



Surface Modification on Magnesium Alloys' Hardness and Microstructure Using Friction Stir Processing – A Review

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Abstract

Low density of magnesium-based alloy is one potential as the lightest structural material for light weight-high strength applications for automotive and aerospace. Severe plastic deformation (SPD) together with thermomechanical processing are proved to be a successful method for attaining desired microstructural modifications through achieving fine and highly misoriented microstructures and creating various structures to the bulk properties of magnesium alloy. The material's deformation can result in an altered microstructure that is gainful to the material's requirements. However, the poor deformability of magnesium and its alloys limits the application of the thermomechanical approach. Controlling over temperature and deformation rate is hard to achieve. Among the thermomechanical processes, friction stir processing (FSP) offers an easy way to achieve process stability and mechanical properties enhancement by heat treatment which results in the closure of porosity and refined grain size. During this process, heat is generated by the rotation of the FSP processing tool. Few process parameters such as rotational and traverse speeds should be controlled to make FSP stay within the defined processing condition. It is critical to set the right tool rotational speed as well as traverse speed to ensure adequate heat generation. As there are no established standards for operating the FSP, the only solution is to experiment with different settings to find the best parameter which will produce better quality on processed magnesium alloy workpiece. This paper explores earlier studies on surface modification via FSP technique to improve the mechanical properties strengthening of magnesium alloy mainly on grain size and hardness. The surface modification was done mostly on popular series of magnesium alloy (AZ series) using different tool material, tool geometry and different parameters combination. A comprehensive view of surface modification on magnesium alloys which includes the FSP tool and workpiece material used, variations of FSP parameters settings as well as the effect on hardness and microstructure analysis will be discussed.

Keywords: Friction Stir Processing, Surface Modification, Magnesium Alloys, Hardness, Microstructure

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1. Introduction

The advancement of Friction Stir Welding (FSW) is expanded by Friction Stir Processing (FSP). It is a source of an innovative solid-state joining technique that was developed by Wayne Thomas from The Welding Institute (TWI Ltd.), Cambridge in 1991 [1]. To create surface composites and alter the surfaces of metallic plates and sheets, friction surfacing and FSP are used.

FSP is a newly developed technology that uses no consumables and emits no gases or waste during the process. In regards, no harmful smoke or fumes are produced during or after the process [2], which is why FSW is known as "green technology" since it is environmental friendly, versatile also energy-efficient [3], [4].

Friction stir processing is a one-stage process that is primarily used to create fine-grained structure, strengthen and alter microstructure in close surface layers of processed metallic material without any

changes in its size and shape [5]. During FSP, dynamic recrystallization and grain boundary pinning improve grain refinement [6]. It is executed at temperatures lower than the melting point of base alloys with low melting points [7], [8]. This technology, in particular, can alloy the categorical components, improve surface or bulk composite production, microstructure homogenization and repair cracks [9].

1.1. Friction Stir Processing Principle

Friction stir processing technology is employed at lower temperatures below melting point of base alloys. With the exception of FSW which is designed to join dissimilar solid materials, FSP modifies the alloy microstructure locally to attain its specific properties.

During the process, a rotating pin is plunged into the material under axial force from the shoulder of the FSP tool. The friction between the tool shoulder and the material generates a lot of heat as the FSP tool moves along the workpiece in the traverse path. In response to the localised heating, the material in the processed area softens before reaching its melting point and undergoes

severe plastic deformation as the pin rotates. The material will flow from the retreating side (RS) to the advancing side (AS). In a nutshell, AS is the position where the rotational tool surface is parallel to the traverse direction whereas RS is the position where the rotational tool surface is not concurrent to the traverse direction [10].

1.2. Magnesium alloy AZ Series

The most popular alloy series is the AZ series for magnesium alloy. Aluminium and zinc are the primary constituent elements of the magnesium alloys in the AZ series. Through alloying the magnesium alloy with rare earth elements plus other purifying methods, the deterioration of the alloy could be controlled. Manganese were incorporated to the AZ series of magnesium alloys to create a biodegradable alloy [11]. AZ91 is a widely recognized magnesium alloy. Aluminium makes up 9% of the weight of this magnesium alloy whereas zinc makes up 1%.

