Open Peer Review on Qeios

Investigating the Mechanical and Tribological Effects of MoS2 Reinforcement in AZ91 Magnesium Alloy: A Comprehensive Experimental Study

Nachimuthu Somasundaram[1], Gokulkumar Sivanantham[2], Gurupranes Sivaraj Vijaya, Karthi Nadarajan[1]

1 Dr. Mahalingam College of Engineering and Technology
2 KPR Institute of Engineering and Technology

Funding: No specific funding was received for this work.

Potential competing interests: No potential competing interests to declare.

Abstract

Magnesium is a lightweight metal with an impressive strength-to-weight ratio and is easy to use. This makes it highly desirable in the transportation industry, particularly for applications in which energy conservation and emissions reduction are key priorities. In recent decades, significant advancements have been made in magnesium-based composites, particularly with the introduction of magnesium matrix nanocomposites. These composites achieved enhanced strength through the incorporation of nanoparticles, while maintaining the original toughness of the matrix. This effectively bridges the gap between strength and flexibility, which is often observed in conventional magnesium composites. This breakthrough in the field of magnesium matrix nanocomposites (MMNCs) represents a new era of material development. However, Mg still faces challenges that hinder its widespread adoption. These include limited elasticity and ductility, susceptibility to creep and wear, and high corrosion rate. This study investigates the impact of the processing parameters on the friction stir processing (FSP) of AZ91 and a Mg alloy reinforced with MoS2. Notably, this experiment used the hole technique instead of the more common groove method. While the groove method dominates FSP applications, this project employs a 2 mm drill technique. The optimal processing conditions were as follows: rotation speed of 1100 rpm, travel speed of 15 mm/min, load of 10 kN, and use of a tungsten carbide tool material owing to its exceptional strength and durability. The surface microstructures and tensile strengths of the FSP-treated areas were analyzed further.

Nachimuthu S[1,*], **Gokulkumar S**[2], **Gurupranes SV**[1], and **Karthi N**[2]

[1] *Department of Mechanical Engineering, Dr.Mahalingam College of Engineering and Technology, Pollachi Coimbatore, Tamil Nadu – 642 003, India.*

[2] *Department of Mechanical Engineering, KPR Institute of Engineering and Technology, Coimbatore, Tamil Nadu – 641 407, India.*

[*]Corresponding author: Nachimuthu S, muthunachi48@gmail.com

Keywords: Magnesium matrix nanocomposites (MMNCs), Friction Stir Processing (FSP), MoS_2 reinforcement, AZ91 and Mg alloy, hole technique, tensile strength, surface microstructure.

1. Introduction

The growing demand for lightweight vehicles in the automotive and aerospace industries necessitates advancements in material design to achieve a significant weight reduction. Replacing heavier materials such as steel with lighter alloys, particularly aluminum alloys, is a crucial step in this direction. However, aluminum alloys often require reinforcement to meet the required rigidity and strength for specific applications. Magnesium and its alloys have emerged as promising alternatives owing to their potential for improving performance and energy efficiency. Compared to conventional building metals, magnesium has a significantly lower density (approximately two-thirds that of aluminum) and superior specific strength (strength-to-weight ratio). These qualities make it highly desirable for applications in which reducing greenhouse gas emissions and fuel consumption is paramount [1][2][3].

Despite these advantages, limitations, such as low strength, poor damage resistance, low modulus (stiffness), and sensitivity to high temperatures, have hindered the widespread adoption of magnesium alloys. Reinforcements are typically added to strengthen the base metal and overcome these limitations. Magnesium composites could potentially serve as viable replacements for currently dominant aluminum-based composites. Friction stir processing (FSP) is a solid-state processing technique derived from friction stir welding (FSW). This offers a means of modifying the microstructure of a material at a localized level. The concept behind FSP is relatively straightforward: a rotating tool stirs and mixes the material while traveling along a designated path, thereby generating a significant plastic deformation and heat exposure. This process significantly alters the microstructure of the treated zone, primarily owing to localized heating.

FSP's versatility of FSP has led to its application in various fields, including the development of metal matrix composites and microstructure enhancement of cast aluminum alloys. The use of FSP to create surface metal matrix composites (SMMCs) with prepositioned reinforcements has been documented in several studies [4]. Careful selection of the processing parameters is crucial to achieve the desired microstructure in the composite layer. Notably, FSP offers the advantage of treating the surface composite at temperatures below the melting point of the substrate, thereby effectively mitigating the potential problems. There is extensive literature exploring various FSP techniques for creating composite surfaces, including placing the powder on top of the surface or within a pre-machined groove. The core principle of FSP involves significant localized plastic deformation to modify material properties. A rotating tool traverses a material surface and generates frictional heat and plastic deformation. This thermomechanical process, known as friction stir processing, enhances the material without altering its phase (solid, liquid, or gas).

Several studies have investigated the impact of FSP on the mechanical properties of the AZ91 magnesium alloy at room and elevated temperatures [5][6]. These studies compared the microstructure and mechanical behavior of samples treated using different FSP techniques (i.e., narrow FSP (NFSP) and SFSP). Similarly, Liu et al. [7] examined the response of

FSP AZ91 alloys to saltwater solutions. Notably, Chai et al. [8] were the first to explore friction stir processing with shear-focused deformation (SFSP) of Mg alloys [9]. Their study compared the microstructure and tensile properties of AZ91 magnesium alloys treated using conventional FSP and SFSP. Similarly, Zhang et al. [6] successfully achieved a fine-grained multiphase structure in a cast AZ91 magnesium alloy using FSP. Iwaszko and Kuda [10] employed FSP to modify the surface layer of an AZ91 magnesium alloy. Raja et al. [11] investigated the development of a layered microstructure and its influence on super plasticity by subjecting a cast AZ91 Mg alloy to multi-pass FSP [12].

