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Invited Review

Surface metal-matrix composites based on AZ91 magnesium alloy via friction stir processing: A review

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Abstract: This monograph presents an overview of friction stir processing (FSP) of surface metal-matrix composites (MMCs) using the AZ91 magnesium alloy. The reported results in relation to various reinforcing particles, including silicon carbide (SiC), alumina (Al₂O₃), quartz (SiO₂), boron carbide (B₄C), titanium carbide (TiC), carbon fiber, hydroxyapatite (HA), *in-situ* formed phases, and hybrid reinforcements are summarized. AZ91 composite fabricating methods based on FSP are explained, including groove filling (grooving), drilled hole filling, sandwich method, stir casting followed by FSP, and formation of *in-situ* particles. The effects of introducing second-phase particles and FSP process parameters (e.g., tool rotation rate, traverse speed, and the number of passes) on the microstructural modification, grain refinement, homogeneity in the distribution of particles, inhibition of grain growth, mechanical properties, strength–ductility trade-off, wear/tribological behavior, and corrosion resistance are discussed. Finally, useful suggestions for future work are proposed, including focusing on the superplasticity and superplastic forming, metal additive manufacturing processes based on friction stir engineering (such as additive friction stir deposition), direct FSP, stationary shoulder FSP, correlation of the dynamic recrystallization (DRX) grain size with the Zener–Hollomon parameter similar to hot deformation studies, process parameters (such as the particle volume fraction and external cooling), and common reinforcing phases such as zirconia (ZrO₂) and carbon nanotubes (CNTs).

Keywords: surface composites; magnesium alloys; friction stir processing; severe plastic deformation; thermomechanical processing

1. Introduction

Magnesium alloys and composites are used in various industries owing to their good specific strength, high damping capacity, good castability, and biodegradability [1–4]. They are considered to be the ultimate choice of lightweight metallic structural materials [5–6]. However, polycrystalline Mg is characterized by poor ductility due to the presence of a hexagonal close-packed (hcp) crystal structure with few slip systems. Moreover, the relatively lower strength of Mg alloys compared to other competing alloys is another major obstacle to their utilization in many potential applications [7]. These shortcomings can be relieved by alloying [8–9], heat treatment [10–11], elevated temperature thermomechanical processing [12–13], severe plastic deformation (SPD) [14], and introduction of reinforcement particles (Mg-based metal-matrix composites (MMCs)) [15–16].

Among SPD techniques, friction stir processing (FSP), which is based on the principles of friction stir welding (FSW), is a viable technique for material processing [17–18]. FSP is applied by pushing a rotating tool into the surface of the workpiece, followed by its translational movement. This nonconsumable tool consists of a cylindrical shoulder and a projecting concentrically located pin (probe). During FSP,

the material is locally softened due to the heat generated by the friction between the tool and workpiece, as well as through auxiliary adiabatic heating due to plastic deformation during the flow of the material [19]. FSP is normally used for microstructural enhancement (e.g., grain refinement by dynamic recrystallization [20–21] and altering the amount, morphology, and distribution of particles [22]), improvement of various properties (e.g., mechanical properties [23] and superplasticity [24]), and processing of composites [25]. FSP is an efficient method for processing surface composites [26], whereby the second-phase particles can be introduced to the surface via narrow grooves or drilled holes before FSP [27], as schematically shown in Fig. 1. After filling the grooves or holes with particles, an optional closing (covering) step can be carried out using a pinless FSP tool to avoid the escape of the particles during the subsequent main FSP step [28]. As shown in Fig. 1, from the stir zone (SZ, or nugget zone, NZ) to base metal (BM), a thermomechanically affected zone (TMAZ) and a heat-affected zone (HAZ) will develop [29]. The primary processing parameters include tool geometry, tool rotation rate (ω , r/min), and traverse/advancing/welding speed (v , mm/min) [30]. A higher ω or lower v normally leads to a higher temperature [31]. The grain size can be refined by decreasing ω at constant v or by

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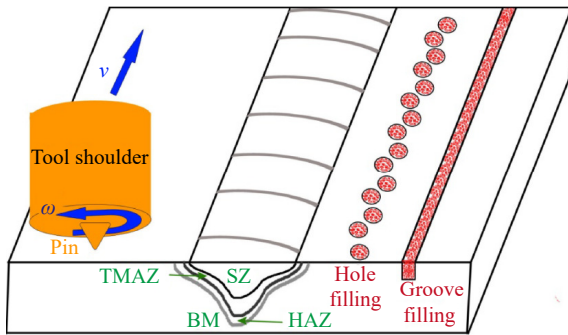


Fig. 1. Schematic representation of the surface composite fabrication by FSP.

increasing v at constant ω [32]. Anyway, adequate heating is always required to produce a defect-free nugget with a refined microstructure [33].

Another FSP-based composite fabrication method for Mg alloys is direct friction stir processing (DFSP) [34–36]. In the DFSP process, the secondary phase is *in-situ* 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 along them just like a planter (Fig. 2) [34]. This method has been successfully applied to AZ31/SiC composite [34]. Moreover, there are some promising and innovative modifications, such as friction stir welding with a pulse current [18]. Furthermore, the potential to use the stationary shoulder tool in FSP as a novel low-heat input tooling system for Mg alloys has been extensively studied by Patel *et al.* [37–39]. As shown in Fig. 3, a rotating tool (consisting of a probe with a small or no shoulder) is housed within a non-rotational shoulder (stationary shoulder)—the tool slides over the joint line during processing to eliminate or minimize the heat generated by the shoulder [39]. As a result, lower and more focused heat will be generated through the thickness [37]. Due to the stationary action of the shoulder, this process generates a smooth surface [40] with a small amount of flash onto the surface [41] and develops uniform grain refinement and, consequently, homogeneous mechanical properties throughout the thickness. In other words, the small temperature gradient across the thickness of the SZ in this process leads to the homogenization of the magnesium alloy microstructure [38].

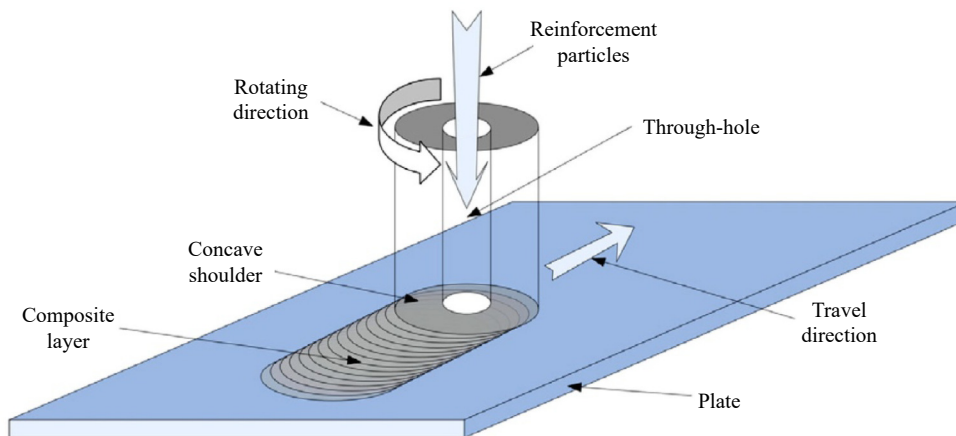


Fig. 2. Schematic representation of direct friction stir processing [34]. Reprinted from *Mater. Des.*, Vol. 59, Y.X. Huang, T.H. Wang, W.Q. Guo, L. Wan, and S.X. Lv, Microstructure and surface mechanical property of AZ31 Mg/SiC_p surface composite fabricated by direct friction stir processing, 274–278, Copyright 2014, with permission from Elsevier.

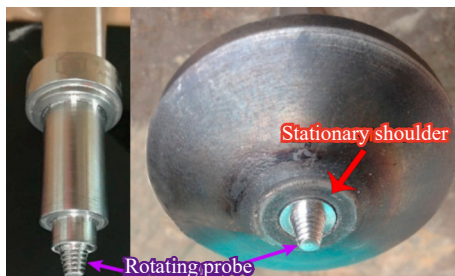


Fig. 3. Stationary shoulder tooling system [39].

The most widely used Mg alloys are based on the AZ series (Mg–Al–Zn) [42], among which the AZ91 (Mg–9Al–1Zn, wt%) is one of the famous alloys [43]. The presence of the eutectic structure (α -Mg/Mg₁₇Al₁₂) deteriorates the mechanical and functional properties of the AZ series Mg alloys [44]. The dissolution of the Mg₁₇Al₁₂ phase during el-

evated temperature processing increases the Al content of the matrix [45], which might result in a major solid solution strengthening effect [46]. Moreover, the fragmentation and dispersion of Mg₁₇Al₁₂ particles during processing reduce their deleterious effects. FSP can simultaneously apply SPD and elevated temperature thermomechanical processing in the solid state [46–47]. Accordingly, recrystallization processes can refine the microstructure and enhance the material properties; while dissolution, fragmentation, and dispersion of particles can effectively amend the adverse effects of intergranular eutectics in AZ91 alloy [48]. Accordingly, FSP can be considered a viable processing method for AZ91 alloy [49]. The processing of fine-grained and high-performance AZ91 composites by FSP is fairly easy. Accordingly, the present overview article is dedicated to summarizing reported works on the FSP of AZ91 composites and indicating key suggestions for future works.