Comparatively to its rivals, non-ferrous metals like titanium alloys and aluminium alloys which have higher strengths than magnesium alloys, the use of AZ91 in various industries is still limited [12]. Additionally, it was partly brought on by the challenge of controlling its microstructure. As it produces relatively poor mechanical properties including their low elastic modulus, low hardness and low wear resistance, this specific alloy system is rarely utilized in its as-cast state [13]. Regrettably, the porosity and inter-dendritic regions that are frequently present in cast alloys lead to a decline in performance as a result of the mechanical properties degrading [14].

2. Research Method

2.1. FSP Process Parameter

In order to test the effects of FSP, Babu et al. [15] varied three traverse speeds: 20, 30, and 40 mm/min for two particular rotational speeds: 600 and 800 rpm. The grain refinement of FSPed AZ31 magnesium alloys can enhance their mechanical properties, making them suitable for superplastic forming. Results show that the rotational and traverse speed combination of 800 rpm and 40 mm/min, the smallest precipitate size is obtained. In comparison to base metal, the corresponding hardness value is about 50% higher at 63 HV. It is discovered that as traverse speed increases, fine precipitates are produced. This is because more precipitate particles are distributed at high traverse speeds than at low traverse speeds. Besides this, it has been encountered that high rotational speeds result in smaller precipitates. More heat input results from high temperatures caused by high rotational speed.

The three primary categories of processing variables that are involved during FSP (Figure 1).

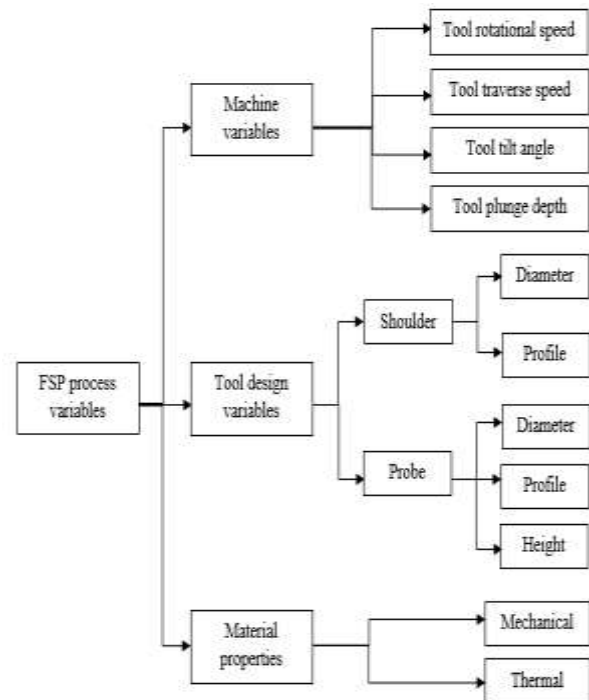


Figure 1. Classification of Friction Stir Process Variables

The FSP tool parameters are crucial in gaining the underlying mechanical properties. Related to FSP, it is simple to control the microstructure and mechanical properties by adjusting the process parameters. Tool rotational speed (w) and tool traverse speed (v) are considered machine variables. During FSP, tool design variables: pin also shoulder diameter certified as the crucial criterion of heat generation on the workpiece [16], [17]. In the defined range of microstructure in magnesium alloys, tool pin profile and tool geometry have a major impact in enhancing the material strength or adaptability [18]. The development of the grain structure as well as subsequent mechanical performance of the stirred zones were identified to be critically dependent on the heat input index ratio in terms of the w/v ratio [19].