This study investigated the influence of processing parameters on the friction stir processing of AZ91 and a Mg alloy reinforced with MoS_2. This study employed the hole technique, which deviates from the more prevalent groove method used in most FSP applications. The experiment utilized a 2 mm drill technique and identified the optimal processing conditions: rotation speed of 1100 rpm, travel speed of 15 mm/min, load of 10 kN, and tool material tungsten carbide (owing to its exceptional strength and durability). The surface microstructures and tensile strengths of the FSP-treated areas were analyzed further.

2. Materials and methods

An AZ91 Mg alloy with dimensions of 125 × 75 × 8 mm was used for this particular task. Using an end mill cutter with a 2 mm diameter and 3 mm depth of cut, the vertical CNC milling machine drills a hole. The spindle (rpm) was then rotated at 400 rpm. Similarly, 42 holes were bored and each hole was filled with MoS_2.

MoS_2 particles were used to increase the tensile strength of the material. The particles in the reinforcing powder were ground with balls to obtain the correct size and proportional distribution. Both goals were achieved using this approach. In the crystal structure of molybdenum disulfide (MoS_2), sulfur atoms are arranged in hexagonal planes next to molybdenum atoms. The van der Waals force, which is relatively weak, holds the layers together. As a result, the layers were physically separated, allowing the creation of two-dimensional MoS_2 sheets. The covalent bonds between the S and Mo atoms are quite strong, and these three planes are stacked on top of each other [2]. MoS_2 is a mineral called molybdenite in its natural state. Pure molybdenum disulfide is black, glossy, and light-reflective. Because the weak connections of the sheets make it easy for them to slide over one another, MoS_2 was used as a lubricant. Thus, MoS_2 is a suitable material for this purpose. MoS_2 is occasionally used in place of graphite when high vacuum is required. However, the maximum temperature at which MoS_2 can operate in these applications is lower than that at which graphite can operate in similar applications. Molybdenum disulfide (MoS_2), a semiconducting bulk material, has an indirect bandgap of ~ 1.2 electron volts. MoS_2 is not essential for optoelectronic applications. In contrast to graphene, molybdenum disulfide (MoS_2) has a bandgap that is not zero. This is only one of the many interesting and perhaps helpful sections of the text. Because MoS_2 is a flexible semiconducting material that can conduct electricity, it is an excellent material for use in electronics and logic circuits. This also enables a significant cost reduction. Furthermore, the extraordinarily broad indirect bandgap can be overcome using MoS_2 in the bulk form. The indirect bandgap changes into a direct bandgap as the size decreases. With this change, it was shown that optoelectronic devices could operate with only one sheet of MoS_2. It has the potential to be utilized in both low-power electrical devices and short-channel field-effect transistors because of its two dimensions (FETs). This is because it is possible to change the electrostatic properties of a material based on its structure. The atomic and molecular makeup values of disulfide nanoparticles are listed in **Table 1.**

Table 1. Chemical Composition of Molybdenum Disulfide Nanoparticles

Element	Content (%)
Molybdenum	59.94
Sulfur	40.06

The FSP tool employs a shoulder fabricated from H-13 tool steel, which is known for its durability and heat resistance during processing. This shoulder measured 18 mm wide and provided a stable platform for material interaction. The tool also incorporated a square-shaped pin, measuring 3.2 mm in length and 6 mm in width. This pin plays a crucial role in the stirring of the material during the FSP. Finally, the assembly process involved precisely inserting the pin into the corresponding hole machined on the shoulder plate. This ensures proper alignment and functionality of the tool. Figures 1 illustrates the FSP tool and its settings.