2. AZ91 composites fabricated by FSP

2.1. AZ91–SiC composites

AZ91–SiC (silicon carbide) composites are among the most widely investigated ones. Asadi *et al.* [50] fabricated composite layers on the surface of as-cast AZ91 Mg alloy using nanosized (30 nm) and micron-sized (5 μm) SiC particles (using groove filling and covering method). With the application of FSP, a fine microstructure was obtained with the dissolution of the eutectic $\text{Mg}_{17}\text{Al}_{12}$ phase in the SZ for as-cast AZ91 Mg alloy and composites, as shown in Fig. 4(a). The

addition of SiC led to more intense grain refinement and enhancement of hardness, where these effects were more pronounced for nanosized SiC particles compared to the micron-sized SiC particles, as shown in Fig. 4(b) and (c). Moreover, lower ω and/or higher v led to a finer grain size and greater hardness, as shown in Fig. 4(b) and (c) [50]. The dependence of the grain size on the FSP parameters has also been reported for AZ91/SiC surface composite (using hole filling or multichamber technology) by Iwaszko *et al.* [51]. In another study, Asadi *et al.* [52] showed that the repetition of the FSP passes leads to more pronounced grain refinement and en-

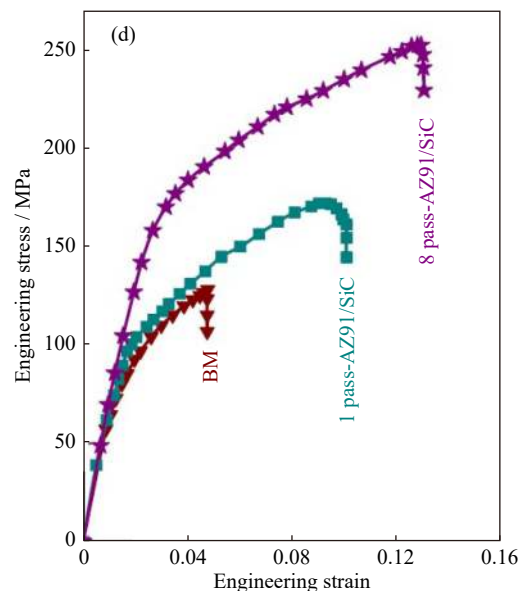
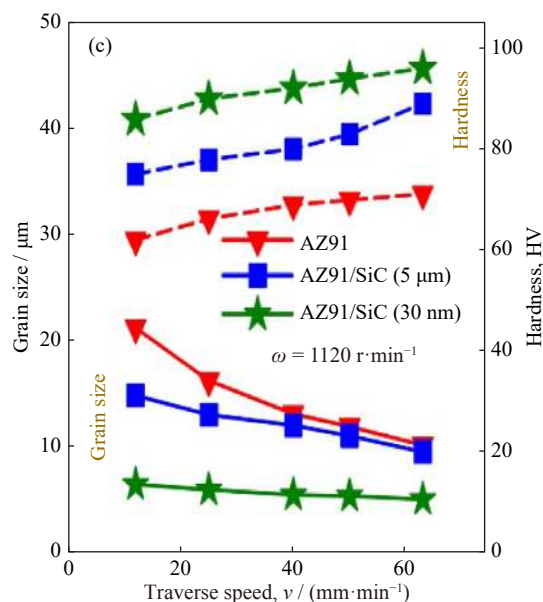
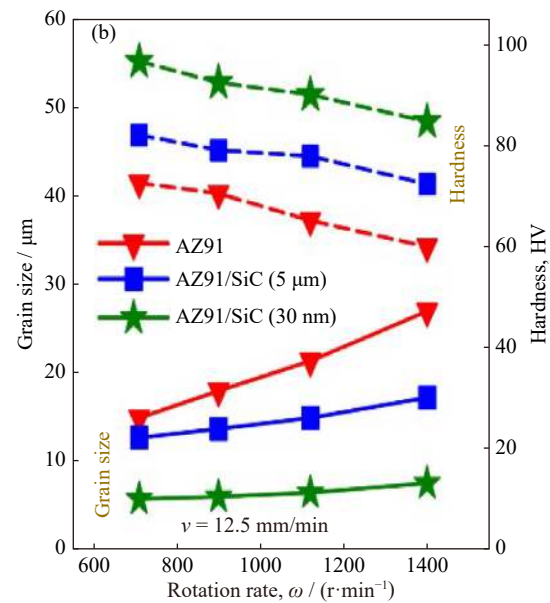
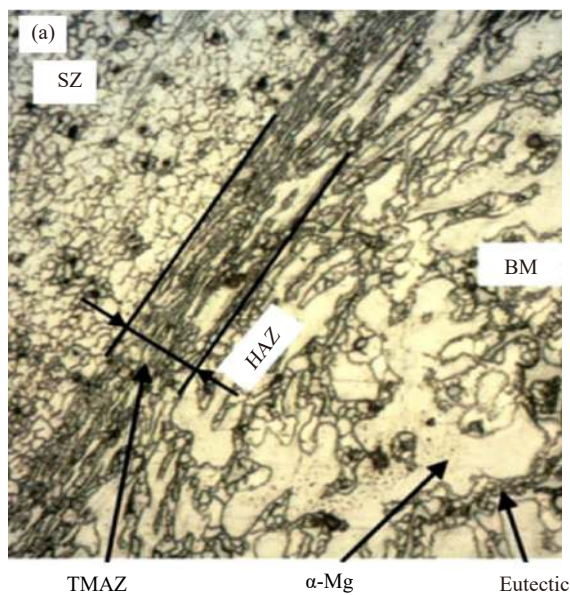


Fig. 4. (a) Representative FSP microstructure, (b, c) dependence of stir zone grain size and hardness on the FSP parameters, and (d) tensile stress–strain curves of FSP AZ91/SiC surface composites (the curves are redrawn) [50,52]. (a–c) Adapted by permission from Springer Nature: *J. Mater. Eng. Perform.*, Effects of SiC particle size and process parameters on the microstructure and hardness of AZ91/SiC composite layer fabricated by FSP, P. Asadi, M.K.B. Givi, K. Abrinia, M. Taherishargh, and R. Salekrostam, Copyright 2011; (d) adapted by permission from Springer Nature: *Metall. Mater. Trans. A*, Experimental investigation of magnesium-base nanocomposite produced by friction stir processing: Effects of particle types and number of friction stir processing passes, P. Asadi, G. Faraji, A. Masoumi, and M.K.B. Givi, Copyright 2011.

hanced mechanical properties (Fig. 4(d)), a finding that was confirmed by Dadaei *et al.* [53] who used the groove filling and covering method.

Bagheri *et al.* [54] proposed friction stir vibration processing (FSVP) as an improved and efficient FSP method for the processing of magnesium alloys and composites. During the process, the workpiece is vibrated in a direction that is normal to the tool translational direction, as shown in Fig. 5(a). The FSP and FSVP techniques were used to process AZ91/SiC surface composites using the groove filling and covering method. A more homogeneous distribution of SiC particles was observed for FSVP compared to FSP. In the FSVP process, workpiece vibration resulted in higher plastic strain in the material, hence promoting dynamic recrystallization. Consequently, finer grains were developed when using FSVP than when using FSP (Fig. 5(b)), indicating that FSVP results in better mechanical properties, as depicted in Fig. 5(c).

Eq. (1) can be used to correlate the dynamically recrystallized grain size (d) with the Zener–Hollomon parameter (Z , Eq. (2)), where A and B are constants, and R is the gas constant. The deformation activation energy (Q) is normally considered to be the activation energy for lattice self-diffusion [55–57]. The deformation temperature (T) and strain rate

($\dot{\epsilon}$) during FSP can be estimated by Eq. (3) [58–59] and Eq. (4) [59–60], where T_m is the absolute melting point of the material; K and α are constants; R_{nugget} and D_{nugget} are the effective (or average) radius and depth of the dynamically recrystallized zone [58–62]. These formulas might also be applied when discussing the effects of modification in the FSP processes. For instance, Bagheri *et al.* [63] have noted that R_{nugget} for the FSVP (Fig. 5(a)) is larger than that for FSP (due to vibration). Accordingly, the strain rate and the Z parameter are higher in FSVP, which is favorable for grain refinement.

$$d = AZ^{-B} \tag{1}$$

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \tag{2}$$

$$\frac{T}{T_m} \approx K \left(\frac{\omega^2}{v \times 10^4}\right)^\alpha, \quad 0.04 \leq \alpha \leq 0.06, \quad 0.65 \leq K \leq 0.75 \tag{3}$$

$$\dot{\epsilon} \approx \pi \omega \frac{R_{nugget}}{D_{nugget}} \tag{4}$$

Another modification for grain refinement is the FSP with external cooling [64–66], which is related to temperature modification and is used to inhibit extensive grain growth and dissolution of precipitates in and around the stirred zone [19]. As shown by Patel *et al.* [67], the backing plate is also

