Investigation on Al alloy MMC with Fly Ash Particulate

Thesis submitted

by

Asim Kumar Dhar

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Jadavpur University

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2. Name, Designation & Institution of the Supervisors:

Prof (Dr.) Goutam Sutradhar

Director, NIT, Jamshedpur – 831014, India

[Lien at Department of Mechanical Engineering,

Jadavpur University, Kolkata – 700032, India]

3. List of Publication (Referred Journals):

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STATEMENT OFORIGINALITY

I, Asim Kumar Dhar registered on 11th Jun 2015, do hereby declare that

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Signature of Candidate:

Date: 29/11/2022

Certified by supervisors:

Prof (Dr.) Goutam Sutradhar

29.11.22

iv

CERTIFICATE FROM THE SUPERVISOR

This is to certify that the thesis entitled "Investigation on Al alloy MMC with Fly Ash Particulate" submitted by Shri Asim Kumar Dhar, who got his name registered on 11.06.2015 for the award of Ph.D. (Engineering) degree of Jadavpur University is absolutely based upon his work under the supervision of Prof (Dr.) Goutam Sutradhar, Director, NIT, Jamshedpur (Lien at Dept. of Mechanical Engineering, Jadavpur University, Kolkata) and that neither his thesis nor any part of the thesis has been submitted for any degree/diploma or any other academic award anywhere before.

[Prof (Dr.) Goutam Sutradhar] 29.11.22

About The Author

Author, Asim Kumar Dhar currently working as an Assistant Professor in Department of Mechanical Engineering at Institute of Engineering & Management, India. He is graduated from Institution of Engineers (I) and Masters from Jawaharlal Nehru Technological University, Hyderabad. He has 15 years of academic & research experience. He has guided many UG students in the area of manufacturing technology. Prior to joining in academics, he served in Indian Air Force in different professional capacity for 20 years. His research area of interest includes composite material characterization and optimization.

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List of Tables

Details	Pag	e no.
Table 3.1:	Chemical composition of LM6 (Al-Si) matrix alloy	38
Table 3.2:	Physical properties of Class F Fly Ash	38
Table 3.3:	Chemical composition of the Fly ash	39
Table 3.4:	Test parameters of dry sliding wear samples	46
Table 4.1:	Tensile properties of composites with various fly ash Weight percentages	55
Table 4.2:	Micro-hardness of MMCs	56
Table 4.3:	Density of MMC with different wt % of fly ash	58
Table 4.4	Design factors and their levels	72
Table 4.5	Ratios of Signal to Noise	73
Table 4.6:	Using Adjusted SS for Tests, Analysis of Variance for SNRA1	75
Table 4.7:	Signal to Noise Ratio Response Table	78
Table 4.8:	Using adjusted SS for tests, an analysis of variance for SNRA1	80

List of Figures

Details		Page no.
Figure 2.1:	Research Methodology	36
Figure 3.1:	Bottom Poured Stir Casting Resistance Furnace	40
Figure 3.2:	Tensile Specimen	40
Figure 3.3:	Instron tensile test machine (Instron-8801)	41
Figure 3.4:	Vickers micro-hardness testing apparatus	42
Figure 3.5:	Optical Microscope (Leica DM 2700M)	43
Figure 3.6:	SEM and EDS Machine (JSM 6360)	44
Figure 3.7:	XRD analysis Machine (ULTIMA III)	45
Figure 3.8:	SEM with EDS	46
Figure 3.9:	Wear Test Specimen (dimensions in mm)	47
Figure 3.10:	Multi Tribo-Tester (Ducom, TR 25)	48
Figure 3.11:	Block diagram of Multi Tribotester	49
Figure 4.1:	Tensile Strength	52
Figure 4.2:	Yield Strength	52
Figure 4.3:	Young's Modulus	52
Figure 4.4:	Tensile Strain	52
Figure 4.5:	Tensile Properties of Composite	53
Figure 4.6:	Micro-hardness	55
Figure 4.7:	Density variation	57
Figure 4.8:	Optical images (500X) of composites (a) Al-2% FA (b) Al-4% FA (c) Al-6% FA (d) Al – 8% FA	A 59

Figure 4.9:	SEM images of composites (a) Al-2% FA (b) Al-4% FA (c) Al-6% FA (d) Al – 8% FA	61
Figure 4.10:	XRD Plot	62
Figure 4.11: E	EDS Spectrum (a) 2% FA (b) 4% FA (c) 6% FA (d) 8% FA	64
Figure 4.12:	Wear at different loads	67
Figure 4.13:	Wear at different speeds	68
Figure 4.14:	COF at different loads	69
Figure 4.15:	COF at different speeds	71
Figure 4.16 :	A wear rate main effect plot	73
Figure 4.17 :	Wear rate interaction plot	74
Figure 4.18:	Residual plot for wear rate	75
Figure 4.19 :	Surface plots of parameters with SNRA1	76
Figure 4.20:	COF's main effect graph	78
Figure 4.21:	COF's Interaction plot	78
Figure 4.22 :	Residual plot for COF	80
Figure 4.23:	Surface plots of parameters with SNRA1	81
	List of Equations	
Equation 3.1:	Density relation	43
Equation 3.2	Taguchi's S/N relation	49
Equation 4.1:	Density relation	56
Equation 4.1	Taguchi's S/N relation	70

Table of Contents

		Page No
Title pa	ge	i
Univers	ity Submission Form	ii
Stateme	ent of Originality	iv
Certific	ate from the supervisors	v
About the	he author	vi
Acknow	vledgment	vii
Dedicat	ion	ix
List of tables		X
List of figures & equations		xi
Table of	f contents	xiii
Abstrac	t	xiv
Chapter1:	Introduction	1-15
1.1	Introduction	1
1.2	Composite Material	2
1.3	Type of Composites	3
1.3.1	Polymer Matrix Composites	3
1.3.2	Carbon Matrix Composites	4
1.3.3	Metal Matrix Composites	4
1.3.3.1	Particulate Reinforced MMCs	6
1.3.3.2	Fiber Reinforced MMCs	6
1.4	Matrix Material	7

1.5	Reinforcement	,	7
1.5.1	Fly Ash as Reinforcement in MMC	:	8
1.6	Manufacturing methods of AMMCs	9	9
1.6.1	Liquid State Processing	9	9
1.6.1.1	Stir casting	9	9
1.6.1.2	Infiltration		10
1.6.1.3	Spray deposition		10
1.6.1.3	In-situ processing		10
1.6.2	Solid state processing		11
1.6.2.1	Powder metallurgy (P/M) processing		11
1.6.2.2	Diffusion bonding		11
1.6.3	Vapour state processing		12
1.6.3.1	Physical vapour deposition		12
1.7	Application of AMMC		12
1.8	Specific Research Problems		13
1.9	Objectives and Scope of Research		14
1.8	Statistical Approach – Taguchi Method		15
1.9	Conclusion		15
Chapter 2:	Literature Survey		16-35
_	•		
2.1	Introduction		16
2.2	Why Aluminum alloys?		17
2.3	Reinforcement used in AMMCs		18
2.4	Manufacturing Methods	2	20
2.4.1	Stir casting		20

2.5	Mechanical Properties	22
2.6	Microstructural Properties	26
2.7	Tribological Behavior	28
2.8	Statistical analysis by Taguchi method	31
2.9	Scope of present work	33
2.10	Research Methodology	35
2.11	Conclusion	35
Chapter 3:	Experimental Methods	36-49
3.1	Introduction	36
3.2	Materials Used	36
3.2.1	Matrix Material	36
3.2.2	Reinforcement Material	37
3.3	Composite Fabrication	38
3.4	Mechanical Properties	39
3.4.1	Tensile Test	40
3.4.2	Vicker's Hardness Test	40
3.4.3	Density	41
3.5	Metallurgical Properties	42
3.5.1	Optical Microscope	42
3.5.2	Scanning Electron Microscope	43
3.6	X-ray Diffraction Analysis	43
3.7	Energy Dispersive X-ray Analysis	44
3.8	Tribological Properties	45
3.9	Taguchi Method for Statistical Analysis	48
3.10	Conclusion	49

Chapter 4:	Results & Discussion	50-82
4.1	Introduction	50
4.2	Mechanical Properties	50
4.2.1	Tensile Test	50
4.2.2	Hardness Test	54
4.2.3	Density Measurement	55
4.3	Metallurgical Properties	57
4.3.1	Optical Microscope	57
4.3.2	Scanning Electron Microscope	60
4.4	XRD	62
4.5	EDS	63
4.6	Tribological Studies	65
4.6.1	Wear Measurement	65
4.6.2	Friction Measurement	68
4.7	Statistical analysis through Taguchi method	71
4.7.1	Wear Analysis	72
4.7.2	COF Analysis	77
4.8	Conclusion	82
Chapter 5:	Summary of Findings	83-86
5.1	Conclusion	83
5.2	Future scope of work	86
Bibliography		87-99

Abstract

Composite materials play a significant role in auto, aviation, marine and defense applications due to their unique properties. Aluminum (Al) alloys have certain advantages over other alloys. In this present research the composite material comprises al alloy LM6 and fly ash $(150 - 175 \mu m)$ has been picked as lattice and supporting materials individually. Magnesium (Mg) is added to lessen the surface pressure and evades the dismissal of the particles from the melts. Liquid state processing through Stir casting procedure was adopted for fabrication of metal matrix composite (MMC) into necessary shape and size according to the ASTM principles by energetically mixing at consistent speed and time. The fly ashes with various syntheses (2%, 4%, 6%, 8%) were added with LM6. The X-ray diffraction (XRD) and energy dispersive X ray analysis (EDX) were used to examine the structural analysis of MMC and optical microscopy and scanning electron micrograph (SEM) were used to investigate the microstructure on MMC. Wear test also carried out on MMC to ascertain the wear rate and coefficient of friction (COF) of different MMCs. There is substantial improvement of mechanical properties like tensile strength 112 MPa to 175.82 MPa, micro hardness 120.3 to 226.2 and density 2.43 to 2.39 kg/m³ of the composite. Optical micrographs and (SEM) revealed that fly ash particles were evenly dispersed throughout the aluminum matrix. The XRD results revealed that very minor changes in the composition of components are occurring. Because there is less segregation during solidification in the 2% and 4% of fly ash MMC samples. They have a more uniform distribution than the other examples. It is apparent that increasing particle dispersion homogeneity leads to improved mechanical behavior. As a result, there is little doubt that the use of this material in the automotive and space sectors will be viable in the future. Friction coefficient for the composites gets reduced with increase in fly ash content for both enhanced variable values. Sliding speed, proportion of fly ash and applied stress all influenced the wear volume of composites. Due to the presence of hard particles, an increase in fly ash content reduced wear volume. A statistical analysis through Taguchi method is carried out with design of experiment (DOE). While wear analysis, it demonstrates that the strongest factor influencing how quickly al-fly ash composites wear out. Whereas load has a moderate impact and speed has the least impact among the three input parameters i.e. weight percent of fly ash, load and sliding speed based on the S/N ratio. When

assessing the wear rate of composites with analysis of variance (ANOVA), the wt. percent has the greatest impact, followed by the two other variables, load and speed. However, no interaction significantly affects the rate at which composites wear out. However COF analysis reveals that load has the greatest influence in limiting the COF of al fly ash composites, whereas speed has a moderate impact and reinforcement has the least impact. While using ANOVA it is observed, that wt. percent has the biggest effect, followed by the other two factors of load and speed in determining the COF of composites. However, no interaction has a major impact on the COF of composites.

Chapter 1

Introduction

Outline of the Chapter: 1.1 Introduction, 1.2 Composite materials, 1.3 Type of composites, 1.3.1 Polymer matrix composites, 1.3.2 Ceramic matrix composites, 1.3.3 Metal matrix composites, 1.3.4 Particle reinforce metal matrix composites, 1.3.5 Fiber reinforced metal matrix composites, 1.4 Matrix material, 1.5 Reinforcement, 1.5.1 Fly ash as reinforcement in MMC, 1.6.1.1 Stir casting, 1.6.1.2 Infiltration, 1.6.1.3 Spray deposition, 1.6.1.4 In-situ processing, 1.6.2 Solid state processing, 1.6.2.1 Powder metallurgy (P/M) processing, 1.6.2.2 Diffusion bonding, 1.6.3 Vapour state processing, 1.6.3.1 Physical vapour deposition, 1.7 Application of AMMC, 1.8 Specific research problem, 1.9 Objective and Scope of Research Work, 2.0 Statistical Approach – Design of experiment, 2.1 Conclusion

1.1 Introduction

With the development of various materials and the methods used in their production, needs for clothing, food, transportation, communication and recreation have been met since the dawn of time. Stone, wood, clay, skins and other natural resources were available to our ancestors, as well. With the passage of time, man developed numerous methods for creating and creating new materials with demanding features. However, a large number of current technologies today require new materials with certain groups of features, which are challenging to achieve with the use of standard materials. It has been noted that there has been a significant demand for engineering materials since the 1950s. Scientists from several fields began to create a variety of super alloys and materials. Engineering materials used in aircraft, automotive and space industries are covered by this case. Therefore, engineers sincerely try to take advantage of chances. The environment is significantly impacted in some way as a result of these developments. Therefore, the creation of environmentally friendly materials continues to be a significant development in the history of material development in the composites sector. The peculiar combination of qualities needed by modern technology cannot be provided by conventional materials. Materials used in the aircraft, automobile, and space industries are a prime example of this. The engineers have access to metal, ceramic, and plastic materials. Another class of materials that is being used more and more frequently is composites, which consists of two or more metals, ceramics, and polymers together. Engineers may be able to create materials with custom qualities using composite materials, which is likely to be unachievable with traditional materials. Composite materials have become a very relevant material in industrial sectors, particularly the automotive, aerospace, and car industries, as society moves toward industry 4.0 and in response to specific needs. Due to its exceptional qualities, such as increased hardness, light weight, low density, high strength to wear ratio, high temperature, high shock resistance (thermal), high corrosive resistance etc., composite materials are in high demand. However, the reality that our civilization has evolved to be highly energy sensitive is more practical. Because of this, there is a rising need for lightweight, stiff and strong structure in many spheres of life. Additionally, composite materials are increasingly offering the solutions.

1.2.1 Composite Materials

In a strict sense, the concept of composite materials is neither novel nor recent. The concept of composite materials is employed frequently throughout nature. There are many historical examples of composites available in the literature. For instance, the coconut palm leaf is essentially a cantilever that makes use of the idea of fibre reinforcement. Cellulose fibres in a lignin matrix make up the fibrous composite known as wood. The lignin matrix links the fibres and provides the stiffness, whereas the cellulose fibres have a high tensile strength but are relatively flexible (i.e. low stiffness). Another example of a natural composite is bone as stated by Chandla [2013]. When a combination of two or more components that are not soluble in one another is seen at the macroscopic level, the result is a structural substance known as a composite. The matrix refers to both the component in which a constituent is embedded and the reinforcing phase, which is one of them. The material used in the reinforcing phase can take the shape of fibres, particles, or flakes. Materials seem to be continuous, used in the matrix phase. Kaw [2006] reported two different composite system named epoxy reinforced with graphite fibres and Concrete reinforced with steel. It is known as a combination when two or more different materials are used to provide unique qualities that cannot be produced by the individual components. A heterogeneous solid made up of two or more different materials that are mechanically or metallurgical bound together is referred to as a composite material. In this perspective, composite materials are merely a

major step in the never-ending quest for material optimization. The needs of today's cutting-edge technology cannot always be addressed by monolithic metals and their alloys. The performance requirements can only be met by combining multiple materials. Composite materials allow for design flexibility since they can be produced to meet the requirements of the best possible designs done by Miracle and Donaldson [2001]. Composite materials are among the most advanced, superior and flexible materials now in use. They are pushing the boundaries of traditional materials and enabling the realisation of cutting-edge designs in the fields of aerospace, automotive, and construction engineering. When these materials were developed for use in engineering applications in the 1970s, they were called "Materials of the Future" by Foltz and Blackman [1997].

1.3 Types of Composites

There are two categories for composites. The first one is based on the composition available in the matrix or base alloy, such as polymer matrix composites (PMC), ceramic matrix composites (CMC) and MMC. The second one is the types of reinforcement, such as particle, whisker, continuous fibre, and laminated weave composites.

1.3.1 Polymer Matrix Composites

Polymers have a significantly more complex structural makeup in comparison to metals or ceramics. They are inexpensive and simple to process. Polymers, on the other hand, have lower strength, modulus, and use temperature limitations. The characteristics of polymers can deteriorate as a result of prolonged exposure to UV radiation and some solvents. Polymers are typically poor conductors of heat and electricity due to their predominance of covalent bonding. However, polymers typically outperform metals in terms of chemical resistance. Reinforcements (fibres, whiskers, or particles) are embedded in a polymer resin matrix in PMCs (e.g. Polyesters, vinyl esters, PEEK, PPS). Due to their qualities at room temperature, ease of production, and low cost, these materials are employed in a vast array of applications and in significant quantities. At the moment, pressure vessels, rocket motor cases, sporting and leisure equipment, and aircraft structural components all make substantial use of carbon fibre in CFRP composites. In an effort to reduce vehicle weight and increase fuel efficiency, the transportation sector is using more PMCs.

1.3.2 Ceramic Matrix Composites

The term "ceramic matrix composites" (CMCs) made of ceramic fibres, particles, or whiskers is enmeshed in a matrix of another ceramic, such calcium aluminium silicate or alumina, and reinforced with fibres made of silicon carbide or carbon. High strength, hardness, high service temperature limits for ceramics, chemical inertness, and low density are all benefits of CMCs. Ceramics, however, have low fracture toughness when used alone. They catastrophically fail when subjected to tensile or impact loading. Ceramics' fracture toughness can be increased by adding fibres like silicon carbide or carbon to them as reinforcement. CMCs can be made using either traditional powder processing methods used to create polycrystalline ceramics or innovative methods created just for CMCs.