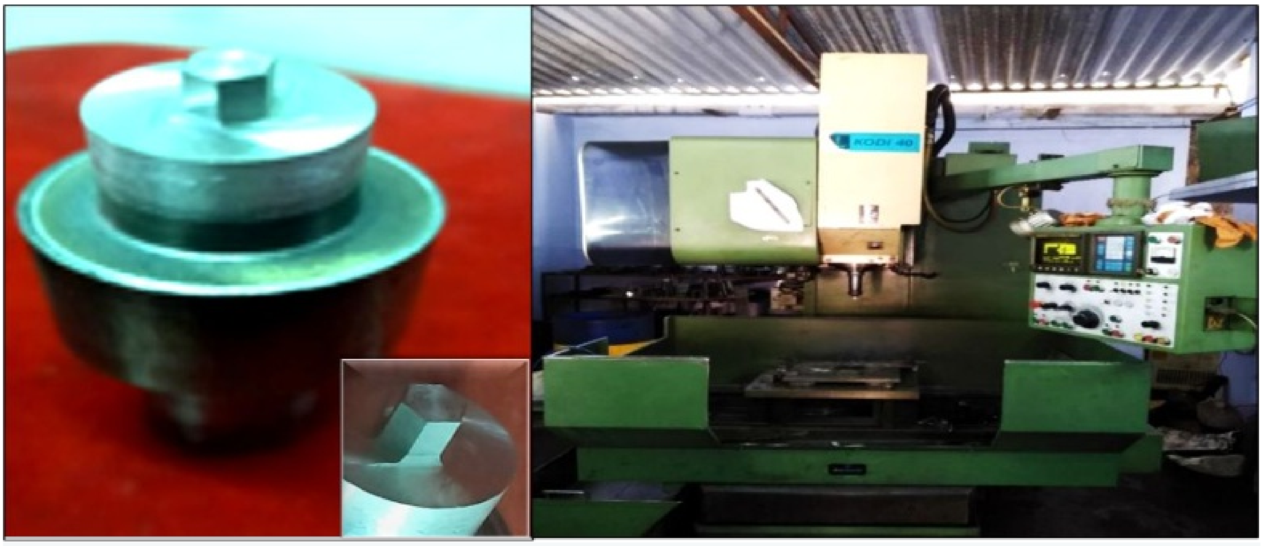


Figure 1. FSP tool and setup

Prior to friction stir processing (FSP), the prepared hole in the AZ91 base plate was filled with 100% concentration MoS_2 powder. Subsequently, an H-13 tool was used to secure the workpiece onto the FSP machine for processing. Table 2 details the specific process parameters employed during FSP. **Figure 1** illustrates the test setup used for the reinforced AZ91 plate. It's important to note that a vertical milling machine served as the platform for the entire FSP process.

Table 2. Process Parameters

S. No	Process parameters	Values
1	Rotational speed (rpm)	1200
2	Traverse speed (mm/min)	20
3	Axial force (kN)	7
4	Number of passes	1

The FSP process begins when the tool plunges into the workpiece. The rotating pin generates frictional heat when it comes into contact with the surface of the material. This heat softens the metal matrix around the pin without reaching the melting temperature. Friction continues to play a crucial role, as the shoulder makes contact with the workpiece. The resulting frictional forces induce significant plastic deformation in the surrounding materials [13]. This stirring action by the pin refines the microstructure, creating a more uniform and finer grain size [14]. Following processing, the material was removed from the machine, polished to remove any surface imperfections, and subjected to a battery of mechanical tests to evaluate the effectiveness of the treatment.

3. Result and Discussions

This section explores how two critical processing parameters in friction stir processing (FSP)–deformation temperature and strain rate–influence the microstructure, tensile behavior, deformation mechanisms, and failure characteristics of FSP-modified AZ91 magnesium alloy [15]. Understanding the interplay between the processing parameters and material properties is crucial for optimizing the FSP technique to achieve the desired properties in the final AZ91 Mg-alloy component [16].

Hardness test

Hardness measurement is a vital tool in materials research, offering a quick assessment of material properties and the effectiveness of processing parameters. It also provides insight into the evolution of the microstructure [17]. In this study, the hardness of the FSP-modified weld zone in the AZ91 magnesium alloy was evaluated at room temperature (25.2°C) using a Brinell hardness tester, as shown in Figure 2. The samples were sectioned from the stir zone and prepared according to ASTM E10 specifications. The hardness was measured at various locations in the weld zone using different indentation sizes with a maximum load capacity of 50 kg. Table 3 lists the average Vickers Hardness Index (HVI) and observed values for each sample categorized by rotational speed (1100 rpm, 900 rpm, or 700 rpm).

Figure 2. Specimen for hardness test

Table 3. FPS observed values of HV10

Sample ID	Observed value HV10			Average
	1	2	3	
1100 rpm	73	73	77	74
900 rpm	74	73	72	73
700rpm	75	80	71	76

The microstructure of the FSP-modified AZ91 magnesium alloy (AZ91-FSP) exhibited significant grain refinement compared with that of the fully annealed baseline material. As shown in Table 3, the average grain size within the stir zone (SZ) of the AZ91-FSP specimens was reduced to approximately 1.4 ± 0.5 μm, compared to the 7.4 ± 1.0 μm of the full-annealed samples. This refinement is likely attributable to the dynamic recrystallization (DRX) effect induced by the high strain rate and the frictional heat generated during the FSP process.

Hardness testing revealed a significant improvement in hardness across all samples processed with FSP and MoS_2 reinforcements. Interestingly, the sample processed at 1100 rpm demonstrated the highest surface zone hardness, ranging from 73 HV_{10} to 77 HV_{10} with an average of 74 HV_{10} (Table 3). Similar trends were observed for the samples processed at 900 rpm and 700 rpm, with average hardness values of 73 HV_{10} and 76 HV_{10}, respectively. These results suggest a positive interaction between the AZ91 matrix and MoS 2 reinforcement, leading to an overall enhanced hardness compared with the non-reinforced material [4][6][16][18].

Tensile test

Following the FSP, the processed workpiece was sectioned using a suitable cutting technique (e.g., waterjet cutting and electrical discharge machining) to obtain samples with the geometry specified for tensile testing according to a standardized test method (e.g., ASTM E8/E8M). To ensure consistency, all the testing parameters, such as the strain rate and gauge length, were maintained constant throughout the experiment. The ultimate tensile strengths (UTS) of both the FSP-modified and non-reinforced (baseline) AZ91 Mg alloy samples were measured using a universal testing machine. This comparison allowed the evaluation of the effectiveness of the MoS$_2$ reinforcement on the tensile strength of the AZ91 alloy.

