

# **EXPERIMENTAL INVESTIGATION ON MECHANICAL AND WEAR CHARACTERISTICS OF AZ91 HYBRID COMPOSITES PRODUCED THROUGH FRICTION STIR PROCESSING**

**A Project Work Report**

**Submitted to**

**JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY, KAKINADA**

**In partial fulfillment of the requirements for the award of the degree of**

**BACHELOR OF TECHNOLOGY**

**IN**

**MECHANICAL ENGINEERING**

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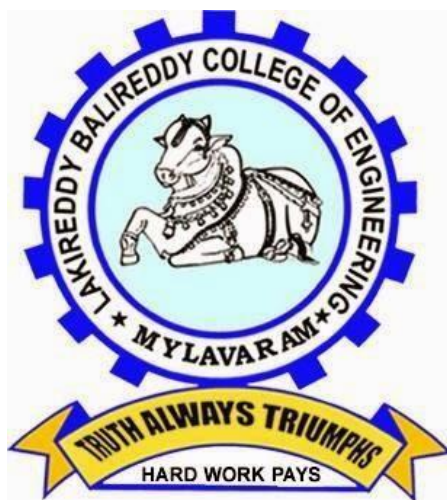
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APRIL - 2024**

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**CERTIFICATE**

This is to certify that the Project Report entitled “**EXPERIMENTAL INVESTIGATION ON MECHANICAL AND WEAR CHARACTERISTICS OF AZ91 HYBRID COMPOSITES PRODUCED THROUGH FRICTION STIR PROCESSING**” that is being submitted for the partial fulfillment of degree on **Bachelor of Technology in Mechanical Engineering to JNTUK, Kakinada** is a bonafide work done by

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## **DECLARATION**

We hereby declare that the work presented in this dissertation report titled “**EXPERIMENTAL INVESTIGATION ON MECHANICAL AND WEAR CHARACTERISTICS OF AZ91 HYBRID COMPOSITES PRODUCED THROUGH FRICTION STIR PROCESSING**” is submitted towards completion of main project in B.Tech (Mechanical Engineering) at Lakireddy Bali Reddy College of Engineering (Autonomous), Mylavaram. It is an authentic record of my original work pursued under the supervision of **Mr. A. Dhanunjay Kumar**, Professor, Department of Mechanical Engineering, LBRCE.

We have not submitted the matter embodied in this dissertation for the award of any other degree.

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## Abstract

The study investigates the application of friction stir processing (FSP), an innovative method derived from friction stir welding, to enhance the properties of AZ91 magnesium alloy composites by incorporating Silicon Carbide (SiC), Aluminium Oxide (Al<sub>2</sub>O<sub>3</sub>), and Graphene (Gn) nanoparticles. Despite the widespread use of FSW and FSP in solid-state welding for magnesium and its alloys, a knowledge gap exists regarding the correlation between FSP parameters and the resulting material properties. This research addresses this gap by examining various FSP parameters, including tool rotational speeds (560, 900, 1400 rpm) and tool transverse speeds (25, 50, 63 mm/min), and their impact on the properties of Magnesium AZ91 alloy composites with SiC, Al<sub>2</sub>O<sub>3</sub>, and Gn nanoparticles. Magnesium alloys are highly favored in diverse industries due to their lightweight nature and exceptional strength. However, traditional processing methods encounter challenges when working with Mg alloys. FSP, on the other hand, offers remarkable efficiency in this regard. Microstructural analysis reveals that grain refinement increases with the addition of Gn nanoparticles. Mechanical tests, including tensile and hardness tests, were conducted on the AZ91 hybrid composite samples. Notably, composition consisting of SiC (66.6% vol.), Al<sub>2</sub>O<sub>3</sub> (25% vol.), and Gn (8.33% vol.), processed at 1400 rpm and 63 mm/min, exhibited superior properties. Wear tests further demonstrated that this composition had somewhat lower wear rate compared to the base material, indicating reduction of almost 58%. Additionally, corrosion characteristics were evaluated through immersion tests, with this composition exhibiting optimal values. The findings underscore the potential of FSP in tailoring the properties of magnesium alloy composites for various industrial applications.



# Chapter 1

# 1. INTRODUCTION

## 1.1 Introduction to Composite Materials

A system of fabric made up of a mixture of two or more micro elements that are insoluble in one another and have different forms or compositions can be referred to as a composite material. These materials are made by combining two or more dissimilar materials such that they work together mechanically. These materials have different properties from their ingredients. These materials may consist of an extremely soft phase encased in a tough phase, or vice versa.

### 1.1.1 Advantages of Composite Materials:

- Low density, Corrosion and wear resistance, High strength, High stiffness, Thermal insulation and conductivity, long fatigue life,

### 1.1.2. Limitations of Composite Materials:

- Design and optimization
- Cost Effectiveness
- Maintenance and Durability

## 1.2 Magnesium AZ91 Alloy

Magnesium (Mg) is the eighth most prevalent element in the planet, with 2.7% of it found in the underside of the earth and 0.13% in the ocean. Magnesium alloys have low densities, which make them useful as structural materials to lighten objects. The low density, high strength to weight ratio, good casting ability, and low melting temperature of magnesium alloys provide them benefits over other materials. One such alloy series is magnesium AZ91 alloy, which combines zinc and aluminum, the two main alloying components, with 1% of zinc by weight and 9% of aluminum by weight. AZ91 exhibits superior mechanical properties, including high tensile strength and impressive stiffness, while retaining its lightweight nature, making it particularly desirable for aerospace, automotive, and electronics sectors. Its excellent castability further enhances its versatility, allowing for intricate designs and complex shapes to be manufactured efficiently. Moreover, AZ91 demonstrates good thermal conductivity, aiding in heat dissipation in electronic devices and facilitating casting processes.

**Table 1.1: Nomenclature of MagnesiumAZ91**

S.NO	PROPERTY	VALUE
1	Atomic Number	12
2	Atomic Weight	24.32
3	Physical State	338C

### 1.2.1 Properties of Magnesium AZ91

- **Low density:** With a density of 1.74 g/cm<sup>3</sup>, magnesium alloys are the lightweight engineering alloys; in comparison, steel (7.8 g/cm<sup>3</sup>) and aluminum (2.7 g/cm<sup>3</sup>) are much heavier.
- **High strength-to-weight ratio:** Magnesium alloys have excellent strength-to-weight ratios, making them up to 70% lighter than stainless steel.
- **Machinability:** Because of their exceptional machinability, magnesium alloys tend to be cast, forged, and extruded with simplicity.
- **Corrosion resistance:** Magnesium alloys have good to excellent corrosion resistance.
- **Damping capacity:** Magnesium alloys have the highest known damping capacity of any structural metal.

- **Cost:** Magnesium alloys are low in cost.

### **1.2.2 Advantages of Magnesium AZ91**

As a result of their relative strength, superb machinability, outstanding corrosion resistance, and remarkable damping capability, magnesium AZ91 alloys have emerged as the most popular and economical modern magnesium alloys.

### **1.2.3 Dis-Advantages of Magnesium AZ91**

The AZ91 magnesium alloy exhibits certain drawbacks, including low wear resistance, reduced cold workability, toughness, and limited strength and creep resistance at high temperatures.

## **1.3 Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>) Nanoparticles**

Aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) nanoparticles are a type of nanoparticles made from metal oxide with a wide range of biomedical uses because of their remarkable physicochemical and structural characteristics, including resistance to mechanical stresses, chemicals, and wear, as well as their advantageous optical qualities and large porous surface area.

**1.3.1 Properties:** High hardness, electric insulation, surface modification, biocompatibility, thermal stability, and extensive surface area.

## **1.4 Silicon Carbide (SiC) Nanoparticles**

High thermal conductivity, high stability, high purity, good wear resistance, and a relatively low thermal expansion coefficient are some of the properties displayed by silicon carbide (SiC) nanoparticles. Furthermore, resistant to oxidation at high temperatures are these particles.

**1.4.1 Properties:** Properties that stem from their small size and high surface area-to-volume ratio. These nanoparticles exhibit high hardness, chemical inertness, and excellent thermal stability, making them ideal for applications requiring abrasion resistance, chemical resistance, and high-temperature performance.

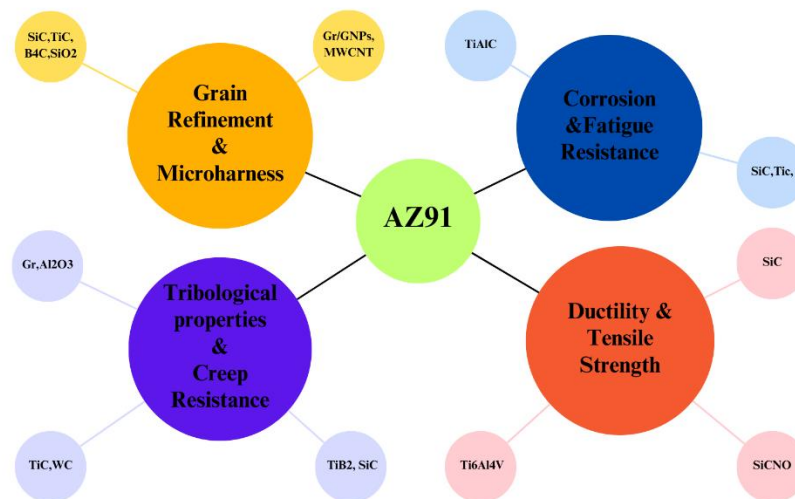
## **1.5 Graphene (Gn) Nanoparticles**

An allotrope of carbon called graphene is made up of a single layer of atoms organized in a nanostructure with a hexagonal lattice. The term "graphite" and the suffix -ene are the sources of the word, which represents the abundance of double bonds seen in the graphite allotrope of carbon.

**1.5.1 Properties:** It is not only the thinnest but also one of the strongest materials, it conducts heat better than all other materials and it is an excellent conductor of electricity.

## **1.6 Reinforcement for AZ91 Alloy**

However, it also possesses several disadvantages. One significant drawback is its susceptibility to corrosion, particularly in environments with high humidity or exposure to saltwater, which can compromise its structural integrity over time. AZ91 also exhibits relatively poor mechanical properties at elevated temperatures, limiting its applicability in high-temperature environments. Additionally, its low modulus of elasticity and poor wear resistance compared to other structural materials can pose challenges in certain engineering applications. To overcome these disadvantages composites can be reinforced with nano ceramic particles like Silicon Carbide (SiC), Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>) and Graphene (Gn).