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1.3.3 Metal Matrix Composites

MMCs are a new generation of materials that were created in response to the demand for materials with high strength, high toughness, and the ability to function well in challenging environments (MMC). MMCs are the materials of the future and this fact has been well recorded. MMCs can achieve desirable mechanical and physical characteristics like thermal stability and high specific modulus strength, have made them this way. MMCs have the greatest strength in shear and compression as well as greater service temperature capabilities because they blend metallic features (high strength and high modulus) [Bienia et al. 2003]. The development of numerous processing routes that produce reproducible microstructures and properties, as well as the availability of relatively inexpensive reinforcements, have increased interest in MMCs for structural uses in the automotive and aerospace industries, and other industries over the past few years. In aerospace applications, it is possible to reduce structural weight by both decreasing alloy density and increasing alloy modulus. Metals that have been reinforced with fibres or particles that are typically stiff, robust, and light in weight are known as MMCs. Metal or ceramic particles or fibres are possible (e.g. SiC, Al₂O₃). Increasing the stiffness/density and strength/density ratios of metals with extremely stiff and strong components is one of the main goals of reinforcement. MMCs are utilised in a variety of industries, like aircraft, automotive, medicine, and sports equipment where it is advantageous to use lightweight structural materials. MMCs are appealing due to a variety of factors. By choosing the right kind and quantity of reinforcing constituent to be included in the MMC, the coefficient of thermal expansion (CTE) of particular MMCs can be customised. Additionally, the components of the enforcements can increase wear resistance, decrease friction and improve thermal conductivity. Compared to organic matrix composites (OMCs), MMCs can be employed at greater temperatures. Metal matrices are necessary for applications at higher temperatures because many organic matrices breakdown at roughly 300°C as reported by Babu et al.[2014]. Additionally, MMCs do not evacuate moisture as effectively as other OMC, which is important for space applications. The development of continuous fibre reinforced high performance hybrid materials using titanium and aluminium matrices and their behaviour were the subject of early studies on MMCs. Sadly, although the results are encouraging, the hefty manufacturing costs linked to the high costs of the reinforcement fibres and manufacturing that requires a lot of work techniques have limited the extensive industrial applications of these composites. Because of this, these materials have only been used in a small number of much specialised applications, mostly related to the military as mentioned by Motgi et al. [2013]. MMCs main purpose is to provide advantages over monolithic metals like steel and aluminium. When low-density metals like titanium and aluminium are utilised as reinforcement, these advantages include increased specific strength and modulus, low value of coefficients of thermal expansion when fibres with low coefficients of thermal expansion, like graphite, are used as reinforcement, and the preservation of properties like strength at high temperatures. MMCs are superior than polymer matrix composites (PMC) in a number of ways. These include improved wear, fatigue and defect resistances, better elastic characteristics, higher service temperatures, insensitivity to moisture, higher electric and thermal conductivities and higher insensitivity to moisture. Higher processing temperatures and higher density are disadvantages of MMCs versus PMCs. MMCs are a diverse class of materials that have at least two constituent parts: a ceramic phase that can take the form of particles, platelets, whiskers as mentioned by Schwartz [2002], short fibres or continuously aligned fibres and a metallic alloy matrix that can be made of al, mg, cu and Ti. MMCs are created via a process that disperses reinforcement across a metal matrix. MMCs have excellent impact and erosive resistance, good impact and electrical conductivity. Adequate stiffness under structural fatigue loads and appealing fracture features. Additionally, MMCs exhibit exceptional wear resistance, stiffness compared to the matrix alloy, strength and a low coefficient of thermal expansion. By carefully selecting the ingredients, additional features like high electrical and thermal conductivities and radiation resistance might be added to a variety of MMCs [Miracle 2005].

Based on nature of reinforcement composites are classified Particulate Reinforced metal matrix composite (PRMMC) and fiber Reinforced Metal matrix composite (FRMMC)

1.3.4 Particulate Reinforced MMCs

Compared to continuously reinforced composites and PRMMCs are substantially less expensive to manufacture. Two further divisions are made. Dispersion strengthened composites and large particle composites. The particle size in a large particle composite is greater than that of the other particles. The movement of the matrix can be controlled if the binding is strong. As a result, performance improvement of the matrix is more affordable than for composites with aligned reinforcements. Ceramic reinforcements that are equiaxed and have a ratio (length/diameter) smaller than roughly five are typically found in these composite materials. Additionally, although continuously aligned composites have extremely anisotropic properties, discontinuously reinforced composites' properties are virtually isotropic. Therefore, less expensive, discontinuously reinforced composites can perform better than continuous fibre reinforced composites in applications demanding isotropic characteristics. Aluminium matrices frequently use the reinforcement materials silicon carbide, alumina, boron carbide and graphite. Small mineral particles found in ceramics and glasses, metal particles like aluminium and amorphous materials like polymers and carbon black are all employed as reinforcing particles.

1.3.5 Fiber Reinforced MMCs

The high strength and stiffness to weight ratio of the composite is due to the fibres. A further division into continuous and discontinuous fibres is possible. Discontinuous fibres have lengths that are less than this, while continuous fibres often have lengths that are 5.3 times of critical crack length. The direction of the discontinuous fibres might either be aligned or random. It should go without saying that continuous fibres will improve the composite's strength and capacity to transfer loads. Examples of certain fibres include carbon, sic, boron, and e-glass fibres.

1.4 Matrix Material

As matrix materials for MMCs, a variety of metals and their alloys may be employed. For an MMC, the choice of a matrix alloy is determined by a number of factors. Whether the composite will be reinforced constantly or intermittently is very crucial. Continuous fibre reinforcements have the potential to transfer the majority of the load to the reinforcing filaments, making the fibre strength the main factor in composite strength. In light of this, it is possible to choose the matrix alloy for a continuously reinforced MMC more for its toughness than for its strength. The key tasks of the matrix alloy are to allow effective load transmission to the fibres and to eliminate cracks in the event that fibre failure occurs. As a result, continuously reinforced MMCs may use matrix alloys with lower strength, greater ductility, and increased toughness. For MMCs with discontinuous reinforcement, the strength of the matrix can be governing. The desired composite strength will therefore be taken into account when choosing the matrix, and greater strength matrix alloys may be needed. Needleman et al., [1993] suggested various matrix materials based on their qualities. Among these materials, magnesium, titanium, and aluminium are frequently employed. One of the lightest structural material now in use, beryllium has a higher tensile modulus than steel. Beryllium is known to be fragile, making it unsuitable for use in most applications. Mg is a light metal, yet it reacts more readily with oxygen in nature. Due to its superior mechanical properties, strong corrosion resistance, high toughness and low density, aluminum is one of the best options for matrix stated by [Baradeswaran et al. 2014]. The matrix should be chosen carefully, taking into consideration its chemical affinity with reinforcements, as it is the main component of MMCs. In addition, compared to other light components, aluminum is not particularly expensive. As a result, improved mechanical qualities, stronger wear resistance at high temperatures, and advanced structural applications, are areas where aluminum-based MMCs have potential as mentioned by Nestler et al.[2015].

1.5 Reinforcement

The material employed as reinforcement increases the composite's stiffness and strength while lowering MMCs' density. The choice of reinforcement is crucial for superior characteristics, and it also depends on the kind, size, and processing method of the reinforcement particles, as well as their chemical compatibility with the metal matrix [Miracle, 2005]. The matrices' uniform distribution, weight and volume percentages, and shape, dimension, and attribute are used to categorize reinforcements. The use of fibre reinforcements improves the characteristics, although the resulting materials are not

isotropic despite this. But the isotropic characteristics and cost effectiveness of the particle reinforced MMCs are positives. A hybrid composite is created by adding two or more reinforcements to the matrix. Additionally, the composite materials can be finished using the existing fabrication techniques such as hot forging, hot rolling, hot extrusion, and machining. It has been demonstrated that improving the mechanical properties of aluminum and aluminum alloys using ceramic particles, are the most effective reinforcement materials [Mahamani, 2014]. These ceramics are typically made of nitrides, oxides and carbides. Aluminum Oxide (A1₂O₃), Boron Carbide (B₄C), Graphite (G), Titanium Diboride (TiB₂), Silicon carbide (SiC) and Thorium are the common ceramic components employed. Graphite is the material of choice for MMC reinforcement because it has excellent mechanical qualities and a high thermal conductivity [Shanmugha sundaram, et al. 2013]. However, poor wetting properties between the reinforcement and aluminum make it challenging to manufacture the material at high temperatures. SiC is a strong reinforcement material that can be utilized for high temperature structural materials, creep and corrosion resistance. Applications in the nuclear sector use B₄C reinforced composites because of their effective neutron absorption properties. There are three different types of reinforcement utilized in metal matrix composites: continuous, particle, and whisker fiber. As reinforcements, commonly utilized materials include graphite, rice husk, fly ash, silicon carbide, aluminum oxide, boron carbide, silicon carbide, titanium carbide and soda ash.

1.5.1 Fly Ash as Reinforcement in MMC

An evenly distributed and suitable reinforcement can be used to give a metal matrix composite the necessary qualities such strength, stiffness, resistance to high temperatures, and low mass density. Fly ash designated as ASTM C 618 is a by product produced by aluminium manufacturing businesses and power stations that is removed from exhaust gases using electrostatic or mechanical precipitators. Fly ash is mostly composed of the following chemical elements: quartz, mullite, magnetite, hematite, spinel, ferrite and alumina [Rohagti 1997]. While cenosphere fly ash particle mass density ranges from 0.4 to 0.6 g/cm³ and precipitated fly ash particle mass density is between 2.1 and 2.3 g/cm³ [Guo et.al. 1997]. Fly ash particles can be added to aluminium matrix in the needed quantity to increase critical characteristics like hardness, wear resistance, damping, stiffness and mass density as reported by many researcher [Rohatgi 1993, Rohatgi 1997, Keshavaram et al. 1984, Sobczak et al. 1998]. In the automotive, small engine and electromechanical industries, aluminium alloy fly

ash composites, commonly referred to as "ash alloys" are frequently utilised as engine blocks, manifolds, valve covers, brake rotors, shrouds, casings, pulleys and pans as reported by Rohatgi et al. [1998]. According to a recent analysis, industrial by products such as fly ash total hundreds of millions of tonnes worldwide. The remaining is either kept for future use or disposed of in controlled landfills, which creates environmental hazards and expensive disposal costs. As a result, it is crucial to create new technologies that will enable creative and environmentally sound uses of waste fly ash [Ahmaruzzaman, 2010]. The following benefits are obtained when fly ash particles are combined with molten aluminium [Rohatgi et al. 2006].

- Affordable Materials
- Savings on energy costs,
- Environmental advantages,
- Low ash disposal costs

1.6 Manufacturing methods of AMMCs

Three major categories can be used to classify industrial AMMC manufacturing techniques. (1) Processing in a liquid state (2) Processing in a solid state (3) Processing in a vapour state. We cover these three classes below in more detail.

1.6.1 Liquid State Processing

This is a very popular and commonly utilised technique for composites made of aluminium and metal. The next sections go through the key liquid state processing methods utilised for MMCs.

1.6.1.1 Stir casting

Incorporating fly ash particles into the molten alloy in the furnace through impeller mixing and allowing the mix to pour in mould and solidify are the steps described in this approach. The key to this process is to create proper vortex before mixing of reinforcement through a mechanical stirrer popularly known as impeller. Proper mixing depends on complete removal of moisture from the reinforcement, mixing time, holding time in the furnace, proper stirring speed etc. Different ratios of fly ash are warmed for around two hours at roughly 450°C. The preheating chamber is affixed to the furnace's side. Preheated fly ash is gradually combined

in the furnace once it reaches liquidus temperature of 750°C and stirring motion begins. The stirring process lasts for 15 minutes at a constant 600 rpm. To encourage wettability 2% of Mg is added to the fly ash as it is being mixed in the furnace. The molten mixture is finally poured directly into a cylindrical mould. Compo-casting is an additional stir casting method variation. In this instance, semi-solid ceramic particles are included into the alloy. The factors that affect the composite's quality are pouring temperature, stirring rate, pouring speed, mould temperature, mould coating, and mould life.

1.6.1.2 Infiltration

In the infiltration process, a preform—an arrangement of small fibers—is injected with liquid metal. The preform is frequently made to have a precise shape so that it can be a crucial component of the end product. Short fibres that have been suspended in liquid are often sedimented to create preforms. The method can also be modified to create particulate MMCs.

1.6.1.3 Spray deposition

Spray deposition processes can be classified into two types depending on whether the droplet stream is created from a molten bath or by continually supplying cold metal into a zone of rapid heat injection. The method was created to build up bulk metallic material by spraying anatomized droplets onto a surface. There has been substantial research towards the adaptation to particulate MMC production by injecting ceramic powder into the spray, but there hasn't been much commercial success. The average droplet velocity is usually between 20 and 40 m/s. On top of the ingot as it forms, there is frequently a thin layer of liquid or semi-solid. The MMC material made in this technique frequently has irregular ceramic particle distributions, and the as-sprayed state porosity is typically between 5 and 10%. Melt atomization methods and thermal spraying are different in a number of ways. Spray deposition aluminium metal matrix composites are reasonably priced, with fabrication costs falling somewhere between stir casting and powder metallurgy methods.

1.6.1.4 In-situ processing

In a number of cutting-edge methods, elements are brought together in such a way that a chemical reaction takes place while the mixture solidifies. In some procedures, liquid metal is added, and it gradually oxidises. For instance, the Dimox method utilises the directional

oxidation of aluminium in a number of other patented processes. Other procedures involve heating numerous elements in combination with the brief creation of a liquid phase to get them to react. The "Exothermic D" processes are a significant subset of these procedures. The reaction between mixed powders of Al, Ti and B to create Al + TiB₂ composites is an illustration of such a process. A near net shape can be formed by creating a powder compact that allows liquid metal to seep in as it reacts with the preform.

1.6.2 Solid state processing

The next sections go through the significant solid-state processing methods utilised for MMCs.

1.6.2.1 Powder metallurgy (P/M) processing

P/M manufacturing entails mixing of the following cold compaction, canning, degassing and a high temperature consolidation stage such as Hot Isostatic Pressing (HIP) or extrusion, aluminium alloy powder with ceramic short fibre or whip is used in either a dry or wet condition. According to the powder's history and the processing circumstances, oxide particles in powder metallurgy-processed AMMCs come in the shape of plate-like particles that are a few tens of nanometres thick and come in volume fractions ranging from 0.05 to 0.5. These tiny oxide particles frequently serve as a dispersion-strengthening agent and significantly affect the matrix's characteristics, especially when subjected to heat. P/M processing is a very flexible method that is frequently employed for AMMCs.

1.6.2.2 Diffusion bonding

Mono-filament reinforced AMMCs have mostly been produced via the diffusion bonding technique (foil-fibre-foil) or the evaporation of relatively substantial coatings of aluminium on the fiber's surface. Composites made of Al 6061 and boron fibre has been produced via foil-fibre-foil diffusion bonding. However, this technique is more typically used to produce Ti-based fibre reinforced composites. Making complicated forms and reinforcements with a high fibre volume fraction requires a challenging technique.

1.6.3 Vapour state processing

The process for the vapour state synthesis of MMCs is discussed below:

1.6.3.1 Physical vapour deposition

Evaporation is a reasonably quick approach that employs target species thermal vaporisation in a vacuum, whereas physical vapour deposition methods are relatively slow. Fabrication of monofilament reinforced by evaporation. In order to generate a thick surface coating, Ti entails passing the fibre through an area where metal to be deposited with a high vapour pressure. By shining a strong electron beam at the feedstock end of a solid bar, vapour is created. The differences in evaporation rates between the different solutes are eventually balanced out by changes in composition of the molten pool formed on the end of the bar when a steady state is reached and the alloy content of the deposit equals that of the feedstock. It's also important to note that there is little to no mechanical disturbance in the interfacial region, which may be important if the fibres contain a diffusion barrier layer or carefully formulated surface chemistry.

1.7 Application of AMMC

Due to its exceptional physical and mechanical qualities, AMMCs have a wide range of applications in fields including electronic engineering and aircraft technology. But recently, AMMCs have also been used to make medical devices. In the automotive industry, reinforced diesel engine pistons, reinforced aluminium engine block cylinder bores, reinforced intake and exhaust valves, drive shafts, brake parts, and reinforced power module components for hybrid and electric vehicles are just a few of the major applications served by AMMCs [Lloyd 1994] and [Kaczmar et al. 2000]. Canning tools, crimp rollers, check valves, hammers, impact dies, bending dies, extrusion dies, and hot forging die inserts are examples of tools that frequently use AMMCs stated by Rosso [2006]. Pistons, automobile racing brakes, filament-wound fuel tanks, drive shafts, engine blocks, clutch plates, push rods, valve guides, frames, suspension arms, body panels, doors, and body panels, among other components, are typically made from silica particle-reinforced aluminium metal matrix composites, which exhibit good strength and high wear resistance reported by Miracle [2005]. Due to their excellent strength to weight ratio, composite materials have been investigated for use in a variety of applications, including aircraft and rocket components. A few examples include pipes, sporting goods and military helmets, helicopter blades, automotive bodies, leaf springs, drive shafts, ladders, pressure vessels, boat hulls, and various other structures. Sports equipment including tennis, squash, and cricket bats, as well as bows and arrows, golf clubs, canoes, oars, skis, and yacht masts, also use composite materials. Composites with favourable stiffness characteristics, biocompatibility and weight reduction are widely used in a variety of medical applications [Strong, A.B, 1989]. Ventral fins, fuel access door covers, rotor blade sleeves and swash plates on various helicopter engines, nose landing gear, and nozzle actuator connections are some major applications in aerospace components [Kunze, 2001]. Payload bay doors, remote manipulator arms, high gain antennas, antenna ribs, and struts are more examples of applications. Fans, blowers, gearboxes, valves, and strainers are components of an engine are a few of the major marine applications [Lee et al., 2004]. For thermal management in electrical and electronic devices such as insulated gate bipolar transistor and rectifier packing, lighting poles, fibre optic tensile members, printed circuit boards, chip carriers, heat spreaders, laser diodes, and integrated circuit packages, AMMCs are employed [Miracle, 2005]. Additionally, AMMCs are employed in the chemical industry for things like composite tanks carrying fuel bottles for the fire service, climbing gear, underground storage tanks, ducts, and stacks, among other things, all use liquid natural gas [Kaczmar et al. 2000]. Tennis rackets, golf club shafts, fishing rods, and bicycle frames are all made with AMMCs for usage in sports [Blawert et al. 2004]. AMMCs could also be used into fishing poles.

1.8. Specific research problem

It is observed that by many researchers preferred ceramic material as reinforcement with aluminum alloy as matrix. Ceramic material as reinforcement provides superior properties for the composite. But it is expensive. There from, search for inexpensive reinforcement started. Fly ash became the best alternative for ceramic material. Fly ash is of two categories. Class C fly ash is primarily made from sub-bituminous and lignite coal with high calcium content. It's shape is of hollow cylindrical. It's research work also quite limited as it is costlier than class F. Class F fly ash is cheap and easily available with shape solid spherical. This category of fly ash (F) is abundantly available in thermal power station. As aluminum alloy with class F type fly ash make composite with many desirable properties in weight reduction in automobiles which lead to improved mileage and reduction in energy consumption. Therefore it has become very pertinent to investigate further on mechanical properties, metallurgical aspect and tribological behavior.

1.9. Objective and scope of Research work

From the previous section, the basic problem as designed is to do some investigation on composites made out of al alloy matrix (LM6) as base metal and fly ash as reinforcement. It is decided that the fabricated composite will provide mechanical properties with different ash content (2%, 4%, 6%, 8%). This composite will also help us to know the distribution of particles through metallurgical analysis. This analysis with validate further through XRD and EDS. This composite will also tell us the tribological phenomena, where COF and wear analysis can be made under some variable. At the last validation is done with Taguchi method using DOE and ANOVA.

2.0 Statistical Approach – Taguchi method

Design of experiments (DOE) is a methodical testing strategy in which desired changes are made to a system's or process' input variables, and the effects on response variables are then assessed. For experimental investigation to identify the physical parameters and numerical simulation models that are unknown, DOE might be helpful. An effective method for increasing the number of instructions derived from a study while minimizing the amount of data that must be assembled is experimental design. For the creation of empirical models, Taguchi developed a collection of statistical and mathematical techniques including design of experiments and ANOVA. Maximizing a response (output variable) that is influenced by a multitude of independent variables is the aim of diligent experiment design (input variables). The DOE and ANOVA approach examines the significance of these process factors in the coupled responses and establishes the relationship between various processing variables and outputs with varied needed circumstances [Myers and Montgomery, 1995]. It is a method for developing and improving an empirical model through successive experiments. The response surface methodology's goal is to create a mathematical connection between the responses and the most common machining parameters. Planning experiments is referred to as experimental design. Through this planned and controlled method of experimental design, the variables that are influencing a response variable are evaluated. The specific setting levels of the factor combinations at which each individual run of the experiment is to be done are specified in the experiment design. The components are varied simultaneously using this multiple variable testing technique. Because the variables can be changed independently

of one another, a causal predictive model can be developed. Data obtained by observation or other methods other than a design of experiments technique can only be used to establish correlations. The classic experimental approach of altering one component at a time has additional drawbacks, such as its inefficiency and inability to identify effects brought on by multiple factors acting together.

