Article

The Effect of Processing Pass on the Microstructure and Mechanical Properties of a Friction Stir Processed As-Cast Mg-6 wt % Sn Alloy

Yang Zhang, Xiaoyang Chen *, Xiyun Qin, Feilong Li, Yalin Lu and Xiaoping Li

Key lab of advanced material design and additive manufacturing of Jiangsu Province, Jiangsu University of Technology, Changzhou 213001, China; zhangyang@jsut.edu.cn (Y.Z.); qxy9509@163.com (X.Q.); lfl_real@163.com (F.L.); jxlyl@jsut.edu.cn (Y.L.); lxp118@jsut.edu.cn (X.L.)

* Correspondence: cxy@jsut.edu.cn; Tel.: +86-519-8695-3210

Received: 14 October 2018; Accepted: 3 November 2018; Published: 5 November 2018

Abstract: In this study, as-cast Mg-6 wt % Sn alloy is subjected to one-pass and two-pass friction stir processing (FSP). The effect of processing pass on microstructure and mechanical properties of FSP Mg-6Sn alloy is investigated. It is found that one-pass FSP leads to the breakage and partial dissolution of the Mg₂Sn phase in the stir zone (SZ) and two-pass FSP leads to the further dissolution and dynamic precipitation of the Mg₂Sn phase. Dynamic recrystallization (DRX) takes place in the SZ of an Mg-6Sn alloy undergoing FSP. Compared to one-pass FSP, two-pass FSP brings about further grain refinement in the SZ. A strong {0001} basal texture is developed in the SZ of a Mg-6Sn alloy from FSP and the change of the sample region or processing pass has little influence on the texture. Compared to an as-cast Mg-6Sn sample, one-pass FSP brings about significant improvement in mechanical properties. Two-pass FSP leads to the further increase in yield strength (YS) and ultimate tensile strength (UTS) but elongation (EL) is reduced. The continuous increase in strength is attributed to the grain refinement and the dissolution and dynamic precipitation of Mg₂Sn phase achieved by FSP.

Keywords: friction stir processing; processing pass; microstructure; texture; mechanical properties

1. Introduction

In the face of challenges from increasing energy consumption and pollution emissions, the application of Mg alloys in transportation and aviation industries has gained considerable attention due to their significant advantages in weight reduction. Therefore, the development of high-performance Mg alloys receives constant attention [1–3]. According to the Mg-Sn binary phase diagram, a binary Mg-Sn alloy has a relatively high eutectic temperature and the second phase in binary Mg-Sn alloy, i.e., Mg₂Sn phase, has a high melting point. Therefore, Mg-Sn alloys have been recognized as potential heat-resistant Mg alloys with an engineering application value in recent years [4–6]. Meanwhile, since the Sn element is an essential trace element in the human body and noncytotoxic, Mg-Sn alloys are promising bio-Mg alloys used as a bio-implant material [7,8]. Therefore, the study of Mg-Sn alloys is of essential research value.

Compared to cast Mg alloys, wrought Mg alloys usually possess a more uniform chemical composition distribution, refined microstructure, and improved mechanical properties. Friction stir processing (FSP) is a typical severe plastic deformation (SPD) method that is developed on the principles of friction stir welding (FSW) [9]. During FSP, metallic materials are subjected to the combined effect of a deformation force and friction heat, which can induce complete dynamic recrystallization (DRX) and results in fine-grained equiaxed structure in the stir zone (SZ) [10–12].

The final microstructure and mechanical properties of FSP samples are largely influenced by the processing parameters, such as rotation speed, travel speed, and processing pass [13–15]. Compared to single-pass FSP, multi-pass FSP can further refine the grains with an increased level of accumulated strain and make the microstructure more uniform. However, the study on multi-pass FSP is mainly focused on its effect on DRX. The mechanical properties of FSP samples are definitely influenced by DRXed grains and second phases. Therefore, the evolution of second phases with high melting point during multi-pass FSP needs to be studied in depth.

The application of FSP can overcome the inherent shortcoming on poor formability of Mg alloys at room temperature induced by the hexagonal close-packed (HCP) structure. Therefore, there are great opportunities to produce high-quality Mg alloys using FSP with appropriate processing parameters. Up to now, studies on the FSP of Mg alloys have mainly focused on Mg–Al, Mg–Zn, and Mg–RE alloys [16–18]. In order to improve the limited mechanical properties of Mg–Sn alloys, plastic deformation methods are often used [19,20]. However, the research work on the FSP of Mg–Sn alloys is limited and there is still no research on multi-pass FSP of Mg–Sn alloys. Compared to single-pass FSP, multi-pass FSP is expected to further improve the mechanical properties of Mg–Sn alloys.