**Figure 1.1:** Several reinforcing materials were employed in studies to enhance AZ91's characteristics.

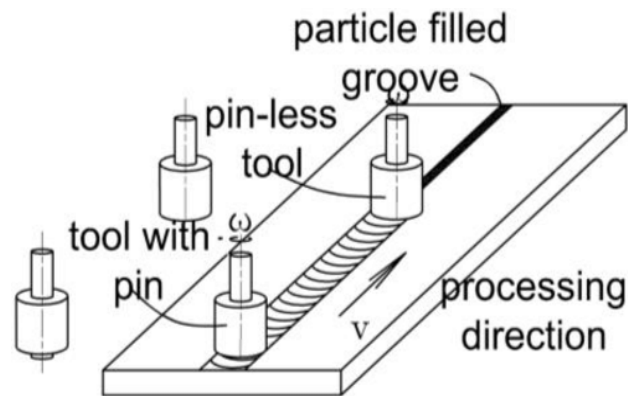
## 1.7 Nano Ceramic Powder Compositions:

The main constituents of nano ceramic particles are metal oxides, carbides, phosphates, and carbonates, which include calcium, titanium, silicon, and other metalloids. Because of their many advantageous qualities, including their excellent heat resistance and chemical inertness, they have many different kinds of uses. A few studies looked at the use of AZ91 in conjunction with the various reinforcing materials shown in figure 1.1. This study is focused on different compositions of Silicon carbide, Aluminium oxide and Graphene. Silicon carbide powder is utilized in the production of metal matrix composites, which are used in various industries for their superior mechanical and thermal properties. It is used in the production of lightweight, high-strength materials for aerospace and automotive applications. A variety of applications have been made possible by SiC's attributes, which include its hardness, resistance to abrasion and corrosion, high force-to-weight ratio, low thermal expansion, high thermal conductivity, and—above all—its ability to maintain elastic resistance at temperatures as high as 1650 °C. Aluminium oxide nano powder is also referred to as alumina nanoparticles. Aluminum and oxygen atoms combine to form aluminum oxide nanoparticles. It belongs to the ceramic nanoparticle class. It has low friction and outstanding durability, making it a good option for additive for any number of composites, as it offers an enhanced wear resistance. Because of its amazing qualities, graphene powder is regarded as a "wonder material" throughout the world. Graphene nano powder is another name for it because of its nano-structural characteristics. It is among the best and strongest materials for conducting heat and electricity. Globally, a wide range of sectors and research are using this incredibly adaptable material more and more. It's among the strongest materials found to date. In terms of tensile strength, it surpasses both diamond and industrial-grade steel. The material is not only extraordinarily strong but also remarkably light.

## 1.8 Friction Stir Processing for fabrication of Composite

Despite the many benefits of magnesium matrix composites reinforced with nano ceramic particles, dispersing the particles evenly into the magnesium matrix proved difficult using traditional liquid or powder metallurgy processes due to the tendency of the substantial surface area of the ceramic nanoparticles to accumulate and the generally poor wetting between the ceramic particles and the magnesium matrix. Research indicates that prolonged high-energy ball milling helps the nanoparticles in the aluminum alloy matrix to disperse uniformly. However, since fine magnesium powders easily oxidize, burn, and explode in the atmosphere, high-energy ball milling needs to be done in a vacuum or under inert gas protection. While there are numerous methods for assisting in the dispersion of nano

ceramic particles in the melt magnesium during the liquid fabrication process, such as mechanical stirring in a semisolid state and ultrasonic wave melt treatment, these particles tend to be pushed rather than captured by the solidification front and ultimately gather at the grain boundaries in the solidified composites.



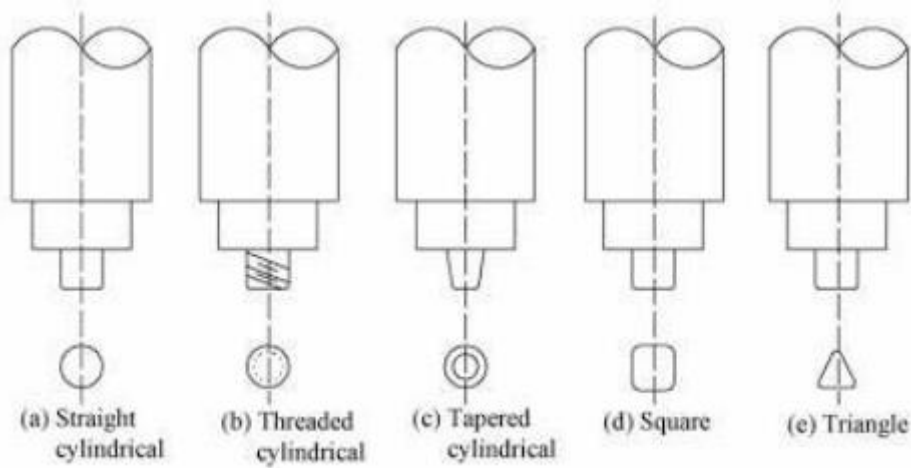
**Figure 1.2:** Friction Stir Processing for a composite filled in groove

In recent years, there has been significant advancement in novel techniques such as FSP (Friction Stir Processing). These processes have emerged as promising methods for enhancing material properties, offering distinct advantages including reduced welding defects, minimal distortion and residual stresses, limited structural alterations, and the generation of a fine-grained structure. Developed by TWI in 1991, FSW was initially introduced to enhance the structural integrity of welded metals, with Friction Stir Processing (FSP) later stemming from this innovation.

FSP is a prominent example of a severe plastic deformation (SPD) method; it is especially useful for dynamic recrystallization, which refines the texture of lightweight alloys such as magnesium and aluminum. A revolving tool with a shoulder and a pin is used in FSP. The shoulder of the pin makes contact with the metal surface as it passes through the workpiece, causing friction that generates heat. Figure 1.2 illustrates how the confined heat refines the microstructure within the stir zone by softening the surrounding material and enabling the pin to stir it.

### 1.8.1 Types of FSP Tools

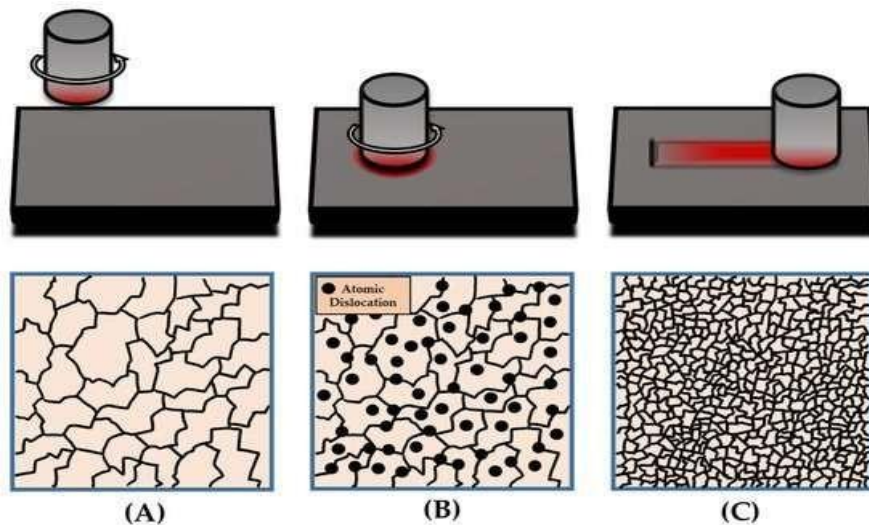
Friction Stir Processing (FSP) employs various tool designs to accomplish material processing objectives these are shown in figure 3. These designs include square cylindrical tools, which feature a square cross-section and are suited for applications requiring square-shaped features. Triangle cylindrical tools, with their triangular cross-sections, offer distinct advantages such as promoting specific flow patterns or improving heat dissipation. Straight cylindrical tools, lacking specific geometric features, are versatile and commonly used across different FSP applications. Threaded cylindrical tools incorporate threads along their length, enhancing material agitation and mixing during processing, particularly beneficial for certain material compositions. Tapered cylindrical tools, characterized by a tapered profile, enable gradual material displacement and provide control over material flow and heat distribution. Each tool design serves specific needs, selected based on material properties and desired processing outcomes in FSP operations.



**Figure 1.3:** Different types of Friction Stir Processing tools

### 1.8.2 Recrystallization Process in FSP

The distinct advantages of FSW and FSP lie in their ability to join materials in a solid-state fashion, minimizing welding defects and structural distortions commonly associated with traditional welding methods. Moreover, the controlled heating and stirring action result in desirable microstructural changes, enhancing the mechanical properties of the materials involved. As such, these techniques represent significant advancements in the realm of material processing, offering versatile solutions for various industrial applications.



**Figure 1.4:** An illustration of the dynamic recrystallization process that shows the following steps: (A) the starting material in its basic form; (B) the first plunge into the working material, which causes significant atomic dislocations (shown by circles); (C) the atomic structure reorientation; and (D) the finalized grain structure.

Variables often have a significant impact on the mechanical, microstructural, wear, corrosion, and friction properties of different materials, as was previously mentioned. Still, the grain's refining customizes these qualities. Dynamic recrystallization (DRX), a microstructure-altering process, is responsible for this. It should be noted that, depending on the kind of material, DRX can be divided into four different sub-mechanisms: discontinuous DRX (DDRX), geometric dynamic recrystallization (GDRX), twinning induced dynamic recrystallization (TDRX), and continuous DRX (CDRX). The working piece needs to undergo strain-induced deformation in order to cause DRX. New grains can

form because of the dislocation of atoms within the working piece, which is dependent on the strain applied to the material. As these grains become re-nucleated, finer grain sizes are created, thus enhancing the microstructural properties of the material. The general concept of DRX via FSP processes is shown in figure 4.

Friction stir processing (FSP) has garnered attention from researchers exploring its effects on magnesium alloys, yielding findings of grain refinement and enhanced hardness within the treated region. Moreover, FSP has emerged as a novel method for producing surface metal matrix composites on magnesium alloys. This technique proves promising for sectors like automotive and aerospace industries, where advancements in materials are crucial for bolstering resistance against wear, creep, and fatigue. Successful applications of FSP encompass various materials such as AA 2519, AA 5083, and AA 7075 aluminium alloys, AZ61 magnesium alloy, nickel-aluminium bronze, and 304L stainless steel.

FSP offers notable advantages in scenarios requiring the amalgamation of two materials. As stated by Ma, FSP represents a concise, solid-state processing method facilitating microstructural refinement, densification, and homogeneity in a single step. Unlike traditional methods involving melting and moulding, FSP enables material modification without drastic alteration. For instance, it allows for the transformation of metal sheets, eliminating the need for melting and moulding for shaping. The microstructure and mechanical properties of the processed zone can be finely tuned through optimization of tool design, FSP parameters, and active cooling/heating. With appropriate tool modifications, a single metal sheet can adapt to diverse requirements. Demonstrably, FSP enhances the malleability of metallic alloys; for instance, an alloy subjected to FSP modification can exhibit a bending capacity of 30 degrees compared to its previous limit of seven degrees.

