

Enhancing the high-cycle fatigue strength of Mg–9Al–1Zn casting by friction stir processing

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Friction stir processing (FSP) of cast Mg–9Al–1Zn alloy plus subsequent aging produced a defect-free recrystallized fine-grained microstructure with fine β -Mg₁₇Al₁₂ particles. Compared to the as-cast parent material, the FSP sample exhibited significantly enhanced fatigue properties, with the fatigue strength being increased from ~45 to ~95 MPa and the fracture mode being changed from quasi-cleavage fracture to dimple fracture. The improvement was attributed to refinement of grains, elimination of porosities and coarse β networks, and precipitation of fine β particles.

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Mg–9Al–1Zn alloy (AZ91) is widely used to produce cast components due to its excellent castability and reasonable mechanical properties. The microstructure of as-cast AZ91 alloy is characterized by Al-lean dendrites with a eutectic Al-rich solid solution and intermetallic β -Mg₁₇Al₁₂ [1], as well as some porosity defects. The eutectic β -Mg₁₇Al₁₂ with a network-like morphology is mainly distributed at the grain boundaries. Due to its special cast structure, the as-cast AZ91 alloy usually exhibits low mechanical properties, especially fatigue [2–6], so it is necessary to modify the morphology, size and distribution of the β -Mg₁₇Al₁₂ phase and to refine the grains of the Mg matrix to improve the mechanical properties.

In the past few years, various research efforts have been made to achieve this purpose [7,8]. The heat-treatment (solution + aging) method was usually been adopted to refine the coarse β -Mg₁₇Al₁₂ phase [7]; however, this procedure has several drawbacks: it takes a long time, and results in surface oxidation and grain growth; more importantly, it cannot eliminate porosity defects. By comparison, the plastic deformation route seems more effective. Multi-pass equal channel angular pressing resulted in significant refinement of both the

β -Mg₁₇Al₁₂ and matrix grains [8], but is not suitable for processing large-sized materials.

Friction stir processing (FSP) is a relatively novel multifunctional metal working method, developed based on the basic principles of friction stir welding (FSW) [9–12]. This method has been successfully used to modify the microstructures of as-cast Al alloys [13–18], Ti alloys [19], NiAl bronze [20] and Mg alloys [21–23]. FSP of the castings resulted in a significant breakup/dissolution of coarse second-phase particles and breakup/refinement of primary dendrites, created a homogeneous distribution of the fine particles in the significantly refined matrix, and eliminated casting porosity, thereby significantly improving the mechanical properties of the castings [15–19].

Poor fatigue property is a hindrance to the as-cast AZ91 alloy being widely used for structural applications, due to coarse cast structure and casting defects. This problem is expected to be solved by FSP through microstructural refinement, densification and homogenization. It was reported that FSP was effective in improving the fatigue properties of AZ91 alloy processed by the high-pressure die-casting method (HPDC) [3]. However, the material in the post-FSP aging condition was not reported, and furthermore, the effect of the β -Mg₁₇Al₁₂ particles on the fatigue properties was not discussed.

A recent investigation by Feng and Ma [24] indicated that two-pass FSP caused significant breakup and dissolution of the coarse eutectic β -Mg₁₇Al₁₂ network and

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Table 1. Tensile properties of as-cast and FSP AZ91D Mg alloy.

| Sample | YS (MPa) | UTS (MPa) | El. (%) |
|-------------------|----------|-----------|------------|
| As-cast | 75 ± 4 | 100 ± 10 | 2.5 ± 0.3 |
| FSP + 168 °C/16 h | 179 ± 11 | 336 ± 20 | 12.5 ± 0.5 |

*Gauge dimension: $10 \times 2 \times 1 \text{ mm}^3$ for FSP sample, $25 \times 4 \times 2.5 \text{ mm}^3$ for cast sample.

remarkable grain refinement of cast AZ91D, and the subsequent aging resulted in the precipitation of fine $\beta\text{-Mg}_{17}\text{Al}_{12}$ particles, thereby producing an excellent combination of strength and ductility. It is interesting to know if the post-FSP aged AZ91D sample with fine grains and fine $\beta\text{-Mg}_{17}\text{Al}_{12}$ particles would exhibit a good fatigue property. In this study, the fatigue behavior of AZ91D samples at cast and post-FSP aging condition was examined. The aim is to understand the mechanism responsible for the enhancement in fatigue property in the FSP AZ91 sample.