The tensile behavior and microstructure of the FSP-modified AZ91 Mg alloy with MoS$_2$ reinforcement were investigated. **Figures 3, 4, and 5** present the results for the samples processed at different rotational speeds (1100, 900, and 700 rpm, respectively). Each figure shows the microstructure of the reinforced alloy at magnifications of 100x and 500x alongside the corresponding stress-strain curve obtained from the tensile test. It is important to note that for all the samples, the testing parameters were kept constant, including a fixed initial strain rate, maximum load capacity of 30 kN, gauge length of 1000 mm, and cross-sectional area of approximately 46 mm² (specific values for each sample can be mentioned if available).

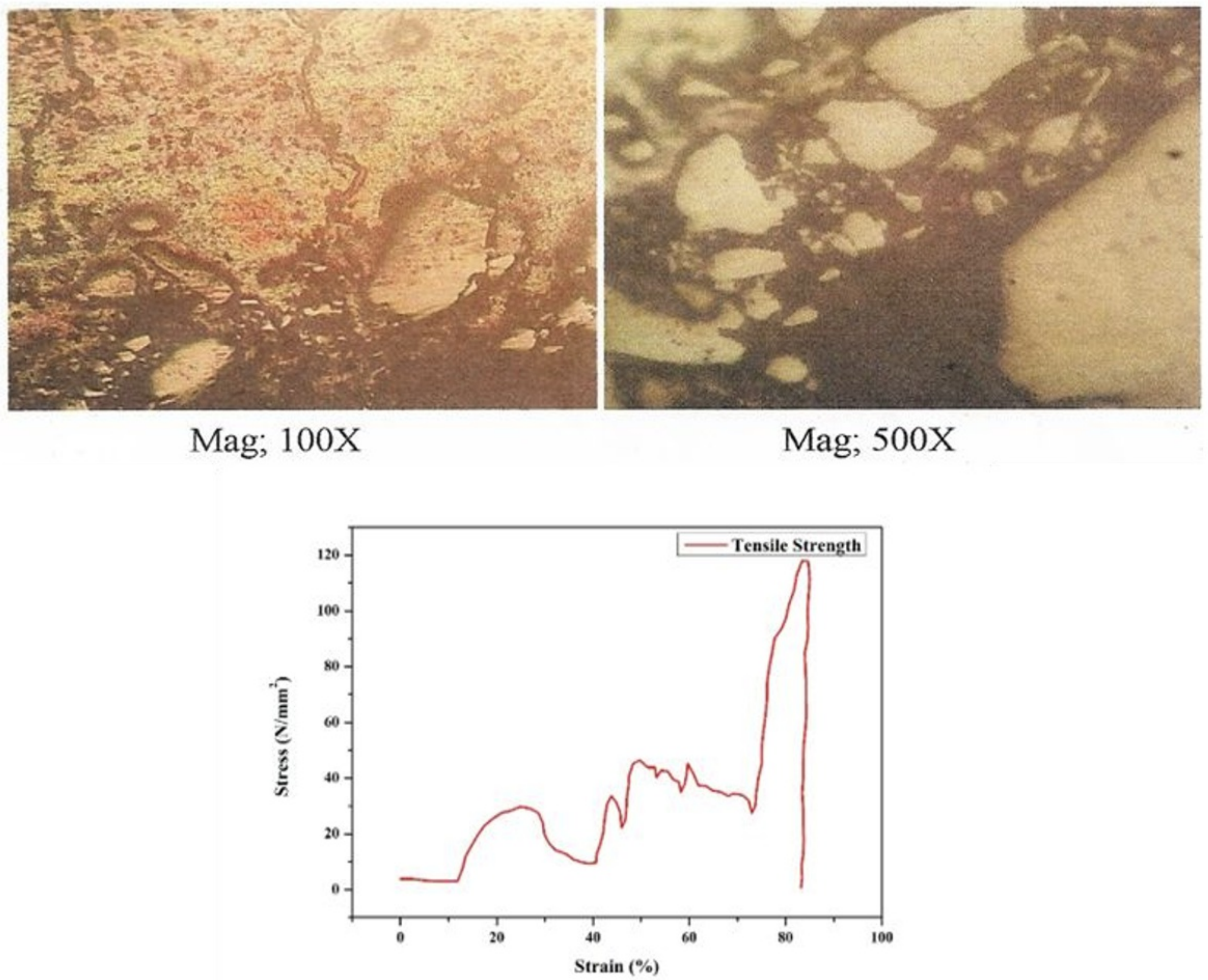


Figure 3. Sample 1100rpm with 100X And 500X And Tensile Stress and Strain Evaluation

Sample obtained at 1100 rpm (Figure 3)

The sample processed at 1100 rpm exhibited a maximum tensile strength of 121.897 N/mm² (or MPa), maximum elongation of 20.742 mm, and a maximum load of 5.59 kN.

Sample centrifuged at 900 rpm (Figure 4)

The sample processed at 900 rpm demonstrated a maximum tensile strength of 117.592 N/mm² (or MPa), a maximum elongation of 5.256 mm, and a maximum load of 5.4499 kN.

Sample centrifuged at 700 rpm (Figure 5)

The sample processed at 700 rpm exhibited the highest tensile strength of the three samples, reaching 132.975 N/mm² (or MPa). However, its elongation was lowest at 5.291 mm, and the maximum load was 4.894 kN. Microscopic observations revealed changes in the microstructure of the alloy after the FSP treatment, suggesting that the process might have reached the recrystallization temperature for magnesium alloys, which is typically approximately 25°C. Further investigation using techniques such as electron backscatter diffraction (EBSD) could provide more detailed information about grain size and texture evolution.