2.1 Conclusion

Chapter 1 gives brief description of composites, composite material, and type of composites, matrix material, reinforcement, different manufacturing methods, and application of AMMC and optimization method.

Chapter 2

Literature Survey

Outline of the Chapter: 2.1 Introduction, 2.2 Why Aluminium alloys. 2.3 Reinforcement used in AMMCs 2.4 Manufacturing Methods 2.4.1 Stir casting 2.5 Mechanical Properties 2.6 Strengthening Mechanism of Composites 2.7 Microstructural Properties 2.8 Tribological Behaviour 2.9 Critical appraisal of existing materials 2.10 Statistical analysis by Taguchi method 2.11 Research Methodology 2.12 Conclusion

2.1 Introduction

This chapter includes a report of the literatures on the research of the mechanical, metallurgical, and tribological behaviour of composites made of aluminium alloy. This section discusses the specifics of the matrix component, reinforced materials, and production techniques established by numerous researchers. In addition, a briefing on metal matrix composites' wet ability and some statistical modelling relating to wear is provided. The thesis's work is organised to accommodate for the gap in the research described in the final section of this chapter. Because they are lightweight and corrosion resistant, a protective layer of alumina oxide exists. Therefore, aluminium and aluminium alloys are typically utilised in numerous mechanical applications, particularly for automobiles, aircraft components, electronics, and the food industry [Callister and David, 2007]. The widespread application of aluminium and its alloys in the automotive and aerospace sectors is a compelling example of the desirable features that these materials may process. However, as technology developed, it became necessary to create materials with these qualities improved. We now have more options for obtaining these desired properties in materials thanks to the field of advanced materials. As a result, we have a variety of composite materials to pick from, each of which is specifically designed to meet a certain requirement. MMCs are the subject of this review, notably aluminium matrix composites (AMCs). One of the components in AMCs is an aluminium/aluminium alloy, known as the matrix phase, which forms a percolating network. The second part, usually non-metallic and frequently ceramic like SiC and Al₂O₃, acts as reinforcement and is integrated in this aluminium / aluminium

alloy matrix. By adjusting the type of elements and their volume fraction, it is possible to alter the properties of AMCs. Greater strength, improved stiffness, lower density, better high temperature qualities, wear resistance, increased abrasion and improved damping capabilities are the main benefits of AMMCs over unreinforced materials [Surappa, 2003]. AMCs are used extensively in the automotive, aerospace, electronics industries due to their exceptional physical and mechanical qualities [Cole, 1995].

2.2 Why Aluminium alloys?

Aluminium is a light metal under a variety of service circumstances that has excellent corrosion resistance. When aluminium surfaces are exposed to the atmosphere, a thin, solid layer of aluminium oxide that deforms quickly stops the metal from oxidising [Schweitzer 2003], [Joseph 1999]. .Metal compounds in which aluminium predominates are known as alloys. Iron, copper, silicon, zinc, magnesium, and silicon are common alloying constituents. A lot of machine parts and structures that need to be lightweight and corrosion-resistant employ aluminium alloys [Nafsin and Rashed 2013]. Precipitation based strengthening is the primary mechanism of strengthening in this type of combination, while hot working can also increase material properties, as seen in Al-Mg-Si and Al-Zn-Mg alloys [McQueen and Celliers, 2007]. Al alloys are chosen as the basic matrix for two reasons. Firstly, aluminium (Al) and aluminium alloys (Al alloys) are widely used in the field of energy efficient transportation because they are simple to work with and efficient in reducing energy consumption due to their light weight and significant strength. Pure aluminium is not robust enough to be used in significant structural applications. However, when strengthened by additional phases, it can demonstrate steel's strength and diverse qualities [Wang et al., 2014]. Al based particle reinforced composites were effectively used by the automobile sector in pistons, engine blocks, disc rotor brakes, drums and callipers, driving shafts, connecting rods [Prasad and Asthana, 2004]. The majority of MMCs produced for commercial applications are composites made from aluminium (Al) alloys [Rittner, 2000]. High structural efficiency and exceptional wear resistance are the most required features in applications including ground transportation, thermal management, the aerospace industry, the automobile industry, and recreational sectors. Al based MMCs satisfy these requirements in engineering materials [Miracle, 2005]. Second, despite an increase in operating temperature, high specific strength must be maintained. Al alloys in peak-aged condition are frequently used to make engine heads, pistons, and related parts for the automotive and transportation industries. Al-alloys have some benefits as a material, to be sure, from a material science perspective. However, before to the development of Al based MMCs, Al alloys were losing ground because, at high operational temperatures, alloy weakening resulted in overage Al. The synergistic interaction of hard ceramic particles with an Al matrix, according to Ceschini et al. [2017], helped to preserve strength and stiffness during high temperature operations. As a result, the utilisation of composites made of Al/Al alloys has advanced. To properly replace aluminium alloy, study of other properties, such as wear, friction, thermal conductivity, and formability of aluminium-based PRMMCs is necessary. Additionally, among all metal matrix composites, aluminium or its alloy composites are the most widely used and necessary for many engineering applications. For this reason, the next part will exclusively discuss aluminium matrix composites (AMC). In comparison to unreinforced materials, Aluminium or its alloy Matrix

Composites (AMCs) have the following key advantages, according to Surappa [2003]:

- Lower density (less weight)
- Greater stiffness
- Thermal/heat control
- Personalized electrical performance that has been improved.
- Enhanced resistance against abrasion and wear
- Increased rigidity
- Better high temperature performance
- Controlled coefficient of thermal expansion
- More effective damping abilities.

Aluminium matrix composites (AMC) are in demand across all industries because to their superior performance, favourable economics, and favourable environmental effects. AMCs are more appealing in the transportation sector as a result of the focus on increased fuel efficiency and strict environmental standards. AMCs are in demand in the transportation sector because they use less fuel and produce fewer airborne emissions and noise. In a variety of automotive and aerospace applications, MMCs compete with plastics, ceramics, smart alloys, and redesigned steel parts. By incorporating reinforcement into the aluminium alloy matrix, certain specialised qualities such as strength, wear, stiffness, creep, and fatigue over base materials, the mechanical and wear behaviour may also be improved.

2.3 Reinforcement used in AMMCs

Recent years have seen an increase in research into the use of fly ash as a filler and reinforcement in MMCs. Al₂O₃, SiC, Y₂O₃, TiC, B₄C, ZnO, ZrO₂, BN, AlN, TiB₂, graphite, grapheme nano-sheets, fly ash, etc. are just a few examples of the numerous inorganic and ceramic particulate reinforcements [Dalmis et al., 2014]; [Casati and Vedani, 2014]. A natural source, inexpensive, and harmful industrial waste particulate reinforcement is fly ash. Precipitator and cenosphere are the two groups of fly ash particles. The majority of fly ash particles in the cenosphere are spherical in shape and typically have smooth exterior surfaces [Rohatgi et al. 2009]. Precipitator fly ash (Solid) and cenosphere fly ash (Hollow) are used to make the composites (Hollow). The density of the aluminium alloy is dramatically reduced when cenosphere fly ash is added. With increasing quantities of fly ash particles, the aluminium alloys' hardness, elastic modulus, and compressive strength rise. Additionally, the addition of precipitator fly ash to the aluminium alloy greatly improves its resistance to abrasive wear. The presence of hard alumina silicate fly ash particles is what causes the improvement in wear resistance [Rohatgi et al. 1995]. Fly ash is typically divided into two types: precipitator fly ash, which refers to solid, spherical fly ash particles, and cenosphere fly ash, which refers to hollow, cylinder-shaped fly ash particles with densities between 0.4 and 0.7 g/cm³. One popular variety of fly ash is typically made up of glassy substances like silica glass, crystalline substances like silica, mullite, and hematite, with various oxides. Precipitator fly ash, which has a density of between 2.0 and 2.5 g/cm³, can reduce the density of some matrix materials while enhancing their wear resistance, stiffness and strength. Cenosphere fly ash, which is composed of hollow fly ash particles, has a substantially lower density than metal matrices, ranging from 1.6 to 11.0 g/cm³. This allows for the production of ultra-light composite materials..

Fly ash is an industrial waste/natural source because a substantial portion of it is currently land filled, it is easily accessible in very big quantities (80 million tonnes per year) at extremely low prices, which is another factor in the inclination to utilise fly ash as reinforcement in metal matrices as analysed by [Matsunaga et al. 2002]. There are several types of fly ash, and researchers have employed them in a variety of research projects. Class C and Class F Fly Ash (fine by-product of coal combustion) are among them. Burning western bituminous coal, sub bituminous coal, or lignite results in the production of Class C fly ash, but burning anthracite coal or eastern bituminous coal results in Class F fly ash [Chugh et al. 2000]. The fly ash obtained from other natural sources, such as neem leaf ash

[Prasanna et al. 2017], rice husk ash by [Narasaraju and Linga Raju, 2015], coconut shell ash [Arulraj and Palani, 2018], breadfruit seed hull ash [Atuanya et al. 2012], bamboo leaf ash [Alaneme et al. 2013] and bagasse ash, is also known as a [Usman et al. 2014]. In order to create Class F fly ash, which has a low CaO level and is better suitable for the synthesis of MMCs, anthracite or bituminous coal are frequently burned [Gikunoo et al. 2005].

2.4 Manufacturing Methods

Harris [1983] divided fabrication techniques into two main groups of manufacturing methods: solid-phase and liquid-phase. There are several methods for solid-phase fabrication, including powder metallurgy, diffusion bonding, extrusion hot rolling, pneumatic impaction, and others. Examples of liquid-phase fabrication techniques include pressure casting, squeeze casting, stir casting, compo casting, spray co- deposition and others. The multistep diffusion bonding procedures may be the most expensive, whereas liquid metallurgy approaches were the least expensive.

2.4.1 Stir-Casting

This procedure entails mixing liquid aluminium melt with ceramic particles and letting the combination set up. The most important thing was to ensure the particle reinforcement was thoroughly moistened and the melting of liquid aluminium alloy takes place. The most fundamental and popular method is stir-casting. In this technique, the matrix material was melted to liquidus temperature. At this stage impeller starts rotating at 600 rpm and vortex motion are created at the melt's surface. The vortex approach involves introducing pre-treated fly ash particles into the revolving impeller's molten alloy for thorough mixing of reinforce with matrix metal [Ye and Liu, 2004]. In addition to bringing particles into the liquid metal, stirring also helps to maintain the particles' controlled state. After that the molten mix gradually poured into prepared die and given time to set. Stirring through impeller action inside the chamber of the furnace is a key aspect of the process. The resulting alloy can be used for permanent mould casting, die casting, or sand casting when it is in molten form and contains other inorganic particles. A homogeneous distribution of secondary particles in the composite matrix was necessary for producing a high reinforcing effect a result of an unequal distribution could lead to early failures in both regions with and without reinforcement as mentioned by [Lim et al., 1999]. Places without reinforcement are typically weaker than other

areas. In certain regions, slip-of dislocations and the start of micro cracks can happen very easily under an applied load, eventually leading to the material failing. Particularly during the melt and later after solidification, particle agglomeration and sedimentation can be brought on by microstructures in homogeneities. The primary issue with the technique of stir-casting is the separation of reinforcement particles brought on during the melting and casting operations, by their settling and surfacing process. [Acilar and Gul, 2004]. These cast composites' inhomogeneous reinforcement distribution may potentially be a concern as a result of interactions between moving solid-liquid interfaces and suspended ceramic particles during solidification [Moustafa, 1995]. The wetting condition in which the particles are with the melt, the mixing strength, the relative density, and the solidification rate are among the factors that affect both the characteristics of the material and the process parameters affect how the signal distribution of the particles in the solid is determined. How the particles are distributed inside the molten matrix depends on the mechanical stirrer's design, the conditions under which it is being used, where it is located in the melt, the temperature at which it is melting, and the characteristics of the additional particles. Generally speaking, a number of different molten aluminium alloys can contain up to 30% reinforcement particles with a size range of 5 to 150 µm. Before fully solidifying, the melt-reinforcement particle slurry can be transported specifically to a shaped mould, or it can solidify into a pre conditioned cylindrical die. Further it can be processed further utilising techniques like investment casting and die casting. Ceramic whiskers or particles smaller than a micron could not be used in the procedure [Ghazali et. al., 2005]. A two-step mixing procedure was an intriguing advancement in stir-casting [Das et al., 2008]. This procedure involved heating the matrix material above the liquidus temperature to completely melt the metal. The melt was then maintained in a semi-solid condition by cooling it to a temperature below the liquidus points. The warmed particles were now added and blended. Once more the molten mix heated above liquidus temperature, the slurry was properly blended. It had been determined that the final microstructure was more consistent than that produced by traditional stirring [Sharma et al., 2007]. The ability of this two-step process to penetrate the gas barrier around the particle surface was largely credited with its usefulness. Because the viscosity of molten mix generates a greater abrasive impact on the surface of the particle than does standard stirring, mixing the particles while they are semi-solid allows for a more effective breakdown of the gas layer. Thus, by fracturing the gas layer, the subsequent mixing in a totally liquid condition is more effective. With the inclusion of a suitable stirring mechanism, such as ultrasonic, centrifugal force, mechanical and electromagnetic stirring. Stir casting is to enable

the use of standard metal processing techniques [Rajan et al., 2007]. The main benefit of stir casting was its viability for mass manufacturing. Stir-casting was the most cost-effective metal matrix composite fabrication method among all those that had been thoroughly tested in comparison with other processes, stir-casting is merely one-third to one-tenth expensive for mass manufacturing. As a result, stir mixing and casting are currently utilised to produce metal matrix particle composites in huge quantities. As the matrix, many metals including Al, Cu, Ni, and Mg had been employed. As reinforcements, numerous materials including SiC, Al₂O₃, SiO₂, Si₃N₄, graphite and ZrSiO₄ had been used. Stir-mixing and casting metal matrix composites together involve unique safety measures, such as design of pouring, temperature control and the gating systems [Rohatgi et al., 2010].

2.5 Mechanical Properties

In industrial applications, the mechanical, corrosion, tri-biological, and thermal properties of developed hybrid composites are crucial. The thermo-mechanical characteristics of a material are primarily what determine which material to use for a given application. This section provides a summary of the literature describing the evaluation methods for gaining access to the thermo-mechanical aspects and the results for various reinforcement materials and the accompanying compositions.

Anandha moorthy et al. [2012] used stir casting to create an alloy of graphite, fly ash, and aluminium. They fixed the weight percentage of the graphite at 3% and varied the fly ash's composition between 3% and 9% it has been observed that the load influences the rate of sliding wear. In addition, the hybrid metal matrix composite has a higher toughness than Al 6061.

As a matrix material Aluminium with 4.5% Cu was used and fly ash with different weight fractions (5 to 15%) was used as the reinforcement material to create the metal matrix composite, according to Mahendra et. al.[2010]. The composite is created using the stir casting method, and as the amount of fly ash raises, so do the composite's impact, compressive, tensile, and hardness strengths. But there is a decline in density and corrosion resistance.

Al alloy's pitting corrosion behaviour and corrosion kinetics were studied by Bienias et. al. [2003]. To create the composite using gravity casting and squeeze casting, they have employed fly ash as the reinforcement and AK12 as the matrix material. In comparison to an unreinforced matrix, fly ash particles cause the AK12/9% fly ash composite to pit more easily.

In order to create the composite by stir casting, Motgi et al. [2013] employed the matrix material as LM25 aluminium alloy, a fixed weight percentage of fly ash (3%), and varying weight percentages of aluminium oxide (5%, 10%, 15%). By examining this sample, it can be shown that the percentage weight of aluminium oxide increases the tensile strength and hardness. However, the main problem is the loss of ductility and impact strength.

In order to create the composite by stir casting, Arun Kumar et al. [2011] chosen to use fly ash with 2 to 8 weight percent and 2 to 6 weight percent of e-glass fibre as the reinforcing along with matrix material as al6061 alloy. As the weight percentage of fly ash rises, so do the material's hardness, tensile strength, and compressive strength. Additionally, an ultrasonic flow detector was used to examine the samples and find any flaws.

Umashankar et al. [2013] chose bottom ash as the reinforcement and Al6061 alloy as the matrix to create the composite via stir casting. With an increase in the weight percentage of bottom ash particles, the composite's micro hardness and tensile strength rise. But the tensile strength and micro hardness start to decline after 9% wt of bottom ash, which is an issue.

In order to create the composite by stir casting, Uthayakumar et al. [2012] used aluminium alloy 6351 serves as the matrix material, and fly ash with a weight proportion of (5 to 15%) serves as the reinforcement. It is clear from the results that the composite does not wear under light loads. And the results show that the applied load has the most influence on the dry sliding wear.

In order to create the composite by stir casting, Bharat et al. [2014] used the eutectic Al-Sialloy LM6 with two different types of cenosphere (fly ash type-A and type-B) as reinforcement and 12.24% Si as the matrix. Type-B fly ash has higher levels of micro hardness, tensile strength, impact strength, and hardness thanks to its minor structural differences. In order to create the composite, Sreenivasa Reddy et al. [2012] used Al 7075 alloy as the matrix material, and fly ash was added to e-glass fibre by adjusting the weight percentage. When compared to a cast specimen, the heat-treated specimen has higher hardness and tensile strength. The amount of fly ash and e-glass fibre can be changed to further improve the mechanical qualities.

In order to create the composite by stir casting, Anilkumar et al. [2013] selected Al 6061 alloy serves as the matrix material, while the reinforcement consists of fly ash with varying weight percentages (10%, 15%, and 20%) and particle sizes [4-25, 45-50, and 75-100 m]. The sample's evaluation reveals that the hardness, tensile strength, and compressive strength all increase with the fly ash weight percentage.

By using appropriate testing techniques, Mani et al. [2014] examined the composite's brittleness, tensile strength, and resistance to wear. They used fly ash as the reinforcement and al 6063 as the matrix material to stir cast the composite. According to the investigation, using fly ash boosts tensile strength, hardness, and wears resistance.

In order to create the composite by stir casting, Prabhu et al. [2014] used fly ash as the reinforcement and aluminium as the matrix material. The best metal removal rate and smallest overcut are determined via electrochemical machining. As a result, more fly ash is added, increasing the al-fly ash's hardness.

By using stir, squeeze, and gravity casting, Lokesh et al. [2013] studied the base alloys and the composite's impact, hardness, compressive, and tensile properties. The above properties are enhanced by the 3% to 12% weight percentage of fly ash in this. The base alloy produced by squeeze casting is less porous than the base alloy produced by gravity casting.

In a study of the mechanical properties of an aluminium alloy (LM6) reinforced with silicon carbide and fly ash conducted by Suragimath et al. [2014], the researchers found that the presence of fly ash tends to boost the wear resistance of the LM6/SiC hybrid composite.

According to Shanmughasundaram et al. [2011], fly ash increases the compressive strength of a material. The composites' compressive strength tends to decline when fly ash content rises

from 20% to 25% weight percent. However, the effect is weakened over 20% weight percent because the fly ash particles interact with one another due to particle clustering.

