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Manufacturing of fine-grained AZ80 surface composites by friction stir processing: A review

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ABSTRACT

This review paper deals with the friction stir processing (FSP) for manufacturing of metal-matrix composites (MMCs) based on the Mg-8Al-0.5Zn (AZ80) magnesium alloy. Accordingly, the reported investigations on the AZ80 surface composites with various reinforcements, including the carbon nanotubes (CNTs), high-entropy alloys (HEAs), hybrid reinforcements, and composite plates, were summarized. Accordingly, the effects of introducing the second-phase particles, tool rotation rate, tool traverse speed, tool design, and vibration were considered. In this regard, the grain refinement by dynamic recrystallization (DRX) and Zener pinning effect, the uniformity of the particle distribution and avoiding agglomeration, and improvement of mechanical properties and wear behavior were critically discussed. Moreover, useful suggestions for future works were proposed, including focusing on the common reinforcing phases and processing parameters, tensile testing to evaluate the strength-ductility balance, metal additive manufacturing processes, and the correlation of DRX grain size with the Zener–Hollomon parameter similar to hot deformation studies.

Keywords: *Surface composites; Friction stir processing; Severe plastic deformation; Thermomechanical processing; Mechanical properties.*

1. Introduction

The lack of proper ductility in pure magnesium is attributed to its hexagonal close-packed (HCP) crystal structure, which has limited slip systems [1,2]. Additionally, the lower strength of Mg alloys compared to other alloys is a major barrier to their use in many applications [3,4]. Alloying [5-9], heat treatment [10-12], thermomechanical processing at elevated temperatures [13-16], severe plastic deformation (SPD) [17-19], and adding reinforcement particles [20-23] can help addressing these limitations.

The last method is used to develop the Mg-based composites, which is an efficient method to improve mechanical properties in terms of strength and stiffness (Young modulus). The Mg-based composites can be processed by different routes in the liquid-state and solid-state. A more recent solid-state process for processing of Mg-based composites is the friction stir processing (FSP), as described in the following.

Using the same principles as friction stir welding (FSW), FSP is considered a useful SPD method for grain refinement by dynamic recrystallization (DRX) [24-29], enhancing the distribution of particles, improving mechanical properties, and processing superplastic microstructures. During FSP, the surface of the workpiece is penetrated by pushing a rotating tool into it. Then, via moving it in a linear direction, the surface of the material can be processed. This tool includes a shoulder and a centrally positioned projecting pin (probe) [30-32]. In FSP, the material is locally heat-softened by the friction between the tool and workpiece and by auxiliary adiabatic heating from the concurrent plastic deformation. Accordingly, the material experiences severe straining at elevated temperatures, which leads to the microstructural refinement via processes such as DRX and fragmentation and dispersion of the particles [33-35].

FSP is also a useful technique for manufacturing surface composites [36-39], in which second-phase particles are incorporated into the surface through narrow grooves or drilled holes prior to FSP, as illustrated in Figure 1. Following the insertion of particles into the grooves or holes, an additional sealing step can be performed using a pinless tool in order to prevent the particles from escaping during the main FSP process. Moreover, the direct FSP is another technique for processing of surface composites, in which the reinforcement particles are introduced to the enclosed space between the shoulder of a pinless tool and the base metal through a hole provided within the FSP tool, followed by pressing them into the workpiece as the rotating tool advances.

According to Figure 1, a thermomechanically affected zone (TMAZ) and a heat-affected zone (HAZ) will form from the stir zone (SZ) to the base metal (BM) [40,41]. The main processing parameters are tool shape, rotation speed (ω, rpm), and traverse speed (v, millimeters per minute). An increase in ω or a decrease in v typically results in an increase in temperature. Accordingly, decreasing ω while keeping v constant or increasing v while keeping ω constant can refine the grain size. However, sufficient heating is always necessary in order to create a flawless nugget [36].

