The Effect of Rapid Deformation Process to Improve Creep and Tensile Resistance of AZ91 Magnesium Alloy Plates

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1. INTRODUCTION

Among the magnesium alloys, the AZ family has excellent properties such as castability at room temperature. However, Mg17Al12 particles with the low melting point (120 °C) on the rough grain boundaries and in the dendritic regions reduce the mechanical properties of these alloys at high temperatures. Friction stir processing (FSP) is a process to improve the tribological and microstructural properties of metallic materials. During FSP, sufficient heat is created to soften the material without melting which causes dynamic recrystallization of rough grains. In fact, due to the mechanical turbulence and stirring action using a rotating tool, the high strain rate deformation occurs. Therefore, FSP should cause a homogenous microstructure, breakdown of the grains, decrease the size of Mg17Al12 eutectic particles, distribute the particles on the grain boundaries, reduce the casting defects and hence improve the mechanical properties [1]. However, the improvement of properties after FSP is highly dependent on the process parameters such as tool rotational speed, traverse speed, axial force, tool dimension and tool tilt angle. Recently, several works attempted to develop the performance of this process and increase the mechanical properties via FSP.

In recent years, many researchers [2-7] have demonstrated the effect of single-pass FSP on the mechanical properties of different alloys, especially cast...
alloys. Buffa et al. [8] reported the effect of water-cooling FSP on microstructure and tensile properties of 304L stainless steel. Their empirical and numerical reports indicate that water cooling increased the strength and reduced the ductility of the material; especially in the thermomechanically-affected zone (TMAZ). Cavaliere and Marco [9] stated that after single-pass FSP on AZ91, the superplasticity behavior improved. Feng [10] reported that single-pass FSP increased the tensile stress of AZ91 alloy at room temperature.

According to Sun and Apelian’s research [11], after performing FSP on aluminum alloy, the size of secondary particles reduced, but the microstructure was not homogenous. Raja and Pancholi [12] reported after single-pass FSP on AZ91 alloy, the coarse structure of the α-Mg field refined slightly, the network of secondary particles dissolved, and the mechanical properties (such as tensile and impact) at room temperature improved. Lua and Zhanhb. [13] reported that after FSP, the β-Mg17Al12 networks broke into smaller particles, but the microstructure of AZ91 alloys was not finely equiaxed.

Heidarpour et al. [14] investigated the microstructure and tensile properties of AZ91 after water-submerged FSP and non-cooled FSP. Their results show that after FSP, the rough casting structure converted into coaxial grains, and the lattice Mg17Al12 eutectic phases converted into pin-like particles on the grain boundaries. The structure of water-submerged FSP samples was more homogenous than that of the non-cooled processed samples; however, the ductility of the specimens after submerged FSP dramatically reduced. Edwin and Shamsudeen [15] studied the effect of tool pin length on microstructure and tensile strength during single-pass FSP. Their report shows that with increasing the pin length, tensile strength improved. However, increasing the length of the pin causes the tool to break quickly. Govindaraju et al. [16] studied the microstructure and mechanical properties of friction stir processed AZ91D with different heat treatment conditions. Wang et al. [2] conducted FSP on as-cast AZ31 to refine grains, homogenize the microstructure, and dissolve the secondary phases. Wang et al. [17] found that after FSP on Mg alloy, with dissolving the rough phases, the tensile and ductility properties were increased.

Mamaghani et al. [18] studied the effect of types of nanoparticles and process parameters on morphological and hardness properties of acrylonitrile butadiene styrene (ABS) plates. For this purpose, a slot with given dimensions (the depth and width) was created on the ABS sheets, and then three types of nanoparticles “nano clay, nano Fe2O3, and multi-walled carbon nanotube” were added to the slot. The workpieces were friction stir processed under different rotational and traverse speeds.

