Fabrication of Magnesium-Based Surface Composite Using Friction Stir Processing

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Abstract — The increasing demand for lightweight, high-performance materials in the transportation sector has accelerated the development of advanced metal matrix composites. Carbon-based nanomaterials are widely utilized to improve the mechanical and microstructural properties of these materials. In the present study, a multi-walled carbon nanotube (MWCNT)-reinforced AZ31 magnesium alloy surface composite was fabricated through friction stir processing (FSP) using a variable revolutionary pitch (RP), while maintaining all other processing parameters constant. The fabricated composites exhibited a more uniform particle distribution and significant grain refinement, with the average grain size decreasing from 33 µm in the base material (BM) to 4.5 µm for 0.1 RP and 5.7 µm for 0.05 RP. Furthermore, the microhardness increased by nearly 72 %, while the tensile strength improved by up to 82% compared to the BM. These enhancements are primarily attributed to grain boundary strengthening and Zener pinning effects induced by dynamic recrystallization (DRX).

Keywords: AZ31, Magnesium, FSP, MWCNT, Revolutionary Pitch, Surface Composite

I. Introduction

Magnesium alloys are increasingly replacing aluminum and steel in the aerospace and automotive industries, as well as plastics in electronic and computer applications, owing to their low density and favorable thermal and electrical conductivity[1]. However, their application in high-performance environments is restricted by relatively poor mechanical strength and wear resistance[2]. To overcome these limitations, magnesium alloys reinforced with Carbonaceous nanomaterials, commonly referred to as magnesium matrix composites (MMCs), have been developed to enhance mechanical and tribological performance[3]. Carbon-based nanomaterials, particularly carbon nanotubes (CNTs), have attracted significant attention due to their outstanding mechanical strength and excellent thermal and electrical conductivity. These unique characteristics make CNTs highly suitable for the development of advanced nanocomposites for a wide range of engineering applications[4]. However, achieving a uniform distribution of CNTs within metal matrix composites (MMCs) remains challenging due to the strong van der Waals attraction between nanotubes, which promotes agglomeration. To overcome this issue, various dispersion techniques such as ball milling, stir casting, ultrasonic dispersion, surfactant-assisted processing, and chemical coating methods have been explored. Despite these efforts, substantial improvement in CNT dispersion within MMCs has not been consistently achieved, highlighting the need for more efficient and reliable processing techniques[2,5]. Friction stir processing (FSP), derived from friction stir welding (FSW), has been recognized as an effective solid-state method for achieving microstructural refinement and homogeneous distribution of reinforcement particles in composites[6].

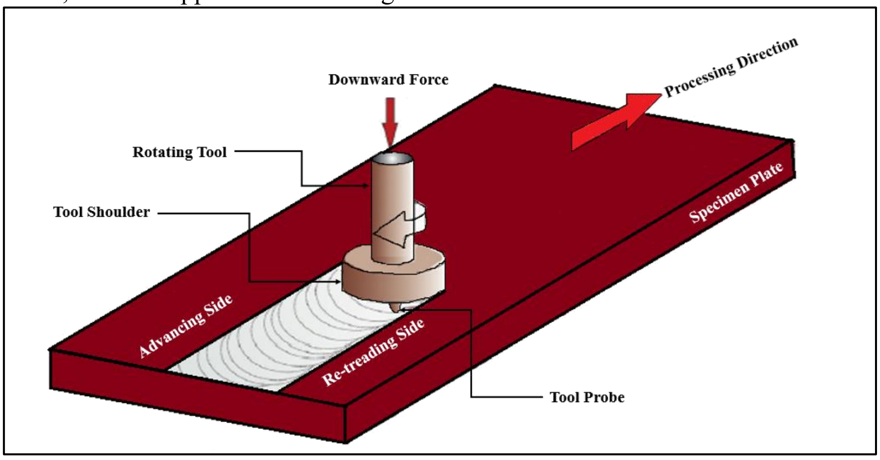

Fig. 1: schematic representation of FSP process.