Among the Mg alloy matrices, the Mg–Al–Zn alloys (AZ series) are more common. The formation of α-Mg/$Mg_{17}Al_{12}$ eutectics in these alloys can deteriorate their properties, especially at high Al contents [42]. Accordingly, processing of these alloys by FSP brings about several advantages. First, partial dissolution of the $Mg_{17}Al_{12}$ phase due to the increased temperature can increase the solute Al to improve the solid solution strengthening effect [43]. Second, the fragmentation and dispersion of the remaining $Mg_{17}Al_{12}$ particles can amend their deleterious effects [44-46]. The severe straining during FSP can lead to the fragmentation of large particles to smaller ones, increasing the number of particles; while the material flow via the rotation of the pin can lead to the good dispersion of these particles in the microstructure. Third, the remarkable grain refinement by DRX boosts the properties in terms of strength-ductility balance [47]. Regarding the restoration processes, it should be noted that Mg has a relatively high stacking fault energy (SFE), and hence, the dynamic recovery (DRX) seems to be only major restoration mechanism during hot working of Mg alloys and the occurrence of DRX is not expected. However, it appears that the limited number of slip systems that can operate as a consequence of its HCP structure result in DRX occurring readily [27,48]. Accordingly, DRX can operate as a grain refining method during elevated-temperature

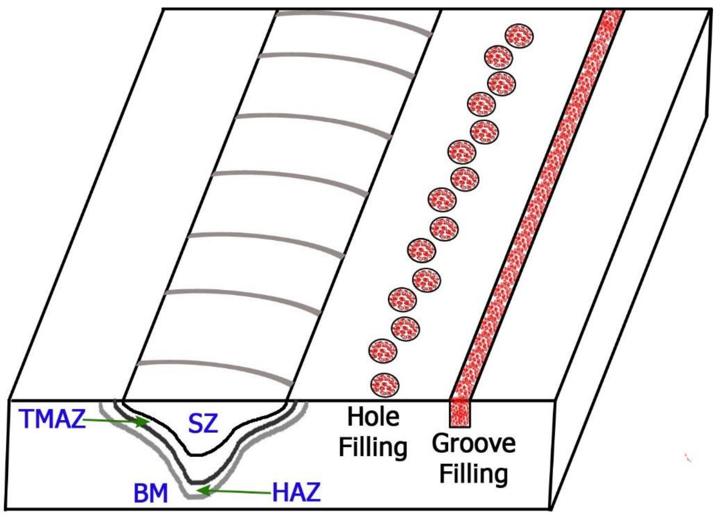

Fig. 1- Processing of surface composites by FSP through grooves or holes, as well as a schematic representation of the formation of different regions during FSP.

thermomechanical processing of Mg alloys.

The AZ80 alloy is a common one in the AZ series. While the processing of fine-grained AZ80 alloys by FSP has been extensively studied, the manufacturing of AZ80 composites by FSP has recently been investigated with quite promising outcomes. Accordingly, the current state-of-the-art in this field should be discussed and summarized. Therefore, the present work summarizes the reported works on the FSP of AZ80 composites, and then, the key suggestions for future works will be indicated. The recently studied AZ80 composites by FSP can be categorized as AZ80/carbon nanotubes (CNTs) surface composites [49,50], hybrid surface composites [51-53], surface composites reinforced by high-entropy alloys (HEAs) [54], and composite plates [55], which are summarized in the following sections.

2. AZ80/CNT composites by FSP

The CNTs are among the common reinforcements for processing of Mg-based composites by FSP. In this regard, there are several reports on the AZ31 [56-58] alloy from the category of Mg-Al-Zn alloys. Recently, Pushpanathan et al. [49] and Chen et al. [50] have investigated the processing of AZ80/CNT composites by FSP.

Pushpanathan et al. [49] investigated the effect of FSP process parameters on the microstructure and microhardness of the processed zone of AZ80 alloy with the addition of multiwalled CNTs (MWCNTs) via the drilled holes. Microstructural examination of the processed zones performed using optical microscopy and the microhardness of the processed zone was assessed using Vickers microhardness tester. The ω values of 1200 and 1400 rpm and v values of 10 and 30 mm/min were used to process the samples. The multipass approach was used and the samples after third FSP pass were considered. The results of hardness measurements are depicted in Figure 2.