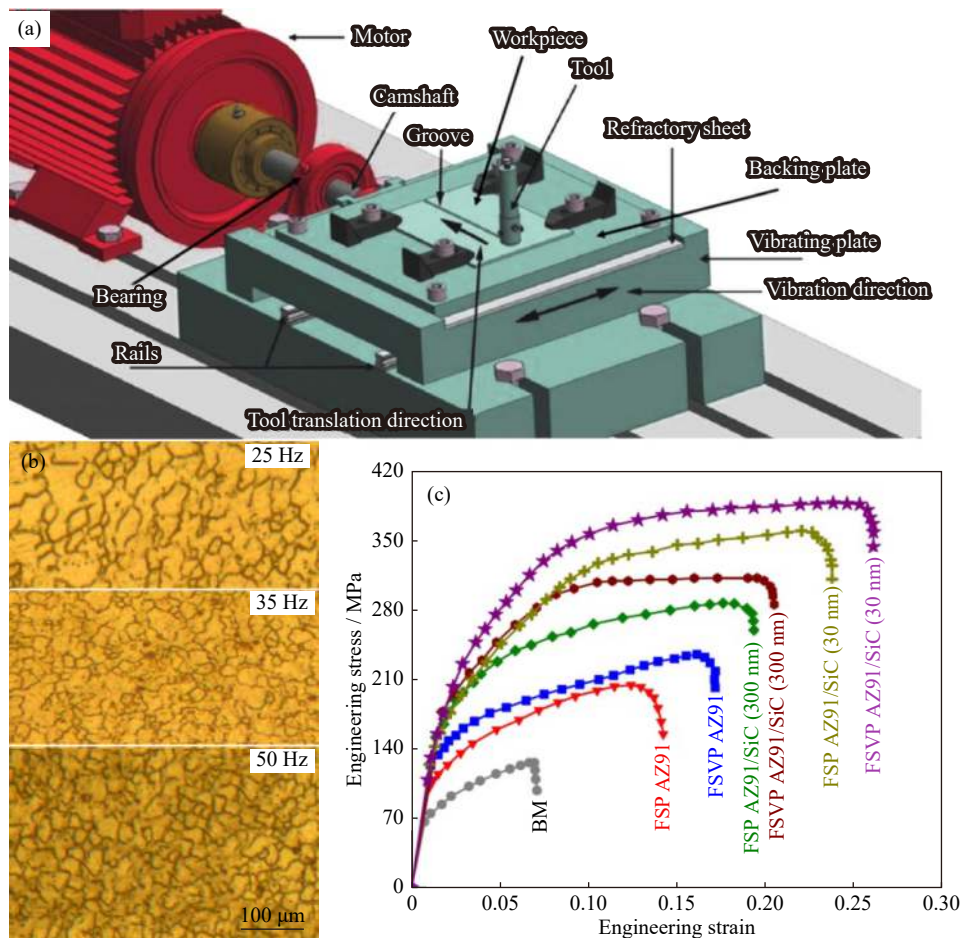


Fig. 5. (a) Schematic representation of FSVP, (b) the effect of vibration frequency on FSVP microstructure, and (c) tensile stress–strain curves of AZ91/SiC surface composites (the curves are redrawn) [54]. Reprinted from *Trans. Nonferrous Met. Soc. China*, Vol. 30, B. Bagheri, M. Abbasi, A. Abdollahzadeh, and S.E. Mirsalehi, Effect of second-phase particle size and presence of vibration on AZ91/SiC surface composite layer produced by FSP, 905-916, Copyright 2020, with permission from Elsevier.

important—a copper backing plate was found to be more effective than a steel backing plate for grain refinement of Mg alloys.

Similar to the results of Asadi *et al.* [50], Bagheri *et al.* [54] also found finer SiC particles to be more conducive to obtaining better mechanical properties. Accordingly, high-performance AZ91 composites can be processed through the addition of nanosized particles combined with FSVP. The favorable effect of increasing vibration frequency on grain refinement can be seen in Fig. 5(b) [54].

Due to the dissolution of the β -Mg₁₇Al₁₂ phase during FSP, the AZ91 alloy might have the next best aging response after FSP. Accordingly, Shang *et al.* [68] examined the aging behavior of FSP AZ91 alloy and the AZ91/SiC composite (50 nm, based on the hole-filling method). With the addition of nanosized SiC particles [69], a more intense grain refine-

ment after FSP was observed (Fig. 6(a)). However, while a higher hardness level was obtained, the age hardening efficiency became inferior for AZ91/SiC composite compared to AZ91 alloy (Fig. 6(b)) [68].

The discontinuous precipitation of the β -phase that dominated the FSP AZ91 alloy was significantly restricted in the composite, and the nanosized SiC particles promoted continuous precipitation of the β -phase. While there was no significant difference in the amounts of precipitated β -phase between the FSP AZ91D and AZ91/SiC (the formation of precipitates during aging can be seen in the X-ray diffraction (XRD) patterns of Fig. 6(c)), the different precipitation behaviors and different strengthening mechanisms were found to be responsible for the difference in aging responses, for which the particle size and distribution might play decisive roles [68].

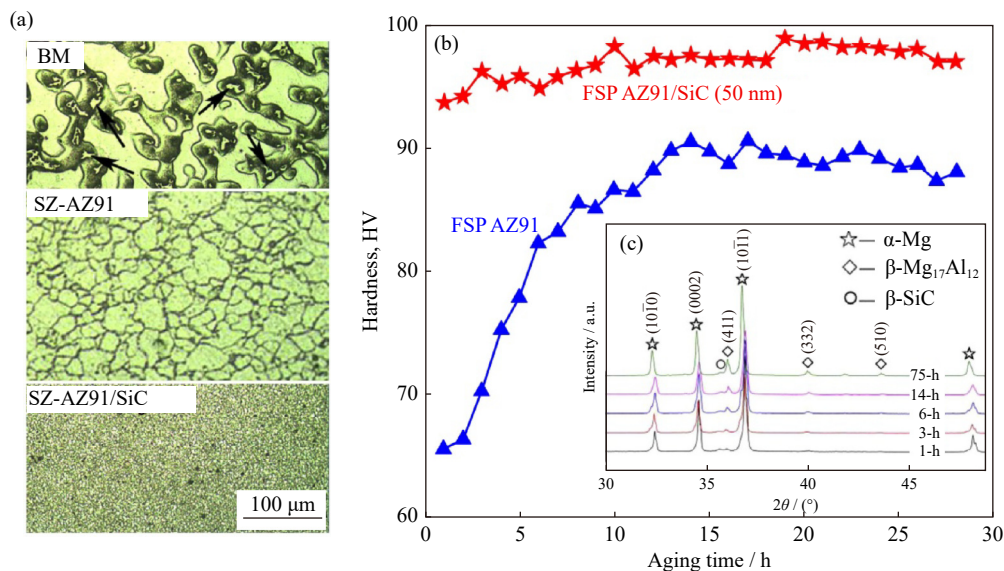


Fig. 6. (a) Representative optical micrographs (the arrows denote the β -Mg₁₇Al₁₂ phase), (b) aging responses of AZ91 alloy and AZ91/SiC composite at 180°C, and (c) XRD patterns of AZ91/SiC composite at different aging times at 180°C [68]. Reprinted from *J. Alloys Compd.*, Vol. 797, J.L. Shang, L.M. Ke, F.C. Liu, F.Y. Lv, and L. Xing, Aging behavior of nano SiC particles reinforced AZ91D composite fabricated via friction stir processing, 1240-1248, Copyright 2019, with permission from Elsevier.

Chen *et al.* [70] investigated the effects of FSP and SiC addition (using the groove filling method) on thixoformed AZ91 alloy. A uniform distribution of SiC particles was obtained via FSP (Fig. 7(a)). The alloy with a composite surface showed higher hardness and wear resistance and lower friction coefficient as compared to the permanent mold cast and the thixoformed alloy without a composite surface, as shown in Fig. 7(b). The composite surface showed good tribological properties due to the strengthening roles played by the particles.

Lee *et al.* [71] also showed the enhancement of wear resistance in AZ91/SiC composites through FSP processing. In another study, Abbasi *et al.* [72] reported that by applying more FSP passes during the processing of AZ91/SiC surface composite, the mechanical properties improved, corrosion resistance increased, and the wear rate decreased. More recently, Abdollahzadeh *et al.* [73] reported the improvement

of wear and corrosion resistance of AZ91/SiC composite layer processed by FSVP compared to FSP.

2.2. AZ91–Al₂O₃ composites

Faraji *et al.* [74–75] fabricated composite layers on the surface of as-cast AZ91 Mg alloy using nanosized Al₂O₃ (alumina) particles (30 nm, using the groove filling method). Fig. 8(a) shows the typical surface appearance of processed composites, in which defects such as voids and cracks cannot be observed [75]. By increasing v at a constant ω of 900 r/min, the grain size was refined, and the hardness increased. Accordingly, the optimum condition for producing a sound and fine-grained surface layer was characterized as $\omega = 900$ r/min and $v = 80$ mm/min. More recently, Ahmadkhaniha *et al.* [76] processed AZ91/Al₂O₃ surface composites using nanosized Al₂O₃ particles (50 nm, using the groove filling and covering method), and the optimum values of $\omega = 800$

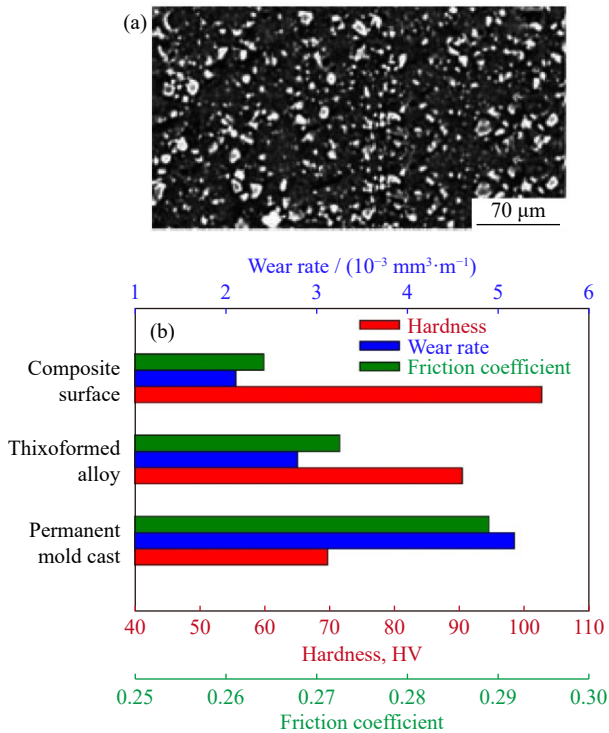


Fig. 7. (a) Scanning electron microscopy (SEM) image of the AZ91/SiC surface composite processed by FSP and (b) the comparison of its properties with AZ91 alloy processed by other techniques [70]. Adapted by permission from Springer Nature: *J. Wuhan Univ. Technol. Mater. Sci. Ed.*, Friction stir processing of thixoformed AZ91D magnesium alloy and fabrication of surface composite reinforced by SiC_ps, T.C. Chen, Z.M. Zhu, Y. Ma, Y.D. Li, and Y. Hao, Copyright 2010.