FSP is a solid-state surface modification technique derived from friction stir welding (FSW), which was originally developed by The Welding Institute (TWI)[6]. During both FSW and FSP, significant microstructural evolution occurs within the stir zone, as shown in Figure 1. Under controlled processing conditions, this evolution promotes substantial grain refinement, thereby improving material properties[7]. From a metallurgical perspective, FSP can be considered a thermo-mechanical treatment capable of tailoring the surface microstructure of metals and alloys. Moreover, FSP can be employed to fabricate surface composites by incorporating secondary-phase reinforcement particles into the stir zone. The process offers several advantages, including refined microstructure, homogeneous particle distribution, solid-state processing, environmental friendliness, and high energy efficiency. Owing to these benefits, FSP has been widely applied to various metallic materials, particularly aluminum and magnesium alloys.

Several studies on the FSP of AZ31 magnesium alloy have focused on microstructural evolution and the fabrication of various surface composites. Morisada et al.[8] fabricated AZ31/SiC surface composites using FSP and reported that the incorporation of SiC particles promoted grain refinement, increased hardness, and enhanced microstructural stability at elevated temperatures (~400 °C). In a subsequent study, the same researchers demonstrated that the distribution of multi-walled carbon nanotubes (MWCNTs) through FSP resulted in microstructural refinement and increased microhardness of the AZ31 alloy[9]. Huang et al.[10] reported the formation of an ultrafine-grained structure in AZ31 magnesium alloy processed by FSP. Similarly, Azizieh et al.[11] fabricated AZ31/Al_2O_3 surface composites using FSP and investigated the influence of tool rotational speed and pin profile on microstructural evolution and particle distribution. Further studies by Mishra et al.[12] on AZ31/CNT nanocomposites produced via FSP revealed significant improvements in hardness and tensile properties. They also observed that repeated FSP passes in the presence of CNT nanoparticles grain refinement occurs in surface composite. Sharma et al.[13] employed a hole-filling technique to introduce MWCNTs and graphene into the AZ31 magnesium matrix. The processed composite exhibited an approximately 130% increase in microhardness, which was primarily attributed to grain boundary refinement and the effective pinning action of the reinforcements.

Based on the available literature, grain formation during FSP is strongly influenced by the combined effect of tool rotational speed and traverse speed, as these parameters govern the heat input and thermal exposure duration during processing. Therefore, selecting the optimum rotational and traverse speeds to achieve superior composite properties remains challenging. In this context, the speed ratio (rotational speed/traverse speed) or revolutionary pitch (RP) (traverse speed/rotational speed) becomes an important parameter, as it represents the combined influence of both processing variables. This parameter controls the heat input, material flow behavior, and degree of plastic deformation, all of which significantly affect grain refinement and the overall microstructural characteristics of the processed zone.

In the present study, the influence of RP, along with a straight-direction pass (SDP) performed during two consecutive passes, on the microstructural and mechanical properties of the fabricated AZ31/MWCNT surface composite was investigated. The resulting variations in composite properties were analyzed in detail, along with the underlying mechanisms responsible for the observed improvements.

II. Experimental Details

A. Materials

The experiments were conducted on rolled AZ31 magnesium alloy plates with dimensions of 100 × 100 × 6 mm³. The chemical composition and mechanical properties of the AZ31 alloy are presented in Table 1. Multi-walled carbon nanotube (MWCNT) nanopowder was used as the reinforcement material, possessing a nominal diameter of 10–20 nm and an average length of approximately 10 µm. As shown in Fig. 3(a), the MWCNTs exhibit a highly entangled morphology with a large aspect ratio.

Mechanical characteristics			Chemical composition						
Hardness (HV)	Tensile strength (Mpa)	Elongation (%)	Al	Zn	Si	Cu	Mn	Fe	Mg
50	248	13.28	2.8	0.8	0.05	0.0045	0.42	0.011	95.5

Table 1: Properties of AZ31 material.

B. Experimental procedure

Surface composite fabrication using FSP follows the steps mentioned in Figure 2. In the initial stage, grooves with dimensions of 1 mm in width and 3 mm in depth were machined into the plates using a vertical milling machine, as illustrated in Figure 3(b). In the subsequent step, the MWCNT powder was mixed with methanol and packed into the prepared groove cavity, maintaining a constant volume fraction of 18.5%, as calculated from Equations (1–3). The volume fraction of the MWCNT reinforcement was determined using the following relation[14].