The evolution of the microstructure was examined at various temperatures. After multi-pass of FSP on as-cast AZ61 alloy, notable grain refinement with an average grain size of $7.8 \pm 6.4 \mu\text{m}$ was attained. The fraction of coarse grains ($> 10 \mu\text{m}$) was above 20%, but the FSP plate's microstructure was inhomogeneous. The inferior ductility of the FSP plate is an outcome of inhomogeneous deformation resulted by the non-uniformity of microstructures. The key challenges to enhance the Mg plates's properties whether at a high temperatures or room temperature seemed to be microstructural inhomogeneity. Further research on overlapping process optimization and controlling heat source during FSP is required for further grain refinement and homogenization [20].

The FSP strategy altered the grain size, microstructure and mechanical properties of the Mg AZ91 alloy. Corrosion resistance was primarily improved by grain refinement with the increment in tool traverse and rotational speed. The resulted fine grains formed in the stirred zone is caused by dynamic recrystallization. The best result of FSPed sample was by using 1250 rpm and 30 mm/min. FSP refined the microstructure of the as-received alloy from 30 ± 9 mm to 16 ± 7.8 mm average grain size. The grain size can be controlled by acknowledging both the grain growth as well as metallurgical development of recrystallization. The existence of these two is primarily caused by the tool's rotational speed conquering higher temperatures. Severe plastic deformation caused significant morphological changes in β phase particles during FSP. The FSPed samples' hardness value increased as the β phase precipitates developed. The removal of casting defects in FSPed Mg AZ91 alloy rising the hardness value. The reduced corrosion rate of Mg AZ91 alloy was primarily because of grain refinement plus hardness increment. Further grain saturation after FSP improved the microhardness of the Mg AZ91 alloy [21].

Luo et al. [22] studied the influence of processing parameters on microstructural texture and evolution as well as mechanical behaviour. The findings demonstrate that FSP can produce fine-grained Mg AZ61 alloy via dynamic recrystallization. Subsequently, the β -Mg₁₇Al₁₂ phase network that existed along the grain boundaries in the base metal of AZ61 cast Mg alloy breaks up then dissolved into the matrix as an outcome of the high temperatures and fragmentation effect combined together during FSP. The 800-240 specimen consists the finest grains (6.5 μ m) at a low rotation rate to traverse speed (ω/v) ratio. An increased of grain size to 12.5 μ m for 1000-60 specimen was noticed when ω/v increased, as did the proportion of high-angle grain boundaries (HAGBs). The FSP specimens' microhardness and tensile properties were drastically boosted when compared to the initial cast alloy (54 HV) because of grain refinement. The average hardness in the SZ for the 800-240, 800-60 and 1000-60 specimens is 73, 69 and 67 HV, respectively. FSP specimens' high strength plus ductility were acquired primarily attributable to the effective grain refinement, dispersed distribution of crystallographic texture as well as a higher fraction of HAGBs.

The variables used in friction stir processing magnesium alloys including machining parameters, workpiece, tool and geometries (Table 1).

Table 1. FSP Variables on Magnesium Alloys

Workpiece/ Tool	Tool Geometry	Rotational Speed (rpm)/Traverse Speed (mm/min)
AZ80/ W18Cr4V	SD: 15 mm PD: 5 mm PL: 3 mm	RS: 375 TS: 118
AZ31 (150x150x4) mm/ High speed steel (HSS)	SD: 18 mm PD: 6 mm	RS: 600, 800 TS: 20, 30, 40
AZ61 (6 mm)	SD: 18 mm PD: 7 mm PL: 5 mm	RS: 1000 TS: 60
AZ91/ High- strength Mo- based alloy	SD: 16 mm PD: 6 mm (b), 4 mm (t) PL: 4.7 mm	RS: 600 TS: 30
AZ31B/ H13 steel	SD: 20 mm PD: 5 mm	RS: 600, 800, 1000, 1200
Magnesium alloy/ H13 steel	SD: 19.95 mm PL: 3.50 mm PD: 7.22 mm	RS: 1500 TS: 0.10 mm/rev
AZ91/ High carbon high chromium steel	SD: 18 mm PL: 3 mm PD: 6 mm	RS: 1100-1400 TS: 30-60