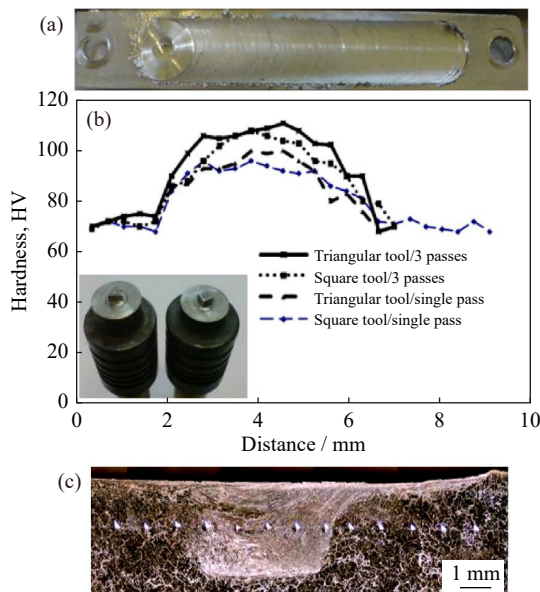


Fig. 8. (a) Top face of the specimen produced by FSP; (b) effect of tool geometry and number of passes on hardness profile (inset shows the used FSP tools); (c) image of the processed region (indicating the indentation locations) [75]. Reprinted by permission from Springer Nature: *J. Mater. Eng. Perform.*, Effect of process parameters on microstructure and micro-hardness of AZ91/Al₂O₃ surface composite produced by FSP, G. Faraji, O. Dastani, and S.A.A. Mousavi, Copyright 2011.

r/min and $v = 40$ mm/min were suggested for obtaining the highest hardness and wear resistance. The authors also proposed the Hall–Petch-type formula that relates hardness (H , HV) to grain size (d , μm), i.e., $H = \frac{55.88}{\sqrt{d}} + 67.453$. Faraji *et al.* [75] also investigated the effects of Al₂O₃ particle size, tool geometry, and repetition of FSP passes. It was found that decreasing the size of Al₂O₃ particles leads to more intense grain refinement and an increase in hardness. Moreover, a higher number of FSP passes using triangular tool geometry can produce a harder surface composite, as shown in Fig. 8(b) and (c). In fact, the triangular tool was more effective compared to the square tool. Increasing the number of passes also enhanced the homogeneity and particle distribution and resulted in a more refined grain size distribution [75].

The AZ91/Al₂O₃ and AZ91/SiC surface composites processed by FSP have been compared by Asadi *et al.* [52], Dadaei *et al.* [53], and Abbasi *et al.* [72]. These studies generally demonstrate that the microstructure of the composite layer created by SiC particles is characterized by finer grains, as well as higher hardness, strength, elongation, corrosion resistance, and wear resistance compared to the composite layer by Al₂O₃ particles. Polarization curves comparing corrosion resistance between AZ91/Al₂O₃ and AZ91/SiC surface composites are shown in Fig. 9 [72], which depict that the AZ91/SiC surface composite has a lower corrosion current density i_{corr} (as can be obtained based on the Tafel extrapolation method [77]) and more positive corrosion potential E_{corr} . By investigating the strength/weight ratios of as-cast and FSP AZ91 alloys, as well as AZ91/Al₂O₃ and AZ91/SiC composites, Dadaei *et al.* [53] proved the positive impact of FSP and significant enhancements for composites at higher FSP passes. Therefore, it can be deduced that the desirable

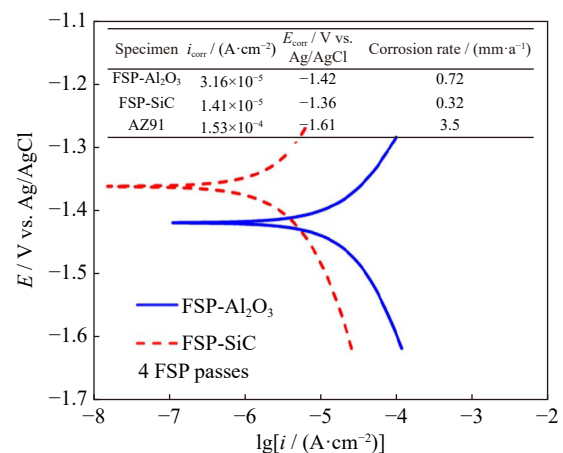


Fig. 9. Polarization curves for FSP AZ91 alloys and composites (the inserted table shows the potentials, current densities, and corrosion rates obtained by Tafel extrapolation analyses of both the FSPed specimens and the original AZ91 alloy) [72]. Reprinted by permission from Springer Nature: *Int. J. Adv. Manuf. Technol.*, The effect of FSP on mechanical, tribological, and corrosion behavior of composite layer developed on magnesium AZ91 alloy surface, M. Abbasi, B. Bagheri, M. Dadaei, H.R. Omidvar, and M. Rezaei, Copyright 2015.

effects of particle addition can be achieved after several FSP passes.

2.3. AZ91–SiO₂ composites

Vignesh *et al.* [78] fabricated a composite layer on the surface of AZ91 Mg alloy using nanosized SiO₂ (quartz) particles (15 nm, using groove filling and covering method). Low tool rotation rate and traverse speed resulted in poor material flow. Composites processed at low tool rotation rates and high tool traverse speed had scalloped surfaces and wormholes. A high tool rotation rate and high tool traverse speed resulted in turbulent material flow, leading to a lack of fusion and root flow defects. However, a high tool rotation rate and low tool traverse speed led to a defect-free processed region with a fine dispersion of the reinforcements in the matrix. Accordingly, the process window for the AZ91/SiO₂ composite was constructed, as shown in Fig. 10(a).

The simulation of temperature change during FSP of AZ91 alloy was performed based on the Comsol Multiphysics 5.0 software; an example is shown in Fig. 10(b) [78], revealing a significant increase in the temperature during FSP. Based on the simulation results, it was found that the peak

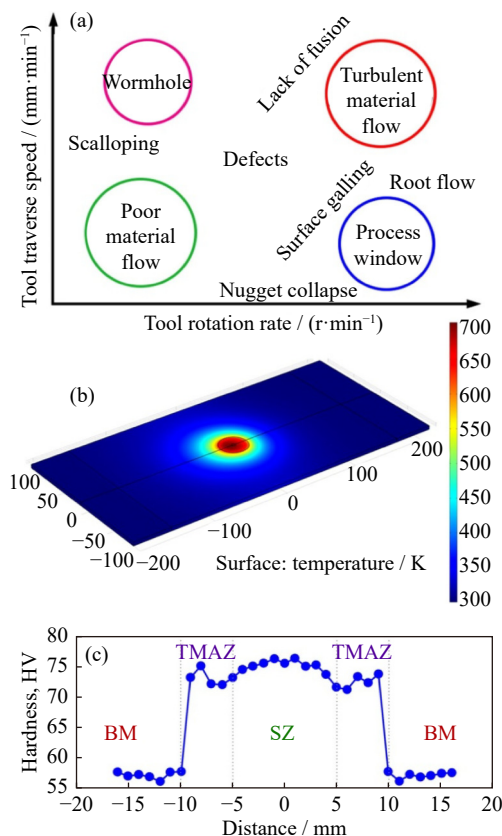


Fig. 10. (a) FSP process window for synthesizing AZ91/SiO₂ composite, (b) simulation of the thermal phenomenon during FSP for $\omega = 600$ r/min and $\nu = 40$ mm/min, and (c) hardness profile along the transverse section for $\omega = 900$ r/min and $\nu = 20$ mm/min [78]. Reprinted by permission from Springer Nature: *Silicon*, Synthesis and characterization of magnesium alloy surface composite (AZ91D–SiO₂) by friction stir processing for bioimplants, R.V. Vignesh, R. Padmanaban, and M. Govindaraju, Copyright 2020.

temperature associated with FSP increases with an increase in tool rotation rate or a decrease in tool traverse speed [78]. In this context, Khayyamin *et al.* [79] reported more intense grain refinement and increased hardness for the AZ91/SiO₂ composite (using the groove filling and covering method) by increasing the traverse speed at a constant tool rotation rate.

The hardness profile along the transverse section is shown in Fig. 10(c) [78]. The figure reveals a much higher hardness in the SZ compared to the hardness of the base metal. This enhancement can be attributed to grain refinement as well as to the presence of a fine dispersion of $\beta\text{-Mg}_{17}\text{Al}_{12}$ phase and nanophase SiO₂ particles. The fine dispersion of SiO₂ accentuated the refinement of the matrix through its effects on the nucleation of recrystallization and inhibition of grain growth [80–81]. The corrosion test results revealed the formation of an adherent layer of calcium hydroxyapatite and calcium–magnesium phosphate in simulated body fluids, which reduced the corrosion rate for bioimplant applications [78].

Khayyamin *et al.* [79] also fabricated composite layers on the surface of the AZ91 alloy using nanosized SiO₂ particles (10 nm). FSP led to the refinement of the matrix grains. Increasing the traverse speed at $\omega = 1250$ r/min resulted in greater grain refinement and enhanced tensile properties, as shown in Fig. 11(a). Moreover, by increasing the number of passes, better uniformity in the particle distribution and finer grain sizes were obtained, which led to the further improvement of the mechanical properties (Fig. 11(b)). In this regard, the dissolution/dispersion of the $\beta\text{-Mg}_{17}\text{Al}_{12}$ phase and the closure of casting defects have also been indicated [79].

In fact, controlling the distribution of reinforcing particles plays a key role in the performance of the fabricated composites [82–83]. An ideal metallic composite should have a homogeneous distribution of particulates and constant inter-particle distance. The resultant composite microstructure should approach this condition in order to exhibit improved mechanical properties [84]. The improvement of particle distribution enhances the material flow and prevents early fracture, thereby improving the tensile ductility of the composite. Conversely, cluster formation might lead to poor tensile strength and elongation [84]. The homogeneous distribution is a result of the rotating tool's effective stirring action [84] as well as the extrusion of the plasticized material due to the movement of the tool [85], which are primarily influenced by the major process parameters, including tool rotational speed and traverse speed [84,86–87]. Traverse speed affects both frictional heat and mechanical stirring simultaneously, leading to poor particle dispersion at increased traverse speed [84,88]. Moreover, there is evidence that increasing the rotational rate leads to improved particle distribution [28,88]. Furthermore, increasing the number of FSP passes can promote the uniform distribution of particles [28,84,88]. In other words, the repeated stirring action and the plastic flow of the material tend to reduce particle agglomeration [84]. Although this is the most effective strategy, a corresponding increase in production costs should also be taken into account [28].