In addition, some researchers investigated the effective strain [19], temperature field [20, 21], microstructural modification [22] and material flow [23, 24] during single-pass FSP and friction stir welding (FSW) using 2-dimensional and 3-dimensional numerical modeling. Nie et al. [25] investigated the residual stresses and temperature field during FSW. Richards developed their model using two separate heat sources defined with Fortran77 DEFLUX subroutines. The modified model has been used to predict the thermal field around the tool [26]. Tutunchilar et al. [23] modeled the flow of material when a cylindrical pin was used for performing this process. They investigated the material behavior with the point tracking method. Rahul et al. [24] showed that during FSP on Mg alloys, the material flow around the pin is asymmetric. Assidi et al. [27] used 3D Forge3 finite element software based on Arbitrary Lagrangian-Eulerian formulation and automatic remeshing for modeling FSP.

Also, in recent years, FSP has been introduced as a relatively new way to create a homogeneous and fine-grained coating with improved resistance to wear and corrosion. Mostafapour et al. [28] studied the deposition of Al7075-T6 coating on Al2024-T351 substrates and investigated the effects of the rotational speed, axial force, and feed rate on the mechanical properties and microstructure of the specimens. Vahdati et al. [29] investigated the production of Al7075 surface composites using reinforcing particles (Al4O3) and FSP process.

Previous studies show that during single-pass FSP, thermal accumulation and asymmetric material flow applied to the samples may result in the growth of the grains adjacent to the process regions, non-homogeneous structure, and imperceptible increase or reduction of mechanical properties. It is possible to prepare fine-grain specimens with improved strength and produce modified wide plates through multi-pass friction stir processing (MPFSP). In this section, some studies on performing MPFSP were reviewed. Chai et al. [30] subjected AZ91 plates with a thickness of 6 mm to two-pass FSP. Their results show that some coarse β-Mg17Al12 phases that existed after the first pass of FSP break and dissolve into the matrix under the action of the second pass of FSP. Lu et al. [31] conducted two-pass FSP (with water cooling) on cast AZ91 plates. Their results show that the microhardness, tensile strength, and elongation of the processed specimens were 94.7 HV, 155.5 MPa, and 31.5%, respectively, which were more than that of the base plates. Allavikutty et al. [32] developed a layered microstructure with three different configurations by MPFSP on AZ91 using three various tools with probe lengths of 4, 5, and 7 mm. The configurations were half thickness processed, surface modified, and full
thickness processed. They concluded that the fatigue properties improved with increasing the fraction of the friction stirred processed regions in AZ91 alloy.

Alavi et al. [33], studied the influence of overlapping ratio on grain size, ductility, and tensile strength at room temperature after performing water-cooling FSP on AZ91. Mansoor and Ghosh [3], carried out the MPFSP on extruded ZK60 Mg plates to improve the mechanical properties. Nakata et al. [34] reported after MPFSP on A383 plate, the casting defects eliminated, microstructure refined, and ductility improved.

Sato and Park [4], mentioned that MPFSP is a process for the modification of AZ91 casting. Venkateswarlu et al. [35] investigated the effect of overlapping ratio (OR) and processing direction on the improvement of the tensile and ductility of AZ31 alloy.

Xicai et al. [5], conducted the two-pass FSP (with water cooling) on AZ61 magnesium alloy to improve the microstructure and dissolution of the β-phases. However, due to the adverse effect of texture evolution, the tensile strength of the two-pass processed workpiece compared to friction stir processed samples decreased.

A review of previous studies shows that the effect of consecutive passes (more than two passes) on mechanical properties and microstructure modification of AZ91 at high temperatures has not yet been investigated. As explained, in recent years, FSP has been the subject of many research studies. However, few studies have been conducted on MPFSP as a process to modify wide surfaces. In this research, the effect of MPFSP with 50% OR on microstructure, microhardness, tensile, and creep strength of AZ91 alloy at several temperatures from 25 to 210 °C were investigated. In the next sections, the description of materials, equipment, and tests are presented. In section 3, the results and discussion are presented and at the end, in section 4, conclusions are given.