Accordingly, in this study, as-cast binary Mg–6Sn (all composition in wt % except otherwise stated) alloy is prepared and subjected to FSP for one-pass or two-passes. The effect of processing pass on microstructure and mechanical properties of FSP Mg–6Sn alloy is investigated. The research aim is to elucidate the microstructure evolution (including DRXed grains and Mg₂Sn phase) during multi-pass FSP and its influence on the mechanical properties of an Mg–6Sn alloy. The relationship between microstructure and mechanical properties is also discussed.

2. Materials and Methods

Commercial pure Mg (99.95%) and Sn (99.99%) were used for the preparation of as-cast Mg–6Sn alloy with vacuum induction melting. Plates used for FSP (base metal, BM) was cut from the as-cast ingots with a size of 130 mm × 75 mm × 8 mm. Oxides on the surfaces of the plates were removed via manual grinding. An FSP machine (FSW-LM-BM16, FSW Technology Co., Ltd., Beijing, China) was used in this study. Figure 1 shows the schematic of FSP in this study. The tool made of H13 steel had a triple helix shoulder of 15 mm in diameter and a conical threaded pin, 4.1 mm long, with a diameter of 6.5 mm and 4.3 mm at the base and the tip, respectively. During FSP, the plunge depth was kept as 0.2 mm. The travel speed and rotation speed were set as 60 mm/min and 1200 rev/min respectively. The Mg–6Sn sample after one-pass of FSP was named as FSP1. For two-pass FSP, the second pass was in the same tool travel direction with the first pass. The travel speed and rotation speed were kept constant and 100% overlap was used. The Mg–6Sn sample after two-pass FSP was named FSP2.

Figure 1. Schematic of friction stir processing (FSP) in this study.

An optical microscope (OM, Zeiss Primotech, Berlin, Germany) and scanning electron microscope (SEM, Simga 500 VP, Zeiss, Oberkochen, Germany) equipped with electron backscattered diffraction (EBSD, Inca, Oxford Instruments, Oxford, UK) were used for microstructure and texture characterization. The gauge size of tensile specimens was 10 mm × 3.5 mm × 2 mm. The up surfaces

of FSP samples were removed and the tensile specimens were taken 2 mm down from the top surface along the processing direction (PD). A universal testing machine (CMT-5205, Wance, Shen Zhen, China) was used for room temperature tensile test and the strain rate was $1.67 \times 10^{-3} \text{ s}^{-1}$.

3. Results

Figure 2 shows the OM and back-scattered electron (BSE)-SEM images of an as-cast Mg-6Sn sample. As shown in Figure 2a, the as-cast Mg-6Sn sample consisted of coarse dendritic α -Mg grains and a eutectic second phase, which is often observed in as-cast Mg alloys. According to the binary Mg-Sn phase diagram, Mg_2Sn phase is the only second phase in binary Mg-Sn alloys. In as-cast Mg-6Sn alloy, the Mg_2Sn phase forms through the eutectic reaction at the end of the solidification. As shown in Figure 2b, the bright eutectic phase was the Mg_2Sn phase. Most of Mg_2Sn phase was connected-network-like and located among the dendrite arms of α -Mg grains. Meanwhile, the difference in contrast of α -Mg grains indicated that the distribution of Sn element in α -Mg grains was inhomogeneous and a generous amount of Sn element aggregated near the grain boundaries.

Figure 2. OM and BSE-SEM images of as-cast Mg-6Sn sample: (a) OM image and (b) BSE-SEM image.

Figure 3 shows the low-magnification optical macrographs of Mg-6Sn samples from FSP (transverse-normal direction plane). No visible pores or other defects were observed in the SZ due to the FSP of Mg-6Sn samples, which indicates that the selected processing parameters in this study were proper for the FSP of as-cast Mg-6Sn alloys. As shown in Figure 3, the outlines and areas of both FSP1 and FSP2 samples show little difference.

Figure 3. Low-magnification optical macrographs of FSP Mg-6Sn samples (TD-ND plane): (a) FSP1 and (b) FSP2.