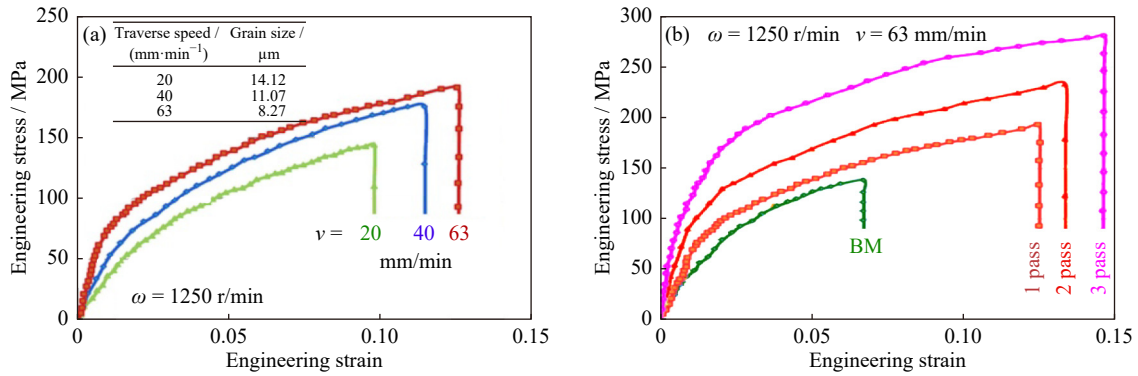


Fig. 11. Tensile stress–strain curves of AZ91/SiO₂ surface composites for (a) various v values at $\omega = 1250$ r/min (the inserted table shows effect of the traverse speed on the final grain size in the SZ of the specimens) and (b) various passes at $\omega = 1250$ r/min at $v = 63$ mm/min [79]. Reprinted from *Mater. Sci. Eng. A*, Vol. 559, D. Khayyamin, A. Mostafapour, and R. Keshmiri, The effect of process parameters on microstructural characteristics of AZ91/SiO₂ composite fabricated by FSP, 217-221, Copyright 2013, with permission from Elsevier.

The effect of the FSP tool might be significant [89]. For instance, a profound effect has been observed on the particle size distribution uniformity when using a tool with a square pin than when using one with a circular pin [28]. Moreover, changing the direction of tool rotation during multipass FSP might also lead to better grain refinement and more homogeneous particle distribution [90]. Furthermore, novel FSP variants such as the bobbin tool FSP (BTFSP) can be used for fabricating two-side composites on the top and bottom sides of the workpiece, as shown in Fig. 12, where good uniformity in the particle distribution can be achieved [91]. A low plunge depth level might lead to insufficient heat generation and cavity formation toward the SZ center. On the other hand, high levels of plunge depth result in the ejection of reinforcement particles and even the sticking of material to the tool shoulder. Thus, an optimal plunge depth is needed in developing defect-free surface composites [92].

The particle volume fraction (f) can be adjusted based on the shape/size of grooves or the depth/number of holes. The microstructure and properties of the composite are dependent on f , where an example is shown in Fig. 13 for FSP AZ31/Ti composite [87]. For groove filling, f can be estimated by Eq. (5) [93]. It is noteworthy that it is difficult to con-

trol the number of particles that can be introduced into the surface, and there may be a nonuniform distribution of the particles in the thickness direction [28]. Another concern is the severe tool wear when FSP is used with composites with a high f , as discussed by Avettand-Fénoël and Simar [30]. Generally, an increase in the particulate content might raise the difficulty of plasticization, which can adversely affect particle distribution homogeneity [84]. On the other hand, an increase in the particulate content can further reduce the ductility of the intrinsically brittle Mg matrix. However, grain refinement of α -Mg might lead to the activation of secondary slip systems, which is advantageous [94]. Using deformable particles such as Ti alloys as reinforcing particles can help maintain ductility [86–87].

$$\begin{cases} f = A_G/A'_p \\ A_G = \text{area of groove} = \text{groove width} \times \text{groove depth} \\ A'_p = \text{projected area of pin} = \text{pin diameter} \times \text{pin length} \end{cases} \quad (5)$$

2.4. AZ91–B₄C composites

Patle *et al.* [95] fabricated composite layers on the surface of as-cast AZ91 Mg alloy using micron-sized B₄C (boron carbide) particles (10–15 μm, using groove filling and covering method). A defect-free processed region was obtained for $\omega = 1400$ r/min and $v = 22$ mm/min. As shown in Fig. 14, the surface composite showed higher hardness compared to the base metal, which was attributed to the dispersed B₄C particles and microstructural refinement in the SZ. Moreover, the processed surface composite showed a lower wear rate. The wear mechanisms were found to be dependent on the sliding velocity. For a sliding velocity of 0.06 m/s, the predominant wear mechanisms were abrasive and severe adhesive wear, along with some degree of oxidative wear; whereas, for a sliding velocity of 0.12 m/s, delamination and oxidative wear were the predominant ones, along with some abrasive and mild adhesive wear. Singh *et al.* [96] also fabricated AZ91/B₄C surface composite by FSP ($\omega = 900$ r/min and $v = 45$ mm/min, using hole filling method) and reported enhancement of wear resistance based on the wear tests performed by the pin-on-disk apparatus.

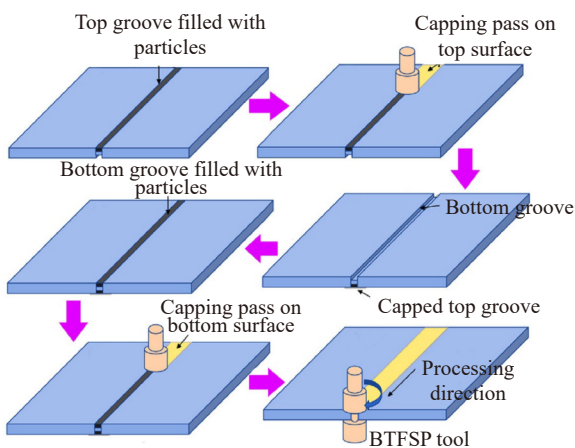


Fig. 12. Schematic diagram of the steps followed for fabricating double-side composite using bobbin tool friction stir processing [91].

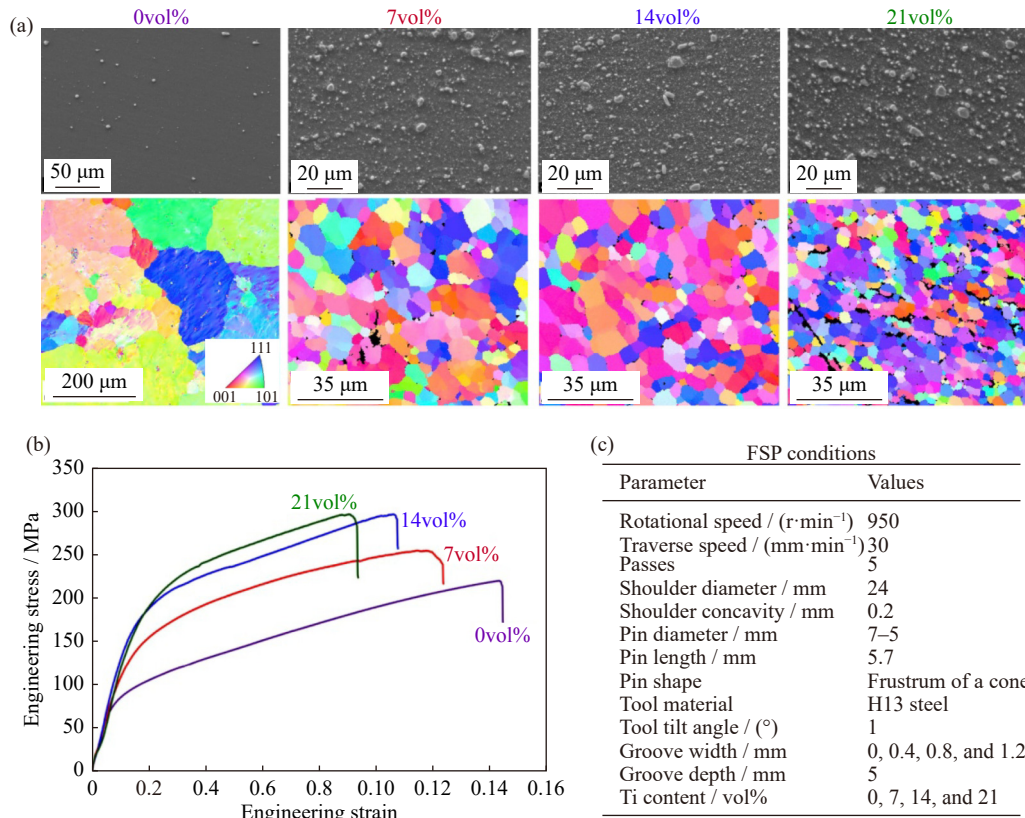


Fig. 13. (a) SEM images and electron backscattered diffraction (EBSD) maps, (b) tensile stress–strain curves of Ti-reinforced AZ31 composites processed by FSP, and (c) the FSP parameters used for processing [87]. Reprinted from *Mater. Sci. Eng. A*, Vol. 772, I. Dinaharan, S. Zhang, G.Q. Chen, and Q.Y. Shi, Titanium particulate reinforced AZ31 magnesium matrix composites with improved ductility prepared using friction stir processing, 138793, Copyright 2020, with permission from Elsevier.

2.5. AZ91–TiC composites

Vijayan *et al.* [97] processed AZ91/TiC (titanium carbide) surface composite using micron-sized TiC particles (4 μm, using groove filling and covering method). Defect-free surface composite with equiaxed recrystallized grains in the nugget zone with homogeneously distributed TiC particles was obtained using ω = 1000 r/min, v = 30 mm/min, and an 8

kN axial load. The surface composite had a peak hardness of almost twice that of the base metal. Accordingly, the pin-on-disk wear test revealed the superiority of wear resistance of the surface composite compared to the base metal, as shown in Fig. 15.