It is observable that the highest microhardness was obtained for the high v value of 30 mm/min and low ω value of 1200 rpm (1200-30 sample); while the generation of additional heat for the low v value of 10 mm/min and high ω value of 1400 rpm (1400-10 sample) resulted in the decreased microhardness due to the coarsening of the DRX grains. Moreover, FSP resulted in a homogeneous distribution of the MWCNTs [49], which is an important aspect in the manufacturing of high-performance composites [59]. These factors contributed to the strengthening of the alloy [49].

It can be concluded that the grain refinement during FSP is quite remarkable and highly depends on the processing parameters such as v and ω, as shown in Figure 2. This is related to the dependency of the deformation temperature (T) and strain rate ($\dot{\varepsilon}$) during FSP on these parameters (Equation 1) [60-63]:

$$\frac{T}{T_m} \approx K\left(\frac{\omega^2}{v \times 10^4}\right)^{\alpha}, \dot{\varepsilon} \approx \pi\omega \frac{R_{nugget}}{D_{nugget}} \tag{1}$$

where K (ranging from 0.65 to 0.75) and α (ranging from 0.04 to 0.06) are constants and T_m is the melting point of the material. Moreover, R_{nugget} and D_{nugget} are the average radius and depth of the dynamically recrystallized zone. It is well-known that the DRX grain size (d) can be correlated to temperature and strain rate via the Zener–

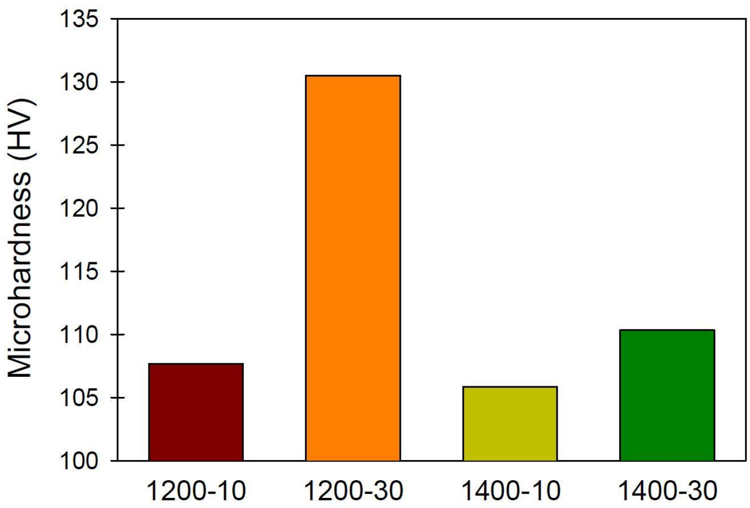

Fig. 2- Microhardness of the processed zone along weld direction for the FSP-manufactured AZ80/MWCNTs composites (data from [49]).

Hollomon parameter (Z) [64-66]:

$$d = AZ^{-p}, Z = \dot{\varepsilon}\exp(Q/RT) \quad (2)$$

where A and p are constants and Q is the deformation activation energy. Based on Equation 2, by increasing Z, the DRX grain size decreases, which is conducive to improve the mechanical properties. These aspects need to be further explored by obtaining the parameters of these equations as well as investigating the mechanical properties (both strength and ductility) by the tensile testing method.

In another study, Chen et al. [50] processed MWCNTs-reinforced AZ80 composites by FSP. A modified X53K milling machine was used to conduct the FSP experiments. After adding varying amounts of MWCNTs to fill the drilled holes, two plates with opposite directions were added together for FSP. This process was repeated five times to ensure uniform distribution of MWCNTs in the matrix. For the 3.1 vol% MWCNTs, the MWCNTs were uniformly distributed in the matrix, and no obvious agglomeration was detected. However, by increasing the MWCNTs content to 4.6 and 7.1 vol%, the agglomeration tendency increased. This was ascribed to the decrease in the frictional heat between the tool and workpiece due to lubricating action of MWCNTs, which reduced the plastic flow during FSP, resulting in the agglomeration of the MWCNTs. Since MWCNTs possess high electrical conductivity, a considerable enhancement in the electrical conductivity was expected. However, the agglomeration of the MWCNTs and the increased density of grain boundaries due to the grain refinement by FSP decreased the electrical conductivity, as summarized in Figure 3 [50], negating the usefulness of the FSP-manufactured AZ80/MWCNTs composites in this regard.