When the weight percentage of fly ash and SiC rose, Mahendra Boopathi et al. [2013] found that hardness increased. The hardest material is found at Al/(10% SiC+10% fly ash). The hardness and deformation of the Al matrix are both improved by the addition of fly ash particles. It has been found that an alloy made of aluminium has a lower hardness than a mixture of SiC and fly ash particles.

The stir casting procedure was used by Vivekananda et al. [2013] to create the aluminium fly ash composite. Fly ash increases the toughness of the composite by acting as a barrier to the migration of dislocations. Additionally, increasing the abrasive wear resistance by mixing fly ash with molten aluminium Particle reinforcement, dispersion reinforcement, and solid solution reinforcement all contribute to the composite's strength.

Garg et al. [2012] created a composite employing SiC, fly ash and using 6061 aluminium as the matrix material Stir casting is used to form the composite in which the fly ash weight fraction (5%), which is fixed, is used to control the silicon carbide weight fraction, which can range from 2.5% to 10%. The analysis shows that the tensile strength and hardness of the composite are increased by increasing the weight percent of SiC.

Aluminium with 4.5% Cu and 3% Mg used as matrix alloy with SiC particle carrying weight percent 5, 10 and 15 for particle reinforced composites have been studied to determine the effects of tensile properties, hardness, microstructure and density, according to Adem Onat et al. [2007]. The findings demonstrated that squeeze casting improved microstructure, reduced porosity, and reduced other casting flaws such shrinkage voids through the application of pressure during solidification. Squeeze casting improved the matrix alloy's tensile and hardness qualities. Hardness of composites rose as the volume fraction of SiC did. Porosity increased along with decreased elongation and area reduction as the volume percentage of the particles increased. Fracture surface study revealed that the matrix and the particles break concurrently in composite materials. Furthermore, this demonstrated that the matrix and SiC particles have a strong link, and the fracture start did not take place at the matrix-particle contact.

2.6 Strengthening mechanism of composite

Rule-Of-Mixture

Most studies concerned with the evaluation of mechanical behaviour of fibre reinforced composites use what is called a "Rule-Of-Mixtures" (ROM) to predict and/or to compare the strength properties of the composite The ROM is an operational tool that uses weighted volume average of the component properties in isolation to obtain the magnitude of the property for the composite. Specifically, in the case of composite containing uniaxial aligned, continuous fibers, the composite stress is written as

$$\sigma_c = \sigma_f V_f + \sigma_m V_m \dots (1)$$

Where σ is the axial stress, V is the volume fraction of the component and the subscripts c, f and m refer to the composite, fiber and matrix, respectively. It is to be noted that $V_f + V_m = 1$ Under conditions of isotactic, i.e, the longitudinal strain in the components being equal, one may write another ROM relationship for the elastic moduli, $E_c = E_f V_f + E_m V_m \dots (2)$

Where E is the elastic modulus and the subscripts represent the components as before. Eq. (2) neglects any transverse strain arising because of the different contractile tendencies of the components (i.e, $\mathbf{v_f} = \mathbf{v}$, where v is Poisson's ratio). However, for metallic systems, the difference in Poisson's ratio of the two components is generally insignificant and the ROM values are generally found to be within the limits of the experimental error. Another example of a property for which ROM works very well is the density ρ . One can write as

 $\rho_c = \rho_f \, v_f + \rho_m \, v_m \,(3)$. It would appear from these studies that the ROM as applied conventionally to the strength properties of composites with metallic matrices is not valid. The whole is more than the sum of individual components in isolation [Chawla KK, 1974].

2.7 Microstructural Properties

In this part, a literature study on the evaluation of the microstructure and characteristics of composite materials was covered. The microstructure had to have a homogeneous distribution of particles, little porosity at the particle-matrix interface, and mechanical qualities that were dependent on the matrix's properties, shape of the reinforcing phase, wettability, reinforcing particle size and amount of reinforcement.

Arun et al. [2013] looked into the properties of a stir-cast composite made of aluminium, fly ash, and alumina. Fly ash is selected as the reinforcement and Al 6061 as the matrix material. According to SEM investigations, the reinforcing particles in Al6061 alloys are dispersed uniformly.

In compared to the matrix alloy produced using the vortex process, Abdel-Azim et al. [1995] found that the matrix microstructure refinement was better in the 2024 Al/alumina composite. The grain size was lowered as the amount of alumina particles in the matrix increased.

According to Surappa's [1997] review, particle-solidification front interactions, homogeneous mixing of the melt-particle slurry, and wetting of the reinforcement by the liquid metal all contribute to the final particle distribution and matrix microstructure. Reinforcing particles had an impact on matrix grain size and dendrite arm spacing (DAS).

Hasim et al. [2001] looked at the challenges of achieving low porosity, high-quality wettability across materials, and uniform reinforcement distribution over the course of stir casting. Cast metal matrix composites were created by experimenting with a number of different parameters, such as stirring speed, holding temperature, pouring time, impeller size, and location in the melt. The inclusion of magnesium improved wettability, but increasing the concentration above 1 weight percent of magnesium caused the slurry's viscosity to rise and impair particle spreading. As the volume % of SiC particles in the matrix alloy grew, the wettability decreased.

Akhlaghi et al. [2004], reported that regardless of pre-heat temperature of the mold or the mold size and amount of SiC particle under semi solid liquid processing provides in lower porosity content and uniform distribution of SiC particulates during microstructural studies on A356/SiC composites.

According to Kok's [2005] observations the density of 2024 Al / alumina composites increased with increasing weight percentage. The realistic pressure applied after pouring improved the wettability and bonding strength between the alumina particles and the aluminium alloy. With smaller particles and a larger weight percentage in the stir-casting procedure, MMCs' tensile strength and hardness increased, while the elongation properties of composites decreased.

Using a scanning electron microscope, IPEK [2005] examined the microstructure of Al 4147 composites and worn-out surfaces (SEM). After performing wear tests on the specimens, the author noted the presence of the moderate wear and acute wear regions.

Onat Adem [2010], examined at the microstructure characteristics and mechanical characteristics of Al-4.5Cu- 3Mg with 15 vol.% SiCp matrix composites produced by the squeeze casting technique. The findings demonstrated that the composites include SiC particles that are evenly distributed and free of pores. Composites fail in both the matrix and the particles at the same time, indicating strong connection between the two. A SEM investigation revealed that the micromachining effects of the reinforcement phase eliminated oxidised and plastically deformed particles from worn surfaces.

Ramesh et al. in [2005] studied microstructure with Al6061 unreinforced ally and its composites. The castings' minor micro porosities were plainly visible on micrographs. Scanner electron microscopy was used to detect the uniform distribution of SiC reinforcement particles throughout the matrix.

Mahadevan et al. [2006] examined the microstructures of composites made of AA60616SiCp. The high wettability of the Al matrix for the SiC particulates was explained by the homogenous dispersion of the SiC particles in the matrix.

Sakthivel et al. [2008] worked on microstructure of 2618 Al-SiC composites formed using the stir casting method had homogeneous dispersion of particles, little agglomeration of particles, and moderate porosity. Additionally, they discovered that the composites' hardness and tensile strength rose as the size and weight of the reinforcing particles were reduced.

According to Rohatgi et al. [2010], in stir-cast A206/Silica sand composites, the dendritic arm space reduces as the volume percentage of the particles rises.

2.8 Tribological behaviour

Generally speaking, tribology analyses how surfaces move in relation to one another. Tribology studies lubrication, wear, and friction. Tribology is used in many different fields. Every mechanical system typically consists of moving parts, indicating the presence of a tribological work area. Friction needs to be minimised somewhere (gear, bearing), whereas friction needs to be maximised someplace (clutch, brake). Numerous manufacturing procedures are necessary, including turning, grinding, rolling, polishing, stamping, and tribology. Tribology is crucial to the transportation industries. Tribology is heavily dependent on the mechanical drive system as well as the contact surface of the wheel and track. Numerous pieces of equipment used in the construction industry, such as oil rigs, tunnelboring drills, excavators, pumps, etc., are dependent on tribological innovation [Stachowiak, 2017]. Friction is a factor in manufacturing for metal forming activities that affects tool wear and required power. Tool replacement, dimensional change, and an increase in working force are all effects of high tool wear. Therefore, there is a strong need for research on tribological improvement. Wear and friction of machine parts such as bearings, clutches, gears, wheels examples of typical tribology research areas. Additionally, the creation of novel, high-quality materials has opened up new avenues for the solution of many tribological issues [Totten, 2006]. Researchers' attention has been drawn to tribological studies of aluminium based metal matrix composites under various experimental settings.

In the production of a range of automotive engine parts, such as cylinder blocks, pistons, cylinder liner and piston insert rings, where adhesive wear (dry sliding wear) is a key process, aluminium metal matrix composites are frequently employed [Deuis et al., 1997]. Materials with strong resistance to wear (under dry sliding conditions) were connected to with the development of fine equiaxial wear debris and a solid tribolayer covering the damaged surface [Sannino and Rack 1995] claim that the type of reinforcement, size and shape of reinforcement, orientation, and percentage of reinforcement plays a vital role in finding parameters that covers tribological aspect that regulate the friction and wear of aluminium composites of discontinuously reinforced type under dry sliding situations. The environment, temperature, counterpart material, applied stress, sliding velocity, sliding distance, and they are together referred to as mechanical and physical parameters, also have a vital role in finding the tribological behaviour of aluminium composites.

Translated from the Hebrew by Onat Adem [2010], This research looked into the squeeze-cast Al-4.5Cu-3Mg/15 vol.% SiCp matrix composites' microstructure traits, mechanical characteristics and dry sliding wear characteristics. Sliding wear tests were conducted against an AISI D2 steel disc at velocities of 0.5, 1, and 2.0 m/s with loads of 5, 10, and 15N over a

distance of 1000 metres. As the weight and sliding speed were increased, the composites' friction coefficient fell. Furthermore, the wear rate increases with both the applied force and the sliding speed.

High temperature wear of Al6063-TiB2 composites was investigated by Natarajan et al. [2009]. At temperatures up to 100 °C, the wear rate of the monolithic alloy and composites increased marginally. A fast increase in wear rate up to about 200 °C indicates specimen seizure; after this point, the increase in wear rate becomes moderate. As the amount of TiB2 increased, the wear rate reduced across the board.

Selvi et al. [2013]'s theoretical and practical investigation of the mechanical characteristics of Aluminium MMCs produced the same result: fly ash particles strengthen the composites resistance to wear. An Evaluation Fly ash containing SiO₂ increases the wear resistance of Al MMC, and sliding wear test results reveal that wear rates vary.

Shivaprasad et al. [2013] have created stir cast composites using fly ash at a 10% weight fraction as reinforcement and aluminium alloy AA2024 as the matrix material. The AA2024+10% fly ash composite with heat treatment has better wear properties than the untreated composite. The water-cooled condition has a lower specific wear rate as compared to the air-cooled condition.

The wear coefficients for Al6061-TiO₂ composites were analysed by Ramesh et al. [2005], who relied on Archard's and Yang's theoretical models. The hardness of composites' increased with increasing volume % of TiO₂and their wear coefficient decreased. At higher loads and greater sliding distances, the wear coefficient dropped for all of the Al6061-TiO₂ composites tested. The matrix alloy and the composite pin surfaces softened, leading to significant deformation, when the temperature of the sliding surface rose proportionally more rapidly at greater sliding distances. Because of this, wear losses on the composites is increased.

Different casting processes have been explored by Prasad et al. [2013] to see how they affect mechanical and tribological properties like friction, wear rate, hardness etc. When it is compared to aluminium alloys made using gravity casting or squeeze casting, the 7.5%

weight fraction of al -fly ash is superior in terms of toughness and wear rate. The gravity casting process results in a sample with poor hardness and a high wear rate.

Increasing the amount of SiC particles present in the A356 alloy (from 5 to 20 vol. %) reduces both the friction and wear rate, as shown by Yalcin and Akbulut [2006]. When tested at 5 N applied load, however, specimens reinforced with 15 and 20 vol.% SiC demonstrated an increase in friction coefficient. The insufficient connection between the SiC particles and the matrix is thought to be the root cause of this rise. Particle segregation and poor bonding can lead to particle transfer at the matrix-WC ball-disc interface, which in turn generates vibration.

In order to manufacture a composite, Prasad et al. [2013] applied pressure to a eutectic -Al -Si -alloy matrix material that contained reinforcements of increasing fly ash (in wt.%).

The composite's sliding wear resistance increases as its fly ash content rises in percentage by weight. Squeeze casting was used to eliminate the voids in the composite and get this result. RL Deuis et al. [1997] Researchers found that good resistance to wear (in dry sliding conditions) is linked to the presence of a solid tribolayer on the damaging surface and the development of fine equiaxed wear particles. The notable parameters in regard to the wear mechanism experienced by some of the factors include the influence of reinforcement fracture toughness, sliding speed, wearing surface hardness, applied load and morphology for adhesive wear. This article reviews recent developments in our understanding of wear, focusing particularly on the ideas, challenges, and mechanisms of counterface wear. Related studies, such as those focusing on the role of the reinforcement phase, are also discussed as they pertain to the topic of adhesive wear of Al-5 alloys and aluminium composites with discontinuous reinforcement phases.

2.9 Statistical analysis by Taguchi method

A strategy for methodically using statistics in experimentation is called design of experiment (DOE). A mathematical model that forecasts how input factors interact to produce output variables or responses in a process or system can be created by experimenters using DOE. Nearly all engineering-related experiments, among many others, can be conducted with DOE. In general, DOE is used to construct a mathematical model in order to derive prediction

equations for response optimization. ANOVA is used to assess for statistical significance, while the prediction model is developed by regression analysis. This methodology examines how changing the intensity at which a set of parameters is used affects the response. The statistical theory of experimental design, which is ideally suited for engineering investigations [Davies 1954, Cochran and Cox 1987, Douglus Montgomery 1990], offers a number of methods. DOE techniques like Taguchi, surface and factorial response have become more significant since they were useful in revealing information on the influence of different parameters in different levels of requests. These methods allow for the decomposition of the combined impacts of many parameters and the discovery of relationship terms.

The impact of Al-4032 based composite reinforced with SiC particle on tribological behaviour (wear characteristics) was examined by Kumar and Singh, [2019]. Analyzing the signal-to-noise ratio and the analysis of variance, the operating parameters of applied load (10, 20, 30 N), rotating time (5, 10, 15 min) and rotational speed (200, 400, 600 RPM) have been attempted to be optimised ANOVA. The results of the ANOVA analysis show that the disc's rotational time and applied load have a substantial impact on the tribological behaviour.

Using the Taguchi technique, Prasad and Ramachandra, [2013] examined the impact of the process variables on wear resistance in squeeze casting of LM6 -fly ash composite. The variables under investigation include squeezing pressure, squeeze time, and percentage weight of fly ash. The percentage of weight fly ash content, squeezing pressure, and squeeze time, in that order, have the greatest influence on the wear rate of the composite.

Al/SiCp composite dry sliding wear behaviour was studied by Basavarajappa et al. in [2007]. Data collection was handled using the Taguchi approach. To evaluate the effects of wear parameters such as normal load, sliding speed, and sliding distance on dry sliding wear of the composites, an orthogonal array and analysis of variance were used.

Using five level factorial design techniques, Charles et al. [2006] created a mathematical model for the machining of hybrid aluminium composites reinforced with SiC and fly ash particles. The regression coefficients were calculated using the ANOVA technique, and the constructed model's significance was also examined. They found that surface roughness rose

as SiC particle volume percentage and current increased, but decreased as pulse length increased.

Using a linear factorial design methodology, Sahin [2003] created a mathematical model for the wear rate of composite. He stated that the direct and indirect impacts of variables on the response were studied using this model. He came to the conclusion that as the applied stress and sliding distance grew, so did the composites wear rate.

Basavarajappa and Chandramohan conducted an experimental investigation on the dry sliding wear characteristics of hybrid composites made of 2219 Al/15%SiC and Al/15%SiC/3% graphite [2006]. In order to develop a linear regression expression that was beneficial in forecasting the wear volume loss for a class of experimental situations, the author used Taguchi's design of experiments. The term was understood to mean that SiC-Gr composites had significantly higher wear resistance than SiC reinforced composites. The wear volume loss was said to have decreased, and the sliding speed of the two composites was said to have increased. In Al/SiC/Gr reinforced composites, the interactive impact between load and sliding-speed predominated, using sliding distance as a point of comparison.

Kok [2010] used the Taguchi technique to investigate the abrasive wear properties of cast composites made of 2024 aluminium alloy supplemented with Al₂O₃. In comparison to abrasive grain size, reinforcement size was determined to have the greatest impact on abrasive wear. While it reduced with increasing reinforcement size and sliding distance, the wear rate of the composites increased with increasing abrasive grain size and applied load.

2.10 Critical appraisal of existing materials

Traditional monolithic materials aren't always able to combine strength, stiffness, toughness, and density well. Composites are the most promising materials of current research because they have the potential to overcome these drawbacks and satisfy the constantly growing demands of modern technology. In comparison to unreinforced alloys, metal matrix composites (MMCs) have much better attributes such as high specific strength, specific modulus, damping capacity, and strong wear resistance. AMMCs are gaining importance due to an increasing demand of stronger, stiffer, newer and yet light-weight applications in the fields of aerospace, automobiles and energy sectors. But these composites are more

expensive, therefore their usage is mostly restricted. There has been an increasing interest in composites containing low density and low cost reinforcements. Typical reinforcement particulate phases used in AMMC's are graphite, mica, silica, zircon, alumina, silicon carbide, boron carbide, quartz etc. Once again their utilizations are restricted due to high cost. Fly ash is one of the most affordable and low density reinforcements available in significant amounts as a solid waste by-product following the burning of coal in thermal power plants, among the several discontinuous dispersoids employed. In order to overcome the cost barrier for widespread use in the automotive and small engine industries, composites containing fly ash as reinforcement are likely to be used. As a result, one of the key innovations needed in the field of materials during the past few decades has been the creation of low cost MMCs. Therefore, it is anticipated that the addition of fly ash particles to aluminium alloy will encourage yet another application for this inexpensive waste by product, while also having the ability to conserve energy-intensive aluminium and lower the price of aluminium products.

According to the published work available on the fabrication, characterization, mechanical, tribological properties of MMCs and Optimatization of tribological properties through Taguchi's technique, the following observations are made:

- Comparatively less literature is available on Al-Si (Eutectic) alloy (LM6) based fly ash composite.
- Comparatively less literature is available, commenting on effect of morphology of reinforcement on mechanical, physical and tribological properties of Al-Si alloy (LM6) base composite reinforced with fly ash.
- Very few work found on optimization technique of tribological properties.

However the present research work has been undertaken with a main objective to overcome the problems during fabrication, characterization and optimization of Al Alloy- Fly Ash composites. The work is focused on the following aspects:

- Judicious selection of fly ash and its characterization (Physical, Chemical)
- Fabrication of Al-Si alloy-Fly ash composites with minimum porosity by stir casting method.
- To analyze the results of various weight fractions of composites specimens.

- Scanning electron microscopy and optical microscopy / microstructure analysis to determine how reinforcements should be distributed. XRD, SEM, and EDS analysis of Al Si alloy-fly ash composites.
- Comparative study of the properties such as tensile strength, yield strength, tensile strain, Young's modulus, density, micro-hardness of Al Si alloy-Fly ash composites with the matrix alloy.
- Investigations on the effect of parameters such as applied load, sliding speed and sliding time of particulates on the dry sliding wear and coefficient of friction for MMCs by using Taguchi technique.