Eight-millimeter thick AZ91D alloy plates were machined from cast billets with a composition of 8.35Al–0.49Zn–0.20Mn–0.045Si–0.0015Cu–0.0013Ni–0.0011Fe (in wt.%). An two-pass FSP, with a 100% overlap and the same forward directions, was conducted at a tool rotation rate of 400 rpm and a traversing speed of 100 mm min^{-1} . A tool with a shoulder 24 mm in diameter and a threaded cylindrical pin 8 mm in diameter and 6 mm in length was used. After FSP, the FSP samples were subjected to aging at 168 °C for 16 h. The microstructure of the cast and FSP samples was examined by optical microscopy (OM) and scanning electron microscopy (SEM). The specimens were polished and etched using a solution of 3 g picric acid, 20 ml acetic acid, 50 ml ethanol and 20 ml water.

Fatigue specimens with a gauge length of 5 mm, a width of 5 mm and a thickness of 5 mm were machined parallel to the FSP direction with the gauge being com-

pletely within the stir zone. Fatigue specimens were ground under a low stress and polished using #5000 abrasive paper along the axial direction. Fatigue tests were conducted on an Instron 8871 tester with a load ratio of $R = -1$ at a frequency of 20–40 Hz. The tensile data in Table 1 were used to guide the fatigue tests. Fracture surfaces were subjected to SEM examination, and in addition some of the failed specimens were mounted, polished and examined by SEM.

The microstructure of the as-cast base metal (BM) was characterized by large grains of about $300 \mu\text{m}$ (Fig. 1a), with a coarse $\beta\text{-Mg}_{17}\text{Al}_{12}$ network being distributed at the grain boundaries (Fig. 1b); furthermore, a few porosities were also found in the BM. After FSP, the coarse microstructure of the BM was refined to about $15 \mu\text{m}$, the coarse $\beta\text{-Mg}_{17}\text{Al}_{12}$ network was significantly broken up and dissolved, and the porosities were eliminated (Fig. 1c). During the subsequent aging process, fine lamellar $\beta\text{-Mg}_{17}\text{Al}_{12}$ particles precipitated out either around the grain boundaries in a discontinuous manner or within the grains in a continuous manner (Fig. 1d). The FSP sample showed higher yield strength and elongation than the BM (Table 1).

Figure 2 shows the S–N curves of the cast and FSP samples, and the arrows indicate the specimens that did not fail. The FSP material showed a significantly higher fatigue strength ($\sim 105 \text{ MPa}$) than the BM ($\sim 45 \text{ MPa}$), which was increased by about 138%. Figure 3 shows the fracture surfaces of the fatigue-failed samples. The cast sample showed the quasi-cleavage fracture mode (Fig. 3a). The fracture surface was rough with the cleavage planes being clearly observed and some cracks and casting voids were distinctly visible (Fig. 3a and b), which were the sources of fatigue crack initiation. By comparison, the FSP sample showed characteristics of dimple fracture (Fig. 3c and d). The fracture surface was relatively even and covered with small dimples with-