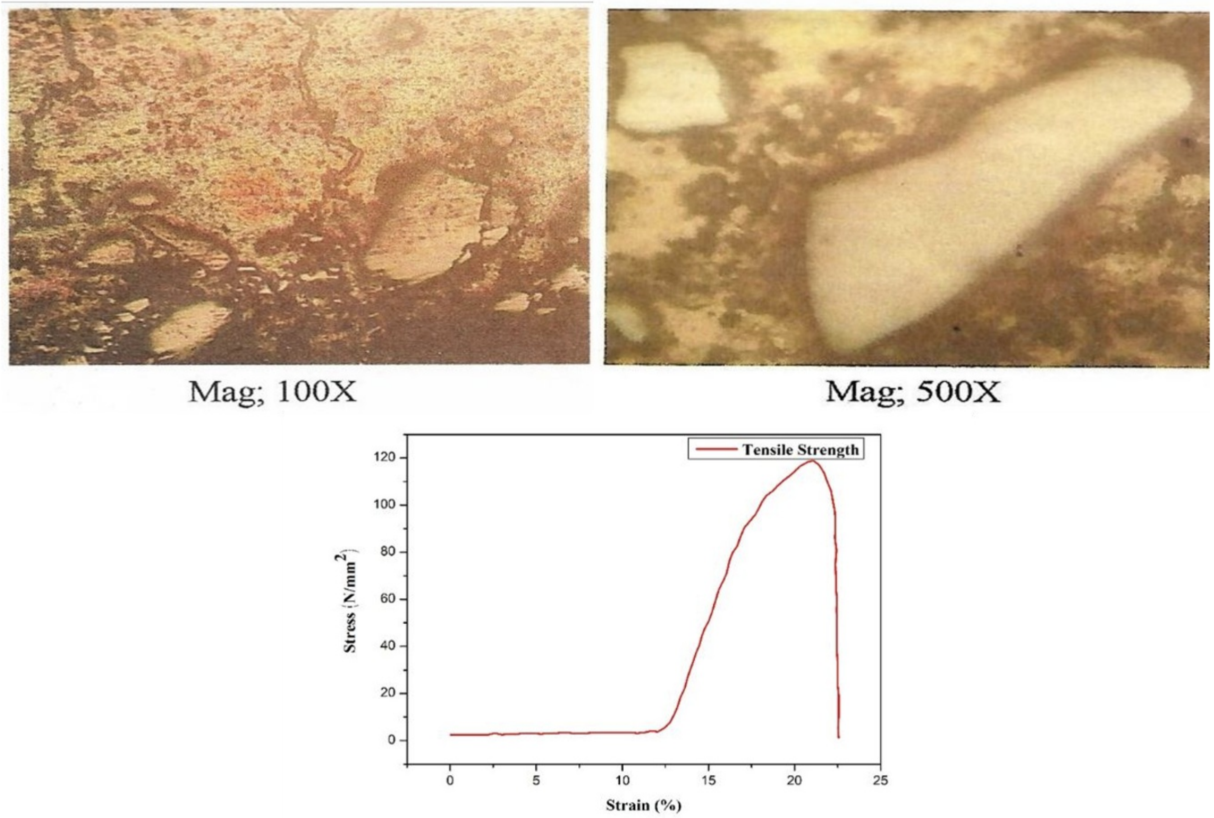


Figure 4. Sample 900rpm with 100X And 500X And Tensile Stress and Strain Evaluation

