

Article

Microstructure Regulation and Strength–Ductility Enhancement of Zn-0.5Mn-0.2Mg Alloy via Equal-Channel Angular Pressing

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Abstract

Zn-Mn-based alloys represent promising material candidates for biodegradable orthopedic implants. Nevertheless, their real-world clinical deployment is severely hampered by unsatisfactory mechanical properties. In this research, a Zn-0.5Mn-0.2Mg (wt.%) alloy was subjected to equal-channel angular pressing (ECAP) with 4, 8 and 12 passes, to systematically explore the influence of ECAP on microstructural evolution and tensile properties. Grain refinement proceeds gradually with additional ECAP passes. The average grain size declines from 1.94 μm (4 passes) to 1.09 μm (8 passes) and further decreases to 0.78 μm (12 passes). While the tilted basal texture remains strong and stable (approximately 15.1–15.2 multiples of uniform distribution, MUD) after 4 and 8 passes, a dramatic drop in texture intensity to 6.7 MUD is detected for the 12-pass sample. Yield strength (YS) and ultimate tensile strength (UTS) increase monotonically with pass number from 279 MPa and 311 MPa (4 passes) to 287 MPa and 323 MPa (8 passes), and further to 298 MPa and 348 MPa (12 passes). Grain boundary strengthening serves as the dominant strengthening mechanism, and dislocation hardening provides supplementary strength contributions. Elongation (EL) also shows a pronounced improvement, increasing from 12% (4 passes) and 17% (8 passes) to a high value of 41% (12 passes). The excellent ductility achieved after 12 passes stems from the combined effects of significant texture weakening and refined second-phase particles. Benefiting from the excellent strength and superior ductility, the 12-pass alloy satisfies the mechanical benchmark for load-bearing orthopedic implantation.

Keywords: Zn alloys; plastic deformation; mechanical property; ECAP; texture; biodegradable alloys



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1. Introduction

Permanent metallic implants, such as stainless steels, titanium alloys and cobalt–chromium alloys, are widely used in clinics yet troubled by complications related to their bioinert properties. These issues cover stress-shielding-triggered osteopenia, periprosthetic inflammation and the requirement for costly secondary implant removal surgery [1–3]. These constraints have stimulated research on biodegradable metals, whose progressive resorption coincides with tissue repair and obviates secondary surgical procedures [4–6]. Zn possesses unique advantages in this material category. Its moderate electrochemical potential leads to corrosion rates between those of Mg and Fe, with a degradation rate of 0.1–0.3 mm/year compatible with bone remodelling cycles [5,7,8]. Zn is also the second richest transition metal in the human organism, serving as a catalytic cofactor for nearly

10% of proteins. Unlike Mg alloys, Zn degradation does not produce hydrogen, effectively eliminating the risk of subcutaneous gas pocket formation [9,10].

Nevertheless, poor mechanical performance restricts the structural application of pure Zn. As-cast pure Zn possesses a YS of merely 10 MPa and nearly no ductility, and wrought pure Zn exhibits a YS of only 33 MPa [11]. These mechanical metrics cannot meet the requirements of load-bearing orthopedic implants ($UTS \geq 300$ MPa, $YS \geq 200$ MPa and $EL \geq 20\%$) [12]. To overcome this limitation, biocompatible alloying elements (Mg, Mn, Ca, Li, Ag, Cu, etc.) have been widely adopted to enhance the mechanical performance [3]. The ternary Zn-Mn-Mg alloy stands out among existing systems. The synergistic addition of Mn and Mg has been demonstrated to enhance the comprehensive mechanical performance of Zn alloys without introducing toxic degradation products [13,14]. Both Mn and Mg are essential trace elements in human physiology. Furthermore, the sustained release of Mn and Mg ions promotes osteogenesis and provides antibacterial properties [15,16].

