m)

Step 3: Determine the gray relational coefficient for the S/N ratio data that have been normalized.

$$Gcij = \frac{\Delta min + \gamma \Delta max}{\Delta ij + \gamma \Delta max} \tag{3.20}$$

Where: *Gcij* = grey relational coefficient for *i*th trial and *j*th response

Δ = absolute difference between Zmax and Zij

Δmax = maximum value of Δ

Δmin = minimum value of Δ

Zmax = maximum value of normalize S/N ratio

γ = distinguishing coefficient that is defined in the range of $0 \leq \gamma \leq 1$

Step 4: Determine grey relational grade (*Gi*)

$$Gi = \frac{1}{m} \sum Gcij \tag{3.21}$$

Where: m = number of responses

Step 5: Execute ANOVA and select best values for parameters by highest mean value of Gij. Such ANOVA receives input parameters like sintering temperature, compaction pressure and wt% reinforcements.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Introduction

Here in this part of the study, the experimental works are described elaborately. The study aimed to create the composite material by incorporating aluminum oxide (Al_2O_3), boron carbide (B_4C) and molybdenum disulphide (MoS_2) into pure aluminum. Hardness test, compressive test, corrosion test, wear test, density measurements and porosity measurements, and characterization of material results of the developed composite through powder metallurgy process were studied and reported. Powder metallurgy process parameters and wt% of reinforcements were optimized for achieving enhanced properties.

4.2 Experimental Results and Discussions

In this part, all the obtained results of experiments were discussed briefly in tables and graphs.

4.2.1 Hardness test

Composites developed hardness was measured using Vickers hardness tester. It was conducted at three points on samples surface. The values of the hardness obtained in Vickers hardness test are displayed in Table 4.1.

Table 4.1 Hardness values of the composite

Sample	Vickers hardness (HV)			Average Hardness	S.Dev
	Trial 1	Trial 2	Trial 3		
S1	136.9	137.7	134.3	136.3	1.78
S2	174.3	176.6	180.1	177	2.38
S3	215.9	213.2	217.5	215.5	1.77
S4	197.4	199.1	200.6	199	1.31
S5	206.5	209.3	207.1	207.6	1.20
S6	164.7	163.7	159.4	162.6	2.30
S7	222.3	221.4	219.8	221.2	1.03
S8	191.8	192.5	194.2	192.8	1.01
S9	154.7	151.3	144.3	150.1	4.33

The finding obtained from (S. K. Sharma et al., 2025) had explained that an increased compaction pressure reduces voids in the green compact reducing porosity and additionally very much, the hardness of the composite was improved. Likewise with the rise in sintering temperature densification has increased with bonding of the composite particles as well as reduced porosity resulting in increased hardness of composites (Sultan et al., 2024). But when we compare wt% of reinforcements, the higher wt% of reinforcements had lower hardness value of composites. This showed that when sintering temperature and compaction pressure was low or medium, and wt% of reinforcements are higher the voids in the sample was increased then the composite hardness decreases as compared to higher hardness value of composites which were obtained with S3 and S7. The influence of those process variables and weight percentage on the hardness of the final composite was given in figure 4.1.

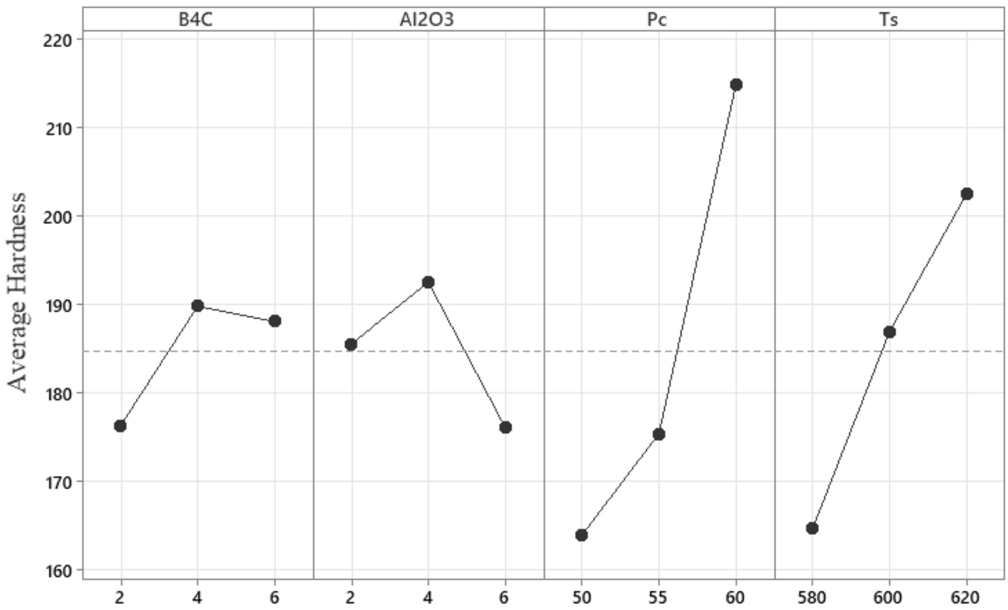

Figure 4.1 Effect of reinforcements and process parameters on hardness

As the compaction pressure, sintering temperature and weight percentage of reinforcements were all increased as revealed by figure 4.2 the hardness were improved from sample one up to sample three. However, sample four registered lower hardness value in comparison to that of sample three. This refers to the reality that a lower compaction pressure or a lower weight percentage of aluminum oxide is most likely to be responsible for this. Although the fourth sample was bigger than the second one, it was less than the third. It shows that with increasing % of weight of B_4C, the hardness rose. Following S1 and S9 the S6 had lower value of hardness. It demonstrated that although the sample has more wt% percentage of reinforcements, it has less hardness and more porosity due to the lesser compacting pressure. Both the S6 and S8 were sintered under the same pressure but the S8 sample had a greater wt% of B_4C and a sintering temperature than the S6. Therefore, the S8 hardness was greater because of the greater wt% of B_4C and sintering temperature.

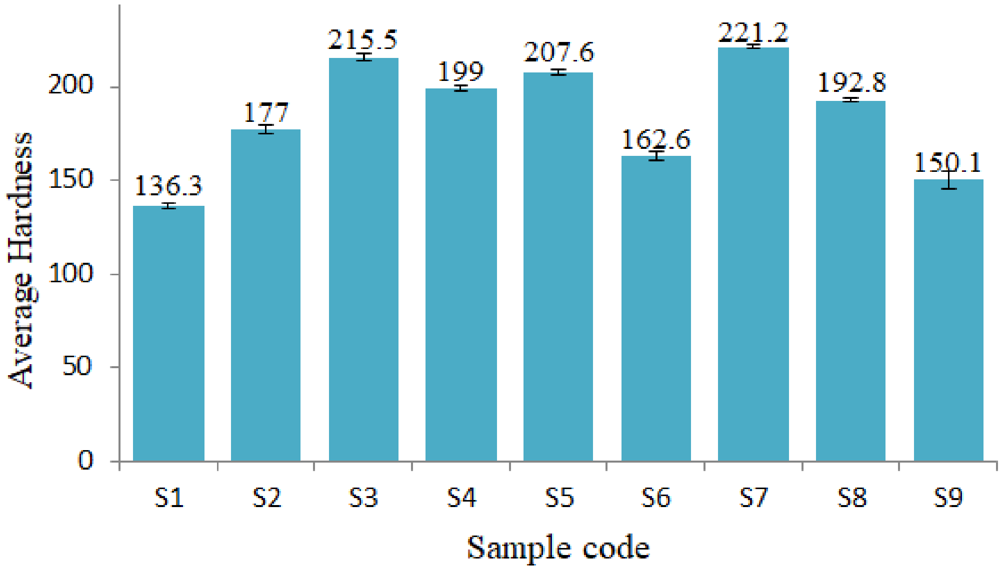

Figure 4.2 hardness value of the composite

Also S1 and S9 were utilized same temperature but more hardness was provided at S9 because of the compaction pressure and wt% of reinforcements increase. Nevertheless, it had less hardness than others. Overall in order to improve hardness the composites reinforcement percentage increment was not enough then compaction pressure and sintering temperature increment were very crucial.

Table 4.2 Response table for signal to noise ratios of hardness

Level	B_4C	Al_2O_3	Pc	Ts
1	44.77	45.19	44.21	44.19
2	45.52	45.67	44.82	45.36
3	45.38	44.81	46.64	46.12
Delta	0.74	0.86	2.43	1.93
Rank	4	3	1	2

As indicated in table 4.2 response table the most significant factor on hardness was compaction pressure which was followed by sintering temperature. But, wt% of reinforcements has less impact on hardness.

Table 4.3 Analysis of variance for hardness

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% contribution
Regression	4	6363.6	1590.9	7.94	0.035	
B4C	1	207.3	207.3	1.03	0.367	2.89
Al2O3	1	133.2	133.2	0.66	0.461	1.86
Pc	1	3881.1	3881.1	19.36	0.012	54.16
Ts	1	2142.0	2142.0	10.68	0.031	29.89
Error	4	801.9	200.5			
Total	8	7165.5				

Table 4.3 revealed that % contribution of Pc, Ts, B4C and Al2O3 on hardness was 54.16%, 29.89%, 2.89% and 1.89 % respectively.

