

**INFLUENCE OF HEAT TREATMENT ON
ELECTRO-MECHANICAL PROPERTIES OF
ALUMINUM COMPOSITES**

MD JALAL UDDIN RUMI

M.SC. ENGINEERING THESIS



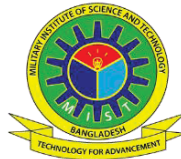
**DEPARTMENT OF AERONAUTICAL ENGINEERING
MILITARY INSTITUTE OF SCIENCE AND
TECHNOLOGY
DHAKA, BANGLADESH**

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MD JALAL UDDIN RUMI (STUDENT ID. 0420220005)

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of
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DECLARATION

I hereby declare that the study reported in this thesis entitled above is my own original work and has not been submitted anywhere for any degree or other purpose. Further, I certify that the intellectual content of this thesis is the product of my own work and that all the assistance received in preparing this thesis and sources have been acknowledged and/or cited in the reference section.

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INFLUENCE OF HEAT TREATMENT ON ELECTRO-MECHANICAL
PROPERTIES OF ALUMINUM COMPOSITES

A Thesis

By

Md Jalal Uddin Rumi

DEDICATION

Dedicated to my Late supervisor Dr. M Muzibur Rahman for his support and
encouraging me to be the best I can be.

ABSTRACT

INFLUENCE OF HEAT TREATMENT ON ELECTRO-MECHANICAL PROPERTIES OF ALUMINUM COMPOSITES

This study presents a detailed exploration of aluminum-based composites reinforced with Al_2O_3 and ZnO nanoparticles, employing a customized stir method, with a primary emphasis on investigating their electro-mechanical properties. Two distinct composites were developed: Al MMC-01 (97.5 wt. % Al, 2.5 wt. % Al_2O_3) and Al MMC-02 (95 wt. % Al, 2.5 wt. % Al_2O_3 , 2.5 wt. % ZnO).

Microstructure analysis through SEM affirmed the uniform dispersion of Al_2O_3 and ZnO within the metal matrix composites. The addition of 2.5% Al_2O_3 notably enhanced the hardness, flexural strength, and impact toughness of the Al composite compared to pure Al. However, Al MMC-02, with an additional 2.5 wt. % ZnO and 2.5 wt. % Al_2O_3 , exhibited increased Vickers microhardness but decreased impact strength, flexural strength, flexural modulus, and electrical conductivity compared to Al MMC-01. SEM fractured surface analysis revealed the brittle nature of Al MMC-02, characterized by cleavage cracks, deep shear dimples, and crystallographic planes.

Subsequent heat treatment at solution temperatures of 510°C, 530°C, and 550°C, coupled with thermal aging between 140°C and 220°C, aimed to enhance hardness and electrical conductivity. Results revealed peak increases in Vickers microhardness (25.92% for Al MMC-01, 17.6% for Al MMC-02) and electrical conductivity (9.57% for Al MMC-01, 12.12% for Al MMC-02) at a common solution temperature of 530°C compared to the as-cast state. The study developed two non-linear mathematical models using a central composite design to predict heat treatment effects on Vickers microhardness (HV) and electrical conductivity (%IACS), achieving R^2 values of 89.29% and 91.50%, respectively.

Characterizing specific wear rates for Al MMC-01, considering parameters like applied load, sliding speed, and duration using Taguchi's Technique, identified applied load as the most impactful factor on the specific wear rate. The study developed a highly accurate regression equation to predict specific wear rates, yielding R^2 and adj R^2 values of 99.85% and 99.76%, respectively. Two confirmation experiments demonstrated minimal errors between experimental and predicted values (2.1% and 6.6%).

INFLUENCE OF HEAT TREATMENT ON ELECTRO-MECHANICAL PROPERTIES OF ALUMINUM COMPOSITES

বর্তমান অধ্যয়ন একটি কাস্টমাইজড স্টিয়ারিং কাস্টিং পদ্ধতি এ Al_2O_3 এবং ZnO সংযুক্ত অ্যালুমিনিয়াম-ভিত্তিক কম্পোজিট তৈরি করে তাদের ইলেক্ট্রো-যান্ত্রিক বৈশিষ্ট্যগুলির তদন্ত প্রতিবেদন করেছে। দুই ধরনের অ্যালুমিনিয়াম কম্পোজিট তৈরি করা হয়েছে, (i) Al MMC-01 যার ৯৭.৫% অ্যালুমিনিয়াম এবং ২.৫% Al_2O_3 এবং (ii) Al MMC-02 যার ৯৫% অ্যালুমিনিয়াম, ২.৫% Al_2O_3 এবং ২.৫% ZnO আছে।

SEM-তে পর্যবেক্ষণ করা Al MMC-1 এবং Al MMC-2 -এর মাইক্রোস্ট্রাকচারগুলি ধাতব ম্যাট্রিক্স কম্পোজিটগুলিতে Al_2O_3 এবং ZnO -এর অভিন্ন বন্টনকে নিশ্চিত করেছে। এটা লক্ষ্য করা গেছে যে ২.৫% Al_2O_3 যোগ করার ফলে পিওর অ্যালুমিনিয়াম তুলনায় Al MMC-01 এর Hardness, Flexural Strength, and Impact Toughness উল্লেখযোগ্যভাবে উন্নত হয়েছে। উপরন্তু, Al MMC-02 -এর Hardness উল্লেখযোগ্যভাবে বৃদ্ধি পেয়েছে। যাইহোক, MMC-02 এর Impact Strength, Flexural Strength, Flexural Modulus and Electrical Conductivity Al MMC-01-এর তুলনায় হ্রাস পেয়েছে কারণ এটি প্রকৃতিতে ভঙ্গুর হয়ে যায়। SEM ভাঙ্গার পৃষ্ঠা বিশ্লেষণ করলে দেখা গিয়েছে যে, Al MMC-02 এর ক্লিভেজ ফাটল, গভীর শিয়ার ডিম্পল এবং ক্রিস্টালোগ্রাফিক প্লেনের উপস্থিতির Al MMC-01 এর তুলনায় অধিক প্রকাশ করা হয়েছে।

অ্যালুমিনিয়াম কম্পোজিট এর ইলেক্ট্রো-যান্ত্রিক বৈশিষ্ট্যগুলির আরও উন্নতির জন্য $550^{\circ}C$, $570^{\circ}C$, এবং $590^{\circ}C$ এর সল্যুশন তাপমাত্রায় এবং $180^{\circ}C$, $160^{\circ}C$, $140^{\circ}C$, $200^{\circ}C$, এবং $220^{\circ}C$ এর থার্মাল এজিং তাপমাত্রায় হিট ট্রিটমেন্ট করা হয়েছিল। বর্তমান তদন্ত অনুসারে, আমরা Al MMC-01 -এর জন্য Vickers micro hardness-এর সর্বোচ্চ বৃদ্ধি ২৫.৯২% এবং Al MMC-02 -এর জন্য ১৭.৬%, Electrical Conductivity Al MMC-01 -এর জন্য ৯.৫৭% এবং Al MMC-02 -এর জন্য ১২.১২% একই সল্যুশন তাপমাত্রায়, $570^{\circ}C$ ঢালাই অবস্থার ফলাফলের তুলনায় বৃদ্ধি লক্ষ্য হয়েছে। Vickers micro hardness (HV) এবং Electrical Conductivity (%IACS) এর উপর হিট ট্রিটমেন্ট প্রভাবের প্রেডিকশন এর জন্য নন-লিনিয়ার ম্যাথমেটিক্যাল মডেল যেখানে HV এবং %IACS এর জন্য R^2 মান ছিল ৮৯.২৯% এবং ৯১.৫০% যথাক্রমে ৯৫% আত্মবিশ্বাসের স্তর এবং ৫% তাৎপর্য স্তরের সাথে। রিগ্রেশন সমীকরণ অনুসারে, তাপ চিকিত্সার অপ্টিমাইজ করা প্রক্রিয়া ভেরিয়েবল যেমন দ্রবণ তাপমাত্রা এবং বার্ষিক তাপমাত্রা হল $571.82^{\circ}C$ এবং $140^{\circ}C$ ।

২.৫% Al_2O_3 সংযুক্ত অ্যালুমিনিয়াম কম্পোজিট Al MMC-01 এর Specific Wear Rates চরিত্রায়ন করেছি। Taguchi এর টেকনিক ব্যবহার করে বিভিন্ন প্রক্রিয়া যেমন Applied load, Sliding speed, and Duration। Taguchi এর Design of experiment (DoE) এবং Analysis of Variance (ANOVA) অনুযায়ী, সবচেয়ে কার্যকরী প্যারামিটার হল Applied load যা অ্যালুমিনিয়াম কম্পোজিটের Specific Wear Rates উল্লেখযোগ্যভাবে প্রভাবিত করে। অ্যালুমিনিয়াম কম্পোজিটের Specific Wear Rates অনুমান করার জন্য একটি রিগ্রেশন সমীকরণ সহ একটি মডেল তৈরি করা হয়েছে যেখানে R^2 এবং adj R^2 যথাক্রমে ৯৯.৮৫% এবং ৯৯.৭৬%। পরীক্ষামূলক এবং পূর্বাভাসিত মানগুলির মধ্যে যথাক্রমে ২.১% এবং ৬.৬% এর ত্রুটি সীমা সহ দুটি নিশ্চিতকরণ পরীক্ষা করা হয়েছিল।

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Chapter 01

INTRODUCTION

1.1 Background of the Study

Aluminum (Al) composites are the most preferred metals used in aircraft construction due to their excellent combination of properties, including a high strength-to-weight ratio, good wear resistance, and enormous availability (Malaki, 2021). However, their low ductility and toughness can limit their use in some applications.

To address these limitations, researchers have explored various methods to enhance the properties of Al matrix composites. One promising approach is the use of nano-sized particles as reinforcement in the matrix. The use of nano-sized particles has been shown to significantly improve composites' mechanical and physical properties (Jebaraj, Isaac and Rajakumar, 2021). The high surface area of these particles provides better interfacial bonding with the matrix, leading to improved mechanical properties.

Aluminum composites are fabricated using different techniques such as stir casting, solid-state processing, spray deposition, etc. (Krishnan, 2022). Among these techniques, stir casting is comparatively simple, low cost, and widely used for the development of Al composites (Kumar and Shiva, 2021). This method involves the addition of reinforcement particles to a molten metal matrix, followed by stirring to distribute the particles uniformly in the matrix. However, the use of nano-sized particles in stir casting can lead to agglomeration and clustering of particles, which can affect the mechanical properties of the composite. Careful selection of process parameters of stir casting can minimize the agglomeration of nano-sized particles in the metal matrix.

Another important factor that can influence the properties of Al matrix composites is the heat treatment process. Heat treatment is an essential process in the synthesis of Al matrix composites, as it can significantly affect the microstructure and mechanical properties of the composite (Zhang, Zheng and John, 2002). Heat treatment involves the application of heat to the composite, followed by controlled cooling to produce the desired microstructure and properties (Totten and Mackenzie, 2000).

In this study, the focus is on the development of Al matrix composites reinforced with nano-sized particles by the stir casting method. The influence of heat treatment on the electromechanical properties of the developed composite is also investigated. The study aims to provide insights into the optimization of the synthesis process of Al matrix composites, with a focus on the influence of processing parameters and heat treatment on the properties of the developed composite. The findings of this study can contribute to the development of high-performance Al matrix composites with enhanced electromechanical properties for various applications.

1.2 Statement of the Problem

Using stir casting to reinforce aluminum matrix composites (Al composite) with nano-sized particles has been demonstrated to be a highly effective way to improve the mechanical and physical characteristics of the composites. However, using nanoparticles in the matrix might cause particle agglomeration and clustering, which can change the composite's mechanical characteristics. Furthermore, there hasn't been enough research done on how heat treatment affects the characteristics of AMCs reinforced with nanoparticles, especially in terms of their electromechanical characteristics.

The issue that needs to be solved in this thesis is to create AMCs reinforced with nanoscale Alumina (Al_2O_3) and Zinc oxide (ZnO) particles using the stir casting method and to investigate how heat treatment affects the electromechanical properties of these materials. Finding the most efficient heat treatment method that affects the distribution of reinforcement particles and improves the electro-mechanical properties of Al composites are some of the specific objectives of the study. Other objectives include determining the optimal volume fraction of nano-sized Alumina (Al_2O_3) and Zinc oxide (ZnO) for achieving the desired properties of Al MMCs.

This study intends to further the development of Al composites with enhanced electro-mechanical properties, which may find use in a variety of sectors, including the aerospace, automotive, and electronics industries.

1.3 Research Objectives

The primary objective of this thesis is to develop Al Composites reinforced with nano-sized particles by stir casting method and investigate the effect of heat treatment on their electromechanical properties. Specifically, the research objectives of this study are:

1. To fabricate Aluminum composites reinforced with Alumina (Al_2O_3) and Zinc Oxide (ZnO).
2. To investigate the electro-mechanical properties of fabricated Al composites.
3. To examine the influence of heat treatment on the electro-mechanical properties of fabricated Al composites.

1.4 Significance of the Study

Aerospace applications demand materials with exceptional electro-mechanical properties to meet the stringent requirements of modern aircraft systems. The study aims to explore the potential of Al composites with nano reinforcements to enhance their mechanical properties, including hardness, flexural strength, and impact toughness while maintaining their lightweight nature. By incorporating these reinforcements into Al composites and optimizing the dispersion, the resulting materials can exhibit enhanced mechanical properties. However, the addition of nano reinforcements affects the microstructure of the Al composite and reduces the electrical conductivity sometimes. One key aspect of the study is also investigating the possible methods to improve the electrical conductivity of the composites while adding Nano reinforcements, such as Alumina (Al_2O_3) and Zinc Oxide (ZnO) at the nanoscale. This is crucial for aerospace applications as it enables efficient transmission of electrical signals, reduces electrical losses, and enhances the performance of electronic components and systems onboard aircraft.

Additionally, the study investigates the effect of heat treatment on the electro-mechanical properties of the Al composites. Heat treatment techniques, such as solutionizing, quenching, and aging, can modify the microstructure within the composites, influencing their hardness and electrical conductivity. Optimizing the heat treatment parameters to achieve the desired material characteristics for aerospace applications will increase the utilization of such Al composites significantly.

Furthermore, the development of Al composites with improved electromechanical properties contributes to the overall weight reduction of aircraft structures. Lightweight

materials are crucial for aerospace applications as they enhance fuel efficiency, increase payload capacity, and improve the overall performance of the aircraft. By incorporating nano reinforcements and optimizing the heat treatment process, the resulting composites can offer enhanced electro-mechanical properties while maintaining their lightweight advantage.

Overall, the significance of the study lies in its potential to advance the development of Al composites with nano-reinforcement particles, optimized through heat treatment, for improved electro-mechanical properties in aerospace applications. The findings of this study can lead to the creation of lightweight, high-performance materials that meet the demanding requirements of modern aircraft systems, ultimately contributing to the advancement and safety of the aerospace industry.

1.5 Scope of this work

The scope of work on the development of aluminum (Al) composites with nano-reinforcement particles and the effect of heat treatment for the improvement of electro-mechanical properties for aerospace applications encompasses several key areas of investigation.

- **Material Selection and Preparation:** The study will begin by identifying suitable nano-reinforcement particles for incorporation into the Al matrix. Various options such as Alumina (Al_2O_3), Zinc Oxide, and Silicon Carbide (SiC) will be considered, considering their compatibility with the Al matrix. The preparation of the Al composites will involve techniques such as stir casting, powder metallurgy, or other appropriate methods.
- **Composite Synthesis:** The scope of work will include the development of a suitable synthesis process for producing Al composites with nano reinforcements. The optimization of processing parameters, such as pre-heating temperature, melting temperature, pressure, stirring speed, and duration will be investigated to achieve uniform dispersion and bonding between the reinforcement particles and the Al matrix. Various heat treatment techniques, including solutionizing, quenching, and aging, will be explored to improve the microstructure within the composites and electro-mechanical properties such as hardness and electrical conductivity.

- **Characterization of Electro-mechanical Properties:** The thesis will involve a comprehensive characterization of the electromechanical properties such as flexural strength, impact toughness, and hardness. Electrical conductivity of the developed Al composites as-casted condition.
- **Influence of Heat treatment:** The fabricated Al composites will undergo various heat treatment processes at different combinations of temperatures. At first, heat treatments will be carried out by a chamber furnace at different solution temperatures followed by water quenching at room temperature. Later, solutionized specimens will be age hardened naturally at room temperature in the laboratory. After the completion of natural aging, thermal aging will be carried out at different temperatures in the same furnace. Later, the same specimens will be aged hardened again naturally at room temperature. After completion of the above heat treatment process, an investigation of electrical conductivity and hardness will be carried out of Al composites. Finally, a comparison will be made on the electro-mechanical properties of Al composites between as casted and heat-treated conditions.
- **Optimization and Prediction of Heat Treatment effects:** The obtained experimental data will be analyzed to understand the relationship between the effect of solution temperature and artificial aging in heat treatment and the resulting desired electro-mechanical properties. Statistical analysis and modeling techniques may be employed to identify the key factors influencing the properties and optimize the heat treatment processes to identify the best heat treatment process. The thesis will also explore the potential synergistic approaches to predict the heat treatment effects on electro-mechanical properties such as hardness and electrical conductivity of developed Al Composites.
- **Characterization of the specific wear rate by Taguchi's Technique:** In this study, characterization of the specific wear rate of developed Al composite reinforced with 2.5.% of Al₂O₃ will be carried out using Taguchi's technique as understanding the wear behavior of the developed Al composite is essential for optimizing their performance and extending their service life in applications that involve sliding or abrasive contact.

In conclusion, the scope of work for the thesis book encompasses material selection, synthesis, processing, and characterization of fabricated Al composites and optimization of heat treatment processes to identify the most suitable effect of heat treatment on their

electro-mechanical properties for aerospace applications. The study aims to contribute to the advancement of lightweight, high-performance materials suitable for the demanding requirements of the aerospace industry.

1.6 Organization of the Thesis

In Chapter 1, the thesis embarks on a comprehensive exploration of the effects of heat treatment on electro-mechanical properties in Al composites. This introductory chapter sets the background of the study, problem statement, research objectives, significance of the study, scope of the work providing context and rationale for the study.

In Chapter 2, a detailed literature review is conducted, focusing on the development of Al composites and the influence of heat treatment on their electro-mechanical properties. This chapter presents a comprehensive overview of the various methods employed for fabricating Al composites and elucidates the heat treatment techniques used to enhance their electro-mechanical properties.

Chapter 3 delves into the materials and experimental setup utilized in the study. It provides an in-depth description of the materials employed for developing Al composites and outlines the experimental apparatus and procedures used for both composite development and subsequent heat treatment processes. It is also dedicated to elucidating the investigation procedure employed to characterize the electro-mechanical properties of the Al composites. This chapter outlines the methodologies and techniques used in the experimental characterization process.

In Chapter 4, the results obtained from the experiments as-casted condition and their subsequent discussions are presented. This chapter offers insights into the outcomes of the research and engages in discussions regarding their implications in relation to the electro-mechanical properties of the Al composites as-casted condition. This chapter also includes the results obtained from the experiments after different heat treatments and their subsequent discussions. This chapter also offers insights into the outcomes of the different heat treatment processes and engages in discussions regarding their implications in relation to the electro-mechanical properties of the Al composites in comparison to as-casted condition.

Chapter 5 focuses on the optimization of various heat treatment parameters, with the aim of identifying the most suitable heat treatment process. Additionally, this chapter delves

into the development of prediction methods and techniques for electro-mechanical properties, such as hardness and electrical conductivity, under different heat treatment conditions.

In this chapter, the investigation of the specific wear rate of the Al composite, utilizing the Taguchi Technique also assesses wear behavior. This chapter provides valuable insights into the wear characteristics of the composites.

In the final chapter, Chapter 6, the thesis culminates with a comprehensive conclusion, summarizing the key findings and contributions of the research. Additionally, this chapter includes a Limitation of the Present works and Recommendations or future works which will be useful to both academics and industries.

CHAPTER 2

LITERATURE REVIEW

2.1 History of Composite Materials

Combining multiple materials to create a new material with performance that is unreachable by the individual constituent is an age-old concept known as composite materials. In this world, almost everything is made of composite materials. The term "composite" generally refers to something that is "made of two or more different parts." A typical chunk of metal, for instance, is a composite (polycrystal) made up of several grains or single crystals. The concept of composite materials is not, strictly speaking, new or recent. A good example of composite material is wood, which is made of cellulose fibers embedded in a lignin matrix. While the lignin matrix connects the fibers and provides stiffness, the cellulose fibers are highly flexible and have high tensile strength. Carbon black in rubber, steel rods in concrete, cement/asphalt combined with sand, fiberglass in resin, and several more contemporary instances are some that came to mind before engineered materials gained popularity (Matthews and Rawlings, 1999).

Metal matrix composites (MMCs), polymer matrix composites (PMCs), and ceramic matrix composites (CMCs) are the three main kinds of composite materials used in man-made composites. These are created by adding different reinforcements, such as fibers, whiskers, or particles, to a metal, polymer, or ceramic substrate, respectively. In the case of metal matrix composites (MMCs), the metal that is utilized most frequently as a matrix can be titanium or aluminum, and the reinforcement can be silicon carbide (SiC) or titanium carbide (TiC) fibers or particles. Polymer matrix composites (PMCs), commonly referred to as FRP - fiber reinforced polymers, are reinforced with a range of fibers, including glass and carbon, and are made of a polymer-based resin as the matrix. One of the polymer matrix composites is fiberglass, which was the first successful modern composite. Additionally, small fibers or particles, such as those formed of silicon carbide and boron nitride, are employed as reinforcement in ceramic matrix composites (CMCs), which are used in very high-temperature conditions (Clyne and Withers, 1993).

The desire for stronger stiffer yet lighter composites in industries as diverse as aerospace, energy, automotive, and civil construction gave rise to the idea of creating composite

materials artificially. A growing amount of research and development has been put into these materials since the early 1960s. Today, we can create a composite material that satisfies or even surpasses the performance criteria thanks to the most effective design, innovative materials, and production techniques. Weight and cost savings account for the majority of the benefits of using these materials. Ratios like stiffness/weight, strength/weight, and cost/weight ratios are used to quantify these (Clyne, 2000).

Over the years, engineers and scientists have developed new, cutting-edge methods that have improved composite materials over monolithic ones. Design and analysis of composite materials have become more adaptable, allowing designers to develop unique materials for each application with an eye on weight and cost reduction. Integration of manufacturing and design at all stages, from ideation through material fabrication, as well as failure analysis, is yet another development.

Thus, compared to monolithic materials, composite materials have a more "controlled" life cycle because they were carefully created for a particular application, making it more likely that they will solve any issues that may arise. While the process of combining materials was first developed by nature, man-made composite materials are the ones that have advanced properties and are created to fill specific engineering roles by utilizing the components' desirable properties while minimizing the negative effects of their less desirable ones. The matrix is a continuous bulk phase that is most frequently present in composite materials, and the reinforcement is a dispersed, non-continuous phase that is typically tougher and stronger. The fundamental idea behind composites is as follows: the bulk phase absorbs the load over a sizable surface area and passes it to the reinforcement, which because it is stiffer, boosts the composite's strength. The importance of this comes from the fact that there are multiple matrix materials and an equal number of reinforcement types, which can be mixed in endless ways to provide the precise qualities that are wanted (Taya and Arsenault, 1989).

2.2 Overview of Metal Matrix Composites (MMCs)

A class of materials called Metal Matrix Composites (MMCs) has demonstrated significant promise for usage in aeronautical applications (Shabestari and Allahkaram, 2018). They are made up of a metallic matrix and a reinforcing phase which may be fibers, metallic or ceramic particles, or even whiskers as shown in Figure 2.1. The properties of common reinforcement materials are given in Table 2.1 MMCs have better

mechanical characteristics, thermal stability, and wear resistance than conventional aerospace materials including metals and alloys (Liu, Li and Chen, 2018).

MMCs are highly sought-after for use in aeronautical applications because of their special qualities (He, Wang and Lin, 2019). For instance, MMCs are perfect for usage in aviation and spacecraft components because of their high strength-to-weight ratio, which emphasizes the importance of weight reduction. MMCs are also ideally suited for usage in high-temperature applications like rocket nozzles and gas turbine engines due to their great thermal stability (Zhou, Xu and Wang, 2021).

By modifying the matrix's composition and structure as well as its reinforcing phases, MMCs can have specific qualities (Shabestari and Allahkaram, 2018). For instance, altering the size and shape of the reinforcing particles can increase wear resistance and fracture toughness while increasing the volume fraction of the reinforcing phase can result in a material with better stiffness and strength.

MMCs offer a number of possible uses in aircraft, according to research (He, Liu and Zhang, 2022). MMCs, for instance, have been utilized to produce lightweight parts for spacecraft and airplanes, including as heat exchangers and engine components. They have also been utilized to create rotorcraft and aircraft high-performance braking discs. The application of MMCs in space travel has also been researched, including the creation of sophisticated heat shields for re-entry vehicles and thermal protection systems for spacecraft. Additionally, MMCs have been researched for usage in satellite parts like solar cells and communication antennas (He, Wang and Lin, 2019). Further advancements in the creation and use of MMCs in aerospace are anticipated as a result of ongoing research (He, Liu and Zhang, 2022).

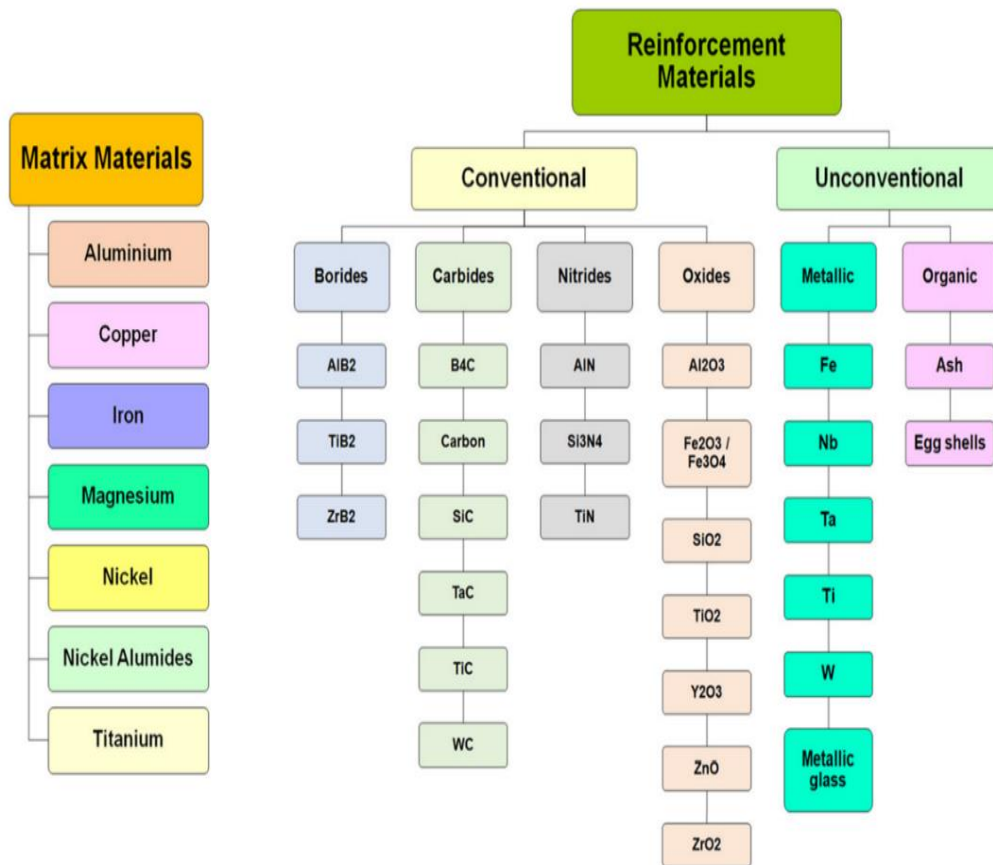


Figure 2.1: Usage of matrix and reinforcement materials in MMCs (Seetharaman, Sankaranarayanan and Gupta, 2021)

Table 2.1: Properties of common reinforcement materials (Seetharaman, Sankaranarayanan and Gupta, 2021)

Material	Specific Gravity	Tensile Strength	Specific Strength	Modulus of Elasticity	Specific Modulus
		[GPa (10 ⁶ psi)]	(GPa)	[GPa (10 ⁶ psi)]	(GPa)
Whiskers					
Graphite	2.2	20	9.1	700	318
Silicon nitride	3.2	5–7	1.56–2.2	350–380	109–118
Aluminum oxide	4	10–20	2.5–5.0	700–1500	175–375
Silicon carbide	3.2	20	6.25	480	150
Fibers					
Aluminum oxide	3.95	1.38	0.35	379	96
Aramid	1.44	3.6–4.1	2.5–2.85	131	91
Carbon	1.78–2.15	1.5–4.8	0.70–2.70	228–724	106–407
E-glass	2.58	3.45	1.34	72.5	28.1
Boron	2.57	3.6	1.40	400	156
Silicon carbide	3	3.9	1.30	400	133
UHMWPE (Spectra 900)	0.97	2.6	2.68	117	121
Metallic Wires					
High-strength steel	7.9	2.39	0.30	210	26.6
Molybdenum	10.2	2.2	0.22	324	31.8
Tungsten	19.3	2.89	0.15	407	21.1

2.3 Types of Metal Matrix Composites (MMCs)

Based on the kind of reinforcing phase, Metal Matrix Composites (MMCs) can be categorized into numerous types as shown in Figure 2.2. A few of the most typical MMC types will be highlighted in this part, along with instances and pertinent studies relevant to the present research objectives.

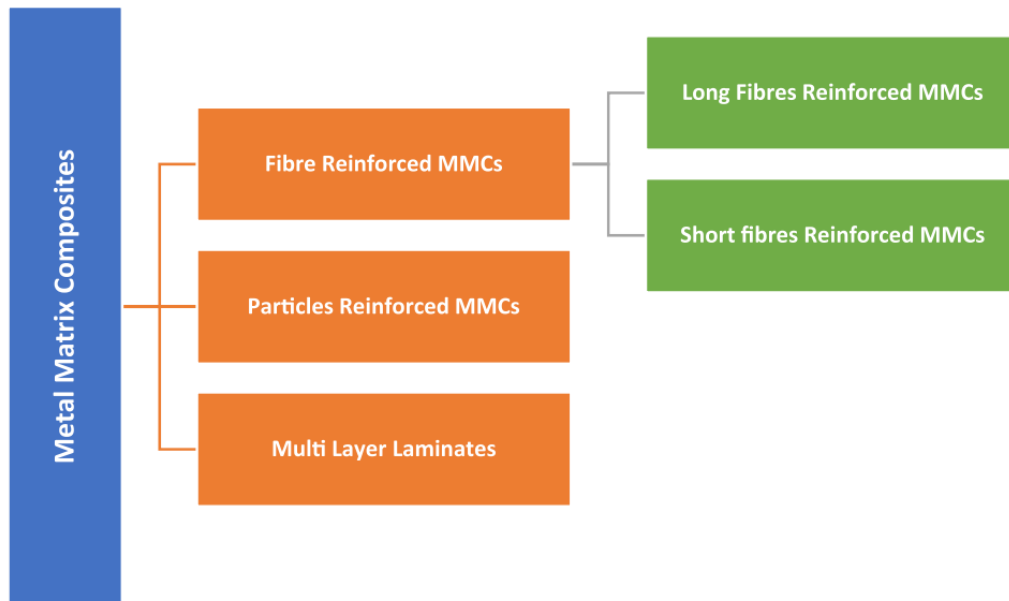


Figure 2.2: Classification of metal matrix composites (Seetharaman, Sankaranarayanan and Gupta, 2021)

2.3.1 Fiber-reinforced MMCs

Fiber-reinforced metal matrix composites (MMCs) are a family of innovative materials that are highly sought after for a range of applications, such as parts for aviation and spacecraft, due to their remarkable mechanical properties, such as high strength, stiffness, and toughness. It is commonly known that by adding reinforcing fibers to the metal matrix, the material properties can be significantly improved. The most often used fibers in MMCs are carbon fibers (CFs), silicon carbide fibers (SiCf), and alumina fibers (Al_2O_3 f) because of their superior mechanical properties (He et al., 2019).

Numerous investigations have been conducted recently on the effects of fiber reinforcements on the properties of MMCs. For instance, Zhang et al. (2020) found that the mechanical properties of aluminum MMCs, such as their ultimate tensile strength and elastic modulus, were greatly improved by the addition of CFs. Similar results were obtained in Yu et al. (2019) work, which investigated the mechanical properties of TiB₂-reinforced MMCs and discovered that the addition of TiB₂ fibers enhanced the composites' tensile strength and ductility.

Fiber-reinforced MMC development has advanced significantly in recent years. For instance, Singh et al. (2018) research on the mechanical properties of Al/SiCf MMCs

showed that SiC fibers significantly improved the composites' tensile and compressive strength. Fiber-reinforced MMCs are cutting-edge materials with exceptional mechanical properties as compared to the base metal matrix. The three fiber types that are most frequently used in MMCs are CFs, SiCf, and Al₂O₃f. Recent research has demonstrated that adding other fiber types, including TiB₂ fibers, can significantly improve the mechanical properties of MMCs. Additionally, the use of hybrid fiber reinforcements like CFs and GNPs has a lot of promise to improve the mechanical properties of MMCs for a number of industrial applications.

2.3.2 Particulate-reinforced MMCs

Modern materials known as Particulate-Reinforced Metal Matrix Composites (MMCs) have better mechanical properties that make them suitable for a wide range of industrial applications. High strength, stiffness, and wear resistance are some of these qualities. Particle reinforcements can be added to the metal matrix to dramatically improve the material's properties. The three most often used types of particle reinforcements are silicon carbide (SiC), alumina (Al₂O₃), and titanium carbide (TiC), all of which have outstanding mechanical properties (Lee and Jang, 2016). According to the study, the properties of particulate-reinforced MMCs can be altered by varying the volume fraction and size of the reinforcing particles.

Numerous studies have looked into how particle reinforcement's affect the properties of MMCs. Jiang et al. (2019) for instance, looked at the mechanical properties of MMCs reinforced with SiC particles based on aluminum. The results demonstrated that adding SiC particles significantly improved the hardness and wear resistance of the composites. Similar findings were made by Singh et al. (2016), who found that the addition of TiC particles improved the tensile strength and elastic modulus of aluminum MMCs.

Particle-reinforced MMCs, a new class of materials, give basic metal matrix improved mechanical properties. The most often used particle reinforcements in MMCs are SiC, Al₂O₃, and TiC. Recent research has demonstrated that the addition of other kinds of particles, including GNPs, can significantly enhance the mechanical properties of MMCs. More research is therefore needed to fully investigate the potential of particulate-reinforced MMCs for various industrial applications.