Accordingly, characterizing the functional properties of the AZ80/CNTs composites by FSP need further investigations, in which the agglomeration of the MWCNTs should be controlled to boost both mechanical and functional properties.

3. Hybrid AZ80 composites by FSP

The processing of hybridized surface composites by FSP has received considerable attention in recent years [67-71]. Regarding the AZ80 surface composites, there are several reports published by Paidar and coworkers [51-53]. For instance, in a comprehensive investigation, Ma et al. [51] manufactured a hybrid AZ80/CeO_2+ZrO_2 composite via FSP using a narrow groove on the surface. The groove was packed with CeO_2+ZrO_2 particles after being securely clamped for the FSP procedure. Then, a tool without a probe was used to fill the groove. Then, FSP using two types of tools, cylindrical pin tool (CPT) and tapered pin tool (TPT), was applied for creating the hybrid composite at v value of 80 mm/min and high ω value of 1400 rpm. Then, Vickers hardness measurements (load of 50 gf), pin-on-disk wear test (applied force of 40 N, rotating speed of 26 rpm, and sliding velocity of 35 cm/s), and shear punch testing (SPT) with a strain rate of 0.001 s^{-1} were used for characterization of the processed alloy. As shown in Figure 4a, using the TPT tool resulted in the removal the flow defects and modification of the nugget shape, vortex width, and surface flash. From the microstructural standpoint,

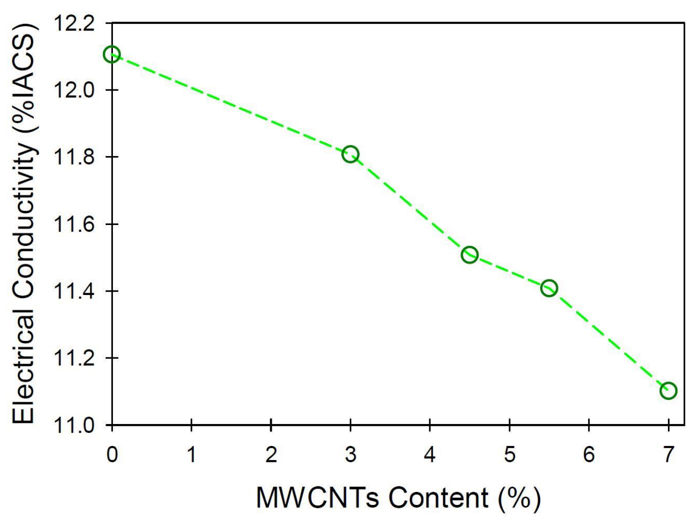

Fig. 3- Effect of MWCNTs content on the electrical conductivity of FSP-manufactured AZ80/MWCNTs composites (data from [50]).