*SD: shoulder diameter; PD: pin diameter; b: bottom; t: top; PL: pin length

3. Result and Discussion

3.1. Effect on HAZ, TMAZ and Stir Zone

At a tool rotational speed and traverse speed of 1400 rpm and 25 mm/min, respectively, a defect-free friction stir of magnesium AZ61 alloy was produced. During processing via FSW, the Mg alloy's elongated grains recrystallized in the stir zone and thermo-mechanically affected zone (TMAZ). In consequence of dynamic recrystallization, fine and equiaxed grains were formed in the stir zone. The grains were observed to be finer on the retreating side of TMAZ than on the advancing side. Hardness improves because of the development of finer grains in the SZ. The study found that the BM: 70 HV has a higher microhardness than the TMAZ but a lower microhardness than the SZ: 81 HV. The experiment also disclosed that the width of the friction stir zone was about 6 mm which equivalent to the tool pin diameter. Furthermore, the advancing and retreating sides of the processed regions have minimum hardness values around 67-69 HV which are lesser compared to the BM hardness number [23].

In AZ31, Ugender [24] analysed the impact of rotational speed on the defects formation of a friction stir processed zone. Rotational speed under 900 rpm forms a worm hole defect because of lack of heat generation and inadequate metal filling while a tunnel

defect is discovered because of excess heat source generated using rotational speed greater than 1400 rpm. Pin holes are spotted with the usage of traverse speed under 25 mm/min because of elevated heat generation. On the other hand, a tunnel defect is found because of lack of heat source due to insufficient metal flow with the usage of traverse speed is above 40 mm/min. The enhanced hardness of the stir zone can be attributed to grain refinement as it is essential in material strengthening since the grain of the SZ is much smaller compared to the BM. The microhardness decreases after further increase of the rotational speed. This is caused by elevated heat source that induces material softening thus reduces microhardness. The stirred zone softened in the magnesium alloys owing to the coarsening and dissolution of strengthening precipitates.

Sevvel et al. [25] examined the significance of optimized process parameters on AZ31B. When using a rotational speed and traverse speed of 500 rpm and 3 mm/min, respectively, coarse grains were formed resulting in tunnel defects and worm holes. Moreover, the optimized process parameter values at 1000 rpm and 0.5 mm/min confirmed that the stirred zone microstructure is finely divided and that grain fragmentation happened appropriately. Plastic flow is also evidenced in the processed regions with uniform grain orientation. Sufficient duration is needed for the grains to deform uniformly which it is only possible at lower feed rates. Further to that, strain hardening factors leading to dislocated stacking up of slipped planes are possible at lower tool rotational speeds. Using a large rotational speed but small traverse speed, sufficient heat is generated leading to a rapid solidification of the grains which can be obtained during processing AZ31B Mg alloys.

3.2. Effect of Friction Stir Processing on Mechanical Properties and Hardness

After a single pass of FSP, fine precipitates of aluminium and magnesium were produced at the grain boundaries. These precipitates build up at the grain boundaries and pin them. As seen in (Figure 2), succeeding passes led to the existence of precipitates at the grain boundaries then, ultrafine grains were formed. The evolution of ultra-fine grain structure is divided into three stages: 1) continuous dynamic recrystallization, 2) discontinuous dynamic recrystallization as well as 3) grain growth limitation via rapid cooling [26].