4.2.2 Density of the Composite

The Archimedes method was carried out for the determination of density of composite samples that were made. Weighing of weights of every nine samples with three trials was performed and their densities were determined by using equation (3.2) and the experimental densities obtained were presented in table 4.4.

Table 4.4 Density of the composite

Sample	Density (g/cm^3)			Average density	S.Dev
	Trial 1	Trial 2	Trial 3		
S1	2.5550	2.5484	2.5468	2.550	0.004
S2	2.6161	2.6101	2.5497	2.592	0.030
S3	2.7074	2.6859	2.6823	2.685	0.011
S4	2.6419	2.6408	2.6295	2.637	0.006
S5	2.5780	2.5631	2.5317	2.558	0.019
S6	2.5354	2.5233	2.4849	2.499	0.022
S7	2.6158	2.5817	2.5666	2.588	0.021
S8	2.5147	2.5234	2.5209	2.520	0.004
S9	2.5060	2.4928	2.4702	2.481	0.015

Since alumina and molybdenum disulphide are denser than aluminum, the addition of those reinforcements into AMMCs increases its theoretical density as recognized by the rule of mixture theory. However, experimental density was influenced by compaction pressure, sintering temperature, and reinforcement properties too. The relationship of experimental density to wt% reinforcements and process parameters was demonstrated by figure 4.3. As can be noticed in figure 4.3, addition of the wt% of aluminum oxide (Al_2O_3) and boron carbide (B_4C) particles lowered the density of processed aluminum metal matrix composites, this due to the hard nature of the reinforcement particles (Annigeri & Veeresh Kumar, 2018),(Ashebir et al., 2022). However as we have seen from figure (4.3) the composites densities were increased with increasing compaction pressure, and sintering temperature. According to (Yuan et al., 2021) explained that with the rise in sintering temperature as well as the density of the composites as a result of densification, there was excellent particle bonding and fewer pores. Apart from this, the density of the composites increased with higher compaction pressure due to more touching surfaces between the particles that initiates densification and decreases porosity (Dixit & Srivastava, 2018).

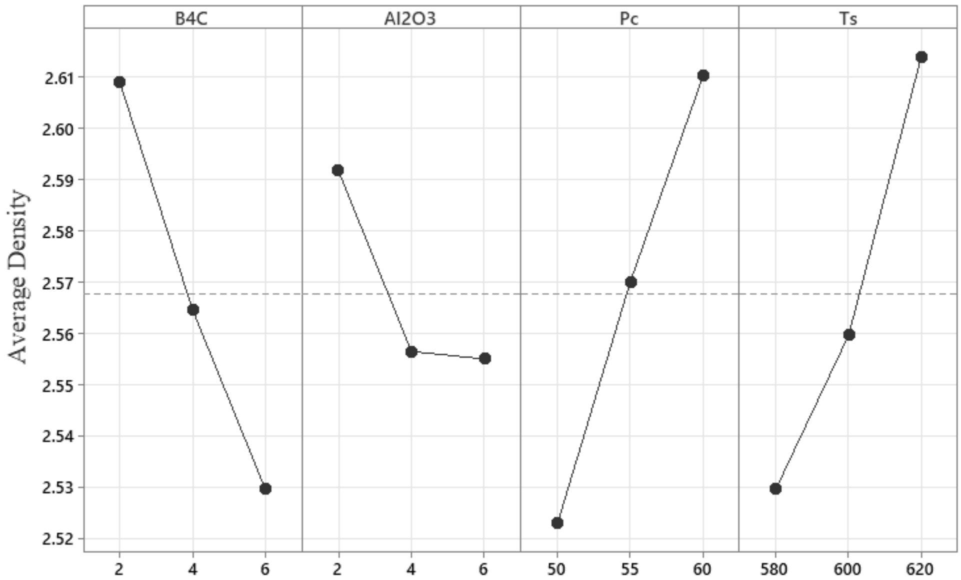

Figure 4.3.The effect of reinforcements and process variables on density.

As depicted in figure 4.4 Sample nine possessed lowest density as compared to sample one, even though it possesses high wt% of reinforcements which increase porosity it was sintered at moderate pressure and low sintering temperature. Apart from this the density of the composites increased from sample one to sample three. This implies that it has direct proportionality with sintering conditions, compaction pressure and wt% of Al_2O_3 reinforcement. But density was lost at sample four by decreased compaction pressure.

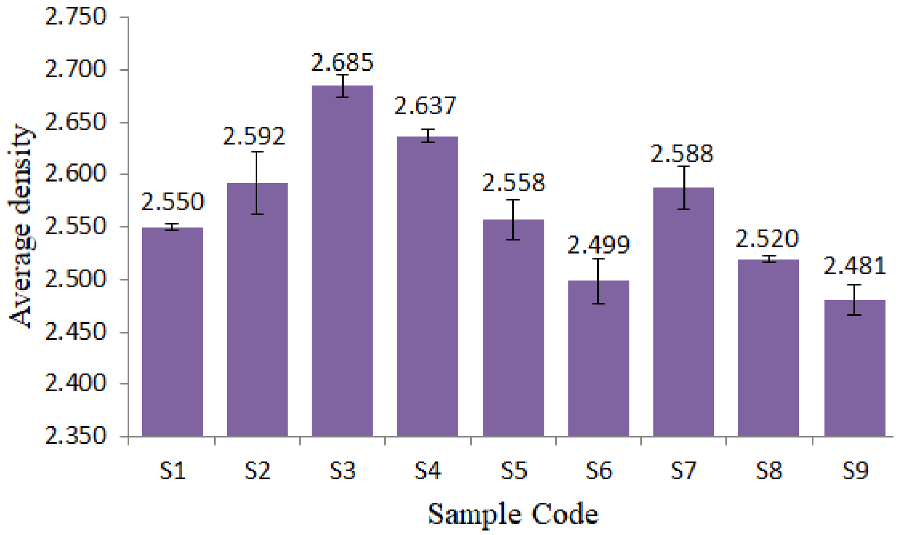

Figure 4.4 Density of the composites

And also sample five was lower density than sample four because of lower sintering temperature. Sample three was the mostly dense, because it was formed under higher sintering temperature and compaction pressure. According to (Gemeda et al., 2022), (Annigeri & Veeresh Kumar, 2018) decreasing compaction pressure, decreasing sintering temperature and increasing the wt% of reinforcement lowers the density of the composite.

As it has been demonstrated from table 4.5 response table of S/N ratio depicts how reinforcement and process variables control density. The primary factor used to influence the density of fabricated composite was compaction pressure. Though compaction forces minimize the porosity but increase the density significantly. Second most important parameter was sintering temperature since particles were appropriately bonded. Lastly, boron carbide reduces the density of processed composite increasing its wt% but alumina has less impact.

Table 4.5 Response table for signal to noise ratios of density

Level	B_4C	Al_2O_3	Pc	Ts
1	-8.328	-8.271	-8.037	-8.060
2	-8.178	-8.152	-8.196	-8.162
3	-8.059	-8.142	-8.332	-8.343
Delta	0.268	0.129	0.294	0.283
Rank	3	4	1	2

Table 4.6 Analysis of variance for density

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% contribution
Regression	4	0.033730	0.008433	35.77	0.002	
B4C	1	0.009494	0.009494	40.27	0.003	27.38
Al2O3	1	0.002041	0.002041	8.66	0.042	5.89
Pc	1	0.011469	0.011469	48.65	0.002	33.08
Ts	1	0.010726	0.010726	45.49	0.003	30.93
Error	4	0.000943	0.000236			
Total	8	0.034673				

Table 4.6 showed that that % contribution of Pc, Ts, Al_2O_3 and B_4C on density was 33.08%, 30.93%, 27.38% and 5.89 % respectively.

4.2.3 Porosity of the composites

Archimedes technique was used to determine porosities of the composites fabricated. It was determined using equation (3.7) as shown in table 4.7 through experimental and theoretical density. Process variables and wt% reinforcement effects on porosity were described in figure 4.5. As indicated in figure 4.5 the porosity of the manufactured composites were rose as wt% of reinforcements were increased.

Table 4.7 Porosity values of the composites

Sample	Porosity (%)			Average Porosity	S.Dev
	Trial 1	Trial 2	Trial 3		
S1	9.212	9.444	9.500	9.385	0.125
S2	7.884	8.094	10.222	8.733	1.056
S3	5.528	6.278	6.402	6.302	0.386
S4	6.002	6.043	6.444	6.163	0.200
S5	9.109	9.635	10.743	9.829	0.681
S6	11.418	11.842	13.183	12.697	0.752
S7	6.812	8.026	8.563	7.800	0.732
S8	11.230	10.922	11.010	11.054	0.130
S9	12.333	12.797	13.586	13.214	0.517

The studies,(Edy et al., 2019), (Bilal et al., 2020) mentioned that with the rising wt% of the reinforcing particles, porosity of the composite also rose. However, with the improvement of compacting pressure and sintering condition, composites porosity were reduced. In the study (Obada et al., 2020) it was explained in detail that porosity of the material was reduced by raising the sintering temperature. According to the (Manohar et al., 2020) due to the existence of good bond between reinforcements particles and matrix at high compaction pressure the composites porosity were reduced.