2.3.3 Multi-Layer Laminate Composites

Composite laminates are constructed from numerous layers of sheet laminates made of matrix and reinforcing materials that are layered and attached in a specific pattern in order to achieve the required strength. Depending on how they are stacked, the composite laminates are separated into angle and cross-ply laminates. These laminates could be symmetric, antisymmetric, or balanced. The design of a thin laminated composite (0.16 kg) intended to replace the aluminum heat sinks (0.29 kg) used on printed circuit boards is shown in Figure 2.3.

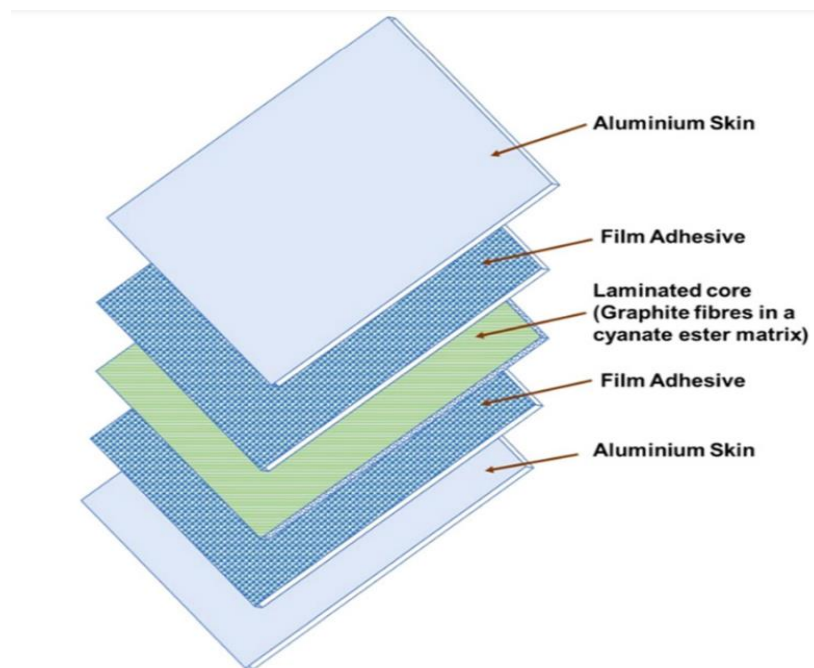


Figure 2.3: Lightweight laminated composite heat sink developed for printed circuit boards (Holz, Niemeyer and Puckett, 2000).

2.3.4 Whisker-reinforced MMCs

Whisker-reinforced metal matrix composites (MMCs), a class of contemporary materials, exhibit excellent mechanical properties including high strength, stiffness, and resistance to wear. Small, solitary crystals, whiskers have a high aspect ratio and a low diameter. Whiskers are typically made from materials such as silicon carbide (SiC), aluminum oxide (Al_2O_3), and silicon nitride (Si_3N_4).

The effect of whisker reinforcements on the properties of MMCs has been the subject of numerous investigations in recent years. For instance, SiC whiskers significantly improved the tensile strength and hardness of the composites, according to Lee et al.

(2019) study, which looked at the mechanical properties of Al/SiC whisker-reinforced MMCs. Similar findings were made by Zhang et al. (2018), who discovered that titanium MMCs' mechanical properties, such as elastic modulus and ultimate tensile strength, were improved by the addition of Si₃N₄ whiskers. The use of hybrid whisker reinforcements, such as the mixing of SiC and Al₂O₃ whiskers, has also been shown to greatly enhance the mechanical properties of MMCs. For instance, Wang et al. (2021) research examined the effects of introducing hybrid SiC and Al₂O₃ whiskers to aluminum MMCs on the mechanical characteristics of those materials, such as ultimate tensile strength and fracture toughness, and discovered a significant effect.

2.3.5 Hybrid-reinforced MMCs

Hybrid-reinforced MMCs consist of a metallic matrix and a number of reinforcing phases, including as fibers and particles. These composites' unique features can be altered by changing the volume fraction and distribution of the reinforcing elements. According to study, hybrid-reinforced MMCs can display exceptional mechanical attributes such high stiffness, strength, and fatigue resistance. Hybrid-reinforced metal matrix composites (MMCs), a new class of materials, having superior mechanical qualities over single-reinforcement MMCs. Two or more types of reinforcements, such as fibers, whiskers, and particles, are mixed in a metal matrix to create hybrid reinforcements (Khan, Zulfiqar and Hameed, 2020). The complementing effects of the multiple reinforcements enhance the mechanical properties of the composite, such as its strength, stiffness, and toughness.

In recent years, a number of researchers have looked into how hybrid reinforcements affect the properties of MMCs. For example, Li et al. (2020) evaluated the mechanical properties of hybrid Al/SiC/Gr-reinforced MMCs and discovered that the addition of graphite (Gr) particles significantly improved the composites' tribological properties. When SiC fibers and particles were introduced to aluminum MMCs, the mechanical parameters of the composites, such as tensile strength and hardness, improved similarly to what Nai et al. (2018) found. Additionally, research has shown that adding hybrid reinforcements improves the way MMCs behave when they become fatigued. For instance, Fu et al. (2018) evaluated the fatigue behavior of Al/SiC/Al₂O₃ hybrid-reinforced MMCs and found that the Al₂O₃ particles' ability to bridge cracks lengthened the composites' fatigue life.

In conclusion, Metal Matrix Composites (MMCs) can be classified into several types based on the nature of the reinforcing phase, including particulate-reinforced MMCs, fiber-reinforced MMCs, whisker-reinforced MMCs, and hybrid-reinforced MMCs. Ongoing research is expected to lead to further advances in the development and application of these types of MMCs.

2.4 Aluminum and Aluminum Alloys

2.4.1 Aluminum

Due to its distinctive qualities, including low density, high strength, strong thermal and electrical conductivity, and exceptional corrosion resistance, aluminum is a material that is extensively employed across a variety of sectors. The characteristics of aluminum are further improved by alloys and composites, increasing its adaptability and usefulness in a range of applications. The properties, production processes, and applications of aluminum, aluminum alloys, and aluminum composites are covered in this section.

2.4.2 Aluminum Alloys

Due to its distinctive qualities, such as its lightweight, high strength-to-weight ratio, great corrosion resistance, and exceptional formability, aluminum, and its alloys are extensively employed in a variety of sectors. Based on their composition, heat treatment, and mechanical characteristics, aluminum alloys can be divided into many groups. The various varieties of aluminum alloys, as well as their uses and qualities, will be covered in this section.

2.4.2.1 1xxx Series Aluminum Alloys

The 1xxx series aluminum alloys are the purest type of aluminum alloys since they have a minimum aluminum content of 99% (Wang, 2019). These alloys are suited for electrical and heat transfer applications because of their outstanding electrical conductivity and high thermal conductivity (Vizureanu and Stefanescu, 2018). The 1xxx series of aluminum alloys are not appropriate for high-stress applications due to their comparatively low strength in terms of mechanical characteristics. However, they are perfect for applications that demand exceptional workability, such as roofing, flashing, and decorative trim, due to their high ductility and great formability (Karthik and Krishna, 2018). The alloy 1100, which is utilized in a variety of applications including

heat exchangers and reflectors, is the most popular one in the 1xxx series (Almarhoon, Alsulaiman and Mansour, 2022).

In summary, the pure aluminum alloys of the 1xxx class have good electrical and thermal conductivity and are very resistant to corrosion. They are excellent for a variety of applications in the building, electrical, and chemical processing industries despite having low strength and considerable ductility.

2.4.2.2 2xxx Series Aluminum Alloys

The 2xxx series of aluminum alloys stand out for their superior fatigue resistance, high strength, and low density. These alloys are frequently employed in industries like aerospace and defense where strength is important. With a minor proportion of magnesium, copper serves as this series' primary alloying ingredient.

Due to its outstanding mechanical qualities, 2024, one of the most popular 2xxx series alloys, has received substantial research. Due to its outstanding fatigue resistance, the 2024 aluminum alloy is frequently employed in airplane structures, including the wings and fuselage (Ye et al., 2018). Its weak corrosion resistance, however, prevents it from being used in several situations. 2219, a 2xxx series alloy with exceptional strength and toughness at cryogenic temperatures, has also drawn attention recently. It is therefore perfect for use in the construction of cryogenic tanks (Zhou et al., 2019). It is also frequently utilized in the production of rocket engine components and has good weldability.

The mechanical properties of alloys in the 2xxx series can be improved through alloying and processing procedures, according to a number of studies. In order to increase the strength and corrosion resistance of 2024 alloy, researchers have looked into the impacts of adding rare earth elements (Sajjadi, Seyedein and Kokabi, 2019). The effects of various processing methods, including hot extrusion, on the mechanical characteristics of 2219 alloy have been examined by researchers (Mahdavian et al., 2021).

2.4.2.3 3xxx Series Aluminum Alloys

The aluminum alloys in the 3xxx series are primarily magnesium-free aluminum-manganese alloys. These alloys have outstanding forging, welding, and corrosion resistance properties. Manganese serves as the main alloying element in the 3xxx series and increases formability and strength. The alloy's strength and resistance to corrosion are

further improved by the addition of magnesium. 3003 is one of the most widely used alloys in the 3xxx series and is utilized in a variety of applications due to its outstanding forming and welding properties as well as its corrosion resistance. It is frequently employed in the creation of pressure vessels and decorative trim. A different popular alloy is 3105, which is comparable to 3003 but has somewhat better strength and corrosion resistance. Zirconium (Zr) can enhance the mechanical properties of 3xxx series aluminum alloys, including their strength and hardness, according to a study by Islam et al. (2018). The scientists discovered that the addition of Zr enhanced the alloy's yield strength and hardness while barely changing its ductility.

In a different study, Lee et al. (2021) examined the impact of annealing temperature on the microstructure and mechanical characteristics of an aluminum alloy from the 3xxx series. They discovered that although lower temperatures led to finer grains and more strength, higher temperatures produced coarser grains and lesser strength. Additionally, according to Zhai et al. (2019) research, the mechanical properties of 3xxx series aluminum alloys, specifically their strength and ductility, can be enhanced by the inclusion of rare earth elements like cerium (Ce) and lanthanum (La). The scientists discovered that Ce and La boosted the alloy's strength and ductility while also enhancing its corrosion resistance.

Overall, the superior formability, weldability, and corrosion resistance of the 3xxx series aluminum alloys make them popular in a variety of applications. Ongoing research aims to enhance their mechanical characteristics and create novel alloys with special features for certain applications.

2.4.2.4 4xxx Series Aluminum Alloys

The predominant alloying ingredient in the 4xxx series aluminum alloys is silicon, with minor amounts of copper, magnesium, or both also present (ASM International, 2018). Due to their exceptional weldability, they are frequently utilized as filler materials in welding applications (Dursun and Yamanoglu, 2020). Due to their weak strength, the alloys in this family are unsuitable for high-stress applications (ASM International, 2018). The automobile and construction sectors both make extensive use of aluminum alloys from the 4xxx class (Lai et al. 2019). They are employed in the automotive sector to weld aluminum body components including fenders and hoods (Cao et al. 2020). They are employed in the construction sector to weld aluminum sheets for roofing and cladding

purposes (Asadi, Tavakoli and Emamian, 2018). The 4xxx series aluminum alloys' low melting point, which makes them suited for use in soldering applications, is one of their main advantages (Abioye and Malabadi, 2019). They are frequently employed in the production of electrical conductors and heat exchangers (Zhang et al. 2020).

The 4xxx series of aluminum alloys' mechanical properties have recently become the subject of investigation. For instance, studies have examined how these alloys' strength and ductility are affected by heat treatment and alloy composition (Zhou et al. 2021). In other investigations, equal-channel angular pressing and other cutting-edge processing methods have been investigated in order to enhance the mechanical properties of these alloys.

2.4.2.5 5xxx Series Aluminum Alloys

Magnesium serves as the main alloying ingredient in the 5xxx series aluminum alloys, with a minor quantity of chromium. They are perfect for producing corrosion-resistant parts because of their outstanding formability, weldability, and corrosion resistance. A set of non-heat-treatable aluminum alloys with outstanding corrosion resistance and formability is known as the 5xxx series. Numerous industrial applications, including those in the automotive, aerospace, and marine industries, frequently utilize these alloys (Zhang and Zhang, 2019). Several research investigations have recently examined the characteristics and processing of aluminum alloys from the 5xxx class.

The impact of alloying elements on the mechanical properties of aluminum alloys from the 5xxx series has been the subject of some investigation. For instance, Liu et al. (2018) looked into how zinc (Zn) and magnesium (Mg) additions affected the mechanical characteristics of Al-Mg-Zn alloys. The findings demonstrated that the alloys' strength and ductility were enhanced by the addition of Zn and Mg. Additionally, studies have looked into the processing of aluminum alloys from the 5xxx family utilizing cutting-edge methods. For instance, Han et al. (2018) examined the impact of friction stir processing (FSP) on the mechanical characteristics and microstructure of Al-Mg alloys. Because of the finer grain size and production of a fine dispersion of strengthening precipitates, the results demonstrated that FSP significantly improved the mechanical characteristics of the alloys.

In conclusion, the superior corrosion resistance and formability of the aluminum alloys of the 5xxx series have led to their widespread use in several industrial applications. Recent

studies have shown that the employment of cutting-edge processing methods and the addition of alloying elements can improve the mechanical properties of the alloys for a variety of industrial applications.

2.4.2.6 6xxx Series Aluminum Alloys

The heat-treatable 6xxx series aluminum alloys are a class of alloys with high strength and exceptional formability. These alloys are frequently employed in a wide range of industrial applications, including those in the construction, aerospace, and automotive sectors (Lu and Lu, 2017). Numerous studies have looked into the characteristics and processing of aluminum alloys from the 6xxx class recently.

The impact of heat treatment on the mechanical properties of aluminum alloys in the 6xxx series has been the subject of some investigation. For instance, the work by Lwin et al. (2019) looked at the impact of aging on the mechanical characteristics of Al-Mg-Si alloys. The outcomes demonstrated that the alloys' strength and ductility improved as a result of the aging treatment. The processing of aluminum alloys from the 6xxx series utilizing cutting-edge methods has also been studied in research investigations. The 6xxx series of aluminum alloys' welding behavior has also been the subject of scientific studies. For example, Rahman et al. (2022) looked into how welding settings affected the microstructure and mechanical characteristics of Al-Mg-Si alloys. The outcomes demonstrated that the alloys' joint strength and toughness significantly increased as a result of the optimal welding parameter selection.

As a result of their high strength and outstanding formability, the 6xxx family of aluminum alloys is widely employed in a variety of industrial applications. Recent studies have shown that the use of heat treatment, sophisticated processing techniques, and suitable welding conditions can result in an improvement in the mechanical properties of the alloys for a variety of industrial applications.

2.4.2.7 7xxx Series Aluminum Alloys

A collection of high-strength aluminum alloys known as the 7xxx series are frequently employed in aerospace and defense applications because of their exceptional strength-to-weight ratio and resistance to the development of fatigue cracks (Ramesh and Ranganath, 2017). Numerous research investigations have recently examined the characteristics and processing of aluminum alloys from the 7xxx class. The impact of alloying elements on

the mechanical characteristics of aluminum alloys from the 7xxx series has been the subject of some investigation. For instance, Zn, Cu, and Mg were examined in the work by Arjmand et al. (2020) to see how they affected the microstructure and mechanical characteristics of Al-Zn-Cu-Mg alloys. The findings demonstrated that the addition of Mg improved the strength and ductility of the alloys whereas the addition of Zn and Cu reduced ductility.

Additionally, investigations have looked into treating aluminum alloys from the 7xxx series utilizing cutting-edge methods. For instance, Arif et al. (2018) looked into how friction stir processing (FSP) affected the microstructure and mechanical characteristics of Al-Zn-Mg-Cu alloys. Because of the finer grain size and production of a fine dispersion of strengthening precipitates, the results demonstrated that FSP significantly improved the strength and ductility of the alloys. Additionally, investigations have concentrated on the 7xxx family aluminum alloys' fatigue characteristics. For instance, the work by Xiong et al. (2020) looked at the impact of temperature on the Al-Zn-Mg-Cu alloys' fatigue crack development behavior. The findings demonstrated that the activation of thermally-activated slip mechanisms caused the fatigue crack propagation rate to rise as the temperature rose.

In conclusion, the exceptional strength-to-weight ratio and resistance to fatigue fracture growth of the 7xxx series aluminum alloys make them a popular choice for aerospace and defense applications. Recent studies have shown that the use of suitable alloying components, cutting-edge processing methods, and comprehension of fatigue behavior can improve the mechanical properties of the alloys for a variety of industrial applications.

2.4.2.8 8xxx series aluminum alloys

Due to its exceptional strength-to-weight ratio and great corrosion resistance, the 8xxx series aluminum alloys are frequently employed in high-strength applications like aerospace and transportation. Recent studies have concentrated on different approaches, like alloying, heat treatment, and microstructural alterations, to improve the mechanical properties of these alloys.

For instance, the study by Xu et al. (2017) looked into how additions of zirconium (Zr) and scandium (Sc) affected the mechanical properties of aluminum alloys from the 8xxx series. The findings demonstrated that the mechanical properties of the alloys, such as

tensile strength, yield strength, and elongation, were improved by the addition of Sc and Zr. Additionally, investigations have looked into how heat treatment affects the mechanical characteristics of aluminum alloys from the 8xxx class.

Recent studies have shown that a variety of techniques, including alloying, heat treatment, and microstructural alterations, can enhance the mechanical properties of aluminum alloys from the 8xxx series for high-strength applications. Future studies in this field should concentrate on creating novel techniques to enhance these alloys' mechanical characteristics for a variety of industrial applications.

2.5 Aluminum Composites and Their Fabrication Techniques

Aluminum composite, a cutting-edge material in the realm of modern construction and engineering, stands as a testament to innovation and versatility. This introductory exploration delves into the fundamental aspects of aluminum composites, elucidating their composition, fabrication processes, mechanical characteristics, and a diverse array of applications across industries. Through a comprehensive examination of the distinctive properties that define aluminum composites, this study aims to provide a foundational understanding of their role in advancing contemporary materials science and engineering practices.

The characteristics of composites are significantly influenced by the production process (Olszówka, Szala and Cwajna, 2001). The main methods used to create Al MMCs are as follows.

- a. Solid State Processes
- b. Liquid State Processes
- c. Deposition Processes

This review study is limited to fabrication techniques based on in-depth applications by numerous researchers. The fabrication techniques examined are shown in Figure 2.4.

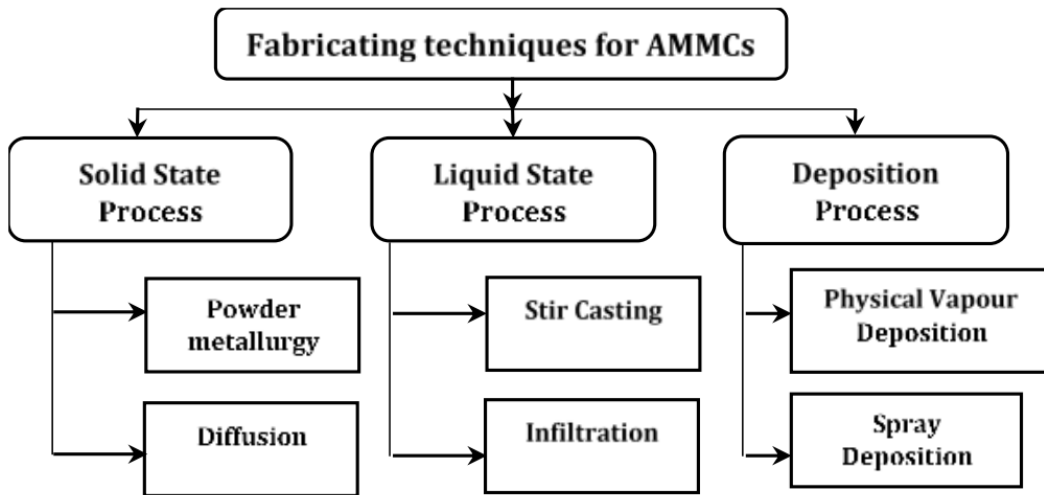


Figure 2.4: Fabrication Techniques for making Al MMCs (Gururaj et al. 2021)

2.5.1 Solid-state processing

2.5.1.1 Powder Metallurgy (PM Process)

These are the primary methods utilized to create AMMCs. Although it is a conventional metallurgy procedure, metal matrix composites are later produced using it (Olszówka, Szala and Cwajna, 2001). The metal matrix composites can be produced, even at lower temperatures, making it a desirable method. This approach makes it simple to regulate kinetic interfaces.

The matrix's alloy compositions can be changed readily in this method as well. A step-by-step description of the powder metallurgical process is shown in the accompanying Figure 2.5.

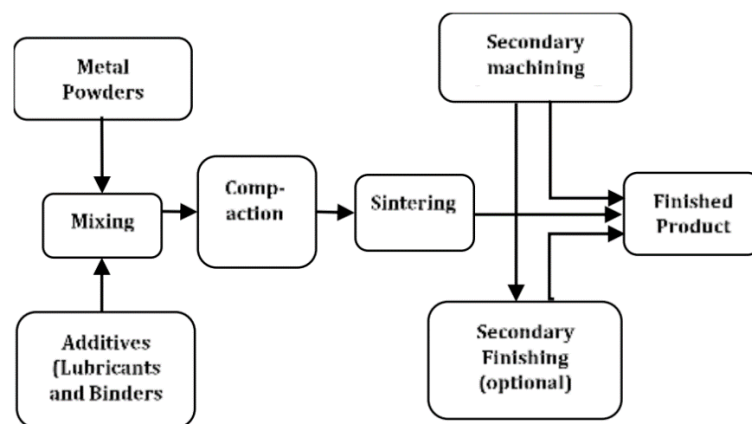


Figure 2.5: Powder Metallurgy Process (Gururaj et al. 2021)

Step 1: Weighing the composition (including any additions, reinforcement, and metal powder)

Step 2: Combine the ingredients listed above.

Step 3: A uni-axial press tool is used to compact the combined powder. A green body is the end product. The green body should have a density of about 85% of the composite.

Step 4: After applying heat at a desired temperature, the green body is sintered (Garg et al. 2016).

The finished product is referred to as a sintered body. It ought to have the same density as the composite. Al- Al_2O_3 composites were sintered to create a body by the PM Process (Olszówka, Szala, and Cwajna, 2001). Here, a vibrating rotary mill is used to create a composite powder with particles between 160 and 380 microns in size. Then, sintering is done inside a container made of graphite that is kept at a vacuum of 2.67 Pa. The sintered body is created for 15 minutes at a temperature of 910K and a pressure of 15MPa. Through the use of a microscope, the microstructure of the composite powder is examined, and a micrograph is provided. The matrix particles are encircling the Al_2O_3 particles in the micrograph, as seen in Figure 2.6. Due to the homogenous microstructure of the manufactured MMCs, it is discovered that the PM approach is a flexible way of MMC manufacturing. But the biggest drawback of this approach is the existence of contaminants.

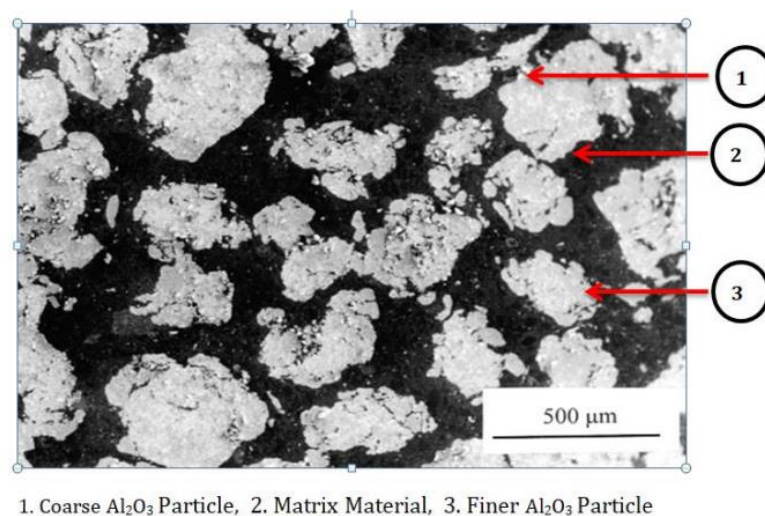


Figure 2.6: Micrograph of Al- Al_2O_3 composite powder (Olszówka, Szala and Cwajna, 2001)

2.5.1.2 Diffusion bonding

It is a method used mostly for materials with persistent oxide layers and high heat sensitivity.

With the addition of the reinforcing particles, this method is also utilized to create composites that are challenging to connect (Feest et al. 1986). Similar to a pressure welding method, diffusion bonding uses pressure and temperature to create interatomic bonds as shown in Figure 2.7. Rough surfaces are present on the materials that go through the diffusion bonding process. The roughness of the substrate is deformed, and the aforementioned surface layers are disrupted and dispersed during the initial stage of the bonding process. Diffusion and grain growth occur to create a new material in the second stage of the bonding process.

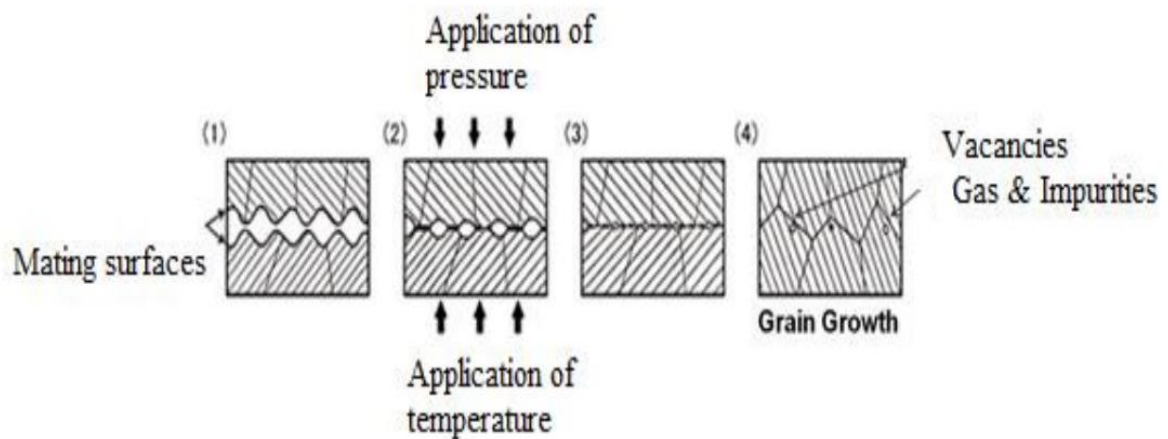


Figure 2.7: Diffusion Bonding Process (Gururaj et al. 2021)

The diffusion bonding mechanism is shown in Figure 2.6.1. Al_2O_3 -Al composite was used as the interlayer in the fabrication of alumina (Al_2O_3) joints and alumina/aluminum MMC ($\text{Al}_2\text{O}_3/\text{Al}$ -MMC) junctions (Li and Xiao, 2002). SEM, X-ray diffraction analysis, and shear testing were each used to analyze the joints' microstructures, phase compositions, and mechanical characteristics. Figure 2.8 displays the SEM image in question.

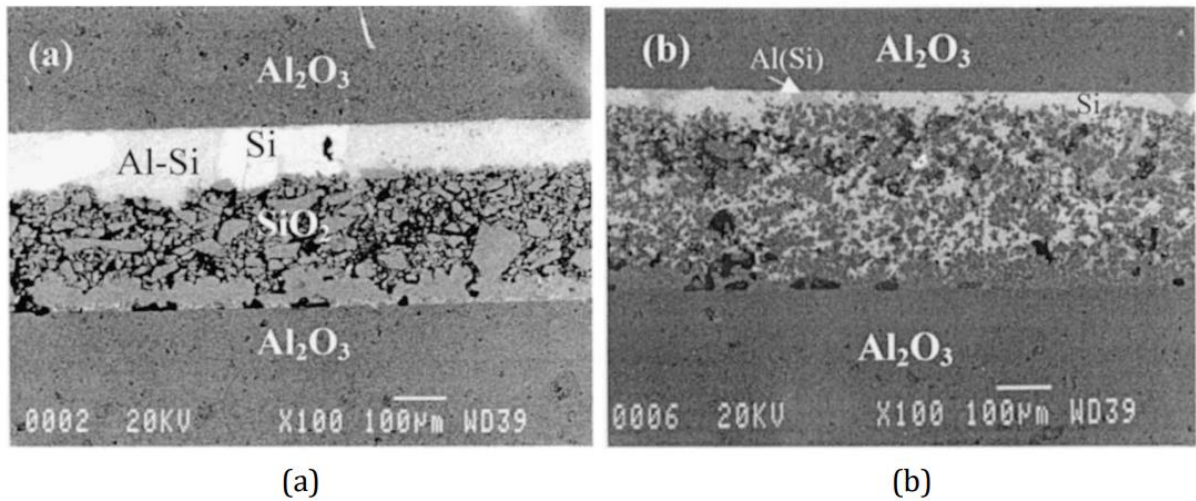


Figure 2.8: SEM images of Cross-section of $\text{Al}_2\text{O}_3\text{Al}-\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ composite; (a) at 850°C , (b) at 1000°C (Li and Xiao, 2002).

2.5.2 Liquid state processing

2.5.2.1 Stir casting

It is the most commonly used liquid state fabrication process. As seen in Figure 2.9, the matrix material is melted in the stir casting setup's crucible or muffle. The electrical filament arrangement provides the temperature. The molten matrix metal is then stirred while the treated reinforcement particles are added. The homogeneous dispersion of the reinforcing particles justifies the churning. Following the stirring is the casting of the desired shape. When compared to the majority of casting procedures, stir casting is the most affordable method (Li and Xiao, 2002). The addition of reinforcement particles uses the vortex approach. The stirrer-created vortex of the molten alloy is filled with the pretreatment reinforcement particles (Contreras, López and Bedolla, 2004). The governing factors for getting a homogeneous mixture to cast are the size of the crucible, the impeller or stirrer, the temperature of the molten metal or alloy, the time and speed of stirring, and the rate at which reinforcement is poured. The two difficulties in this method are as follows (Lakshmi and Gupta, 1998). Some reinforcing particles are not moistened by the molten metal. Therefore, pre-treatment is required due to the density difference between the reinforcement particles and the molten metal; otherwise, the uniform dispersion would not be at the desired level and the reinforcement particles would tend to float or sink inside the molten metal. The Stir casting technique is found to be the best method due to its ease of use, low production costs, ability for mass manufacturing, etc.

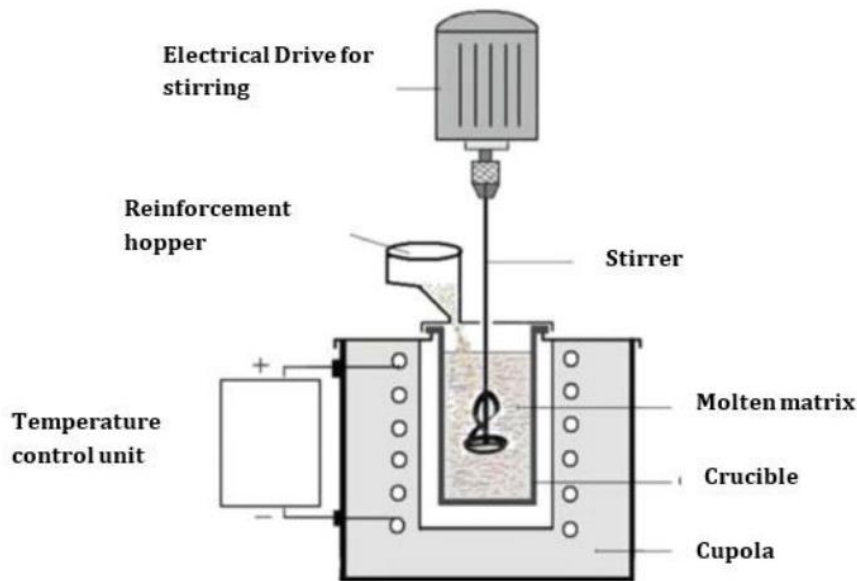


Figure 2.9: Stir Casting Equipment (Gururaj et al. 2021)

2.5.2.2 Infiltration Process

In the infiltration process, a preform is prepared with the use of reinforcement material with high porosity. The porosity of the reinforcing preform is then allowed to fill with the molten metal. If the molten metal wets the preform, no outside power is required to fill the preform pores with the molten metal. The capillary force is sufficient. If not, external mechanical force is used to counteract the capillary force and drag. The molten metal is then combined with the filled preform by applying pressure. The process of infiltration is depicted in Figure 2.10.

In an experiment, a medium-pressure infiltration procedure is used to create an AMMC with continuous fibers of alumina as reinforcement and pure aluminum powder as a matrix. For the aforementioned AMMC, a micrograph is provided (Contreras, López and Bedolla, 2004). Both the forced and spontaneous infiltration processes are used to create metal matrix composites.

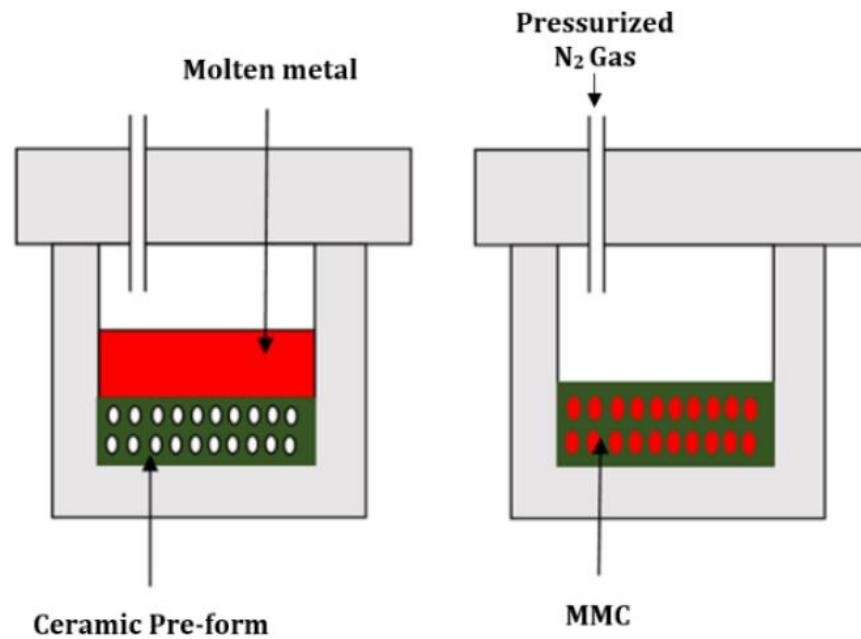


Figure 2.10: Infiltration Process (Gururaj et al. 2021)

2.5.3. Deposition Process

2.5.3.1. Physical Vapour Deposition (PVD)

This method can also be used to create metal matrix composites. It moves along pretty slowly. The metal is vaporized using an electron beam, and the fiber material is then passed over the area of intense metal vapors. Metal vapours condense over the fiber materials, and as a result, the fiber is thickly coated. The average deposition rate is 5 to 10 μm . The coated fibers are then put in rows and put through a hot-pressing process to create metal matrix composites. Physical Vapour Deposition with an electron beam is the approach mentioned above. There is also another approach, called the sputtering technique. The metal item is coated with coating materials using the sputtering technique, which sprays ions from an inert gas field over it. The inert gas ions in the coating substance broke apart the metal piece's atoms and covered it.