Sahoo *et al.* [98] processed AZ91/TiC–TiB₂ *in-situ* hybrid composite by stir casting, whereby the TiC–TiB₂ reinforcements were formed *in-situ* [99] via the addition of ball-

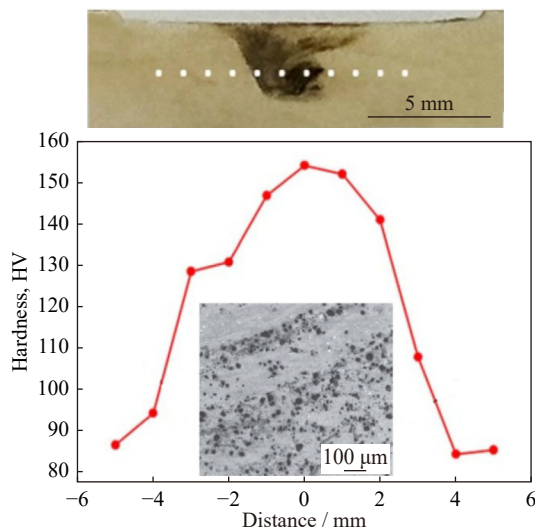


Fig. 14. Hardness profile along the transverse section and a representative SEM image from the processed area of FSP AZ91/B₄C composite [95].

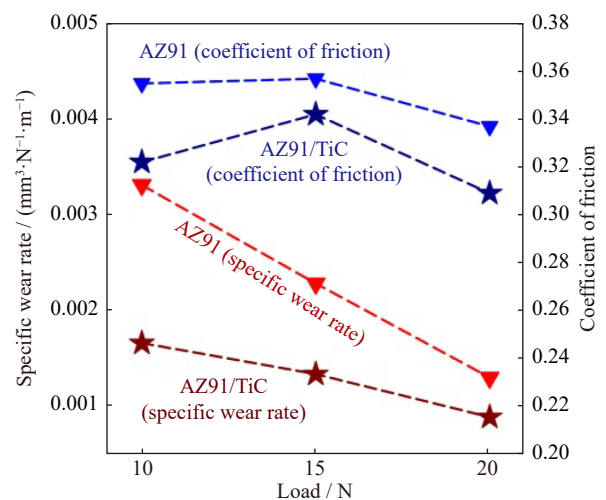


Fig. 15. Specific wear rate and coefficient of friction versus load for FSP AZ91/TiC composite compared to those obtained for AZ91 alloy [97].

milled Ti-B₄C powder. The as-cast *in-situ* composite was then subjected to FSP for microstructural refinement. From the EBSD maps shown in Fig. 16 for AZ91 and AZ91/TiC-TiB₂, FSP led to remarkable grain refinement. The SEM images in Fig. 16 also show that applying FSP and its repetition (pass 2) resulted in the formation of a more uniform microstructure due to the elimination of the continuous network of the β-Mg₁₇Al₁₂ phase of the as-cast state. The presence of the *in-situ* TiC-TiB₂ reinforcement particles in the AZ91/TiC-TiB₂ composite can effectively result in grain growth resistance at the grain boundaries; hence a finer grain size has been achieved for the AZ91/TiC-TiB₂ composite.

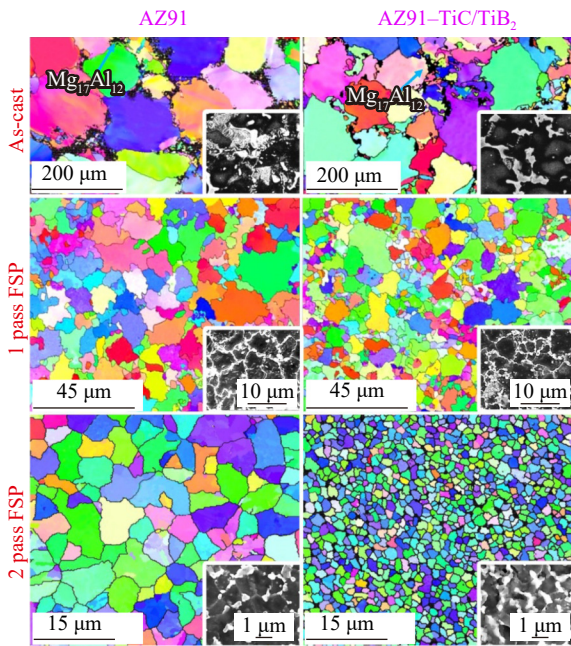


Fig. 16. EBSD maps and SEM images (insets) of as-cast and friction stir processed AZ91 alloy and AZ91/TiC-TiB₂ composite [98]. Reprinted from *Mater. Sci. Eng. A*, Vol. 724, B.N. Sahoo, F. Khan, S. Babu, S.K. Panigrahi, and G.D.J. Ram, Microstructural modification and its effect on strengthening mechanism and yield asymmetry of *in-situ* TiC-TiB₂/AZ91 magnesium matrix composite, 269-282, Copyright 2018, with permission from Elsevier.

As can be seen in the transmission electron microscopy (TEM) images of Fig. 17 [98], material deformation during FSP has led to increased refinement and more homogeneous distribution of the *in-situ* TiC-TiB₂ reinforcing particles, which is a favorable outcome. The tensile stress-strain curves are shown in Fig. 17 [98]. The as-cast AZ91 and AZ91/TiC-TiB₂ exhibited low strength and ductility due to the presence of casting defects and the effects of size, quantity, shape, and distribution of network-type intergranular β-Mg₁₇Al₁₂ phase (Fig. 16). However, major improvements in mechanical properties were realized with FSP due to the fragmentation of the coarse β-Mg₁₇Al₁₂ phase, grain refinement, and elimination of the inhomogeneous microstructure.

Arora *et al.* [100] fabricated AZ91/TiC-Al₂O₃ hybrid composite by FSP using ball-milled particles that were added by the hole-filling method. Different cooling conditions

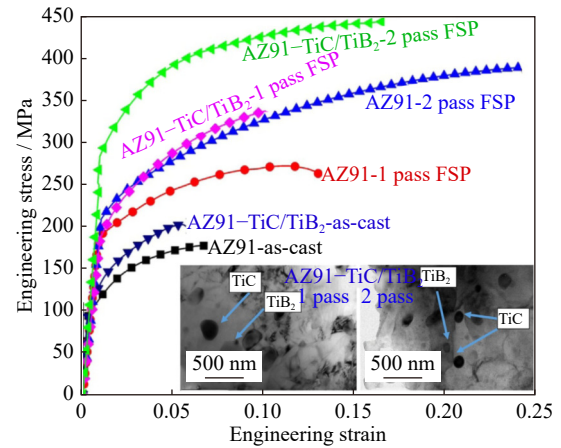


Fig. 17. Tensile stress-strain curves of as-cast and friction stir processed AZ91 alloy and AZ91/TiC-TiB₂ composite as well as some representative TEM images [98]. Reprinted from *Mater. Sci. Eng. A*, Vol. 724, B.N. Sahoo, F. Khan, S. Babu, S.K. Panigrahi, and G.D.J. Ram, Microstructural modification and its effect on strengthening mechanism and yield asymmetry of *in-situ* TiC-TiB₂/AZ91 magnesium matrix composite, 269-282, Copyright 2018, with permission from Elsevier.

were applied for enhanced performance of the material. The results are summarized in Fig. 18, which shows that the hybrid composites have finer grain size and higher hardness compared to the AZ91 alloy. Moreover, the rapid cooling conditions (undersurface cooling using coolant at -20°C) had a greater enhancement effect on both grain refinement and hardness compared to the conventional ambient cooling conditions. FSP processing of AZ91/TiC-Al₂O₃ hybrid composite also led to enhanced scratch resistance [100].

2.6. AZ91-carbon fiber composites

Afrinaldi *et al.* [101] fabricated carbon fiber reinforced AZ91 composite via the addition of chopped carbon fibers with a length of ~1 mm and subsequent FSP (using groove filling and covering method based on a narrow slit). Chopped

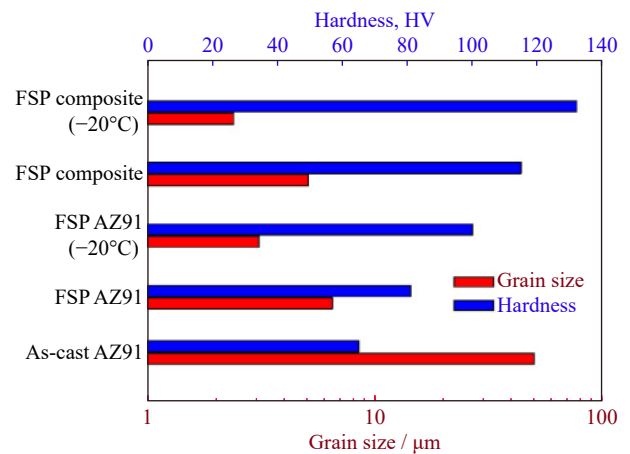


Fig. 18. Grain size and hardness of as-cast and friction stir processed AZ91 alloy and AZ91/TiC-Al₂O₃ composite under different cooling conditions [100]. Adapted by permission from Springer Nature: *Trans. Indian Inst. Met.*, Some investigations on friction stir processed zone of AZ91 alloy, H.S. Arora, H. Singh, B.K. Dhindaw, and H.S. Grewal, Copyright 2012.

carbon fibers were fragmented (to a length of less than 20 μm) and dispersed in the SZ during the FSP process. The effect of the FSP tool was also investigated, where the 3-flat pin tool better reduced the size and number of defects in the SZ compared to the conventional threaded pin tool (Fig. 19(a)). As shown in Fig. 19(b), the fatigue strengths of the carbon fiber-reinforced AZ91 composite were comparable to those of the as-cast counterparts but lower than those of the FSP AZ91 samples without carbon fibers. In fact, fatigue cracks were initiated at the agglomerations of carbon fibers, where the adverse effect of the inhomogeneous distribution of the carbon fibers was also noted.