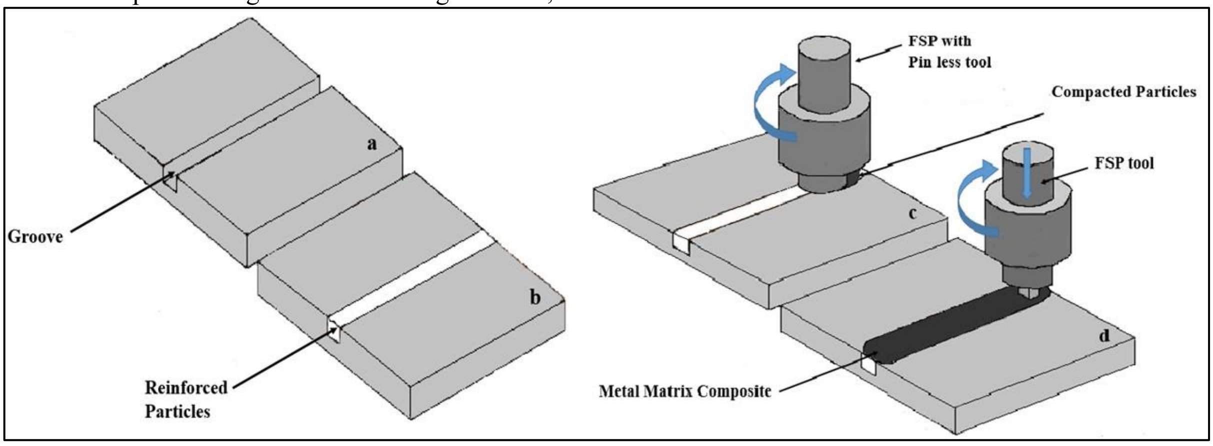

Fig. 2: Schematic illustration of the fabrication procedure

of AZ31/MWCNT surface composite using friction stir processing: (a) groove preparation on AZ31 plate, (b) filling of reinforced MWCNT particles into the groove, (c) groove sealing using a pinless FSP tool, and (d) fabrication of surface composite using the FSP tool.

$$Area\ of\ groove(Ag) = width \times depth \quad (1)$$

$$Projected\ area\ of\ pin(Ap) = pin\ dia \times pin\ length \quad (2)$$

$$Volume\ of\ fraction\ of\ the\ reinforcement(Vf) = \frac{Ag \times\ pin\ length\ advanceing\ distance}{Ap} \quad (3)$$

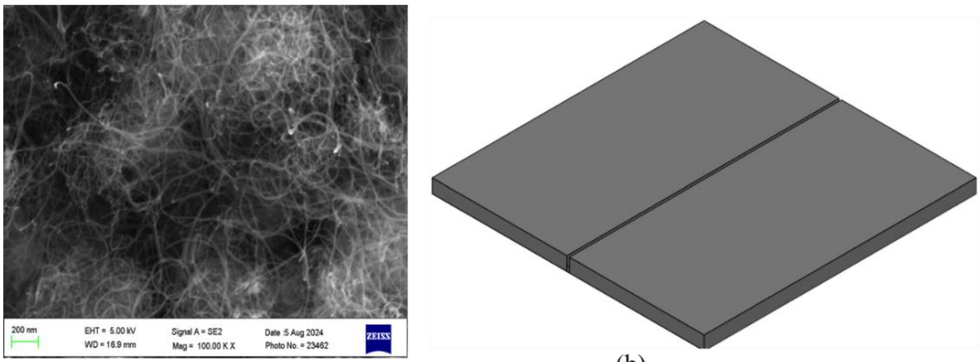

Fig. 3: (a) SEM of MWCNT powder, (b) Groove on AZ31 plate

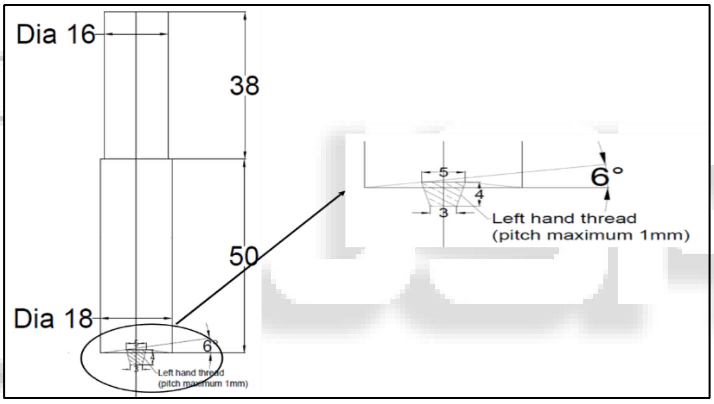

Fig. 4: configuration of H13 tool, featuring a conical threaded pin with a concave shoulder.