While compositional design establishes the thermodynamic foundation, severe plastic deformation (SPD) offers a potent metallurgical avenue for microstructural refinement. ECAP is preeminent among SPD techniques, imposing intense cumulative shear strain through repeated pressings along an angular die channel, thereby generating ultrafine-grained (UFG) architectures without dimensional loss [17]. Building on these fundamentals, ECAP has been extensively applied to biodegradable Zn-based alloys in recent years, consistently delivering simultaneous grain refinement and mechanical-property enhancement [18–25]. Bednarczyk et al. [20] reported that ECAP refines the grains of Zn-0.42Mn alloy to around 1 μm , endowing the alloy with very superior ductility ($EL = 93\%$) but a low UTS of 188 MPa. Zhao et al. [23] demonstrated that lowering the ECAP processing temperature is an effective strategy for further improving the strength–ductility balance of Zn alloys. A Zn-3.5Ag-0.08Mg alloy processed by ECAP at RT showing a very high UTS of 466 MPa and an excellent EL of 44% [23]. Xu et al. [26] reported a synergistic improvement of strength (UTS 399 MPa) and ductility in ECAP-processed Zn-Mg alloys through the refinement of grains and eutectic structures. Martynenko et al. [27] confirmed that ECAP-strengthened Zn-1Mg-0.1Dy alloys retain satisfactory biocompatibility both in vitro and in vivo, underscoring the clinical relevance of the ECAP route. Collectively, these studies confirm the efficacy of ECAP for the microstructural regulation of biodegradable Zn alloys. ECAP pass number is a pivotal processing parameter dominating the microstructural evolution and mechanical response of deformed alloys [28]. Nonetheless, systematic studies examining ternary Zn-Mn-Mg alloys across comparable deformation regimes remain conspicuously absent, leaving a critical research gap for the performance optimization of such biodegradable alloys.

This study was accordingly designed to bridge these knowledge gaps by systematically investigating the influence of ECAP pass number (4, 8, and 12) on the microstructure and mechanical properties of a Zn-0.5Mn-0.2Mg alloy. Characterization via scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) was employed to delineate microstructure evolution, with tensile testing establishing quantitative structure–property correlations. The findings are anticipated to furnish critical insights into the optimization of ECAP protocols for Zn-Mn-Mg alloys and to advance the development of mechanically tailored biodegradable implants for orthopedic applications.

2. Materials and Methods

The Zn-0.5Mn-0.2Mg alloy was fabricated by induction melting of pure Zn (99.99%), a Zn-10Mn master alloy, and pure Mg in a graphite crucible at 680 °C under a flowing CO_2/SF_6 shielding gas to inhibit oxidation. The melt was cast into a preheated carbon steel mold (200 mm $\times$ 75 mm $\times$ 50 mm) and permitted to cool in still air. Rectangular

prisms measuring $19.5 \text{ mm} \times 19.5 \text{ mm} \times 45 \text{ mm}$ were sectioned from the cast ingot via wire electrical discharge machining. These billets were subjected to a homogenization anneal at 330°C for 12 h, then immediately quenched into water. The billets were ECAP-pressed at ambient temperature for cumulative passes of 4, 8, and 12. A rotary-die ECAP setup was employed, with an ECAP die with an internal channel angle of 90° and an outer corner angle of 0° , as shown in Figure 1. For this die geometry, the equivalent strain per single pass is ~ 1.15 [29]. Accordingly, the cumulative equivalent strains reached ~ 4.6 , ~ 9.2 and ~ 13.8 for 4, 8 and 12 passes, respectively. ECAP Route Bc was implemented: the billet was rotated 90° about the pressing axis with the same rotation sense between successive passes. No back-pressure was applied. The ram pressing speed was fixed at 0.2 mm/s . A mixture of MoS_2 and graphite was used as lubricant between billet and die channel. The billet longitudinal axis coincided with the extrusion direction (ED). The time interval between consecutive passes was approximately 1 min. The resulting materials are referred to herein as 4P, 8P, and 12P, respectively.