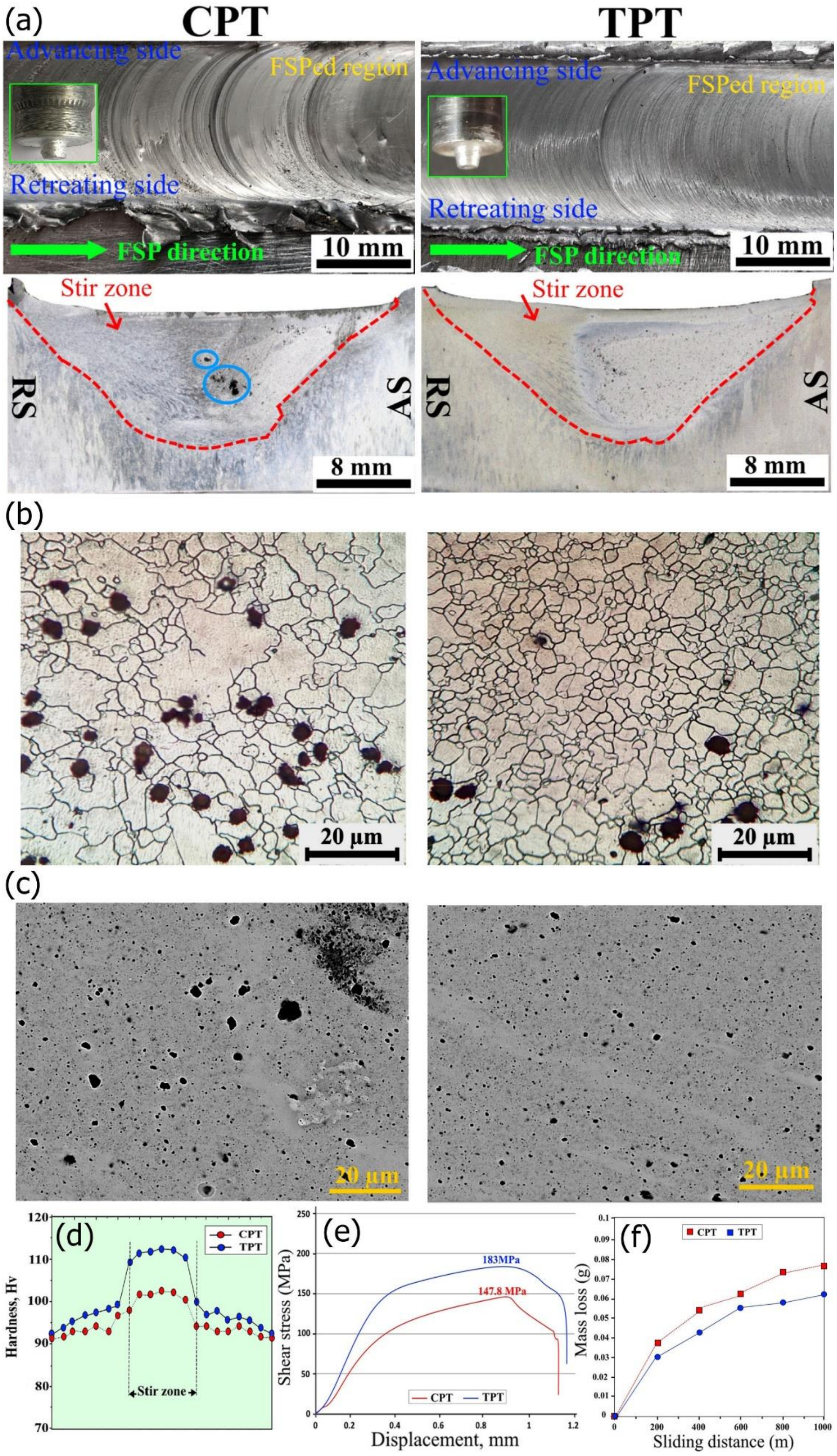

Fig. 4- Hybrid AZ80 Mg/CeO_2+ZrO_2 composite via FSP: (a) surface and cross-sectional views, (b,c) microstructures, (d) hardness, (e) SPT results, and (f) mass loss during wear test [51].

Figure 4b reveals that the change of the tool from CPT to TPT refined the average grain size from 7.48 μm to 5.72 μm due to enhanced tool-assisted plastic deformation and flow, DRX, and the Zener pinning effect. Moreover, the particle clustering was eliminated, as shown in Figure 4c.

As shown in Figure 4d, change of the tool from CPT to TPT resulted in the improved hardness of the processed zone, which was subsequently confirmed by the higher ultimate shear strength in the shear punch tests (Figure 4e). This can be related to the better grain refinement, elimination of defects, and more appropriate particle distribution for the composite processed by the TPT tool. These factors also played a main role in the improved wear resistance, as shown in Figure 4f [51].

In a related study, Wei et al. [52] also processed a hybrid AZ80/CeO_2+BN composite via FSP with similar outcomes, which proved the importance of the tool design. It was revealed that by changing the pin shape from cylindrical to conical, flow problems during FSP and particle crushing were reduced and microstructural refinement was enhanced, where the average grain size of the composite refined from 6.47 to 4.08 μm. Moreover, the enhanced hardness, wear resistance and shear punch strength were achieved.