2.5.3.2. Spray Deposition Method

This technique creates the composite material by forming tiny droplets of liquid metal and spraying them over the substrate. The Osprey method and the Thermal Spray method, often known as the plasma spray method, are the two different types of spray deposition techniques. The Osprey technique uses inert gases to create tiny metallic droplets, which

are then sprayed over the metallic substrate. The following factors affect the coating's quality.

- a) Distance from the nozzle to the substrate (flight length),
- b) Type of atomizing inert gas
- c) Spray angle
- d) Atomizing pressure and
- e) Coating rate.

2.5.4 Comparison of Different Fabrication Techniques for Al Composites

According to a review of the market, pure aluminum, aluminum alloys, and aluminum composites have an excellent track record due to their desirable qualities, such as high modulus of strength, high fracture strength, high compressive strength, and low thermal strength expansion coefficient. The output of Al-based composites is particularly high when compared to other metallic composites. The cost-performance ratio of aluminum products is the biggest worry among the aforementioned benefits. Due to their marketability, several metal matrix composites are available. Al-based composites can compete even when their cost-to-performance ratio is not at an acceptable level. Table 2.2 lists the comparison of various fabrication methods based on the area of applications.

Table 2.2: Comparison on Different Fabrication Techniques of Al Composites (Garg et al. 2019)

Fabrication route	Cost of fabrication	Field of Applications
Stir casting	Very least	It is suitable for the mass production of AMMCs and commercial products
Infiltration Process	Low	The building materials like rods and some other uniaxial components are produced by this method.
Powder metallurgy	Medium	Small mechanical components such as bolts, valves, and pistons, etc., are produced using this fabrication technique
Diffusion bonding	High	The sheets having large areas, blades and turbine vanes, etc., are produced by this method

2.6 Strengthening Mechanisms of Al Composites

2.6.1 Selection of Fabrication Technique

Researchers around the world are continuously applying different innovative techniques to develop Aluminum-based metal matrix composites with the inclusion of various reinforcement particles in nano and micro sizes. Al composites can be fabricated using a variety of processing methods, including stir casting, ultrasonic assisted casting, compo-casting, powder metallurgy, etc. (Sharma, Bhandari and Bretotean, 2021), (Efzan, Syazwani and Bakri, 2016) and (Rajesh et al. 2016). Among these techniques, stir casting is relatively easy to use and inexpensive for the production of Al composites. Up to 30% volume fractions of reinforcement, aluminum composites can be developed using the stir casting method (Gebre, 2020) and (Kumar and Jayahari, 2018). However, the fundamental drawback of the stir-casting method is the porosity that results from density variations and the distribution of the reinforcement materials in the matrix, which may prevent it from being fully homogeneous (Sable and Deshmukh, 2012). The non-homogeneous dispersion of reinforcement particles in the matrix reduces the material properties of the composite in many aspects. Few techniques, such as two-step stir casting, can mitigate or at least reduce the drawbacks of single stir-casting (Reddy and Srinivas, 2018) and (Gopalakrishnan and Murugan, 2012). These criteria led to the two-step stir casting method being chosen as the fabrication method to develop Al composites. The stir-casting process and parameters such as stirring speed, stirring duration, casting temperature, preheating temperature, the effect of squeeze pressure, reinforcement size, etc. can have effects on developed Al composites, which need to be identified and addressed.

2.6.2 Selection of Process & Parameters

The stir-casting process and associated parameters such as stirring speed, stirring duration, casting temperature, preheating temperature, the effect of squeeze pressure, reinforcement size, etc. have a variety of effects on developed Al composites which will be discussed here sequentially. In the case of stirring speed, there is no room for the reinforcement particles (dispersed phase) to scatter evenly throughout the matrix due to reduced shearing force (Aqida, Ghazali and Hashim, 2003). With a faster stirring speed, there is a potential that the matrix's porosity will increase as the gas particles move

around inside it. Therefore, an optimum stirring speed should be chosen to avoid such circumstances. The duration of stirring is crucial in ensuring that the dispersed phase is distributed evenly throughout the matrix. The clustering of reinforcement particles is brought on by shorter stirring times (Mathur and Barnawal, 2013).

The casting temperature has one of the factors that have the biggest impact on the stir casting process. The viscosity of the matrix metal reduces as the temperature rises, and the particle distribution is also impacted. By raising the melt temperature, the chemical reaction between the metal matrix and the reinforcing particles is sped up (Kumar and Parshuram, 2013). According to the microstructure analysis of numerous study articles, the reinforcing particles were discovered to be evenly distributed between 750°C and 800°C for casting. Due to variations in the viscosity of the liquid Al matrix, the particle agglomerations were seen at processing temperatures of 700°C, 850°C, and 900°C (Sozhamannan, Prabu and Venkatagalapathy, 2012).

Preheating the base metal and reinforcing material is essential to reducing porosity. Base metal is preheated to 500°C for an hour because preheating is needed to release the trapped gases from the metal and reinforcing particles (Saravanakumar et al. 2016). The MMC strength is impacted by the size of the reinforcement. The size of the reinforcement has an inverse relationship with strength. Strength improves as reinforcement size decreases (Kumar, Singh and Chaudhary, 2019). With the heat being dissipated from various dies, the squeeze pressure accelerates cooling (Sozhamannan, Prabu and Venkatagalapathy, 2012). Additionally, it reduces the nucleation of gas bubbles, which decreases porosity (Seo and Kang, 1995).

2.6.3 Selection of Reinforcement

The following types of reinforcements can be applied to aluminum composites to further improve their mechanical properties:

2.6.3.1 Ceramic-based Reinforcements:

To improve the mechanical properties of aluminum composites, ceramic-based reinforcements like silicon carbide (SiC), zinc oxide (ZnO), aluminum oxide (Al₂O₃), and titanium diboride (TiB₂) have been widely used. For instance, Han et al. (2017) created Al/SiC composites and discovered that SiC particles greatly enhanced the composites' mechanical properties, such as tensile strength, yield strength, and elastic modulus.

Al₂O₃-reinforced aluminum composites exhibited better mechanical properties, such as hardness, compressive strength, and wear resistance, according to Zhang et al. (2021).

2.6.3.2 Carbon-based Reinforcements:

The mechanical properties of aluminum composites have also been significantly improved by the addition of carbon-based reinforcements such as carbon nanotubes (CNTs) and graphene. As an illustration, Huang et al. (2019) created Al/CNT composites and discovered that the CNT addition considerably enhanced the composites' mechanical properties, such as tensile strength, yield strength, and hardness. Similarly, to this, Zhang et al. (2020) stated that aluminum composites reinforced with graphene oxide (GO) displayed enhanced mechanical properties, such as tensile strength and yield strength.

2.6.3.3 Metal-based Reinforcements:

The mechanical properties of aluminum composites have also been improved by the addition of metal-based reinforcements including Mg, Zn, and Cu. For instance, Wang et al. (2019) created composites made of Al and Mg and discovered that the addition of Mg particles considerably increased the composites' hardness and tensile strength.

In summary, the mechanical properties of aluminum composites can be improved by using reinforcements made of metal, carbon, and ceramic materials. Aluminum composites frequently use SiC, ZnO, Al₂O₃, TiB₂, CNTs, graphene, Mg, Zn, and Cu as reinforcements. To attain the desired mechanical properties of aluminum composites, additional study is required to optimize the size, distribution, and composition of the reinforcements.

2.6.4 Size of Reinforcement Particles

Reinforcement particle size is an essential factor that affects the mechanical properties of aluminum composites. The incorporation of reinforcement particles of different sizes can lead to different strengthening mechanisms and affect the overall properties of the composites. The effect of reinforcement particle size on the mechanical properties of aluminum composites are as follows:

2.6.4.1 Nano-sized Reinforcements

The impact of nanoscale reinforcements on the mechanical characteristics of aluminum composites has been the subject of numerous investigations. According to Jia et al.

(2018), the addition of nano-sized aluminum nitride (AlN) particles greatly improved the yield strength and ultimate tensile strength of aluminum composites. Similarly to this, Wang et al. (2020) discovered that silicon carbide (SiC) nanoparticles added to aluminum composites increased their ductility and strength. In addition, according to earlier research (Jiang et al., 2017; Huang et al., 2020), adding nano-sized reinforcements can enhance the wear resistance, corrosion resistance, and fatigue strength of aluminum composites.

2.6.4.2 Micro-sized Reinforcements

In-depth research has also been done on the use of micro-sized reinforcements in aluminum composites. The hardness and wear resistance of aluminum composites supplemented with micro-sized silicon carbide (SiC) particles were shown to be greatly improved by the addition of SiC particles by Zhang et al. (2018). Similarly, to this, Zhao et al. (2019) found that the tensile strength and ductility of aluminum composites were increased by the inclusion of micro-sized titanium diboride (TiB₂) particles. The addition of micro-sized reinforcements can improve the heat conductivity and fatigue strength of aluminum composites, according to additional research (Li et al., 2018; Dong et al., 2021).

As a whole, aluminum composites mechanical properties can be enhanced by adding reinforcements that are both nano- and micro-sized. Aluminum composites can benefit from nano-sized reinforcements to increase their strength, ductility, and wear resistance, while micro-sized reinforcements can boost their hardness, tensile strength, and thermal conductivity. Depending on the particular application and the desired mechanical properties of the aluminum composite, the ideal size of reinforcing particles will vary.

2.6.5 Weight % volume fraction of Reinforcement Particles

Due to their enhanced mechanical qualities, such as increased strength, stiffness, and wear resistance, nanoparticles have emerged as a desirable material for the reinforcement of aluminum composites. Al₂O₃ and ZnO have been employed frequently as reinforcing elements for aluminum composites among the numerous types of nanoparticles. In this examination of the literature, we will talk about the weight percentages of ZnO and Al₂O₃ nanoparticles utilized to make aluminum composites between 2017 and 2022.

Al₂O₃ nanoparticles were employed to strengthen aluminum matrix composites in a study by Kumar et al. (2017). Between 0.5 and 3 weight percent of Al₂O₃ nanoparticles were

utilized by the researchers. They discovered that the mechanical properties of the composites were improved by the inclusion of Al₂O₃ nanoparticles, with the largest improvement seen at 2 wt% of Al₂O₃ nanoparticles.

ZnO nanoparticles were used as a reinforcing material for aluminum matrix composites in a different work by Li et al. (2018). The ZnO nanoparticles utilized by the researchers ranged in weight percentage from 0.5 to 3 wt%. They discovered that adding ZnO nanoparticles improved the composites' mechanical characteristics, with the largest increase shown at 1 weight percent of ZnO nanoparticles.

In a more recent study, Al₂O₃ nanoparticles were employed to strengthen aluminum matrix composites by Zhao et al. (2021). Al₂O₃ nanoparticle weight percentages ranging from 0.5 to 4 wt% were used by the researchers. They discovered that the mechanical properties of the composites were improved by the inclusion of Al₂O₃ nanoparticles, with the largest improvement seen at 2 wt% of Al₂O₃ nanoparticles.

ZnO nanoparticles were utilized as a reinforcing element for aluminum matrix composites in a different recent study by Wang et al. (2022). The ZnO nanoparticles utilized by the researchers ranged in weight percentage from 0.5 to 2 wt%. They discovered that adding ZnO nanoparticles improved the composites' mechanical characteristics, with the largest increase shown at 1 weight percent of ZnO nanoparticles.

According to the literature, the ideal weight percentage of ZnO and Al₂O₃ nanoparticles for the creation of aluminum composites lies between 1 and 2 wt%. The ideal weight %, however, may change depending on the kind and size of nanoparticles employed, as well as the fabrication technique for the composites.

2.7 Influence of Heat Treatment

2.7.1 Purpose of Heat Treatment on Aluminum Composites

Material is heated and cooled during a heat treatment procedure to change its mechanical and physical properties. Heat treatment has many advantages and benefits for aluminum composites. Heat treatment is primarily used to enhance the mechanical characteristics of aluminum composites, such as strength, hardness, ductility, toughness, and corrosion resistance. This is accomplished by altering the material's microstructure through heat treatment.

The careful tuning of the grain structure is one of the key advantages of heat-treating aluminum composites. The mechanical qualities of the material are improved by the heat treatment procedure, which encourages the production of smaller, more uniform grains. The bonding between the aluminum matrix and the reinforcing particles can also be strengthened through heat treatment, which boosts performance in general.

Heat treatment also has the substantial advantage of making a material stronger and harder. Precipitation hardening is the process of rapidly cooling a material to a supersaturated solid solution after heating it to a predetermined temperature. As a result, precipitates start to form, which makes the material stronger and harder.

Additionally, residual tensions that might have arisen during the production process can be relieved by heat treatment. This could enhance the material's overall functionality and toughness. Additionally, heat treatment can improve a material's thermal and electrical conductivity. By reducing the number of alloying elements present in the material, solution treatment can increase electrical conductivity.

Refinement of the grain structure, greater strength, and hardness, improved bonding between the matrix and the reinforcing particles, stress relief, and improved electrical and thermal conductivity are all advantages and enhancements of heat treatment on aluminum composites. The composition of the aluminum composite, the intended purpose, and the desired qualities will all influence the precise heat treatment method that is employed.

2.7.2 Types of Heat Treatment on Aluminum Composites

Depending on the precise material composition and the desired qualities, a variety of heat treatment methods can be applied to aluminum composites. Typical heat treatment procedures for aluminum composites include the following:

- I. **Solution Treatment:** To dissolve any precipitates and produce a uniform solid solution, the material is heated to a high temperature, usually over the melting point. In order to stop the development of fresh precipitates, the material is then quickly cooled to room temperature.
- II. **Precipitation Hardening:** Also referred to as age hardening, this method entails heating the substance to a particular temperature in order to encourage the production of precipitates inside the aluminum matrix. The precipitates are then trapped in the aluminum matrix by quenching the material to room temperature, which increases its strength and hardness.

- III. Annealing: In this procedure, the material is heated to a specified temperature, held there for a predetermined amount of time, and then slowly cooled to room temperature. The material is softened during this procedure, which also increases the material's ductility, toughness, and machinability.
- IV. Stress Reduction: To release any lingering tensions that might have formed during the manufacturing process, the material is heated to a temperature below its melting point. The possibility of distortion is then reduced by allowing the material to progressively cool to ambient temperature.
- V. Homogenization: This procedure involves heating a material to a high temperature, holding it there for a set amount of time, and then slowly cooling it to eliminate chemical segregation in the substance. The distribution of alloying elements in the material becomes more uniform as a result of this procedure.

To achieve the necessary qualities and performance, each of these heat treatment procedures can be customized to the unique requirements of the aluminum composite material.

2.7.3 Process of Heat Treatment:

The process of heat treatment on aluminum composites are:

- i. Heat the material: The material is then heated to a specific temperature to dissolve any precipitates and create a homogeneous solid solution. The exact temperature and time of the heat treatment process will depend on the specific composition of the material and the desired properties.
- ii. Soak the material: The material is held at the specified heat treatment temperature for a specific period of time to allow for the dissolution of the precipitates.
- iii. Quench the material: The material is rapidly quenched in water or a suitable quenching medium to prevent the formation of new precipitates and freeze the material in the desired solid solution state.
- iv. Age the material: In some cases, the material may be subjected to a subsequent aging process to promote the precipitation of new phases within the aluminum matrix and further improve the material's mechanical properties.

The process parameters of heat treatment can have a significant impact on the electro-mechanical properties of the material. The process followed by other researchers on heat treatment of Al composite is shown in Table 2.3.

Table 2.3: Process Parameters of Heat Treatment on Al Composites

SL	Alloy	Heat Treatment Details				Reference
		Solution Temp(⁰ C)	Duration (Hr.)	Aging Temp (⁰ C)	Duration (Hr.)	
1	Al 6063	412	1	181	2	Özyürek et al. (2014)
		520	1	181	2	
2	Al Alloy LM4	510, 520, 530	1/2,1, 2	160,170,180	2,4,6	Salleh et al. (2020)
3	Al 356	540	1	180	1	Hashim et al. (2019)
4	Al 6061	558	1	140,160,180,200,220,240	0,0.5,1,2,3,4,5,6,8,10,20	Rajasekaran et al. (2012)
5	Al LM25	520	8	165	4,6,8	Sam et al. (2020)
6	Al 356	540	12	155	5,10,15	Kumar and Sharma (2018)
7	Al 356	465,500, 535	8,7,6	65	8	Bazilah et al. (2020)
8	Al 6061	555	8	175	2,4,6,8,10	Manjunatha et al (2017)
9	Al 7075	490	8	130/150/170/200/240	6	Tiwari et al. (2017)

2.8 Response Surface Methodology (RSM)

The optimization of heat treatment procedures and the prediction of the electro-mechanical properties of Al composites following heat treatment have both been done using a variety of prediction and optimization methods. Response surface methodology (RSM) has been widely utilized by scientists and engineers to determine the best parameter settings to enhance process and equipment designs since it was first proposed by Box and Wilson in 1951 (Myers, Montgomery and Anderson, 2016). The design of the experiment (DoE) approach is used by the RSM to gather data and pinpoint important

interactions and factors that affect the process response. The development of a mathematical model to represent the causal relationships between causes and responses is then done using RSM. In order to get optimal factor settings, RSM ultimately optimizes the causality model as the objective function. This technique, which is frequently used for process optimization, is effective in settings where engineers have full control over the levels and treatments of the factors, such as in laboratory experiments, applications of the scientific method, computer experiments, and any other research environments where there are controllable factors at play. RSM gives engineers a way to determine the ideal parameter settings for enhancing the characteristics of particular industrial processes or design optimization. The optimal way for the RSM to function is to be based on experimentation activities, provided engineers have the opportunity to set the process/equipment parameter.

However, carrying out planned trials for continuous process/production is difficult. Changing the parameters while a process is operating can disrupt production, increase the number of nonconforming products, and raise costs (Sukthomya and Tannock, 2005). Using observational data as the input to the RSM is one of the other solutions when direct testing is not practical (Chien, Chang and Wang, 2014), (Sadati, Chinnam and Nezhad, 2018). Intelligent data-acquiring systems are frequently added to some high-tech companies, enabling them to track changes in process or equipment parameters in real-time. These real-time recorded data are used as the input for a mathematical model to produce predictions, such as a system for anticipating maintenance schedules or product quality (Cerquitelli, 2021). Numerous studies (Hussain et al., 2021), (Fazeli, Afkari and Taghinezhad, 2020) on chemical engineering and food production have shown that observational-data-based RSM (RSM-OD) provides a fitted mathematical model to determine the ideal factor configuration. As demonstrated in the work of (Garg and Singh, 2017) for steel production and (Mahmoodi, Taghizadeh and Taghizadeh, 2019) with pollution removal operations, other studies employed the observed data from a running process or piece of equipment as the input for RSM-OD.

A researcher's ability to manipulate their factor levels, as the DoE ideally allows, is nonetheless limited by observational data and their similarities, including real-time recording data and already completed experiment data. Observational data are assumed to comprise circumstances that are serially correlated, high volume, and variable (Demchenko, Laat and Membrey, 2014). As a result, various adjustments to the selection

of the observations are necessary before the data are used in the RSM analysis, including the adaptive RSM model and optimization approaches, while still taking into account the ideal RSM concept. By choosing a subset of observations and identifying stages within the data, the authors of Chien, Chang and Wang (2014) have successfully adopted observational data for the DoE; similarly, Refs Khoei, Masters and Gethin (2002) also provide alternatives by matching the data with specific DoE to ensure orthogonality. Furthermore, the current growth of big data should be noted as having hastened the usage of observational data. Once the process parameters and product attributes are present in the obtained dataset, RSM-OD should be taken into account as an optimization tool. However, the literature that has already been written on RSM-OD takes a distinctive approach to handle the observational data and changing the RSM model or processes; as a result, there is still room for developing a well-established RSM-OD.

2.8.1 RSM Overview for Response Optimization

A thorough analysis of the traditional RSM revealed that this approach has recently made important contributions. Strong theoretically based analysis and interpretation will result from a defined experiment-based RSM with satisfied statistical assumptions. Nevertheless, as several works of literature (Berni 2003) have shown that observational data can be successfully included into the RSM, the idea of integrating it should not be disregarded.

2.8.2 Classic RSM

According to Figure 2.11, the three techniques that make up traditional RSM are combined in a sequential analysis. The DoE is implemented in the first stage using traditional RSM. The DoE participates in experiment preparation, data collection, analysis, and interpretation in this step and makes sure the experiment achieves its goals. The predetermined process parameters can be evaluated separately among other parameters thanks to the DoE matrix's orthogonality fulfillment. Second, traditional RSM uses a particular mathematical model to fit the data gathered by the DoE. The link between components or parameters as inputs and responses as outputs are captured by this model. Because of its straightforward interpretation and formal statistical inference of all its necessary assumptions during the modeling step, the classic RSM typically prefers to adopt a linear model. Finding the factor (or parameter) setting to optimize the answer is how the optimization stage, third, operates.

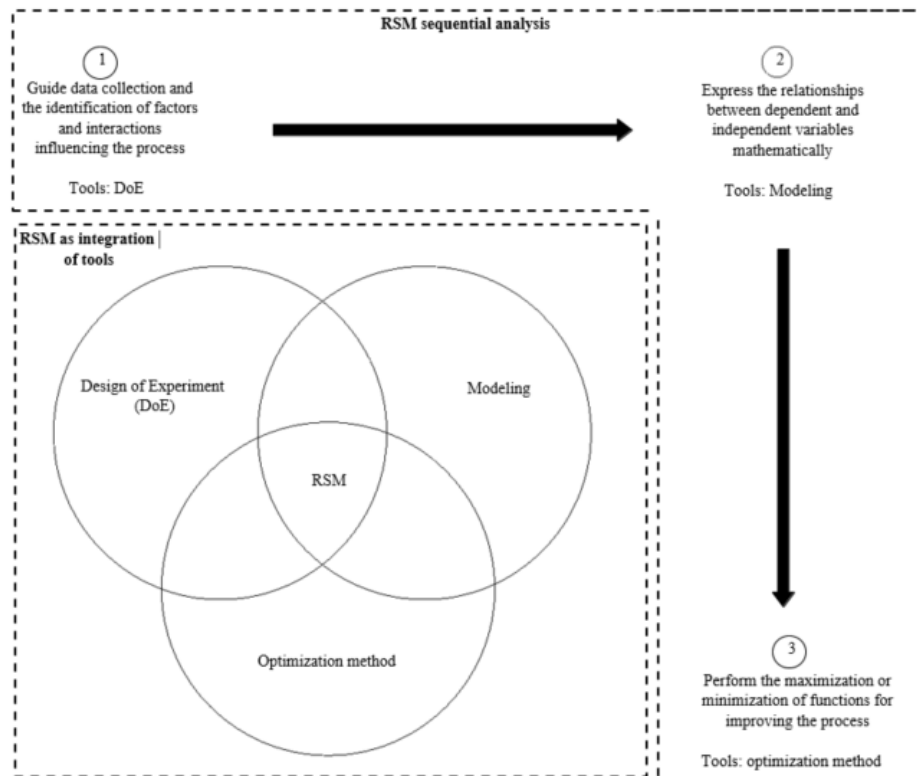


Figure 2.11: Overview of RSM (Oliveira et al. 2019).

The classic RSM has been effectively used for many years in numerous research fields as a well-established tool for planned, experimentally-based optimization. More than 48,000 Scopus-indexed studies used traditional RSM as a starting point after the idea put forth by Box and Wilson (1951).

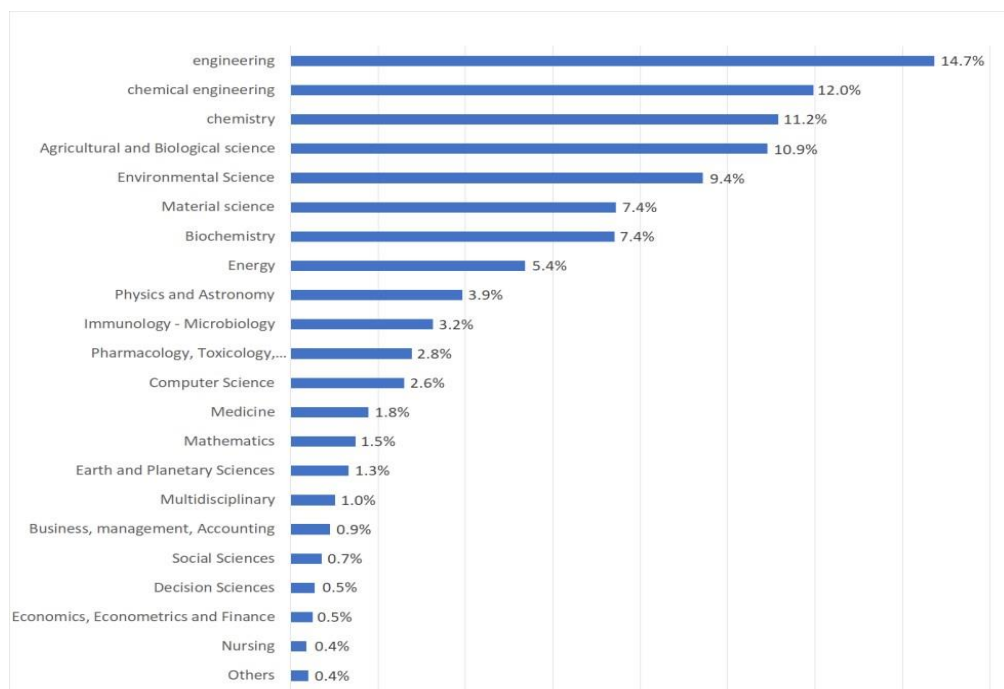


Figure 2.12: Research fields applying RSM (Hadiyat, Sopha and Wibowo, 2022).

Figure 2.12 demonstrates that the percentage of RSM applications is dominated by general engineering fields. This indicates that RSM is a key optimization tool in many research fields and that RSM has undergone significant progress to address current research concerns.

CHAPTER 3

EXPERIMENTAL SETUP AND CHARACTERIZATION TECHNIQUES

3.0 Introduction

In this study, the influence of Alumina (Al_2O_3) and Zinc Oxide (ZnO) nanoparticles on the microstructure and electro-mechanical properties such as flexural strength, impact toughness, hardness, and electrical conductivity of aluminum metal matrix composites (Al MMCs) will be carried out. Two types of Al composites will be fabricated by a customized two-step stir casting method, (i) Al MMC-01 having 97.5 wt. % of Al and 2.5 wt. % of Al_2O_3 and (ii) Al MMC-02 having 95 wt. % of Al, 2.5 wt. % of Al_2O_3 and 2.5 wt. % of ZnO . The fabricated Al composites will undergo various heat treatment processes at different combinations of temperatures. After completion of the above heat treatment process, an investigation of electrical conductivity and hardness will be carried out of Al composites. Finally, a comparison will be made on the electrical conductivity and hardness of Al composites between as-casted and heat-treated conditions.

3.1 Materials Under Study

The base metal used for the casting of composites is aluminum ingots collected from RUSAL of Russia. The reinforcement particles used for the synthesis of composites are nanoparticles such as Alumina (Al_2O_3) and Zinc Oxide (ZnO) provided by Hebei Suoyi New Material Technology Co. Ltd. in China. The details of these components/ingredients used for the present study are briefly described below.

3.1.1 Aluminum

Aluminum (Al) has been becoming increasingly popular across a variety of industries, including aerospace, automotive, space, etc. due to their superior strength-to-weight ratio, cost-effectiveness, and ample supply of Al on a worldwide scale. The chemical composition analysis of the Aluminum used for the synthesis of Al composites is analyzed by the XRF Analyzer of Olympus, Model: Vanta C Series and the report obtained from the XRF analyzer is attached in Appendix A-1. The chemical composition results are also presented below in Table 3.1.

Table 3.1: Composition of Aluminum

Element	Al	Si	Fe	Cu	Zn	Zr	Pb
Percentage (%)	99.052	0.614	0.323	0.002	0.008	0.0007	0.0009

3.1.2 Aluminum Oxide (Al₂O₃)

Aluminum oxide, more commonly referred to as alumina, is a chemical compound composed of aluminum and oxygen, characterized by the chemical formula Al₂O₃. Alumina holds considerable significance in the production of aluminum metal matrix composites. This is primarily due to its abrasiveness, stemming from its remarkable hardness, as well as its refractory properties, resulting from its exceptionally high melting point.

A comprehensive technical specification detailing the properties of alumina (Al₂O₃) can be found in Appendix B, generously provided by Hebei Suoyi New Material Technology Co. Ltd. in China. In Table 3.2, we present the physical characteristics of the Al₂O₃ material acquired for this study.

Table 3.2: Physical Properties of Alumina (Al₂O₃) as per maker's manual

Grain Size (nm)	20
Melting point (°C)	2072
Boiling point(°C)	2977
Limit of application (°C)	1175
Hardness (Moh's Scale)	7.5
Linear coefficient of expansion (µm/m ⁰ C)	4.5
Fracture toughness (MPa-m ^{1/2})	3.5
Colour	white
Molecular weight (g/mol)	101.96
Thermal conductivity (W.m ⁻¹ -K ⁻¹)	30

3.1.3 Zinc Oxide (ZnO)

ZnO is a white powder that is essentially water-insoluble. To customize the mechanical properties of aluminum metal matrix composites, ZnO is employed as reinforcement. The

detailed technical specification of Zinc Oxide (ZnO) is attached in Appendix-C which was provided by Hebei Suoyi New Material Technology Co. Ltd. in China. Table 3.3 shows the physical characteristics of ZnO collected for the present study.

Table 3.3: Physical Properties of Zinc Oxide (ZnO) as per maker's manual

Grain Size	30 nm
Melting point ($^{\circ}\text{C}$)	1,974
Boiling point($^{\circ}\text{C}$)	1,974
Hardness (Moh's Scale)	4.5
Bulk Density (g/cm^3)	.15-.3
Fracture toughness ($\text{MPa}\cdot\text{m}^{1/2}$)	2.44
Colour	White
Molecular weight (g/mol)	81.406
Thermal conductivity ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$)	50
Odor	Odorless
Flashpoint ($^{\circ}\text{C}$)	1,436

3.2 Experimental Setup

In this experiment, stir casting has been selected for the synthesis of Al MMCs among a variety of processing methods, including ultrasonic assisted casting, compo-casting, and powder metallurgy as per section 2.5.4 as stir casting is relatively easy to use and inexpensive for the production of Al composites. A dedicated stirring mechanism has been designed and developed to apply the stir-casting process and parameters such as stirring speed, stirring duration, casting temperature, preheating temperature, reinforcement size, etc. can have effects on developed Al composites.

3.2.1 Casting Arrangement

On the basis of the notable factors and their parameters on the effects of stirring casting after studying the various research works as per section 2.6.2 Selection of Process & Parameters such as stirring speed, stirring duration, casting temperature, preheating temperature, reinforcement size, etc., the finalized parameters of Stir casting for the development of Al composites are shown in Table 3.4.

Table 3.4: Selected Process & Parameters for Stir casting of Al Composites

SL No	Casting Arrangement	Casting parameter
1	Base Metal	99% pure Al
2	Casting Method	Two Step Stir Casting
3	Base Metal Preheat	500°C
4	Base Metal Preheat Time	60 minutes
5	Casting Temp	800°C
6	Reinforcement	Al ₂ O ₃ and ZnO
7	Reinforcement Particle Size	Al ₂ O ₃ : 20 nm and ZnO: 30 nm
8	Reinforcement Preheat	300°C
9	Reinforcement Preheat Time	120 mins
10	Stirrer RPM	400 RPM
11	Stirring Time	05 minutes in two steps

3.2.2 Design and Development of Stirring Mechanism

A dedicated small version of the stirring mechanism has been designed and developed to apply the effects of selected casting parameters such as stirring speed, stirring time, Squeeze Pressure, etc. during the synthesis of Al composites for the present research purpose. The stirring mechanism was developed using two major parts, i.e., a power drive and a stirring rod with an impeller (mixer head) as shown in Figure 3.1. For the power drive part, one multi-speed handheld mixer machine motor was used having a rated voltage of 220V, power of 2100w, frequency of 50/60 Hz, and for the stirring part, a rod of about 1220 mm length was connected with an impeller of 150 mm m diameter. This mixer machine had 6 gears to operate at different rotational speeds, i.e., 100 rpm to 600 rpm with an interval of 100 rpm for each gear. For the present work of the synthesis process, the 4th gear having 400 rpm was selected as the optimum one after a few trials with speed variations. A detachable arrangement was made to remove the mixer head from the motor part for cleaning purposes.

Since the crucible furnace was supposed to be set at 800°C for casting the metal matrix composite, the materials were selected in such a way that the stirring rod and mixer head can sustain at such an elevated temperature. Therefore, the mixer head was made of stainless steel (SS) and the mixer rod was made of mild steel (MS) as their melting point

are about 1500°C and 1300°C, respectively. Figure 3.1 shows the driving part and the stirring part along the dimensions of developed stirring gear used for casting purposes in the crucible furnace.