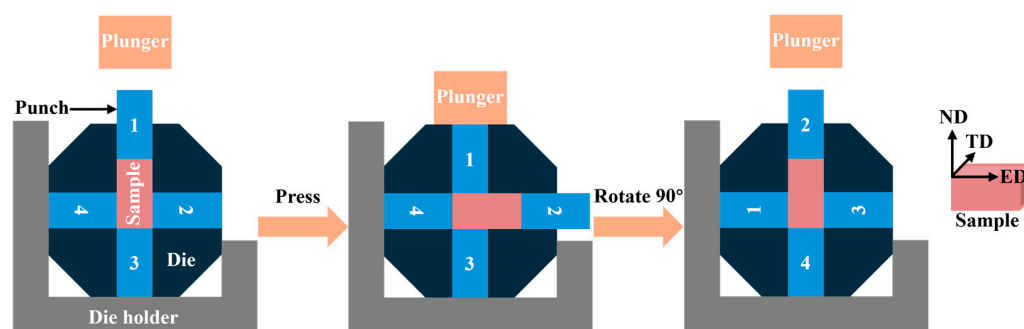


Figure 1. Schematic of the rotary-die ECAP setup, with ED, ND, and TD denoting the extrusion, normal, and transverse directions, respectively.

Mechanical behavior was evaluated through uniaxial tension at room temperature using a universal testing machine. Flat dog-bone specimens with a 6 mm gauge length were extracted from the central region of the pressed billets such that the tensile axis coincided with the ED. Tests were conducted under displacement control at a crosshead speed of 0.5 mm min^{-1} . For each processing state, no fewer than three specimens were tested, and the mean values of YS, UTS, and EL together with corresponding standard deviations of YS, UTS, and EL were computed. The reported EL values are elongation-to-failure.

For metallographic examination, samples were prepared by sequential grinding on SiC papers up to 2000 grit, polishing with $2.5 \mu\text{m}$ diamond paste, and chemical etching in an aqueous solution of 2 g CrO_3 and 0.15 g Na_2SO_4 per 10 mL H_2O . These surfaces were examined via SEM coupled with energy-dispersive spectroscopy (EDS). For EBSD, the final surface preparation involved electropolishing in a perchloric acid–ethanol solution (5 vol.% HClO_4 , 95 vol.% $\text{C}_2\text{H}_5\text{OH}$) at 450 mA cm^{-2} for 4.5 min. EBSD characterization was performed with an accelerating voltage of 20 kV. The EBSD mapping step size was set to $0.05 \mu\text{m}$.

3. Results

Figure 2a displays engineering stress–strain curves of the as-cast, 4P, 8P, and 12P alloys. Table 1 exhibits the YS, UTS and EL of the studied alloys. The as-cast alloy exhibits inferior mechanical performance, with very low strength (YS = 87 MPa and UTS = 98 MPa) and limited ductility (EL = 3%). It should be noted that all ECAP-processed samples were subjected to homogenization annealing and water quenching before deformation. Hence, the direct comparison between as-cast and ECAP-processed states includes contributions from both heat-treatment and ECAP deformation. Our analysis of ECAP-induced property

evolution is primarily based on the trend across 4P, 8P and 12P, which possess identical pre-ECAP heat-treatment history. After ECAP, both strength and ductility are progressively enhanced with increasing ECAP passes. The 4P alloy achieves a YS of 279 MPa and a UTS of 311 MPa. Although strength is dramatically improved relative to the as-cast alloy, the EL is still low (12%). When the ECAP pass number increases to 8, the YS and UTS rise to 287 and 323 MPa, respectively, accompanied by a moderate improvement in ductility with EL reaching 17%. The 12P alloy delivers optimal mechanical properties, with the maximum strength (YS = 298 MPa and UTS = 348 MPa) and a dramatically elevated EL of 41%, exceeding the mechanical benchmark of medical implants [12]. It should be emphasised that adequate corrosion resistance and cytocompatibility still need to be validated in follow-up investigations. Figure 2b compares the UTS-EL performance of the present Zn-0.5Mn-0.2Mg alloy with recently reported Zn-Mn based alloys. The 12P alloy is positioned in the high-performance zone of the scatter plot. Its outstanding strength–ductility synergy is superior to recently developed Zn-Mn based alloys.