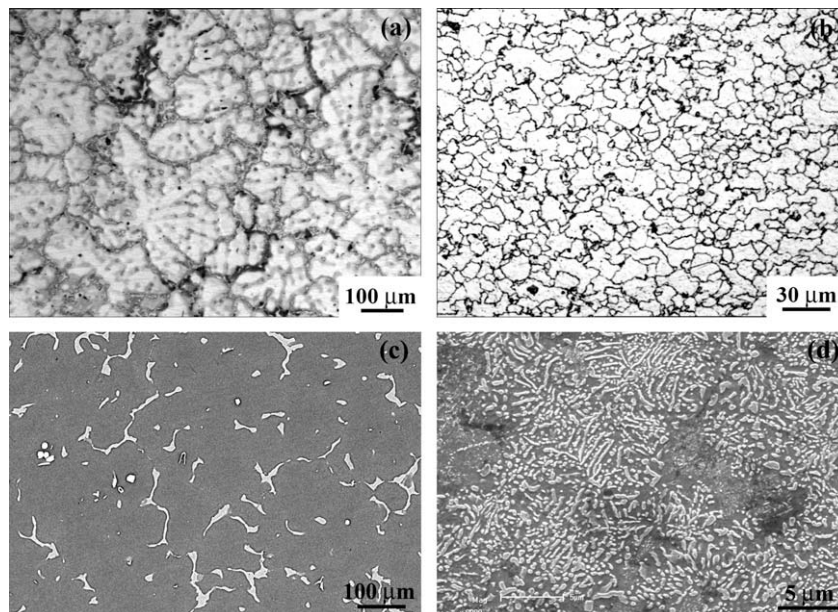


Figure 1. OM images showing grain structure of AZ91D samples: (a) as-cast and (b) FSP; SEM images showing $\beta\text{-Mg}_{17}\text{Al}_{12}$ phase: (c) as-cast and (d) FSP + aging.

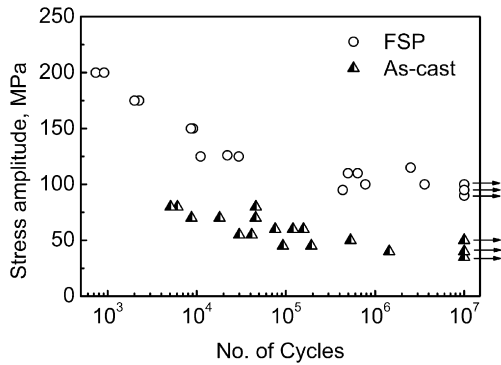


Figure 2. S–N curve of as-cast and FSP AZ91D samples.

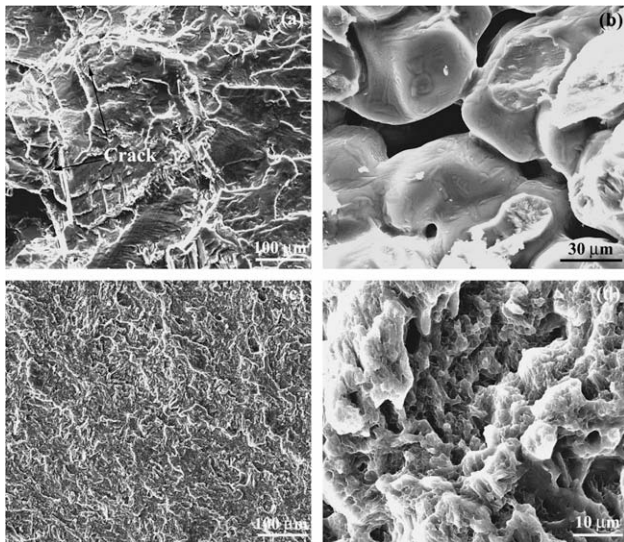


Figure 3. SEM fracture surfaces of fatigue-failed samples: rough surface with (a) cracks and (b) casting voids in as-cast sample (40 MPa); even surface without (c) cracks and (d) dimples in FSP sample (95 MPa).

out cracks and voids being observed, and the crack source came from the subsurfaces of the specimen. It can also be observed that most of the cast specimens exhibited a fracture path deflected from the loading direction, whereas all the FSP specimens failed perpendicular to the load direction (not shown).

Figure 4 shows the cross-sectional views of the fatigue-failed specimens near to the fracture tip. There was distinct evidence of crack nucleation and growth in the BM. The cracks occurred preferentially within the coarse $Mg_{17}Al_{12}$ particles but not on the interface between the particles and the Mg matrix, and some cracks stopped at the interface where many extremely fine $Mg_{17}Al_{12}$ particles precipitated (Fig. 4a and b). For the FSP sample, no crack was observed in the region near to the failure site (Fig. 4c and d).

Compared to the cast sample, the FSP sample exhibited significantly improved fatigue strength and life. This is mainly attributed to grain refinement, porosity elimination and $Mg_{17}Al_{12}$ phase refinement in the FSP sample. First, the fine-grained FSP microstructure can effectively prevent fatigue cracks from being initiated

and propagated. Sharma et al. [17,18] reported that FSP improved the fatigue properties of A356 Al alloys partly due to grain refinement. Pilchak et al. [19] found that the equiaxed fine-grained FSP structure had higher compressive yield stress which is more resistant to fatigue crack initiation, thus dramatically increasing the fatigue properties of cast Ti–6Al–4V alloys. De et al. [25] considered that the refinement of grains reduced the crack propagation rates in the samples and increased the barrier effect of grain boundaries, which is primarily responsible for the improvement in the fatigue life of Al alloy.