In order to characterize the microstructure of the SZ of Mg-6Sn samples from FSP in detail, two representative regions with different depths were selected from the center of the SZ of both FSP1 and FSP2 samples, i.e., SZ-2 mm (2 mm down from the top surface) and SZ-4 mm (4 mm down from the top surface) regions, as can be seen in Figure 3. Figure 4 shows the BSE-SEM images of SZ-2 mm and SZ-4 mm regions of Mg-6Sn samples created using FSP. As shown in Figure 4a, after one-pass of FSP, obvious plastic deformation streamlines were formed in the SZ-2 mm region of the FSP1 sample. The eutectic Mg_2Sn phase was broken into particles and distributed along the streamlines. However, in the SZ-4 mm region of the FSP1 sample (seen in Figure 4c), no plastic deformation streamlines were observed. Most of the Mg_2Sn phase disappeared after one-pass of FSP while only several fine Mg_2Sn particles were observed in the SZ-2 mm region of the FSP1 sample. As shown in Figure 4b, the obvious streamlines were also observed in the SZ-2 mm region of the FSP2 sample. Two-pass FSP led to the further dissolution of Mg_2Sn particles and the volume fraction of the Mg_2Sn phase was reduced compared to the FSP1 sample. As shown in Figure 4d, after two-pass FSP, numerous fine Mg_2Sn particles were observed in the SZ-4 mm region of FSP2 sample. The number density of fine Mg_2Sn particles in the SZ-4 mm region of the FSP2 sample increased significantly compared to the FSP1 sample. Table 1 presents the volume fraction of the Mg_2Sn phase in SZ-2 mm and SZ-4 mm regions of the Mg-6Sn samples due to FSP. The results of the quantitative metallographic analysis confirm that the Mg_2Sn phase was partially dissolved into an $\alpha\text{-Mg}$ matrix during FSP in different degrees. For the SZ-2 mm region, the volume fraction of the Mg_2Sn phase decreased from 2.55% in the as-cast sample to 2.10% in the FSP1 sample, and it further decreased to 1.81% in the FSP2 sample. However, for the SZ-4 mm region, the volume fraction of the Mg_2Sn phase first decreased to a relatively low value (0.46%) in the FSP1 sample, but then increased to 0.80% in the FSP2 sample. The abnormal increase of the volume fraction of the Mg_2Sn phase in the SZ-4 mm region of the FSP2 sample indicated the dynamic precipitation of the Mg_2Sn phase during the second-pass of FSP.

Figure 4. BSE-SEM images of the SZ-2 mm and SZ-4 mm regions of Mg-6Sn samples from FSP: (a) SZ-2 mm region of FSP1 sample, (b) SZ-2 mm region of FSP2 sample, (c) SZ-4 mm region of FSP1 sample, and (d) SZ-4 mm region of FSP2 sample.

Table 1. Volume fraction of Mg₂Sn phase in the SZ-2 mm and SZ-4 mm regions of Mg-6Sn samples from FSP.

Sample	Region	Volume Fraction of Mg ₂ Sn Phase (vol.%)
As-cast	-	2.55
FSP1	SZ-2 mm	2.10
	SZ-4 mm	0.46
FSP2	SZ-2 mm	1.81
	SZ-4 mm	0.80

Figure 5 shows the inverse pole figure maps of the SZ-2 mm and SZ-4 mm regions in Mg-6Sn samples created using FSP obtained by EBSD. For comparison, the inverse pole figure map of an as-cast Mg-6Sn sample is also shown in Figure 5a. As shown in Figure 5a, coarse grains were observed in the as-cast Mg-6Sn sample and the orientation was relatively random. It was found that, during FSP, dynamic recrystallization (DRX) took place in the SZ regions of Mg-6Sn samples from FSP. Compared to an as-cast Mg-6Sn sample with an average size of α -Mg grains over 200 μm , α -Mg grains in Mg-6Sn samples from FSP were significantly refined. Based on the inverse pole figure maps, two basic rules can be concluded. First, compared to the FSP1 sample, two-pass FSP brought about further grain refinement in both the SZ-2 mm and SZ-4 mm regions of the FSP2 sample. Second, in both the FSP1 and FSP2 samples, the DRXed grains in the SZ-2 mm regions were coarser than those in the SZ-4 mm regions. Table 2 presents the average size of DRXed grains in the SZ-2 mm and SZ-4 mm regions of Mg-6Sn samples created using FSP. The average size of DRXed grains in the SZ-2 mm region decreased from 5.46 μm in the FSP1 sample to 4.46 μm in the FSP2 sample. The average size of DRXed grains in the SZ-4 mm region decreased from 4.67 μm in the FSP1 sample to 3.24 μm in the FSP2 sample. It was also found that, after FSP, the orientation of DRXed grains in the SZ of the FSP1 and FSP2 samples turned out to be highly consistent. The {0001} plane of most DRXed grains was nearly parallel to PD.

Figure 5. Cont.

Figure 5. Inverse pole figure maps of an as-cast Mg-6Sn sample and the SZ-2 mm and SZ-4 mm regions in FSP Mg-6Sn samples obtained by EBSD: (a) as-cast Mg-6Sn sample, (b) SZ-2 mm region of the FSP1 sample, (c) SZ-2 mm region of the FSP2 sample, (d) SZ-4 mm region of the FSP1 sample, and (e) SZ-4 mm region of the FSP2 sample.

Table 2. Average size of the DRXed grains in the SZ-2 mm and SZ-4 mm regions of Mg-6Sn samples created with FSP.