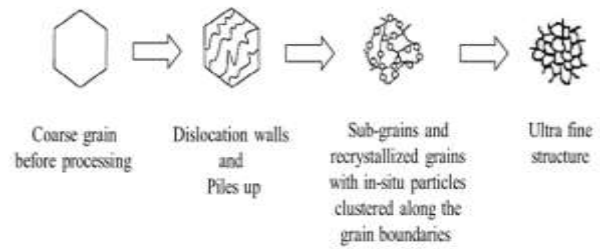


Figure 2. The *in-situ* Particles Affecting the Evolution of Ultrafine Structure

Impact of FSP parameters and tool profile on the microstructure properties of AZ80A were investigated [27]. The micrographs revealed that the base metal has inhomogeneous distribution of intermetallic compounds and the particles are rather coarse. The outcomes in the stir zone positively demonstrate that big-sized coarse grains in the BM: 29 μm were reshaped into equidistant small-sized grains: 8 μm after FSP.

A report by Iwaszko et al. [28] stated that recrystallization caused by plastic strain and also frictional heat during processing results in very fine and equiaxed grains of 2-10 μm . In the FSP trials at 3,500 rpm and 4,500 rpm, the average grain size in the stir zone was about 6 μm and 9 μm , respectively. The existence of highly elongated grains distributed along the flow line in the TMAZ implies that dynamic recrystallization failed to take place. The as-received and FSPed zones had average hardness of $58 \pm 5 \text{ HV}_{0.05}$ and $77 \pm 10 \text{ HV}_{0.05}$, respectively. The maximum microhardness was noticed with the usage of tool rotational speed: 3,500 rpm rather than 4,500 rpm. Increasing the rotational speed causes less structure refinement and weakened the material. This matter should be associated with variations in grain size resulting from varied tool rotational speeds, alongside the onset of temperature variations in the material through friction modification. The temperature is highest at the surface, allowing grain growth to accelerate.

Zhen et al. [29] looked into the impact of various rotation rates on the cladding layers of an AZ91 magnesium alloy subjected to FSP. Furrow defect is visible after FSP at a rotation rate of 100 r/min. The refined grains in the stirred zone plus the existing long strip second phases were crushed and dispersed into the α -Mg matrix, leading in fine-grain strengthening as well as second-phase dispersion strengthening. The investigation additionally demonstrated the absence of a statistically considerable difference in the distribution of the second phases under varying rotational rates. When compared to the before FSP, the average grain size for each parameter was refined. This is a result of the combined effects of fine-grain strengthening and second phase dispersion strengthening. The average microhardness of the

stirred zone before FSP was 73.2 HV 0.1 and it markedly improved after FSP based on the researchers' analyses. With each increase in rotation rate, the average microhardness first ascended and then dropped.

FSPed AZ80 magnesium alloy's mechanical properties, static and electrochemical corrosion, stress corrosion cracking behaviour as well as microstructure were studied using a cylindrical stir tool with shoulder diameter: 15 mm, cylindrical pin diameter: 5 mm and pin length: 3 mm. The study found that FSP produced a consolidation of fine and coarse grains in the specimens with an average grain size (7.1 μm). In the stirred zone, the specimen's average microhardness was 69.4 HV. The texture softening effect sped up the emergence of pitting corrosion leads to lower performance of stress corrosion cracking [30].

The outcome of microstructural factors on corrosion resistance of as-cast and FSPed AZ91 Mg samples also discussed. The FSP approach was capable of substantially alter the Mg AZ91 alloy's microstructure, together with notable refinement of grain size (~1.5 μm), favourable grain orientations and also the formation of uniformly distributed fine β-Mg₁₇Al₁₂ phase. The FSPed AZ91 Mg alloy has a relatively homogenous microstructure with no observable dendritic β phase. The β phase was clearly fragmented into a large fraction of finer particles and evenly dispersed into the α-Mg matrix. This remarkable morphological adaptation of β phase is principally caused by the severe plastic deformation that occurs during FSP [31].