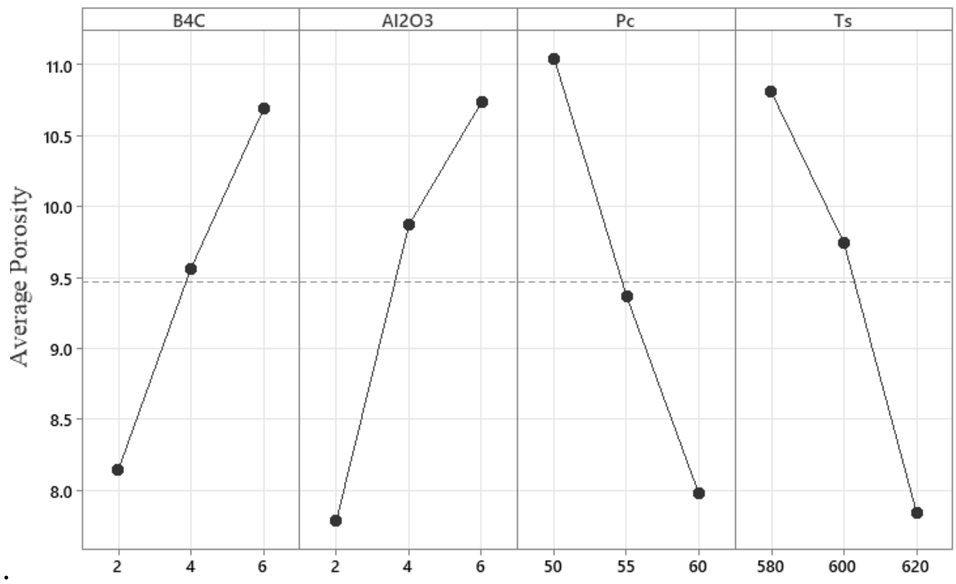

Figure 4.5 The effect of reinforcements and process variables on porosity

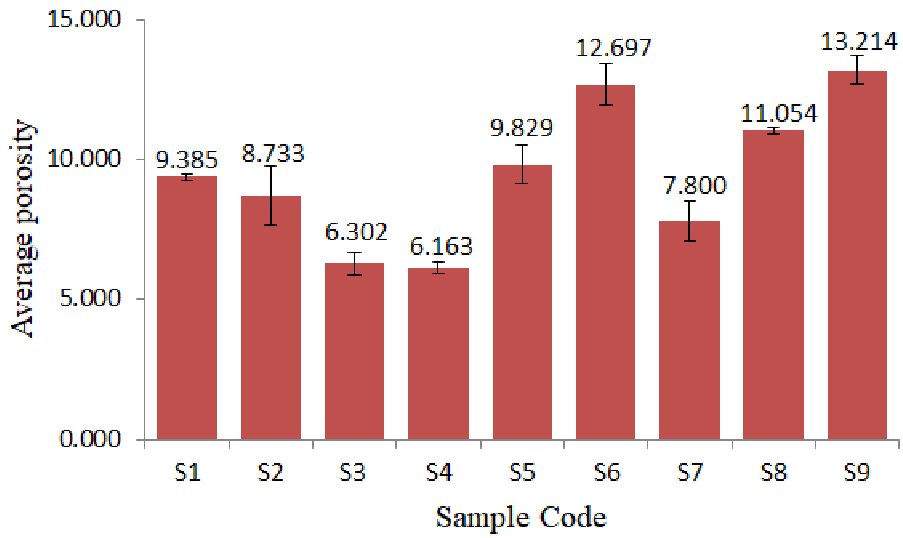

Figure 4.6 Porosity of the composite

The percentage porosity of the formed composites were described in figure 4.6, this figure indicated that the porosity decreased from sample one to sample four with increasing sintering temperature and compaction pressure but density was increased. This revealed that the porosity and density were inversely related. Minimum porosity was calculated on sample four which was developed under moderate compaction pressure and high sintering temperature. Since sample nine was processed under low sintering conditions and medium compaction pressure, it had maximum porosity even though it had higher wt% of reinforcement particles. Sample seven had low percentage of porosity after sample three due to the medium temperature and high compacting pressure. In addition, when the compaction pressure and sintering temperature were reduced the porosity percent of the fabricated composites were greater, or when they raised the porosity of the composite was less.

Table 4.8 Response table for signal to noise ratios of porosity

Level	B_4C	Al_2O_3	Pc	Ts
1	-18.09	-17.70	-20.80	-20.57
2	-19.24	-19.85	-19.01	-19.58
3	-20.38	-20.16	-17.89	-17.55
Delta	2.29	2.47	2.90	3.02
Rank	4	3	2	1

The response table for signal to noise ratio of porosity tabulated in table 4.8 showed that the most significant factor on porosity was sintering temperature and then followed by compaction pressure.

Table 4.9 Analysis of variance for porosity

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% Contribution
Regression	4	50.195	12.5487	42.41	0.002	
B4C	1	9.748	9.7477	32.94	0.005	18.97
Al2O3	1	13.097	13.0965	44.26	0.003	25.49
Pc	1	14.122	14.1221	47.72	0.002	27.49
Ts	1	13.228	13.2284	44.70	0.003	25.75
Error	4	1.184	0.2959			
Total	8	51.378				

The percentage contribution of the factors on porosity was determined in table 4.9. Thus the compaction pressure had the highest % contribution followed by sintering temperature and aluminum oxide.

4.2.4 Compression Test

Following ASTM Standard B925-15, the compression strength test was performed on the sample processed at optimal conditions with the aid of UTM. The maximum compressive strength of the composite was 390.23MPa, which indicated that the incorporation of reinforcements (Al_2O_3, B_4C, and MoS_2) enhanced the capacity of aluminum to sustain compressive loads without failure. In Figure 4.7, the stress-strain graph for this test was displayed. The study, (Tenali et al., 2019), (Alam et al., 2023)reported that as the wt% of particles increased, it was evident that the strength of composites to compression had increased gradually.

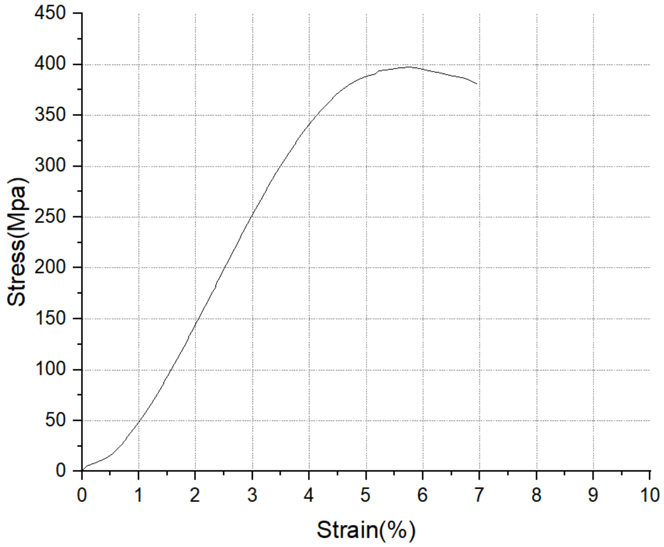

Figure 4.7 Stress – strain graph of the developed composite

4.2.5 Wear Test

Pin-on-disc wear tester machine has been employed to measure wear strength of the composite developed. It was performed at room temperature in the absence of lubrication. This test was applied on the composite sample prepared at optimal level and pure aluminum and the sample size was prepared with 12mm length and 6mm diameter and it was polished to provide better results. The experimental results were analyzed in table 4.6. Using equations (3.11),(3.12),(3.13),(3.14) and (3.15) the experimental results weight loss, wear rate, volume loss, volumetric wear rate and COF were determined respectively. As we have seen from the results achieved the processed composite has lower friction coefficient and wear rate than pure aluminum. The coefficient of friction and wear rate were improved by 30.2% and 32% respectively.

Table 4.10 Experimental results of wear test

Samples	Weight loss (g)	Wear rate (g/m)	Volume loss (mm^3)	Volumetric wear rate (mm^3/m)	Coefficient of friction
Pure Al	0.0017	$7.22x10^{-6}$	0.63 mm^3	$2.67x10^{-3}$	0.53
AMMC	0.0011	$4.67x10^{-6}$	0.407mm^3	$1.73x10^{-3}$	0.37

Incorporation of ceramic hard particles into the composites reduced material loss resulting from wear through increasing the surface hardness and reinforcement improve surface integrity through a decreased in the volume of material removed this concurs with (Annigeri & Veeresh Kumar, 2018). In addition to this increase in wt% reinforcement particles of Al_2O_3 reduces the wear rate of the composites according to (Al-Salihi et al., 2019). Since Al_2O_3 and B_4C possess greater wear resistance compared to pure aluminum, and MoS_2 used as solid lubricant for the reduction of friction the wear strength of the composite aluminum was increased. The fluctuation of wear rate with time after testing at 250rpm and applied load of 30N was indicated in figure 4.8.