Sample	Region	Average Size of DRXed Grains (μm)
FSP1	SZ-2 mm	5.46
	SZ-4 mm	4.67
FSP2	SZ-2 mm	4.46
	SZ-4 mm	3.24

Figure 6 shows the $\{0001\}$ pole figures of the SZ-2 mm and SZ-4 mm regions in Mg-6Sn samples from FSP obtained using EBSD. It was confirmed that, after FSP, a strong $\{0001\}$ basal texture was developed in the SZ-2 mm and SZ-4 mm regions of both the FSP1 and FSP2 samples. The intensity of the $\{0001\}$ basal texture was relatively high for all four regions (ranging from 43.1 to 59.9). For all four regions, the intensity peaks were tilted from PD to ND slightly. The pole figures of different SZ regions from the FSP1 and FSP2 samples were almost the same, which proved that the change of sample region or processing pass had little influence on the texture except for the intensity.

Figure 6. Pole figure maps of the SZ-2 mm and SZ-4 mm regions in the Mg-6Sn samples created using FSP obtained using EBSD: (a) SZ-2 mm region of the FSP1 sample, (b) SZ-2 mm region of the FSP2 sample, (c) SZ-4 mm region of the FSP1 sample, and (d) SZ-4 mm region of the FSP2 sample.

Figure 7 presents the mechanical properties of as-cast and Mg-6Sn samples from FSP. The mechanical properties of an as-cast Mg-6Sn sample are relatively low, with its yield strength (YS), ultimate tensile strength (UTS), and elongation (EL) of 50.1 MPa, 116.6 MPa, and 10.4% respectively. One-pass FSP brought about comprehensive improvement in the mechanical properties and YS, UTS, and EL of the FSP1 sample reached 68.3 MPa, 139.8 MPa, and 24.2%, respectively, which were an increase of 36%, 20%, and 133%, respectively, compared to an as-cast Mg-6Sn sample. Two-pass FSP further improved the strength of the Mg-6Sn sample and YS and UTS of the FSP2 sample reached 91.0 MPa and 154.4 MPa respectively, which were an increase of 33% and 10%, respectively, compared to FSP1 sample. However, two-pass FSP was harmful to the ductility of the Mg-6Sn alloy and EL of the FSP2 sample was decreased to 17.5%. The results of the mechanical properties show that one-pass and two-pass FSP can improve the strength of an Mg-6Sn alloy continuously.

Figure 7. Mechanical properties of as-cast and Mg-6Sn samples created using FSP.

4. Discussion

In this study, the dissolution and dynamic precipitation of the Mg_2Sn phase was observed in different SZ regions of Mg-6Sn samples from FSP. The dissolution and dynamic precipitation were induced by the thermal-mechanical effect during FSP. Due to the inhomogeneous distribution of the deformation force and friction heat in the SZ [21], the size and relative content of the Mg_2Sn phase in different SZ regions exhibited a distinct difference.

Although the SZ-2 mm region, which was near the top surface of the SZ, was subjected to a higher heat input than the SZ-4 mm region, the dissolution of the Mg_2Sn phase in the SZ-4 mm region of the FSP1 and FSP2 samples was more significant than the SZ-2 mm region. Since the SZ-2 mm region was deformed under the combined effect of shear deformation using a rotating pin and compressive deformation at the shoulder, the plastic deformation force subjected to the SZ-2 mm region was lower than the SZ-4 mm region. These results indicated that the deformation force rather than the elevated temperature was the main determining factor for the dissolution of the second phase during FSP [22]. Therefore, the SZ-4 mm region provided more proper conditions for the dissolution of the Mg_2Sn phase than the SZ-2 mm region. Compared to a conventional solid solution treatment [23], the dissolution of the Mg_2Sn phase was significantly accelerated during FSP in this study. Due to the high-speed friction, a relatively high temperature could be generated via FSP in the SZ. More importantly, the Mg_2Sn phase in as-cast sample was broken into small particles, which increased the contact area between the Mg_2Sn phase and the α -Mg matrix. High density dislocation and vacancies were formed in the SZ regions and a high diffusion path was created for the Sn element. Therefore, the diffusion of Sn in the α -Mg matrix was accelerated significantly [24]. The dissolution of the second phase during FSP was also observed in other Mg alloys. For example, $Mg_5(Gd, Y)$ phase in as-cast Mg-10Gd-3Y-0.5Zr alloy and $Mg_{12}Nd$ phase in as-cast Mg-2.9Nd-0.18Zn-0.4Zr was found to dissolve completely during FSP [25,26]. The results in this study also confirm that two-pass FSP can further promote the dissolution of a broken Mg_2Sn phase in the SZ-2 mm regions.