Mertens *et al.* [102] applied FSP on a sandwich obtained by stacking one layer of C fabric between two sheets of AZ91 alloy. This technique is known as the sandwich method, in which the secondary phase is placed as a laminate or layer between workpieces for subsequent FSP processing [28]. High ω (1500 r/min) and high v (300 mm/min) led to the heterogeneous distribution of the reinforcing phase and high porosity. These defects were more severe for high ω (1500 r/min) and low v (80 mm/min). However, low ω (500 r/min) and low v (80 mm/min) led to the development of a sound processed region with a homogeneous distribution of carbon fibers [102]. These processing conditions were also applied on a sandwich obtained by stacking one layer of C fabric between two sheets of AZ31 alloy. In this case, High ω (1500 r/min) combined with high v (300 mm/min) or low v (80 mm/min) resulted in a sound processed region with a homogeneous distribution of carbon fibers. However, low ω (500 r/min) and low v (80 mm/min) led to the heterogeneous dis-

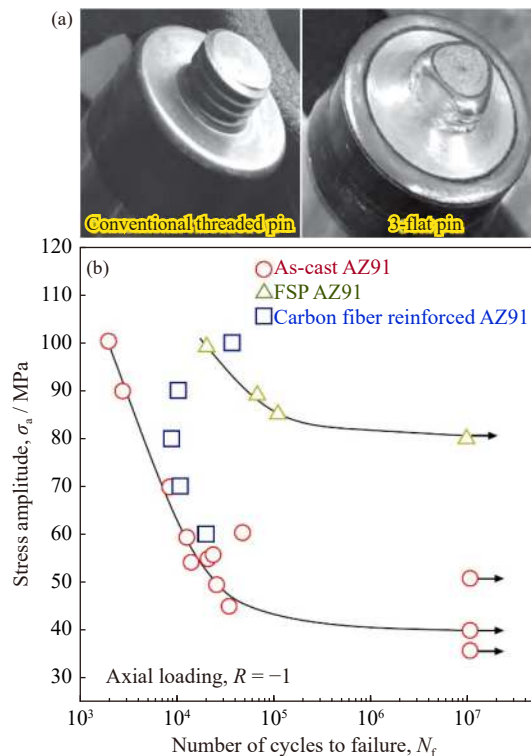


Fig. 19. (a) FSP tools and (b) fatigue $S-N$ diagrams of as-cast and friction stir processed AZ91 alloy and AZ91/carbon fiber composite (R represents load ratio) [101].

tribution of the reinforcing phase, while low ω (500 r/min) and high v (300 mm/min) resulted in significant porosity. Therefore, while both AZ31 and AZ91 alloys are similar materials, the presence of a high amount of $\beta\text{-Mg}_{17}\text{Al}_{12}$ (as well as its dissolution/fragmentation) seems to be an important determinant for the differences as well as for the much smaller processing window for AZ91 composite compared to the AZ31 composite. The AZ91 composite was also found to be capable of age hardening due to the precipitation of the $\beta\text{-Mg}_{17}\text{Al}_{12}$ phase (Fig. 20), thus allowing for mechanical behavior improvement after processing [102].

2.7. AZ91–hydroxyapatite composites

Yousefpour *et al.* [103] processed AZ91/hydroxyapatite (HA) bionanocomposites by multipass FSP using the hole filling and covering method. The 2nd and 3rd passes were performed with a 100% overlapping strategy. As shown in Fig. 21, the samples' hardness and strength increased with an increasing number of passes due to better grain refinement and more uniform dispersion of HA powder. Moreover, the introduction of HA powder to AZ91 alloy was found to result in better grain refinement and better mechanical properties. The results of this study clearly show that the particle distribution in the AZ91/HA nanocomposites is significantly affected by the number of FSP passes [103]. In a related study, Yousefpour *et al.* [104] added the hybrid HA/Ag powder, which led to improved grain refining efficiency. Moreover, this sample had the highest texture parameter for the $\{10\bar{1}1\}$ orientation as the high corrosion resistance texture, which was due to the promotion of the nonbasal slip caused by the dissolution of Ag particles in the matrix.

2.8. Other reinforcing phases

Fly ash can be used as an effective reinforcement for fabricating low-cost and environmental-friendly MMCs, as shown by Dinaharan *et al.* [105–106]. In this regard, Patle *et al.* [107] processed AZ91/fly ash surface composite by adding fly ash particles and FSP. Better mechanical and wear properties were realized, but the composite had decreased corrosion resistance for the processed surface composite. Farghadani *et al.* [108] introduced Cu and CuO micropowders on the surface of AZ91 alloy for processing by FSP.

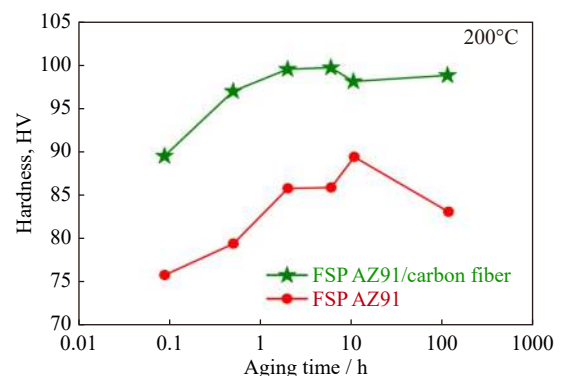


Fig. 20. Hardness at mid-thickness as a function of aging time for friction stir processed AZ91 alloy and AZ91/carbon fiber composite [102].

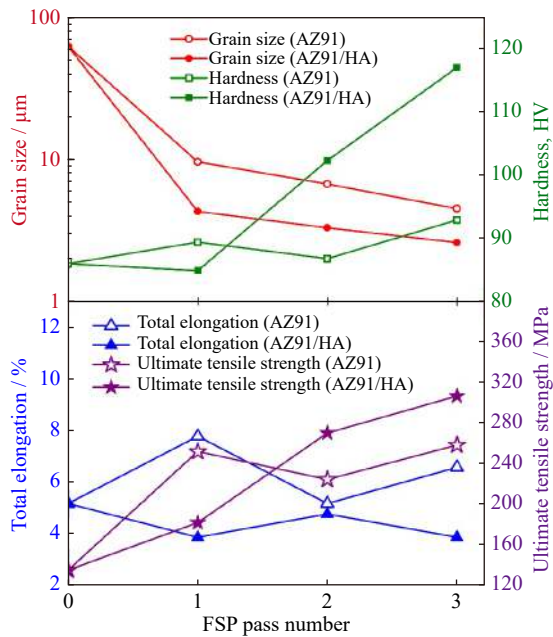


Fig. 21. Grain size and mechanical properties of friction stir processed AZ91 alloy and AZ91/HA composite [103]. Adapted from *J. Mech. Behav. Biomed. Mater.*, Vol. 125, F. Yousefpour, R. Jamaati, and H.J. Aval, Investigation of microstructure, crystallographic texture, and mechanical behavior of magnesium-based nanocomposite fabricated via multi-pass FSP for biomedical applications, 104894, Copyright 2022, with permission from Elsevier.

The AZ91/Cu nanocomposite was reinforced by the *in-situ* formation of the Mg_2Cu compound, while the CuO particles in AZ91/CuO nanocomposite were reduced, and MgO and $MgCu_2$ reinforcing particles were formed alongside the Mg_2Cu compound. Accordingly, grain refinement and *in-situ* formation of reinforcing particles significantly improved the mechanical properties and wear resistance of the composite. Bhadouria *et al.* [109] processed AZ91 hybrid composites via the addition of nano-WC–Co–Cr and multiwalled carbon nanotubes (CNTs) by multipass FSP using the grooving method. The hybrid composites showed better grain refinement, higher hardness, and greater wear resistance compared to the AZ91 alloy and the AZ91/WC–Co–Cr and AZ91/CNTs composites.

3. Future scope/prospects

In the previous section, the reported works on FSP-processed AZ91 composites with SiC, Al_2O_3 , SiO_2 , B_4C , TiC/TiB₂, carbon fiber, hydroxyapatite, and other reinforcements are summarized. Most of the works are focused on SiC. However, there are many common and effective particles that need to be investigated for AZ91 composites processed by FSP. One of the most common particles is ZrO_2 , which has systematically been investigated for FSP AZ31 composites by Chang *et al.* [110], Navazani and Dehghani [111], Mazaheri *et al.* [112], Zang *et al.* [113], and Qiao *et al.* [114]. These studies showed that ZrO_2 is a promising particulate for FSP Mg alloys, and hence, systematic in-

vestigation of the AZ91/ ZrO_2 surface composites by FSP is recommended for future studies. Other important reinforcing phases are CNTs, as demonstrated in the reports of Morisada *et al.* [115], Jamshidijam *et al.* [116], Nia and Nourbakhsh [117], and Arab *et al.* [118] for AZ31 alloy. Based on the results of Bhadouria *et al.* [109], the introduction of CNTs to AZ91 should be further investigated in future works, where issues associated with CNTs, such as agglomeration, need special attention. Recently, Dinaharan *et al.* [86] introduced the titanium particulate-reinforced AZ31 composites for pure Ti [84] and Ti–6Al–4V alloy. Both Ti and Mg have an hcp crystal structure that ensures good compatibility. Moreover, the Ti particulate is deformable and is characterized by a higher elastic modulus, melting point, hardness, and corrosion resistance. Furthermore, the solubility of Ti in magnesium is negligible. As a result, Ti-based particulates seem to be good choices for the processing of Mg-matrix composites. Accordingly, their introduction to AZ91 is an interesting practice for future work. Many other potential reinforcing phases can be added to this list, including graphene nanoplatelets (GNPs) [119–120], ZrB_2 [121], and graphite [122]. Due to their favorable properties, such as wear resistance [123], hybrid composites (with more than one reinforcing phase) have gained considerable attention in recent years [124–125]. The hybrid surface MMCs with more than one reinforcing phase gained attention in material processing due to their noble tribological behavior and surface properties, which cannot be attained in mono composites [99,123]. Several investigators have introduced hybrid reinforcements to the matrix of Mg alloys via FSP, such as Sharma *et al.* [126] (MWCNT–graphene), Jalilvand and Mazaheri [127] (ZrO_2 /WC/ B_4C), Lu *et al.* [128] (Al_2O_3 –CNT), Sahoo *et al.* [98] (TiC–TiB₂), Arora *et al.* [100] (TiC– Al_2O_3), Yousefpour *et al.* [104] (HA–Ag), and Bhadouria *et al.* [109] (WC–Co–Cr–CNTs). Due to the favorable effects of hybrid reinforcements on enhancing the properties of AZ91 alloy, more research in this field is suggested.