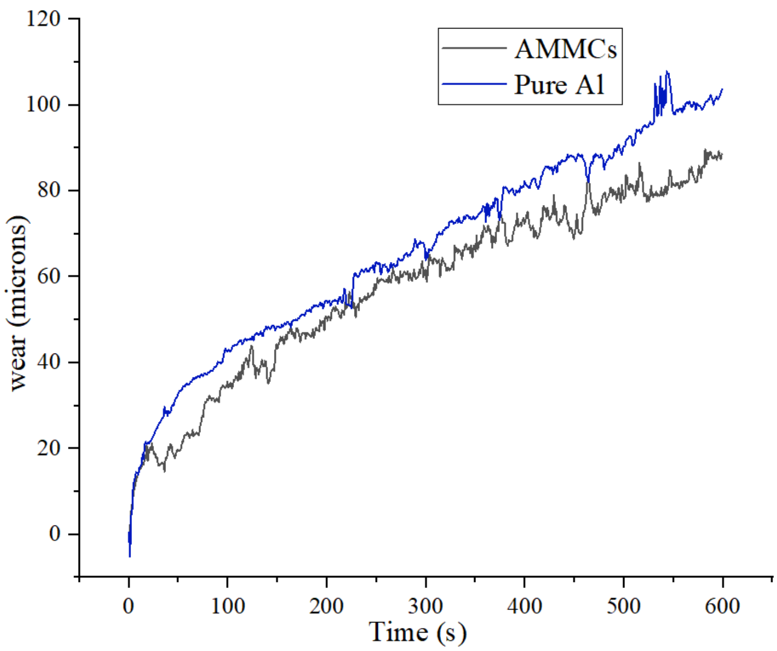

Figure 4.8 Graph of wear rate with time

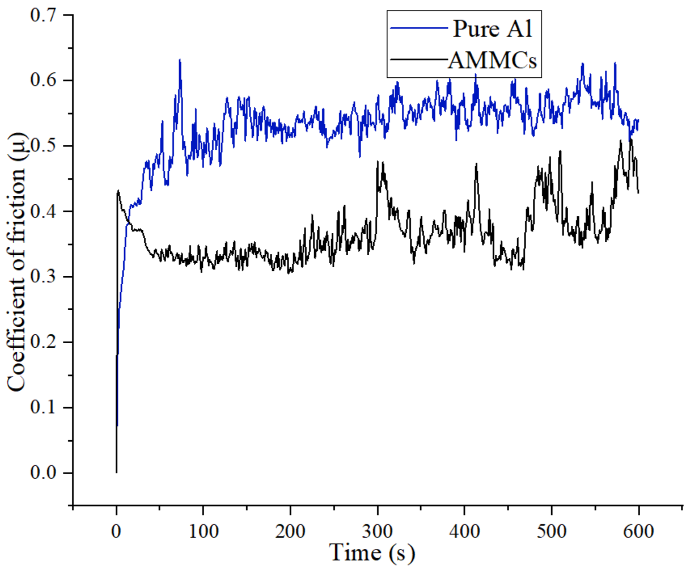

Figure 4.9 The fluctuation of friction coefficient with time

4.2.6 Corrosion Test of the composite

The corrosion of the developed composite was tested through electrochemical cell. And the corrosion rate was calculated using equation (3.10). The computed corrosion rate of the optimum sample and pure aluminum were 0.0023 mmpy and 0.0042 mmpy respectively. Using origin software the corrosion current densities and corrosion potentials of both samples which evaluated were discussed in the figure 4.11 and 4.12.

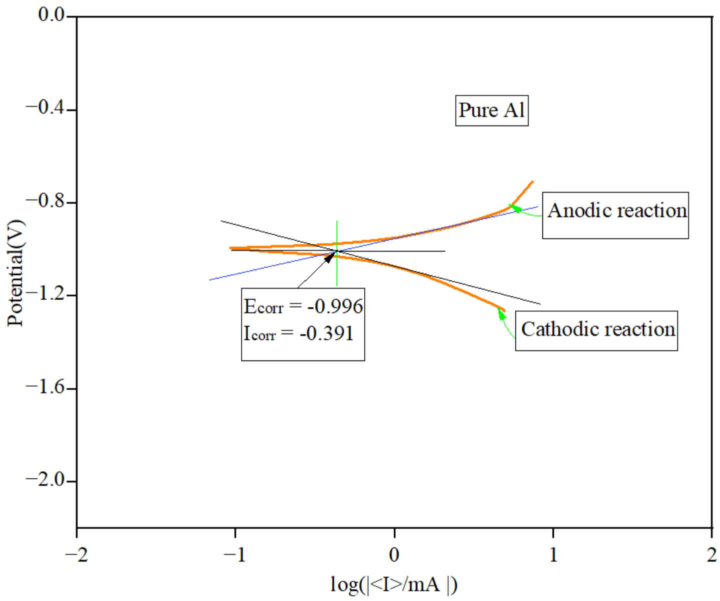

Figure 4.10 Tafel curve analysis of pure aluminum

The determined corrosion rate indicated that lower corrosion rate was obtained on the sample fabricated at the optimum level rather than that of pure Al sample. The studies (Alshammri et al., 2023);(Xin et al., 2025) reported that the addition of particles in to the matrix improved the corrosion resistance of the composites due to creation of protective barrier for prevents it from being directly exposed to corrosive conditions. Additionally, high corrosion potential was obtained on optimum sample in relation to pure sample; this shows that the Al composite was more corrosion resistant than pure Al. Similarly, the optimum samples corrosion current density was less than that of pure sample; this suggests that its rate of corrosion was slower than pure sample.

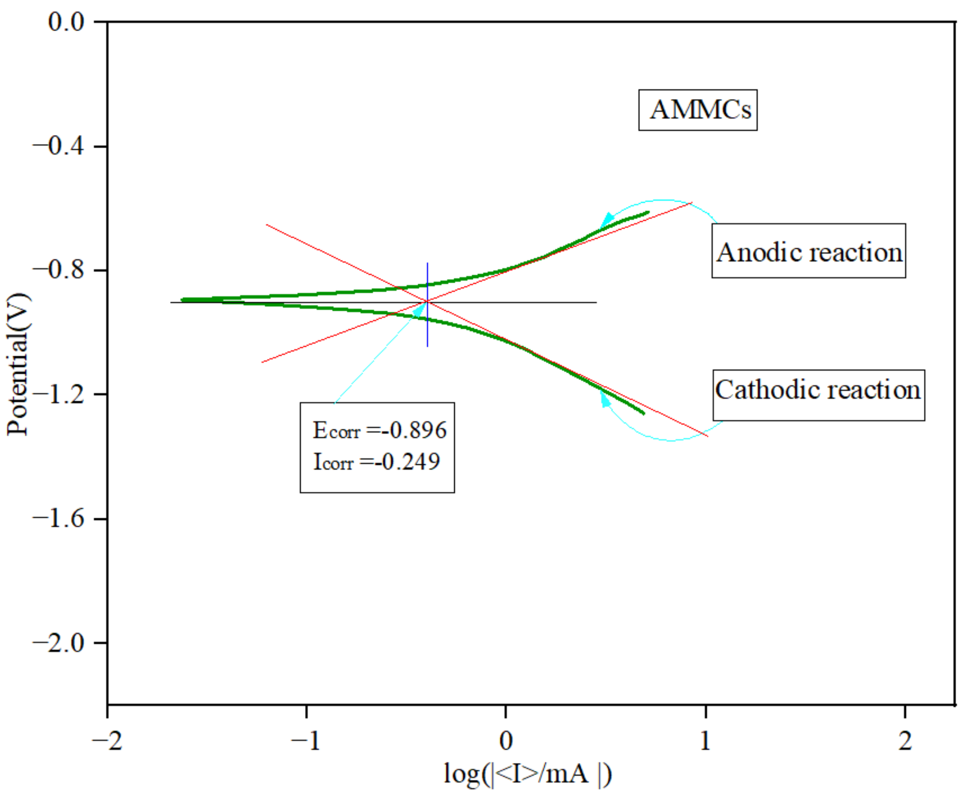

Figure 4.11 Tafel curve analysis of aluminum composite

4.3 Optimization method

The improvement of developed aluminum composite wt% reinforcements and process parameters for strengthening mechanical properties was being deliberated under this subsection. Optimization process was implemented for enhancing hardness, compressive strength and density of developed samples by reducing its porosity. Taguchi based-Grey relational analysis (GRA) and ANOVA technique were applied for process parameter and wt% of reinforcement's optimization.

4.3.1 Grey relational analysis

To carry out grey relational analysis, experimental results obtained were initially converted to S/N ratio. Since larger hardness was required, larger is better was used for conversion to signal to noise ratio equation (3.17) and smaller is better was utilized for porosity and density since lower was required equation (3.16). Data of the results converted to signal to noise ratio were presented in table 4.11. Following the transformation of the S/N ratio the normalization of the results obtained in S/N ratio was done through equation (3.19) for hardness since bigger result was required, and equation (3.18) for porosity and density since smaller results were required. Afterwards, as shown in table 4.12 the grey relational coefficient (Gcij) and grey relational grade (Gi) were determined using equation (3.20) and (3.21) respectively.