## 1.9 Applications of Mg Alloys and FSP

The diverse applications of magnesium-based alloys and the innovative FSP techniques across various industries. These technologies are prized for their ability to provide high strength-to-weight ratios, making them invaluable in numerous sectors.



**Figure 1.5:** The applications for FSP technology include construction of ships and marine, renewable energy, railway, the aviation, aerospace, and defense sectors.

In the construction industry, magnesium alloys find utility in critical infrastructure such as pipelines, bridges, and reactors for power plants, alongside structural frameworks. Likewise, railway applications encompass container bodies, trams, wagons, and underground carriages, where the lightweight yet durable nature of magnesium alloys enhances efficiency and longevity.

In land transportation, magnesium-based materials feature prominently in diverse components including track bodies, engine chassis, wheel rims, and fuel tankers, offering advantages in terms of weight reduction and corrosion resistance. Similarly, shipbuilding and marine industries leverage magnesium alloys for marine structures, decks, masts, and hulls, benefiting from their strength and corrosion resilience in harsh maritime environments.

The aerospace sector capitalizes on magnesium alloys for their unique properties, employing them in cryogenic tanks, wings, fuselages, and fuel tanks for space vehicles and aircraft. This application underscores the critical role of magnesium-based materials in advancing aerospace technology.

### **1.10 Objective of Research**

- Investigate the application of Friction Stir Processing (FSP) on Magnesium AZ91 alloy composites by incorporating Silicon Carbide (SiC), Graphene (Gn) nanoparticles and Aluminium Oxide ( $\text{Al}_2\text{O}_3$ )
- Examine the influence of FSP processing parameters on the surface properties of Mg alloy composites, particularly focusing on the correlation between these parameters and resultant properties.
- Evaluate the corrosion resistance behavior of Magnesium AZ91 alloy composites produced through FSP, considering the addition of SiC, Gn and  $\text{Al}_2\text{O}_3$  nanoparticles.
- Assess the mechanical properties, including wear properties and hardness, of the FSP-treated AZ91 alloy composites and compare them with the properties of the base metal.
- Contribute to bridging the existing gap in knowledge regarding the correlation between FSP parameters and material properties of Mg AZ91 alloy composites with SiC,  $\text{Al}_2\text{O}_3$  and Gn nanoparticles.



# Chapter 2

## 2. LITERATURE REVIEW

### 2.1 Literature Survey

1. **Journal Title:** Mishra, R. S., Ma, Z. Y., & Charit, I.'s Seminal: A foundation laid for understanding FSP's capabilities and its impact on surface composite fabrication.
  - Friction-stir processing (FSP), an extension of friction-stir welding (FSW) pioneered by The Welding Institute (TWI) of the UK, has garnered attention for its ability to induce localized microstructural changes.
  - Mishra et al. [1] introduced FSP as a novel technique capable of refining grains and preparing nano-crystalline structures in aluminum (Al), magnesium (Mg), and copper (Cu)-based components. Recent studies highlight FSP's potential for fabricating surface composites with enhanced properties. By refining grains and introducing nanoparticles, FSP enables the formation of metal matrix composites (MMCs), leading to improved mechanical, tribological, and corrosion resistance properties.
  - This technique offers versatility in processing various alloys, paving the way for applications in aerospace, automotive, and other industries.
2. **Journal Title:** Heidarzadeh, A., Mironov, Kaibyshev, R. Çam, G. Simar, Gerlich, A. Withers. Friction stir welding/processing of metals and alloys: A comprehensive review on microstructural evolution.
  - Heidarzadeh et al. [2] emphasized that Friction-stir processing (FSP) offers a unique opportunity to refine and homogenize microstructures locally while facilitating the incorporation of nanoparticles to form metal matrix composites (MMCs). These nanoparticles activate particle-stimulated nucleation (PSN) recrystallization, followed by pronounced Zener-pinning, resulting in an ultrafine-grained structure.
  - Their comprehensive review highlights the potential of FSP in microstructural evolution. The process enables tailored material properties through precise control over grain size and distribution, essential for enhancing mechanical performance. The activation of PSN and subsequent Zener-pinning mechanisms underscores FSP's capability to achieve superior structural refinement, crucial for advancing the development of high-performance materials.
  - This review serves as a valuable resource for understanding the intricacies of microstructural evolution during FSP and its implications for the fabrication of advanced metal alloys and composites.
3. **Journal Title:** Soorya Prakash K, Balasundar P, Nagaraja S, et al. Mechanical and wear behaviour of Mg–SiC–Gn hybrid composites.
  - K. Soorya Prakash et al. [3] pioneered the development of magnesium–SiC–Gn hybrid composites via powder metallurgy, resulting in heightened hardness compared to the base material due to the robust SiC presence.
  - However, a slight decline in hardness occurred compared to Mg–SiC composites due to the addition of softer Gn particles. Tribological properties were evaluated via pin-on-disc wear tests under dry sliding conditions, revealing significantly improved wear resistance for the composites compared to pure magnesium.
  - Soorya Prakash et al.'s study sheds light on the mechanical and wear behavior of these hybrid composites, offering valuable insights into their potential applications.

4. **Journal Title:** Hsu, C. J., Chang, C. C. Y., Kao, P. W., & Ho, N. J. (2006). Al-Al<sub>3</sub>Ti nano composites produced in situ by friction stir processing.
  - Hsu et al. [4] employed friction-stir processing (FSP) to create Al/Al<sub>3</sub>Ti nano-composites, enhancing the Young's modulus and tensile strength of aluminum alloys.
  - The study revealed that increasing the volume percentage of Al<sub>3</sub>Ti resulted in a significant elevation in Young's modulus, indicating the potential of ceramic particles to enhance wear resistance in metal matrix composites.
  - This research underscores the effectiveness of FSP in producing nano-composites with improved mechanical properties, offering insights into the optimization of composite materials for various applications.
5. **Journal Title:** Abbasi M, Bagheri B, Dadaei M, Omidvar H R, Rezaei M: The effect of FSP on mechanical, tribological, and corrosion behavior of composite layer developed on magnesium AZ91 alloy surface.
  - Abbasi et al. [5] investigated the impact of SiC and Al<sub>2</sub>O<sub>3</sub> particles on mechanical, tribological, and corrosion properties of surface composites on AZ91 magnesium alloy via Friction-stir processing (FSP).
  - They found that specimens containing SiC particles exhibited higher strength, ductility, and superior corrosion resistance compared to those with Al<sub>2</sub>O<sub>3</sub> particles. This suggests the potential of FSP in enhancing the performance of magnesium alloys through the incorporation of suitable reinforcing particles.
  - The study underscores the importance of particle selection in optimizing the properties of surface composites, providing valuable insights for the development of advanced materials with improved mechanical and corrosion characteristics.
6. **Journal Title:** A. Atrens, Z. Shi, S.U. Mehreen, S. Johnston, G.L. Song, X. Chen, F. Pan, J. Magnes: Review of Mg alloy corrosion rates
  - In recent years, there has been a growing interest in the use of nanoparticles such as Al<sub>2</sub>O<sub>3</sub>, SiC, and carbonaceous materials as reinforcing agents in metal matrix composites (MMCs). Andrej Atrens et al. [6] highlight this trend, noting the emerging role of nanoparticles in enhancing the physical, mechanical, and corrosion properties of MMCs. These nanoparticles offer a promising avenue for improving the performance of materials across various applications.
  - The research underscores the potential of incorporating nanoparticles into MMCs to achieve desired material characteristics, reflecting ongoing efforts to innovate in the field of composite materials. As the understanding of nanoparticle-reinforced MMCs continues to evolve, further exploration of their synthesis, processing, and performance is essential to unlock their full potential in advancing material science and engineering.
7. **Journal Title:** Faraji, G., & Asadi, P. Characterization of AZ91/alumina nanocomposite produced by FSP.
  - Faraji and Asadi (2011) explored AZ91/Al<sub>2</sub>O<sub>3</sub> nano-composite fabrication via friction stir processing (FSP), revealing insights into grain size, hardness, and wear resistance. They found that the distribution pattern of nano-alumina particles, rather than heat production, significantly influenced final grain size and hardness.
  - Additionally, FSP of AZ91 magnesium alloy with nano-alumina particles notably enhanced wear resistance, transitioning the wear mechanism from intensive to moderate. Moreover,

optimizing particle distribution at high W/V ratios led to reduced grain size and increased hardness.

- This study underscores the importance of nanoparticle distribution in FSP-produced composites and highlights its potential for enhancing mechanical properties, particularly wear resistance, in magnesium alloys.

8. **Journal Title:** O. Guler, Y. Say, B. Dikici, The effect of graphene nanosheet (GNS) weight percentage on mechanical and corrosion properties of AZ61 and AZ91 based magnesium matrix composites.

- Guler et al. [8] explored the impact of Graphene Nanosheets (GNS) on AZ61 and AZ91 magnesium alloys, revealing enhanced compressive strength with increased GNS content. However, achieving optimal corrosion resistance necessitates precise control over the GNS ratio.
- The study underscores the potential of GNS as a reinforcement material for magnesium matrix composites, offering improvements in mechanical properties. By examining the mechanical and corrosion characteristics of GNS-incorporated alloys, the research contributes valuable insights into the design and optimization of composite materials. These findings have implications for various applications where magnesium alloys are utilized, such as aerospace, automotive, and biomedical industries, highlighting the importance of tailoring GNS content to achieve the desired balance between mechanical performance and corrosion resistance.

9. **Journal Title:** Chen, H., & Alpas, A. T. (2000). Sliding wear map for the magnesium alloy Mg-9Al-0.9 Zn (AZ91).

- Chen and Alpas [9] explored the wear behavior of AZ91 magnesium alloy, revealing two distinct wear regimes: mild and severe. Their study, drawing from previous investigations on pure magnesium wear by Hiratsuka et al. [10], observed an oxidation wear mechanism predominant in magnesium when subjected to dry sliding in air.
- This research contributes to understanding the complex wear mechanisms inherent in magnesium alloys, providing valuable insights for designing materials with improved wear resistance. The findings underscore the importance of considering environmental factors such as air exposure in evaluating wear performance.
- This study serves as a foundational framework for further research aimed at enhancing the wear properties of magnesium-based materials for various industrial applications.

10. **Journal Title:** Heidarzadeh, A., Mironov, S., Kaibyshev, R., Çam, G., Simar, A., Gerlich, A. Withers, P. J. (2021). Friction stir welding/processing of metals and alloys: A comprehensive review on microstructural evolution.

- Haramritpal Sidhu et al. [11] highlighted the significant impact of microstructural features such as second phase distribution, grain size, and grain texture on the corrosion behavior of Mg alloys. Corrosion resistance was found to improve with decreased grain size, attributed to the refinement of  $\alpha$  and  $\beta$  phases leading to increased  $\beta$  phase precipitation. Additionally, the study elucidated that the enhanced wear resistance of Friction Stir Processed AZ31 magnesium alloy samples compared to the base material was due to intensified grain refinement.
- This finding aligns with Heidarzadeh et al.'s [12] comprehensive review, which discusses the microstructural evolution in friction stir welding/processing of metals and alloys, providing valuable insights into the mechanisms governing grain refinement and its implications for material properties.