As for SZ-4 mm regions, compared to the FSP1 sample, the volume fraction of the Mg_2Sn phase in the SZ-4 mm region of the FSP2 sample was increased, which indicated the occurrence of dynamic precipitation. The solid solubility of Sn in the α -Mg matrix was largely influenced by the temperature and it decreased from 14.85 wt % at 561 °C to 0.45 wt % at 200 °C. The drastically varied solid solubility with temperature was the basis for the dynamic precipitation. The strain-induced dynamic precipitation has been reported in several wrought Mg alloys [27,28]. For example, a dynamically precipitated $Mg_{17}Al_{12}$ phase was observed in DRXed grains of rolled Mg-8Al sheets [29]. In this study, after one-pass of FSP, most of the Mg_2Sn phase in the SZ-4 mm region was dissolved into the α -Mg matrix and led to the formation of a supersaturated solid solution. The distribution of Sn in the α -Mg matrix also became relatively uniform. Compared to one-pass FSP, two-pass FSP further increased

the amount of crystal defects, including dislocation and vacancies. The crystal defects could provide effective nucleation sites for Mg_2Sn phase and the absorbed deformation energy could promote the precipitation. Therefore, numerous fine Mg_2Sn particles were dynamically precipitated in the SZ-4 mm region during two-pass FSP.

It is well-accepted that FSP is able to induce DRX and significant grain refinement in BM with coarse grains. Multi-pass FSP is reported to be able to further refine the DRXed grains in the SZ. For example, Luo et al. found that, after two-pass submerged FSP, the average grain size of AZ61 alloy was decreased from 5.2 μm to 4.6 μm [30]. However, the effect of multi-pass FSP on grain refinement is still inconsistent up to now. This is because the heat input and accumulated strain induced by multi-pass FSP has the opposite effect on grain refinement. The accumulated strain can promote further DRX and corresponding grain refinement in the SZ. However, the grain refinement via multi-pass FSP may be counteracted by accompanied grain coarsening since the increased heat input may result in the significant coarsening of DRXed grains. Therefore, the final grain size in the SZ of multi-pass FSP samples is determined by the competition between the heat input and accumulated strain. In this study, as shown in Figure 5, two-pass FSP promoted the further refinement of the α -Mg grains in both the SZ-2 mm and SZ-4 mm regions. These results indicated that the grain refinement achieved by accumulated strain was more significant than the grain coarsening induced by the increased heat input during the second pass FSP in this study. The final microstructure of the FSP samples was largely influenced by the processing parameters. Previous research on single-pass FSP of Mg-6Sn-2Zn alloy showed that, when the travel speed increased from 60 mm/min to 120 mm/min, the average size of DRXed grains first increased and then decreased [22]. The influence of processing parameters on the final grain size was attributed to the contrary effect of severe plastic deformation and thermal exposure. According to the calculation, the travel speed showed little influence on the strain rate while the heat input was reduced with the increase of travel speed, which indicated that thermal exposure was the main determining factor of final grain size. However, in this study, the multi-pass FSP induces a continuous decrease in the grain size and these results indicate that severe plastic deformation is the main determining factor of final grain size.

The results in Figure 7 confirm that the mechanical properties of Mg-6Sn alloy were influenced by FSP significantly. The variation of mechanical properties, including strength and ductility, was related to the microstructure evolution and texture.

YS of as-cast Mg-6Sn alloy is relatively low and FSP leads to the continuous increase of YS. The improvement in YS of Mg-6Sn alloy is mainly attributed to the grain refinement achieved by FSP. According to the classic Hall–Petch equation, YS is proportional to $d^{-1/2}$, where d is the average diameter of grains. In this study, coarse α -Mg grains with an average diameter over 200 μm are formed in as-cast Mg-6Sn alloy. After one-pass of FSP, the coarse α -Mg grains are broken into fine grains via DRX and two-pass FSP leads to the further grain refinement in FSP2 sample. As a consequence, the YS of Mg-6Sn alloy is improved continuously. Moreover, the dissolution and dynamic precipitation of the Mg_2Sn phase also influences the YS of the Mg-6Sn alloy. In as-cast Mg-6Sn alloy, a eutectic Mg_2Sn phase is coarse and mainly distributed at the grain boundaries. The strengthening effect of a coarse and connected eutectic phase in as-cast Mg alloys is relatively low. During FSP, the Mg_2Sn phase is broken into fine particles under severe plastic deformation. Part of the Mg_2Sn phase is dissolved into an α -Mg matrix and contributes to the solid solution strengthening. Meanwhile, the number density of Mg_2Sn particles in Mg-6Sn alloy created using FSP, including the residual and dynamically precipitated Mg_2Sn phase, is much higher than the as-cast Mg-6Sn alloy. It is believed that the fine and dense precipitates is beneficial to YS, since it is able to provide more obstacles to dislocation movement [31]. Compared to the FSP1 sample, the DRXed grains in the SZ of FSP2 sample are more refined and the broken and precipitated Mg_2Sn particles are denser. Therefore, the YS of the FSP2 sample is higher than that of the FSP1 sample.