Table 4.11 S/N ratio of hardness, porosity and density

Samples	S/N of Hardness	S/N of Density	S/N of Porosity
S1	42.69	-8.13	-19.45
S2	44.96	-8.27	-18.89
S3	46.67	-8.60	-15.68
S4	45.98	-8.42	-15.80
S5	46.35	-8.16	-19.87
S6	44.22	-8.01	-21.71
S7	46.89	-8.26	-17.88
S8	45.70	-8.03	-20.87
S9	43.52	-7.92	-22.22

Grey relational coefficient is helpful in decision-making and ranking since it helps in quantifying the degree to which various elements belong to the optimum solution. Grey relational grade gives overall evaluation of the alternatives, which enables them to pick the most desirable option from amongst several.

4.3.2 Analysis of variance for grey relational grade

After determination of grey relational grade the ANOVA test was performed on grey relational grade to find out the most dominant factor or to find out percentage contribution on it by process variables (Pc and Ts) and reinforcement weight percentages (B_4C and Al_2O_3 wt%). In order to investigate the impact of input variables on response results, Taguchi L9 orthogonal array was set up. Parameters wt% boron carbide (2, 4 and 6 wt%), alumina (2, 4 and 6 wt%), sintering temperature (620, 600 and 580 °C) and pressure (50,55 and 60 Mpa) were considered.

Table 4.12 Grey relational coefficient, normalized S/N ratio and grey relational grade results

Sample	Normalized S/N ratio (*Zij*)			Grey relational Coefficient (*Gcij*)			Grey Relational Grade (*Gi*)
	HV	Density	Porosity	HV	Density	Porosity	
S1	0.000	0.307	0.576	0.333	0.419	0.541	0.431
S2	0.539	0.516	0.490	0.521	0.508	0.495	0.508
S3	0.947	1.000	0.000	0.903	1.000	0.333	0.746
S4	0.782	0.738	0.018	0.697	0.656	0.337	0.563
S5	0.870	0.345	0.641	0.793	0.433	0.582	0.603
S6	0.364	0.128	0.921	0.440	0.364	0.864	0.556
S7	1.000	0.496	0.336	1.000	0.498	0.430	0.643
S8	0.717	0.153	0.793	0.638	0.371	0.708	0.572
S9	0.197	0.000	1.000	0.384	0.333	1.000	0.572

The grey relational grade's variance analysis was performed using Minitab software as stated in table 4.13.

Table 4.13 Analysis of variance

Source	DF	Seq SS	Adj SS	Adj MS	P-Value	Contribution (%)
Regression	4	0.054948	0.054948	0.013737	0.023	
B4C2	1	0.001734	0.001734	0.001734	0.321	2.87
Al2O3	1	0.009362	0.009362	0.009362	0.058	15.51
Pc	1	0.031248	0.031248	0.031248	0.009	51.77
Ts	1	0.012604	0.012604	0.012604	0.038	20.88
Error	4	0.005409	0.005409	0.001352		
Total	8	0.060357				

The contribution % obtained from analysis of variance in table 4.8 for boron carbide weight percentage, aluminum oxide weight percentage, sintering temperature and compaction pressure were 2.87%, 15.51%, 20.88% and 51.77% respectively. Hence the compaction pressure had a great effect on the grey relational grade. This suggests a parameter which is most affecting variable for hardness, porosity, and density was compaction pressure, followed by sintering temperature and aluminum oxide wt%.

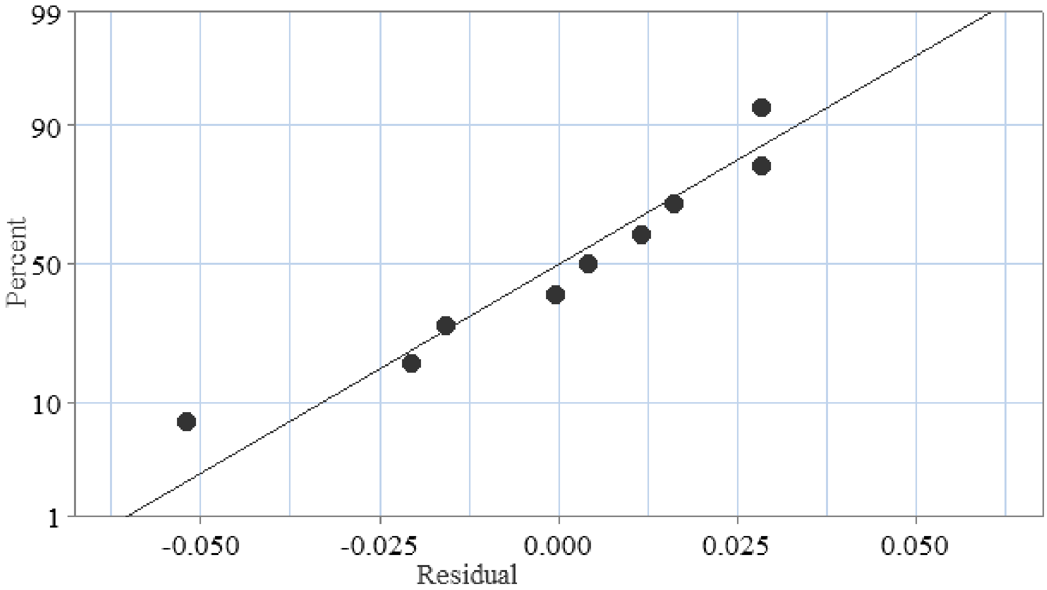

Figure 4.12 Normal probability plot

Grey relational grade normal probability plot was illustrated as in figure 4.12. It shows the plot points to be nearly normally distributed. This establishes the validity of the regression model.

4.3.3 Confirmation of sample developed at optimum

The sample that was created under the optimum manufacturing conditions was used for the confirmation test. To check if samples processed at the optimum level were greater than samples that had already been processed, this test was conducted. The optimum levels used were 60Mpa of compaction pressure, 620°C of sintering temperature, weight percentage of reinforcements (boron carbide 6 wt% and aluminum oxide 6 wt%). Since optimization technique was carried out on hardness, density and porosity the optimal level of specimen were obtained from them as shown in table 4.9. A decrease in porosity percentage, improvement in density, and a better hardness were indicated by the outcomes of experiments that were achieved. Porosity percentage in the composites decreased compared to the previous result. But the optimum density was enhanced.

Table 4.14 The experimental outcome of the confirmation test

Trial	Hardness	Density	Porosity
1	226.1	2.757	3.562
2	218.9	2.773	3.005
3	229.4	2.75	3.786
Average	224.8	2.76	3.451

4.4 Analysis of Microstructure of the Composite

Microstructure was regarded as the primary test during material characterization. The true properties of tested samples can be recognized from visible microstructure patterns. X-ray diffractometer, scanning electron microscopy and optical microscopy were used in this study to analyze the phase of the material, reinforcement distribution and microstructure.

4.4.1 Optical Microscope

The reinforcement particle distribution within the aluminum metal matrix composites was investigated with optical microscope as described in figure 4.13. To examine the distribution of reinforcement within the matrix the developed sample at the optimal level was mounted beneath the optical microscope. The uniform distributions of reinforcement particles in the matrix were revealed at various magnification levels. This indicated that the carried out mixing processes were effective.

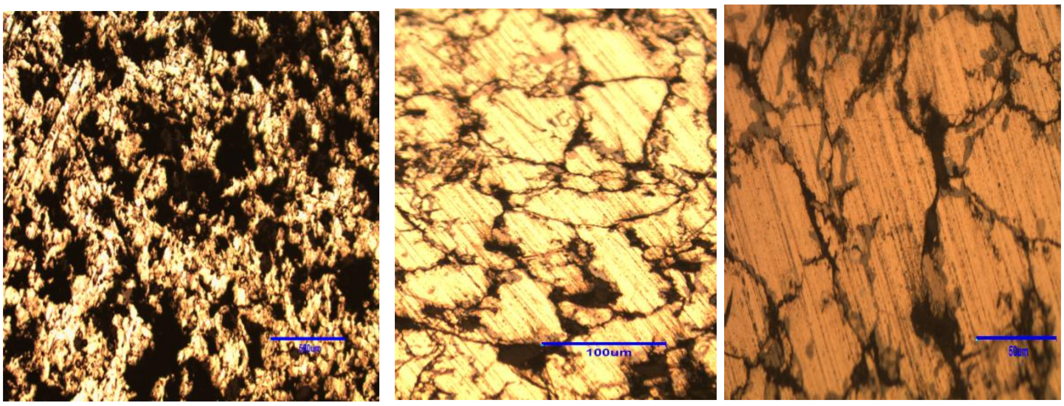

Figure 4.13 The micrograph of developed composite by OM

The average grain size was determined using optical microscopic image by image-J software analysis. Based on the image obtained from OM the grain size was determined from 5.617 µm to 10.755 µm using image analysis. The average grain size was determined using five trials from different image of specimen location was 7.89 µm.

4.4.2 Scanning Electron Microscope

In this analysis of microstructure a concentrated electron beam was utilized to scan a samples surface and produce photographs of it. Electron-atom interactions generate signals that indicate the composition and morphology of the samples surface, which are subsequently utilized to form an image. Under method detailed surface of the produced sample at optimum was revealed, which cannot be shown by optical microscope. Then the surface morphology of the sample was revealed by utilizing scanning electron microscope at different levels of magnifications as illustrated in the figure 4.14. The porosity, reinforcing particles and the matrix were revealed through this method.