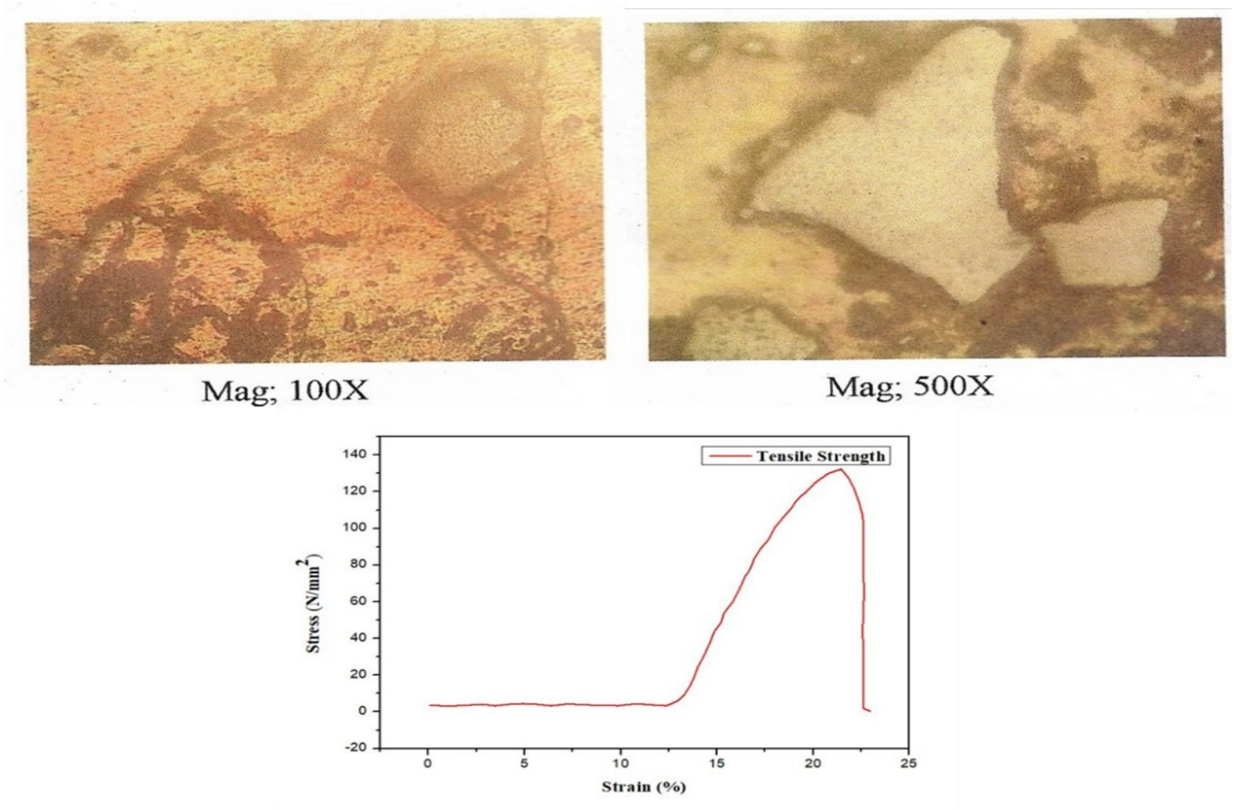
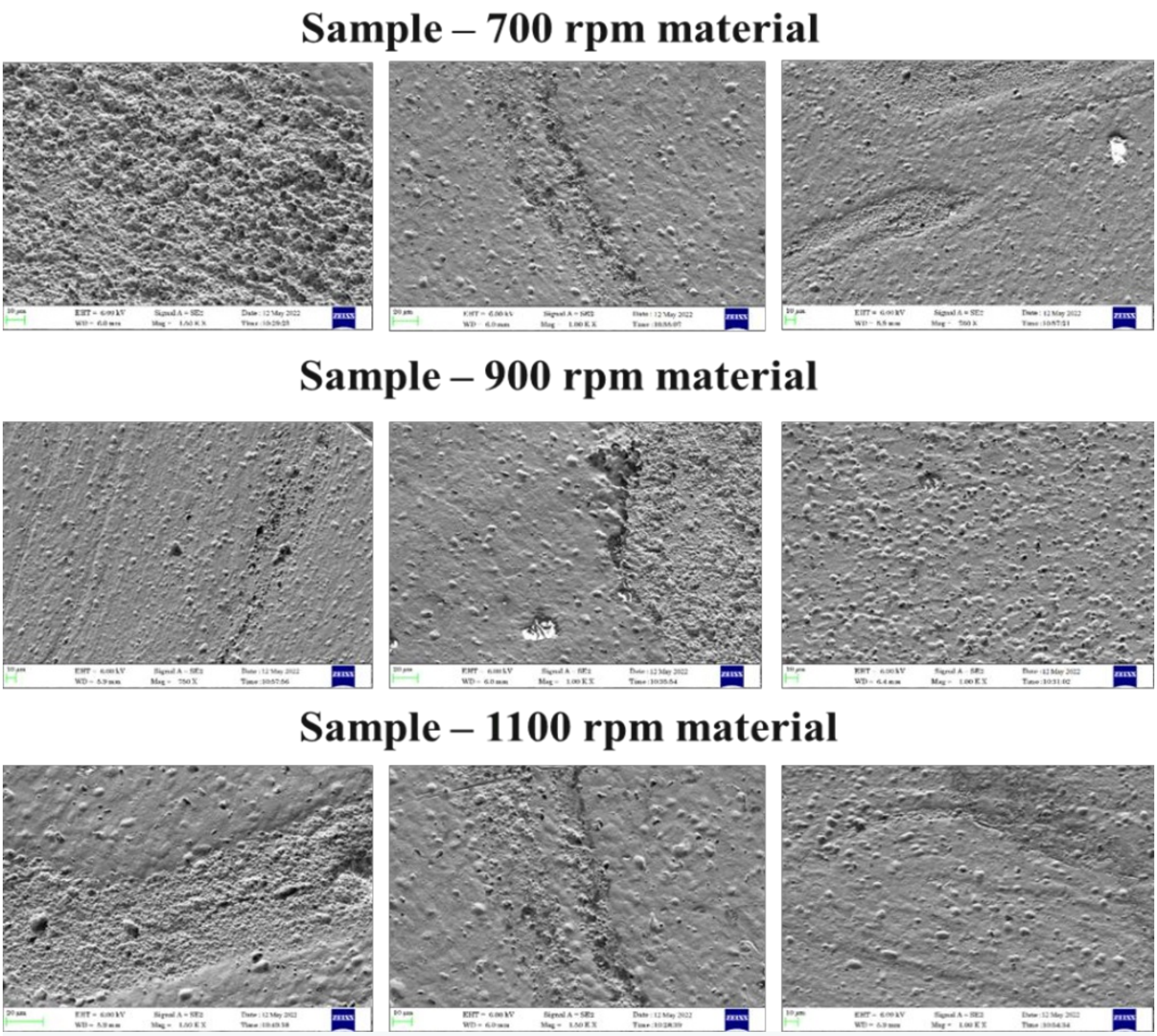


Figure 5. Sample 700rpm with 100X And 500X And Tensile Stress and Strain Evaluation

SEM Analysis

The elemental composition of the FSP-modified AZ91 Mg alloy with MoS2 reinforcement was investigated using EDS analysis**. Figure 6** presents the results for samples processed at different rotational speeds (1100, 900, and 700 rpm). Each figure shows EDS spectra obtained at specific locations within the modified zone. Specific measurement distances are indicated in the Figures (e.g., 10 µm and 20 µm).