Second, the densified FSP microstructure eliminated the possibility of crack initiation at the defects. Casting defects such as porosities are especially harmful to the fatigue property of castings due to preferential initiation and propagation of cracks at these sites [5,17]. This has been proved by SEM examination (Fig. 3b). For the BM, the crack path was generally rough and uneven, indicating that the crack followed defects in the microstructure. FSP eliminated the porosities in the cast sample and densified the material greatly, thus increasing the fatigue properties.

Third, the fine $\beta-Mg_{17}Al_{12}$ particles decreased the possibility of crack initiation due to the breakup of the coarse $\beta-Mg_{17}Al_{12}$ phase, and furthermore, the fine particles had an inhibiting effect on crack propagation. For the cast BM, the coarse $\beta-Mg_{17}Al_{12}$ network could not be deformed in consonance with the Mg matrix, and the plastic deformation during fatigue produced great stress concentration around the network, resulting in crack initiation and propagation within the $\beta-Mg_{17}Al_{12}$ particles (Fig. 4a). As presented above, the post-FSP aged sample was characterized by fine $\beta-Mg_{17}Al_{12}$ particles. This reduced the possibility of crack initiation at the $\beta-Mg_{17}Al_{12}$ phase. Moreover, the uniformly distributed fine $\beta-Mg_{17}Al_{12}$ particles might restrain the propa-

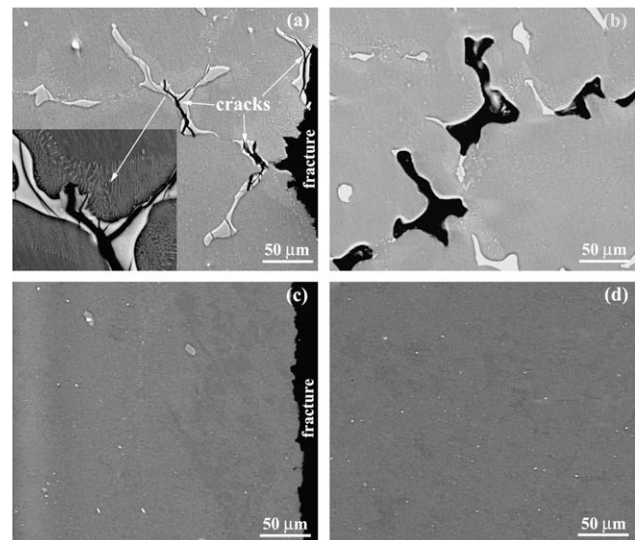


Figure 4. Longitudinal cross-sectional views of failed sample near to fracture tip: (a) as-cast (40 MPa) and (c) FSP (95 MPa), and transverse cross-sectional views (about 1–2 mm below fracture surface): (b) as-cast and (d) FSP.

gation of cracks, and this effect is similar to that due to the fine $Mg_{17}Al_{12}$ particles around the coarse $Mg_{17}Al_{12}$ networks in the BM (Fig. 4a). In fact, a similar effect has been found in cast Al–Si alloys, in which the refined Si particle laden interdendritic regions acted as barriers to small fatigue crack propagation and hence decelerated small fatigue cracks [26,27].

Cavaliere and Marco [3] reported that FSP improved the fatigue properties of HPDC AZ91 alloy. The samples was small ($2 \times 2 \times 12 \text{ mm}^3$) and the test was conducted in the tension loading model ($R = 0.1$). The FSP and the solutionized FSP samples showed similar fatigue life of about 7×10^6 cycles at 80 MPa. The enhancement in the fatigue property was attributed to the grain refinement and especially the elimination of casting defects, which is consistent with the present results. However, the effect of the coarse β - $Mg_{17}Al_{12}$ particles, which are likely to be mainly responsible for the failure of the HPDC sample, on the fatigue properties was not discussed. Furthermore, because the FSP sample was not aged to precipitate fine β - $Mg_{17}Al_{12}$ particles, the effect of the fine particles was not studied. Because of different workpiece thicknesses, FSP conditions and testing methods, it is impossible to compare the present results to the previous one. Although the difference in crystal structure among Al, Ti and Mg alloys may lead to differences in deformation micromechanisms which can affect the fatigue life, it seems that FSP is a promising way to increase the fatigue property of cast alloys.