2. MATERIALS AND METHODS

The workpiece material selected for this study was magnesium alloy AZ91 in the form of casting plates with the dimensions of 600×300×10 mm. The chemical composition of this alloy is listed in Table 1. The physical and mechanical properties of AZ91 are given in Table 2 [5, 36]. The tool was prepared from H13 tool steel with a pin size of 4mm diameter, 4mm length, and a flat shoulder with the diameter equal to 18mm. The tool properties are shown in Table 3 [37].

The tool was rotated clockwise and tilted 3 degrees opposite to the processing direction with 1mm of penetration depth. The process was performed using a universal milling machine DECKEL FP4M with different rotational and traverse speeds. The traverse speeds were 40, 60, and 80mm/min. Also, the tool rotational speeds of 1000 and 1200 rpm were selected. To find suitable values for process parameters, the processed specimens were inspected for process defects. Small defects on the surface could be detected using the liquid penetrate test method according to the ASME-Section V standard (article 6). In addition, for the detection of internal defects, the radiography test according to EN1435 standard using panoramic XXG300s equipment was employed. Process defects were observed as shown in Figures 1 and 2. Finally, the rotational speed of 1200 rpm and the traverse speed of 60 mm/min were selected for producing defect-free samples. The friction stir processed workpiece, with the selected parameters is shown in Figure 3.

The OR is used to determine the overlapping area between two consecutive passes and is defined by Equation (1) [35].

\[
OR = 1 - \left( \frac{l}{d_{pro}} \right)
\]

where \(l\) and \(d_{pro}\) are “the distance between the centers of two consecutive passes” and “the pin diameter”, respectively. After performing MPFSP on AZ91 plates with the selected parameters, the optical microscopy (OM, OLYMPUS CKX53 model) and scanning electron

| TABLE 1. The Chemical Composition of the AZ91 alloy |
| Al | Zn | Mn | Si | Fe | Ni | Mg |
| 8.8 | 0.7 | 0.2 | 0.03 | 0.002 | 0.0002 | Bal. |

| TABLE 2. The material properties of AZ91 at 25 °C |
| The important properties of AZ91 | Value |
| “Young’s modulus of elasticity (GPa)” | 46 |
| “Poisson’s ratio” | 0.33 |
| “Thermal conductivity (Wm⁻¹K⁻¹)” | 72 |
| Coefficient of thermal expansion (°C⁻¹) | 2.4×10⁻⁵ |
| Density (kg m⁻³) | 1810 |
| Specific heat capacity (J Kg⁻¹ °C⁻¹) | 1050 |
| Solidus temperature(°C) | 470 |
| Liquids temperature (°C) | 595 |

| TABLE 3. The material properties of H13 |
| The essential properties of AZ91 | Value |
| “Emissivity” | 0.7 |
| Coefficient of thermal expansion (°C⁻¹) | 1.17×10⁻⁵ |
| “Poisson’s ratio” | 0.3 |
microscopy (SEM, VEGA TESCAN-XMU model) were used to study the microstructure of the processed samples. In addition, the Vickers microhardness of the samples was measured on the cross section perpendicular to the processing path under 100 g loading for 15 s.

Standard tensile test specimens with 20 mm length and the width of 6 mm were cut using wire cut electro-discharge machining according to the ASTM standard E8/E8M [38]. These samples were parallel and perpendicular to the processing path (as shown in Figure 4). After preparing sub-size samples, the tensile tests were performed using the Zwick Roell testing device with the strain rate of $10^{-3}$ s$^{-1}$, at several temperatures of 25, 140, 170, and 210°C.

For conducting the impression creep test, the test samples with dimensions of 8x8x8 mm were prepared. The details of the impression creep test were explained by Mahmudi et al. [39]. The creep-testing device equipped with a controllable temperature furnace was used to carry out the constant-temperature, and constant-load impression creep tests with a simple cylindrical indenter having 2 mm diameter. Also, the impression tests were performed at several temperatures (25, 140, 170, and 210°C).

3. RESULTS AND DISCUSSION

3.1. The Microhardness Test

The hardness profiles in the stirred zone-up and stirred zone-down are shown in Figure 5. The profiles show that after MPFSP, generally, the hardness of processed workpieces increased to 87 VHN (Vickers hardness number), which was 23% more than the hardness of the base workpiece (71 VHN). As shown in Figure 5, the hardness of the stirred zone-up is more than the hardness of the stirred zone-down.