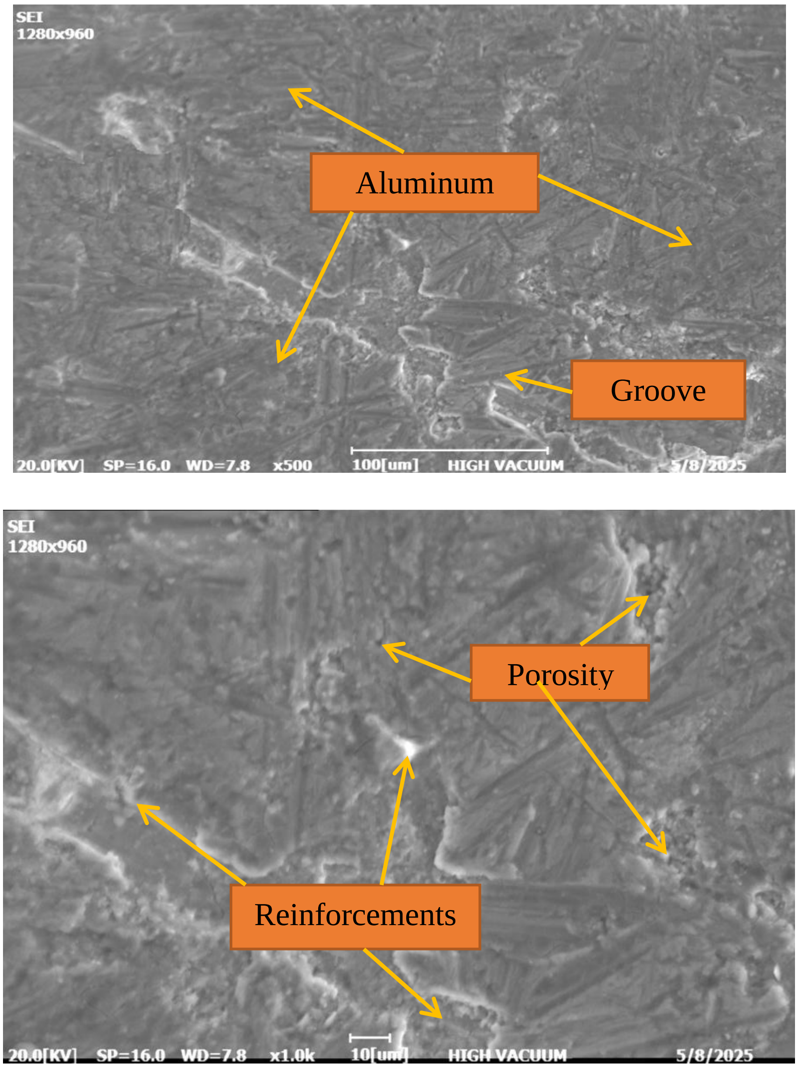

Figure 4.14 SEM analysis of the fabricated composite

4.4.3 X-Ray Diffraction (XRD)

This test was conducted on the developed sample at an optimum level to study the existence of the reinforcements in the aluminum matrix composites. The tested result illustrates the presence of the alumina, boron carbide and molybdenum disulphide as shown in the figure 4.9. Data acquisition system of XRD machine automatically calculated the outcome of the experiment after XRD test as presented in the table 4.8. Matching interplanar spacing and diffraction angle with standard reference patterns of powder diffraction file data base prepared by International Centre of Diffraction Data (ICDD), the phases appeared were identified.

Table 4.15 XRD experimental result

Peaks	2θ (degree)	d(Å)	FWHM
1	25.5366	3.48538	0.392
2	28.9977	3.07676	0.33762
3	36.2237	2.47786	0.2295
4	38.5560	2.33317	0.29445
5	44.8171	2.02068	0.32062
6	56.2021	1.63536	0.30774
7	65.2370	1.42901	0.36397
8	78.3915	1.21888	0.41374
9	82.6128	1.16697	0.38361

Their card numbers are aluminum oxide (00-046-1212), molybdenum disulphide(00-037-1492) boron carbide(00-035-0798) and aluminum (00-004-0787). In comparison with reference standard patterns peaks (4,5,7,8 and 9) were showed phases of aluminum. Their miller indices were (111,200,220,311 and 222) respectively. Peaks (2and 6) revealed boron carbide phase with miller indices (021,110). Similarly peaks (1 and 3) showed phases of aluminum oxide and molybdenum disulphide respectively. Their miller indices are (111 and 101) respectively.

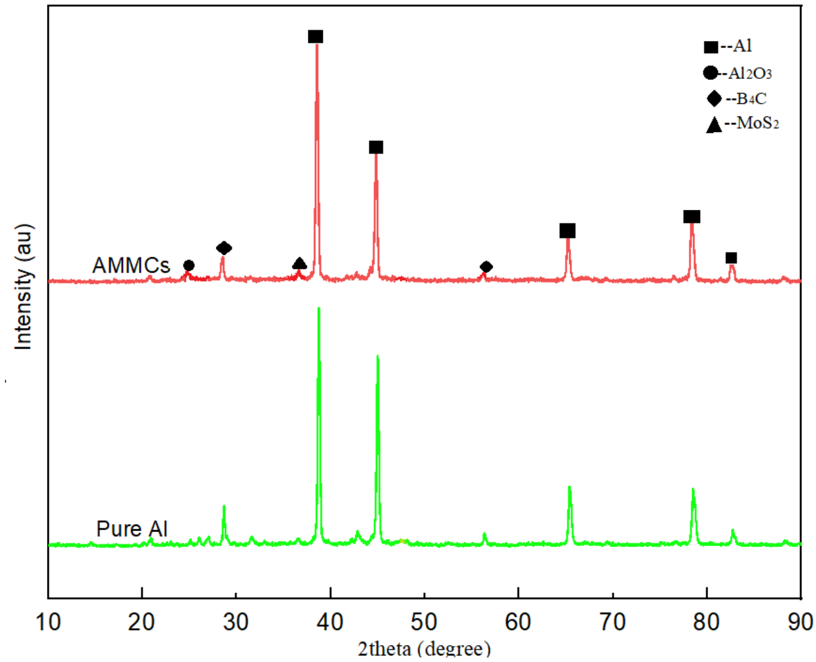

Figure 4.15 XRD analysis of the Al composites and pure Al

4.4.4 Energy Dispersive X-ray Spectroscopy (EDS)

The elemental mapping of the developed composite were revealed by utilizing energy dispersive x-ray spectroscopy as shown in figure 4.16.

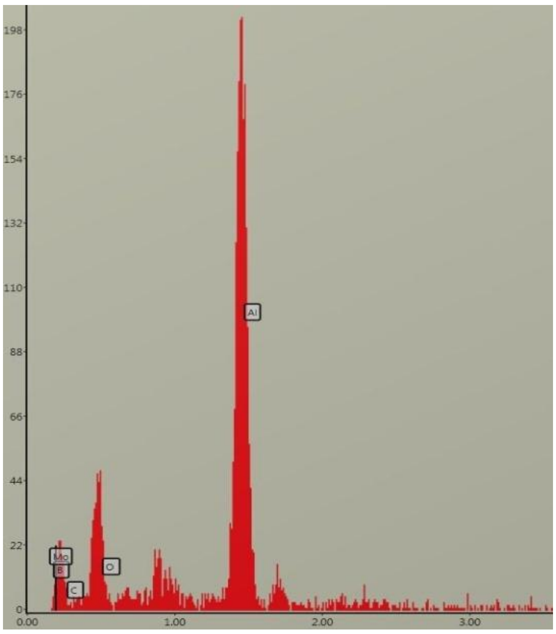

Figure 4.16 Compositional analysis the composite using EDS

4.5 Comparison of findings of this study to published research

In this part the properties of AMMCs in this work were compared to the previously fabricated AMMCs properties through different researchers.

Table 4.16 Comparison of properties of developed composites with previous research

Properties	Pervious Research	Current research
Hardness	212.5(Behera et al., 2021)	224.8
Corrosion rate	0.029(N. Kumar et al., 2023)	0.0023
Compressive strength	269.54 (Raghu et al., 2017)	390.23
Wear rate	0.09(Abd & Saber, 2015)	0.0017
Coefficient of friction	0.418(Behera et al., 2021)	0.37

This developed optimum sample composite also align with aluminum alloys like A6061 and A7075 having 224.8 HV which is greater than hardness of these alloys and also it has 2.76 g/cm^3 which is greater than that of A6061 BOYI technology.(2024).