In summary, FSP improved the fatigue properties of the cast AZ91 sample significantly, due to the refinement of grains, elimination of coarse $Mg_{17}Al_{12}$ network and casting porosities, and precipitation of fine $Mg_{17}Al_{12}$ particles. Accordingly, FSP changed the fracture mode from quasi-cleavage fracture to dimple fracture.

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[1] G. Eisenmeier, B. Holzwarth, H.W. Hoppel, H. Mughra-
bi, Mater. Sci. Eng. A 319–321 (2001) 578.

- [2] B. Wolf, C. Fleck, D. Eifler, Int. J. Fatigue 26 (2004) 1357.
- [3] P. Cavaliere, P.P. De Marco, Mater. Charact. 58 (2007) 226.
- [4] P. Venkateswaran, S.G.S. Raman, S.D. Pathaka, Y. Miyashita, Y. Mutoh, Mater. Lett. 58 (2004) 2525.
- [5] H. Mayer, M. Papakyriacou, B. Zettl, S.E. Stanzl-Tschegg, Int. J. Fatigue 25 (2003) 245.
- [6] M.F. Horstemeyer, N. Yang, K. Gall, D.L. McDowell, J. Fan, P.M. Gullett, Acta Mater. 52 (2004) 1327.
- [7] Y. Wang, G. Liu, Z. Fan, Scripta Mater. 54 (2006) 903.
- [8] K. Mathis, J. Gubicza, N.H. Nam, J. Alloy Compd. 394 (2005) 194.
- [9] W.M. Thomas, E.D. Nicholas, J.C. Needham, M.G. Murch, P. Templesmith, C.J. Dawes, GB Patent Application No. 9125978.8, December 1991.
- [10] C.J. Dawes, W.M. Thomas, Weld. J. 75 (1996) 1.
- [11] W.M. Thomas, R.E. Dolby, in: S.A. David (Ed.), Trends in Welding Research, ASM Int, Park, OH, USA, 2003, p. 203.
- [12] R.S. Mishra, Z.Y. Ma, Mater. Sci. Eng. R 50 (2005).
- [13] Z.Y. Ma, S.R. Sharma, R.S. Mishra, Mater. Sci. Eng. A 433 (2006) 269.
- [14] Z.Y. Ma, R.S. Mishra, M.W. Mahoney, in: K.V. Jata, M.W. Mahoney, R.S. Mishra, S.L. Semiatin, T. Lienert (Eds.), Friction Stir Welding and Processing II, TMS, Warrendale, PA, 2003, p. 221.
- [15] Z.Y. Ma, S.R. Sharma, R.S. Mishra, Metall. Mater. Trans. A 37A (2006) 3323.
- [16] M.L. Santella, T. Engstrom, D. Storjohann, T.Y. Pan, Scripta Mater. 53 (2005) 201.
- [17] S.R. Sharma, Z.Y. Ma, R.S. Mishra, Scripta Mater. 51 (2004) 237.
- [18] S.R. Sharma, R.S. Mishra, Scripta Mater. 59 (2008) 395.
- [19] A.L. Pilchak, D.M. Norfleet, M.C. Juhas, J.C. Williams, Metall. Mater. Trans. A 39A (2008) 1519.
- [20] K. Oh-Ishi, T.R. McNelley, Metall. Mater. Trans. A 35A (2004) 2951.
- [21] C.I. Chang, C.J. Lee, J.C. Huang, Scripta Mater. 51 (2004) 509.
- [22] W. Woo, H. Choo, D.W. Brown, P.K. Liaw, Z. Feng, Scripta Mater. 54 (2006) 1859.
- [23] D.T. Zhang, M. Suzuki, K. Maruyama, Mater. Sci. Forum 539–543 (2007) 3739.
- [24] A.H. Feng, Z.Y. Ma, Scripta Mater. 56 (2007) 397.
- [25] P.S. De, R.S. Mishra, C.B. Smith, Scripta Mater. 60 (2009) 500.
- [26] K. Gall, N. Yang, M.F. Horstemeyer, D.L. McDowell, J. Fan, Fat. Fract. Eng. Mater. Struct. 23 (2000) 159.
- [27] K. Shiozawa, Y. Tohda, S.M. Sun, Fat. Fract. Eng. Mater. Struct. 20 (1997) 237.