11. **Journal Title:** Molla Ramezani, N., Davoodi, B., Aberoumand, M., Rezaee Hajideh, M. (2019). Assessment of tool wear and mechanical properties of Al 7075 nanocomposite in friction stir processing (FSP).
- Molla Ramezani et al. [13] examined tool wear and mechanical properties in Al 7075 nanocomposite using friction stir processing (FSP). They found that increasing rotational speed reduced tool wear, with a 52.9% impact on tool wear attributed to rotational speed. Higher rotational speed led to greater strain rates and material elongation, resulting in smaller grain sizes and consequently increased material strength and hardness.
  - This study highlights the intricate relationship between process parameters, microstructure, and mechanical properties in FSP, offering insights into optimizing processing conditions for enhanced performance in metal matrix composites.
12. **Journal Title:** Y. Mazaheri, M.M. Jalilvand, A. Heidarpour, A.R. Jahani, Tribological behavior of AZ31/ZrO<sub>2</sub> surface nanocomposites developed by friction stir processing.
- Mazaheri et al. [13] conducted a study on Friction Stir Welding (FSW) of AZ31 and AZ91 alloys, employing an H13 Tool Steel Flat shoulder with a hybrid tool pin. They varied rotation speed (700-1000 rpm), welding speed (30-50 mm/min), and shoulder diameter (15-21 mm). Their findings revealed that higher rotation speeds and shoulder diameters led to larger grain sizes in the welded materials.
  - This research contributes to understanding the relationship between process parameters and microstructural evolution during FSW, which is crucial for optimizing weld quality and mechanical properties. Additionally, Mazaheri et al. [13] demonstrate the potential for developing surface nanocomposites, as indicated in their previous work on tribological behavior in AZ31/ZrO<sub>2</sub> nanocomposites processed by friction stir processing
13. **Journal Title:** Sudhakar, M., Stanley, A. I., Suri, K. S. V., Sai, B. S., Anil, D., Rao, Y. V., Basha, S., Vikas, C., & Charan. Friction Stir Processing Of AZ91 Magnesium Alloy and Effect of Process Parameters.
- Sudhakar et al. [14] investigated the effects of Friction Stir Processing (FSP) on AZ91 magnesium alloy, varying tool rotational speeds (760 and 1130 rpm) and traverse speeds (11, 22, 48 mm/min). Their study revealed that FSP significantly improved the microstructure and hardness of the alloy. The optimized process parameters led to enhanced grain refinement and increased hardness, indicating improved mechanical properties.
  - This research contributes valuable insights into the influence of process parameters on the microstructural evolution and mechanical behavior of AZ91 magnesium alloy during FSP. Sudhakar et al.'s findings provide a basis for further optimization of FSP parameters to tailor the properties of magnesium alloys for specific applications.
14. **Journal Title:** Asadi, P., Besharati Givi, M. K., Parvin, N., Araei, A., Taherishargh, M., & Tutunchilar. On the role of cooling and tool rotational direction on microstructure and mechanical properties of friction stir processed AZ91.
- Faraji and Asadi [15] demonstrated that Friction Stir Processing (FSP) effectively alters microstructures, with optimal outcomes achieved using a square tool, 900 rpm rotational speed, and 40 mm/min traverse speed. These parameters notably influence grain size and particle distribution, crucial factors determining material properties.
  - The study contributes valuable insights into the optimization of FSP parameters for enhanced microstructural refinement and mechanical performance. Additionally, Asadi et al. [15] investigated the effects of cooling and tool rotational direction on the microstructure and

mechanical properties of friction stir processed AZ91, further enriching our understanding of the FSP process and its outcomes.

15. **Journal Title:** Iwaszko J and Kud1a K. Microstructure, hardness, and wear resistance of AZ91 magnesium alloy produced by friction stir processing with air-cooling.

- Iwaszko and Kud1a [16] investigated the enhancement of AZ91 magnesium alloy surface using Friction Stir Processing (FSP). Employing a jet cooling nozzle during single-pass FSP resulted in fine-grained microstructures, particularly notable in nozzle-cooled samples compared to natural cooling. Both cooled samples exhibited significantly higher hardness than the base material, with FSPed samples displaying slightly better wear resistance.
- The study underscores the effectiveness of jet cooling during FSP, as evidenced by improved microstructural refinement and mechanical properties. This research sheds light on the potential of cooling strategies to optimize FSP outcomes and enhance the performance of magnesium alloys in various applications.

16. **Journal Title:** Vivek Patel, Wenya Li b, Joel Andersson a,” Enhancing grain refinement and corrosion behavior in AZ31B magnesium alloy via stationary shoulder friction stir processing”

- Vivek Patel et al. [17] explored the fabrication of surface matrix composites (MMCs) using stainless powder on AZ61 magnesium alloy through friction-stir processing (FSP), leading to a notable increase of up to 12.1% in mechanical properties and improved corrosion resistance. Sithole et al. [6] detailed the influential factors in FSP, highlighting nine combinations and four essential processes including rotational speed, travel speed, depth, and tilt angle.
- Their study, conducted on pure magnesium alloy, demonstrated changes in material properties with parameters set at 1600 rpm, 63 mm/min, 0.1 depth, and 2° tilt angle. This research collectively contributes to understanding FSP's role in enhancing grain refinement and corrosion resistance in magnesium alloys, providing valuable insights for future process optimization and material development.

17. **Journal Title:** Patel, P., Patel, S., & Shah, H. (n.d.). Design and Experimental study of Friction stir welding of AA6061-T6 Alloy for optimization of welding parameters by using Lathe Machine.

- Patel et al. investigated the butt joining of aluminum plates via friction stir welding (FSW) using a light-duty lathe machine, eliminating the need for additional setup besides the tool and fixture. Their study, focusing on two key process parameters - tool speed and transverse speed - utilized Al alloy 6061 for the base plate and EN8 for the tool.
- The research aimed at optimizing welding parameters for AA6061-T6 alloy. By employing a simplified experimental setup, they aimed to streamline the FSW process and enhance efficiency.
- This study contributes valuable insights into parameter optimization for FSW, particularly in the context of using readily available equipment like a lathe machine, potentially facilitating broader adoption of this welding technique in manufacturing settings.

18. **Journal Title:** B.Sharma, Ujjwal Prakash, B.V. Manoj Kumar: Surface composites by friction stir processing: A review

- B. Sharma [19] investigated vertical milling machine-based friction stir welding of thin aluminum sheets, finding that the 1000 RPM setting produces the highest-quality welds. Suggested areas for further research include the development of robust tools and fixtures, preheating to enhance weld quality, and addressing issues such as tool vibration and workpiece fixture materials to improve future weld quality.

- Additionally, Sharma, along with Ujjwal Prakash and B.V. Manoj Kumar, conducted a comprehensive review on surface composites fabricated through friction stir processing (FSP). This review likely covers various aspects of FSP, including its applications, process parameters, and resulting material properties. Such investigations contribute to a deeper understanding of FSP's capabilities and potential for enhancing the mechanical and structural characteristics of materials.

19. **Journal Title:** Bagheri, B., Abbasi, M., Abdollahzadeh, A., & Mirsalehi. Effect of second-phase particle size and presence of vibration on AZ91/SiC surface composite layer produced by FSP.

- Bagheri et al. [21] explored the impact of submerging Friction Stir Processing (FSP) on grain size in Al-6061-T6 alloy compared to FSP in air. Submersion resulted in enhanced grain refinement due to accelerated cooling. The researchers employed a boundary migration model, integrating measured thermal profiles, to predict grain size.
- This study sheds light on the influence of processing conditions on microstructural evolution during FSP. Additionally, Bagheri et al. [21] delved into the effects of second-phase particle size and the presence of vibration on AZ91/SiC surface composite layers produced by FSP [22], further contributing to understanding the factors influencing microstructure and mechanical properties in FSP-produced materials.

## 2.2 Research Gap

- Despite the widespread use of Friction Stir Processing (FSP) in solid-state welding for magnesium and its alloys, a significant research gap exists in understanding the co- relation between FSP processing parameters and the resultant surface properties of Magnesium alloy composites with Silicon Carbide (SiC), Aluminium Oxide ( $\text{Al}_2\text{O}_3$ ) and Graphene (Gn) nanoparticles.
- The current study focuses on AZ91 Mg alloy composites, aiming to enhance their properties through FSP. The lack of comprehensive research on the corrosion behavior, microstructures, and mechanical properties like wear resistance of AZ91 alloy composites produced through FSP, especially with the addition of SiC,  $\text{Al}_2\text{O}_3$  and Gn nanoparticles. Provide potential advancements in Magnesium alloy applications.

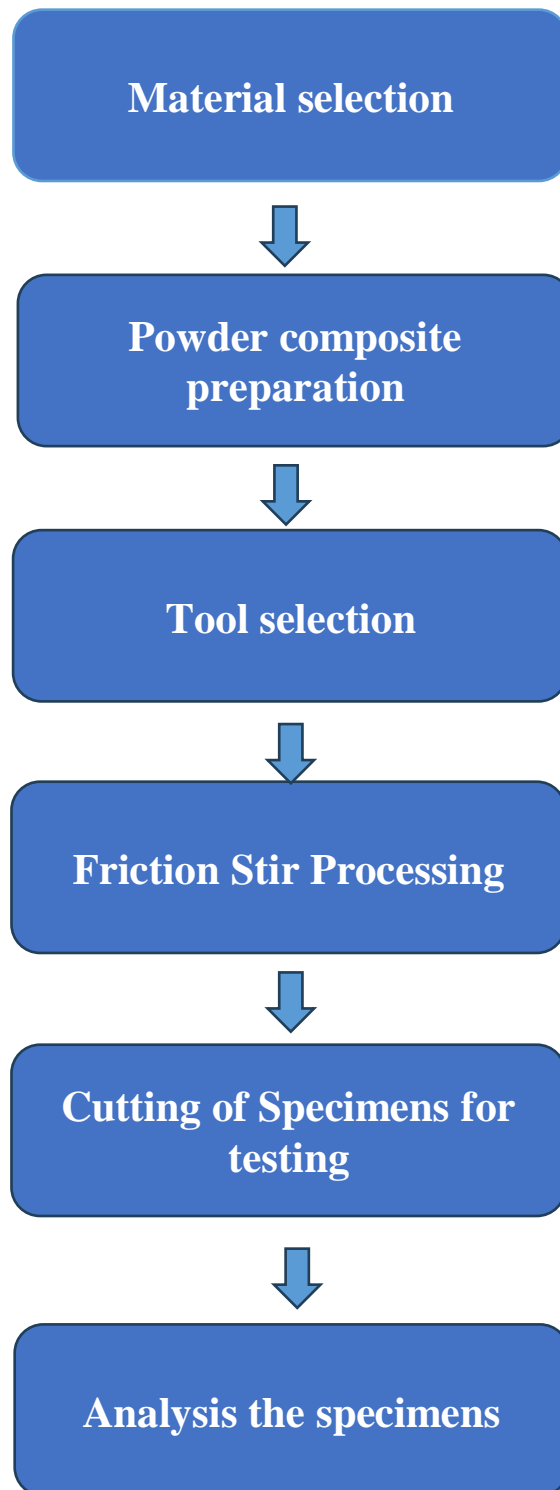
# Chapter 3



### **3. EXPERIMENTATION SETUP AND METHODOLOGY**

#### **3.1 Experimental Procedure**

The following flow chart represents different steps involved in the experimentation setup.