3.2. The Microstructures

The metallographic samples were prepared through the standard polishing method and then etched. The microstructure of the casting plates and multi-pass processed samples were studied using OM and SEM. The casting defects such as tunnels, grooves, cavities, and nonadhesion between the field and secondary particles are observed in Figure 6a. In addition, Figure 6b shows the microstructure of the cast workpiece that consists of non-homogenous grains and the network of eutectic phases in the α-Mg field.
After MPFSP, the microstructure refined, the grain size decreased, the eutectic lattice phases converted into the spherical shape particles, and the casting defects reduced as shown in Figure 7. After MPFSP, as shown in Figures 7b and 7c, the microstructure was non uniform. Because of intense deformation at the tool shoulder-workpiece interface, grains in the stirred zone-up can be coarser than those in the stirred zone-down, and the dislocations density in stirred zone-up is more than that in the stirred zone-down [40]. Since the microhardness of the processed samples is affected by the microstructural properties, dynamic recrystallization, texture changes, and especially the dislocations density [40]; therefore, the hardness in the zone-up is more than that in the stirred zone-down.

The average grain size of cast samples is about 98 µm. During FSP, the grains are refined. The average grain size of the MPFS Processed AZ91 alloy is about 11 µm and homogenous grains with reduced casing defects were observed. The CLEMEX commercial software was used for grain size measurement.

In a similar paper, El-Danaf et al. [41] investigated the effect of FSP on the grain size of cold-rolled sheets of AA5083. They reported that decreasing the grain size causes increase in hardness from 80 to 95HV on the nugget center. Nascimento et al. [42] studied the effect of FSP on the aluminum alloy (AA5083) for obtaining a uniform hardness using one-pass and multi-pass processes.

### 3.3. Tensile Test

The tensile test results for the base metal and the processed samples are given in Figure 8. As shown, the yield and ultimate stresses of the single-pass and MP processed samples at room temperature (parallel to the processing paths) increased by about 21 and 29% compared to the base workpiece. Improving the mechanical properties of the processed samples can be attributed to the refinement of the grains size in the processed zone and modifying the microstructure after MPFSP. In a similar research on AZ31, Feng and Ma [10] concluded that reducing the grain size increases strength. On the other hand, the recrystallization process reduces the density of dislocations. The competition between reducing the strength caused by decreasing the dislocations density and increasing the strength caused by reducing the grain size affects the hardness and tensile properties. In this research, the effect of reducing grain size is more effective, and the strength increased. Besides, after performing FSP, because of the elimination of defects and crushing of the unstable lattice intermetallic phases into the matrix, the microstructure and mechanical properties, especially at high temperatures improved. As shown in Figure 8, the yield and ultimate stresses of multi-pass processed samples at 140, 170, and 210°C increased by about 23 and 31% compared to the base workpieces. Reducing the tensile strength at a perpendicular direction to the processing path can be due to the development of firm basal texture in several non-perpendicular directions [43].

### 3.4. Creep Test

The creep behavior of base workpiece and the processed samples was investigated by impression testing in which the indenter is a circular cylinder with a flat end. After loading, the penetration depth of the indenter was measured automatically depending on time up to 4000s. The power-law equation defines the creep rate in the steady-state stage. When the impression test is used to determine creep resistance, the strain creep rate is calculated using the “impression depth of indenter"/time slope (\(V_{imp}=\frac{dh}{dt}\)) and equivalent stress is defined using the relation between the force applied to the punch and diameter of indenter.