As discussed in the previous section, the effects of reinforcement type and particle size, FSP tool geometry, rotation rate, traverse speed, and the number of FSP passes have been investigated for FSP AZ91 composites. However, there are many other variables involved in composite fabrication by FSP [129], as summarized in Table 1. In this regard, the special tool pin profiles have been shown to be favorable [89,130], which need to be investigated for various AZ91 composites. Moreover, much more attention is needed to the effects of particle volume fraction for AZ91 Mg composites [87,109].

For the introduction of reinforcement particles to AZ91 composites, groove filling, hole filling, sandwich method, stir casting, and formation of *in-situ* particles have been applied so far. In fact, the introduction of *in-situ* formed particles in Mg alloys has been observed in several famous systems. The Mg–Si system is the most recognizable one. In this system, Mg_2Si forms during processing [131–132]. Applying FSP on

Table 1. Summary of the variables involved in composite fabrication by FSP

| Process parameters | Tool variables | Reinforcement/matrix characteristics |
|---------------------|-------------------|--------------------------------------|
| Tool rotation rate | Shoulder diameter | Reinforcement type |
| Tool traverse speed | Shoulder profile | Reinforcement size |
| Axial force | Pin profile | Reinforcement volume fraction |
| Tool tilt angle | Pin diameter | Reinforcement strategy |
| Plunge depth | Pin length | Mechanical properties of matrix |
| Number of passes | Tool material | Thermal properties of matrix |

the Mg₂Si system is quite effective for enhancing microstructure and mechanical properties through microstructural refinement and modification of primary/eutectic Mg₂Si, as shown by Taghiabadi and Moharami [133] and Raeissi and Nourbaksh [134]. Therefore, it might be interesting to apply FSP to the Si-containing AZ91 alloys [135]. Moreover, the *in-situ* formation of phases is prevalent in many Mg alloys

[136], and hence, these alloys might be viewed as composites. For instance, applying FSP to the Mg–Al–Ca system [137] leads to the formation of a fine-grained composite with well-dispersed intermetallic particles (such as the Al₂Ca compound), as shown in Fig. 22 [29]. Therefore, applying FSP to the *in-situ* formed composites is expected to receive considerable attention in the future.

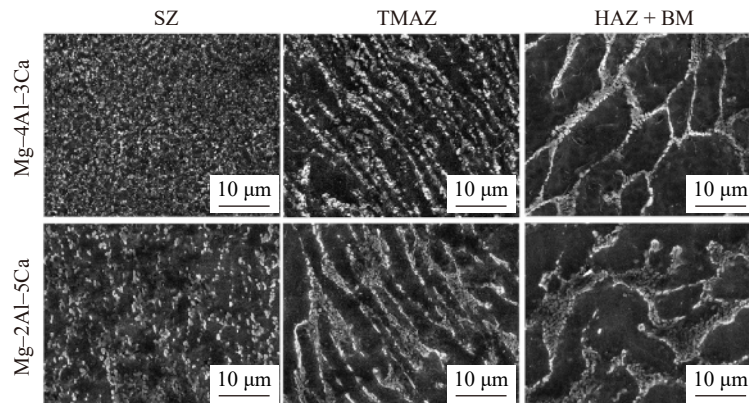


Fig. 22. SEM images of Mg–Al–Ca alloys processed by FSP [29]. Reprinted from *Mater. Lett.*, Vol. 296, Z. Nasiri, M.S. Khorrami, H. Mirzadeh, and M. Emamy, Enhanced mechanical properties of as-cast Mg–Al–Ca magnesium alloys by friction stir processing, 129880, Copyright 2021, with permission from Elsevier.

The dynamically recrystallized grain size is not correlated to the Zener–Hollomon parameter for FSP AZ91 composites (based on Eq. (1)). There is a need for greater attention in future works to determine the effects of particle type and characteristics on grain refinement. The effect of grain size on mechanical strength can be represented by the classical Hall–Petch relationship [138]. This area also needs more research on AZ91 composites to enable a comparison of the results with the obtained values of the Hall–Petch slope for Mg alloys, as summarized by Yu *et al.* [139].

Superplasticity is the ability of a fine-grained polycrystalline material to exhibit very high elongations ($\geq 400\%$) prior to failure. Since the grain boundary sliding (GBS) is the governing deformation mechanism, the strain rate sensitivity index ($m = \frac{\partial \lg \sigma}{\partial \lg \dot{\epsilon}}$ based on $\sigma \propto \dot{\epsilon}^m$) during superplastic flow is ~ 0.5 [59,140–141]. These large elongations are usually achieved at high temperatures and relatively low strain rates; hence, the grains might become coarse, and superplasticity might be lost due to the replacement of GBS with a dislocation creep mechanism [59]. Accordingly, microduplex or pseudosingle phase alloys are usually considered superplastic materials [142–145]. Friction stir-processed AZ91

magnesium alloy shows better superplastic properties compared to AZ31 alloy due to the higher content of the β -Mg₁₇Al₁₂ phase [146]. In fact, FSP can refine the microstructure in the processed region, which is characterized by a high proportion of high-angle grain boundaries as well as particle fragmentation and dispersion. All of these attributes accentuate superplasticity. However, the β -Mg₁₇Al₁₂ phase is unstable at elevated temperatures, and its dissolution becomes a drawback to grain growth restriction. As shown in Fig. 23 [147], increasing the deformation temperature from 300 to 350°C accentuates the superplastic properties due to the favorable effect of deformation temperature in obtaining superplasticity at high strain rates (superplasticity at strain rates $\geq 0.01 \text{ s}^{-1}$) [2,59,148]. However, a further increase to 375°C leads to a sharp drop in ductility due to the rapid grain growth. Accordingly, the fine-grained AZ91 nanocomposites processed by FSP with thermally stable reinforcing particles (for grain growth inhibition) might be useful materials for superplastic forming, and this subject needs to be investigated.

Additive manufacturing (AM), which is suitable for the fabrication of a wide range of complex geometries at fine resolutions, is based on the progressive addition of thin layers of materials from 3D model data [149–150]. Besides the most

widely used processes in the categories of powder bed fusion and directed energy deposition [151–152], metal AM processes based on friction stir engineering have received considerable attention [153]. These approaches are useful, especially in obtaining Mg alloys that are defect-free with fine-grain size and good mechanical/functional properties [149]. Friction stir AM (FSAM) and additive friction stir deposition are two widely used methods for Mg alloys [150]. These processes seem to be suitable for the processing of composites, which have been examined by Ho *et al.* [154]. Fig. 24 shows the processing of AZ31/hydroxyapatite composites based on FSAM. Accordingly, the applicability of these promising methods for the processing of AZ91 composites is yet to be investigated.

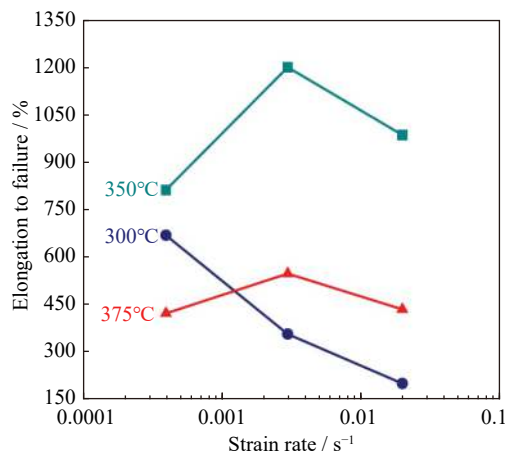


Fig. 23. Tensile elongation vs. strain rate at different temperatures for the submerged friction stir processed AZ91 alloy [147]. Reprinted from *Mater. Sci. Eng. A*, Vol. 568, F. Chai, D.T. Zhang, Y.Y. Li, and W.W. Zhang, High strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing, 40–48, Copyright 2013, with permission from Elsevier.

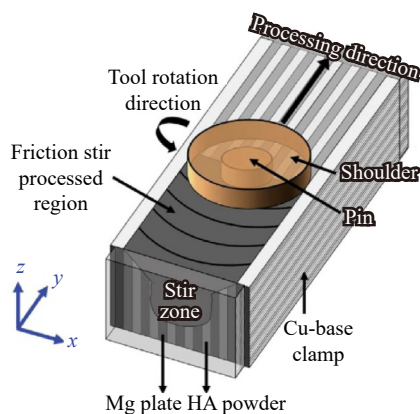


Fig. 24. Processing of AZ31/hydroxyapatite composites by FSAM [154].

4. Summary

In summary, this study reviewed FSP of surface MMCs using the AZ91 alloy, whereby AZ91 composites with various reinforcing phases such as SiC, Al₂O₃, SiO₂, B₄C, TiC,

carbon fiber, hydroxyapatite, *in-situ* formed phases, and hybrid reinforcements were critically discussed. FSP composite fabrication methods were discussed, including grooving, hole filling, sandwich method, stir casting followed by FSP, and formation of *in-situ* particles. The effects of introducing second-phase particles and FSP process parameters such as tool rotation rate, traverse speed, number of passes on the microstructural modification, grain refinement, mechanical properties, wear/tribological behavior, and corrosion resistance were also discussed. Finally, useful suggestions were given to shed light on the important issues and to highlight research prospects for future works.

Conflict of Interest

The author declares no conflict of interest.

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