The effects of rotational and traverse speeds on the Mg AZ91 alloy's crystallographic texture, microstructural evolution and also mechanical properties were explored [32]. In the study, the tool rotational and traverse speeds applied were 400, 800, 1200 rpm and 20, 40, 60 mm/min, respectively. In response to the occurrence of dynamic recrystallization (DRX), the average grain size was significantly reduced from 61.6 μm (as-cast condition) to far less than 10 μm (FSPed samples). The grain size was decreased by lowering the rotational speed but raising the tool traverse speed. The fine particles at the grain boundaries restrain grain growth. After the FSP, the fraction of β-Mg₁₇Al₁₂ markedly reduced as a result of fragmentation and dissolution. When associated with its higher peak temperatures, the 800-40 and 1200-40 samples had a high strain rate that accelerated the dissolution of β-Mg₁₇Al₁₂ throughout FSP. Given its extremely small grains (1.1 μm), the 800-20 sample had the maximum microhardness (104.4 HV).

The influence of FSP parameters in enhancing the mechanical properties and the microstructure of the FSPed magnesium alloys are summarized in (Table 2).

Table 2. Summary Reviews on Tool FSP Parameters, Defects, Grain Size and Hardness Improvement of Magnesium Alloys

Workpiece/ Tool	Rotational Speed (rpm)/Traverse Speed (mm/min)	Findings
AZ61/ H13	RS: 1400 TS: 25	<ul style="list-style-type: none"> •Friction stir zone's width was about 6 mm which equal to pin's diameter •BM: 70 HV [higher than TMAZ] •SZ: 81 HV
AZ31	RS: 900, 1120 and 1400 TS: 25-75 Optimum: 1120, 40	<ul style="list-style-type: none"> <900 rpm: worm hole >1400 rpm: tunnel defect <25 mm/min: pin hole >40 mm/min: tunnel defect
AZ31B/ M35 HSS	RS: 45-1500 TS: 0.25-500 Optimum: 1000, 0.5	Formation of coarse grains leading to tunnel defects and worm holes when using 500 rpm and 3 mm/min.
AZ31/H13	RS: 2000 TS: 40.50	<ul style="list-style-type: none"> •Tunnel defect increases in all specimens as the welding towards the retreating side. •2000 rpm, 40 mm/min: Tunnel defect •2000 rpm, 50 mm/min: Excessive flash
AZ80A/M3 5 HSS	RS: 750 TS: 75	29 to 8 μm
AM60/H13	RS: 3500-4500 TS: 16 mm/min Optimum: 4000 rpm	<ul style="list-style-type: none"> •In TMAZ, highly elongated grains distributed along flow line. •6-9 μm •BM: 58±5 HV0.05 •SZ: 77±10 HV0.05
AZ91	RS: 200, 300, 500, 800, 1000, 1200 TS: 60	<ul style="list-style-type: none"> •3.4, 5.6, 7.4, 11.8, 14.6, 14.9 μm •82.4, 85.6, 84.1, 83.9, 80.9, 80.7 HV •100 rpm: Furrow defect
AZ91/H13	RS: 400, 800, 1200 TS: 20, 40, 60	800-20 sample has 104.4 HV due to its very fine grains (1.1 μm)

4. Conclusion

Magnesium alloys are widely used in diverse industries. However, there are some limitations. To meet each of the requirements, numerous strategies were adopted to upgrade the microstructure and mechanical properties of this alloy. Each technique, however, has advantages and disadvantages. FSP approach is preferred by many researchers in boosting the magnesium alloys' properties. The microstructure and mechanical properties of the material will be modified because of the deformation and temperature faced by material during the process. A literature review of machine variable, microstructure and mechanical properties in FSP was described in this paper in order to design the future proposed FSP research. The highest priority ought to be given to the machine variable being investigated as it is required during FSP to attain the superior performance of microstructural and mechanical properties of magnesium AZ91 alloy.

Acknowledgements

The authors gratefully appreciate Universiti Malaysia Pahang, the Ministry of Higher Education Malaysia and the Ministry of Science, Technology and Innovation (MOSTI) for providing support in technical and financial aspects through the Fundamental Research Grant Scheme FRGS/1/2019/TK03/UMP/02/17 (RDU 1901140). Additionally the authors would like to extend appreciations to Universitas Putra Indonesia YPTK Padang for providing supports for this publication.

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