In this study, it was found that FSP also led to the significant improvement in ductility of the Mg-6Sn alloy. Compared to the as-cast sample, the EL of FSP1 sample was improved by $\approx 133\%$ while

the two-pass FSP led to a slight decrease in the EL of the FSP2 sample. However, the EL of the FSP2 sample was still much higher than that of the as-cast sample. The significant improvement in ductility of the FSP samples are also observed in other alloys [32,33]. The increased ductility is attributed to the grain refinement, the breakage of coarse eutectic second phase, and the formation of strong basal texture achieved by FSP. In this study, due to the formation of strong basal texture in the FSP samples, the texture played an important role in improving the ductility. During tensile testing, the dislocation slip on the basal plane was able to accommodate a large strain. Moreover, the basal slip could also lead to the formation of $\{10\bar{1}2\}$ extension twinning and contributed to the strain accommodation. Therefore, high ductility was achieved in Mg-6Sn samples from FSP in this study. The comparison between the EL of the FSP1 and FSP2 samples shows that the further grain refinement led to the reduction in ductility of the Mg-6Sn samples created using FSP. This phenomenon is also observed in several severe plastic deformed Mg alloys. For example, W. Yuan and Mishra [34] found that the EL of an AZ31 alloy created using FSP decreased from 65% to 10% when the average size decreased from 9.6 μm to 0.8 μm . The reduction in ductility was related to a low degree of work hardening and a high rate of recovery or stress localization depending on the DRXed grain size. When the grain size decreased from 9.6 μm to 2.8 μm , it was attributed to stress localization, and when the grain size decreased from 2.8 μm to 0.8 μm , it was attributed to a low degree of work hardening and high rate of recovery. Accordingly, in this study, the reduction in ductility from the FSP1 sample to the FSP2 sample could be attributed to the stress localization. The further refined grains may have led to the formation of inhomogeneous strain localization zones in terms of large shear bands during tensile testing. The fine shear bands were beneficial to the ductility while the large ones were harmful. Therefore, the ductility of the FSP2 samples was deteriorated. The study on multi-pass FSP of AZ61 alloy showed that the two-pass FSP induced significant ductility improvement while the strength was slightly reduced [30]. This was attributed to the texture modification and a high Schmid factor. After two-pass FSP, the intensity of the texture of an AZ61 alloy from FSP decreased from 23.2 to 16.4, the tilted angle between *c*-axis and PD increased to 19° from 35°, and the average Schmid factor increased from 0.313 to 0.410. The preferred texture orientation could promote a basal slip easily and the ductility was consequently improved. However, in this study, the change of processing pass showed little influence on the texture of the SZ in Mg-6Sn samples from FSP. As shown in Figure 6, two-pass FSP even led to a slight increase in the texture intensity and the tilted angle was almost the same. Therefore, two-pass FSP could not contribute to the ductility improvement in this study.

5. Conclusions

In this study, as-cast Mg-6Sn alloy was subjected to FSP for one-pass or two-passes and the effect of processing pass on microstructure and mechanical properties of an Mg-6Sn alloy created using FSP was investigated. The main conclusions are listed as follows.

(1) The microstructure in the SZ of an Mg-6Sn alloy created using FSP was inhomogeneous. One-pass FSP led to the breakage and partial dissolution of the Mg_2Sn phase. Two-pass FSP led to the further dissolution and dynamic precipitation of the Mg_2Sn phase in different regions of the SZ.

(2) DRX took place in the SZ regions of Mg-6Sn samples created using FSP. Compared to one-pass FSP, two-pass FSP brought about further grain refinement in the SZ. A strong $\{0001\}$ basal texture was developed in the SZ regions of Mg-6Sn samples created using FSP. The change of sample region or processing pass had little influence on the texture except for the intensity.

(3) Compared to an as-cast Mg-6Sn sample, one-pass FSP brought about comprehensive improvement in mechanical properties. Two-pass FSP led to the further increase in the YS and UTS but the EL was reduced. The continuous increase in strength was attributed to the grain refinement and the dissolution and dynamic precipitation of Mg_2Sn phase achieved using FSP.

Author Contributions: Conceptualization, X.C.; Data curation, X.C.; Formal analysis, X.C.; Funding acquisition, Y.Z., Y.L., and X.L.; Investigation, Y.Z., X.Q., and F.L.; Methodology, X.Q. and F.L.; Project administration, Y.L. and X.L.; Resources, Y.L. and X.L.; Writing—Original draft, Y.Z. and X.C.

Funding: This research was financially supported by National Natural Science Foundation of China (No. 51601076) and Natural Science Fund for Colleges and Universities in Jiangsu Province (No. 15KJA430003, 16KJB430013, 17KJA430005).

Conflicts of Interest: The authors declare no conflict of interest.