In the third stage, the MWCNT-filled grooves were sealed to prevent particle expulsion during processing. For this purpose, a cylindrical pinless tool made of hardened H13 steel with an 18 mm shoulder diameter was employed to perform the capping pass. In the fourth stage, after successful sealing of the grooves, the stirring operation was carried out using a tool with an 18 mm shoulder diameter, a 6° concave shoulder profile, and a 4 mm-long conical pin with left-hand threads, as shown in Fig. 1(c). The surface composites were fabricated using one capping pass followed by three stirring passes. All stirring passes were performed using the SDP approach with a tool tilt angle of 3°, while the remaining FSP parameters are summarized in Table 2. The entire experimental procedure was conducted using a specially designed 3-axis automated universal milling machine.

Sample id	FSP Pass direction	Tool rotational speed (rpm)	Tool traverse speed (mm/min)	RP (mm/rev)
A	SDP	545	31.5	0.05
B		765	78	0.1

Table 2: Processing parameters for the FSP.

C. *Testing procedure*

Specimens were cut using electrical discharge machining (EDM) to investigate microstructural and mechanical properties. Microstructural sample preparation was performed using standard metallographic procedures, including grinding, polishing, and etching. The etchant solution consisted of 70 ml of ethanol, 10 ml of distilled water, 10 ml of acetic acid, and 4.2 g of picric acid. The microstructure of the stir zone was examined by Optical microscopy (OM). The microhardness of the fabricated surface composites was measured using a Vickers hardness tester under a load of 100 g with a dwell time of 10 s. The indentations were made at a depth of 1 mm below the top surface with an interval spacing of 0.5 mm across the processed zone. Tensile testing was performed using a universal testing machine (UTM) in accordance with ASTM

E08 standards at a crosshead speed of 0.5 mm/min to evaluate the mechanical behavior of the fabricated composites.

III. RESULT AND DISCUSSIONS

A. Microstructure Studies

	**Sample-A** Groove 545 rpm / 31.5 mm/min SDP
	**Sample-B** Groove 765 rpm / 78 mm/min SDP

Fig. 5: Surface Appearance of the fabricated surface composite using FSP

The surface appearance of the FSPed AZ31/MWCNT surface composites is shown in Figure 5. Both processed specimens exhibited a smooth, continuous surface without visible cracks, tunnel defects, voids, or flash formation, indicating successful fabrication of defect-free surface composites. The formation of regular surface ripples along the processing direction confirms stable material flow during FSP.

The specimen processed under lower RP conditions exhibited finer, more uniform surface ripples, suggesting improved plasticised material flow and enhanced stirring action. In contrast, the higher RP showed relatively irregular flow marks, indicating comparatively non-uniform material transportation within the SZ. The smoother surface morphology obtained in the lower RP condition can be attributed to adequate heat generation and effective consolidation of the material around the rotating tool.

The absence of severe surface defects further indicates that the selected process parameters generated sufficient frictional heat to soften the AZ31 alloy and facilitate proper incorporation of MWCNTs into the matrix. Moreover, the repeated stirring action during FSP promoted better closure of the groove region and minimized defect formation.