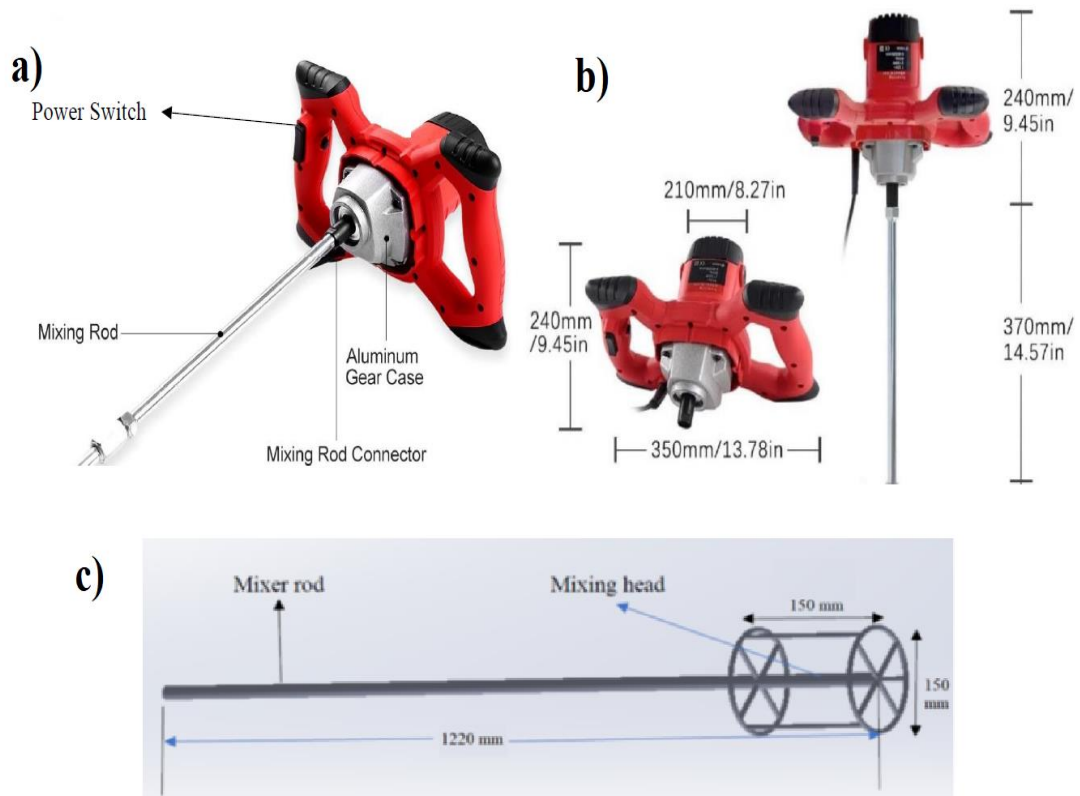


Figure 3.1: Design and Development of Stirring Mechanism; (a) Component details of Stirrer, (b) Dimensions of Stirrer machine, (c) Dimension of mixer rod & head

3.2.3 Synthesis of Aluminum Composites

The stir casting was done in a gas-fired crucible furnace which could sustain very high temperatures even more than 3500°C. An external air blower was used to supply a sufficient amount of air for maintaining a steady temperature during gas burning. The step-by-step process of Stir casting is shown in Figure 3.2.

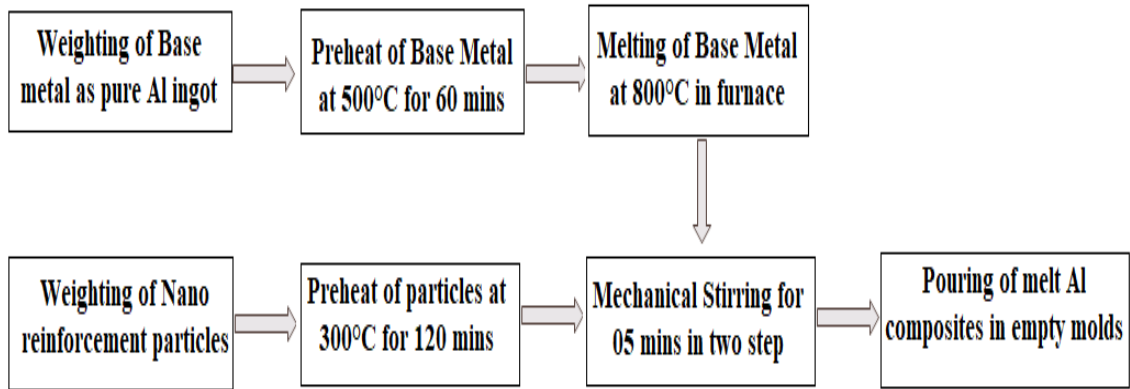


Figure 3.2: Flowchart of the Stir Casting Process

The aluminum was initially placed in the crucible and heated without activating the blower, gradually reaching a temperature of around 300°C. After 15 minutes, the electric blower was turned on, and the base metal was preheated at 500°C for 60 minutes. Simultaneously, reinforcement particles were preheated in an oven at 300°C for 120 minutes. The metal took approximately 60 minutes to completely melt at a casting temperature of 800°C. Subsequently, a stirring machine was employed for two 5-minute cycles to ensure proper mixing with Al₂O₃, as depicted in Figure 3.3. Once the metal reached the desired state, it was poured into a sand mold, as shown in Figure 3.4(a), to create the Al composite reinforced with Al₂O₃ (Al MMC-01) as illustrated in Figure 3.4 (b). Following the casting of Al MMC-01, ZnO reinforcement particles were added to the crucible and mixed using the same two-step stirring mechanism for 5 minutes. Once the metal was ready again, the Al composite reinforced with both Al₂O₃ and ZnO (Al MMC-02) was poured into a separate sand mold, as illustrated in Figure 3.4 (c). The mixing of Al₂O₃ and ZnO in the crucible was maintained at a rate of 20 gm/minute.

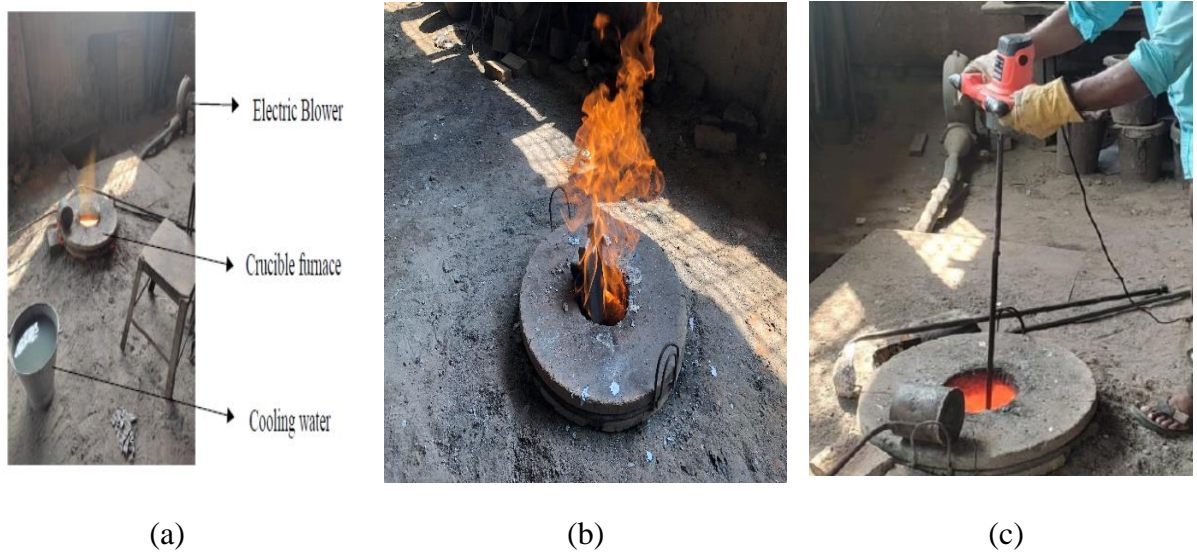


Figure 3.3: Synthesis steps; (a) Casting Arrangement, (b) Base metal preheating and (c) Stirring during casting.

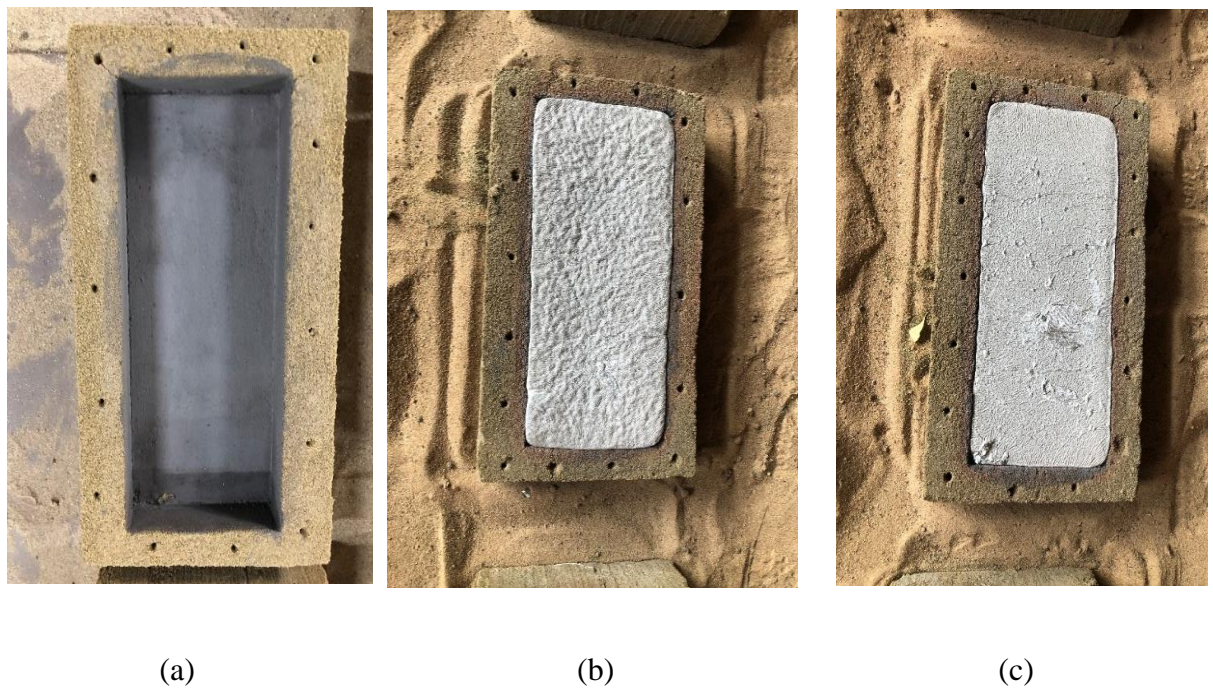


Figure 3.4: Sand Mold; (a) Empty, (b) Al MMC-1 pouring and (c) Al MMC-2 pouring.

3.3 Preparation of Test Specimen

Test specimens for flexural strength, impact toughness, hardness, and electrical conductivity have been designed in Solid Works as per ASTM standards and prepared by a CNC machine.

3.3.1 Design of Test specimens as per ASTM standard

The ASTM standards for test specimens of Impact Charpy, flexural, Hardness (Rockwell, Vickers Micro, and Brinell), and Electrical conductivity are followed as mentioned in Table 3.5. These test specimens are designed in Solid Works as shown in Figure 3.5 as per ASTM standards mentioned in Table 3.5.

Table 3.5: ASTM test standards for Al Composites

Test Name	Sample Dimension	ASTM Standard
Impact (Charpy)	55mm × 10 mm × 10 mm	ASTM E23-18
Flexural	80 mm × 10 mm × 04 mm	ASTM D790-17
Vickers Micro Hardness	20 mm × 20 mm × 08 mm	ASTM E92-17
Electrical Conductivity	20 mm × 20 mm × 08 mm	ASTM E1004-17
Wear Test	8mm×12mm×5mm	ASTM G99-17

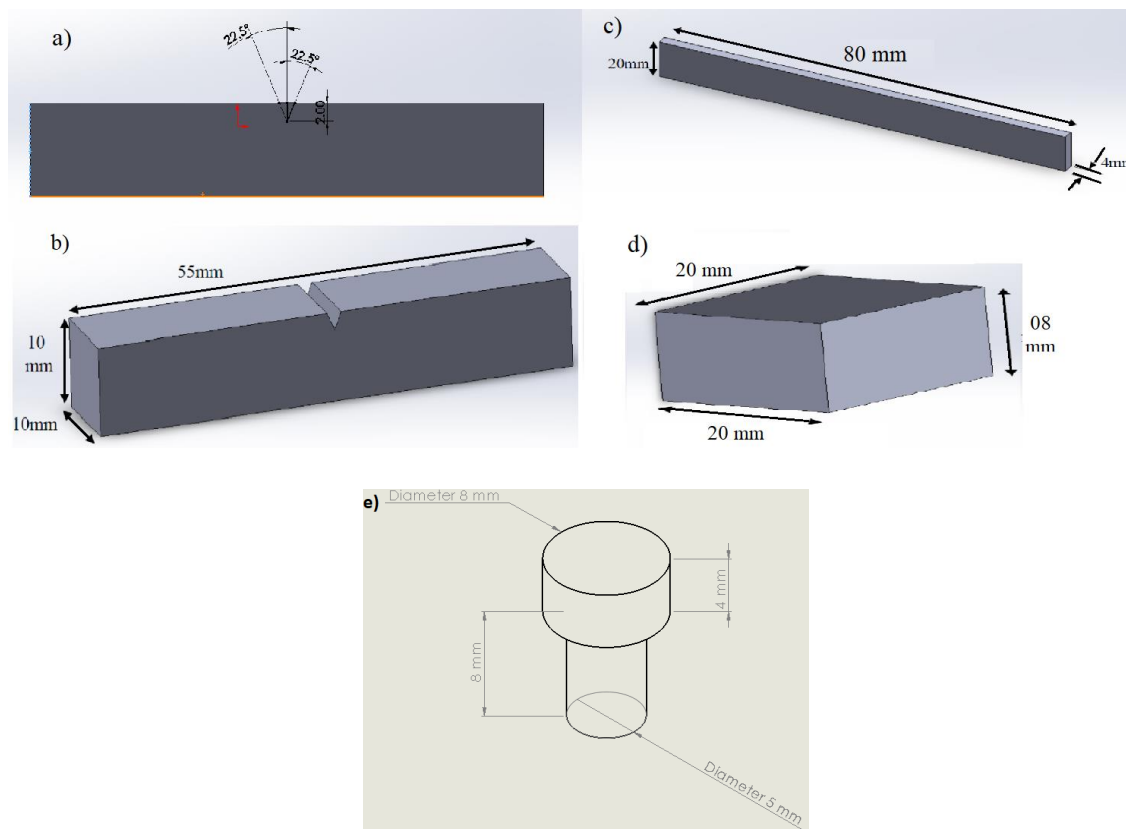


Figure 3.5: Solid works design of specimens as per ASTM Standard; (a) V-notch of Impact (Charpy), (b) Impact (Charpy), (c) Flexural, (d) Hardness and Electrical Conductivity Test, (e) Pin for wear test

3.3.2 CNC Machining for Test Specimens

Test specimens were prepared by a CNC machine, Model VF-2 type as shown in Figure 3.6(a). The cutting tools used for surfacing and machining were respectively 12 mm and 6mm coated with bronze as shown in Figure 3.6(b). The finished smooth surface of the Al composite is shown in Figure 3.6(c). Test specimens for Impact Charpy and flexural were prepared respectively as per ASTM standards E23-18 and D790-10 as shown in Figure 3.8(d)-(e). The dimension of Impact Charpy and flexural test specimens are respectively 55 mm×10 mm×10 mm and 80 mm×10 mm×4 mm. Test specimens for hardness (Brinell and Vickers micro) and electrical conductivity were prepared respectively as per ASTM standards E10-18, E18-20, E92-17, and E1004-17 with the dimensions of 20 mm×20 mm×08 mm. The specimens are demonstrated sequentially in Figure 3.6(f).

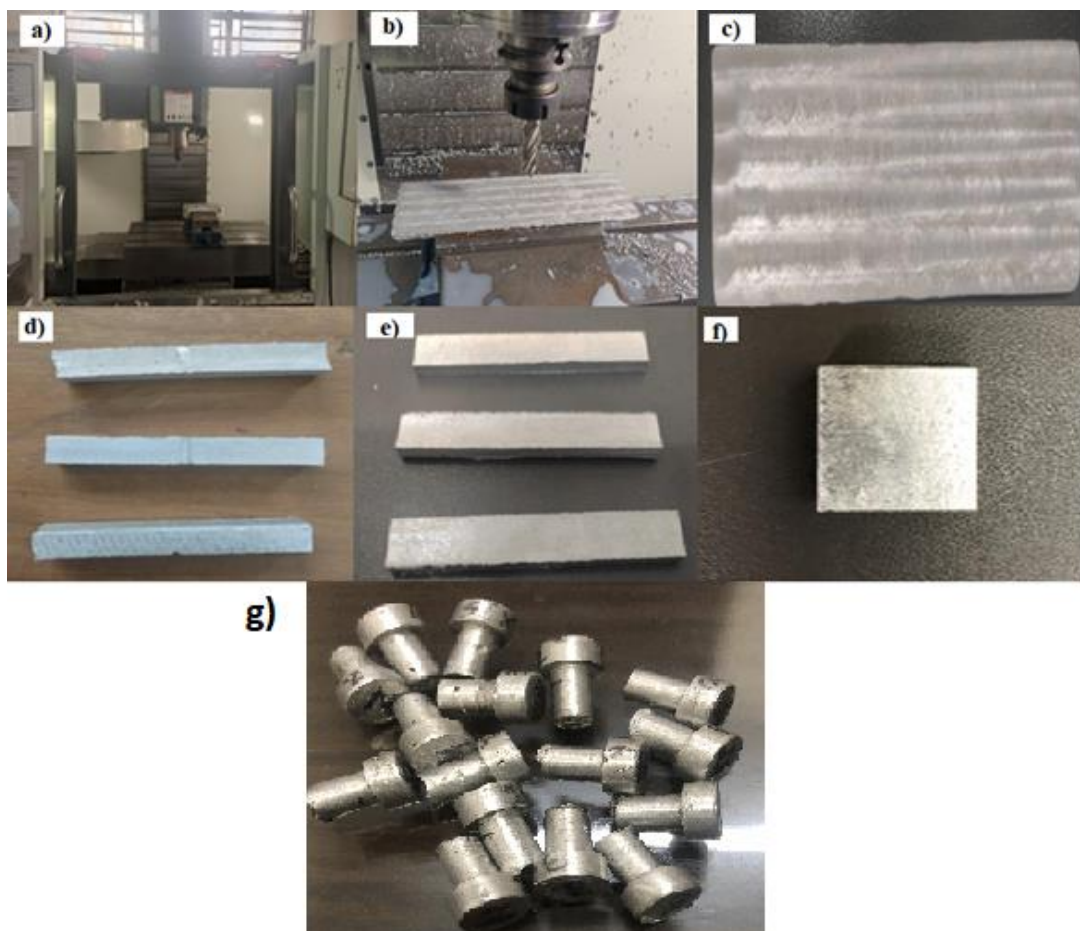


Figure 3.6: (a) CNC machine set up, (b) End Mill cutter, (c) Finished Al composite, (d) Impact (Charpy), (e) Flexural, (f) Hardness and Electrical Conductivity, (g) Cylindrical Pin

3.4 Investigation Procedure

The investigation procedure in this study encompasses a comprehensive evaluation of the mechanical and electrical properties of the fabricated aluminum composites. It involves a range of testing techniques, including Impact Charpy, flexural testing, and the assessment of hardness using Vickers Micro. Additionally, the electrical conductivity of the materials is examined. These investigations are essential for characterizing the performance of the composite materials and gaining valuable insights into their suitability for various applications.

3.4.1 Vickers Micro Hardness

The Vickers microhardness test was carried out by a Vickers hardness tester model: TMHV-10MDT auto turret Vickers hardness tester. The ASTM standard for the test followed was ASTM E92-17. These test methods cover the determination of the Vickers hardness of metallic materials. This standard also provides the requirements for Vickers hardness machines and the procedures for performing Vickers hardness tests. The test specimens for Vickers microhardness were prepared as per ASTM standard E92-17 with a dimension of 20 mm×20 mm×08 mm. The tests were carried out with a 500-gm load for 10 seconds duration. The average value of HV was taken from ten readings.

Micro indentation Vickers hardness is typically determined using indentation test forces in grams-force (gf) and indentation diagonals measured in micrometers (μm). The Vickers hardness number, in terms of gf and μm , is calculated using equation (3.2) as per ASTM E92-17 given as follows:

$$\text{HV} = 1854.4 \frac{\text{F}}{\text{d}^2} \quad (3.1)$$

where:

F= indentation test forces in grams-force (gf) and

d = mean Vickers indentation diagonal length (mm).

3.4.2 Impact Toughness

The Impact Charpy test was carried out as per ASTM E23-18. The test was carried out by an Impact testing machine of Model: AIT300 from Turkey at a room temperature of 26⁰C. The purpose of the test is to investigate the energy being absorbed and ascertain the durability of the developed composite samples. The specimens used for the Charpy test had

a dimension of 55mm×10mm×10mm as per ASTM E23-18. The average value of impact toughness was taken from three readings. To conduct the test, we prepared the machine by raising the pendulum to the latched position, prepared the indicating device, positioned the specimen in the test position, and released the pendulum.

3.4.3 Flexural Strength

The flexural test was carried out as per ASTM D790-17. The purpose of the flexural test is to determine the flexural behavior based on the simple beam load-bearing capacity of developed Al composite materials. It was conducted using test specimens by a Universal Testing Machine as shown at a room temperature of 26⁰C under three-point bending conditions. The dimension of the test specimens was 80mm×10mm×4mm as per ASTM D790-17. During the flexural test, test specimens of the fabricated Al composite are supported at two points, and a load is applied at a third point between the supports, causing the sample to bend as per the figure. The amount of deflection is measured at the midpoint of the sample, and the force required to cause the bending is recorded.

From these tests, mechanical properties of the Al composites such as Ultimate Flexural Strength (UFS), Modulus of Elasticity, etc. were obtained the values of Ultimate Flexural Strength (UFS), and Modulus of Elasticity were obtained as per the calculation methods using Equation 3.3 and 3.4 respectively provided in ASTM D790-17 guideline. The values obtained are taken on average from three test readings. In this regard, the following mathematical expressions were employed as per ASTM D790-17.

$$\text{Flexural Stress } (\sigma_s) = \frac{3PL}{2bd^2} \quad (3.2)$$

Where:

σ = stress in the outer composite at the midpoint, MPa (psi),

P = load at a given point on the load-deflection curve, N (lbf),

L = support span (mm),

b = width of beam tested (mm) and

d = depth of beam tested (mm).

$$\text{Modulus of Elasticity } E_B = \frac{L^3m}{4bd^3} \quad (3.3)$$

Where:

E_B = modulus of elasticity in bending, MPa (psi),

L = support span (mm),

b = width of beam tested (mm),

d = depth of beam tested (mm) and

m = slope of the tangent to the initial straight-line portion of the load-deflection curve, N/mm of deflection

3.4.4 Electrical Conductivity

The electrical conductivity of fabricated Al composites was tested as per ASTM E1004-17. This test method covers a procedure for determining the electrical conductivity of nonmagnetic metals using the electromagnetic (eddy current) method. The procedure has been written primarily for use with commercially available direct-reading electrical conductivity instruments. The test was carried out by an Eddy Current Conductivity meter, model: 12Z from ZAPPITEC PTY LTD, Australia.

The test specimens were prepared as per ASTM E1004-17 with a dimension of with dimension of 20 mm×20 mm×08 mm. The electrical conductivity of a metal depends on several factors, such as its chemical composition and the stress state of its crystalline structure. Also, the conductivity of metals changes significantly with temperature. To allow easy comparison between different metals, conductivity values were taken with a standardized temperature of 20⁰C. The measurement is made in %IACS units, an acronym that means “percent of International Annealed Copper Standard”.

3.4.5 Heat Treatment Process

A Carbolite laboratory chamber furnace, model CWF 13/13 from the United Kingdom, with a maximum operating temperature range of 1300⁰C, was used to perform the heat treatment. During the heat treatment process, the chamber furnace's reported heating rate was 16.66⁰C per minute. The step-by-step procedure of heat treatment is shown in Figure 3.7.

Heat treatment involves solution treatment of test specimens at different temperatures from 500⁰C to 550⁰C and thermal aging at different temperatures from 120⁰C to 240⁰C. All heat

treatment procedures took place for 60 minutes, were followed by water quenching, and were then allowed to naturally age for 72 hours at room temperature in both solution treatment and thermal aging. After the aforementioned heat treatment steps were finished, tests on Vickers micro hardness, Rockwell hardness, and electrical conductivity were done.

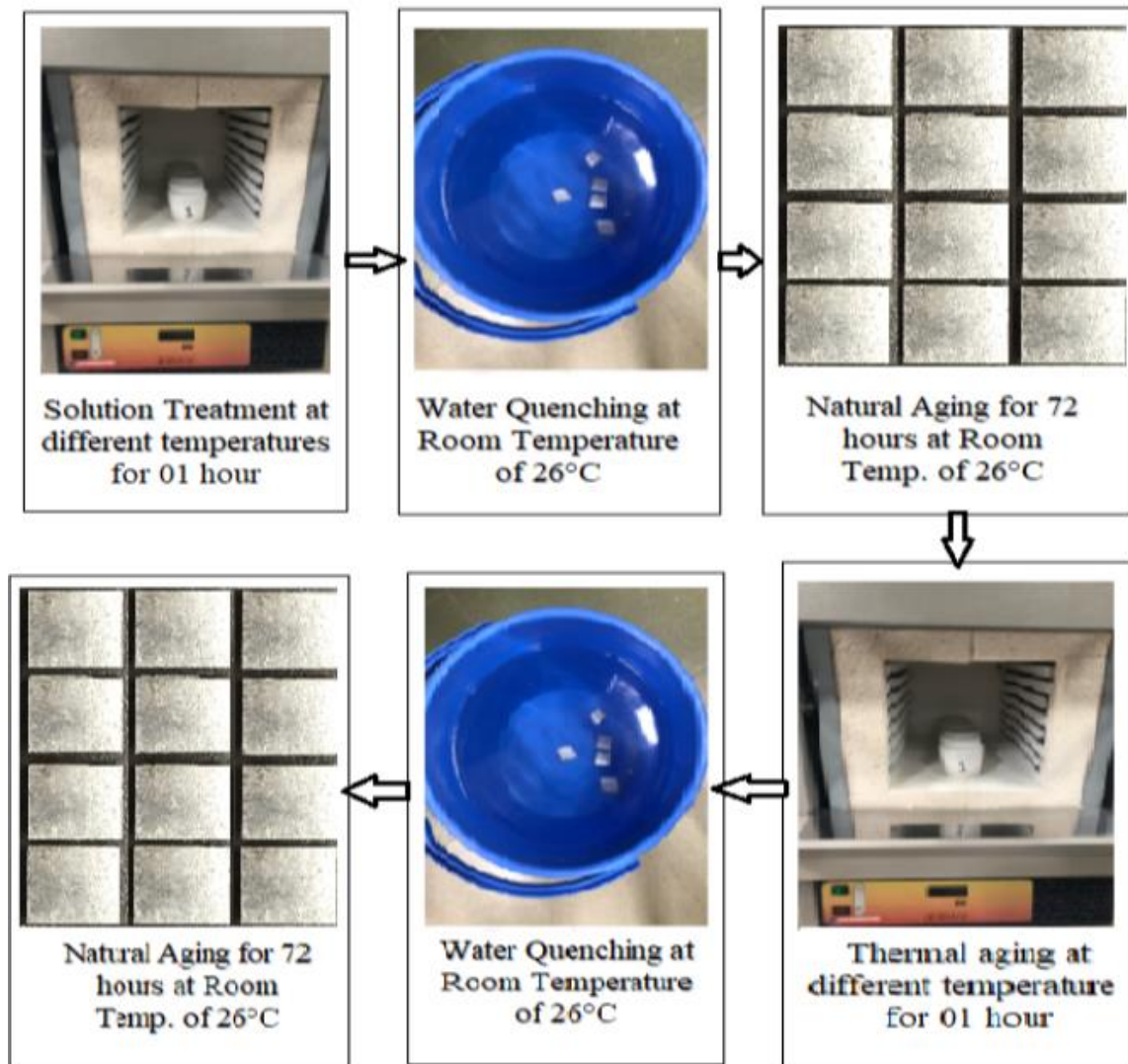


Figure 3.7: Heat Treatment Process of Al Composites

CHAPTER 4

MICROSTRUCTURE AND ELECTRO-MECHANICAL PROPERTIES OF AL COMPOSITES

4.0 Introduction

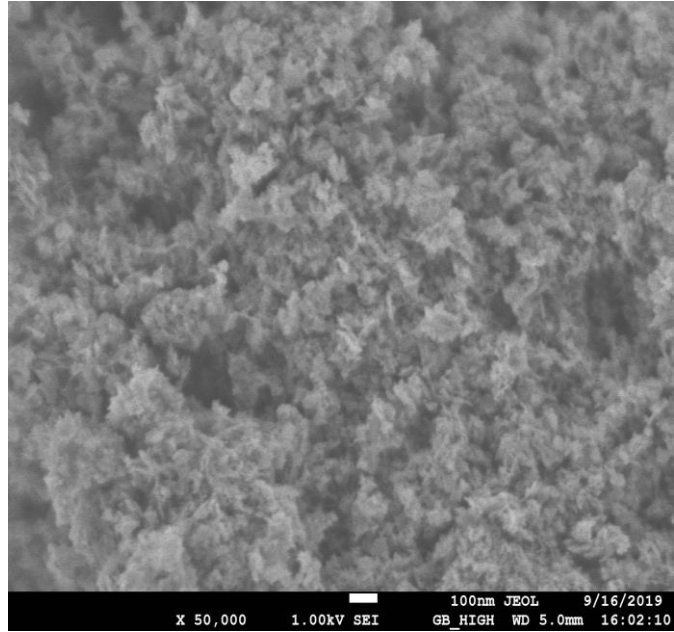
This chapter delves into a comprehensive analysis of the electro-mechanical characteristics of the fabricated materials through an intricate examination of the impact resistance, flexural behavior, and hardness properties of as-casted conditions. Furthermore, the microstructure observation provides critical insights into the distribution and homogeneity of reinforcement particles. This chapter also delves into the transformative effects of heat treatment on the electro-mechanical characteristics and microstructure of Al composites, shedding light on how controlled thermal processes can unlock their full potential. Understanding these changes is crucial for tailoring Al composites to specific needs and optimizing their utility across a spectrum of industries.

4.1 Microstructure Properties As-casted Condition

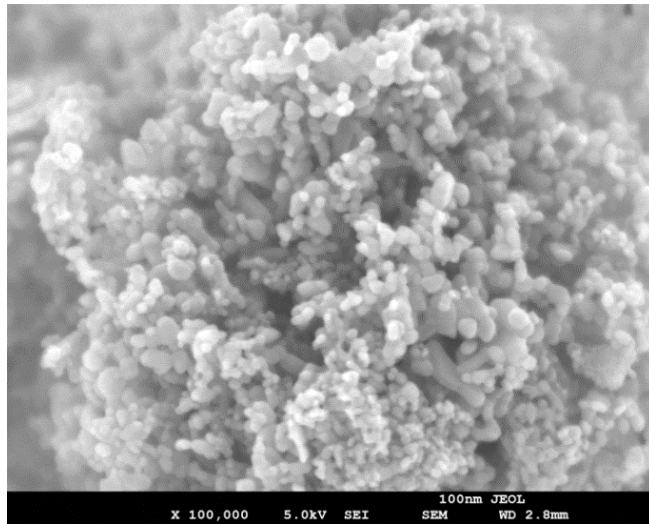
The microstructure observation plays a crucial role in enhancing our understanding of SEM pictures of fabricated Al MMC reinforced with nanoparticles. The microstructure offers essential insights into the distribution, morphology, and interfacial characteristics of nanoparticles within the matrix. This information aids in evaluating the quality of the manufacturing process, ensuring uniform dispersion of nanoparticles, and identifying potential clustering issues that can impact the material's performance.

4.1.1 Microstructure of Nanoparticles

By studying the microstructure of nanoparticles, a deeper understanding is gained of how they interact with the aluminum matrix, contributing to improved control over material properties, structural integrity, and the optimization of Al MMC for specific applications. This examination is vital for understanding how the reinforcement particles such as Al_2O_3 and ZnO affect the overall properties of the fabricated Al MMC-01 and Al MMC-02, including mechanical and electrical characteristics.



(a)



(b)

Figure 4.1: SEM of Reinforcement particle; (a) Al_2O_3 (20 nm) and (b) ZnO (30 nm)

The microstructure observation of the Al_2O_3 nanoparticles (20 nanometers in size) as shown in Figure 4.1 (a) and ZnO nanoparticles (30 nanometers in size) as shown in Figure 4.1 (b) supplied by Hebei Suoyi New Material Technology Co. Ltd. in China is a critical aspect of this study.

4.1.2 Surface Smoothness and Microstructure of Test Specimens

The cutting speed, feed rate, and depth of cut for the CNC machining were maintained as 375m/min, 400m/min, and 1 mm respectively during facing and preparation of test specimens for hardness, flexural & impact strength, and electrical conductivity. The surface roughness of a test specimen is the prediction factor for mechanical performance. Mainly surface irregularities contribute to the breakage and initial formation of corrosion.

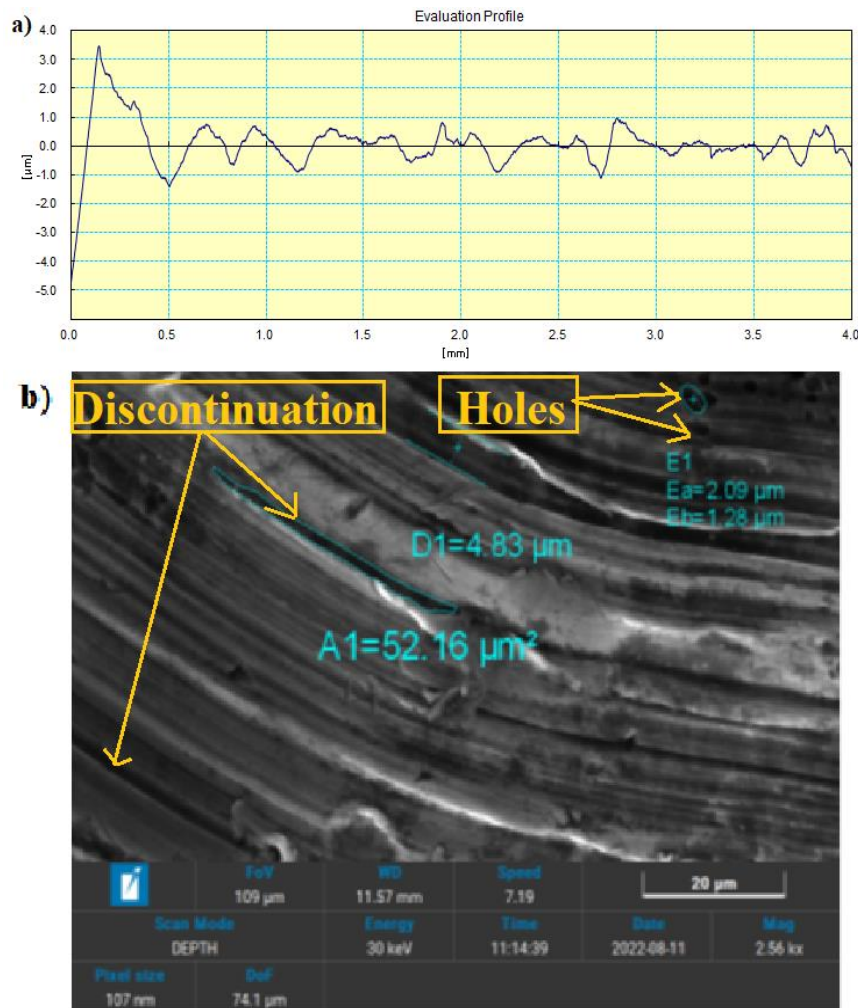


Figure 4.2: Test Specimen of Al MMC-01; (a) Surface roughness, (b) Microstructure observation by SEM

A sample of test specimen from Al MMC-01 has been taken to conduct the surface roughness test by a Mitutoyo roughness tester, Model SJ-210 to identify the possible imperfection and obtained an overall R_a of $0.492 \mu\text{m}$. The evaluation profile of measured surface roughness is shown in Figure 4.2(a).