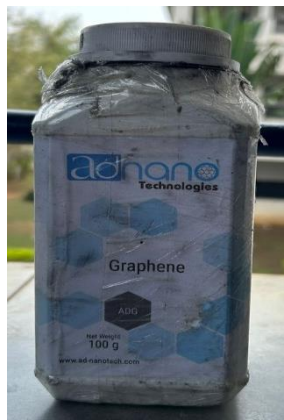


### 3.2 Selection of Material

Three plates of Magnesium AZ91 measuring  $100 \times 100 \times 8 \text{ mm}^3$  were utilized as shown in the fig 3.1. Three nano powders: Silicon Carbide, Aluminum Oxide, and Graphene are taken which are shown in fig 3.2.



**Figure 3.1:** Mg AZ91 Plates



**Figure 3.2:** Composite powders

### 3.3 Preparation of Powder Composite

Three nano powders: Silicon Carbide, Aluminum Oxide, and Graphene. combined in varying proportions as outlined in Table 3.1. Achieving a homogeneous mixture of the nano powder composite involved blending on a Lathe machine for one hour per composition as shown in figure 3.3.



**Figure 3.3 (a):** Composite material

**Figure 3.3 (b):** Blending of powder on lathe

**Table 3.1:** Weight ratios of Compositions

Composite Material	I	II	III
Silicon Carbide SiC	4	6	8
Aluminium Oxide Al <sub>2</sub> O <sub>3</sub>	1	2	3
Graphene Gn	1	1	1

### 3.4 Preparation of Grooves

The preparation of the grooves for place the composite powder on fsp Each plate underwent the creation of three grooves, measuring 2 mm x 2 mm x 100 mm, utilizing a shaper machine. These grooves facilitated the placement of the nano powder composite, ensuring its integration with the magnesium plate.



**Figure 3.4:** Shaper Machine and Work piece with grooves

### 3.5 Tool Selection

The tool shape determines the mixing of composite, processing speed and tool strength. The tool material determines the rate of friction heating, tool strength and working temperature.



**Figure 3.5:** FSP Tapered Cylindrical Tool



### 3.6 Friction Stir Processing on Vertical Milling

Friction Stir Processing (FSP) was conducted using a Vertical Milling machine equipped with a conical-shaped tool probe as shown in figure 3.6. Preceding the FSP, a flat-shaped tool was passed over the grooves of the plate containing the composite to prevent powder escape. Subsequently, a conical-shaped tool, as depicted in the figure, was employed to execute the FSP. Different tool parameters were applied for each composition, ranging from 560 to 1400 rpm and 25 to 63 mm/min, as detailed in Table 3.2. The tilt angle for the tool remained constant at 2°. Post-processing involved the removal of excess material generated during the FSP.

**Table 3.2:** Design of Experimentation

Sl. No.	Composite	Rotational Speed (RPM)	Transvers Speed (mm/min)	Angle
1	I	560	25	2°
2	I	900	50	2°
3	I	1400	63	2°
4	II	1400	63	2°
5	II	560	25	2°
6	II	900	50	2°
7	III	900	50	2°
8	III	1400	63	2°
9	III	560	25	2°



**Figure 3.6:** Set up of vertical milling machine



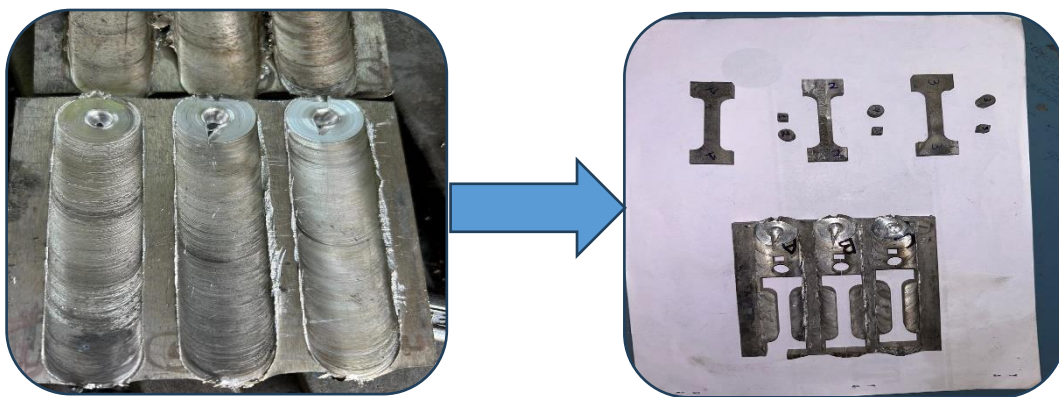
**Figure 3.7:** Process of friction stir process

### 3.7 Cutting of Specimens

To facilitate various tests on the processed composites, markings were made on the FSPed Plates. Utilizing the Wire electrical discharge machining (WEDM) method, different specimens were cut, as illustrated in figure 3.8. WEDM uses a metallic wire to cut or shape a work piece, often a conductive material, with a thin electrode wire that follows a precisely programmed path.



**Figure 3.8 (a):** Wire electrical discharge machining (WEDM)



**Figure 3.8 (b):** Wire cutting of specimens

### 3.8 Hardness Test

The Brinell hardness test measures the diameter of the impression left on the surface of the material being tested after the load is released. It involves forcing a hard steel ball indenter into the substance under test under predetermined load and time parameters using a calibrated equipment. The body of the hardness testing machine is made of cast iron. The internal working components are shielded from some dust and external elements by the enclosed design. Furthermore, shielding the primary screw is a rubber bellow. The weights and levers comprise the basic system. In order to determine the hardness value of the work piece, the weights under the hydraulic dash-pot time control are applied to the free end of the lever. This transfers the pressure to the plunger. Tightly clamping the workpiece during the test—something that is occasionally impossible to verify under normal circumstances—is made possible by a clamping device.



**Figure 3.9:** Brinell Hardness Testing Machine and Microscope

### 3.9 Tensile Test

A basic mechanical test used to determine how materials react under tension is called a tensile test. Key characteristics including yield strength, ultimate tensile strength, elongation, and modulus of elasticity are frequently ascertained using it. In a tensile test, a regulated tensile force or load is applied to a sample of the material, increasing it gradually until the sample cracks. Measurements are made during the test to track the applied force and the specimen's subsequent deformation, which is often elongation. Afterwards, a stress-strain curve that depicts the behavior of the material under tension is created using this data. While the strain is ascertained by measuring the change in length with respect to the specimen's initial length, the stress is computed by dividing the applied force by the specimen's original cross-sectional area. Tensile tests are essential in many fields, including as materials research, manufacturing, and construction, since they offer important information about the behavior and mechanical characteristics of materials under diverse loading scenarios.





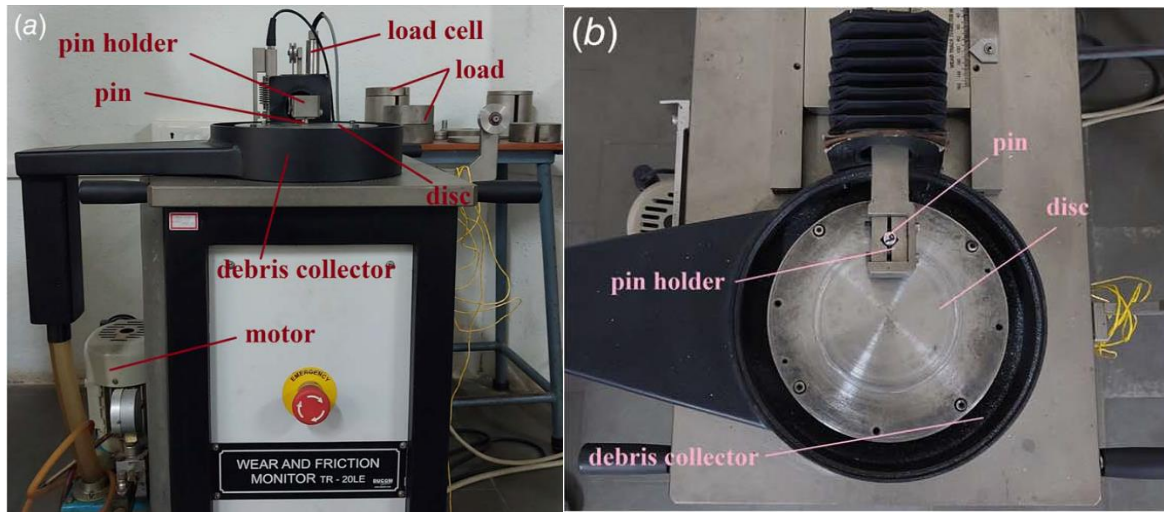
**Figure 3.10:** Tensile Testing Machine

### 3.10 Wear Test

Materials are often tested for friction and wear using tribometers, with one of the most common methods being the pin-on-disk test. This test is popular because of its simplicity and can mimic many real-world situations where materials rub against each other. It involves a pin pressing against a rotating disk, simulating friction between materials. Standardized testing procedures, like ASTM G99, ASTM G133, and ASTM F732, are followed during these tests to ensure consistency and accuracy. These tests help scientists and engineers understand how different materials perform under stress and choose the best ones for specific applications. The wear testing machine used in this study is DUCOM TR-20LE as shown in figure 3.11. The different parts of machine are as followed:

1. **Pin:** This is a small cylindrical or conical-shaped specimen that is attached to the wear testing machine. It represents one of the materials being tested for wear resistance. The pin is typically made from the material of interest, or it can be coated with a specific material for testing purposes.
2. **Disc:** The disc is the counterpart to the pin and serves as the stationary surface against which the pin rubs. Like the pin, the disc can be made from the material under investigation or coated with a different material. It is usually larger in size compared to the pin.
3. **Testing Machine:** This is the equipment used to conduct the wear test. It includes mechanisms to hold the pin and disc, apply a specific load or force, and control the relative motion between the pin and disc. The machine may also have sensors or instruments to measure parameters such as frictional force, wear rate, and temperature.
4. **Load System:** The load system applies a controlled force or pressure between the pin and disc during the test. This force is essential for simulating real-world conditions where materials experience stress due to contact and motion.
5. **Motion Control System:** This system regulates the movement of either the pin, the disc, or both. It determines the speed, direction, and type of motion (e.g., linear, rotational) during the wear test. Controlling the motion allows researchers to mimic specific operating conditions relevant to the application.
6. **Environmental Control:** Maintaining a consistent testing environment is crucial for obtaining reliable results. Temperature and humidity levels may be controlled to simulate different operating conditions, such as dry or lubricated environments.