2.11 Research Methodology

Based on the literature survey it is decided to carry out investigation on aluminium alloy as matrix (LM6) with a reinforcement of fly ash (Class F) particulate. Composite fabrication will be carried out in Stir cast bottom poured furnace. Fabrication to be done with four variants of 2%, 4%, 6%, 8% of fly ash. Our objective is to carry out investigation on mechanical properties, microstructure properties, and tribological behaviour. Optimization is also carried out with the Taguchi's DOE and ANOVA method.

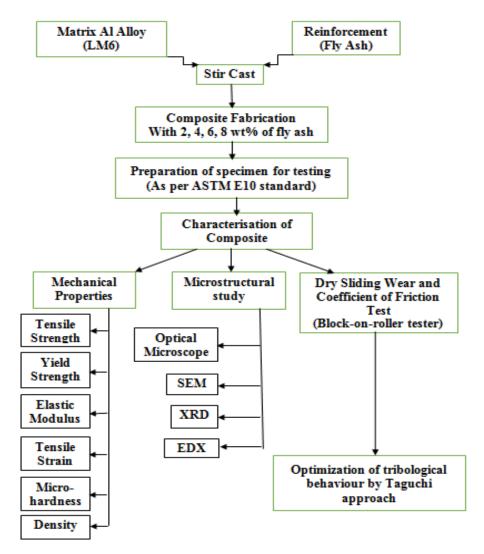


Figure 2.1: Research Methodology

Figure 2.1 is self explanatory to know the details of different kind of test to be performed. While preparing test specimen, several numbers of sample were made to get mean data.

2.12 Conclusion

This chapter deals with extensive literature survey on selection of base alloy, selection of reinforcement, fabrication method used for this work. Extensive reviews on existing literature are also made related to mechanical properties, metallurgical properties, tribological behaviour and optimization of dry sliding wear and friction variables through Taguchi method of aluminium fly ash composite. Finally, based on optimisation with DOE and ANOVA, the most likely priority variables are decided for optimum result.

Chapter 3

Experimental Methods

Outline of the Chapter: 3.1 Introduction, 3.2 Materials used, 3.2.1 Matrix material, 3.2.2 Reinforcement material, 3.3 Composite Fabrication 3.4 Mechanical Properties, 3.4.1 Tensile Test, 3.4.2 Hardness Test, 3.4.3 Density Measurement, 3.5 Metallurgical Properties, 3.5.1 Optical Micrograph, 3.5.2 SEM Micrograph, 3.6 XRD Plot, 3.7 EDS Analysis, 3.8 Tribological Studies, 3.8.1 Wear and Friction measurement, 3.9 Statistical analysis through Taguchi method, 3.10 Conclusion

3.1 Introduction

In this chapter the discussion is made on matrix material, reinforcements, fabrication method of composite. Different experimental procedures as well as equipment used for every property of the compositearealso explained. For finding out the mechanical properties UTM, Vickers hardness tester equipment and test procedure is explained. For metallographic, the process and equipment details of optical microscope and scanning electron microscope are discussed. For validation XRD and EDS technique are explained. For wear and COF analysis block on roller tribotester is explained. At the last Taguchi's DOE method is explained for optimisation.

3.2 Materials used

3.2.1 Matrix Material

In this study, as a matrix material aluminium alloy (LM6) was chosen. The chemical composition shown in **Table 3.1** reflects Silicon contains at high percentage of around 10 - 13. Therefore, the alloy has good strength at high temperature, high strength to wear, good thermal conductivity, and better corrosion and wears resistance properties. The main advantage of this material is that fabrication can be managed easily at reasonable expenditure without preheating.

Table 3.1: Chemical composition of LM6 (Al-Si) matrix alloy

Compo	Pb	Mg	Zn	Ni	Cu	Sn	W	Mn	Fe	Si	Al
sition											
%	0.1	0.1	0.1	0.1	0.1	0.5	0.2	0.05	0.6	10 - 13	Rem.
											1

3.2.2 Reinforcement material

The by-product of burning coal in a steel or thermal power plant is called fly ash. When anthracite or bituminous coal is burned in a power plant, fly ash is produced which ispozollanic and is known as class F type fly ash. In modern coal fired power plants, ash is collected through electrostatic precipitator or other filtration equipment before the flue gas reaches the chimney. Ash which cannot fly is known as bottom ash. According to the type of coal used in the furnace, fly ash has a variety of chemical components. But all type of fly ash contains silicon di-oxide (SiO₂), calcium oxide (CaO) and others. Availability of toxic components in the fly ash depends on the kind of coal bed used. However, following elements are found in some percent: arsenic, strontium, vanadium, beryllium, thallium,lead, manganese, mercury, molybdenum, selenium, boron, cadmium, chromium, cobalt, cobalt, chromium etc.In the present work, class F type fly ash of 150 – 175 micron particle size is collected from Mecheda thermal power station, Kolkata. The physical properties of class F fly ash are shown in **Table 3.2**and of chemical composition in **Table 3.3**. Loss on ignition (LOI) is a measurement of the amount of unburned carbon in fly ash. It's an important characteristic of fly ash, especially for concrete applications.

Table 3.2: Physical properties of Class F Fly Ash

Product name	Class F Precipitator Fly ash				
Density	2.2 gm/c.c				
Color	Grey				
Form	Solid Spherical				
Particle size	150 μm				

Table 3.3: Chemical composition of the Fly ash

Comp	SiO ₂	Al ₂ O	Fe ₂ O	Mg	CaO	K ₂ O	TiO	Na ₂	P ₂ O	Mn ₂ O	SO_3	LOI
osition		3	3	Ο			2	Ο	5	3		
%	49.4	29.6	10.7	1.3	3.47	0.54	1.76	0.31	0.53	0.17	0.27	1.45
	5	1	2									

3.3 Composite Fabrication

Aluminum alloy (LM6) of density 2.68 gm/cc is considered as base material. Fly ash with average particle size 150 µmwith density 2.2 g/cc is considered as reinforcement. Table 3.2 shows the physical characteristics of fly ash. In this, metal matrix composites are created using a bottom-poured stir casting resistance furnacewith different percent (2%, 4%, 6% and 8%) of fly ash. There are two separate chamber fitted with the furnace. One is mechanical stirring unit and another is particle or reinforcement heating unit. Figure 3.1 shows the furnace set up. Aluminum alloyis put in the crucible of the furnace. The temperature in the panel is set at 750°C so that aluminum ingots get melted. Required proportion of fly ash is placed inside the particle preheater unit for a temperature of 500°C so as to remove moisture content in fly ash and to form an oxide layer over it. In most cases, fly ash particles become entangled in gas bubbles, resulting in poor distribution and porosity. On observation when it is found that all alloy ingots melted at 750 °C, a mechanical stirrer is run at high speed (500 rpm) to create a vortex in the chamber. In the particle pre heating unit fly ash particle is already removed with moisture and heated for 450°C. Then pre-heated fly ash particles are added gradually. [Banerjee et al. 2019]. Magnesium of 1.5% is added into the mold for enhancing the wetting action between the constituents of the composite. Reduced wettability leads to porosity, as well as non-uniform reinforcing particle dispersion, are common side effects of lower Mg concentrations. Stirring speed of the impeller is maintained at 600 rpm and continued for 15 – 20 min. Finally melt was poured into 50 mm cylindrical mold. After the die has cooled, the composite bar is taken out of the die and machined to create the necessary samples [Banerjee et al. 2019].

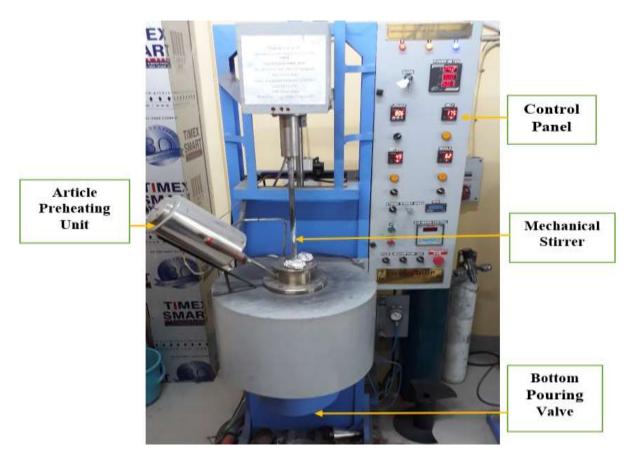


Figure 3.1.Bottom Poured Stir Casting Resistance Furnace [Courtesy Smart Foundry Lab: JU]

3.4 Mechanical properties

Composite specimen is prepared according to ASTM E10 standard. Tensile properties i.e UTS, yield stress, Young's modulus, tensile strain, density, micro-hardness of the composite under different weight percent of fly ash are measured. Comparative study for the composite over base alloy is analyzed.

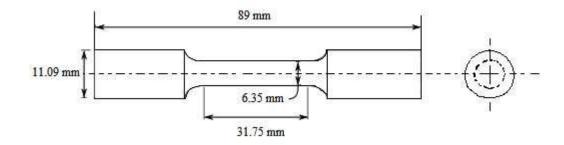


Figure 3.2: Tensile Specimen

3.4.1 Tensile test

In this present examination Tensile test was carried out on composites in UTM. Specimens were prepared by following standard ASTM E-10 [ASTM standard, 2004]. All of these specimens underwent tensile testing in the lab in accordance with ASTM E-10 standard. The tests were conducted utilising a computerised Instron-8801 electromechanical testing machine as shown in **Figure 3.3**. Strain rate is defined as the change in strain (or deformation) of a material with respect to timeat ambient temperature and 1 mm/min crosshead speed. A rate of 1% per minute on a specimen having 100 mm length results in a crosshead speed 1 mm/min. Tensile strength and percentage elongation data were computed using load-displacement graphs produced from an X-Y recorder.



Figure 3.3: Instron tensile test machine (Instron-8801)[Courtesy: JU]

3.4.2 Vickers hardness test

In current study, Vickers's micro-hardness of the composites is evaluated as per ASTM standard E92 using a UHL make micro-hardness tester (VMHTOT, Technische Mikroskopie). Example surfaces are indented subjected to full load of 50 gf for duration of 10 to 15 seconds. Five times

in five separate regions, each sample is indented. A microscope is used to measure the indentation left in the material's surface when the load has been removed. The test apparatus used for this purpose is shown in **Figure 3.4**. The micro hardness tester's built-in touch screen is used to operate it, and a digital camera is used to capture and upload photographs of the indentions to a computer. Utilizing specialised computer software, the indentation image is processed to produce the hardness values. To do this, the lengths of the diagonals of the indents formed on the surfaces of the specimens are measured and analysed using an optical microscope [Poria et al. 2016]. The hardness analyzer is actually linked to a computerized system that uses a high-tech camera to capture space marks. Examining space impressions determines the hardness value. Beforehand, the surfaces are polished with fine SiC-grit paper prior to hardness tests since lighter indenter requires smoother surface to obtain good results. The specimens are mounted in the fixed platform and held perpendicular with the indenter.

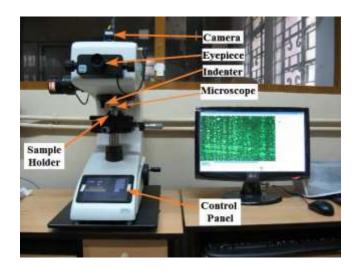


Figure 3.4: Vickers micro-hardness testing apparatus[Courtesy: JU]

3.4.3 Density

The Archimedes principle was used to achieve the density of the composite samples. The hypothetical intensity was calculated using the mixtures rule, as per the mass fraction reinforcement. The density of as-cast composites is investigated using the Archimedes principle.

$$\rho = \frac{Wair}{(Wair - Wwater)} \rho W$$

Equation: 3.1. Density Relation of composites

3.5 Metallurgical properties

Composites prepared from matrix alloy and fly ash reinforcement is carried out microstructural analysis through optical microscope and scanning electron microscope. Three specimens each are prepared from composites with 2%, 4%, 6%, 8% weight of fly ash.

3.5.1 Optical microscope

An optical magnifying instrument was utilized to look at the microstructure of as-projected composites in this examination. Optical microscope is one of the most important tools to study the microstructure, despite evolution of many other sophisticated instruments.



Figure 3.5: Optical Microscope (Leica DM 2700M)[Courtesy: JU]

This can be used to analyze as-polished or etched metallographic specimens. Emery paper of grades 120, 180, 400, 800, 1200, 1500, 2000, 2500, 3000 was used to polish the sample. For a scratch-free mirror finishing polish, use a velvet cloth and non-ferrous polishing solvent. Further, the specimens are etched with Keller's etchant, which consists of 2.5 ml of nitric acid, 1.5 ml of hydrochloric acid, and 1 ml of hydrofluoric acid in 95 ml of distilled water. The specimen must be properly prepared for correct observation. Etchant is normally used to reveal the grain structure. Microscopic examination is carried out in Optical Microscope in model Leica DM 2700M as shown in **Figure 3.5**.

3.5.2 Scanning electron microscope (SEM)

SEM micrograph taken in FEG QUANTA 250 as shown in Figure 3.6 using Emission current 102.1 uA, chamber strain 80 Pa, gun pressure 2.49 x10⁻⁷ Pa, voltage 20 kV – 25 kV, working distance 10 mm, cut gap 40 um, Detector utilized ETD and LFD.To create a mirror-like surface finish for micro structural investigation, a series of grinding and polishing procedures were conducted on the samples' surface. Before the test, the samples were polished with grade 600, grade 1000, and grade 1200 abrasive paper, then etched with Keller's reagent (95 ml water, 2.5 ml HNO₃, 1.5 ml HCl, 1.0 ml HF) and swabbed for 10–20 seconds.



Figure 3.6: SEM and EDX Machine (JSM 6360)[Courtesy: JU]

3.6 X-ray diffraction analysis (XRD)

X-rays bear small wavelength and high energy electromagnetic radiation, of the order of the solids' interatomic gaps. A non-destructive approach is X-ray diffraction, in which study properties, degree of crystallinity, texture, crystalline phases of the composite can be made with the help of monochromatic X-ray beam. The whole principle of working based on Bragg Law. The XRD method was utilized to investigate the prepared composite materials to affirm the presence of different phases. It demonstrates the connection between relative intensity and diffraction angle (2θ). With a scan rate of 0.02 ° (2 Hz), the diffraction angle range of 10 to 90° was maintained. The scan rate is a measure of how long the spectrometer takes to scan a mass spectrum. So the analysis was conducted at a rate of 40 kV voltages and 30 mA current. **Figure 3.7** shows the experimental set up.



Figure 3.7: XRD analysis Machine (ULTIMA III) [Courtesy: JU]

3.7 Energy dispersive X-ray analysis (EDS)

The use of EDX was employed to confirm the construction of a uniform component distribution. In EDAX GENESIS, EDX was completed. **Figure 3.8** shows the experimental set up.



Figure 3.8: SEM with EDS [Courtesy: JU]

3.8 Tribological properties

3.8.1 Block – on – roller Triboster test for wear and friction measurement

Table 3.4: Test parameters of dry sliding wear samples

Test parameter	Unit	Values			
Weight percentage of fly ash	percent	2, 4, 6			
Load	N	25, 50, 75			
Sliding speed	RPM	400, 500, 600			
Duration	Seconds	1800			
Temperature	0 C	Ambient			

A Computerized block – on – rollertribotesteras shown in **Figure 3.11** isutilized to examine the characteristics of the composite under dry sliding wear condition with three varying loads under

three varying speeds with three different weight percent of fly ash. The COF of the Al – Fly ash matrix alloy and composites varies with varied loads (25N, 50N, 75N) at 500 rpm and varied speeds (400 rpm, 500 rpm, 600 rpm) at 75 N. As the load was raised, COF of the composites decreased. The friction coefficient decreases as fly ash weight percentage rises. Rana et al. [1989] examined the friction coefficient between Al-1.5 percent Mg alloys reinforced with SiCpthey found that the friction coefficient reduced as the volume proportion of SiC particles rose. In Cu reinforced with Al₂O₃ particles, friction and wear were studied by [Saka et al. in 1985]. They discovered that the friction coefficient dropped as the alumina percentage rose. The coefficient of friction, wear rate, and mechanical behavior of the manufactured composites change as the weight percentage of fly ash in the composites increases [Zhenfeng et al. 1994] reported a similar observation. When the weight percentage of fly ash particles was raised, the hardness, ultimate tensile strength, and wear resistance of the composites improved, but the coefficient of friction and density of the composites decreased. The rise in temperature of the contact surface rises and softens the surface of the pin, resulting in a drop in coefficient of friction as the load increases. As a result, the friction coefficient falls. Another cause could be that as the load increases, the pin surface wears out more, and the wear debris becomes lodged between the pin and the counter surface, acting as a roller ball. As a result, the COF falls.

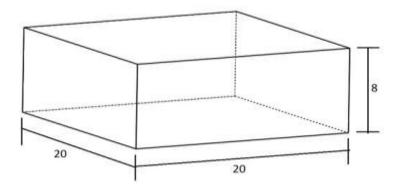


Figure 3.9: Wear Test Specimen (dimensions in mm)

Wear test specimen for the test is shown in Figure 3.9. The machine was incorporated with a LVDT for wear rate measurement. [Basavarajappa et al. 2007]. Volumetric loss and sliding distance were divided to determine the wear rate (Wr = Vr/SD). The tangential force (F_T) to normal force (F_N) ratio is used to compute the coefficient of friction (μ). The load cell installed in the pin-on-disk device provides the tangential force. Additionally, a strain gauge force

measuring device was put on the apparatus to assist in determining the COF.All of the specimens were put through a minimum of ten minutes of testing at various sliding velocities of 400, 500 and 600 revolutions per minute with normal weights of 25 N, 50 N and 75 N. A controller was used to record the outcomes on a PC. Each test was run three times, and the average results were used to determine the wear rate of the composite and COF.



Figure 3.10: Multi Tribo-Tester (Ducom, TR 25)[Courtesy: JU]

At room temperature, a block-on-roller machine of the Ducom, India, type was used to evaluate the wear of composite samples at various speeds (400, 500, and 600 rpm) and under various weights (25, 50, and 75 N). The tests, which lasted 30 minutes, looked at how the composite samples behaved with regard to wear and coefficient of friction (COF). As a counter body, a revolving roller disc made of EN31 hardened steel with an RC 62 hardness was used. **Figure 3.11** displays the Multi Tribo-Tester (TR-25) of the wear testing apparatus.

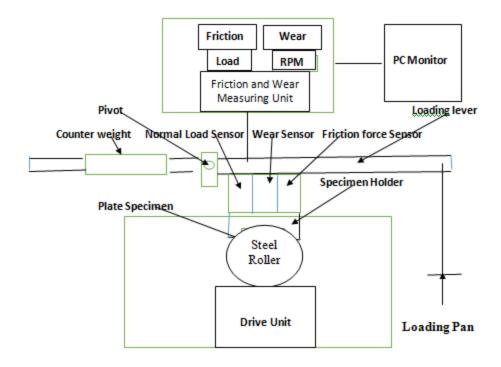


Figure 3.11: Block diagram of Multi Tribotester(Courtesy: JU)

3.9 Taguchi method for statistical analysis on tribological behavior

A systematic way to ascertain the relationship between the variables influencing a process and its result is called design of experiments (DOE). ANOVA study and signal to noise ratio (S/N) analysis are made to do statistical analysis of wear rate and COF of Al – fly ash composites. In order to obtain optimality, Taguchi recommended using S/N ratio analysis rather than simple averages of experimental findings because S/N ratio analysis can investigate the variability of experimental results within the experimental condition. As a result, in this study, the S/N ratio was used to analyze wear rate and COF with the smaller being the better criterion. The S/N ratio is calculated using the formula:

Where, n = number of experiments and y = observed data

$$\frac{s}{N} = -10\log\left(\frac{1}{n}\sum_{i=0}^{n} Y_i^2\right)$$

Equation: 3.2. Taguchi relation

Where n denotes the number of experiments and Y denotes the observed data

Wear Analysis

It reveals that the most important factor in controlling the wear rate of composites is reinforcement based on S/N ratio. Whereas load has a moderate impact and speed has the least impact. With the help of the main effect plot and interaction plot most influencing and least influencing variables are identified. Therefore optimality of variables can be calculated. ANOVA table ascertains the optimality obtained from S/N ratio, main effect plot and interaction plot.