References

- Ren, L.B.; Quan, G.F.; Xu, Y.G.; Yin, D.D.; Lu, J.W.; Dang, J.T. Effect of heat treatment and pre-deformation on damping capacity of cast Mg-Y binary alloys. *J. Alloys Compd.* **2017**, *699*, 976–982. [\[CrossRef\]](#)
- Zhang, Y.; Rong, W.; Wu, Y.; Peng, L.; Nie, J.F.; Birbilis, N. A comparative study of the role of Ag in microstructures and mechanical properties of Mg-Gd and Mg-Y alloys. *Mater. Sci. Eng. A* **2018**, *731*, 609–622. [\[CrossRef\]](#)
- Zhang, Y.; Yang, L.; Dai, J.; Ge, J.; Guo, G.; Liu, Z. Effect of Ca and Sr on the compressive creep behavior of Mg-4Al-RE based magnesium alloys. *Mater. Des.* **2014**, *63*, 439–445. [\[CrossRef\]](#)
- Cheng, W.; Tian, L.; Wang, H.; Bian, L.; Yu, H. Improved tensile properties of an equal channel angular pressed (ECAPed) Mg-8Sn-6Zn-2Al alloy by prior aging treatment. *Mater. Sci. Eng. A* **2017**, *687*, 148–154. [\[CrossRef\]](#)
- Huang, X.; Du, Y.; Li, W.; Chai, Y.; Huang, W. Effects of Ag content on the solid-solution and age-hardening behavior of a Mg-5Sn alloy. *J. Alloys Compd.* **2017**, *696*, 850–855. [\[CrossRef\]](#)
- Khalilpour, H.; Miresmaeili, S.M.; Baghani, A. The microstructure and impression creep behavior of cast Mg-4Sn-4Ca alloy. *Mater. Sci. Eng. A* **2016**, *652*, 365–369. [\[CrossRef\]](#)
- Zhao, C.Y.; Pan, F.S.; Pan, H.C. Microstructure, mechanical and bio-corrosion properties of as-extruded Mg-Sn-Ca alloys. *Trans. Nonferrous Met. Soc. China* **2016**, *26*, 1574–1582. [\[CrossRef\]](#)
- Shuai, C.; Zhou, Y.; Lin, X.; Yang, Y.; Gao, C.; Shuai, X.; Wu, H.; Liu, X.; Wu, P.; Feng, P. Preparation and characterization of laser-melted Mg-Sn-Zn alloys for biomedical application. *J. Mater. Sci. Mater. Med.* **2017**, *28*, 13. [\[CrossRef\]](#)
- Ma, Z.Y. Friction stir processing technology: A review. *Metall. Mater. Trans. A* **2008**, *39*, 642–658. [\[CrossRef\]](#)
- Yang, Q.; Xiao, B.L.; Ma, Z.Y.; Chen, R.S. Achieving high strain rate superplasticity in Mg-Zn-Y-Zr alloy produced by friction stir processing. *Scr. Mater.* **2011**, *65*, 335–338. [\[CrossRef\]](#)
- Khodabakhshi, F.; Simchi, A.; Kokabi, A.H.; Nosko, M.; Simančík, F.; Švec, P. Microstructure and texture development during friction stir processing of Al-Mg alloy sheets with TiO₂ nanoparticles. *Mater. Sci. Eng. A* **2014**, *605*, 108–118. [\[CrossRef\]](#)
- Cao, G.; Zhang, D.; Chai, F.; Zhang, W.; Qiu, C. Superplastic behavior and microstructure evolution of a fine-grained Mg-Y-Nd alloy processed by submerged friction stir processing. *Mater. Sci. Eng. A* **2015**, *642*, 157–166. [\[CrossRef\]](#)
- Aktarer, S.M.; Sekban, D.M.; Saray, O.; Kucukomeroglu, T.; Ma, Z.Y.; Purcek, G. Effect of two-pass friction stir processing on the microstructure and mechanical properties of as-cast binary Al-12Si alloy. *Mater. Sci. Eng. A* **2015**, *636*, 311–319. [\[CrossRef\]](#)
- Yang, Q.; Xiao, B.L.; Ma, Z.Y. Influence of process parameters on microstructure and mechanical properties of friction-stir-processed Mg-Gd-Y-Zr casting. *Metall. Mater. Trans. A* **2012**, *43*, 2094–2109. [\[CrossRef\]](#)
- Al-Fadhalah, K.J.; Almazrouee, A.I.; Aloraier, A.S. Microstructure and mechanical properties of multi-pass friction stir processed aluminum alloy 6063. *Mater. Des.* **2014**, *53*, 550–560. [\[CrossRef\]](#)
- Chai, F.; Zhang, D.; Li, Y. Microstructures and tensile properties of submerged friction stir processed AZ91 magnesium alloy. *J. Magnes. Alloys* **2015**, *3*, 203–209. [\[CrossRef\]](#)
- Vargas, M.; Lathabai, S.; Uggowitzer, P.J.; Qi, Y.; Orlov, D.; Estrin, Y. Microstructure, crystallographic texture and mechanical behaviour of friction stir processed Mg-Zn-Ca-Zr alloy ZKX50. *Mater. Sci. Eng. A* **2017**, *685*, 253–264. [\[CrossRef\]](#)
- Zheng, F.Y.; Wu, Y.J.; Peng, L.M.; Li, X.W.; Fu, P.H.; Ding, W.J. Microstructures and mechanical properties of friction stir processed Mg-2.0Nd-0.3Zn-1.0Zr magnesium alloy. *J. Magnes. Alloys* **2013**, *1*, 122–127. [\[CrossRef\]](#)
- Elsayed, F.R.; Sasaki, T.T.; Ohkubo, T.; Takahashi, H.; Xu, S.W.; Kamado, S.; Hono, K. Effect of extrusion conditions on microstructure and mechanical properties of microalloyed Mg-Sn-Al-Zn alloys. *Mater. Sci. Eng. A* **2013**, *588*, 318–328. [\[CrossRef\]](#)