The microstructure of the same test specimen from Al MMC-01 was also observed by Scanning Electron Microscope (SEM), Model: TESCAN VEGA 4 from the Czech Republic. As shown in Figure 3.9 (b), a sample of test specimen with a dimension of 05mm×05mm×05mm was observed in SEM with FoV: 109 μm, WD:11.57 mm, Speed: 7:19, Energy: 30KeV, Mag: 2.56Kx, Pixel Size: 107 nm, DoF: 74.1 μm. As shown in Figure 4.2 (b), we identified a few discontinuities and a few holes with elliptical sizes of 2.09 μm and 1.28 μm.

4.1.3 Microstructure of Al Composites

After the preparation of the Aluminum metal matrix composite by two-step stir casting, microstructure observation was carried out by SEM in order to confirm the mixing of reinforcement particles (Al_2O_3) and (ZnO) into a base material (99% Al). A sample of Al- Al_2O_3 (Al MMC-01) and Al- Al_2O_3 -ZnO (Al MMC-02) with a dimension of 05mm×05mm×05mm prepared for microstructure observation by Scanning Electron Microscope (SEM), Model: TESCAN VEGA 4.

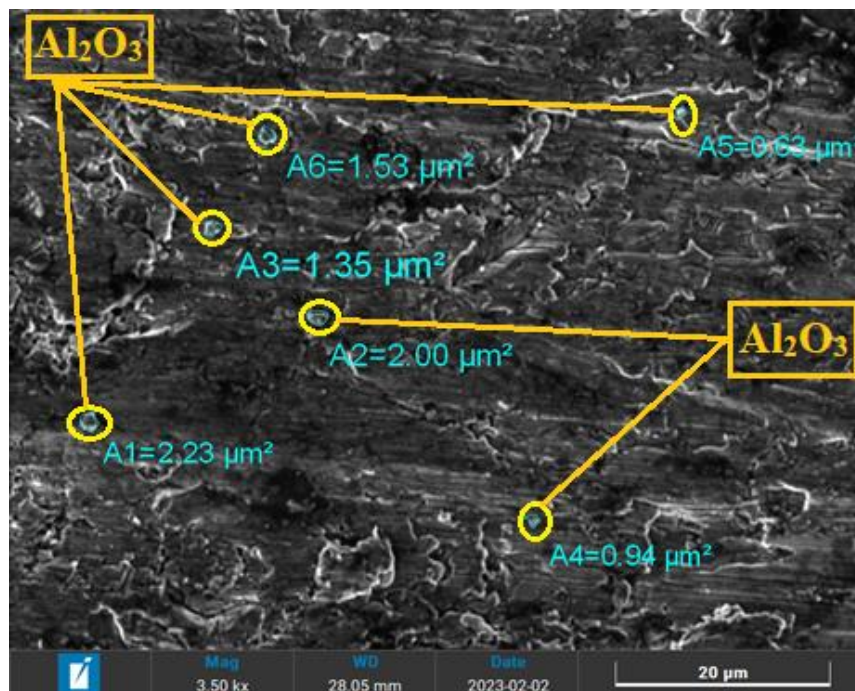


Figure 4.3: Microstructure observation by SEM of Al composite having 97.5% of Al and 2.5% of Al_2O_3 (Al MMC-01)

As shown in Figure 4.3, the morphology of Al_2O_3 particles is mainly irregular or nearly elliptical. The Al_2O_3 particles were uniformly distributed in the Al/ Al_2O_3 (Al MMC-01) as

casted-condition. The elliptical area immersed by the reinforcements (Al_2O_3) in Al MMC-01 is approximately $5.45 \mu\text{m}^2$ and $6.29 \mu\text{m}^2$ which is investigated through image processing as shown in Figure 7. Also, a few clustering or agglomerations of Al_2O_3 particles were perceived in the Al/ Al_2O_3 matrix composite.

As shown in Figure 4.4, the morphology of Al_2O_3 and ZnO particles are also irregular or nearly elliptical in shape. The Al_2O_3 and ZnO particles were uniformly distributed in the Al/ Al_2O_3 -ZnO (Al MMC-02) as a casted condition. As shown in Figure 4.4, the elliptical area immersed by the reinforcements Al_2O_3 and ZnO in Al MMC-02 are respectively 0.37 to $2.78 \mu\text{m}^2$ and $.92$ to $5.76 \mu\text{m}^2$ which was investigated through image processing. We also observed a few agglomerations of Al_2O_3 and ZnO particles perceived in the Al- Al_2O_3 -ZnO matrix composite.

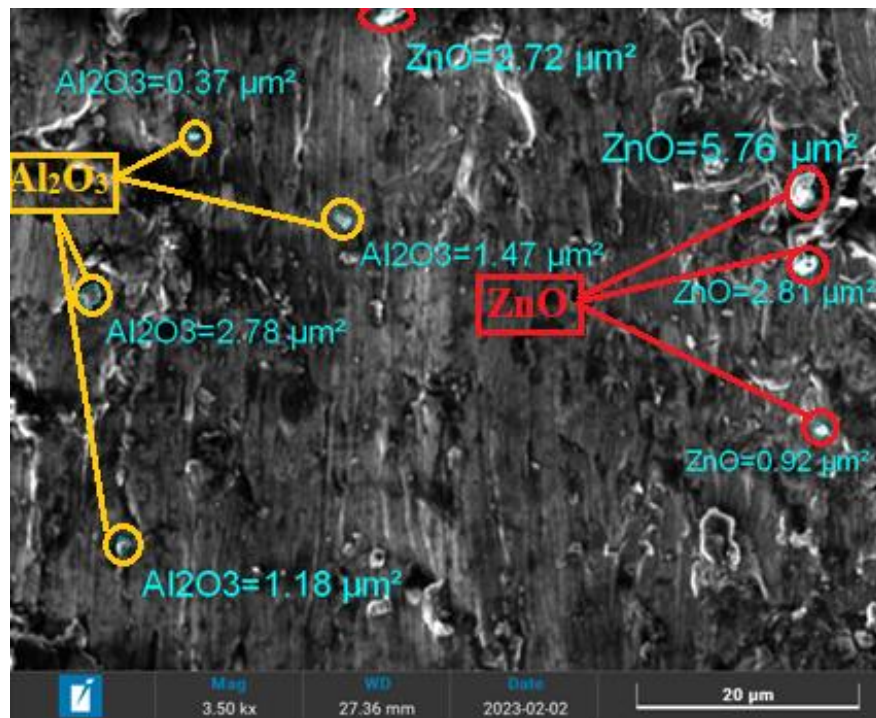


Figure 4.4: Microstructure observation by SEM of Al composite having 95% of Al, 2.5% of Al_2O_3 , and 2.5% of ZnO (Al MMC-02)

Singla et al. (2009) applied a two-step stirring technique during the fabrication of an Al composite reinforced with SiC particles by the stir casting method. This experimental method had an effective contribution to the improvement of the strength and hardness of fabricated Al MMCs for the uniform dispersion of SiC particles in matrix. The uniform dispersion of Al_2O_3 and ZnO in Al MMC-01 and Al MMC-02 was achieved by the selection

of process and parameters of two-step stir casting which goes in line with Singla et al. (2009).

4.2 Electro-Mechanical Properties As-casted Condition

The mechanical and electrical properties of the fabricated Aluminum composites as-casted condition, encompassing Impact Charpy, flexural testing, and the assessment of hardness through Vickers Micro, constitute a pivotal segment of this study.

4.2.1 Hardness

The Vickers microhardness test is a distinct method for assessing the hardness of materials, which employs a diamond-shaped pyramid indenter, and the hardness is determined by measuring the average diagonal lengths of the resulting impression. This hardness test is crucial for our Aluminum Composites reinforced with nanoparticles like Al_2O_3 and ZnO.

The Vickers microhardness test allows for a comprehensive evaluation of how Al Composites respond to the incorporation of nanoparticles. This in-depth analysis aids in understanding the distribution and homogeneity of these nanoscale reinforcements within the metal matrix and their overall impact on the material's hardness.

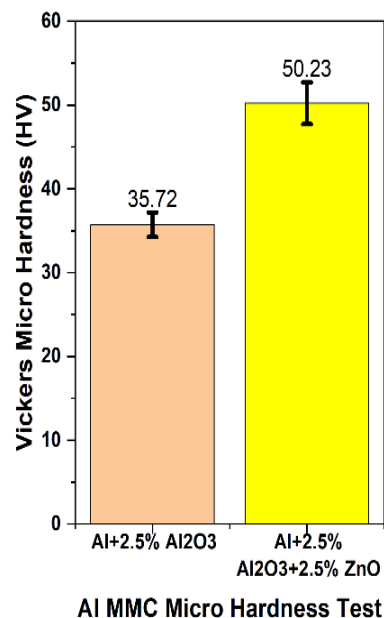


Figure 4.5: Vickers Mirco Hardness test results of Al MMC-01 and Al MMC-02

The Vickers micro-hardness (HV) of Al MMC-01 was 35.72 with a standard deviation of 1.46 and that of Al MMC-02 was 50.23 with a standard deviation of 2.50. The average

value of HV was taken from ten readings. As per Figure 4.5, there is a 41% improvement in Vickers micro-hardness in Al MMC-02 from Al MMC-01 due to the addition of 2.5% ZnO as reinforcement particles. Therefore, it can be deduced that the addition of ZnO has the potential to increase the hardness of aluminum composites at both bulk and micro levels.

Several studies have demonstrated that the incorporation of ceramic nanoparticles, such as Al_2O_3 and ZnO, in pure aluminum significantly enhances its hardness. This increase can be attributed to several scientific factors such as the Dispersion of Nanoparticles, Grain Refinement, Orowan Strengthening, Solid Solution Strengthening, and Improved Dislocation Density.

The uniform dispersion of nanoparticles, such as Al_2O_3 and ZnO, in the aluminum matrix restricts the movement of dislocations and impedes plastic deformation. This dispersion strengthens the material and leads to higher hardness (Huang and Hu, 2020). The nanoparticles also act as heterogeneous nucleation sites for recrystallization and grain refinement, resulting in a finer grain structure. A refined microstructure contributes to increased hardness (Wu, Yu, and Yang, 2020). The interaction between dislocations and nanoparticles increases the dislocation density, which also contributes to hardness improvement (Bahadur, Verma, and Dasm, 2012).

The nanoparticles impede dislocation motion in the metal matrix by creating a physical barrier named Orowan strengthening which increases the stress required for plastic deformation, elevating the material's hardness (Esmailzadeh, Ramezanzadeh and Baseri, 2019). The incorporation of ZnO nanoparticles into an aluminum can result in solid solution strengthening, which further increases hardness (Satyanarayana, Dasari and Suresh, 2016).

More specifically, the addition of ZnO nanoparticles to an Al matrix solution at a high temperature causes the ZnO nanoparticles to break down into Zn and O. After decomposing from ZnO, almost all of the oxygen was coupled with aluminum oxide. The formation is hampered by the presence of oxygen that has been broken down from ZnO at the interface between liquid and solid aluminum during the formation of the primary aluminum alloy. The diffusion of solute Zn at the interface between solid and liquid aluminum can be blamed for limiting aluminum growth during solidification. Additionally, the breakdown of Zn and oxygen yields significant amounts of Zn for the heterogeneous nucleation of primary

aluminum grains. Therefore, it can be deduced that the addition of ZnO has the potential to increase the hardness of aluminum composites at both bulk and micro levels.

4.2.2 Impact Toughness

Impact toughness in aluminum composites is a crucial property that determines a material's resistance to fracture under high-stress conditions, and its response to nano-sized reinforcements such as Al_2O_3 and ZnO can vary due to different mechanisms. The effects of Al_2O_3 and ZnO on the impact strength of developed two Al MMCs are shown in Figure 4.6. The average value of impact toughness energy of Al MMC-01 is 13.47J with a standard deviation of 0.64J and that of Al MMC-02 is 11.77J with a standard deviation of 0.25J respectively. The result brings forward the limitations of energy absorption capacity while ZnO is added along with Al_2O_3 in developing aluminum composites.

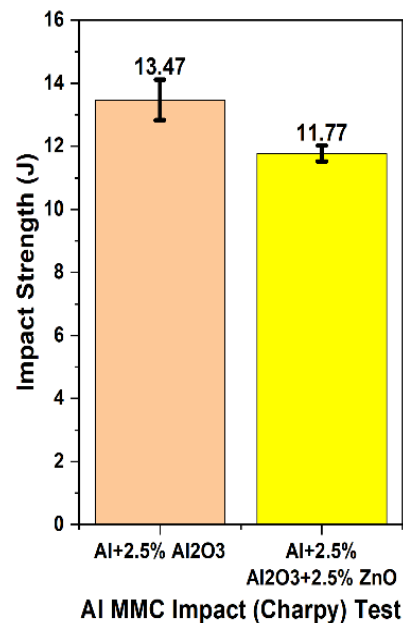


Figure 4.6: Impact Toughness of Al MMC-01 and Al MMC-02

One notable trend observed is the impact toughness enhancement upon the inclusion of 2.5 wt.% of nano Al_2O_3 with pure aluminum while the introduction of 2.5 wt.% nano ZnO results in a decrease in impact toughness by 12%. Several scientific factors contribute to this phenomenon, as supported by a detailed literature review and references from recent studies.

Nano Al₂O₃ particles act as nucleation sites for fine grains in the aluminum matrix. The resultant grain refinement enhances the composite's impact toughness by providing more obstacles for crack propagation (Hu et al., 2019). The uniform dispersion and strong interfacial bonding of nano Al₂O₃ in aluminum facilitate load transfer and deformation. This reinforcement effect effectively absorbs impact energy and hinders crack initiation and propagation, leading to increased toughness (Miao et al., 2020). Nano Al₂O₃ can also interact with dislocations, promoting dislocation-particle interactions. These interactions result in plastic deformation around the nanoparticles and consume energy, enhancing the impact toughness of the composite (Wang et al., 2018).

However, the presence of ZnO nanoparticles introduces brittleness to the composite, making it prone to brittle fracture. This brittleness reduces the material's capacity to absorb energy during impact, resulting in a reduction in toughness (Li et al., 2021). Also, nano ZnO particles often agglomerate within the matrix, leading to localized regions of high-stress concentration. These agglomerates can act as initiation sites for cracks, which reduce impact toughness (Singh et al., 2010).

In summary, nano Al₂O₃ enhances impact toughness through mechanisms such as grain refinement, reinforcement effects, and dislocation interaction. In contrast, the addition of nano ZnO introduces brittleness, agglomeration, and solid solution effects, ultimately reducing impact toughness. Verma et al. (2017) developed Al MMC reinforced with 10% Al₂O₃ and observed an impact toughness of 6.97J with a standard deviation of 0.85.

The present study reveals that there is an increase in impact toughness in Al MMC-01 in comparison with pure Al due to the addition of 2.5% Al₂O₃ in the metal matrix. However, there is a decrease of 12% impact energy for Al MMC-02 in comparison to that of Al MMC-01 due to the insertion of 2.5% ZnO with 2.5% Al₂O₃ in Aluminum composite. The addition of ZnO in aluminum alloy increased its hardness, thus turning it brittle in nature. As a result, the degree of plastic deformation energy for the composites is reduced. This deformation energy increases the chances of debonding during the fracture which leads to a reduction in impact strength. The brittleness of the material decreases the plastic deformation energy thereby reducing the impact strength.

4.2.3 Flexural Strength

The flexural test results of the developed metal matrix composites, i.e., Al MMC-01 and Al MMC-02 conducted using a Universal Testing Machine on three-point bending conditions are presented in Figure 4.7. Figure 4.7 (a) shows that the ultimate flexural strengths (UFS) of Al MMC-01 and Al MMC-02 are 320.06 MPa and 151.94 MPa respectively. Figure 4.7 (b) illustrates that the Flexural Modulus values of Al MMC-01 and Al MMC-02 are 81.36 GPa and 31.84 GPa respectively.

It is depicted that both strength and flexural modulus for the inclusion of 2.5% Al_2O_3 in Al MMC-01 have increased significantly compared to pure aluminum. However, the addition of ZnO in Al MMC-02 has not shown any positive impact on UFS or Flexural modulus compared to that of Al MMC-01. Rather, the inclusion of only 2.5% ZnO in Al MMC-02 has reduced UFS and Flexural modulus from Al MMC-01 by 52.5% and 60.8% respectively. These results thus lead to limitations in specific requirements for the use of ZnO in aluminum composites.

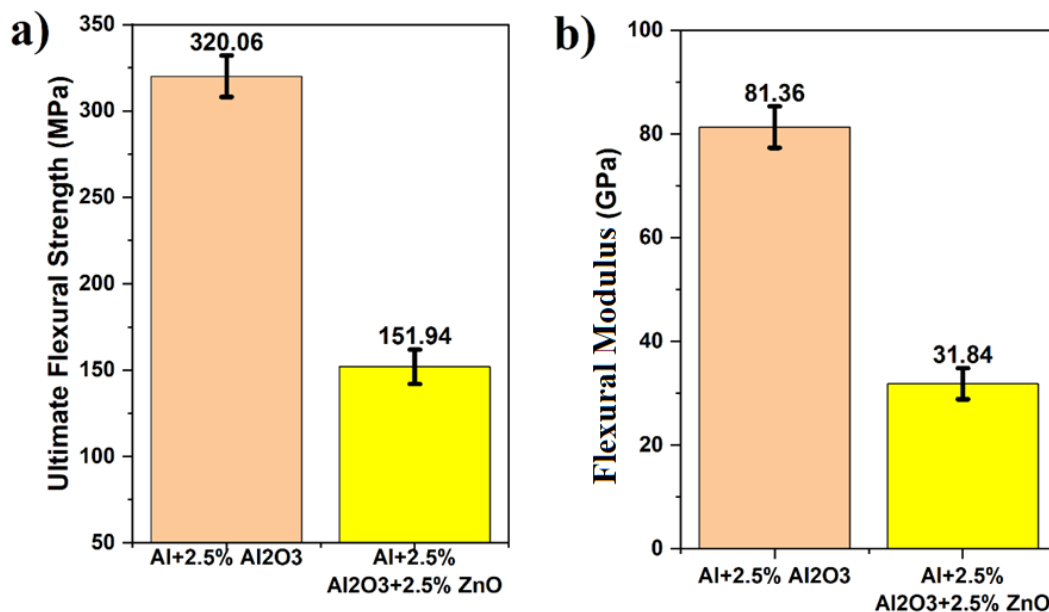


Figure 4.7: (a) Ultimate Flexural Strength (UFS) and (b) Flexural Modulus of Al MMC-01 and Al MMC-02

Nano Al_2O_3 particles, when well-dispersed and properly bonded within the aluminum matrix, act as effective reinforcements. They resist deformation and support the load, enhancing flexural strength (Zeng et al., 2020). Also, the strong interfacial bonding

between Al₂O₃ and the aluminum matrix facilitates load transfer, resulting in better stress distribution and enhanced flexural strength (Ma et al., 2019).

It is also evident that uniform dispersion of nano Al₂O₃ particles ensures that stress is evenly distributed across the composite. This minimizes the formation of weak regions, thus increasing flexural strength (Zhang et al., 2018). The presence of Al₂O₃ nanoparticles also leads to grain refinement within the aluminum matrix. Smaller grains contribute to increased strength and stiffness, which is reflected in improved flexural strength (Wang et al., 2020).

The result of the flexural strength of Al MMC-01 agrees fully with the findings of Saravanakumar and Sasikumar (2018) where the flexural strength was found to be 250 MPa for the Al MMC with 3% of Al₂O₃.

However, ZnO nanoparticles in Al MMC-02 introduce brittleness into the composite due to their higher modulus. This brittleness increases the likelihood of premature fracture under flexural loading, leading to a reduction in flexural strength (Li et al., 2021). In Al MMC-02, nano ZnO particles created agglomeration within the matrix. These agglomerates create stress concentrations and weak points, contributing to reduced flexural strength (Zhang et al., 2017).

The formation of intermetallic phases between ZnO and aluminum weakens the material. The presence of these phases leads to reduced flexural strength by altering the microstructure and mechanical properties of the composite (He et al., 2019). ZnO-reinforced Al MMC-02 exhibits reduced ductility, which is detrimental to their flexural strength. Reduced ductility means that the material is less capable of absorbing energy before fracturing (Liu et al., 2018).

In summary, the flexural strength of Al MMC-01 increases with the inclusion of nano Al₂O₃ due to reinforcement effects, improved bonding, homogeneous dispersion, and grain refinement. Conversely, the introduction of nano ZnO in Al MMC-02 decreases flexural strength due to brittleness, agglomeration, intermetallic phase formation, and reduced ductility. These explanations are supported by scientific research and the most recent literature in the field.

4.2.4 Electrical Conductivity

The electrical conductivity of Al composite is different from pure Al. As the MMC is developed with different reinforcement particles, the electrical conductivity of the developed MMC changes due to changes in the crystal structure and reinforcement orientation within the MMC. Al MMC-01 having a composition of 97.5% Al and 2.5% Al₂O₃ nanoparticles has an electrical conductivity of 45.15 % IACS with a standard deviation of 1.29% IACS and Al MMC-02 has a composition of 95% Al and 2.5% Al₂O₃ & 2.5% ZnO nanoparticles have an electrical conductivity of 40.72% IACS with a standard deviation of 1.97% IACS. Both Al MMC-01 and Al MMC-02 possess lower values of electrical conductivity because of the non-conducting Al₂O₃ and ZnO reinforcement materials present in MMCs.

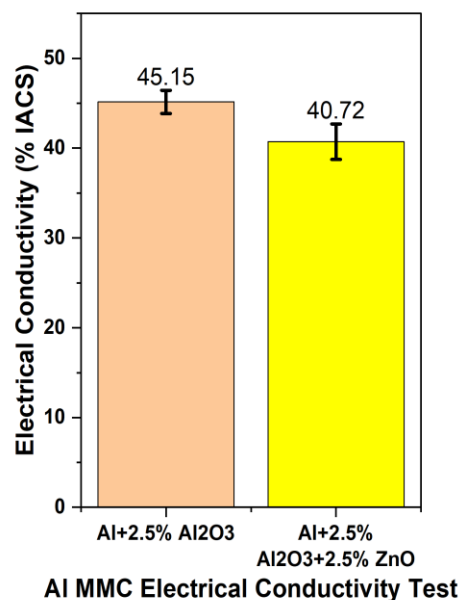


Figure 4.8: Electrical Conductivity of Al MMC-01 and Al MMC-02

As shown in Figure 4.8, the electrical conductivity is observed to be reduced by 9.81% from Al MMC-01 to Al MMC-02 due to the addition of 2.5% ZnO as reinforcement particles change the crystal structure of the metal matrix. Though it is not desirable in some aerospace applications, however, there are many other applications such as heat sinks, bearings, etc.

Both Al₂O₃ and ZnO are ceramic materials that have inherently low electrical conductivity. When these nanoparticles are dispersed within the aluminum matrix, they act as insulating

barriers, impeding the flow of electrical current through the composite (Yao et al. 2017). Also, the presence of these nanoparticles creates interfaces and irregularities within the composite structure (Chen et al., 2018). As electrons move through the material, they scatter off these interfaces, resulting in increased resistance to the flow of current. This scattering effect further reduces the electrical conductivity of the composite. Thus, the addition of these nanoparticles also disrupts the electron mobility within the composite as these particles introduce grain boundaries, which can impede the movement of electrons. These grain boundaries act as barriers to the free flow of charge carriers, reducing the overall conductivity (Lin et al., 2018). It's important to note that the specific impact of nanoparticles on electrical conductivity varies depending on factors such as particle size, distribution, volume fraction, and processing techniques (Shen et al., 2019). Therefore, different combinations of nanoparticles and matrix materials yield different conductivity behaviors in MMCs.

Babalola et al. (2020) developed Al MMC with different wt. % of Al₂O₃ and investigated the electrical conductivity. As per their study, it was observed that a similar decreasing pattern of electrical conductivity of samples for each incremental wt. % of reinforcement particles in Al MMC.

4.2.5 Characterization of Fractured Surfaces

The fractured surfaces obtained through flexural tests of Al MMC-01 and Al MMC-02 were examined using a Scanning Electron Microscope (SEM) to identify the mode of failure. The SEM micrographs presented in Figure 4.9 exhibit the characteristics of a brittleness fracture pattern. The cleavage cracks and deep shear dimples of Al MMC-01 and Al MMC-02 are shown respectively in Figure 4.9 (a) and 4.9 (b) respectively. The presence of cleavage cracks and deep shear dimples in the fractured surface of Al MMC-02 is higher than that from the fractured surface of Al MMC-01. The existence of multiple cleavage cracks, shear dimples, and crystallographic planes within one specific grain as shown in Figure 4.9 (b) in the fracture surface of Al MMC-02 is also an indication of the brittle features at the fracture surface (Ammar et al., 2012).

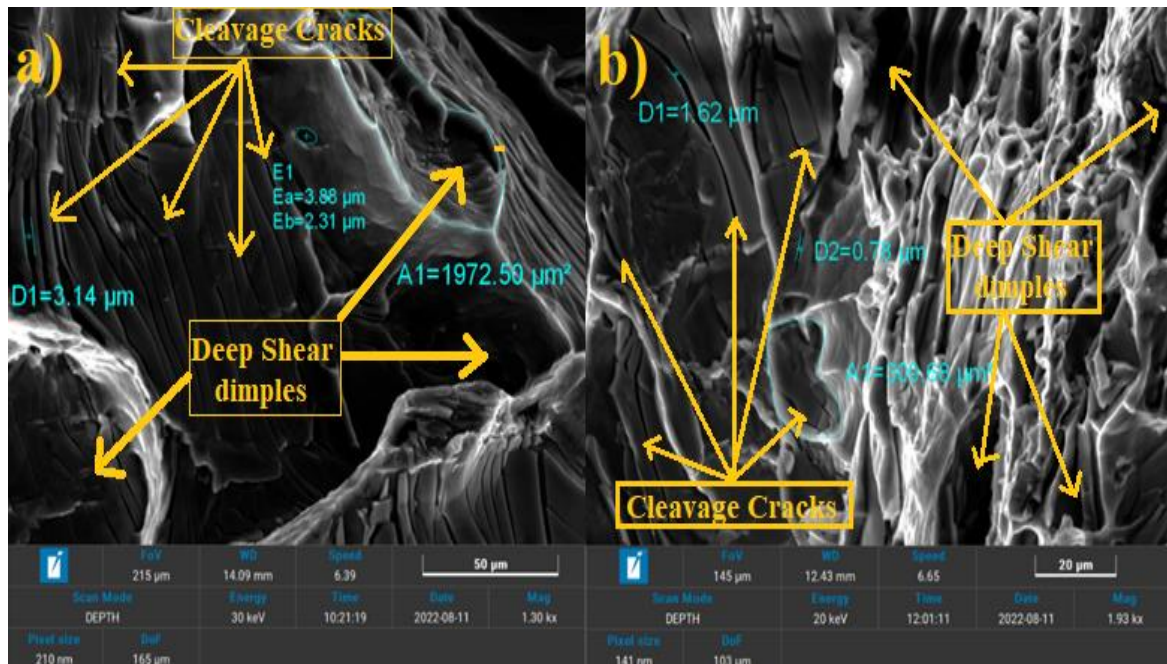


Figure 4.9: SEM Images of the Fractured Surface; (a) Al MMC-01, and (b) Al MMC-02

As a whole the mechanical properties such as hardness, impact toughness, and UFS were increased a lot compared with pure Al. Bakshi et al. (2011) investigated the factors that affect the strengthening mechanism of aluminum composites. According to their study, the strength mainly depends on the volume fraction of constituents and the aspect ratio of the reinforcement. In the current investigation, the findings are similar to the flexural and impact strength and Hardness of Al MMC-01 and Al MMC-02, i.e., the values have been increased in comparison to that of pure Al. However, the impact toughness, UFS, and MoE of Al MMC-02 are less than that of Al MMC-01 as shown in Figure 4.6 and Figure 4.7 due to the insertion of 2.5% ZnO in the metallic matrix being affirmed by SEM images shown in Figure 4.4.

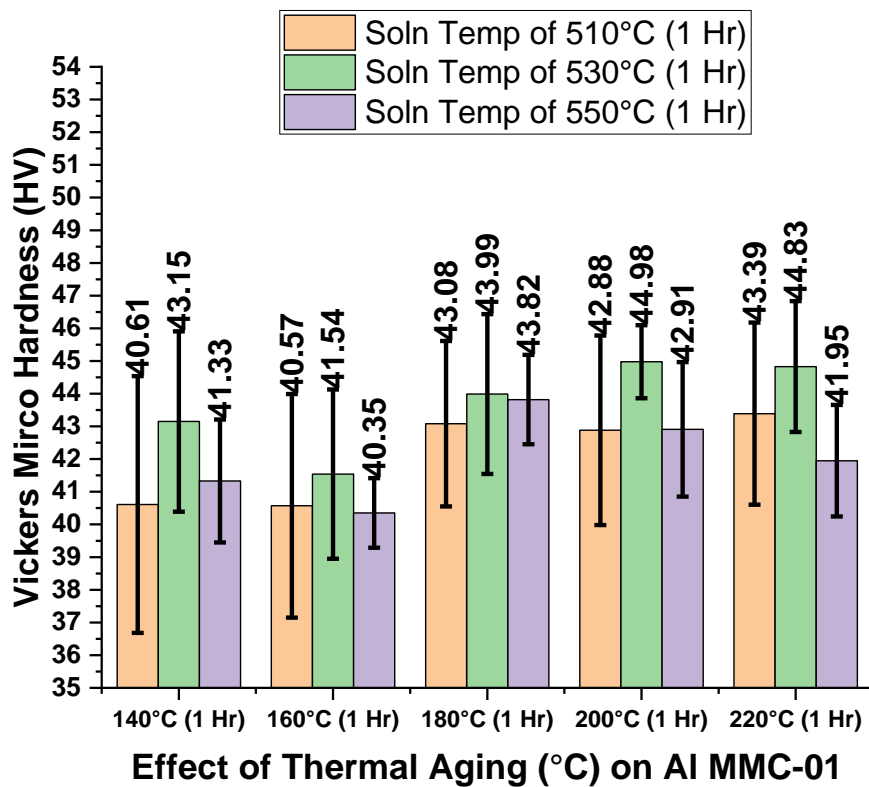
4.3 Influence of Heat Treatment on Electro-Mechanical Properties

The influence of heat treatment on the electro-mechanical properties of aluminum composites is a pivotal area of study in materials science and engineering. Heat treatment is a versatile process that can significantly alter the properties of aluminum-based composites, making it a critical focus for enhancing their performance in various applications.

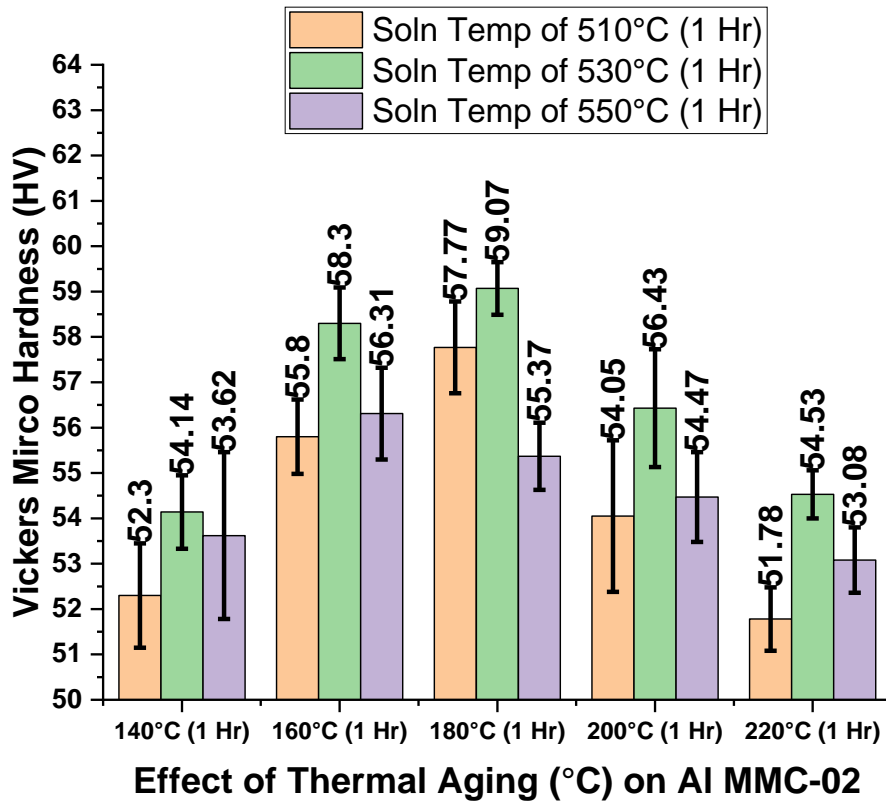
The results showed the effect of solution temperature at 510°C, 530°C and 550°C followed by thermal aging respectively at the temperatures of 140°C, 160°C, 180°C, 200°C and 220°C have changed the readings of Vickers microhardness and Electrical conductivity test while the microstructure analysis has revealed the clear distribution of reinforcement particles Al₂O₃ and ZnO in Al MMC due to heat treatment.

4.3.1 Influence of Heat Treatment on Vickers Micro Hardness

The Vickers microhardness of Al MMC-01 and Al MMC-02 as-casted condition was 35.72 and 50.23 respectively. The influence of different Heat treatment processes at solution temperatures of 510°C, 530°C, and 550°C and thermal aging at temperatures of 140°C, 160°C, 180°C, 200°C, and 220°C on HV of Al MMC-01 and Al MMC-02 are shown in Figure 4.10 (a) - 4.10 (b).