COF analysis

It demonstrates that the most that load has the greatest influence in limiting COF of Al – fly ash composites based on S/N ratio, whereas speed has a moderate impact and reinforcement has the least impact. With the help of the main effect plot and interaction plot most influencing and least influencing variables are identified. Therefore optimality of variables can be calculated. ANOVA table ascertains the optimality obtained from S/N ratio, main effect plot and interaction plot. Further statistical analysis is made from residual plot which describes about normal probability plot, versus fits plot, versus order and histogram.

3.10 Conclusion

This chapter describes about the machines and methods used for mechanical tests, metallurgical observation, tribological behaviour. It also describes about the specimen, matrix material and reinforcement properties. It gives brief description about design of experiment which is used to find composite wear and COF and its most influencing variables.

Chapter 4

Results & Discussion

Outline of the Chapter: 4.1 Introduction, 4.2 Mechanical Properties, 4.2.1 Tensile Test, 4.2.2 Hardness Test, 4.2.3 Density Measurement, 4.3 Metallurgical Properties, 4.3.1 Optical Micrograph, 4.3.2 SEM Micrograph, 4.4 XRD Plot, 4.5 EDS Analysis, 4.6 Tribological Studies, 4.6.1 Wear measurement, 4.6.2 Friction measurement, 4.7 Statistical analysis through Taguchi method, 4.7.1 Wear analysis 4.7.2 COF analysis 4.8 Conclusion

4.1 Introduction

As Fly ash of micron size (µm) is involved in composite, proper fabrication method (Stir casting resistance furnace) is used. All the variables i.e. temperature of melt, preheat of fly ash, stirring time, stirring speed, pouring time etc are taken care for the preparation of composite. Specimens for mechanical properties are prepared for tensile test, micro-hardness test, density measurement. Specimen for metallurgical studies and tribological studies are made as per standard procedure and sizes for appropriate result.

4.2 Mechanical properties

A composite obtained through liquid metallurgy route in specific metallic mold is further made into samples for analyzing mechanical properties. In this, tensile stress, yield stress, young's modulus, tensile strain, micro-hardness and density measurement of composite are carried out for analysis.

4.2.1 Tensile test

The test is carried out on specimens made as per ASTM-E10 in INSTRON 8801 universal testing machine.

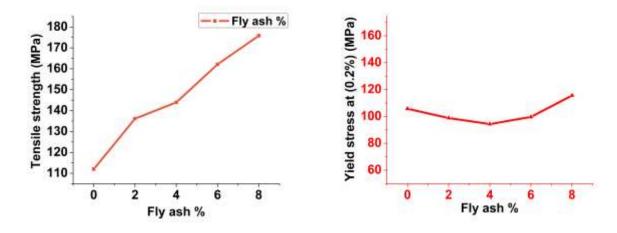


Figure 4.1: Tensile Strength of composite Figure 4.2: Yield Stress of composite

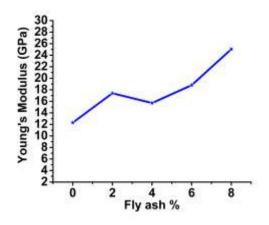


Figure 4.3: Young's Modulus of composite

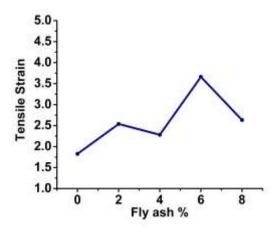


Figure 4.4: Tensile Strain of composite

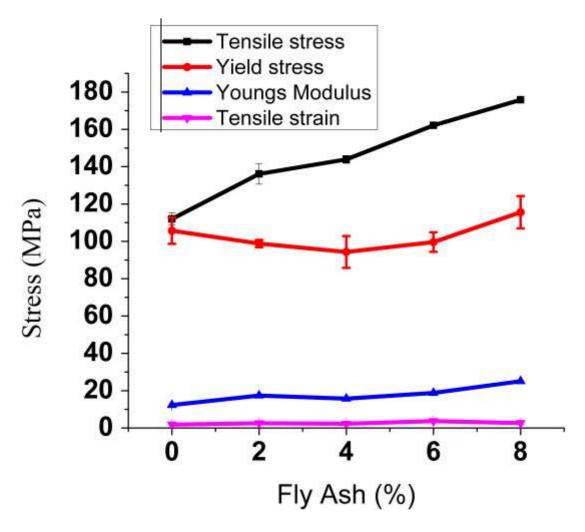


Figure 4.5: Tensile Properties of Composite

Figure 4.1 to 4.5 shows a graphical representation of the UTS values of composite materials containing 2, 4, 6 and 8% fly ash particulates and table 4.1 shows the data obtained from all these experiments.

Experimental research on composites reinforced with fly ash demonstrates that tensile strength of the composite increases as weight % of fly ash increases. Due to the fact that it is typically significantly stiffer than the matrix, the reinforcing phase in metal matrix composites carries a sizable portion of the stress. Stress concentrations in the matrix at the poles of the reinforcement and/or at sharp corners of the reinforcing particles have been suggested as the cause of micro plasticity in MMCs, which occurs at relatively low stress as observed by [Wilkinson and Corbin, 1994]. Due to an increase in the number of stress concentration

locations, the volume fraction of reinforcing particles at first reduces the micro yielding stress. [Chawla, 2006]. Tensile strength has reportedly increased from 112 MPa to 175.82 MPa, thereby an increase by an amount of 57% compared to the basic alloy, as shown in Figure 4.1 & 4.5. The microstructure plays a major role in particulate reinforced composites. There can be decrease in grain size with the rise in weight % of fly ash thereby increasing the tensile strength. Increased dislocation density and higher tensile strength are both caused by variations in the coefficient of thermal expansion (CTE) between the matrix and the particle reinforcement. The presence of ash particles that function as safeguards against the dislocations when absorbing the applied load might also be attributed for this rise in tensile stress. The majority of the alumina, silica, and hematite found in fly ash particulates are truly hard by nature. The matrix is strengthened as a result of the rigid fly ash particles blocking the moving dislocation front. The filler fly ash's increased strength is what's responsible for the composite's observed increase in tensile strength.

For base alloy yield stress value is 105.7 MPa. However it gradually increased to 115.56 MPa on increasing reinforcement from 2 to 8 weight percent as shown in Figure 4.2 & 4.5. Therefore it is reported 9.32% increase in the property; however it is observed that during the range of 0-4 % reinforcement yield stress fall by equal amount due to grain realignment in the composite.

Young's modulus is one of the parameters in particle reinforced MMCs that has been observed to significantly increase when the fly ash % increases. The elastic modulus for base metal as 12.31 GPa shown in Figure 4.3 & 4.5. It rose to 25.04 GPa at 8 % of reinforcement. Therefore, it is observed while finding out Young's modulus, there is substantial rise in it. Zhang et al. [2002] and Rohatgi et al. [2006] reported similar phenomena with increasing fly ash content. At first, they noticed that the elastic modulus decreased as the weight fraction of fly ash rose. However, after 4 wt% fly ash, the elastic modulus started to rise with rising fly ash content. Similar findings were observed by Kolukisa et al. [2003]. Fly ash-reinforced aluminium MMCs with equal stiffness and lower cross sections can be employed in composite applications where stiffness is a key design criterion due to higher elastic modulus, which denotes greater rigidity. This will eventually result in a smaller component mass.

The amount of lengthening a work piece experiences under tension is known as tensile strain or elongation. It has been discovered that when the quantity of fly ash in an aluminum alloy increases, the ductility of the alloy reduces, and as a result, the elongation percentage of various specimens falls. Figure 4.4 & 4.5 demonstrates that when the weight fraction of fly ash increased, the composites' ductility reduced. This is brought on by the fly ash particles' hardness or clustering. Even in composites that are defect-free, several elements such as particle size and the weight percentage of reinforcement have an impact on the percentage of elongation of the composites. It is found that marginal high tensile strain occurring for the composite during 6% of reinforcement. Due to particle clustering and porosity, it has been found that adding fly ash to the composite reduces % elongation. The same was reported in the work of Gikunoo et al. [2005].

Table 4.1: Tensile properties of composites with various fly ash weight percentages

Mechanical Properties/ Fly Ash %		2	4	6	8
Tensile stress at Maximum Load (MPa)	112	136.14	143.93	162.15	175.82
Yield stress at Yield (Offset 0.2 %) (MPa)	105.7	98.79	94.3	99.6	115.56
Young's Modulus (MPa)	12310	17400	15706	18800	25046
Tensile strain under maximum load	1.827	2.54	2.282	3.66	2.633

4.2.2 Hardness test

Figure 4.6 demonstrates the graphical representation of the composite materials' hardness with different weight percent of fly ash particulates. It is observed that the micro-hardness of the

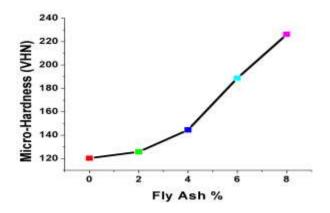


Figure 4.6: Micro-hardness

Composites, with an increase in fly ash weight percentage, increases. The hardness of the composite has increased as a result of the hard ceramic phase's presence in the ductile matrix. The increase in the weight % of the hard and brittle phase of the fly ash particles, which were obtained from TiO₂, Al₂O₃, Fe₂O₃, and SiO₂ of the chemistry made up of the particles, is

ultimately ascribed to these increases. Additionally, the dislocation density at the particle-matrix interfaces is increased by the fly ash particles in the alloy. When the weight fraction of fly ash particle is increased to 8%, the micro hardness of the alloy improves substantially by 88 percent over its base alloy, as shown in the Figure 4.6 and table 4.2.

Table 4.2: Micro-hardness of MMCs

Fly ash %	0	2	4	6	8
Micro hardness	120.3	125.8	144.5	184.8	226.2

4.2.3 Density measurement

Table 4.3 and Figure 4.7 shows density measurements of composites. The density of fly ash particles is often low in nature. In this investigation, precipitator type fly ash having a density of less than 2.2 gm/cm³ was used. The Archimedes method was used to determine the density of the composite specimens. After weighing the little pieces cut from the specimens in air and subsequently in water, the density values were calculated using the following expression as shown in Equation 4.1.

$$\rho = \frac{\text{weight in air}}{(\text{weight in air - weight in water})} \times \rho_{\text{water}}$$

Equation 3.1: Density relation

With the addition of fly ash particles, the density of the Al-fly ash composites dropped linearly. The difference between experimental and theoretical densities is shown to exist. It is as a result of the composite's voids and pores. Additionally, Bhaskar et al. [2017] showed that the addition of fly ash increased porosity and that this increase was less than the predicted density of MMC. The reduced density of fly ash particles in composites can be due to the lower density of

unreinforced Al. According to Mahindra et al. [2007], adding reinforcement lowers density. It is obvious that as the presence of fly ash increases, the porosity of all the composites increases.

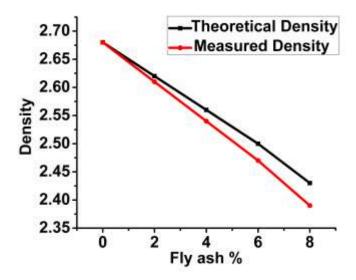


Figure 4.7: Density variation

These findings are consistent with the studies conducted by these scientists (Hanumanth and Irons, [1993]; Kok, [2000]; Ghosh and Ray, [1988]. The following factors contribute to porosity formation:

- (a) Gas entrapment during stirring.
- (b) Air bubbles getting inside the melt-matrix substance.
- (c) The development of hydrogen.
- (d) The variation in reinforcement and matrix size and shape.

The prepared castings are dense and sound, indicating that the interface between matrix and reinforcement was practically perfectly bonded.

Table 4.3: Density of MMC with different wt % of fly ash

Fly Ash	LM6	2	4	6	8
Theoretical density	2.68	2.62	2.56	2.50	2.43
Measured Density	2.68	2.61	2.54	2.47	2.39

4.3 Metallurgical properties

Metallurgical observation of the composite under different weight percentage of the fly ash is carried out with optical microscope and scanning electron microscope for validation with morphology of different constituents' and its significance.

4.3.1 Optical microscope

The surface structure, force, kind of particulate reinforced and availability of the particulates all affect the properties of composite materials. The solidified speed, fluidity, type of reinforcement and technique of incorporation are the factors that control molecule dispersal. The cuboids, the needle, and the irregular polygon are three distinct morphologies that represent the Silicon-rich phase. In some cases, the main -Aluminum phase surrounds the Silicon cuboids. These silicaterich cuboids could constitute the main silicate phase. Pena and Lozano corroborated this outcome [Pena and Lozano, 2006]. They claim that the basic Silicon phase has shape similar to cuboids. Sometimes they act as the principal -Al phase nucleating sites. The irregular polygons display a morphology that is comparable to the initial Silicon phase. The creation of an aluminum dendrite network structure is visible in the microstructure at 2 wt% fly ash and is caused by the composite's super-cooling during solidification. The growing Silicon-rich (black region) phase will become more noticeable when 4 wt% fly ash is added, as shown in Figure 4.9b. In the manufactured AMCs, Silicon needles and Silicon cuboids have accumulated in one location whereas a large area of eutectic aluminum matrix has developed. The Silicon needles started to clump up close to one another. However, the Silicon rich (black region) and aluminum rich regions of the composites' microstructure continue to predominate when fly ash addition of the order of 6 wt% (bright- region). As seen in Figure 4.9c, there were also some dark structures that resembled petals and Silicon-cuboids.

The majority of the structure was dominated by the Silicon-needles, which were evenly dispersed throughout. A smaller portion of the structure was dominated by the eutectic Al matrix. Figure 4.9d, on the other hand, displayed the microstructure of composite reinforced with 8 weight percent of fly ash. Figure 4.9d of microstructure also reveals a region where Silicon polygons and petal-like dark structures are rising, whilst Silicon-needle structures are decreasing.

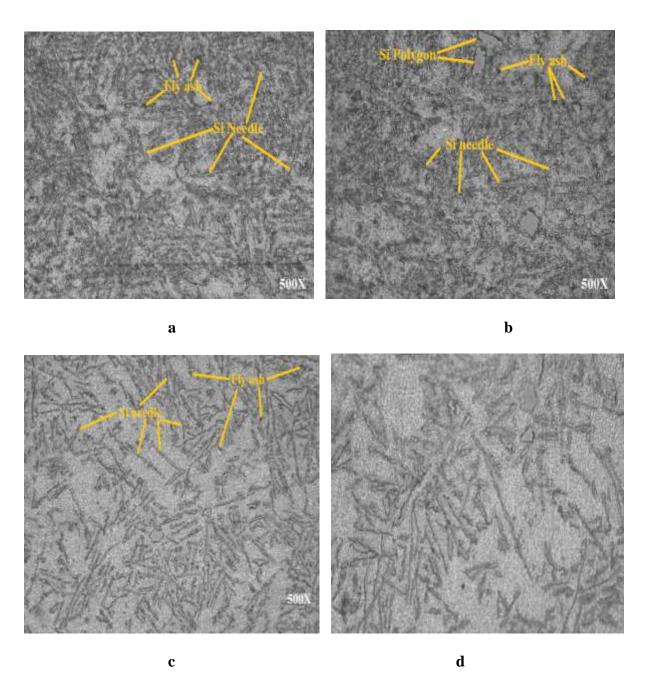


Figure 4.8: Optical images (500X) of composites (a) Al-2% FA (b) Al-4% FA (c) Al-6% FA (d) Al -8% FA

In contrast, the brightness of the eutectic – aluminum matrix has grown. The reason is the uniform distribution of reinforcement throughout the manufactured AMCs. The needle like structure, which lowers the mechanical properties of aluminum silicon alloys, is usually referred

to as sharp eutectic Silicon embedded in the eutectic -aluminum matrix [Pena and Lozano, 2006]. Therefore, it is advantageous that there are less Silicon needles in the structures as fly ash is incorporated at a higher weight percentage since it will improve the characteristics of the manufactured AMCs. It is demonstrated that the microstructure of manufactured AMCs can be improved by the reinforcement of fly ash particles. This is because it is anticipated that adding Si to composites will improve their characteristics [Ervina et.al, 2014]. The particulates are spread similarly all through the fabrication process utilizing wettability during the assembling system and consequently isolation/agglomeration of particulates happens during the pouring system. Magnesium was added to further develop wettability. At different spots, the molecule appropriation of microstructure was inspected. Figure 4.9 portrays the optical microstructure of aluminum alloy combination fly ash composites (a to d). The grain limit was identified later the carving system, and the grounded modules were found before the drawing strategy, in view of these pictures. Due to the evenly distributed fly ash in the metal matrix composites, the optical microscopic image of the samples under analysis reveals a uniform surface pattern.

The conveyance of fly ash particulates is generally homogeneous. It exhibits that the grid stage and reinforcement stage have solid interfacial association. As the level of fly ash builds, some agglomeration happens. Examination on Optical micrographs additionally uncovers the presence of equi-hacked out grains in produced composites. The presence of equi-axed grains demonstrates that fly debris particulates caused recrystallization. In the microstructural examination of MMC, comparable conveyances of particulate matter of fly ash were distinguished. The mechanical and actual properties of composite materials are intensely affected by molecule dispersion.

4.3.2 Scanning Electron Microscope

A FEG QUANTA 250 scanning electron microscope was used to capture the scanning electron micrographs (SEM) as shown in Figure 4.10 (a, b, c & d). To create a mirror-like surface finish for microstructural investigation, a series of grinding and polishing procedures were conducted on the samples' surface. Before the test, the samples were polished with grade 600, grade 1000, and grade 1200 abrasive paper, then etched with Keller's reagent consisting of 95 ml water, 2.5 ml nitric acid (HNO₃), 1.0 ml hydrogen fluoride (HF), 1.5 ml hydrochloric acid (HCl) and swabbed for 10–20 seconds.