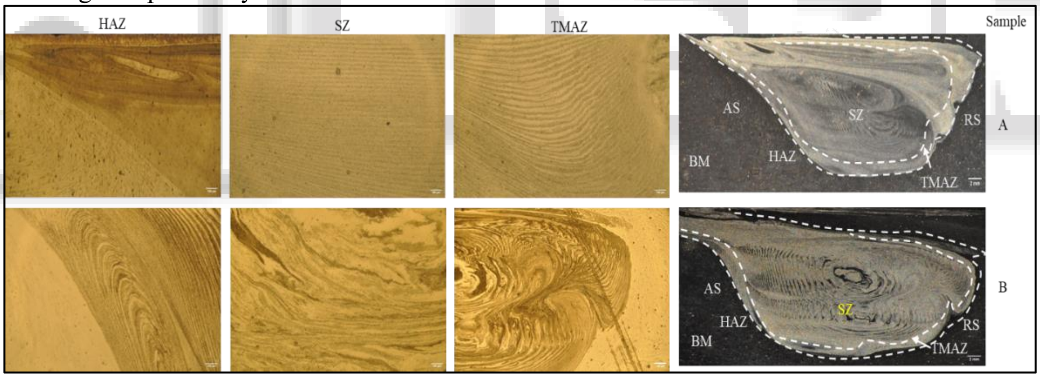

Fig. 6: Microstructure of SZ shows material flow and distribution (a) Sample A, (b) Sample B.

The macrostructural analysis of the AZ31/MWCNT surface composites revealed distinct regions: SZ, thermo-mechanically affected zone (TMAZ), heat-affected zone (HAZ), and base material (BM) as illustrated in Figure 6. A defect-free stir zone without tunnel defects, voids, or cracks was successfully produced under the selected processing conditions, indicating sufficient heat input and effective material consolidation during FSP. The SZ exhibited characteristic onion-ring patterns and vortex-like flow structures, confirming intense plastic deformation and cyclic material flow generated by the rotating tool. Compared with the higher RP condition, the lower RP condition exhibited a more homogeneous and symmetric stir zone with smoother flow contours, indicating improved material transport and enhanced MWCNT dispersion within the matrix.

Optical micrographs further revealed significant grain refinement in the SZ, attributable to severe plastic deformation and dynamic recrystallisation during processing. The repeated stirring action fragmented the material layers and promoted uniform redistribution of MWCNTs throughout the processed region. In addition, the presence of MWCNTs restricted grain growth via a grain-boundary pinning effect, resulting in a finer, more stable microstructure. The asymmetric flow behaviour between the advancing side (AS) and retreating side (RS) observed in the processed region is typical of FSP magnesium alloys and reflects the difference in heat generation and deformation characteristics on both sides of the tool. Overall, the refined and homogeneous microstructure confirms the effective incorporation of reinforcement and improved material flow behaviour during FSP, which is expected to contribute positively to the mechanical performance of the AZ31/MWCNT surface composite.

B. Mechanical Properties

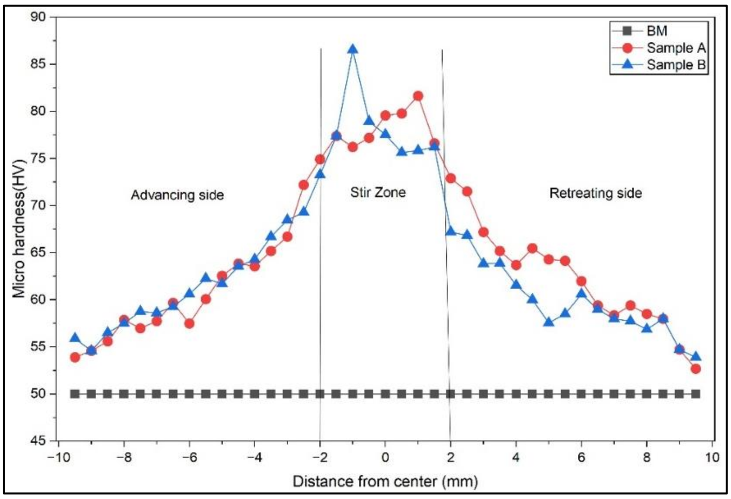

Fig. 7: Microhardness distribution in SZ of fabricated surface composites.

Figure 7 shows the microhardness distribution across the transverse cross-section of the AZ31/MWCNT surface composites from the advancing side (AS) to the retreating side (RS). The base material (BM) exhibited a nearly constant hardness value of about 50 HV throughout the measured region, whereas both processed samples demonstrated a significant increase in hardness within the stir zone (SZ). The hardness gradually increased from the AS toward the SZ, reached a maximum near the center of the processed region, and then decreased toward the RS. Sample B exhibited the highest peak hardness of approximately 86 HV, while Sample A showed a maximum hardness of nearly 82 HV.