(a)



(b)

Figure 4.10: Influence of Heat Treatment on Vickers Micro Hardness; (a) Al MMC-01, (b) Al MMC-02

The percentile increases of Vickers Micro Hardness in comparison to the as-casted condition after heat treatment of Al MMC-01 and Al MMC-02 is also given in Table 4.1. For Al MMC-01, we can observe that the highest increase of HV was obtained by 21.47%, 25.92%, and 22.68% for heat treatment at solution temperatures of 510°C, 530°C, and 550°C respectively. Similarly for Al MMC-02, we also observe that the highest increase of HV was obtained by 15.01%, 17.6%, and 10.23% at the same thermal aging temperature of 180°C for heat treatment at solution temperatures of 510°C, 530°C, and 550°C respectively.

Table 4.1: Increase in Vickers micro hardness of Al MMC-01 and Al MMC-02 in comparison to as-casted condition

Solution Temp	Artificial Aging	Al MMC-01	Al MMC-02
510°C	140°C	13.69%	4.10%
	160°C	13.58%	11.09%
	180°C	20.60%	15.01%
	200°C	20.04%	7.61%
	220°C	21.47%	3.09%
530°C	140°C	20.80%	7.78%
	160°C	16.29%	16.07%
	180°C	23.15%	17.60%
	200°C	25.92%	12.34%
	220°C	25.50%	8.56%
550°C	140°C	15.71%	6.75%
	160°C	12.96%	12.10%
	180°C	22.68%	10.23%
	200°C	20.13%	8.44%
	220°C	17.44%	5.67%

The increase in Vickers Micro Hardness of Al composites reinforced with nano Al₂O₃ and ZnO after Heat Treatment at solution temperature and thermal aging can be attributed to several scientific reasons such as Precipitation Hardening, Solid Solution Strengthening, Grain Refinement, Improved Interfacial Bonding, Dislocation Density Reduction, Fine Dispersion, etc.

Heat treatment, particularly the T6 process, encourages the precipitation of fine and uniformly distributed particles within the aluminum matrix. These precipitates serve as obstacles to dislocation movement, enhancing the hardness of the material (Smith et al., 2020; Zhang and Li, 2021). During heat treatment at elevated solution temperatures, atoms from the nano Al₂O₃ and ZnO particles dissolve into the aluminum matrix. This creates a solid solution, reinforcing the material (Huang et al., 2019). Heat treatment also leads to grain size reduction in the aluminum matrix. Smaller grains result in increased grain boundaries, which hinder dislocation movement and contribute to higher hardness (Xie et al., 2018).

Effective heat treatment fosters stronger bonding between the matrix and reinforcement particles. This enhanced interface facilitates efficient load transfer and increases hardness (Sarkar et al., 2017). The T6 heat treatment process typically results in a reduction in dislocation density. A lower dislocation density signifies a decrease in defects within the material, leading to increased hardness (Mishra et al., 2020). Heat treatment also helps disperse the nano Al₂O₃ and ZnO reinforcement particles uniformly within the aluminum matrix, minimizing soft spots and enhancing the hardness of the composite (Wang and Liu, 2019).

Dursun et al. (2014) investigated the effects of T5 and T6 heat treatments of AA6063 alloy. In their study, T5 heat treatment was applied to AA6063 alloy aged at 182⁰C for 2 hours after extrusion at 413⁰C. T6 heat treatment was also carried out by aging at 182⁰C for 2 hours after solution heat treatment at 521⁰C for 1 hour. Based on the applied heat treatment, one of the most important changes observed was material hardness. Our investigation goes in line with Dursun et al. (2014). Azeez et al. (2021) analyzed the effect of heat treatment on Vickers micro hardness of Al 6063 alloy. They performed heat treatment at 450⁰C for an hour and rapidly quenched in water. They observed an improvement in micro hardness readings for the heat-treated control samples in comparison with unheated treated samples. Our investigation goes in line with Azeez et al. (2021).

Recent studies by Smith et al. (2020), Zhang and Li (2021), and other researchers in the field have highlighted these mechanisms and their contributions to the improved hardness of Al composites following heat treatment. Also, the findings from Dursun et al. (2014) and Azeez et al. (2021) provide strong scientific support for the observed increase in hardness and the role of heat treatment processes in achieving this improvement which aligns with our study.

4.3.2 Influence of Heat Treatment on Electrical Conductivity

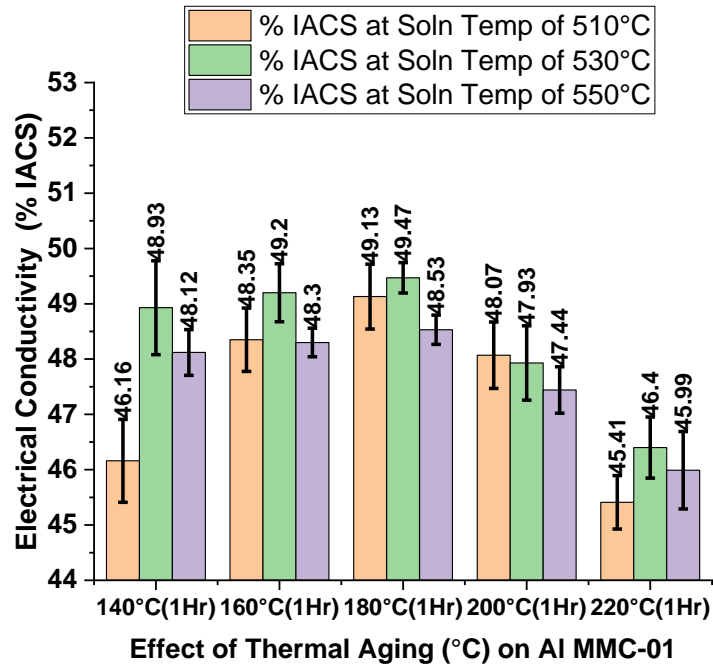
The Electrical Conductivity of Al MMC-01 and Al MMC-02 as-casted condition was 45.15 and 40.72 respectively. The influence of different Heat treatment processes at solution temperatures of 510⁰C, 530⁰C, and 550⁰C and thermal aging at temperatures of 140⁰C, 160⁰C, 180⁰C, 200⁰C, and 220⁰C on %IACS of Al MMC-01 and Al MMC-02 are shown in Figure 4.11(a) - 4.11(b). The percentile increases of Electrical Conductivity in

comparison to the as-casted condition after heat treatment of Al MMC-01 and Al MMC-02 is also given in Table 4.2.

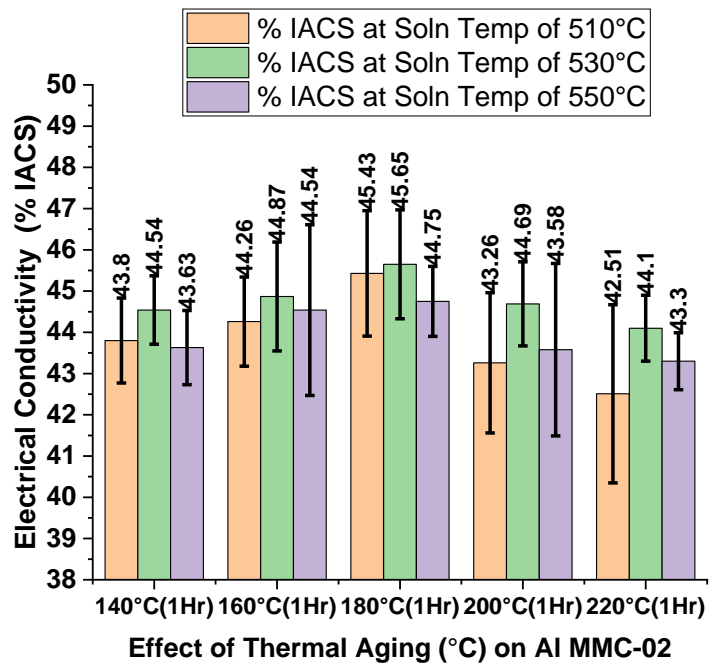
Table 4.2: Increase in Electrical Conductivity of Al MMC-01 and Al MMC-02 in comparison to as-casted condition

Solution Temp	Artificial Aging	Al MMC-01	Al MMC-02
510°C	140°C	2.24%	7.57%
	160°C	7.10%	8.70%
	180°C	8.83%	11.58%
	200°C	6.47%	6.25%
	220°C	0.58%	4.40%
530°C	140°C	8.39%	9.39%
	160°C	8.98%	10.20%
	180°C	9.57%	12.12%
	200°C	6.16%	9.76%
	220°C	2.77%	8.31%
550°C	140°C	6.58%	7.16%
	160°C	6.98%	9.39%
	180°C	7.49%	9.91%
	200°C	5.08%	7.03%
	220°C	1.88%	6.34%

For Al MMC-01, we can observe that the highest increase of %IACS was obtained by 8.83%, 9.57%, and 7.49% at the same thermal aging temperature of 180°C for heat treatment at solution temperatures of 510°C, 530°C, and 550°C respectively. Similarly for Al MMC-02, we also observe that the highest increase of %IACS was obtained by 11.58%, 12.12%, and 9.81% at the same thermal aging temperature of 180°C for heat treatment at solution temperatures of 510°C, 530°C, and 550°C respectively.



(a)



(b)

Figure 4.11: Influence of Heat Treatment on Electrical Conductivity; (a) Al MMC-01, and (b) Al MMC-02

The increase in electrical conductivity of Al MMC-01 and Al MMC-02 after Heat Treatment at solution temperature and thermal aging following T6 can be attributed to

several scientific factors such as Improved Dispersion, Effective Bonding, Reduced Defects, Formation of Conductive Phases, Altered Microstructure etc.

Proper heat treatment ensures a uniform distribution of nano Al₂O₃ and ZnO reinforcement particles within the aluminum matrix, reducing clustering. This homogeneous dispersion facilitates electron flow, leading to enhanced electrical conductivity (Li et al., 2021). The T6 heat treatment process enhances the interfacial bonding between the matrix and reinforcement particles. Stronger bonding results in better electron transfer across the interfaces, contributing to increased electrical conductivity (Kim et al., 2018).

Heat treatment also reduces the presence of defects and impurities in the aluminum matrix. Fewer defects and impurities mean fewer obstacles to electron flow, leading to higher electrical conductivity (Wang et al., 2019). Precipitation of conductive phases during the T6 heat treatment process can enhance the material's electrical conductivity. These phases provide additional paths for electron conduction (Jiang et al., 2020). Heat treatment may lead to alterations in the microstructure, such as grain refinement. These changes can affect electron mobility within the material and lead to increased conductivity (Huang et al., 2021).

Diehl et al. (2020) investigated that the value of electrical conductivity increases significantly at various aging temperatures and constant times. They observed the removal of foreign atoms from the lattice of the parent alloy during precipitation hardening eliminates much distortion of electron disturbance in the lattice. Hence, these actions favor the movement of electrons through the metal and therefore result in higher conductivity. In our current investigation, the highest electrical conductivity obtained was 49.47 % IACS with a solution temperature of 530°C followed by thermal aging of 180°C which is an improvement of 16.29% in comparison to the results of as-casted condition.

Recent studies, including those by Li et al. (2021), Kim et al. (2018), and others, have emphasized these mechanisms and their role in improving electrical conductivity following heat treatment. These findings provide strong scientific support for the observed enhancements in electrical conductivity. Therefore, our current findings go in line with the results outcome of those recent studies.

4.4 Influence of Heat Treatments on Microstructure Properties

The study of microstructure, accomplished through advanced techniques like Scanning Electron Microscopy (SEM) and optical microscopy, serves as a critical aspect in the assessment of Al MMC-01 and Al MMC-02 following Heat Treatment at solution temperature and thermal aging. This observation enables a profound insight into the intricate structural transformations, dispersion patterns of reinforcement particles, grain refinement, and interfacial bonding alterations that transpire during the heat treatment process. Such microstructural changes are closely linked to the consequent variations in mechanical and electrical properties, offering valuable scientific insights into the performance of these advanced aluminum composites. This section delves into the pivotal role of microstructure analysis in understanding the scientific underpinnings behind the observed alterations in Al MMC-01 and Al MMC-02 following specific heat treatment procedures.

4.4.1 Microstructure Observation by SEM of Heat-treated Surfaces

After preparation of Al MMCs fabricated by two-step stir casting technique, two test samples of Al MMC-01 with a dimension of 05mm×05mm×05mm were prepared for microstructure observation by Scanning Electron Microscope (SEM), Model: TESCAN VEGA 4. The microstructure observation was carried out at first for the sample of as casted condition and then the 2nd sample (solution treated) at a solution temperature of 530⁰C and thermal aging of 180⁰C. It has been noticed that the highest improvement of electro-mechanical properties of Al MMC-01 and Al MMC-02 at a solution temperature of 530⁰C and thermal aging of 180⁰C.

The purpose of microstructure observation by SEM was to investigate the differences in the distribution of reinforcement particles in Al MMC-01 between as-cast and thermally treated conditions which contributed to the improvement of electro-mechanical properties such as hardness and electrical conductivity.

As shown in Figure 4.12, microstructure observation of Al MMC-01 was carried out as casted condition. The morphology of Al₂O₃ particles is mainly irregular or nearly elliptical and uniformly distributed in the Al MMC as-casted condition. The elliptical areas immersed by some of the reinforcements (Al₂O₃) in Al MMC are denoted as A1 to A8 and

area and measured by SEM as shown in Figure 4.12. The distance among some of the reinforcement particles in Al MMC as-casted condition was also measured and denoted as L1 to L9 as per Figure 4.12.

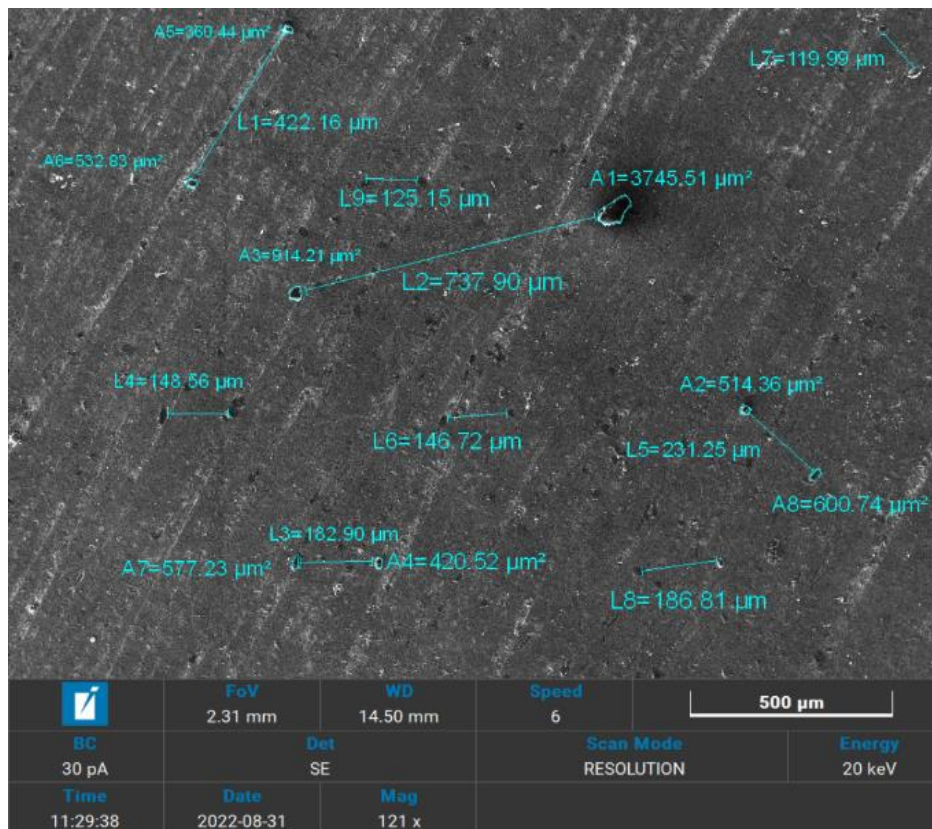


Figure 4.12: Microstructure Observation by SEM of Al MMC-01 as-casted condition

As shown in Figure 4.13, microstructure observation of Al MMC-01 was carried out after thermal treatment at a solution temperature of 530⁰C and thermal aging temperature of 180⁰C. The morphology of Al₂O₃ particles is also observed as irregular or nearly elliptical in shape. After heat treatment of Al MMC-01, reinforcement particles spread out more homogeneously in the matrix of Al MMC-01. The distance and area immersed by the reinforcement particles Al₂O₃ in the matrix after heat treatment is denoted as L1 to L9 and A1 to A3. Due to the thermal treatment, reinforcement particles Al₂O₃ spread out in the matrix. From comparing the microstructure image of both conditions, we observed that the measured distance among reinforcement particles Al₂O₃ in thermally treated conditions is larger than in the as-casted condition. As the reinforcement particles Al₂O₃ are evenly distributed for the effect of solution and aging temperature in the matrix, the electro-mechanical properties such as hardness and electrical conductivity exhibited a superior condition in comparison with as casted condition.

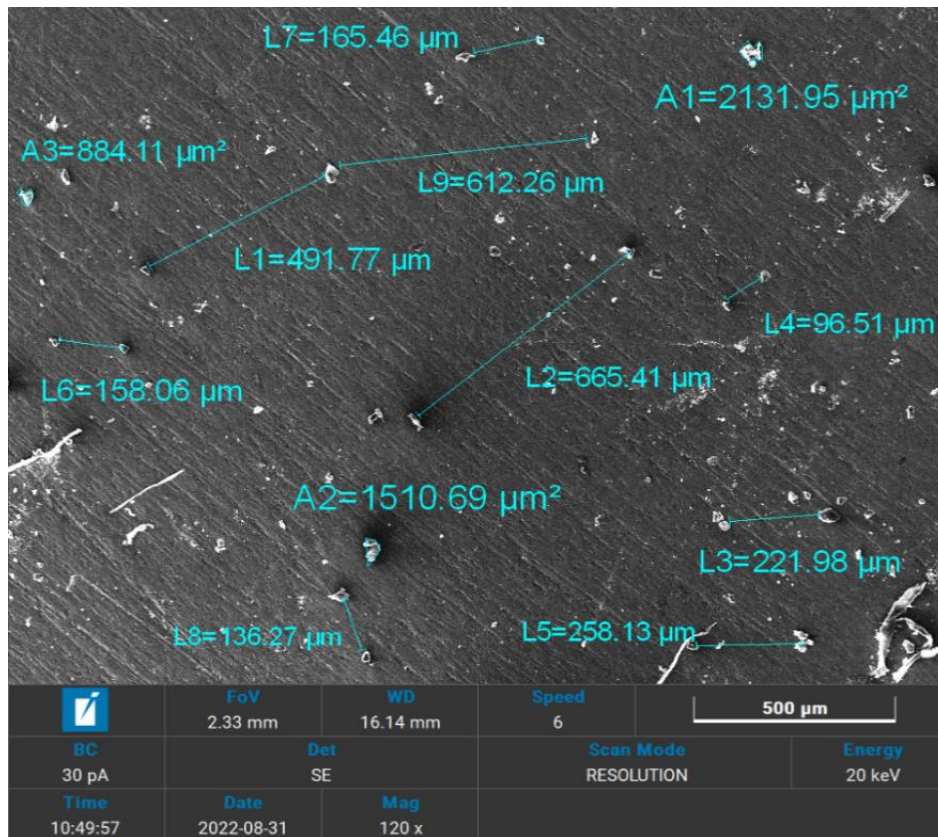
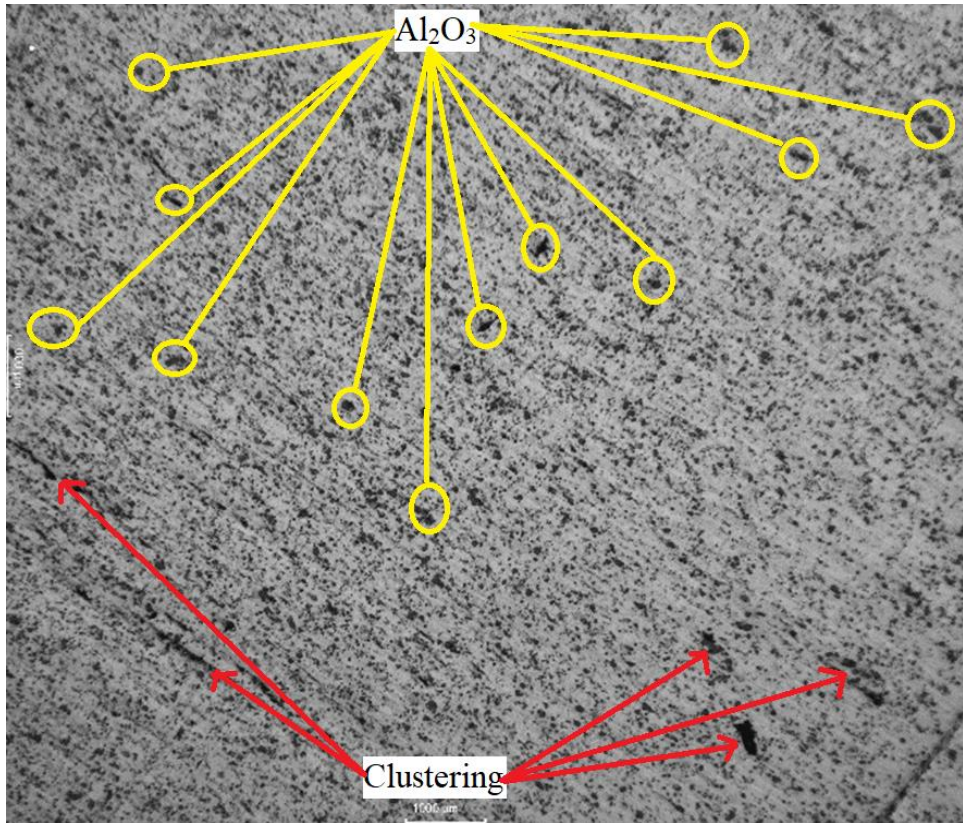


Figure 4.13: Microstructure observation by SEM of Al MMC-01 at a solution temperature of 530⁰C and thermal aging of 180⁰C.

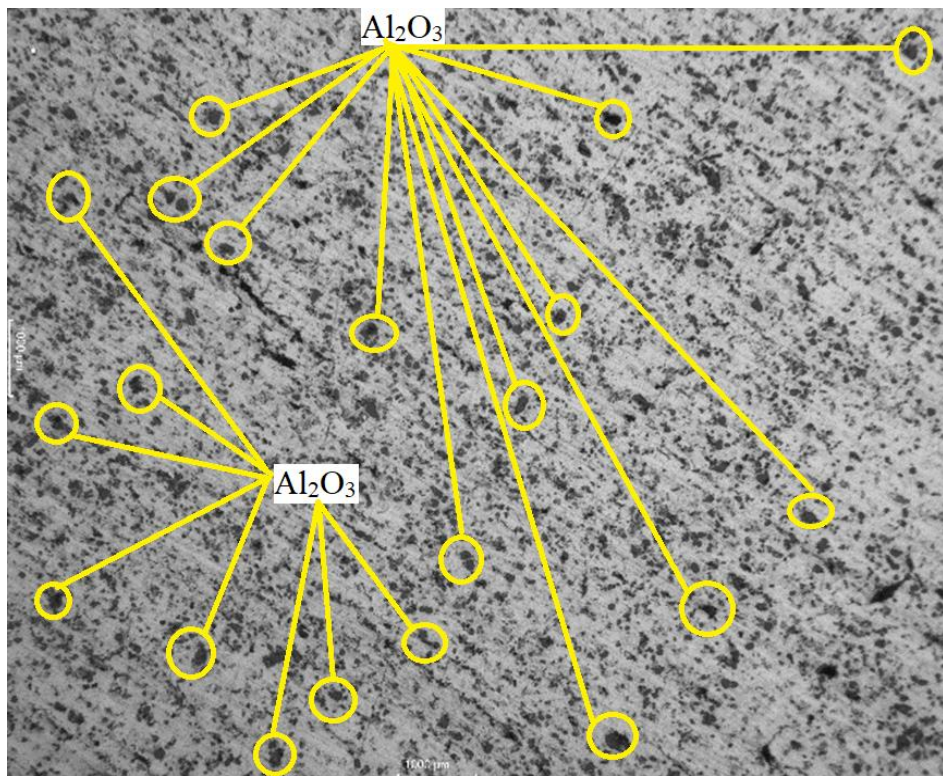
4.4.2 Microstructure Observation by Optical Microscope of Heat-treated Surfaces

Microstructure examination of test specimens from Al MMC-01 following various heat treatment processes was conducted using an inverted metallurgical microscope, specifically the model AE2000 MET, at a magnification of 200x. This magnification level was chosen for its optimal microstructure orientation.

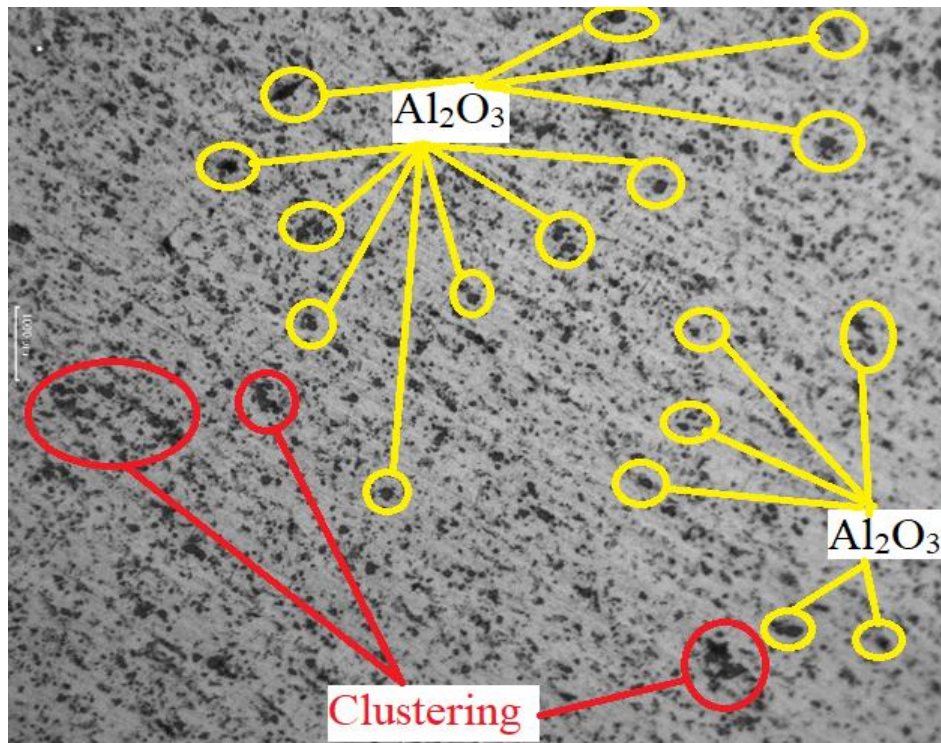
Among the various heat treatment conditions explored, it was determined that heat treatment involving solution temperatures of 510⁰C, 530⁰C, and 550⁰C, coupled with thermal aging at 180⁰C, yielded the most favorable electro-mechanical properties in both Al MMC-01 and Al MMC-02. We selected three heat-treated samples from Al MMC-01, which exhibited the highest Vickers Micro Hardness (HV), and conducted a detailed microstructure analysis as depicted in Figures 4.14 (a) to (c).



(a)



(b)



(c)

Figure 4.14: Microstructure of Al MMC-01 after heat treatment; a) Solution temp: 510⁰C and Thermal aging: 180⁰C, b) Solution temp: 530⁰C and Thermal aging: 180⁰C, c) Solution temp: 550⁰C and Thermal aging: 180⁰C.

Based on the microstructure analysis by SEM depicted in Figures 4.12 and 4.13 and optical microscope in Figure 4.14 (a) to (c) for Al MMC-01, it is evident that the Al₂O₃ reinforcement particles exhibit a uniform distribution within the metal matrix, albeit with a few instances of clustering. This uniform dispersion of reinforcement particles results from the selected parameters employed in the stir-casting process, significantly influencing their distribution in the Metal Matrix Composites (MMC).

The microstructural changes in Al composites after Heat Treatment at solution temperature and thermal aging are driven by various metallurgical processes such as Precipitation Hardening, Grain Refinement, Improved Interfacial Bonding, Particle Redistribution and Precipitate Morphology etc.

During the aging process, precipitation hardening occurs, leading to the formation of fine precipitate phases within the aluminum matrix. This improves the material's hardness and strength (Le and Singh, 2016). Heat treatment also results in grain size reduction within the aluminum matrix. Smaller grains enhance the material's mechanical properties, including

hardness (Zhao et al., 2019). Heat treatment can enhance the bonding between the matrix and the reinforcement particles, promoting load transfer and strengthening the material. This improved interfacial bonding is addressed by J. Ma et al. (2018).

Heat treatment aids in the uniform redistribution of reinforcement particles, such as Al₂O₃ and ZnO, ensuring a more homogenous microstructure and improved mechanical properties (Haider et al., 2021). The heat treatment process can also alter the morphology of precipitates within the composite, leading to improved hardness (Li et al., 2021). These processes lead to improved mechanical and electrical properties.

Praveen et al. (2016) investigated the effect of reinforcement on aluminum-based matrix composites. They reported the influence of morphology, density, type of reinforcement particles, and distribution which depends on the property of a particulate composite. Azeez et al. (2021) analyzed the effect of heat treatment on the microstructure properties of Al 6063 alloy. They performed heat treatment at 450⁰C and soaked for an hour using a heat treatment furnace and rapidly quenched water taking into consideration the size and dimension of the sample specimen. The morphology of their composites revealed the balanced and homogenous distribution of the reinforcement particles within the matrix of Al 6063 alloy for heat treatment.

These reasons are supported by recent literature review references, providing comprehensive insights into the microstructural changes observed in Al composites after heat treatment as a result, electro-mechanical properties improve in comparison to as-casted condition. In our microstructure observation by SEM and optical microscope, we also notice that nano Al₂O₃ is uniformly distributed in our developed metal matrix composite for its lower density, type, and size of reinforcement of particles which goes in line with the findings of the above researchers.

CHAPTER 5

REGRESSION ANALYSIS OF ELECTRO-MECHANICAL PROPERTIES OF AL COMPOSITE

5.0 Introduction

To optimize and predict the properties of Al composites, it is crucial to employ effective experimental design and statistical analysis techniques such as Response Surface Methodology (RSM) and Taguchi's technique. The main objective of this chapter is to apply the Response Surface Methodology to optimize and predict the hardness and electrical conductivity of Al composites after heat treatment. Also, the specific wear rate of Al Composite will be characterized using Taguchi's technique.

Furthermore, this chapter aims to establish predictive models that can accurately estimate the response variables based on the input variables, thus enabling efficient and cost-effective optimization. The findings of this research can benefit industries that utilize Al composites, such as aerospace, automotive, and electronics, by providing guidelines for process optimization and enhanced material performance.

5.1 Prediction and Optimization of Heat Treatment Effects on Electro-Mechanical Properties

Response Surface Methodology (RSM) is a powerful statistical tool that has been widely used for modeling, optimization, and prediction in various engineering fields. RSM allows the investigation of multiple factors simultaneously and provides a systematic approach for exploring the relationship between input variables and response variables. By utilizing RSM, we aim to identify the optimal combination of heat treatment parameters that maximize the desired mechanical and electrical properties.

5.1.1 Response Surface Methodology

RSM (response surface methodology) is a factorial/experimental design for optimizing a strong bond between one or more target variables. This pragmatic method has been recommended by Box and Wilson (1951). Moreover, it is the key component for gaining the finest results through an array of exclusive experiments. To achieve it, normally, the

second-degree polynomial model is used. Even though RSM is a hypothetical model, scientists and researchers practice it for estimations.

RSM helps examine the connection between the input variables and any process or system's responses (output). Its main intention is to advance the response time or to reach the scope of betterment in the work. The response surface methodology (RSM) is used to analyze the rapport when the input factors are quantitative. Additionally, the variables (x_1) and (x_2) maximize the yield of a process (Y). To put it briefly, the variables influence the process yield, as mentioned below:

$$y = f(x_1, x_2) + \varepsilon$$

(5.1)

Indeed, RSM is expedient in creating and analyzing the system, as the aim is to optimize the impact response by various factors.

As RSM is very important in the formulation, creation, and implementation of new engineering research and products, it is more predominant in the fields of industrial, manufacturing, material development, aeronautical engineering, and engineering sciences. It is a known fact that it has many applications; therefore, researchers explore its origin. In addition, many researchers have used RSM for manufacturing and engineering applications. For example, surface roughness and cutting force components are modelled analytically by using the response surface methodology (RSM). Its outcome determines the workpiece's feed rate and its hardness impacts the cutting force components. Nevertheless, the surface roughness is affected by both the workpiece's feed rate and hardness (Nandan, 2021).

RMS's efficiency depends on the accuracy of y at various points throughout the response surface; therefore, the researcher determines its optimal or improved system's reaction. From the very beginning of the research, the researcher studies the components or variables of the response surface. Correspondingly, non-significant independent variables are isolated from the critical ones with a view to conducting a good experiment. Also, it is necessary to focus on the essential components before the examination study. In addition, Hill and Hunter divide the response surface analysis into four phases (Myers, Khuri and Carter, 1989). The specifics are as follows:

- The experiments can be planned as statistically valid experiments.
- Compute the coefficients for the response surface equation.

- Test the equation by using the lack-of-fit test to see if this is correct.
- Test the response surface regions target by examination.

RSM manipulates the precise design of experiments (DoE), which has recently acquired favor for formulation. Along with this, its statistical approach is used to assess the interaction effect between the process factors than the traditional approach.

RSM (response surface methodology) is a computational and scientific technique for modelling and analyzing situations that consider various factors that impact the desired response and attempt to maximize the result (Montgomery, 2013). The connection between the output and the independent variables is unknown in most of the RSM issues (Noordin et al., 2004). In a similar fashion, RSM estimates the outcome variable ‘y’ and the set of independent variables ‘x’ first. A low-order polynomial is commonly used in several portions of the response variable. The functional approximation in the first-order model is well represented by the independent parameters and it is given below (Montgomery, 2013):

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \dots + \beta_kx_k + \varepsilon$$

(5.2)

When the structure has curvature in the second-order form, a higher-grade polynomial is used (Montgomery, 2013). Approximation polynomials’ parameters are estimated by using the least-square approach. So, a surface response analysis is performed on the linked surface. Besides that, the analysis of the installed surface is compared with the analysis of the actual system, when the installed surface is a reasonable approximation to the genuine response function (Palanikumar, Karunamoorthy and Karthikeyan, 2006).