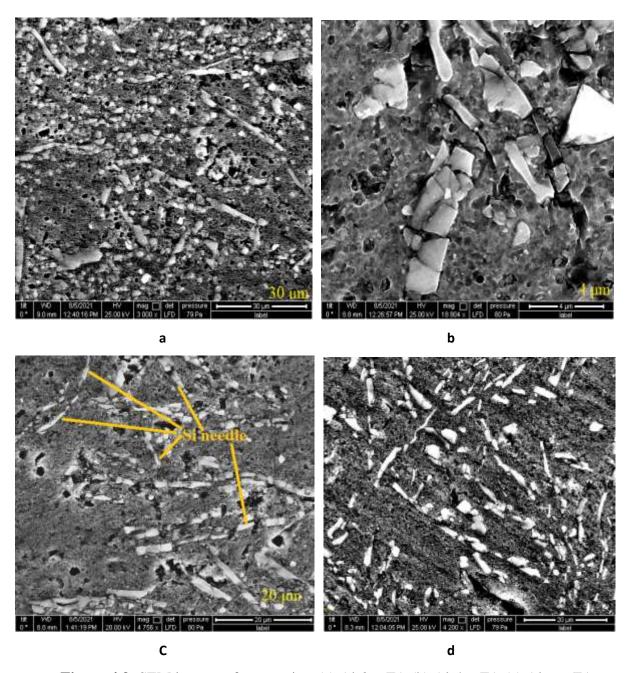


Figure 4.9: SEM images of composites (a) Al-2% FA (b) Al-4% FA (c) Al-6% FA (d) Al -8% FA

Figure 4.9 (a–d) shows SEM micrographs of LM6 alloy composites augmented with fly ash. The reinforced fly ash precipitator particles are very evenly distributed in the matrix, as shown in Figure 4.9 (b - d). The morphology of the precipitator integrated matrix is seen via SEM thermionic emission in Figure 4.9 (b). Precipitator inside the Al matrix can be seen in secondary electron mode micrographs. The micrograph confirms the uniform distribution of fly ash inside

the Al matrix. The fly ash particles are uniformly dispersed in the matrix, with no voids or flaws visible in the micrographs of LM6 – fly ash composites. In the low percentage content, there was an excellent dispersion of matrix material and fly ash particles, with very little agglomeration of fly ash. The difficulties of fly ash particle agglomeration and flaws, on the other hand, rapidly grow as the fly ash content rises. Initial magnification of 500 times reveals a homogeneous distribution of silicon carbide (SiO₂) and aluminum oxide (Al₂O₃) in the fly ash. So the microstructure seems gleaming, there are no pores in the form of dark patches, and there is only a blunt edge after failure, indicating that the material is elastic. However, as the level of reinforcement added to the composite increases, the shiny nature of LM6 decreases. Because fly ash provides a dark grey nature to the microstructure, blunt ends become sharp edges, and the number of dark pores formed increases, as shown in Figure 4.9, implying an increase in the brittle nature of the MMCs.

4.4 XRD

It is a noble, non-destructive technique for chemical analysis. In a mixture of substances, each substance produces its own pattern independently of the others, the same substance always produces the same pattern, and every crystalline substance produces a pattern. It establishes the direction of a single crystal as well as the typical spacing between atom layers or rows, the crystal structure of an unidentified substance, and the size, shape and internal stresses of small Crystalline regions. XRD analysis, which was utilized to confirm the mineralogical constituents, revealed a mineralogical synthesis that included X-ray diffraction samples of fly ash particles, alumina Al₂O₃, silica SiO₂, and hematite Fe₂O₃. The distinctive pinnacles of the fly debris are the pinnacles that are addressed with mill operator data. The minute difference in peaks matching to crystallite size and preparation processes. The figure shows that all of the peaks are identical. Different pinnacles could be due to a different era of any precursor materials or contaminants, or they could be due to any form of halfway point. X-ray diffraction was used to study composite materials to see if there were any special blends.

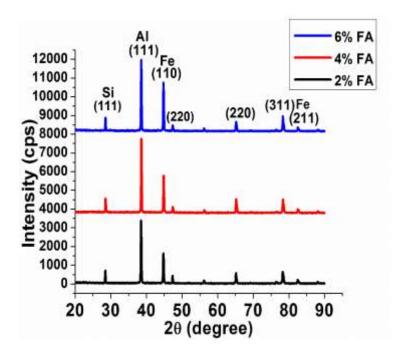


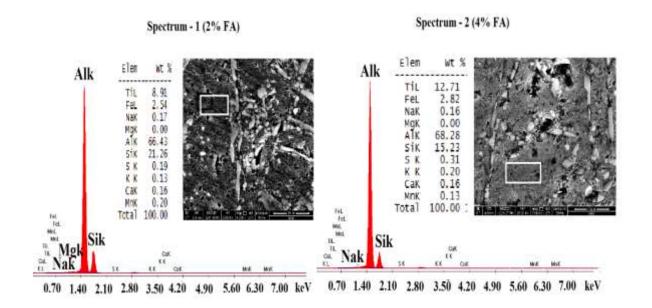
Figure 4.10: XRD Plot

4.5 EDS analysis

The following are the uses of EDS, a chemical method used in conjunction with SEM:

- To identify the elements
- To find out the contents of each material present in the composite
- to ascertain the homogeneity and the distribution of its elements in the synthesized structure

The weight percentage or atomic percentages of the EDS results are shown in Figure 4.12. It shows the elemental composition and its peaks i.e. Al, Si, Mg, Mn for different constituents in the composite. The data produced by EDS analysis consists of spectra with peaks matching to the elements of the sample's actual composition. EDS analysis is used to confirm the compositional features of composites. Figure 4.12 shows the EDS result. In order to confirm the fly ash composition, An EDS analysis was done. Different locations were focused on during the EDS analysis and the relevant peaks are shown in Figure 4.11 (a, b, c, d).



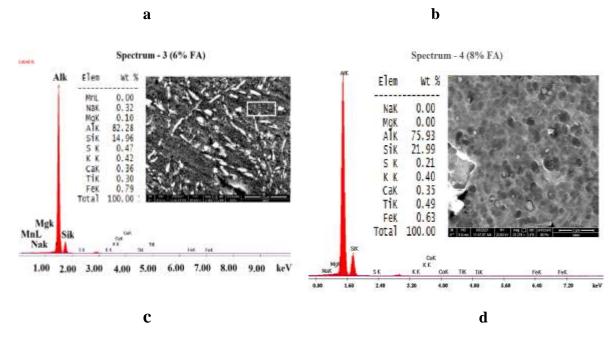


Figure 4.11: EDS Spectrum (a) 2% FA (b) 4% FA (c) 6% FA (d) 8% FA

Fly ash contents and composition of alloy i.e. Al, Si, Mg, Na, K etc are available in the generated composite structure in the EDS spectrum. S, K, and Ca were present in spectrums 1 and 2, respectively, in amounts of 0.19, 0.13, and 016 and 0.31, 0.20, and 0.16. However, the values in spectrum 3 were 0.47, 0.42, 0.36. Details of four EDS spectra of the composite in weight % are seen in Figure 4.11 (a, b, c, d).

EDS examination demonstrates that fly ash particles are present in all composite types. The EDS investigation merely verifies that the weight % of fly ash components has gradually grown with greater fly ash particle assimilation. All of these EDS layouts contain elements of the LM6 alloy. However, because the particle size is at the micron level, the exact weight % of each element does not match the EDS. The current findings, on the other hand, essentially indicate that aluminum matrix contains fly ash particles.

4.6 Tribological studies

Wear in the current experiment was caused by contact between two surfaces, one moving and the other immovable. A hardened roller steel disc serves as the moving surface, while a "block-of-sample" serves as the fixed surface. A consistent load was applied to the fixed block of sample as it was pressed against the moving roller disc. Because the steel disc in this investigation has a far higher hardness than the composite specimen, there was very little disc wear. The roller disc's contact area is significantly larger than the sample block's area. The following factors were used to analyse the wear characteristics of composite samples.

The experimental variables were

- (i) weight fraction of fly ash reinforcement
- (ii) applied load
- (iii) sliding speed
- (iv) duration

4.6.1 Wear measurement

Influence of weight fraction of fly ash, applied load and Speed on Wear rate

Figures 4.12 (a, b, c) depict the wear of composites in micron measured at 25 N, 50 N and 75 N of load separately for a sliding speed of a range of 400, 500, and 600 rpm under different weight fraction of fly ash. Figure 4.13 (a, b, c) describes the wear in micron for a sliding speed of a range of 400, 500, and 600 rpm for 25 N, 50 N, and 75 N load under varied fly ash weight percentages. Figure 4.12 illustrates the degree of wear is more for 2 % fly ash and less for 6 % fly ash initially, thereafter as the load or speed grew, the wear increases simultaneously for all

samples. As there is an increase in fly ash components, wear increases. The rate of growing wear is a function of temperature for a constant amount of fly ash because the tougher fly ash components resist dislocation motion. The roller softens as there is increase in temperature due to prolonged running of slider. The fly ash particles pull out of the matrix as a result of the aluminum alloy LM6 softening because the interfacial stress is greater than the bonding strength between the matrixes and reinforced. These fly ash particles are encircled by the roller surface of the tougher counter face, where they are crushed into tiny particles. This is an agreement with studies of some researchers Basavarajappa et al., [2006]; Sahin & Ozdin, et al. [2008]. A similar observation was examined by Alpas et.al., [1992)] and Moustafa, et al. [1995]. The variance in wear with varied weight loads percent at various sliding speeds can be further explained by two factors. First, the abrasive, which initially comes into contact with the matrix and is less hard than the angular silica sand (abrasive) particles, has a lower hardness. Material removal is exceptionally high at that specific time because the ratio of Ha (refers to abrasive particle)/Hs (refers to surface) is substantially higher than unity, that causes matrix damage. The specific wear rate is higher as a result. Fly ash particles give superior resistance to the abrasion process with reduced the wear rate when the load increases because they come in contact with abrasive particles when the Ha/Hs ratio is greater than unity. Second one: the wear rate is decreased by the work hardening of the pin contact surface caused by the continuous rise in load, similar to the outcomes found by Rohatgi et al. [1997], Rohatgi and Guo [1997], Rohatgi et al. [2009], and Rajan et al. [2007].

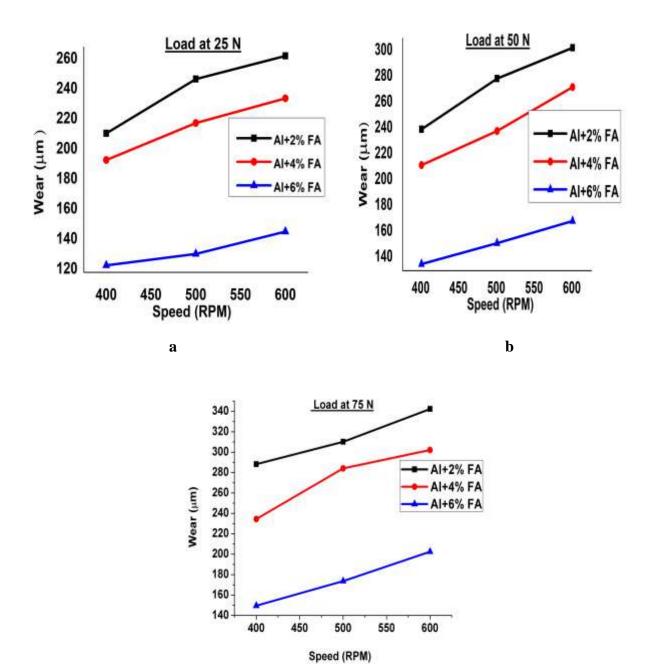


Figure 4.12: Wear at different loads

 \mathbf{c}

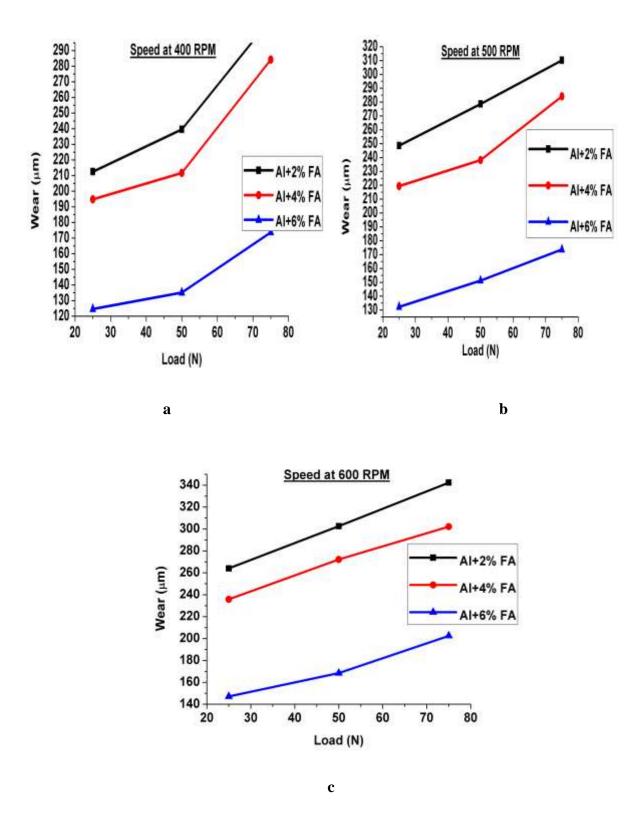
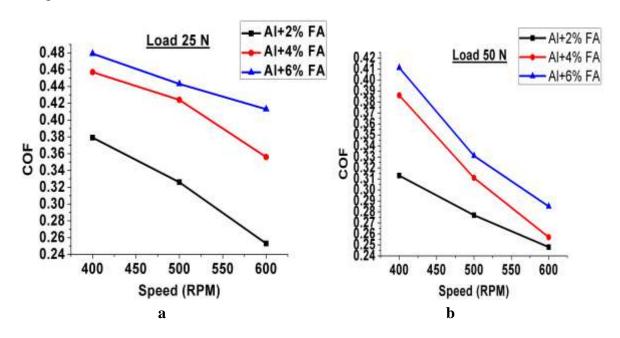


Figure 4.13: Wear at different speeds

4.6.2 Friction measurement

Effect of reinforcement weight percent of load & sliding speed on COF

The COF of the Al-Fly ash matrix alloy and composites varies with different loads (25N, 50N, 75N) at 600 rpm and speeds (400 rpm, 500 rpm, 600 rpm) at 25 N, as shown in Figures 4.14 (a, b, c) and 4.15 (a, b, c). The COF of the matrix alloy and composites reduced as the load was increased. As the Fly ash weight percent increases, the friction coefficient drops. The rise in temperature of the contact surface rises and softens the surface of the roller, resulting in a drop in COF as the load increases. As a result, the friction coefficient falls. Another cause could be that as the load increases, the roller surface wears out more, and the wear debris becomes lodged between the roller and the counter surface, acting as a roller ball. As a result, the COF falls. This is in agreement with the work of some researchers Rohatgi et al. [1997]; Rohatgi and Guo [1997], Rana et al., [1989], Komvopoulos et al. (1985). Further it can be added that at lower loads the sliding wear increases. However with the increase in fly ash and load, wear increases and friction coefficient decrease. As the sliding speed increases, it was also observed that the composites are mildly worn. The outcomes were connected to and validated by tests carried out by numerous studies. [Ramachandra and Radhakrishna 2007], [Shanmughasundaram et. al. 2011].



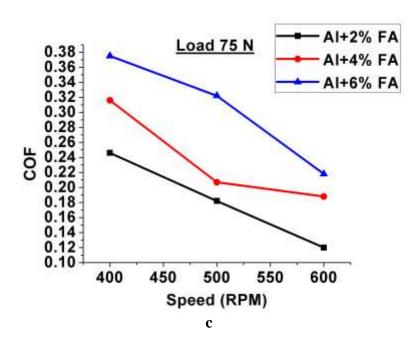
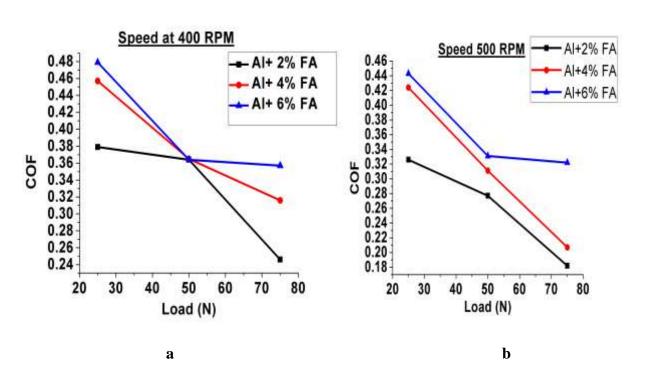


Figure 4.14: COF at different loads



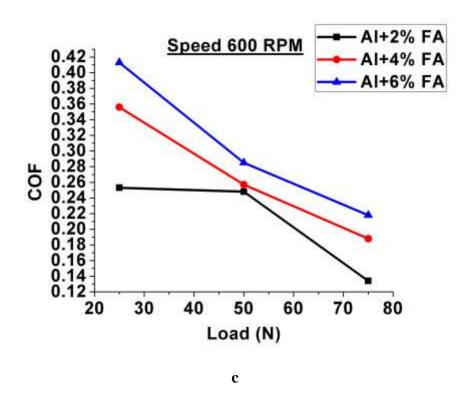


Figure 4.15: COF at different speeds

4.7 Statistical analysis through Taguchi method

Al-fly ash composites' wear rate and coefficient of friction (COF) are statistically analyzed using S/N analysis and analysis of variance studies. S-N ratio analysis may examine the variability of experimental results under different working conditions, hence Taguchi advised employing it rather than only taking the average of the experimental data to achieve optimality. The wear rate and COF were therefore analyzed in this study using the S/N ratio, with a smaller value being a better criterion. S/N ratio is calculated based on the following formula:

$$\frac{S}{N} = -10\log(\frac{1}{n}\sum_{i=0}^{n} Yi^2)$$

Where, n denotes the number of experiments and y denotes the data that were observed.

Equation 4.1: Taguchi's Formula

4.7.1 Wear Analysis

The various design variables and their levels are displayed in Table 4.4. The experimental data that predicts the delta value and S/N ratios based on smaller is the better, are displayed in Table 4.5. It also demonstrates that the strongest factor influencing how quickly Al-fly ash composites wear out is reinforcing. Whereas load has a moderate impact and speed has the least impact.

Taguchi Analysis: Wear versus Wt.%, Load, Speed

Table 4.4: Design factors and their levels

Factor	Type	Levels	Values
Wt.%	fixed	3	2, 4, 6
Load (N)	fixed	3	25, 50, 75
Speed (rpm)	fixed	3	400, 500, 600

Table 4.5 Ratios of Signal to Noise

Level	Wt.%	Load	Speed
1	-48.75	-45.63	-45.67
2	-47.65	-46.62	-46.77
3	-43.65	-47.80	-47.61
Delta	5.10	2.17	1.94
Rank	1	2	3

Examining the effects of input variables on output variables involves using the main effect plot of S/N ratios and the interaction plot. The main effect graphic shows the process in its ideal state. The slope of the curve in a main effect plot can be used to forecast how the parameter will affect

the data. The parameter with the sharpest slope has the greatest impact, whereas the parameter with the horizontal curve has the least. Figures 4.16 and 4.17, respectively, display the study's main effect plot and interaction plot.