CHAPTER FIVE

RECOMMENDATION AND CONCLUSIONS

5.1 Conclusion

In this study work, through the incorporation of Al_2O_3, B_4C and MoS_2 reinforcement particles the aluminum metal matrix was successfully developed using powder metallurgy method. The optimization of process variables and reinforcement weight percentage were implemented through Taguchi-based grey relational analysis depending on result of the experiment from sample developed by varying wt% of Al_2O_3(2,4 and 6), wt% of B_4C(2,4 and 6), compaction pressure (50, 55 and 60MPa), sintering temperature (580,600 and 620°C) and taking constant wt% of MoS_2(4wt%). The influence of process variables and reinforcements on tribological and mechanical properties were examined and given as follows:

- A better reinforcement distribution was found when the optimum sample was characterized using OM. The distribution of reinforcement and the existence of micro voids were confirmed from SEM examinations. XRD examination showed that the optimum composite contained a number of phases.
- As shown from Vickers hardness tester machine the hardness of the fabricated composite was enhanced as the porosity was reduced. The highest hardness 224.8 HV was obtained on the sample developed at the optimum level. Also the compressive strength obtained was 390.23 MPa.
- The density increased from sample one (S1) to sample three (S3) as the wt%of aluminum oxide (Al_2O_3), sintering temperature, and compaction pressure were increased. Nevertheless, the density varies unevenly along with the sintering temperature and compaction pressure.
- Furthermore, the sintering temperature, and compaction pressure had the biggest effects on the porosity percentage. As wt% of reinforcement increased and the sintering temperature and compaction pressure were decreased, the percentage of porosity increased as well. The lowest porosity (3.45%) and the highest density (2.76 g/cm^3) were obtained at optimum sample. Hardness and compressive strength are mechanical properties that are typically enhanced by optimal density and minimal porosity making suitable composites for engineering applications.

- The optimum sample composite exhibited a 32% improvement in wear resistance, 30.2% decreased in the friction coefficient and 45% reduction in corrosion rate in comparison to pure aluminum.
- The optimization method was implemented by utilizing Taguchi-based grey relational analysis which converts multi-response to single response by combining both grey relational analysis and Taguchi. The parameters of the optimum sample used were 6 wt% of B_4C, 6wt% of Al_2O_3, 620°C of sintering temperature and 60MPa of compaction pressure.

5.2 Recommendations

The following suggestions were made depending on the results and conclusions obtained in this current work.

- The aluminum properties were improved by the incorporation of Al_2O_3, B_4C and MoS_2. Examine aluminum composites with different reinforcements to obtain better properties.
- Optimization of weight % of reinforcement particles and process variables must be performed. Due to the fact that the composites fabricated at optimum levels showed better properties.
- This study demonstrated the homogeneous distribution of reinforcements, which improves the composites' mechanical and tribological properties. Then further investigation through advanced fabrication method is needed, to enhance consistence of reinforcement distribution.
- The improved properties of developed aluminum composites were suitable for engineering applications such as aerospace, automotive sectors which require high corrosion and wear resistance, high hardness.

5.3 Future Work

Even though this study had improved composite properties, some areas still need more research.

- Sintering is an essential process in powder metallurgy that improves the integrity and mechanical properties of composite materials. Compressed powders are subjected to a heat treatment procedure that improves strength, density, and durability by promoting particle bonding. It is expected from future research to sinter compacted samples under controlled conditions (inert or vacuum environment) and compacting the powder at higher pressure using hot isostatic pressing.
- In order to adequately generalize the impacts of the process variables of PM and percentage of reinforcing particles, a large sample size of composites would need to be investigated. Results from a large sample size could be straightforward and simple to examine.
- Since the other process parameters of the PM (mixing speed, mixing time, compacting time and sintering time) have impact on the properties of the composites samples, examining them by varying their values the improved properties will be found.
- To properly assess the mechanical capabilities of aluminum composites, more study is required to examine its properties under several loading situations such as simulation based testing for fatigue or impact loading. Evaluation of practical applications demands testing thermal behavior.

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APPENDIX

Appendix A

A.1 Equipment and tools utilized in this study

- Ball mill /mixer machine
- Weighing machine
- Hydraulic pressing machine
- Furnace
- Polishing and grinding
- Optical microscope (OM)
- Scanning electron microscope (SEM)
- X-ray diffractometer (XRD)
- Vickers hardness machine
- Universal Testing machine
- Pin-on-disc machine
- Potentiostat

A.2 The required mass of matrix and reinforcements within each sample in grams

	Reinforcements			Matrix
Samples	B_4C (gram)	Al_2O_3 (gram)	MoS_2 (gram)	Al (gram)
S1	0.24	0.24	0.48	11.04
S2	0.24	0.48	0.48	10.8
S3	0.24	0.72	0.48	10.56
S4	0.48	0.24	0.48	10.8
S5	0.48	0.48	0.48	10.56
S6	0.48	0.72	0.48	10.32
S7	0.72	0.24	0.48	10.56
S8	0.72	0.48	0.48	10.32
S9	0.72	0.72	0.48	10.08

A.3 Weight measurements of samples in fluid and air

Samples	W_{air}			W_{fluid}		
	T1	T2	T3	T1	T2	T3
S1	3.9312	3.9314	3.9264	2.3953	2.3915	2.3875
S2	3.9168	3.9071	3.9170	2.4223	2.4129	2.3835
S3	3.9279	3.9481	3.9469	2.4797	2.4808	2.4781
S4	3.9380	3.9360	3.9371	2.4501	2.4482	2.4425
S5	3.8797	3.8801	3.8792	2.3775	2.3690	2.3497
S6	3.9522	3.9530	3.9526	2.3962	2.3892	2.3648
S7	3.9111	3.9124	3.9150	2.4186	2.3997	2.3924
S8	3.8841	3.8852	3.8834	2.3423	2.3483	2.3457
S9	3.9082	3.9080	3.9068	2.3515	2.3431	2.3281

Based on the above result the sample's densities were determined as follows:

$$\rho\text{sample} = \frac{m\text{air}}{m\text{air} - m\text{water}} * \rho\text{water}$$

$$\rho\text{sample} = \frac{3.9312}{3.9312-2.3915} *0.9982=2.555 \text{ g/cm}^3$$

The other value of density also determined similarly.

The porosity percentage can be determined using:

$$\Phi = \frac{\rho t - \rho A}{\rho t} * 100$$

$$\rho_t = (W_m*\rho_m)+(W_r*\rho_r)$$

$$\text{For trial 1: } \rho_t = ((2.52*2)+(3.99*2)+(5*4)+(92*2.7))/100 = 2.8142 \text{g/cm}^3$$

$$\Phi = \frac{2.8142-2.555}{2.8142} * 100 = 9.212$$

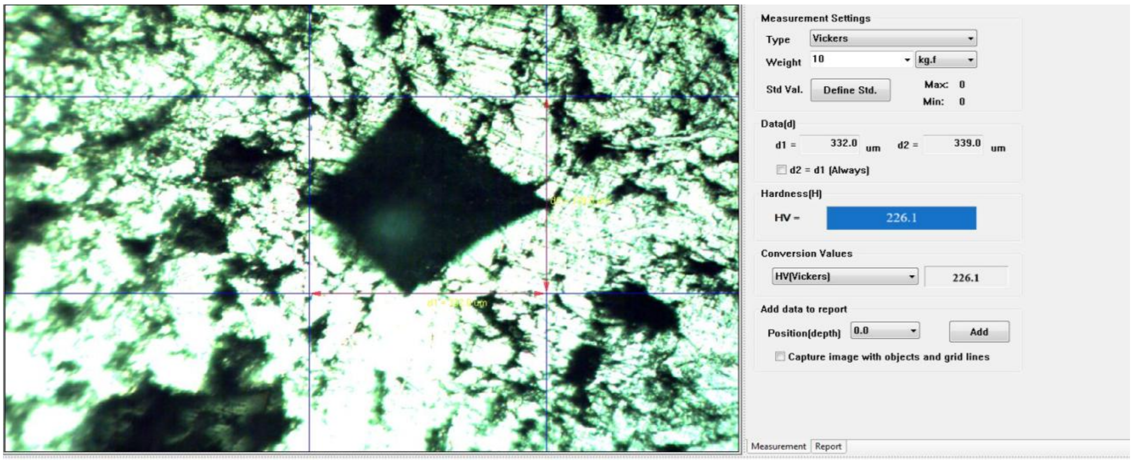
Similarly, the other values of porosity and theoretical density can be calculated.

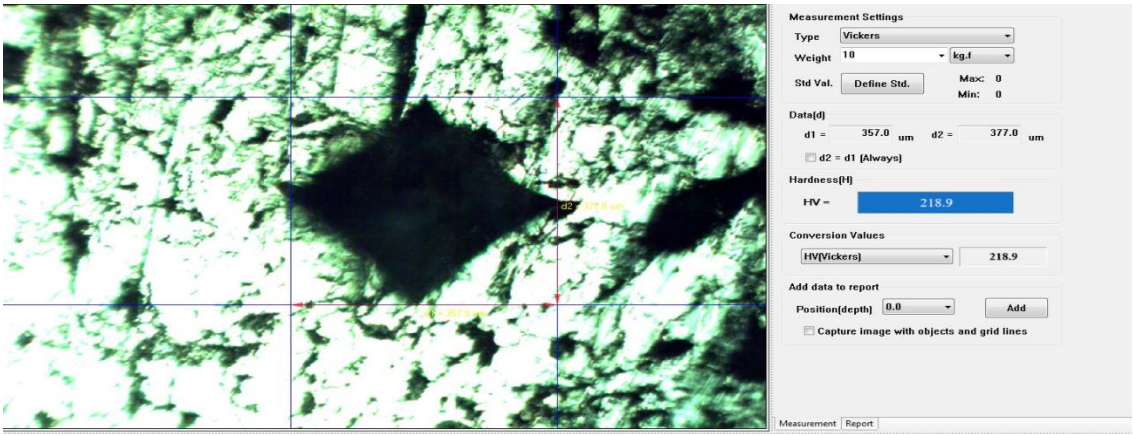
A.4 Wear test result from the experiment