In another investigation, Mao et al. [53] assessed the impact of the vibration during FSP for processing of AZ80/MnO+ZrO_2 composite. It was revealed that using vibration during FSP effectively enhances the material flow, results in the enlargement of the stir zone, and leads to a more uniform distribution of reinforcement particles. It was also discovered that vibration positively influenced the grain refinement and could improve dislocation density, which boosted wear performance and shear properties.

These investigations revealed the positive effect of FSP on the microstructural refinement and improved properties of the hybrid AZ80 composites. However, the characterization of the mechanical properties by the tensile testing method has remained to be investigated to unravel the effect of FSP and its processing variables on the strength-ductility balance.

4. Composites reinforced by high-entropy alloys

Due to their unique properties, the HEAs have received a considerable attention by the materials science community [72-75]. In this regard, the incorporation of HEAs as the reinforcement particles for other alloys is one of the most sought-after topics [76,77]. Accordingly, the processing of composites reinforced by HEAs is a hot topic, where there is a recent report on the AZ80 composites reinforced by HEAs [54].

Guo et al. [54] used the friction stir vibration processing (FSVP), which is known as an improved and efficient FSP method for the processing of magnesium alloys and composites [78-80]. This fact can simply be evaluated based on Equation 1, as Bagheri et al. [80] have noted that R_{nugget} for FSVP due to the application of vibration is larger than that for FSP. Accordingly, the strain rate and the Z are higher, which is favorable for grain refinement according to Equation 2. In this regard, the effects of vibration and AlCrFeMoNb HEA particles on the microstructure, hardness, shear strength, and wear properties of a novel FSP-manufactured AZ80/AlCrFeMoNb composite was investigated by Guo et al. [54]. After the HEA powders were poured into the grooves, a FSP tool without a pin was used to seal the top surface and prevent the powders from spilling out. An H13 steel tool with a cylindrical shape, featuring a pin length of 4 mm, a pin diameter of 6 mm, and a shoulder diameter of 18 mm, was used for performing FSP and FSVP at v of 100 mm/min and ω of 900 rpm. It was revealed that FSVP improves grain refinement and inhibits grain growth, resulting in the formation of equiaxed fine grains with a greater degree of recrystallization. Increased fragmentation of AlCrFeMoNb particles distributed in the matrix was noted for the FSVP route, resulting in a reduction of the average grain size in the stir zone. FSVP significantly enhanced the material flow in the stir zone, which in turn decreased the likelihood of particle aggregation and led to a uniform distribution of the AlCrFeMoNb particles [54]. Accordingly, applying vibration during FSP resulted in a significant enhancement of hardness, shear strength, and tribological properties.

5. Composite plates by FSP

The processing of composite plates by FSP is an interesting practice. Regarding the AZ80 plates, the processing of AZ80/Al composite plates has been reported by Liu et al. [55]. A pure aluminum plate with a thickness of 1.25 mm was polished with SiC paper, cleaned with acetone, and then, it was mounted on an AZ80 plate and fixed on the working table of a revamped milling machine. The FSP tool had a shoulder diameter of 12 mm, with a pin diameter of 3 mm and length of 1.6 mm. The processing parameters were ω in the range of 1800 to 1850 rpm, v of 20 mm/min, and plunge depth of 2.0 mm.

It was revealed that a composite layer of aluminum with fine grains as well as a small amount of Al_2Mg intermetallic compound at the Mg/Al interface form during FSP. The strong bond between the AZ80 substrate and 1060 pure Al layer was shown to be due to the metallurgical bonding achieved through FSP. The Al layer/AZ80 interface showed increased microhardness compared to surrounding areas, suggesting the presence of Al-Mg intermetallic compounds and Al-Mg solid solution. The electrochemical corrosion test in a 5 wt.% NaCl solution indicated that the composite Al layer provided superior protection for the AZ80 alloy in corrosive environments [55]. These results reveal the opportunities that the composite AZ80 plates processed by FSP can offer to control the properties.