20. Park, S.H.; Kim, S.H.; Kim, H.S.; Yoon, J.; You, B.S. High-speed indirect extrusion of Mg-Sn-Al-Zn alloy and its influence on microstructure and mechanical properties. *J. Alloys Compd.* **2016**, *667*, 170–177. [[CrossRef](#)]
21. Mishra, R.S.; Ma, Z.Y. Friction stir welding and processing. *Mater. Sci. Eng. R* **2005**, *50*, 1–78. [[CrossRef](#)]
22. Chen, X.; Dai, Q.; Li, X.; Lu, Y.; Zhang, Y. Microstructure and Tensile Properties of Friction Stir Processed Mg-Sn-Zn Alloy. *Materials* **2018**, *11*, 645. [[CrossRef](#)] [[PubMed](#)]
23. Li, W.; Huang, X.; Huang, W. Effects of Ca, Ag addition on the microstructure and age-hardening behavior of a Mg-7Sn (wt %) alloy. *Mater. Sci. Eng. A* **2017**, *692*, 75–80. [[CrossRef](#)]
24. Wang, Y.B.; Huang, Y.X.; Meng, X.C.; Wan, L.; Feng, J.C. Microstructural evolution and mechanical properties of Mg-Zn-Y-Zr alloy during friction stir processing. *J. Alloys Compd.* **2017**, *696*, 875–883. [[CrossRef](#)]
25. Xiao, B.L.; Yang, Q.; Yang, J.; Wang, W.G.; Xie, G.M.; Ma, Z.Y. Enhanced mechanical properties of Mg-Gd-Y-Zr casting via friction stir processing. *J. Alloys Compd.* **2011**, *509*, 2879–2884. [[CrossRef](#)]
26. Han, J.; Chen, J.; Peng, L.; Zheng, F.; Rong, W.; Wu, Y.; Ding, W.J. Influence of processing parameters on thermal field in Mg-Nd-Zn-Zr alloy during friction stir processing. *Mater. Des.* **2016**, *94*, 186–194. [[CrossRef](#)]
27. Chen, C.; Chen, J.; Yan, H.; Su, B.; Song, M.; Zhu, S. Dynamic precipitation, microstructure and mechanical properties of Mg-5Zn-1Mn alloy sheets prepared by high strain-rate rolling. *Mater. Des.* **2016**, *100*, 58–66. [[CrossRef](#)]
28. Du, Y.; Zheng, M.; Jiang, B.; Zhou, K. Deformation-Induced Dynamic Precipitation and Resulting Microstructure in a Mg-Zn-Ca Alloy. *JOM* **2018**, *70*, 1611–1615. [[CrossRef](#)]
29. Guo, F.; Zhang, D.; Yang, X.; Jiang, L.; Pan, F. Strain-induced dynamic precipitation of Mg₁₇Al₁₂ phases in Mg-8Al alloys sheets rolled at 748 K. *Mater. Sci. Eng. A* **2015**, *636*, 516–521. [[CrossRef](#)]
30. Luo, X.; Cao, G.; Zhang, W.; Qiu, C.; Zhang, D. Ductility Improvement of an AZ61 Magnesium Alloy through Two-Pass Submerged Friction Stir Processing. *Materials* **2017**, *10*, 253. [[CrossRef](#)] [[PubMed](#)]
31. Wang, B.; Pan, F.; Chen, X.; Guo, W.; Mao, J. Microstructure and mechanical properties of as-extruded and as-aged Mg-Zn-Al-Sn alloys. *Mater. Sci. Eng. A* **2016**, *656*, 165–173. [[CrossRef](#)]
32. Han, J.; Chen, J.; Peng, L.; Tan, S.; Wu, Y.; Zheng, F.; Hong, Y. Microstructure, texture and mechanical properties of friction stir processed Mg-14Gd alloys. *Mater. Des.* **2017**, *130*, 90–102. [[CrossRef](#)]
33. Darras, B.; Kishta, E. Submerged friction stir processing of AZ31 magnesium alloy. *Mater. Des.* **2013**, *47*, 133–137. [[CrossRef](#)]
34. Yuan, W.; Mishra, R.S. Grain size and texture effects on deformation behavior of AZ31 magnesium alloy. *Mater. Sci. Eng. A* **2012**, *558*, 716–724. [[CrossRef](#)]

© 2018 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>).