Consecutively, the model variables envisage the most suitable experimental designs for data collection. Eventually, the surface design is considered as the suitable responsive surface. Undoubtedly, RSM is a method for identifying the system’s optimal process parameters or a factor space area that successively meets (Ali, Naseh and Ommi, 2020). Likewise, simultaneous analysis of multiple responses commences with appropriate response surface models for each result. Later, it pursues to optimize a set of operating conditions and keeps all responses within the necessary range at the lower limit (Yadav 2017).

5.1.2 Flow chart of RSM

To comprehend the total strategy in Response Surface Methodology, the accompanying stream graph is shown in Figure 5.1. It tends to be done by utilizing the following accompanying advances:

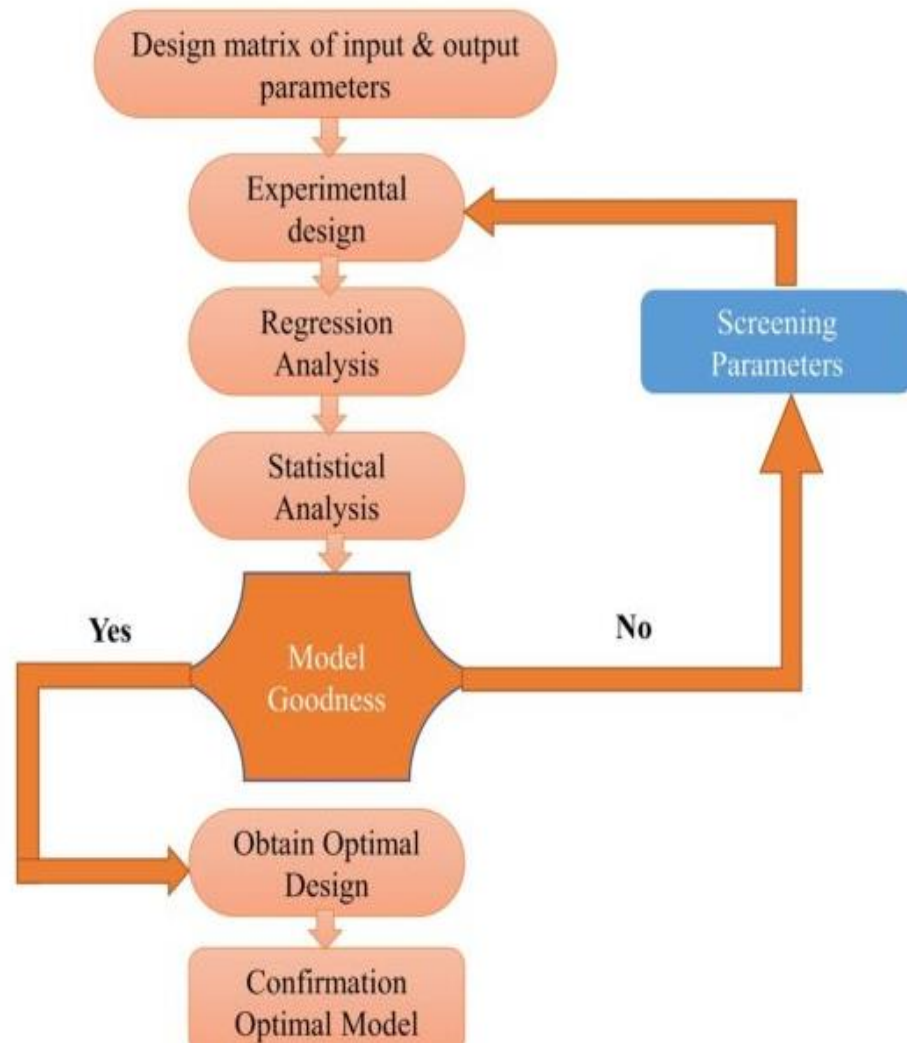


Figure 5.1: Flowchart of RSM

1. Distinguishing proof of the significant autonomous factors with their upper and lower limits.
2. Improvement of the planning framework.
3. Perform explores according to runs produced by programming.
4. Put the upsides of reaction boundaries in the plan network.

5. Check the model fitting either (straight, 2 FI, or quadratic).
6. Foster a proposed demonstration and ascertain the relapse coefficients.
7. Really look at the sufficiency of the proposed model.
8. Test the meaning of coefficients.
9. Break down the outcomes.
10. Mathematical improvement

5.1.3 Central Composite Design (CCD)

A central composite design (CCD) is a statistical and mathematical technique employed in experimental design and analysis, particularly in the context of optimizing various processes. CCD is a type of response surface design that helps to explore the relationships between multiple input factors (independent variables) and their impact on one or more response variables (dependent variables). The following are key components of a CCD as shown in Figure 5.2:

- **Factorial Points:** CCD begins with a factorial design that covers the low and high levels of input factors to capture linear effects.
- **Axial Points:** These points are added to the design to assess potential quadratic effects.

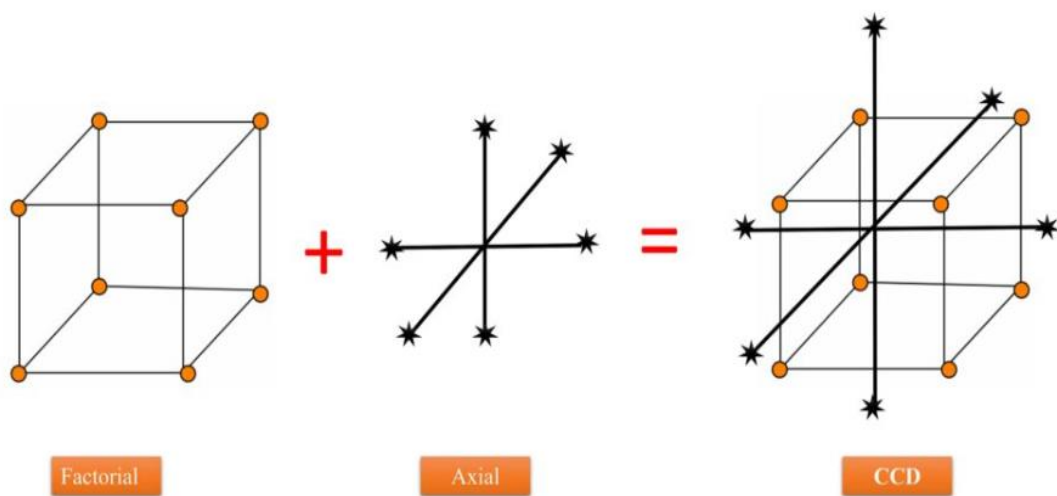


Figure 5.2: key Components of a Central Composite Design (CCD)

CCD systematically vary heat treatment parameters such as solution temperature and thermal aging temperature. The variations of solution temperature and thermal aging temperature impact the electro-mechanical properties of Al composites (e.g., hardness and electrical conductivity). CCD works as follows:

- **Factors (Independent Variables):** The factors would be the heat treatment parameters. For example, solution temperature and thermal aging temperature.
- **Response Variables:** These are the properties to be studied, such as Vickers microhardness, Rockwell hardness, and electrical conductivity.
- **Experimental Runs:** CCD allows to systematically design a series of experiments to determine how changes in the heat treatment parameters affect your response variables.
- **Model Fitting:** Using the data obtained from these experiments, statistical models can be fitted to predict how changes in the input parameters influence the response variables. These models can then be used to optimize the heat treatment conditions for desired properties.
- **Response Surface:** In RSM, a response surface is a graphical representation of the relationships between your input factors and the response variables. This helps visualize the optimal regions for the desired properties.

By applying CCD and RSM to the heat treatment process, the best combination of heat treatment parameters can be efficiently determined to achieve the desired mechanical and electrical properties in Al composites. This approach can significantly improve the efficiency and effectiveness of research in optimizing the heat treatment process for Al composites.

5.1.4 Design Matrix

The goal of the current study was to use Response Surface Design, based on Central Composite design, to simulate and optimize the various heat treatment processes of manufactured Al composite. The relationship between the answer and a few independent variables is mathematically modeled. The coefficients of mathematical modeling based on the response surface regression form were determined using the MINITAB program version 18. The selected Design Matrix is shown in Table 5.1.

Table 5.1: Design matrix of Central Composite Design (CCD)

Type of Design	Central Composite Design	
Number of Input Variables	02	
Input Variable 01: Solution Temperature (°C)	510°C (Low)	550°C (High)
Input Variables 02: Thermal Aging Temperature (°C)	140°C (Low)	220°C (High)
Number of Output Variables	02	
Output Variables 01	Vickers Micro Hardness	
Output Variables 02	Electrical Conductivity	

5.1.5 Experimental Results

As indicated in Table 5.2, Central Composite design of RSM, DOE ran a total of 14 experiment runs using the optimized model of heat treatment for Al MMC. According to the best results obtained from DOE, a confirmation experiment was carried out. Table 5.2 displays the experimental findings for the specified matrix for Vickers microhardness (HV) and electrical conductivity (%IACS).

Table 5.2: Experimental results of Central Composite design (CCD)

Run Order	Pt Type	Blocks	Solution Temperature(°C)	Thermal Aging Temperature(°C)	HV	%IACS
1	0	2	530.00	180.00	43.99	49.47
2	-1	2	501.72	180.00	42.08	48.53
3	-1	2	530.00	123.43	42.15	48.33
4	-1	2	530.00	236.57	43.83	45.70
5	-1	2	558.28	180.00	43.42	47.93
6	0	2	530.00	180.00	43.99	49.47
7	0	2	530.00	180.00	43.99	49.47
8	0	1	530.00	180.00	43.99	49.47
9	1	1	510.00	140.00	40.61	46.16
10	1	1	550.00	220.00	41.95	45.99
11	1	1	550.00	140.00	41.33	48.12
12	1	1	510.00	220.00	43.39	45.41
13	0	1	530.00	180.00	43.99	49.47
14	0	1	530.00	180.00	43.99	49.47

The non-linear mathematical model based on CC has been developed for the response of HV, RHN, and %IACS having a continuous factor of Solution temperature (lower level: 510°C and higher level: 550°C) and Thermal aging (lower level: 140°C and higher level: 220°C). Significance and ANOVA tests have been carried out to check the statistical adequacy of the models.

5.1.6 Statistical Analysis

Statistical analysis is a critical component of research, especially in fields where experimental design and optimization play a pivotal role. In the context of current research on the heat treatment of Al composites, the application of Analysis of Variance (ANOVA) under the umbrella of Response Surface Methodology (RSM) is crucial for understanding the impact of various heat treatment parameters on the electro-mechanical properties of Al Composite.

Understanding the intricate relationships between heat treatment parameters such as solution temperature and thermal aging along with the electro-mechanical properties of aluminum composites is a fundamental pursuit. To achieve this, Statistical Analysis, particularly Analysis of Variance (ANOVA), stands as a robust and indispensable tool. This statistical technique, when harnessed under the framework of Response Surface Methodology (RSM), enables a comprehensive investigation into the influence of solution temperature, thermal aging temperature, and other critical factors.

Through ANOVA, the significance of each factor and their interactions with properties like hardness and electrical conductivity can be rigorously assessed. This research explores the application of ANOVA within RSM to discern the optimal heat treatment conditions for achieving superior electro-mechanical properties in Al composites.

5.1.7 Analysis of Electro-mechanical Properties

According to the established model, 89.29% and 80.12%, respectively, are the derived values of R^2 and $\text{adj } R^2$ for Vickers microhardness. Additionally, for electrical conductivity, R^2 and $\text{adj } R^2$ values were determined to be 91.5% and 84.22%, respectively. The polynomial performs better at characterizing the system's behavior when the R^2 values are higher. This set of parameters is the only set where the model is valid (solution temperature and thermal aging temperature).

5.1.8 Study of Variance

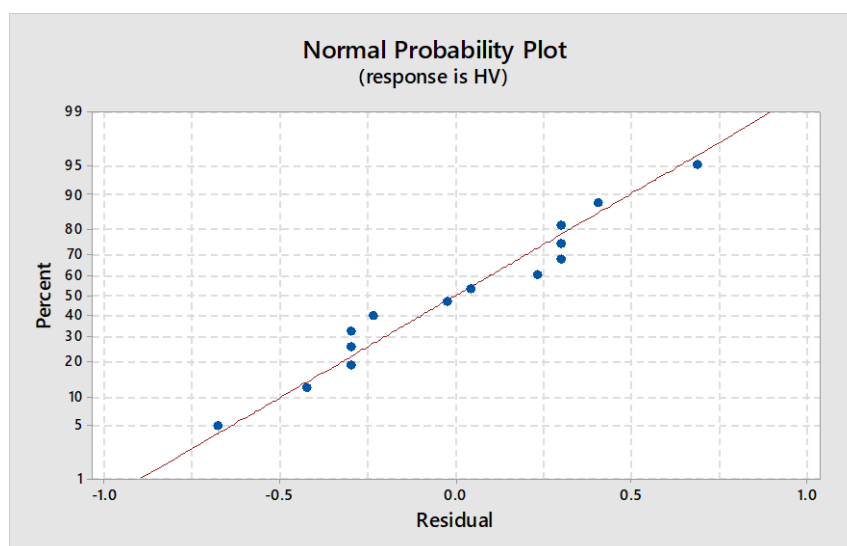
A 95% confidence level and a 5% significance level were used in the regression analysis for Vickers microhardness (HV) and electrical conductivity (%IACS). The relevance of numerous aspects, including the regression model, linear terms, 2-way interaction terms, and lack of fit, was determined by analysis. To determine whether or whether the results are statistically significant, one uses the P-value. Regression equations (5.3) through (5.4) were determined based on the analysis, and they are as follows for Vickers microhardness and electrical conductivity:

$$\text{Regression Equation of HV} = -663 + 2.467 \text{ ST} + 0.547\text{TA} - 0.002206 \text{ ST}*\text{ST} - 0.000477 \text{ TA}*\text{TA} - 0.000675 \text{ ST}*\text{TA} \quad (5.3)$$

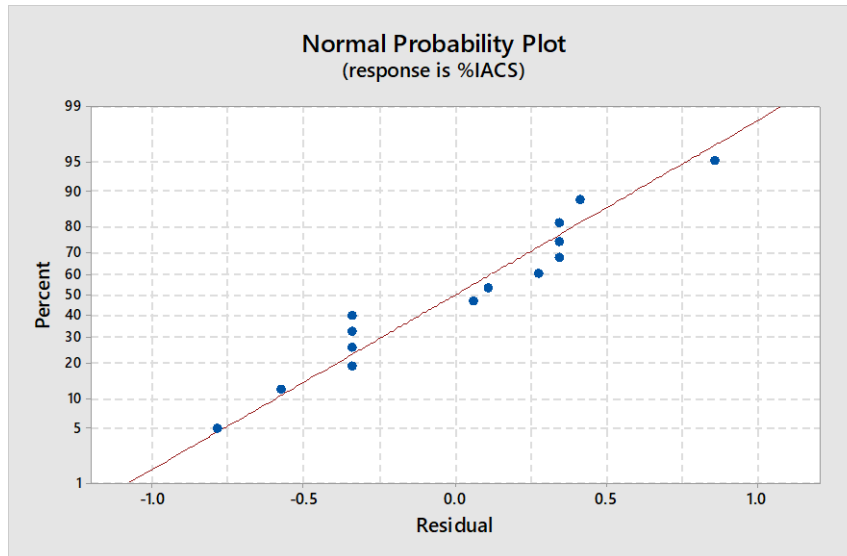
$$\text{Regression Equation of \%IACS} = -670 + 2.524 \text{ ST} + 0.550 \text{ TA} - 0.002298 \text{ ST}*\text{ST} - 0.000954 \text{ TA}*\text{TA} - 0.000429 \text{ ST}*\text{TA} \quad (5.4)$$

Where ST is Solution temperature at °C and TA is Thermal Aging at °C.

Vickers microhardness and electrical conductivity of the manufactured Al MMC are all impacted by linear and two-way interactions. Eqs (5.3) – (5.4) show that the HV and %IACS are affected by positive sign parameters for increasing and negative sign parameters for decreasing, respectively. The normal probability plots for Vickers microhardness and electrical conductivity are shown in Fig. 5.3 (a) through (b) respectively.



(a)



(b)

Figure 5.3: Normal Probability Plot; (a) Vickers micro hardness and (b) Electrical Conductivity

In the context of our research, the construction of normal probability plots stands as an essential component, offering valuable insights into the electro-mechanical properties of aluminum composites. Vickers microhardness and electrical conductivity are key parameters that are rigorously examined.

These plots as shown in Figures 5.3 (a) to 5.3(b) provide a visual representation of data distribution, allowing us to evaluate the normality of the data, which is pivotal for accurate statistical analysis. As such, we precisely assessed the effects of various heat treatment parameters, including solution temperature and thermal aging, on the Vickers microhardness and electrical conductivity of aluminum composites.

5.1.9 Comparison between Experimental and Predicted Results

Mathematical modeling provided Equation (5.3) - (5.4) for the prediction of Vickers microhardness (HV) and Electrical conductivity (%IACS) respectively for fabricated Al MMC. For each solution temperature (ST) and aging temperature (AT), Eqs (5.3) - (5.4) provide a predicated value of HV and %IACS respectively as shown in Table 5.3. We also calculated the Error percentage (%) of predicated HV and % IACS with respect to the experimental results as below. The negative (-) sign in Error % Calculation means that predicated results are found higher in some cases than the experimental results.

Table 5.3. Analysis of Experimental and Predicated Results with % of Error

Factor 1	Factor 2	Experimental Results		Predicted Results		% of Error	
		HV	%IACS	HV	%IACS	HV	%IACS
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%
501.72	180.00	42.08	48.53	41.49	47.23	1.41%	2.69%
530.00	123.43	42.15	48.33	40.94	47.50	2.88%	1.72%
530.00	236.57	43.83	45.70	42.92	45.15	2.08%	1.21%
558.28	180.00	43.42	47.93	41.89	47.85	3.52%	0.17%
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%
510.00	140.00	40.61	46.16	40.43	47.20	0.46%	-2.26%
550.00	220.00	41.95	45.99	42.11	45.97	- 0.39%	0.05%
550.00	140.00	41.33	48.12	41.79	48.32	- 1.11%	-0.43%
510.00	220.00	43.39	45.41	42.91	46.22	1.11%	-1.79%
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%
530.00	180.00	43.99	49.47	43.45	49.38	1.22%	0.18%

5.1.10 Analysis of Response Optimization

It is possible to derive the Vickers microhardness (HV) and electrical conductivity (%IACS) and optimality searches based on the proposed second-order response surface equations, i.e., Eqs (5.3) – (5.4). This is done to determine the best electro-mechanical parameter combination and how it will affect the required response criterion (Llorca, 2002). Response surface methodology is the foundation of the optimality search model for the various process variable positions for optimizing the %ICAS and HV values.

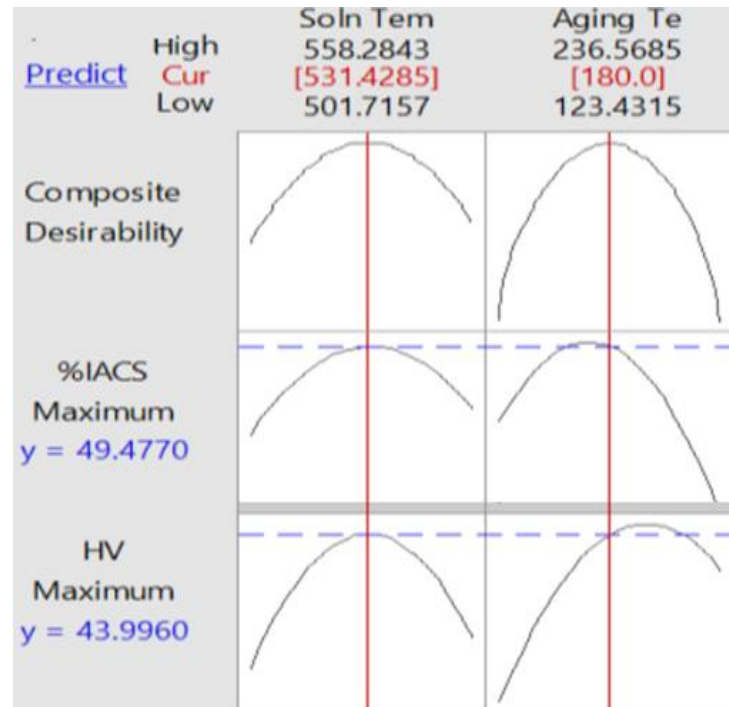


Figure 5.4: Optimum Response results for maximum %IACS and HV

Figure 5.4 shows that Solution Temperature of 531.428⁰C and Aging Temperature of 180⁰C as the most favorable values of Electrical Conductivity (%IACS) and Vickers micro hardness (HV) which are 49.4770 % IACS and 43.9960 HV, respectively, through the optimized parametric combination.

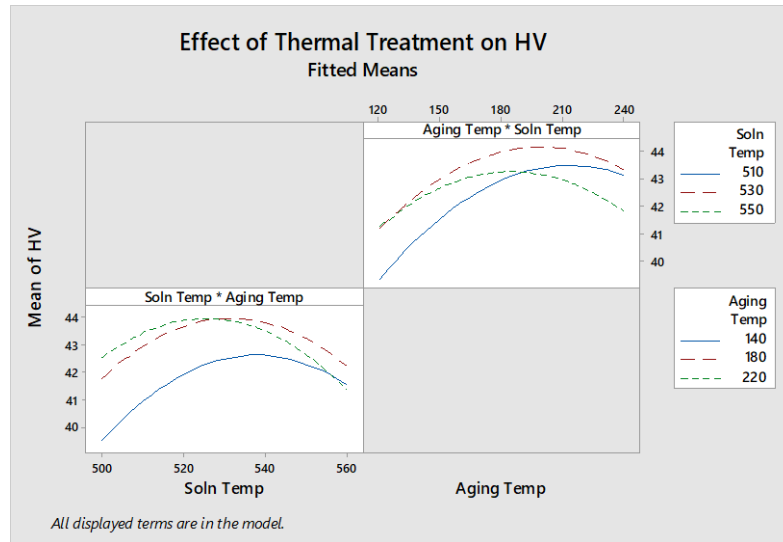
5.1.11 Fitted Means and Surface Plot

As per the central composite (CC) design of RSM, a total of 14 set heat treatments were carried out with different solution temperatures from 500⁰C to 560⁰C and aging temperatures from 120⁰C to 240⁰C for 1 hour for investigation of Rockwell hardness. The highest Vickers microhardness (HV) and Electrical conductivity (%IACS) were obtained for the solution temperature of 530⁰C and thermal aging at 180⁰C through Fitted means and surface plot.

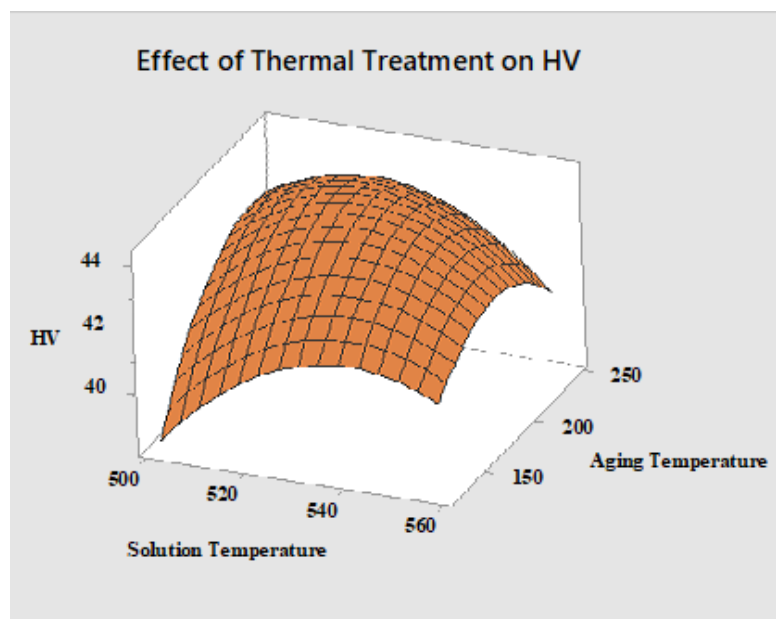
5.1.12 Fitted Means and Surface Plot of Vickers Micro Hardness

As per Figure 5.5 (a) and 5.5 (b), the highest Vickers microhardness of 43.99 HV was obtained for the solution temperature of 530⁰C and thermal aging at 180⁰C which is an improvement of 23.15% of HV in comparison to as-casted condition. We also observed that aging at higher temperatures, especially after 200⁰C led to softening of the alloy, and

the ductility also decreased. As a result, hardness also decreased with an increase in aging temperature.



(a)



(b)

Figure 5.5 Vickers microhardness of Al Composite; a) Fitted means, (b) Surface plot

The results of this study indicate that heat treatment has a significant effect on the hardness of the aluminum composite. The hardness of the composite increases with an increase in the aging temperature, up to a certain point, and then decreases with a further increase in the aging temperature. This behavior can be explained by the precipitation of hardening

phases during the aging process, which increases the strength and hardness of the composite up to a certain point.

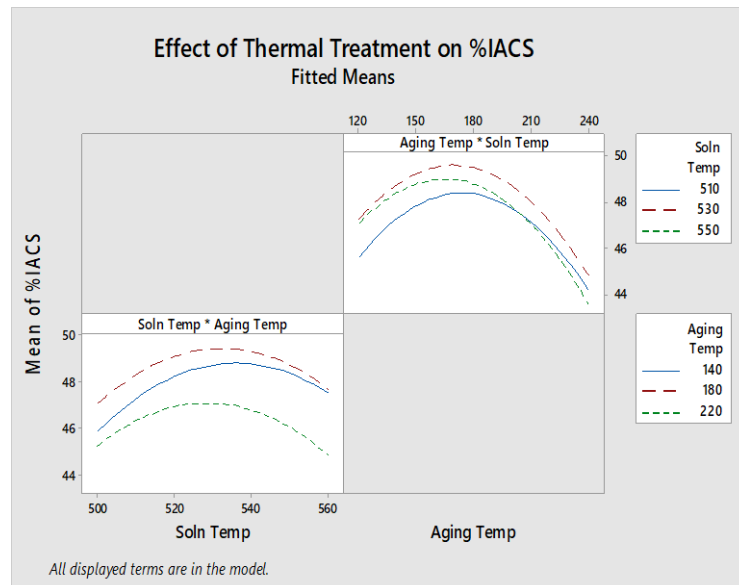
Beyond this point, over-aging can lead to the coarsening of these phases, which can decrease the hardness and strength of the composite. The results also show that the composite exhibits higher hardness values when subjected to a combination of solution treatment and aging compared to just aging. This can be attributed to the fact that the solution treatment allows for a more uniform distribution of the hardening phases, leading to an overall increase in the hardness of the composite.

Farokhpour et al. (2022) investigated the heat treatment effect of aluminum alloy at an aging temperature from 180⁰C to 230⁰C and observed the hardness and microstructure. As per their investigation, they observed the highest hardness values of an aluminum alloy at an aging temperature of 180⁰C. Our findings go in line with Farokhpour et al. (2022).

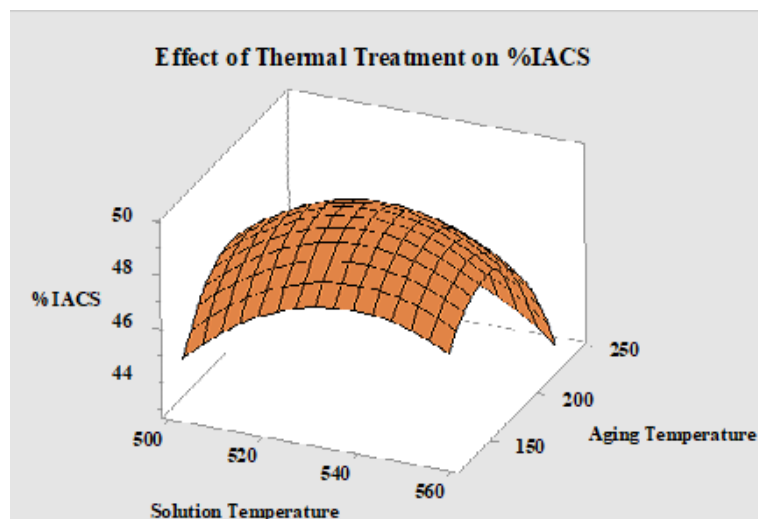
5.1.13 Fitted Means and Surface Plot of Electrical Conductivity

As per Figure 5.6(a) and Figure 5.6(b), the highest electrical conductivity of 49.47 %IACS was obtained for the solution temperature of 530⁰C and thermal aging at 180⁰C which is an improvement of 9.57% of electrical conductivity in comparison to the as-casted condition.

Debih et al. (2023) investigated the influence of heat treatment on mechanical properties and electrical conductivity of AA6101 Aluminum alloy. In their experiment, they performed heat treatment of Al alloy in three categories namely natural aging, artificial aging, and a combination of natural aging. As per their investigation, they observed the highest improvement of microhardness (HV) and electrical conductivity (%IACS) a combination of natural aging at 20⁰C for 72 hours and artificial aging at 180⁰C for 6 hours. In our present research work, we performed a combination of natural aging of 72 hours and artificial aging of a constant time of 1hr at different temperatures from 120⁰C to 240⁰C as per CC of RSM. Here the progressive improvement of electrical conductivity up to an aging temperature of 180⁰C. Therefore, our current findings go in line with the results outcome of Diehl et al. (2023).



(a)



(b)

Figure 5.6: Electrical conductivity of Al composite (a) Fitted means; (b) Surface plot

5.2 Characterization of Specific Wear Rate Using Taguchi's Technique

In the field of materials engineering, the characterization of wear rates plays a crucial role in determining the performance and durability of various materials. Specifically, in this study, we focus on the characterization of the specific wear rate of developed Al composite using Taguchi's technique. Al composites are widely used in numerous industrial applications due to their excellent strength-to-weight ratio, corrosion resistance, and

thermal conductivity. However, understanding the wear behavior of the developed Al composite is essential for optimizing their performance and extending their service life in applications that involve sliding or abrasive contact.

5.2.1 Taguchi's Technique

Taguchi's technique, developed by Dr. Genichi Taguchi, provides a powerful and systematic approach for conducting experiments to optimize process parameters and minimize variations in performance. By utilizing Taguchi's methodology, we aim to comprehensively analyse the specific wear rate of Al composite under varying experimental conditions. The specific wear rate is a fundamental parameter that quantifies the material loss per unit sliding distance or load, providing valuable insights into the wear behavior of materials. Through this study, we seek to identify the key factors affecting the specific wear rate of Al composite and determine their optimal levels, leading to enhanced performance and reduced wear.

The findings from this study will contribute to the understanding of wear mechanisms in aluminum composites, guide the selection of optimal parameters for specific applications, and provide valuable insights for future material development and design optimization. Ultimately, this research aims to enhance the overall performance and durability of aluminum composites in wear-critical environments, ensuring their successful application in various industries.

One of the most palatable statistical methods for examining the effects of numerous process parameters simultaneously is the design of experiments (DOE) found by Radhika et al. (2011). Using Taguchi's L9 orthogonal array, Suryakumari and Ranganathan (2018) investigated the wear behavior of an aluminum hybrid composite reinforced with 2.5% Al₂O₃ under applied loads ranging from 10 to 30 N and sliding velocities ranging from 500 to 1050 RPM. Prakas et al. (2014) investigated the sliding wear behaviour of Al₂O₃-reinforced metal matrix composites with applied loads ranging from 15 N to 45 N and sliding speeds ranging from 390 RPM to 780 RPM. Poovalingam Muthu (2020) used the Grey-Taguchi method to analyze the wear behavior of Aluminum MMCs while applying loads ranging from 10 N to 30 N and sliding between 780 RPM and 1050 RPM.

In the present study, the wear properties of developed Al composite reinforced with 2.5 wt.% of Al₂O₃ were investigated at different parameters such as applied load varying from 20N to 50N, sliding duration from 5 to 15 minutes, and sliding speed at RPM of 100 to 200. Also, a Taguchi-based L9 orthogonal array was chosen for the prediction and optimization of those process parameters.

5.2.2 Pin-on-Disc Wear Setup

A pin-on-disc apparatus was utilized in the experiment, as depicted in Figure 5.7, and sample specimens were made in accordance with ASTM G99-17. The fabricated Al composite was used to create cylindrical pins with dimensions of 5 mm in diameter and 12 mm in height. The fabricated Al composite had a Brinell hardness of 139.32 and Vickers hardness of 35.72 respectively. All the pins were thoroughly cleaned and dried before the wear tests. The disc used for this experiment was from Muzibur and Reaz (2021) having SS 309s material containing 60% Fe, 23% Cr, 14% Ni, 2% Mn, 0.84% Si, 0.08% C, 0.05% P, and 0.03% S as the counter-body. The hardness of the stainless-steel disk was found on average as 168 HV made of stainless steel having a diameter of 0.049m. The pin and disk weights were calculated using an electronic balance with a 0.001 mg accuracy for the wear test.

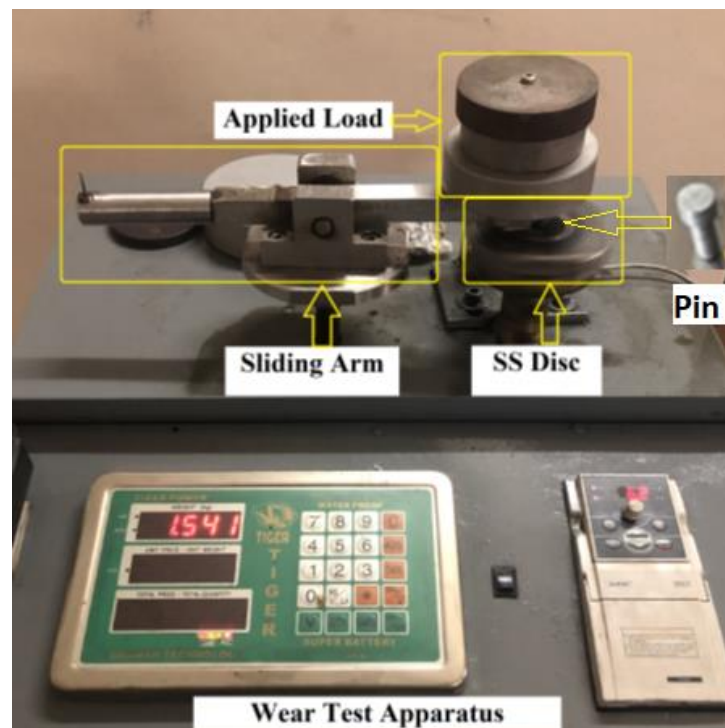


Figure 5.7: Pin-on-Disc Wear Setup

5.2.3 Design of Experiments (DOE)

DOE can examine the effects of numerous process parameters simultaneously. For monitoring the results of those tests, each experiment requires a combination of the process parameters and their levels. For determining the impact of those process parameters, the Taguchi technique uses the process parameters in specified orthogonal arrays. The planning stage, conducting stage, and analysis stage are the three key phases that make up the DOE. By analyzing the results of the experiments, the S/N ratio is utilized to identify the process parameters that would produce the best results.