![Unprocessed specimen of AZ91 alloy](image)

**Figure 6.** Unprocessed specimen of AZ91 alloy

![Microstructure](image)

**Figure 7.** The microstructure of a) processed specimen of AZ91 alloy using SEM, b) stir zone-down region, c) stir zone-up region

![Stress-strain diagrams](image)

**Figure 8.** The Stress-strain diagrams for all sample
impression depth/time slope, the softening of eutectic phases, especially at unprocessed samples is investigated. The penetration depth of indenter, the creep rate can be investigated. Figure 9 shows the steady state stage, the effect of FSP and temperature on creep of the base and processed samples was more than that in the stirred zone (at room temperature). The microhardness in the stirred zone is far more significant than that at room temperature. The yield and ultimate stresses of multi-pass processed samples at 140, 170, and 210°C increased by about 23% and 31% compared to the base workpiece.

The microstructure investigation shows that after FSP with the selected parameters, defects such as cracks, cavity, interconnection, non-adhesion between the field, secondary phase particles, and rough network phases, which cause stress concentration and lead to accidental and bad time failure are removed, and the microstructure is modified.

4. CONCLUSIONS

According to the experimental results, the following conclusions could be given:

1. The microhardness of the processed samples is more than that of the base material by about 23%.
2. The microhardness in the stirred zone-up is more than that in the stirred zone-down by about 14%.
3. The yield and tensile strength of specimens after processing increased by about 21 and 29% compared to the base workpiece (at room temperature).
4. The main factors for increasing the tensile strength of the MPFSP samples are considered to be the casting and processing defects, homogenous microstructure, and dissolution of the β-phases.
5. Improvement of tensile and creep properties at high temperatures is far more significant than that at room temperature. The yield and ultimate stresses of multi-pass processed samples at 140, 170, and 210°C increased by about 23% and 31% compared to the base workpiece.
6. The creep resistance of friction stir processed samples was more than that of the unprocessed ones by about 38%.
7. The microstructure investigation shows that after FSP with the selected parameters, defects such as cracks, cavity, interconnection, non-adhesion between the field, secondary phase particles, and rough network phases, which cause stress concentration and lead to accidental and bad time failure are removed, and the microstructure is modified.

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تاثیرات برای افراد نسبت ایجاد انکسار به وقوع امواج الکترومغناطیسی و میلای ارتعاشات، آزمایش کاربردی آلاینگی های میلیمتری از جمله AZ91 در صنایع مختلف مانند

فناها، نظاره، خودروسازی و کشی سازی در دمای بالا به دلیل اینکسار اندازه‌ای در زیر دانه و مصالح دوگانه، استحکام کشی تنها و مقاومت خود آلیاژی در میانی در مداه‌ای بالای کاهش می‌یابد. برای بهبود این خواص در دمای بالا از آزمایشات غیر شکل دادن منابع فرایندهای امکان‌پذیر استفاده می‌شود. در این تحقیق، تأثیر فرایندهای امکان‌پذیر بهبود بهبودی، بررسی کشی و مقاومت خود آلیاژی AZ91 از نظر مقاومت فرنگی این آلیاژ در دمای بالا از تعداد نیوک فلزاتی بهبودی، استحکام کشی و مقاومت خود آلیاژی به ترتیب حداکثر 39.2 و

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هافری گره‌ای های آلیاژی در دمای بالا یکی از تأثیرات ممکن برای افراد نسبت به وقوع امواج الکترومغناطیسی و میلای ارتعاشات، آزمایش کاربردی آلاینگی های میلیمتری از جمله AZ91 در صنایع مختلف مانند

فناها، نظاره، خودروسازی و کشی سازی در دمای بالا به دلیل اینکسار اندازه‌ای در زیر دانه و مصالح دوگانه، استحکام کشی تنها و مقاومت خود آلیاژی در میانی در مداه‌ای بالای کاهش می‌یابد. برای بهبود این خواص در دمای بالا از آزمایشات غیر شکل دادن منابع فرایندهای امکان‌پذیر استفاده می‌شود. در این تحقیق، تأثیر فرایندهای امکان‌پذیر بهبود بهبودی، بررسی کشی و مقاومت خود آلیاژی AZ91 از نظر مقاومت فرنگی این آلیاژ در دمای بالا از تعداد نیوک فلزاتی بهبودی، استحکام کشی و مقاومت خود آلیاژی به ترتیب حداکثر 39.2 و

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