7. **Data Acquisition System:** This system collects data throughout the test, including measurements of wear, friction, and other relevant parameters. It enables researchers to analyze the performance of materials and evaluate their wear resistance characteristics.



**Figure 3.11:**(a) Wear Testing Machine and (b) Workpiece attached to pin holder



# Chapter 4

## 4. RESULTS AND DISCUSSIONS

### 4.1 Microstructure

The goal of the current work is to improve the characteristics of AZ91 Mg alloy composites by means of FSP. The absence of comprehensive investigations on the microstructures, mechanical properties like tensile strength, hardness, wear resistance, and corrosion behavior of AZ91 alloy composites made by FSP, especially when SiC, Al<sub>2</sub>O<sub>3</sub>, and Gn nanoparticles are encompassed. Provide desirable enhancements in the applications of magnesium alloys. The microstructural analysis provided sheds light on the intricate correlation of FSP parameters, material compositions, and resulting mechanical properties. Optimizing FSP parameters and material compositions is crucial for achieving desired surface properties, microstructural characteristics, and mechanical properties in AZ91 alloy reinforced with SiC, Al<sub>2</sub>O<sub>3</sub>, and Gn. Dynamic recrystallization induced by high rotational and transverse speeds shows a significant role in refining grain size and enhancing material properties.

#### 4.1.1 Effect of Processing Parameters:

- High rotational speed (1400 rpm) and transverse speed (63 mm/min) coupled with the third composition led to the formation of a microstructure with the highest microhardness.
- The application of high transverse seed and high transverse speed induces severe plastic deformation, leading to dynamic recrystallization, and reducing grain size significantly.

#### 4.1.2 Influence of Material Composition:

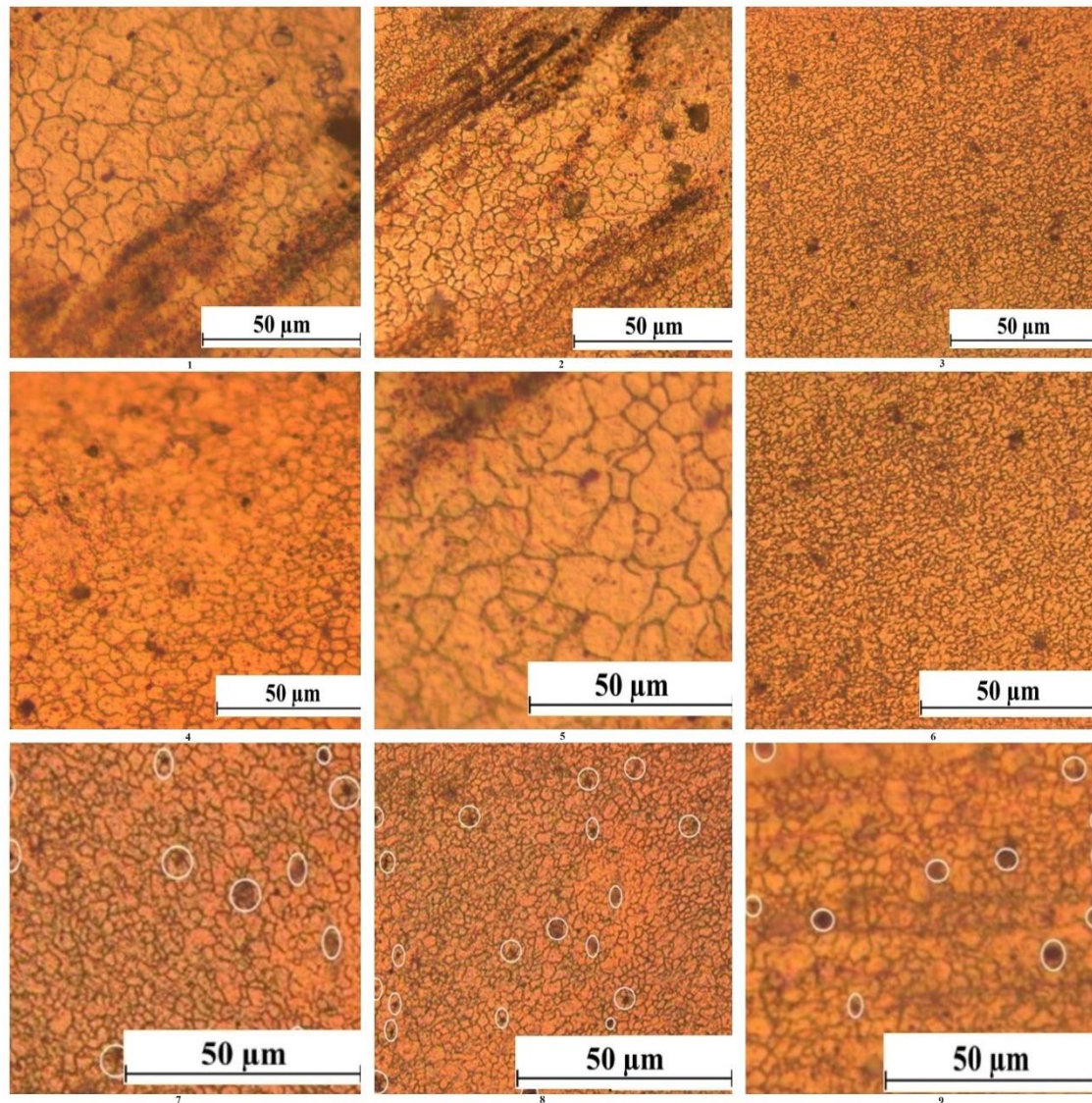
- Because of the higher strain and pinning effect, the presence of SiC, Al<sub>2</sub>O<sub>3</sub>, and Gn particles hinders grain development during recrystallization. Small, equiaxed granules characterize the resulting microstructure.
- Higher percentages of Al<sub>2</sub>O<sub>3</sub> and Gn, combined with increased transverse speed, adversely affect the microstructure, but contribute to a more uniform grain distribution.

#### 4.1.3 Observations from Micrographs:

- The micrographs reveal large strained/strained Gn particles in the second sample, indicating stretching due to a transverse speed of 50 mm/min and the formation of coarse grains.
- In contrast, the fourth sample's micrographs show refined grains and a uniform distribution of SiC, Al<sub>2</sub>O<sub>3</sub>, and Gn particles as a result of dynamic recrystallization induced by increased heat creation from a rotating speed of 1400 rpm.

#### 4.1.4 Implications:

- Coarse grains formed at lower transverse speeds create permissible corrosive sites when less heat is produced, whereas greater transverse speeds and increased percentages of reinforcing particles contribute to a more uniform microstructure.



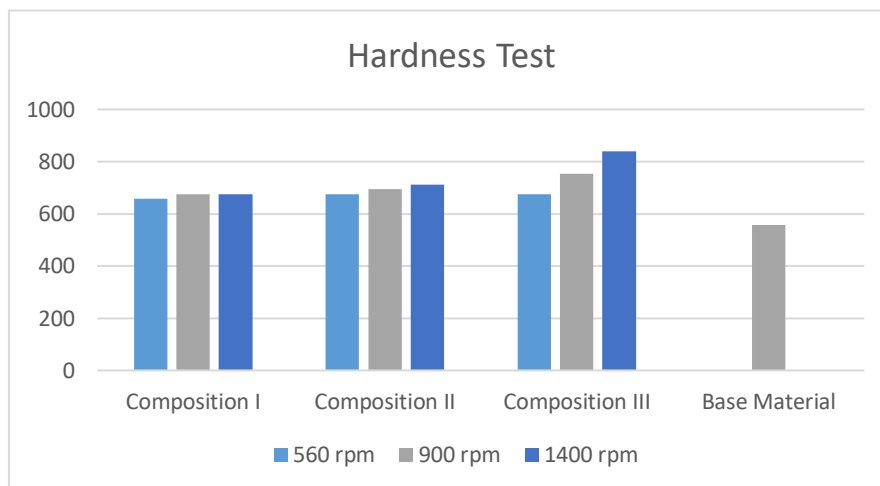
**Figure 4.1:** Micrographs of FSPed composite specimens

## 4.2 Brinell Hardness Test

Brinell Hardness Testing Machine is used to determine the hardness of the Friction Stir Processed Magnesium alloy. First, a 5 mm diameter ball indenter is attached in the hardness tester's chuck. The work piece is firmly set on the testing bench while the lever is in position "A." Once the dial gauge displays the little pointer at '3' (red spot) and the long pointer at '0' on the outer scale, the hand wheel is rotated clockwise to push the indenter, designated with diameter 'D'. Move the lever slowly from position "A" to position "B" to apply the entire 250 kgf load steadily and without jerking. Return the lever to position A gently to remove the weights off, leaving only the initial load engaged, once the long pointer on the dial gauge stabilizes, signifying the completion of the indentation. After that, the specimen is moved from the testing table to the surface plate, and a Brinell microscope is used to determine the impression diameter, or "d." To ensure reliable and consistent results throughout the process, this method is performed for all future tests on the work specimens. Different hardness values for compositions at different rotational and transversal speeds, ranging from 560-1400 rpm and 25-63 mm/s, respectively, are provided in table 4.1.