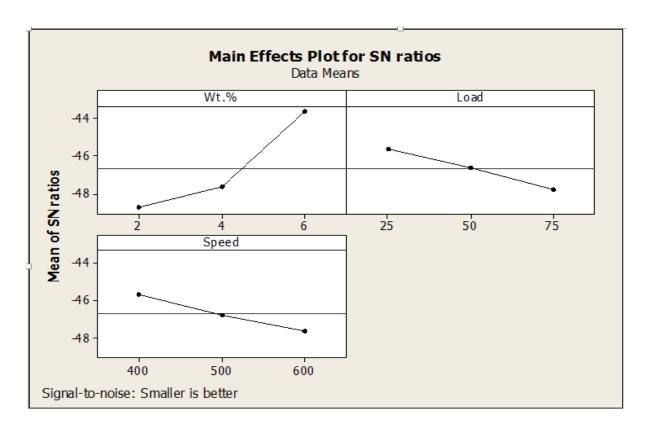


Figure 4.16: A wear rate main effect plot

In a major effect plot, the wt% of fly ash has the steepest inclination, whereas the other two variables, load and speed, have a mild slope. As a result, reinforcement has the greatest impact on composite wear rate, whereas the other two have a moderate to minor impact. The level 3 fly ash weight percentage and level 1 value for the other two variables had the highest S/N ratios. A3B1C1 will therefore be the preferred optimality for this inquiry. In an interaction plot, non-parallelism indicates that there is interaction between the input parameters, but an intersecting line denotes that there is significant interaction. Figure 4.17 for the current experiment displays the wear rate interaction plot. The graph shows a moderate level of factor interaction. An ANOVA (Table 4.5) analysis demonstrates the importance of specific input variables, their interactions, which primarily determine the variance level of experimental data.

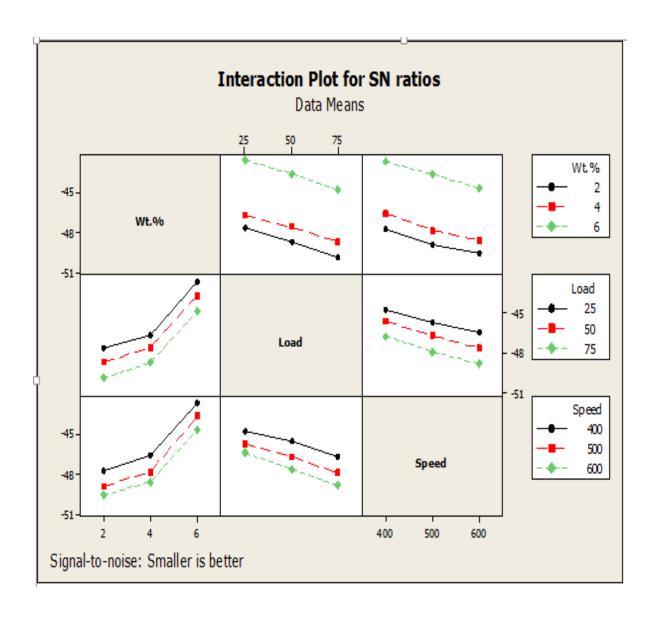


Figure 4.17: Wear rate interaction plot

The table 4.6 displays all of the parameters' F-ratios, P-values, degrees of freedom and interactions. [Banerjee et al. 2019] reported similar observation. When assessing the wear rate of composites, the wt percent has the greatest impact, followed by the two other variables, load and speed. However, no interaction significantly affects the rate at which composites wear out. The wear rate's S/N ratio residual plots are shown in Figure 4.18. Residual plot for SNRA1 describes about a normal probability plot, versus fit, histogram and versus plot. Probability plot depicts the experimental data distribution, and the data sets checked for normality.

Table 4.6: Using Adjusted SS for Tests, Analysis of Variance for SNRA1

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Wt.%	2	129.5847	129.5847	64.7923	848.9	0.000
Load	2	21.3063	21.3063	10.6532	139.58	8 0.000
Speed	2	17.0055	17.0055	8.5028	111.40	0.000
Wt.%*Load	4	0.0679	0.0679	0.0170	0.22	0.918
Load*Speed	4	0.1795	0.1795	0.0449	0.59	0.681
Wt.%*Speed	4	0.1517	0.1517	0.0379	0.50	0.739
Error	8	0.6106	0.6106	0.0763		
Total	26	168.9063				

S = 0.276268 R-Sq = 99.64% R-Sq(adj) = 98.83%

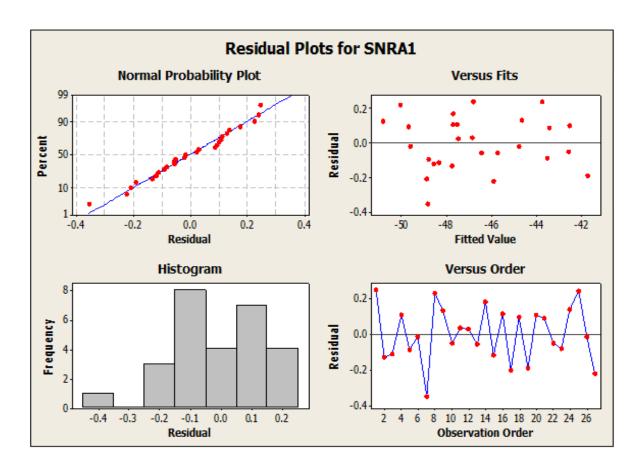
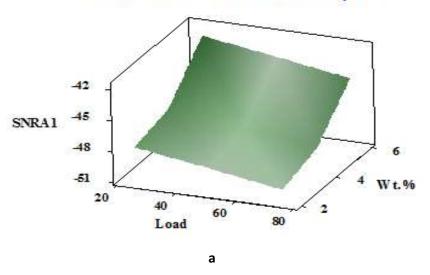


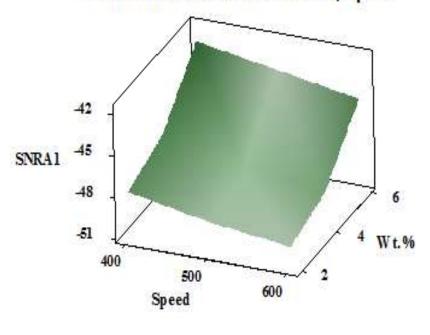
Figure 4.18: Residual plot for wear rate

A straight line is formed from the normal probability plot that shows the model adequate in the current study practically forms. In the case of other plots, the observed data are dispersed with regard to the residuals. The observation order is plotted on the x-axis and the residuals are plotted on the y-axis. The versus order graphic shows that there is no relationship between the observation data sets and their residuals. Figure 4.19 (a, b, c)'s surface plots of SNRA1 versus the input variables confirm each of the afore mentioned observations.

Surface Plot of SN RA1 vs Wt.%, Load



Surface Plot of SN RA1 vs Wt.%, Speed



b

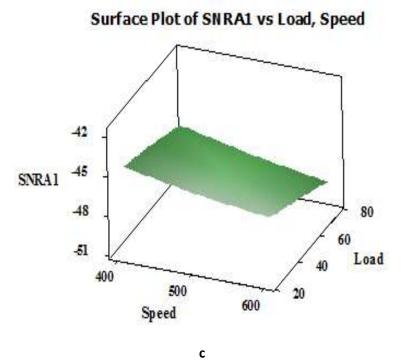


Figure 4.19: Surface plots of parameters with SNRA1

4.7.2 COF analysis

Table 4.7 shows the different design factors and their levels. Table 4.8 shows the experimental data as well as the S/N ratios. It also reveals that load has the greatest influence in limiting COF of Al – fly ash composites, whereas speed has a moderate impact and reinforcement has the least impact.

Taguchi Analysis: COF versus Wt.%, Load, Speed

Table 4.7: Signal to Noise Ratio Response Table

Level	Wt.%	Load (N)	Speed (rpm)
1	11.896	8.275	8.852
2	10.493	10.764	10.361
3	9.244	12.594	12.421
Delta	2.653	4.320	3.569
Rank	3	1	2

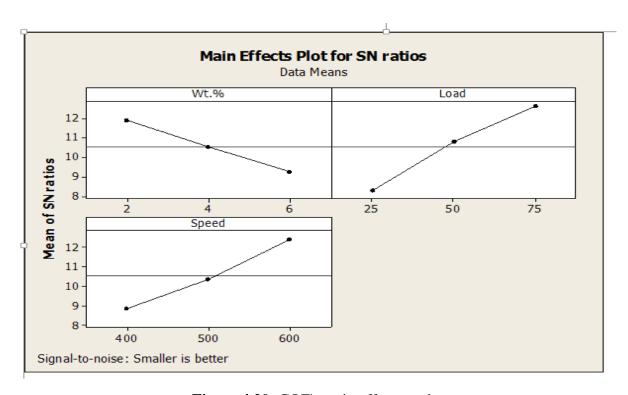


Figure 4.20: COF's main effect graph

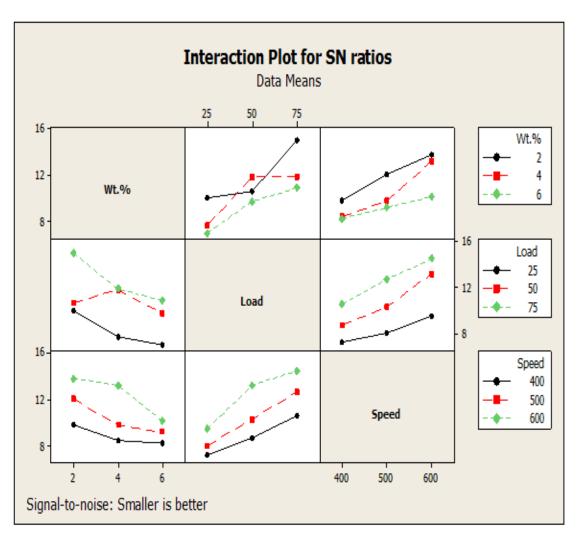


Figure 4.21: COF's Interaction plot

In a major effect plot of COF, the load and speed has the steepest inclination, followed by wt% with mild slope. As a result, reinforcement has the least impact and load, speed have greater influence on composite COF. The highest S/N ratio is found from the response table 4.7 at level 3 for given fly ash and for the other two variables at level 1. As a result, A3B1C1 will be the desired optimality for this investigation too. The interaction plot for COF is shown in Figure 4.21 for the current investigation. The graph depicts a moderate interaction between the factors. An ANOVA (Table 4.8) analysis demonstrates the importance of specific input variables and their interactions that primarily determine the variance of experimentally obtained data. The table 4.8 displays all of the parameters' F-ratios, P-values, degrees of freedom, and interactions. The wt

percent has the biggest effect, followed by the other two factors of load and speed in determining the COF of composites. However, no interaction has a major impact on the COF of composites.

Table 4.8: Using adjusted SS for tests, an analysis of variance for SNRA1.

Source	DF	SeqSS	Adj SS	Adj MS	F	P
Wt.%	2	31.700	31.700	15.850	15.30	0.002
Load	2	84.622	84.622	42.311	40.85	0.000
Speed	2	57.785	57.785	28.892	27.90	0.000
Wt.%*Load	4	16.444	16.444	4.111	3.97	0.046
Load*Speed	4	4.420	4.420	1.105	1.07	0.433
Wt.%*Speed	4	8.617	8.617	2.154	2.08	0.175
Error	8	8.285	8.285	1.036		
Total	26	211.873				

S = 1.01768 R-Sq = 96.09% R-Sq(adj) = 87.29%

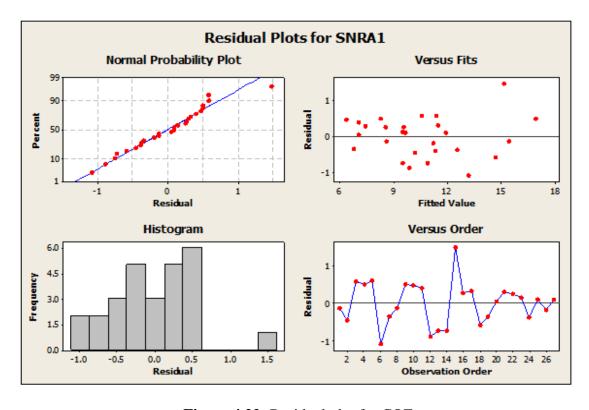
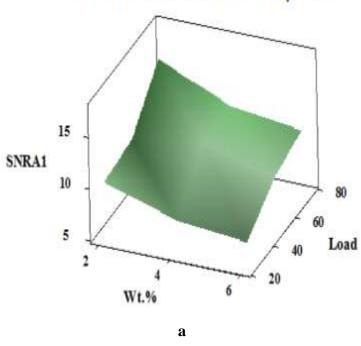


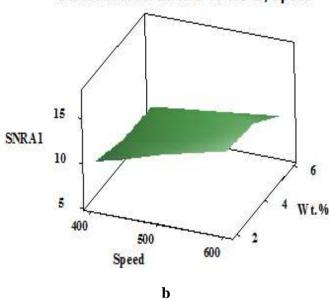
Figure 4.22: Residual plot for COF

Figure 4.22 shows residual plots for the S/N ratio of COF. Surface plots of SNRA1 vs. input variables, as shown in Figure 4.23 (a, b, c), verify all of the preceding observations.

Surface Plot of SNRA1 vs Load, Wt.%



Surface Plot of SN RA1 vs Wt.%, Speed



Surface Plot of SN RA1 vs Load, Speed

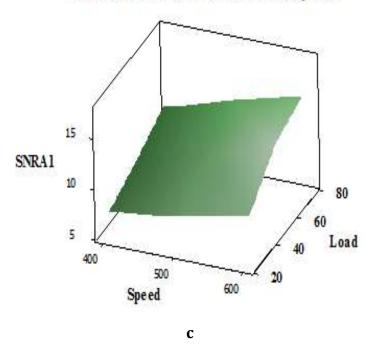


Figure 4.23: Surface plots of parameters with SNRA1

4.8 Conclusion

This chapter provides thorough description of complete research work carried out. Firstly it investigates the mechanical properties of the composite under four different condition of weight percentage. Secondly it analyses the optical microstructure and SEM micrograph. Thirdly Composites mineralogical constituent and their phase alignment is measured in XRD. Fourth, EDS analysis is carried out to find the materials present in the composite, its homogeneity and distribution. Fifth, design of experiment is carried out to find the optimality among the variables in wear analysis and COF measurement.

Chapter 5

Summary of Findings

5.Summary of findings 5.1 Conclusion 5.2 Future Scope of work

5.1 Conclusion

In the present work, stir casting method has been used to successfully produce LM6 and fly ash reinforced composites with different reinforcement weight percentages. Investigations have been made into microstructural characterization, mechanical characteristics, and tribological behavior under dry sliding conditions. For desired optimality in wear rate and COF analysis, a statistical analysis for tribological parameters using the Taguchi technique is also conducted. In order to create a lightweight material that has improved properties and can take the place of the current material, the major goal of this effort is to correlate changes in the microstructure and properties brought on by different reinforcing. The outcomes of the trials can be used to draw the following conclusion:

- Thermal power plant waste, fly ash can be utilized effectively as filler/reinforcement to create light weight composite. As a result, using fly ash to make composites can transform industrial waste into economical worth.
- In this work, composites made of Al-Si alloy (LM6) and fly ash are fabricated by bottom
 poured stir casting technique. Studies using SEM, EDS and XRD are employed to
 describe the composite.
- A fly ash addition to an Al alloy (LM6) results in a 7.5% reduction in the density of the composite.
- As the weight level of the fly ash particle increases, the mechanical properties of the composites are also enhanced.
- Uniform distribution of fly ash throughout the aluminium alloy matrix increase the composite's toughness. This will happen as a result of a noticeable increase in strength

brought on an increase in the surface area between the fly ash particles and the matrix material.

- Hardness and wear resistance directly correlate with an increase in fly ash weight percentage. Fly ash addition increases the wear resistance and the hardness of the composite by limiting matrix material deformation.
- The optical microscope revealed that the fly ash particles in the AMCs were evenly distributed. The wettability of a semi-solid aluminium alloy was increased by the addition of fly ash particles. On the other hand, micro hardness of the composites increases 88% over its base alloy with 8% of fly ash. As the fly ash percentage gradually increases, the resulting effect of the mechanical properties intensifies even further.
- The matrix or composites don't show any voids or discontinuities, and a look at the microstructures shows that the fly ash particles are distributed uniformly.
- While the SEM images of composite with 8% fly ash showed a dispersed, broken oxide layer in the Al matrix, an image of pure Al alloy showed a distributed, cracked oxide layer in the Al matrix. Fly ash had homogeneous granules and narrow grain boundaries. Successful casting was achieved by adding the proper amount of fly ash to the mixture, which increased wettability throughout the casting process.
- Fly ash particles were equally distributed throughout the aluminum matrix, according to optical and scanning electron micrographs. The XRD data showed that only very slight alterations in component composition are taking place. The 2 and 4% fly ash composite samples show a more uniform distribution than the other samples because there is less segregation during solidification. It is obvious that better mechanical behavior results from increased particle dispersion uniformity. There is therefore little question that this material's usage in the automobile and space industries will be practical in the future.
- The availability of mineralogical ingredients at various weight fractions of the composite was shown by XRD. The graph demonstrates that each peak is identical.
- To determine the homogeneity and elemental distribution in the synthesized structure, EDS is used in conjunction with SEM to identify the elements contained in the

composite. For various constituents in the composite, the EDS result displays the elemental composition and its peaks, namely Al, Si, Mg and Mn. EDS research supports the existence of fly ash particles in all composite types.

- The coefficient of friction reduces as the amount of fly ash particles in composite materials rises. The wear volume of the composites was affected by the applied load, the amount of fly ash, and the sliding speed. Because fly ash comprises tougher particles, its content decreases as it gets higher.
- In order to lower the composite wear rate and COF, the Taguchi approach is utilized to perform a tribological study and optimize the stated input parameters, such as sliding speed, applied load, and weight percent of reinforcement. An analysis of variance (ANOVA) is used to determine the significance of the input variables and their interactions. The following conclusions can be taken from this study:
 - The weight fraction of the reinforcement is the variable that has the greatest influence, according to the main effect plot. The other two, however, have the least impact. As a result, based on the S/N ratio discovered, A3B1C1 will be the required optimality for minimizing wear rate from this experiment.
 - In a similar way, the main effect plot shows that the applied load is the factor that influences COF consideration the most. The impact of the other two parameters is minimal. As a result, based on the measured S/N ratio, A1B3C1 will be the desired optimality for minimizing COF from this experiment.
 - As a result, ANOVA analysis reveals that the applied load and weight percent of reinforcement are the two most important parameters for the COF and wear rate analyses, respectively. The final two factors are still only marginally important for optimality.

5.2 Future Scope of work

Following recommendations can be made for the expansion of current work with aluminium matrix and fly ash composite on the basis of the current investigation. The following areas may be the focus of more research:

- 1. To examine the impact of nanoparticles on the mechanical and tribological properties of the LM6 fly ash composite, the size of the particulate reinforcement can be adjusted from micro to nano.
- 2. The tribological behaviour for dry sliding circumstances is explored in this work. Future research can be conducted on the same compositions for abrasive, slurry, erosive, and corrosive/stress corrosive wear.
- 3. To determine the corrosion rate, corrosion experiments can be carried out in salt and fresh water solutions at various pH levels and temperatures. It is also possible to research how thermally treated fly ash affects the rate at which metal matrix composites corrode.
- 4. Investigations will be conducted utilising additional fabrication techniques, such as squeeze casting and powder metallurgy, to determine the volume of reinforcement for thermally treated fly ash in different particle sizes.
- 5. The mechanical characteristics, wear, and microstructural behaviour of AMCs are examined in the current work. Future studies may examine mechanical qualities including fatigue strength, creep, and shear strength as well as thermal behaviour, such as thermal conductivity, coefficients of thermal expansion, specific heat, and thermal stress.
- 6. Endless to investigate the stress distribution in the constructed composite for various percentage compositions of reinforcement, element models may be devised. Another area of research that might be examined to understand how these materials behave during solidification is computer simulation and modelling.
- 7. Non-destructive testing (Radiography) can be used to see whether there are any pores that could cause the specimen's strength to decrease.

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