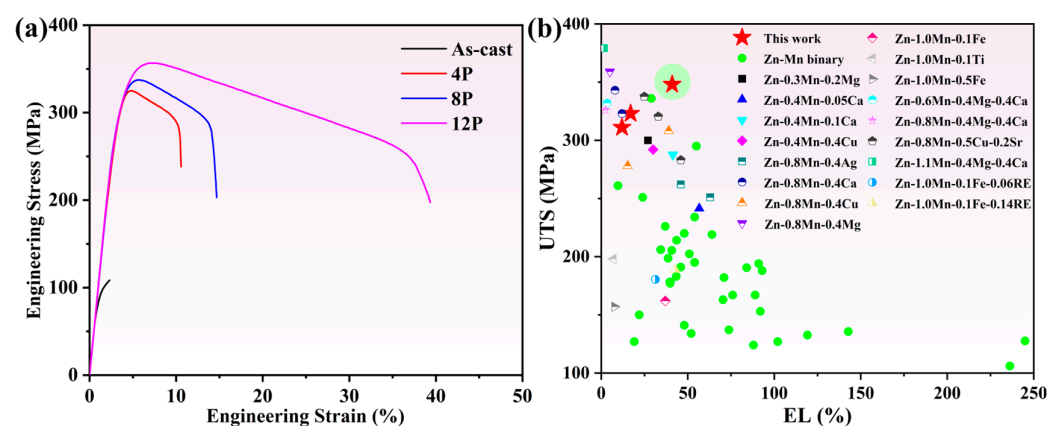


Figure 2. (a) Engineering stress–strain curves of as-cast, 4P, 8P, and 12P alloys, (b) EL and UTS of 4P, 8P, and 12P alloys, compared with recently developed wrought Zn-Mn based alloys [16,20,22,30–47].

Table 1. YS, UTS, and EL of the as-cast, 4P, 8P, and 12P alloys.

Alloy	YS (MPa)	UTS (MPa)	EL (%)
As-cast	87 ± 6	98 ± 5	3 ± 1
4P	279 ± 9	311 ± 7	12 ± 4
8P	287 ± 11	323 ± 9	17 ± 3
12P	298 ± 8	348 ± 6	41 ± 6

Figure 3 presents SEM micrographs illustrating the microstructural evolution of ECAP-processed Zn-0.5Mn-0.2Mg alloy subjected to 4, 8 and 12 passes. Numerous particles are distributed within the Zn matrix of 4P, 8P, and 12P alloys. Two categories of intermetallic phases are observed. Phase assignments to MnZn_{13} and $\text{Mg}_2\text{Zn}_{11}$ are proposed based on local EDS point-composition analysis (Table 2). The occurrence of MnZn_{13} and $\text{Mg}_2\text{Zn}_{11}$ intermetallic phases have been widely reported in previous investigations on comparable biodegradable Zn-Mn-Mg alloys [43,48,49]. MnZn_{13} phases exist in the 4P alloy in two distinct morphologies: large block-shaped particles with an average size exceeding 5 μm , and fine particles with an average dimension of approximately 1 μm . Increasing the number of ECAP passes to 8 triggers fragmentation of coarse MnZn_{13} intermetallics, whereas the dimensions of fine MnZn_{13} particles remain nearly unchanged. As ECAP passes rise to 12, coarse MnZn_{13} particles are further fragmented, and fine MnZn_{13} counterparts exhibit negligible size evolution. This trend confirms that accumulated shear strain induced by multi-pass ECAP effectively breaks down coarse intermetallic phases, consistent with

the established mechanism of SPD-mediated particle fragmentation reported in previous biodegradable Zn alloy studies [49,50]. The elemental mapping presented in Figure 4 corroborates the phase identification. In all three processing states, Zn is homogeneously distributed across the matrix, whereas Mn and Mg exhibit localized enrichment at the particle sites.