**Table 4.1:** Hardness values of samples

Sample No.	Hardness Values		Average Hardness Value
	H <sub>1</sub>	H <sub>2</sub>	
1	642.485	675.106	658.7955
2	675.106	675.106	675.106
3	675.106	675.106	675.106
4	747.812	675.106	711.459
5	675.106	675.106	675.106
6	747.812	642.485	695.1485
7	675.106	832.264	753.685
8	747.812	931.143	839.4775
9	675.106	675.106	675.106

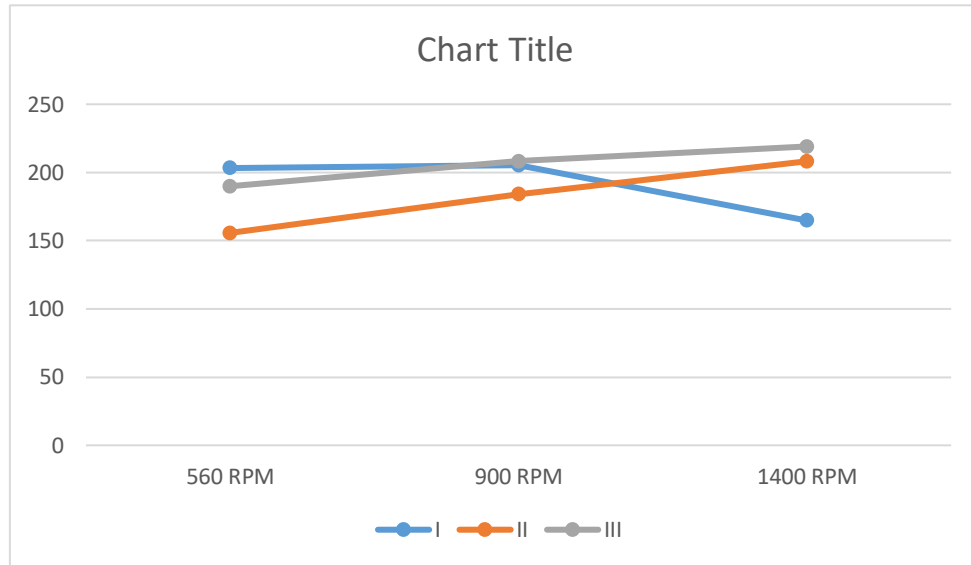
**Graph 4.1:** Result of the Hardness test

The hardness of base material Mg AZ91 was found to be 557.016 N/mm<sup>2</sup> and the composite with lowest hardness was 1<sup>st</sup> sample (500 rpm and 25 mm/sec) with 658.796 N/mm<sup>2</sup>. The hardness value was found highest for 8<sup>th</sup> sample (1400rpm and 63 mm/sec) with 839.478 N/mm<sup>2</sup>.

### 4.3 Tensile Test

The ASTM A370 test technique was used to perform the tensile test on an Instron 5500R. In order to calculate the cross-sectional area at its smallest point, the specimen's dimensions were first measured and documented. This cross-sectional area was then used for all engineering stress calculations. The gauge length can be indicated with a center punch, scribe markings, or an ink drawing. The percentage of elongation at break can be computed by measuring the distance between the gauge marks after they have broken. Prior to installing the specimen in the grips, the testing apparatus was zeroed without the specimen inside. The sample was loaded and the testing speed was determined in one of three ways:

either by measuring the rate of crosshead movement when not under load, the rate of specimen stressing, or the rate of crosshead separation under load. One can determine the yield strength and tensile strength at any convenient speed up to half of the specified yield point and as low as 1/10 of the specified maximum rates. The specimen was tested until it failed or fractured. The broken sample was then taken out of the machine and the shattered ends were put together. The gauge markings' distances were then measured, rounding to the nearest 0.05 millimeter.

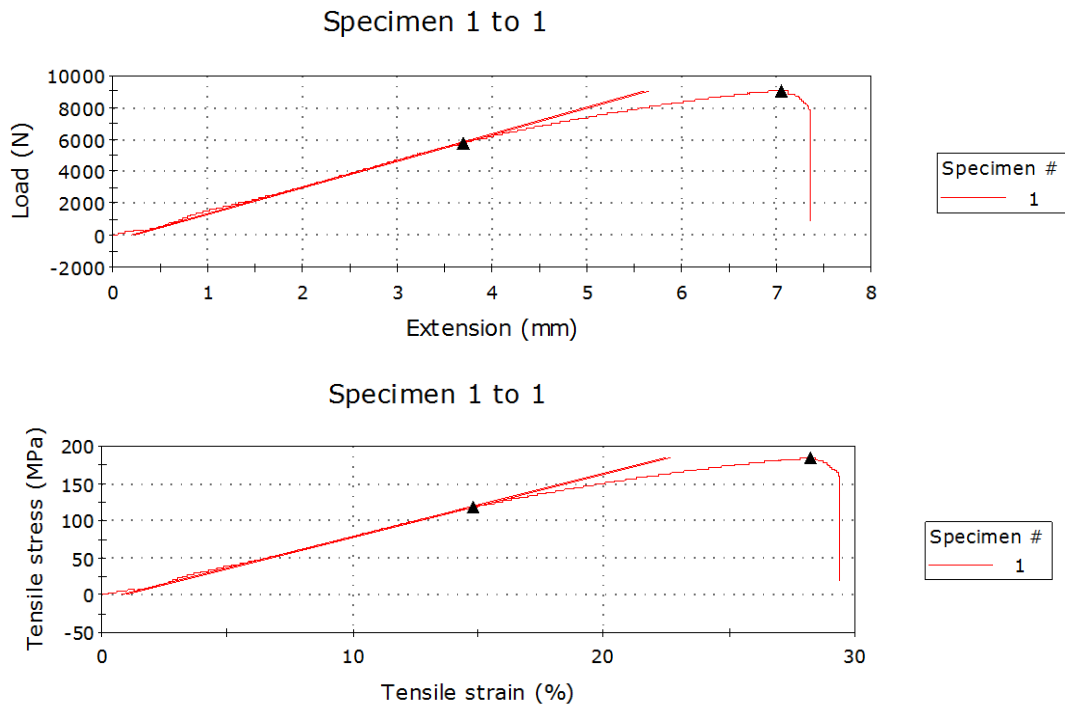


**Graph 4.2:** Result for Tensile test

**Table 4.2:** Outputs of tensile test of various samples

Sample No.	Tensile Strength TS (Mpa)	Yield Strength YS (Mpa)	Elongation E (%)
1	203.4	172.92	9.92
2	155.71	84.91	4.44
3	189.92	156.06	14.84
4	202.21	176.64	10.12
5	205.36	174.16	12.28
6	184.09	157.57	10.05
7	208.23	185.66	12.48
8	219.08	184.21	14.12
9	164.77	125.02	8.48

The results for tensile test are shown in the table 4.2. Graph 4.2 replicates the tensile strength of each sample specimen of different compositions and process parameters. It can be concluded that sample 8 has highest tensile strength (TS) of 219.08 MPa, yield strength (YS) of 184.21 MPa and elongation (E) of 14.12%. Figure 4.2 shows the curves of sample 8.



**Figure 4.2:** Load vs Extension and Stress vs Strain curves for sample 8

#### 4.4 Wear Test

The Pin-on-disk wear test is carried out with ASTM-G99 standards. In the present investigation, the factors of reinforcement percentage (R), distance (D), load (L), and velocity (V) were chosen. The primary objective was to assess the Wear Rate (WR), which was designated as the outcome. The current study delves into the examination of different FSP parameters and compositions of nano powder composite and their influence on the WR. The experiments were executed according to ASTM-G99-27 by utilizing a pin-on-disk wear testing equipment with 100 mm track diameter in a normal laboratory environment at a humidity level (50–60%) and temperature (28– 35 °C). While experimentation the contact surfaces underwent preparation through the process of grinding with SiC paper, followed by cleaning using alcohol. The disc was cleaned with alcohol after each run, and then the surface was finished with an alumina abrasive wheel to an average Ra value of about 0.2  $\mu\text{m}$ . This procedure was done to remove accumulated debris on the wear track. Following each run, the specimens underwent thorough cleaning using alcohol, and their weight was subsequently measured using a precise electronic balance with a precision of 0.001 g.

**Table 4.3:** Wear Rate of FSPed composites

<i>Sample No.</i>	<b>Load-L (N)</b>	<b>Speed-V(m/s)</b>	<b>Distance-D(m)</b>	<b>Wear Rate (<math>\times 10^{-3} \text{ mm}^3/\text{m}</math>)</b>
1	30	2	1500	3.384
2	30	2	1500	3.148
3	30	2	1500	3.073
4	30	2	1500	2.981

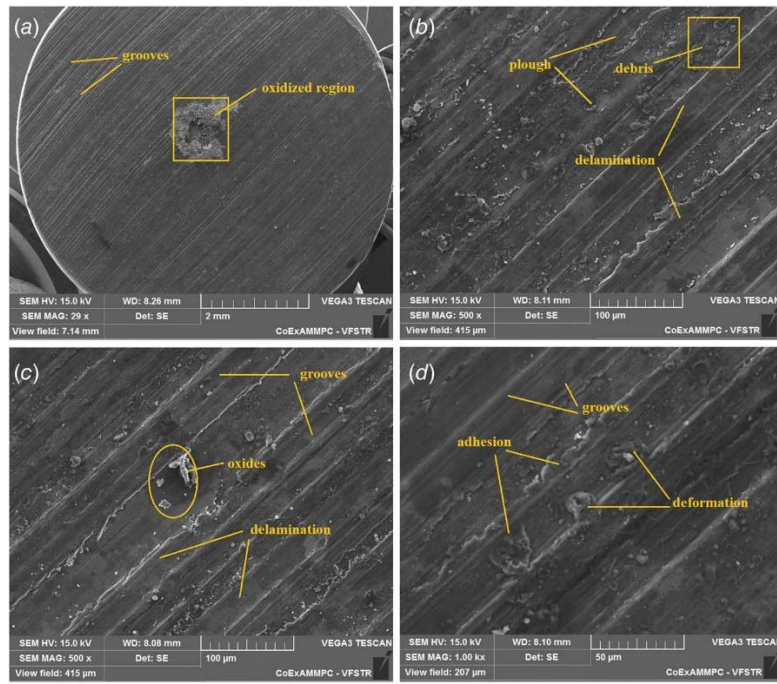


5	30	2	1500	3.296
6	30	2	1500	3.092
7	30	2	1500	2.948
8	30	2	1500	2.856
9	30	2	1500	3.047
<i>B.M</i>	30	2	1500	6.953