The purpose of the current work was to determine the minimum specific wear rate as much as possible. In this experiment, the Diameter of the sliding disc (D) was kept fixed at 0.049m. Sliding Distance (SD) and Specific Wear Rate (WR) were determined respectively using Equation (5.5) and Equation (5.6) followed from Muzibur and Reaz (2021).

$$\text{Sliding Distance (SD)} = \pi \times D \times N \times t \quad (5.5)$$

Where,

D= Diameter of the sliding disc (m),

N= Sliding speed (RPM)

T= Sliding Duration (Minutes)

$$\text{Specific wear rate (WR)} = \frac{\Delta W}{SD \times L} \quad (5.6)$$

Where,

WR= Specific Wear Rate (mg/Nm)

ΔW = Wear Loss (mg)

SD= Sliding Distance (m)

L= Applied Load.

In the current investigation, the L9 orthogonal arrays of Taguchi's approach were used to create the experimental plan as per selected process parameters and their levels are shown in Table 5.4. The ANOVA method was used to analyze these process parameters.

Table 5.4: Parameters and their levels for L9 orthogonal arrays of Taguchi's approach

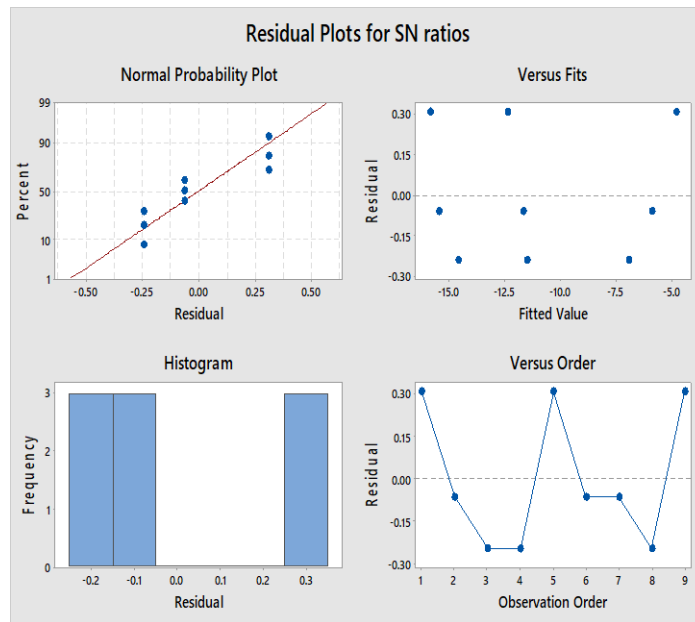
Level	Applied Load (L)	Sliding Speed (N)	Sliding Duration (t)	Sliding Distance (SD)
Units	N	RPM	Minutes	Meter
1	20	100	5	77
2	35	150	10	231
3	50	200	15	462

A total of 09 experiments were conducted as shown in Table 5.5 as per L9 orthogonal arrays of Taguchi's approach for different process parameters. The Specific wear rate was determined using equation (2). Signal-to-noise (SN) ratio and mean are given from the experimental outcomes of Taguchi's approach. Residual plots for SN ratio and mean are shown in Figures 5.8(a) and 5.8(b).

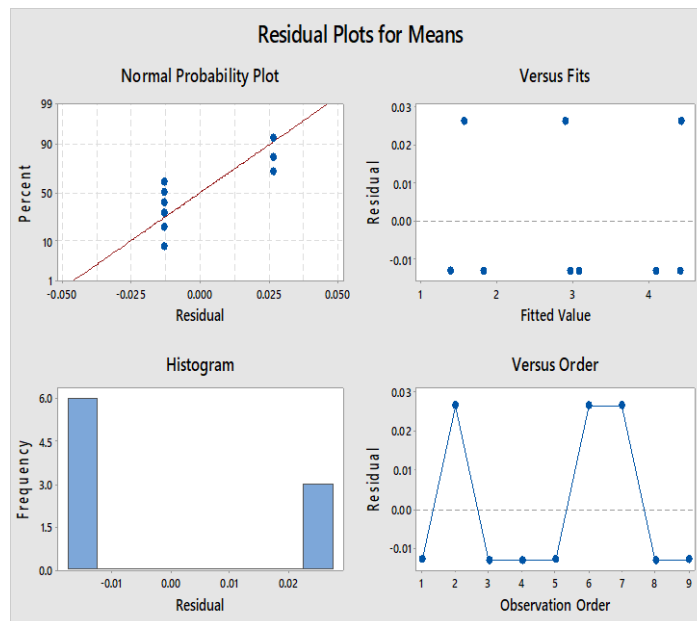
Table 5.5: Specific Wear Rate, Signal-to-noise ratio and Mean Using Taguchi's Technique

Applied Load (N)	Sliding Speed (RPM)	Sliding Duration (Mins)	Specific Wear Rate (mg/Nm)	Signal to noise Ratio	MEAN
20	100	5	2.3377	7.38	2.34
20	150	10	2.7706	8.85	2.77
20	200	15	3.2035	10.11	3.20
35	100	10	5.4545	14.74	5.45
35	150	15	5.6566	15.05	5.66
35	200	5	5.4545	14.74	5.45
50	100	15	8.4416	18.53	8.44
50	150	5	7.7922	17.83	7.79
50	200	10	8.4416	18.53	8.44

The crucial aspect of this analysis involves the examination of residual plots for signal-to-noise ratios and mean values as shown in Figure 5.8 (a) and Figure 5.8 (b) respectively. These residual plots play a pivotal role in assessing the effects of various experimental factors and their interactions on the specific wear rate of Al composites reinforced with nano Al₂O₃. By scrutinizing these plots, a deeper understanding of the variations in the signal-to-noise ratios and mean values has been gained, which are essential for identifying the optimal combination of input parameters in order to minimize wear rates.



(a)



(b)

Figure 5.8: Residual Plots; (a) Signal-to-Noise Ratios, (b) Mean values.

5.2.4 Analysis of Variance (ANOVA)

ANOVA was used to assess the experimental results and the effects of the process parameters that were taken into consideration, such as the applied load, sliding speed, and sliding time, which have a significant impact on the wear rate. This investigation was carried out using a 95% confidence level and a significance level of 5%. Response for Signal to noise ratio and Means were ranked and shown in Table 5.6 and Table 5.7.

Table 5.6: Response for signal-to-noise ratios with smaller is better

Level	Load	Speed	Duration
1	5.872	10.595	10.360
2	11.857	10.947	11.072
3	15.296	11.482	11.592
Delta	9.424	0.887	1.232
Rank	1	3	2

Table 5.7: Response for means

Level	Load	Speed	Duration
1	1.594	2.923	2.790
2	2.978	2.912	2.978
3	4.302	3.039	3.107
Delta	2.708	0.127	0.316
Rank	1	3	2

Tables 5.6 and 5.7 show that the applied load is the process variable that has the greatest impact on the wear rate of Al₂O₃ reinforced Al composite. Bhuvanesh and Radhika (2017) found that the dominating factor was applied load to the wear rate in their experimental study on the tribological properties of nitride-reinforced aluminum metal matrix composites. Consequently, our research supports Bhuvanesh and Radhika's findings (2017).

5.2.5 Regression Analysis

A regression model that illustrates the link between the independent variable and the response variable was produced by the statistical program "MINITAB 18". Equation (5.7) illustrates the interaction between applied stress, sliding speed, and distance on specific wear rate through ANOVA analysis. As per the analysis, the R² and adj R² values for this model are 99.85% and 99.76%, respectively. The combination settings of applied load, sliding speed, and duration have behavioral patterns described as the values of R² achieved 99.85%. Figure 5.9 represents the residual plot for specific wear rates.

The regression equation of specific wear rate can be expressed as follows:

$$WR = -1.863 + 0.18182 L + 0.002886 SD + 0.05724 t \quad (5.7)$$

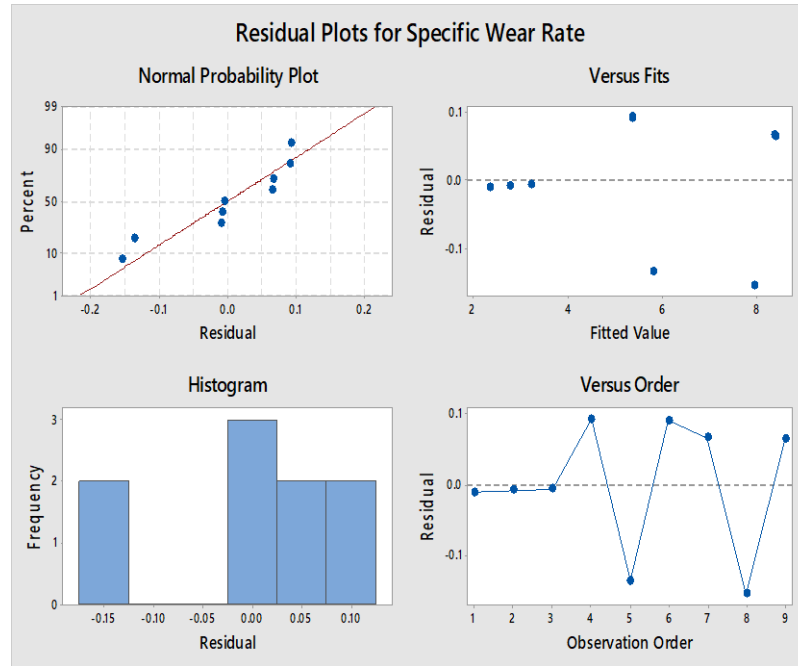


Figure 5.9: Residual plots for Specific Wear Rate

5.2.6 Study of Variance

Table 5.8 displays the significance of terms related to this model at a 5% significance level and a 95% confidence level. The significance of several aspects, including the regression model, terms, and lack of fit, is investigated using this analysis. By looking at the P-value, it is possible to assess whether the model's results are statistically significant.

Table 5.8: Regression coefficients for specific wear rate

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-1.863	0.208	-8.96	0.000	
Applied Load	0.18182	0.00317	57.33	0.000	1.00
Sliding Speed	0.002886	0.000951	3.03	0.029	1.00
Sliding Duration	0.05724	0.00951	6.02	0.002	1.00

5.2.7 Confirmation Test

In this current work, the confirmation test was carried out by choosing the process parameters as given in Table 5.9. The tests were carried out and the outcomes were compared with the predicted value given by the regression equation (5.7). The comparison between the experimental and predicted values is exhibited in Table 5.9.

Table 5.9: Confirmation test of specific wear rate with % of Error

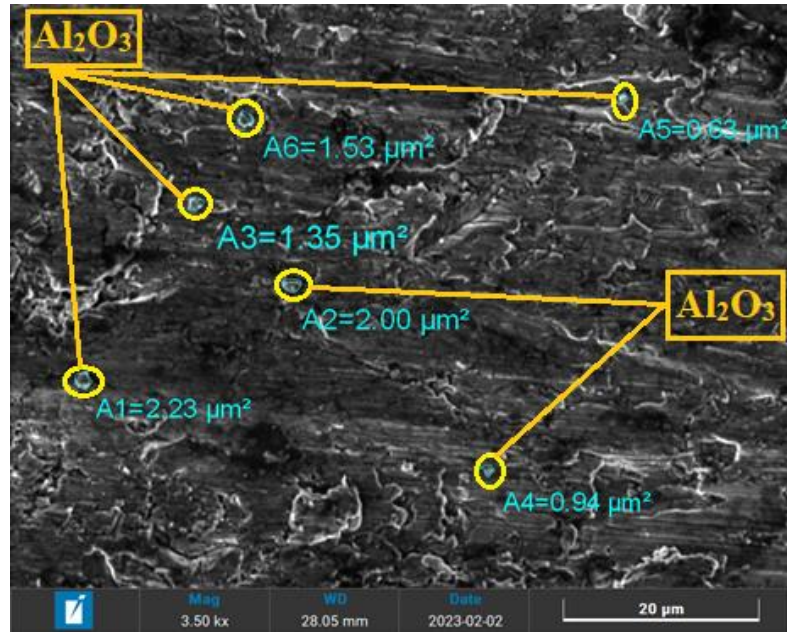
Applied Load (N)	Sliding Speed (RPM)	Sliding Duration (Mins)	Experimental	Prediction	Error (%) Calculation
			Specific Wear Rate (mg/Nm)	Specific Wear Rate (mg/Nm)	
30	125	12	4.5455	4.64	2.1%
40	180	20	6.6378	7.07	6.6%

As per Table 5.9, we observed a 2.1% error between experimental and predicted values for the applied load of 30 N, sliding speed of 125 RPM for a duration of 12 minutes. Also, 6.6% error between experimental and predicted values for the applied load of 40 N, sliding speed of 180 RPM for a duration of 20 minutes. Rajesh et al. (2013) performed a confirmation test during their MOORA-Based tribological studies on red mud-reinforced aluminum metal matrix composites and observed a 4.2% difference between initial parameter and optimal parameters settings.

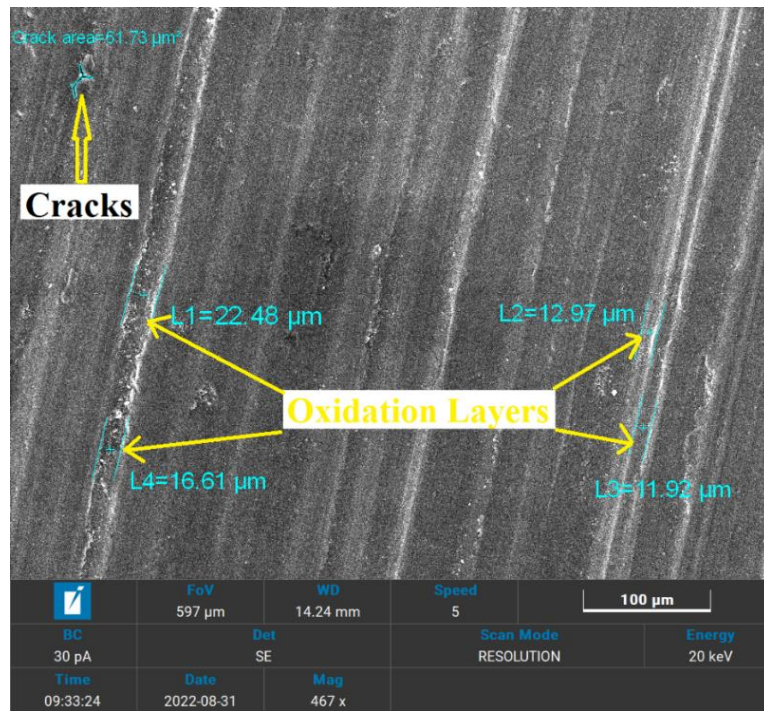
5.2.8 Microstructure Observation

After the preparation of Al composite by two-step stir casting, microstructure observation was carried out by Scanning Electron Microscope (SEM) in order to confirm the mixing of reinforcement particles (Al_2O_3) into a base material (99% Al). A sample of Al- Al_2O_3 with a dimension of 05mm×05mm×05mm was prepared for microstructure observation by Scanning Electron Microscope (SEM), Model: TESCAN VEGA 4.

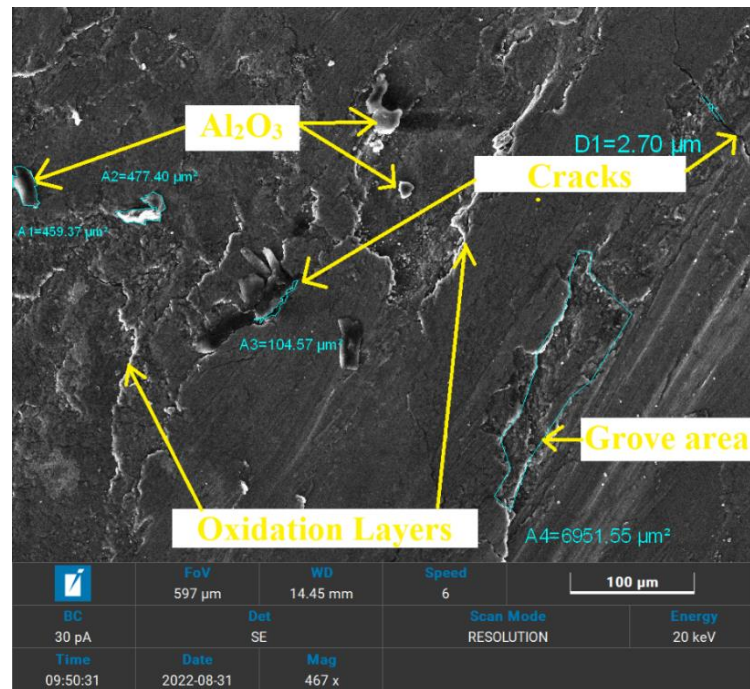
As shown in Figure 5.10 (a), the morphology of Al_2O_3 particles is mainly irregular. The Al_2O_3 particles were uniformly distributed in the Al- Al_2O_3 . The elliptical area immersed by the reinforcements (Al_2O_3) in Al composite is approximately $5.45 \mu m^2$ and $6.29 \mu m^2$ which is investigated through image processing as shown in Figure 7.4(a). Also, a few clustering or agglomerations of Al_2O_3 particles were perceived in the Al/ Al_2O_3 matrix composite.



(a)



(b)



(c)

Figure 5.10: Microstructure observation of (a) Material with reinforcement dispersion; (b) worn-out surfaces of the cylindrical pin at applied load: 20 N, sliding speed: 100 RPM and sliding duration: 5 mins; (c) worn-out surfaces of the cylindrical pin at applied load: 50 N, sliding speed: 200 RPM and sliding duration: 10 mins.

The microstructure of the worn-out surface of the cylindrical pins having the lowest and highest specific wear rates was also examined by SEM with a diameter of 5 mm as shown in Figure 5.10 (b) and Figure 5.10 (c) respectively. The process parameters of the pin having the lowest specific wear rate were 20 N as applied load, 100 RPM as sliding speed, and 5 minutes as sliding duration. The process parameters of the pin having the highest specific wear rate were 50 N as applied load, 200 RPM as sliding speed, and 10 minutes as sliding duration.

In Figure 5.10 (b), a worn-out surface was observed with minor grooves developed for adhesive wear. The minor grooves and adhesive wear marks were due to the selected process parameters. We identified a minor crack of 51.73 μm² and a few groove lines measured through SEM image processing denoted as L1 to L4 in Figure 5.10 (b).

In Figure 5.10 (c), the delamination observed at the worn-out surface of the pin having maximum specific wear rate was for the selection of process parameters such as 50 N as applied load, 200 RPM as sliding speed, and 10 minutes as sliding duration. As per SEM,

we observed deep grooves along with the surface level. The transition from minor grooves to deep grooves increased because of increasing applied load from 20N to 50 N, sliding speed from 100 RPM to 200 RPM, and sliding duration from 05 minutes to 10 minutes. As shown in Figure 5.10 (c), we identified 02 cracks at the worn surface of the pin, one having a width of 2.70 μm and another one having 104.57 μm^2 . We also observed a few chips of surface whose area measured as 477.40 μm^2 (A1) and 459.437 μm^2 (A2). One of the deep grooves measured 6951.55 μm^2 .

Rajesh et al. (2013) performed a tribological study on red mud-reinforced aluminum metal matrix composites. At the applied load of 20N load and a constant sliding distance of 3000 m, they identified formation of reinforcement particles at the surface which reduces the specific wear rate of the composite. As shown in Figure 7.4(b), we also observed a similar formation of reinforcement particles on the worn-out surface of the pin for which the specific wear rate was minimum at the applied load of 20 N, sliding speed of 100 RPM, and duration of 5 minutes. Natrayan and Kumar (2019) investigated the wear behavior on AA6061/Al₂O₃/SiC metal matrix composite. As per their investigation, it was perceived that the increase in applied load and sliding speed increases the wear rate of the composites due to the formation of larger grooves on the worn surfaces. They observed cavities and a few dimples formed for the applied load of 30N and at the sliding speed of 200 RPM for a sliding distance 1200 m. As per Figure 7.4(c), we also observe similar wear conditions for the process parameters such as 50 N as the applied load, 200 RPM as the sliding speed, and 10 minutes as the sliding duration. Therefore, we understand that our fabricated Al composite reinforced with Al₂O₃ has less abrasive wear.

5.2.9 Effect of Applied Load on Specific Wear Rate

The escalation of specific wear rate is observed with the increase of load from 20 N to 50 N nearly in linear proportion as shown in Figure 5.11. This trend in the plot is due to an increase in touching pressure between the composite pin and the disc which is the basis of greater surface damage. When the given load increases up to 35 N, wear is developed because of rubbing between cylindrical pins and SS disc. Also, acute wear is developed for adhesion when the applied load escalates from 35 N to 50 N and a similar pattern is observed by Radhika et al. (2014) at an applied load of 30N, sliding speed of 286 RPM for a sliding distance of 1500m. Prasat et al. (2011) observed that at low load and sliding speed, the worn pin surface showed shallow grooves in the direction of sliding. However, at higher loads and sliding speed, the alumina particles get fractured, and these particles act as sharp

asperities to remove more material from the wear surface revealed by Prasat et al. (2011) which is a similar finding as per the present microstructure observation shown in Figure 5.10 (b) and Figure 5.10 (c).

5.2.10 Effect of Sliding Speed on Specific Wear Rate

The escalation of the specific wear rate was also noticed with the increase of speed from 100 RPM to 200 RPM as shown in Figure 5.11. As the sliding speed of the disc escalates up to 150 RPM, the reinforcement particles of nano Al_2O_3 develops a thin layer on the surface which withstands high stress, and therefore, it reduces the sliding wear up to a sliding speed of 150 RPM as shown in Figure 5.11. Poovalingam Muthu (2020) applied a sliding speed of 780 RPM to 1050 RPM and studied the wear behavior of Aluminum MMCs using the Grey-Taguchi method. As per their study, a similar thin oxidation layer developed on the surface which withstood high stress and reduced the sliding wear after a certain duration. However, at higher sliding speeds, the temperature rises due to friction of the pin and SS disc. As a result, the material becomes soft and gets easily removed. Thus, the wear rate again increases at this condition which indicates a severe wear regime and delamination also becomes maximum at this condition investigated by Radhika et al. (2014).

5.2.11 Effect of Sliding Duration on Specific Wear Rate

An escalation of specific wear rates with an increase in duration was observed. Figure 5.11 exhibits a direct interrelation between the duration and specific wear rate. This phenomenon is for the presence of nano Al_2O_3 reinforcements that projects out of the pin surface and escalates the groove on the touching surface area. Tazari and Siadati (2019) observed the wear mechanism of material removal in an aluminum composite for 50 minutes and observed major adhesion and abrasion marks on the surface. As per Figure 5.10 (c), we also observe minor to major grooves for the duration from 5 minutes to 15 minutes which goes in line with the findings of Tazari and Siadati (2019).

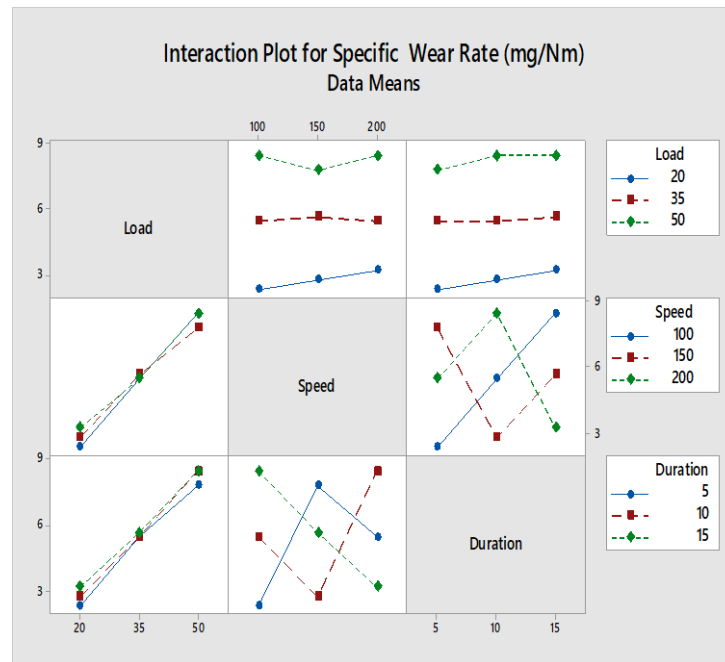


Figure 5.11: Interaction plot of Specific wear rate holding for a duration of 10 mins.

As per Taguchi's DOEs and ANOVA, the most effective parameter is the applied load which remarkably affects the specific wear rate. Next comes the sliding duration/distance and sliding speed can be considered as the least effective parameter for specific wear rate. A model has been developed as a regression equation to predict specific wear of nano- Al_2O_3 reinforced Al composite. As per the developed model, R^2 and adj R^2 are 99.85% and 99.76% respectively. Two confirmation experiments were done, and the outcomes of those experimental data were compared with the predicted value provided by the regression equation (5.7). The comparison between the experimental and predicted values from two confirmation tests shows an error of 2.1% and 6.6% respectively. Microstructure has shown a distribution of Al_2O_3 in the metal matrix with less agglomeration. Also, the microstructure of the worn-out surface has revealed narrow grooves and a minor crack on the surfaces for 20 N load, 100 RPM, and 5 minutes duration. Conversely, the microstructure of the worn-out surface having the highest specific wear rate displayed deep grooves and several cracks on the surfaces for the load of 50 N, sliding speed of 200 RPM, and sliding duration of 10 minutes. The effect of different sliding parameters on the specific wear rate of fabricated Al composite will assist to identify the ideal wear resistance applications of fabricated Al composite to the aerospace industry.

CHAPTER 6

CONCLUSION

6.1 Conclusions

The notable outcome and findings of the investigations are summarized as follows:

1. The microstructure observation of Two Al MMCs developed by a two-step stir casting method having compositions of 97.5% Al and 2.5% Al₂O₃ (Al MMC-1) and 95% Al, 2.5% Al₂O₃ and 2.5% ZnO (Al MMC-2) displayed an almost uniform distribution of nanoparticles with fewer agglomeration. The selection of process parameters was observed to influence the properties of developed Al composites.
2. The inclusion of 2.5% ZnO nanoparticles along with 2.5% Al₂O₃ in Al MMC-02 has increased surface hardness (Vickers Micro) from Al MMC-01 having 2.5% Al₂O₃ nano-particles in Al matrix by 41% respectively. This enhanced property of Al MMC-02 can contribute to the application of machine parts, bush bearings, etc.
3. Since flexural strength, impact toughness, modulus of elasticity, and electrical conductivity values were reduced by 12%, 52.5%, 60.8%, and 9.81% in Al MMC-02 in comparison to Al MMC-01 due to the presence of multiple cleavage cracks, deep shear dimples, etc. the applications of Al MMC-02 will not be at par of Al MMC-01.
4. For Al MMC-01, the highest increases of Vickers Mirco hardness were by 21.47%, 25.92%, and 22.68% for solution temperatures of 510°C, 530°C, and 550°C respectively in comparison to the results of as casted condition. For Al MMC-02, the highest increases of Vickers Mirco hardness were by 15.01%, 17.6%, and 12.10% for solution temperatures of 510°C, 530°C, and 550°C respectively in comparison to the results of as casted condition.
5. For Al MMC-01, the highest increases of electrical conductivity were by 8.83%, 9.57%, and 7.49% for solution temperatures of 510°C, 530°C, and 550°C respectively in comparison to the results of as casted condition. For Al MMC-02,

the highest increases of electrical conductivity were by 11.58%, 12.12%, and 9.91% for solution temperatures of 510°C, 530°C, and 550°C respectively in comparison to the results of as casted condition respectively.

6. Two non-linear mathematical models have been developed through Central Composite design based on response surface methodology (RSM) for prediction of Vickers microhardness and Electrical conductivity of manufactured Al MMC where R^2 obtained for HV and % IACS are respectively 89.29% and 91.50%.
7. The most favourable values of HV and %IACS were achieved at the optimized parametric combination of solution temperature of 531.428⁰C and aging temperature of 180⁰C.
8. As per the regression equation, the lowest and highest error (%) calculated between the experimental and prediction of HV are respectively 0.39% and 3.52%. For Electrical conductivity, the lowest and highest error (%) calculated between the experimental and prediction of %IACS are respectively 0.05% and 2.69%.
9. As per Taguchi's DOEs and ANOVA, the most effective parameter is the applied load which remarkably affects the specific wear rate of Al composite reinforced with 2.5 wt.% of Al₂O₃. A model has been developed with a regression equation to predict the specific wear rate of Al composite where R^2 and adj R^2 are 99.85% and 99.76% respectively. Two confirmation experiments were carried out having an error limit of 2.1% and 6.6% between the experimental and predicted values respectively. The microstructure of the worn-out surface has revealed narrow grooves and a minor crack on the surfaces for 20 N load, 100 RPM, and 5 minutes duration. Conversely, the microstructure of the worn-out surface having the highest specific wear rate displayed deep grooves and several cracks on the surfaces for the load of 50 N, sliding speed of 200 RPM, and sliding duration of 10 minutes.

6.2 Limitations of Present Study

It is imperative to acknowledge certain limitations that have impacted the scope and comprehensiveness of the present study. One constraint was the insufficient availability of nanoparticles, which hindered the preparation of tensile test specimens. Consequently, the tensile tests could not be conducted. Additionally, the experimental dataset utilized for the prediction and optimization of heat treatment effects on electro-mechanical properties was constrained by its size. The relatively small dataset may have implications for the generalizability and robustness of the findings, urging caution in extrapolating the results to broader contexts. Despite these limitations, the present study serves as a valuable foundation, laying the groundwork for future investigations to build upon and address these challenges for a more nuanced understanding of the subject matter.

6.3 Recommendations/Future Study

The insights from this study are expected to be a valuable reference for both academics and industries. While the current work establishes a solid foundation for understanding material characteristics, there are unexplored avenues for future research. Investigating the influence of nanoparticles on the tensile behavior of aluminum metal matrix composites, especially after various treatments, holds promise for informing composite design. Another crucial area for future exploration is the development of model-based predictions for the tensile properties of synthesized aluminum metal matrix composites, offering a significant tool for researchers and engineers. These avenues aim to address current gaps and foster innovation in material science, contributing to the ongoing advancement of knowledge in aluminum metal matrix composites.

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APPENDIX-A

Chemical Composition Analysis of Pure Aluminum

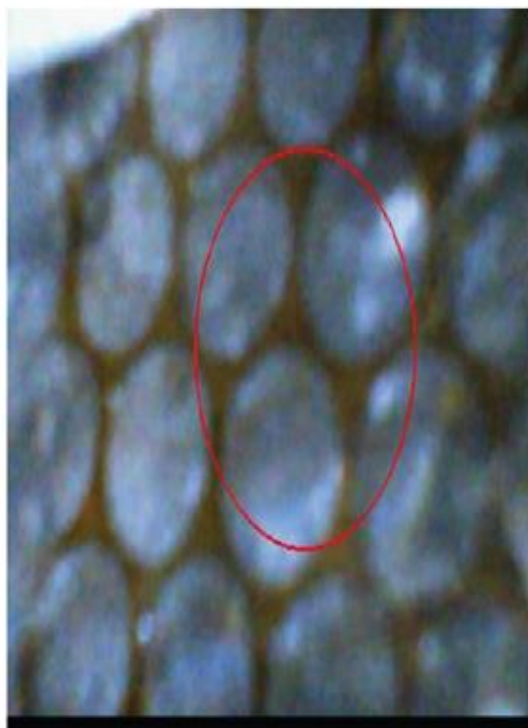


Method : AlloyPlus
 Aiming Image : 13
 Daily ID

Chemistry

Full Test Req'd; suggest 15 + seconds testing, Mg 0.4-0.9% cor

El	%	+/- 3σ	6063
Mg	ND	<0.42	
Al	99.052	0.072	
Si	0.614	0.068	
Cr	ND	<0.006	
Mn	ND	<0.005	
Fe	0.323	0.023	
Cu	0.002	0.002	
Zn	0.008	0.002	
Zr	0.0007	0.0005	Resid. 0.1
Pb	0.0009	0.0007	Resid. 0.1



APPENDIX-B

Certificate of Physical Properties of Alumina (Al₂O₃)



Hebei Suoyi New Material Technology co.,LTD.

NO.378 ZhongHua North Street,CongTai District,Handan City,Hebei Province

Tel:+86.310.6932202

Fax:+86.310.4560155

Sample name: <u>Nano Aluminium Oxide</u>	Quantity <u>1000kg</u>
Product name : <u>Nanometer alumina</u>	Batch NO <u>2021061801</u>
Formula: <u>Al₂O₃</u>	Test date) <u>20210619</u>
MOL WT.: _____	Item NO.) <u>VK- L20Y</u>

Inspection	Standard	Test Results
Appearance	White powder solid	White powder
PH value	6-9	Pass
Al₂O₃% ≥ Assay	99.99	99.99
Crysatl Structure and Type	gamma	gamma
Grain size(nm)	20	Pass
BET, m²/g (800°C 2h)SSA,	100-160	Pass
Bulk density g/cm³	0.1 -0.3	Pass
Si	≤0.003%	Pass
Fe	≤0.003%	Pass
Na	≤0.001%	Pass
Conclusion: <div style="text-align: center; margin-top: 20px;"> </div>		

Tester: 王晓斌

Re-tester: 钱敏敏

APPENDIX-C

Certificate of Physical Properties of Zinc Oxide (ZnO)



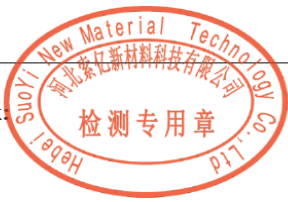
Hebei Suoyi New Material Technology co.,LTD.

NO.378 ZhongHua North Street,CongTai District,Handan City,Hebei Province

Tel:+86.310.6932202

Fax:+86.310.4560155

Product name : <u>Zinc Oxide</u>	Quantity <u>1000kg</u>
	Bach NO <u>20211025</u>
Formula: <u>ZnO</u>	Test date <u>20211026</u>
MOL WT.: _____	Item NO.: <u>SY-J30</u>

Inspection	Standard	Test Results
	white powder	Conform
Appearance		
Grain size, nm	30	Conform
ZnO% ≥	99.9	Conform
Density, g/cm ³	0.15-0.3	Conform
BET m ² /g	10-50	Conform
(%) ≤ Loss on dry	0.8	Conform
L. O. I (%) ≤ Sulphated assay	1.8	Conform
Conclusion:	Conform	
Remark:		

检验者(Tester): 陈超

审核者: (Re-tester): 杨智