6. Summary and future prospects

The present work summarized the reports on the FSP-manufactured AZ80 composites. It was revealed that the FSP is a viable approach for the processing of high-performance AZ80 composites. The available works have dealt with the CNTs [49,50], hybrid reinforcements [51-53], and HEAs particles [54], as well as the composite plates [55]. However, there are many common and effective particles that need to be investigated for the FSP-manufactured AZ80 composites, including SiC [81-84], Al_2O_3 [85-87], SiO_2 [88,89], B_4C [90,91], TiC/TiB_2 [92,93], ZrB_2 [94,95], hydroxyapatite [96,97], Ti alloys [98,99], Fly ash [100,101], graphene nanoplatelets (GNPs) [102,103], nanodiamonds [104,105], and other HEAs. These particles have widely been investigated for the composites based on the AZ31 and AZ91 alloys with quite promising outcomes. Accordingly, much more investigations on the manufacturing of AZ80 composites based on these particles are expected in the future. On the other hand, due to the favorable effects of hybrid reinforcements in enhancing the properties of AZ80 alloy, more research in this field is suggested. Currently, the CeO_2+ZrO_2 [51], CeO_2+BN [52], and MnO+ZrO_2 [53] hybrid composites have been studied. However, other combinations of reinforcement particles need to be investigated.

Another aspect that needs future investigations is the evaluation of the mechanical properties of FSP-manufactured AZ80 composites. For instance, the reported works have evaluated the mechanical properties mainly by the hardness and SPT methods. However, comprehensive investigations on the tensile properties of FSP-manufactured AZ80 composites is required to unravel the effect of FSP, introducing particles, and other factors on the strength-ductility balance. On the one hand, the tensile testing can be used to evaluate the improvement of Young modulus by the introduction of particles through FSP. On the other hand, the improvement of yield stress, ultimate tensile strength (UTS), and total elongation can be tracked, which is an important aspect. Therefore, it is expected that the FSP-manufactured AZ80 composites show superior mechanical properties compared to the composites processed by other methods.

Regarding grain refinement by DRX, the exact DRX mechanisms can be discussed in future works, including the discontinuous DRX (DDRX), continuous DRX (CDRX), and Twin-induced DRX (TDRX). Moreover, Equation 2 has been proposed for the grain refinement by DRX, which can be applied to the FSP investigations using Equation 1. In this way, the effect of the main processing parameters (ω and v) on the grain refinement efficiency can be formulated and quantified, which is an important aspect. Moreover, the properties, especially the mechanical properties, can be correlated to the obtained grain size by FSP. In this regard, the Hall-Petch relationship [106-108] can be used, which is applicable for the hardness, tensile properties, and shear properties. In this respect, there is no report on the FSP-manufactured AZ80 composites.

The effects of ω, v, vibration, and tool design have been investigated for the FSP-manufactured AZ80 composites, as described above. However, the effect of multi-pass FSP needs to be unraveled, which can significantly affect the microstructure and mechanical properties [109-111]. Another modification for grain refinement is the FSP accompanied by external cooling [112-115], which pertains to temperature adjustment and serves to prevent extensive grain growth [116-121] and the dissolution of precipitates within the stirred zone [122]. According to Patel et al. [123], the backing plate plays a crucial role—a copper backing plate proved to be more efficient than a steel backing plate in refining the grain structure of Mg alloys. On the other hand, because of the dissolution of the β-$Mg_{17}Al_{12}$ phase during FSP, the AZ80 alloy could exhibit a good aging response following FSP [124]. Moreover, the volume fraction of the reinforcement particles is one of the main variables of the composites [99,125], which needs to be investigated for the FSP-manufactured AZ80 composites.

Additive manufacturing, ideal for creating a variety of intricate shapes, relies on the sequential layering of thin materials derived from 3D models [35,126-128]. In addition to the most commonly utilized methods of powder bed fusion and directed energy deposition, metal additive manufacturing techniques based on the friction stir engineering have garnered significant interest. Friction stir additive manufacturing (FSAM) and additive friction stir deposition (AFSD) are two commonly utilized techniques for magnesium alloys. These methods are beneficial, particularly for producing defect-free Mg alloys and composites with a fine-grain structure and excellent mechanical/functional properties [130-136]. Accordingly, it is expected that future investigations deal with the FSAM and AFSD of AZ80 composites.

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