Figure 7 depicts the EDS results for the samples processed at different rotational speeds (1100, 900, and 700 rpm). Here, the focus seems to be on analyzing the potential variations in the chemical composition of the magnesium alloy within the FSP-modified zone (stir zone) at different locations. This analysis helps understand how the processing parameters and MoS2 reinforcement affect the elemental distribution within the material.

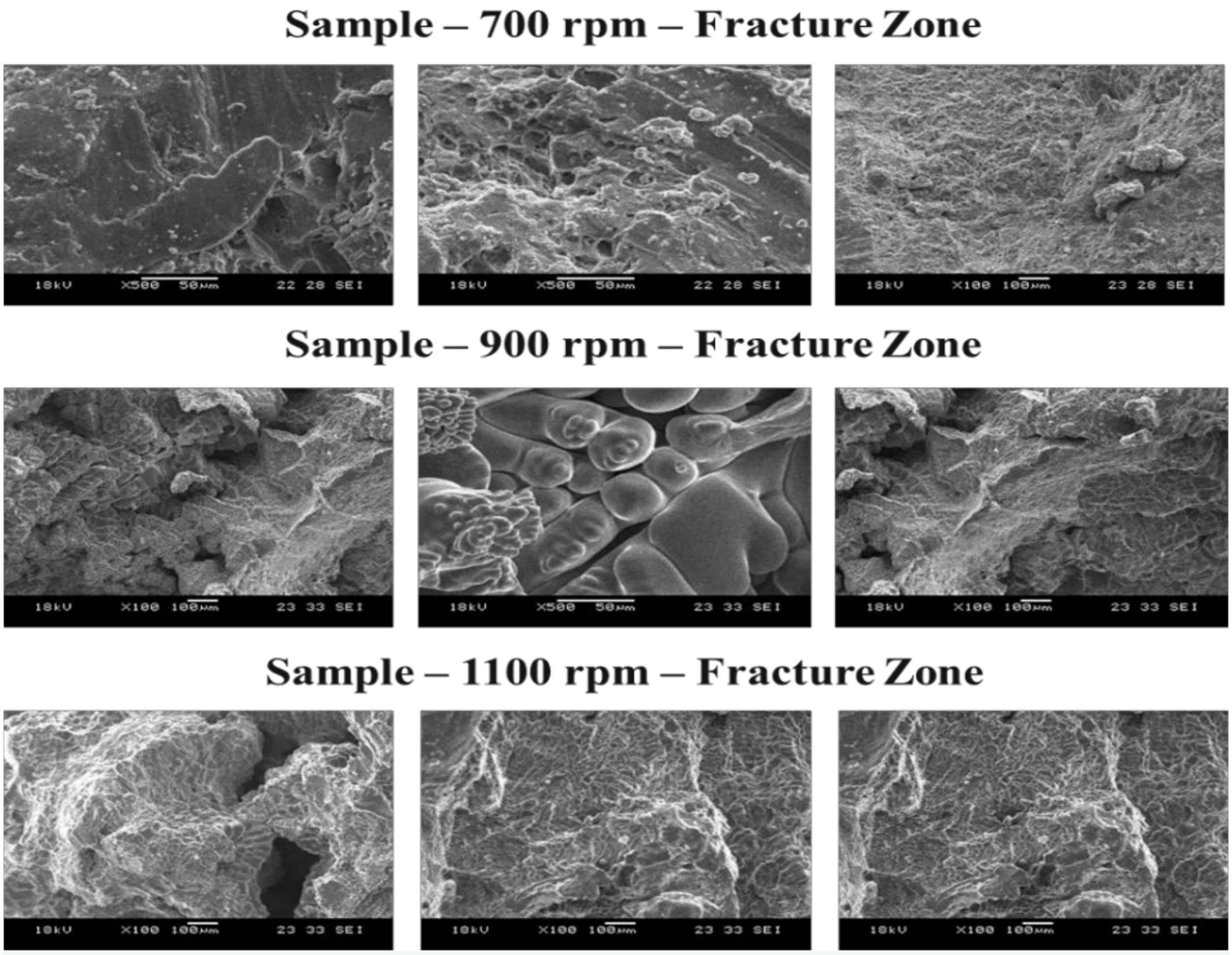


Sample – 700 rpm material

Sample – 900 rpm material

Sample – 1100 rpm material

Figure 6. Sample Materials of 700 rpm, 900 rpm and 1100 rpm



Sample – 700 rpm – Fracture Zone

Sample – 900 rpm – Fracture Zone

Sample – 1100 rpm – Fracture Zone

Figure 7. Fracture Zone of 700 rpm, 900 rpm and 1100 rpm

4. Conclusion

This study investigates the influence of FSP on the microstructure and tensile properties of an AZ91 magnesium alloy composite reinforced with MoS_2 particles. The AZ91 Mg alloy workpiece had dimensions of 125 × 75 × 8 mm (length × width × thickness). A vertical CNC milling machine was used to create a series of holes in the AZ91 Mg alloy plate for MoS_2 reinforcement. These holes were drilled with a 2 mm diameter end mill cutter at a depth of 3 mm. The spindle speed was maintained at 400 rpm for all the holes. A total of 42 holes were created and filled with MoS_2 particles. This approach utilizes a through-hole reinforcement technique, which differs from the more common groove technique typically employed in FSP. The optimal FSP processing parameters identified in this study were a rotational speed of 1100 rpm, transverse speed of 15 mm/min, downward force (load) of 10 kN, and tungsten carbide tool material (owing to its high strength and durability). This study focused on evaluating the impact of FSP on two key aspects of the AZ91 + MoS 2 composite. The microstructure of the FSP-treated zone was analyzed to understand the grain size, morphology, and potential presence of intermetallic phases, and tensile testing was conducted to assess the strength, ductility, and overall tensile behavior of the material. This study successfully achieved the FSP of AZ91 alloy reinforced with MoS_2 particles using a through-hole technique. The results demonstrated that the combination of AZ91 Mg alloy and MoS_2 reinforcement led to enhanced surface hardness and improved wear resistance. The potential of AZ91 + MoS_2 composites for aerospace applications is promising, owing to the combination of the lightweight properties of Mg and the beneficial effects of MoS_2 reinforcement. Further research is recommended to explore the broader mechanical properties and performance of this composite material for various aerospace applications.

Statements and declarations

Funding

No funding or financial interests were involved in the study.

Conflict of interest

The authors declare that they have no conflict of interest. This work has not been published or presented elsewhere.

Authors contribution

All authors are equally contributed

Acknowledgments

We would also like to express our appreciation to the faculty members in the Department of Mechanical Engineering, Dr. Mahalingam College of Engineering and Technology, for their technical support.

References

1. ^*Gupta A, Singh P, Gulati P, et al. Effect of Tool rotation speed and feed rate on the formation of tunnel defect in Friction Stir Processing of AZ31 Magnesium alloy. Materials Today: Proceedings 2015; 2: 3463–3470.*