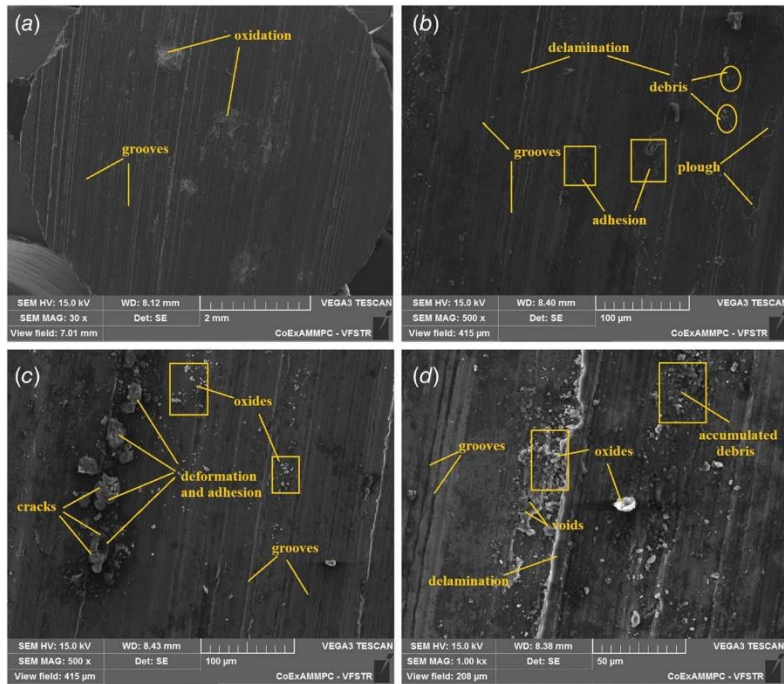


**Graph 4.3: Wear Rate of Composites**

From the results of wear test, we can conclude that the Composition three has lowest wear rate with  $2.856 \times 10^{-3} \text{ mm}^3/\text{m}$  as compared with the base material with  $6.953 \times 10^{-3} \text{ mm}^3/\text{m}$ . The sample 8 has lowest wear rate with almost 58% less than the base material. And the highest wear rate was observed in sample 1 with  $3.384 \times 10^{-3} \text{ mm}^3/\text{m}$ . Overall, a variety of wear mechanisms affect composites, including oxidation, adhesion, abrasion, and delamination. In Figure 4.3 and 4.4, the parallel grooves suggest abrasive wear. Usually, the stiff counter face that had displaced the sample's smoother surface was which caused the grooves to be recognized. During this procedure, the material outer layer was removed, causing the surface to develop the defined grooves. The heat produced by the friction on the pin's surface led to oxidative wear. It was also discovered that the plastic deformation was the reason why the debris had gathered on the pin's surface. In addition, it was seen that when the wear particles separated, tiny cracks and parallel layers developed. Delamination wear is the name given to this stage of wear. In addition, material delamination is the cause of the cavities and fissures on the worn surface. Delamination is the term used to describe a material's surface deformation, fracture initiation, and subsequent crack propagation. Ploughing and crater formation were also evident on the worn-out surface. This extraordinary event reveals the existence of plastic deformation. Under wear conditions, the matrix material deforms plastically, exposing reinforcement particles to the pin's contact surface and further reducing the plastic deformation. In comparison to the lower wear surface of sample 8 shown in figure 4.4, the higher wear surface sample 1 shown in figure 4.3 had more widely spaced grooves and ploughed areas.



**Figure 4.3:** SEM micrographs of high worn-out sample 1 with different magnifications



**Figure 4.4:** SEM micrographs of low worn-out sample 8 with different magnifications

The depth and width of the grooves were found to be enhanced under wear circumstances, suggesting that the abrasion mechanism dominates with the decrease in rotational speed. The oxygen content found on the worn surfaces validates the occurrence of oxidative wear. The reason for this appearance can be traced back to the heat generated during the sliding process, which led to surface oxidation. Significant levels of frictional heat are produced when sliding motion occurs, mostly because of the high concentration of reinforcements. This increased frictional heat makes surface oxidation more likely. By reducing contact between the rough surfaces of sliding components and enhancing wear resistance, the oxide layer serves as a protective barrier.

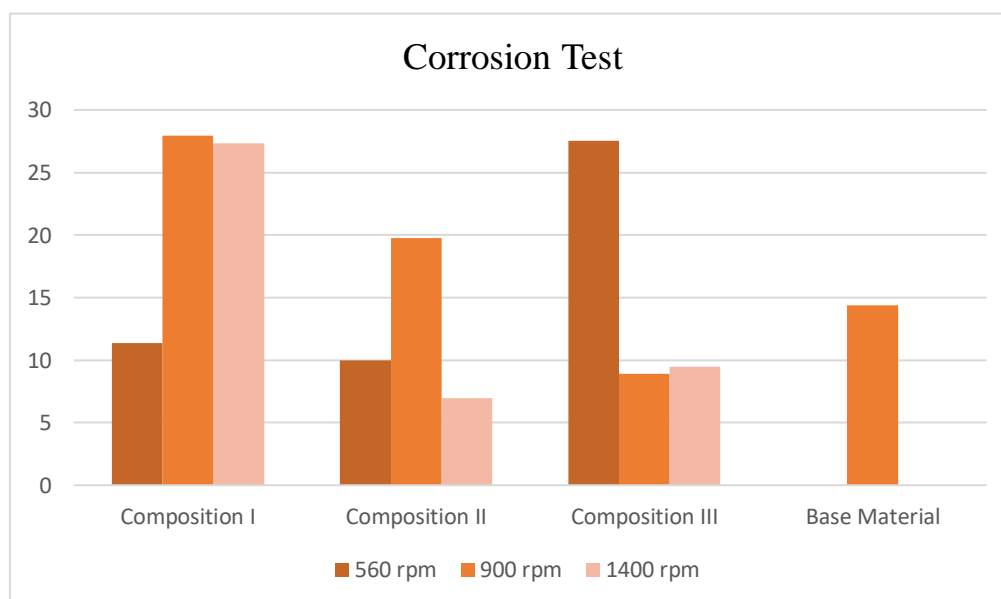


## 4.5 Corrosion Test

The AZ91 alloy, which underwent friction stir processing, underwent a rigorous evaluation method to guarantee precise measurement of corrosion. Samples were first taken out of the treated zone with a wire electrical discharge machine, and then they were uniformly ground with emery sheets. immersion testing for 48 hours in a 3.5 weight percent NaCl solution that was made by dissolving 35.24 grams of powdered NaCl in 1000 milliliters of distilled water. All surfaces of the samples were immersed in the solution to ensure complete submersion. Samples were carefully taken out of the immersion time and cleansed with distilled water to get rid of any remaining solution. Samples were polished with emery paper to remove any corrosion elements, and then they were cleaned again with distilled water. The dried samples were then weighed once more in order to measure the weight variations brought on by corrosion. The obtained information was compiled in Table 4.4 and showed the corrosion rate. The goal of this methodical approach was to offer accurate insights into the AZ91 alloy's corrosion behavior following friction stir processing. From the graph 4.4 it can be concluded that the sample 4 has least weight loss with only 6.97% loss and samples with equal amount of aluminium oxide and graphene content have highest weight loss of about 27.96% which is more than the base material's weight loss which is 14.36%.

**Table 4.4:** Weight loss due to corrosion

Sl. No.	Initial Weight	After Test Weight	Weight loss	Weight loss in %
1	2.346	2.079	0.267	11.38107
2	2.106	1.517	0.589	27.96771
3	2.044	1.485	0.559	27.34834
4	2.780	2.586	0.194	6.978417
5	3.980	3.582	0.398	10
6	2.335	1.874	0.461	19.74304
7	2.090	1.904	0.186	8.899522
8	2.825	2.558	0.267	9.451327
9	2.160	1.565	0.595	27.5463
B.M	2.896	2.480	0.416	14.36464



**Graph 4.4:** Weight loss % due to corrosion of specimens

# Chapter 5

## 5. CONCLUSION






The comprehensive study of AZ91 Mg alloy composites using friction stir processing (FSP) has produced significant insights about improving their mechanical characteristics for a wide variety of application. Significant increases were found by Brinell hardness testing; the 8th sample had the maximum hardness, measuring 839.478 N/mm<sup>2</sup>, compared to the basis material's 557.016 N/mm<sup>2</sup>. Significant improvements in tensile strength were observed in the tensile testing; sample 8 had the highest result, 219.08 MPa, combined with a Yield Strength (YS) of 184.21 MPa and an elongation of 14.12%. Furthermore, wear tests carried out in compliance with ASTM-G99 guidelines demonstrated encouraging outcomes, suggesting improved wear resistance of the composite materials. These results highlight how crucial it is to optimize FSP parameters and material compositions, including Gn, Al<sub>2</sub>O<sub>3</sub>, and SiC nanoparticles, for customizing the mechanical and microstructural properties of AZ91 alloy composites. These developments improve magnesium alloy composites' performance and open the way for their use in severe industries like electronics, aircraft, and the automobile industry, where strong, lightweight materials are in great demand. All things considered, our work offers a strong basis for more investigation and advancement targeted at realizing the maximum potential of magnesium alloy composites treated with FSP.

# Chapter 6

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## ACTIVITY CHART

<b>Activity Description</b>	<b>October 1<sup>st</sup>-4<sup>th</sup> Week</b>	<b>November 1<sup>st</sup>-4<sup>th</sup> Week</b>	<b>December 1<sup>st</sup>-4<sup>th</sup> Week</b>	<b>February 1<sup>st</sup>-4<sup>th</sup> Week</b>	<b>March 1<sup>st</sup>-4<sup>th</sup> Week</b>	<b>April 1<sup>st</sup> Week</b>
<b>Problem identification</b>						
<b>Problem definition</b>						
<b>Literature Review</b>						
<b>Experimentation</b>						
<b>Result Analysis &amp; Report</b>						



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**Batch: 2019-23**

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- 2. To provide training programs that bridge the gap between academia and industry.**
- 3. To create a conducive environment and facilities to improve the overall personality development of the graduates.**
- 4. To make the graduates aware of the role and responsibilities of an engineer in society.**

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PO3: Design / Development of Solutions: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for public health and safety, and cultural, societal, and environmental considerations.

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PO5: Modern Tool Usage: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.

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PO7: Environment and Sustainability: Understand the impact of professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.

PO8: Ethics: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.

PO9: Individual and Team Work: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.

PO10: Communication: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.

PO11: Project Management and Finance: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's work, as a member and leader in a team, to manage projects and in multidisciplinary environments.

PO12: Life-long Learning: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

**Course Outcomes (COs):** After completion of the project work, students will be able to:

CO1: Apply the engineering knowledge to prepare the prototype and experimental setup layouts. (Apply level–L3)

CO2: Analyse the complex engineering problems relevant to society, industry, environment, and sustainability. (Analyse level -L4)

CO3: Design and develop a prototype models and experimental setups with the knowledge of mathematics, science and engineering. (Design and development level –L6)

CO4: Implement the project management and modern IT Tools to make a project report with results and discussions. (Analysis level -L4) CO5: Exhibit the individual and teamwork skills with professional and ethical values and communicate the project works effectively with society. (Apply level- L3)



Project work: CO-PO Articulation matrix

	<b>Program Outcomes (POs)</b>												<b>PSOs</b>		
<b>POs→</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>	<b>1</b>	<b>2</b>	<b>3</b>
<b>CO1</b>	2	2	2	1	-	-	-	-	3	2	2	2	-	3	-
<b>CO2</b>	2	2	2	1	-	-	-	-	2	1	2	2	-	2	-
<b>CO3</b>	2	2	2	1	-	-	-	-	2	1	2	2	-	2	-
<b>CO4</b>	-	-	-	-	-	-	-	-	2	1	3	2	-	-	-
<b>CO5</b>	-	-	-	-	-	-	-	-	2	3	-	-	-	-	-

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