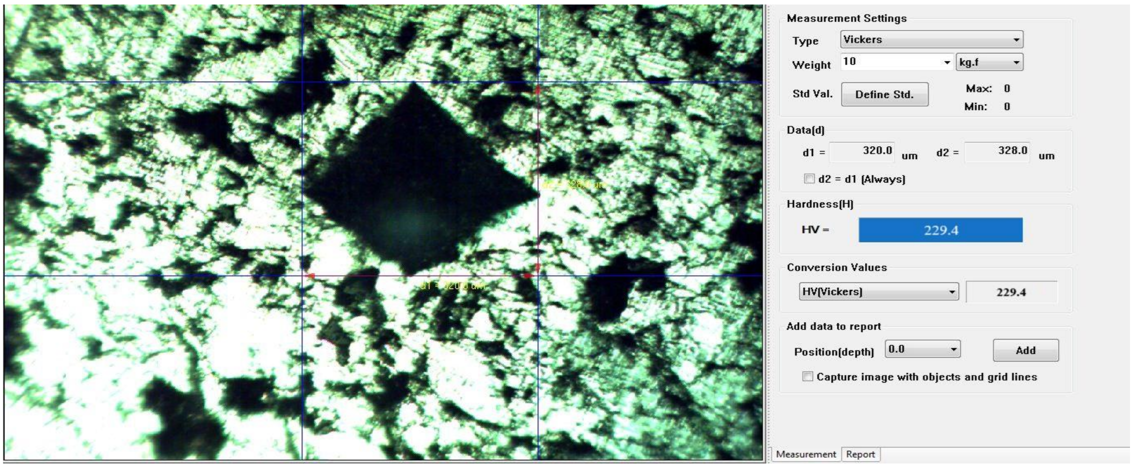
Samples	Weight before test (g)	Weight after test(g)	Sliding distance (m)	Friction force (N)
Pure	0.6708	0.6691	235.62	16
AMMCs	0.6597	0.6586	235.62	11.1

Appendix B

B.1 Experimental result of Vickers hardness for optimum







Appendix C

C.1 Composite fabrication activities

a) Compacting the powder

b) Grinding the sample

c) Polishing the sample

C.2 Developed composite samples

C.3 Testing activities of developed composite

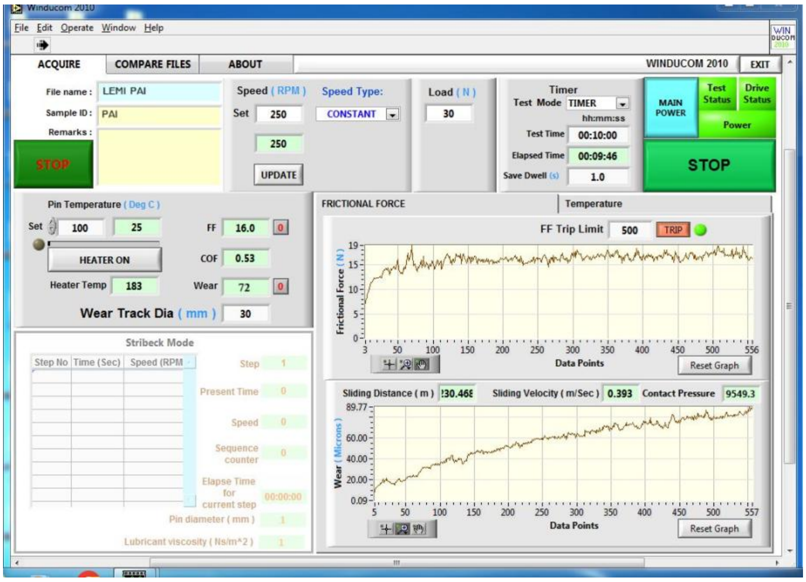
a) Compression testing (sample dimension was prepared as per ASTM E9, d=12.7mm,h=18mm)

a) Wear testing

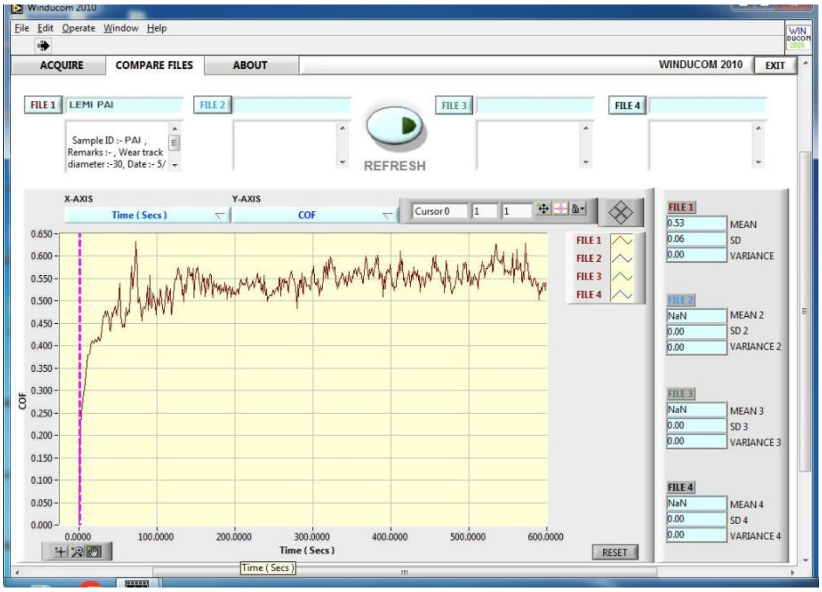
c) Hardness testing

Appendix D

D.1 Pure aluminum graphs of friction force, wear rate and coefficient of friction generated while testing wear

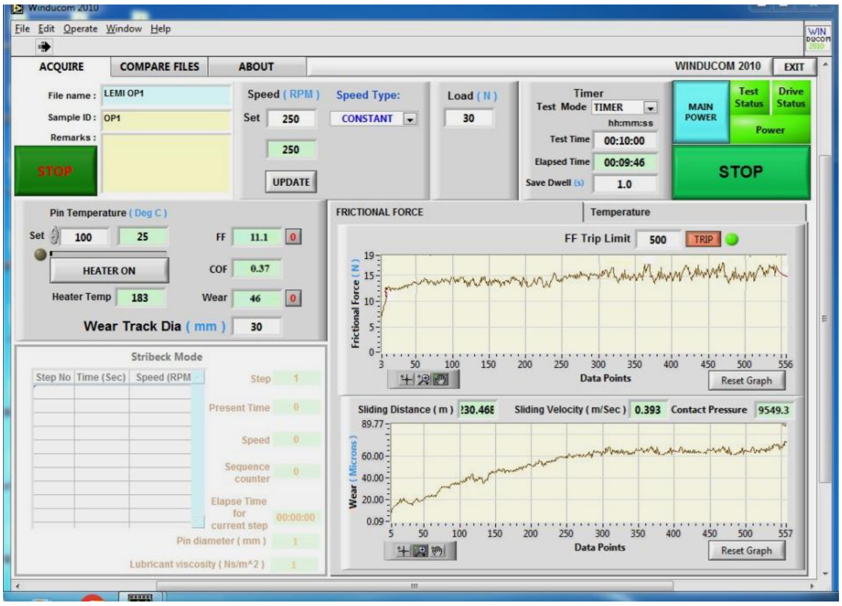


a) Graph of friction force and wear rate for pure Al

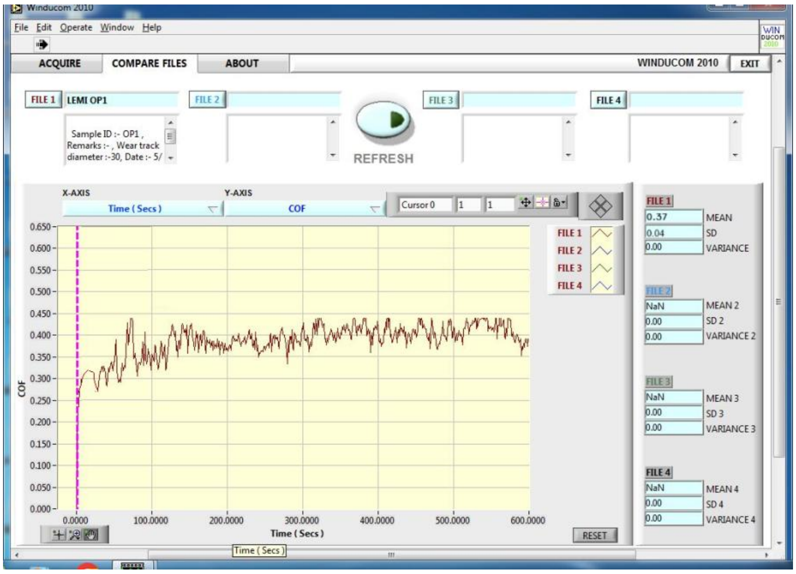


a) Graph of coefficient of friction for pure Al

D.2 Wear rate, friction force and coefficient of friction for fabricated Al composites



a) Graph of friction force and wear rate for AMMCs



b) Graph of coefficient of friction for AMMCs