2. a, b*Babu J, Anjaiah M, Mathew A. Experimental studies on Friction stir processing of AZ31 Magnesium alloy. Materials Today: Proceedings 2018; 5: 4515–4522.*
3. ^*Chang CI, Wang YN, Pei HR, et al. Microstructure and Mechanical Properties of NanoZrO2 and Nano-SiO2 Particulate Reinforced AZ31-Mg Based Composites Fabricated by Friction Stir Processing. Key Engineering Materials 2007; 351: 114–119.*
4. a, b*Balakrishnan M, Dinaharan I, Palanivel R, et al. Synthesize of AZ31/TiC magnesium matrix composites using friction stir processing. Journal of Magnesium and Alloys 2015; 3: 76–78.*
5. ^*del Valle JA, Rey P, Gesto D, et al. Mechanical properties of ultra-fine grained AZ91 magnesium alloy processed by friction stir processing. Materials Science and Engineering: A 2015; 628: 198–206.*
6. a, b, c*Zhang D, Wang S, Qiu C, et al. Superplastic tensile behavior of a fine-grained AZ91 magnesium alloy prepared by*

friction stir processing. Materials Science and Engineering: A 2012; 556: 100–106.

7. ^*Liu Q, Ma Q, Chen G, et al. Enhanced corrosion resistance of AZ91 magnesium alloy through refinement and homogenization of surface microstructure by friction stir processing. Corrosion Science 2018; 138: 284–296.*
8. ^*Chai F, Zhang D, Li Y, et al. High strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing. Materials Science and Engineering: A 2013; 568: 40–48.*
9. ^*Sahu G. A BRIEF REVIEW ON MG ALLOYS THEIR PROPERTIES AND APPLICATION. Vol No.*
10. ^*Iwaszko J, Kudła K. Microstructure, hardness, and wear resistance of AZ91 magnesium alloy produced by friction stir processing with air-cooling. Int J Adv Manuf Technol 2021; 116: 1309–1323.*
11. ^*Raja A, Biswas P, Pancholi V. Effect of layered microstructure on the superplasticity of friction stir processed AZ91 magnesium alloy. Materials Science and Engineering: A 2018; 725: 492–502.*
12. ^*Isaac CW, Ezekwem C. A review of the crashworthiness performance of energy absorbing composite structure within the context of materials, manufacturing and maintenance for sustainability. Composite Structures 2021; 257: 113081.*
13. ^*Jin L, Lin D, Mao D, et al. Mechanical properties and microstructure of AZ31 Mg alloy processed by two-step equal channel angular extrusion. Materials Letters 2005; 59: 2267–2270.*
14. ^*Lorenzo-Martin MC, Ajayi OO. Surface Layer Modification of 6061 Al Alloy by Friction Stir Processing and Second Phase Hard Particles for Improved Friction and Wear Performance. Journal of Tribology; 136. Epub ahead of print 9 July 2014. DOI: 10.1115/1.4027860.*
15. ^*Yang M, Xu C, Wu C, et al. Fabrication of AA6061/Al2O3 nano ceramic particle reinforced composite coating by using friction stir processing. J Mater Sci 2010; 45: 4431–4438.*
16. [a], [b]*Dinaharan I, Zhang S, Chen G, et al. Titanium particulate reinforced AZ31 magnesium matrix composites with improved ductility prepared using friction stir processing. Materials Science and Engineering: A 2020; 772: 138793.*
17. ^*Asadi P, Givi MKB, Parvin N, et al. On the role of cooling and tool rotational direction on microstructure and mechanical properties of friction stir processed AZ91. Int J Adv Manuf Technol 2012; 63: 987–997.*
18. ^*Chai F, Zhang D, Li Y. Microstructures and tensile properties of submerged friction stir processed AZ91 magnesium alloy. Journal of Magnesium and Alloys 2015; 3: 203– 209.*