The enhanced hardness in the SZ is mainly attributed to severe plastic deformation and dynamic recrystallization during friction stir processing, which resulted in substantial grain refinement. In addition, the incorporation and dispersion of MWCNTs within the AZ31 matrix contributed to strengthening through grain boundary pinning, dislocation generation, and load transfer mechanisms. The comparatively higher hardness observed in Sample B indicates more effective material flow and improved reinforcement distribution under optimized processing conditions. The slight reduction in hardness toward the AS and RS regions is associated with lower deformation intensity and partial thermal softening outside the stir zone. Overall, the hardness profile confirms successful microstructural modification and strengthening of the AZ31/MWCNT surface composites after friction stir processing.

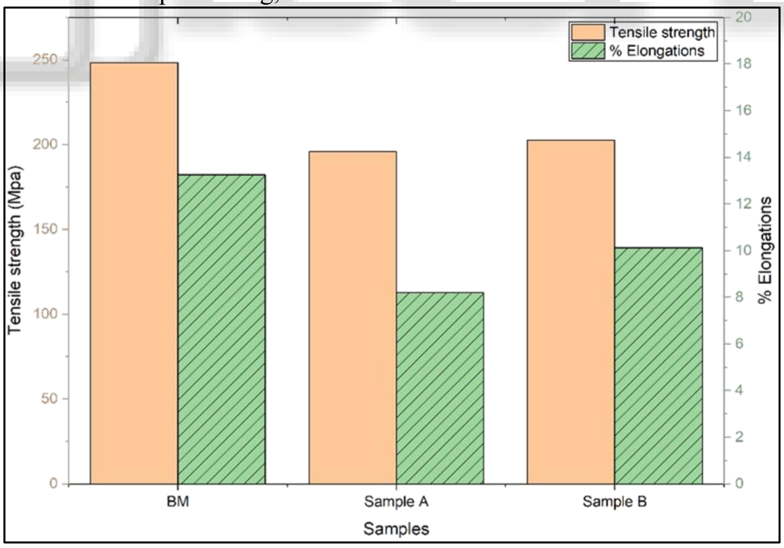

Fig. 8: Tensile strength and % elongation of fabricated surface composites.

Figure 8 illustrates the tensile strength and percentage elongation of the base material (BM) and the fabricated AZ31/MWCNT surface composites. The BM exhibited the highest tensile strength of approximately 248 MPa along with an elongation of nearly 13%, indicating superior ductility compared to the processed samples. After friction stir processing and incorporation of MWCNTs, both Sample A and Sample B showed a reduction in tensile strength and elongation. Sample A exhibited the lowest tensile strength (~195 MPa) and elongation (~8%), whereas Sample B demonstrated comparatively improved mechanical performance with a tensile strength of approximately 202 MPa and elongation close to 10%.

The reduction in tensile properties after processing may be attributed to localized reinforcement agglomeration, interfacial discontinuities, and the formation of residual stresses during friction stir processing. However, the comparatively higher strength and ductility of Sample B suggest improved material flow and more uniform dispersion of MWCNTs within the stir zone. The enhanced performance of Sample B may also be associated with better consolidation and a more homogeneous refined microstructure produced under optimized processing conditions. Although the tensile properties of the composites remained lower than the BM, the processed samples still exhibited reasonable ductility, indicating successful composite formation without severe brittleness.

IV. Conclusion

The study successfully fabricated AZ31/MWCNT surface composites via FSP under different RP conditions, demonstrating the significant impact of RP on microstructural evolution and mechanical properties. The composites exhibited defect-free surfaces with uniform MWCNT distribution and notable grain refinement, particularly at lower RP values, thereby enhancing material flow and promoting homogeneous microstructures. Microhardness improved substantially due to grain boundary strengthening and effective reinforcement dispersion. Although tensile strength and elongation decreased relative to the base material, likely due to reinforcement agglomeration and residual stresses, the sample processed at higher RP showed comparatively better mechanical performance, indicating that optimised processing parameters improve reinforcement distribution and composite consolidation. These findings confirm that controlling RP during FSP is crucial for tailoring the microstructure and mechanical behaviour of AZ31/MWCNT surface composites, enabling the development of advanced lightweight materials with enhanced surface properties.

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