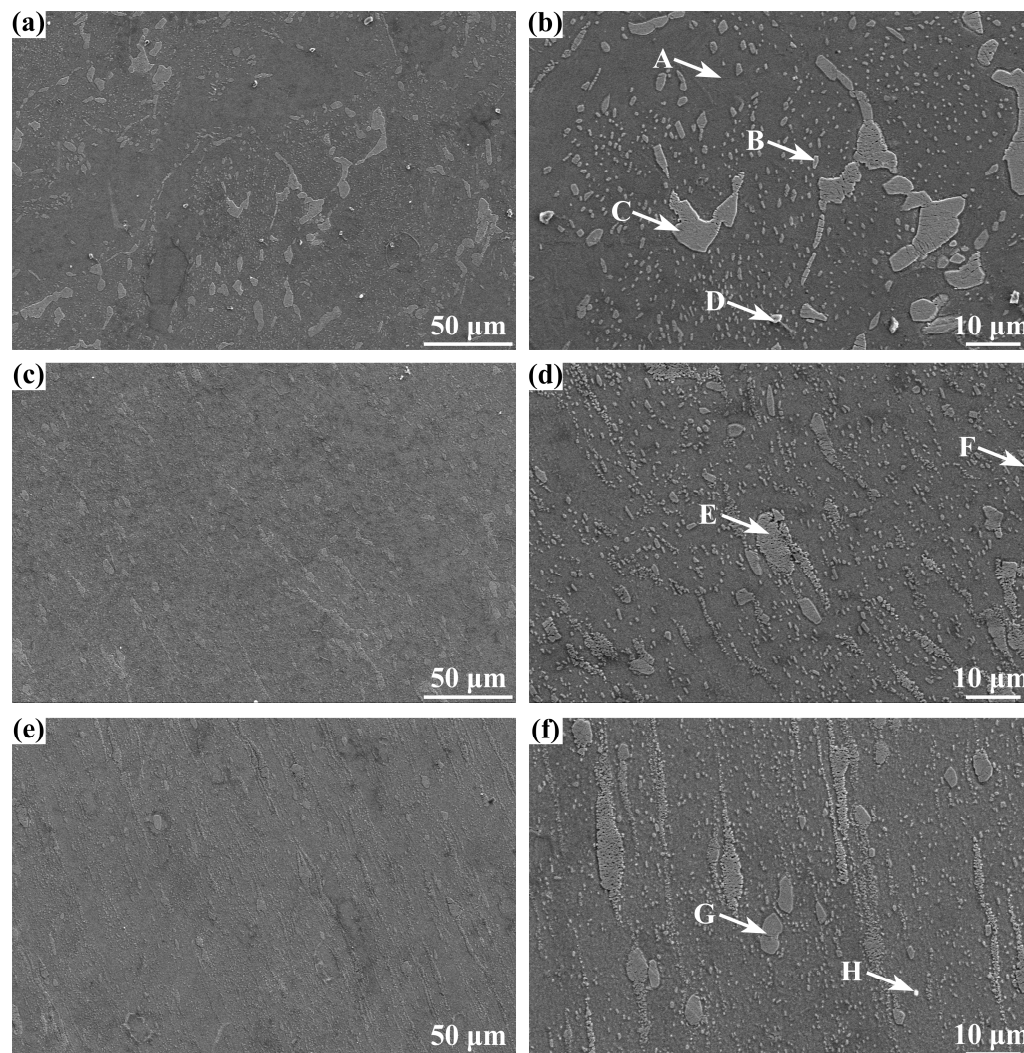


Figure 3. SEM images of (a,b) 4P, (c,d) 8P, and (e,f) 12P alloys. EDS results of points A–H are given in Table 2. Tensile data for the as-cast alloy are included for reference, while microstructural characterisation was not conducted on the as-cast specimen in this study.

Table 2. Chemical composition of points A–H in Figure 3.

Point	Zn (at.%)	Mn (at.%)	Mg (at.%)	Phase
A	99.27	0.52	0.21	η -Zn
B	90.86	8.01	1.13	MnZn ₁₃
C	92.15	7.57	0.29	MnZn ₁₃
D	84.86	0.83	14.31	Mg ₂ Zn ₁₁
E	91.40	8.50	0.10	MnZn ₁₃
F	84.73	0.74	14.53	Mg ₂ Zn ₁₁
G	91.35	8.07	0.58	MnZn ₁₃
H	82.91	0.91	16.18	Mg ₂ Zn ₁₁

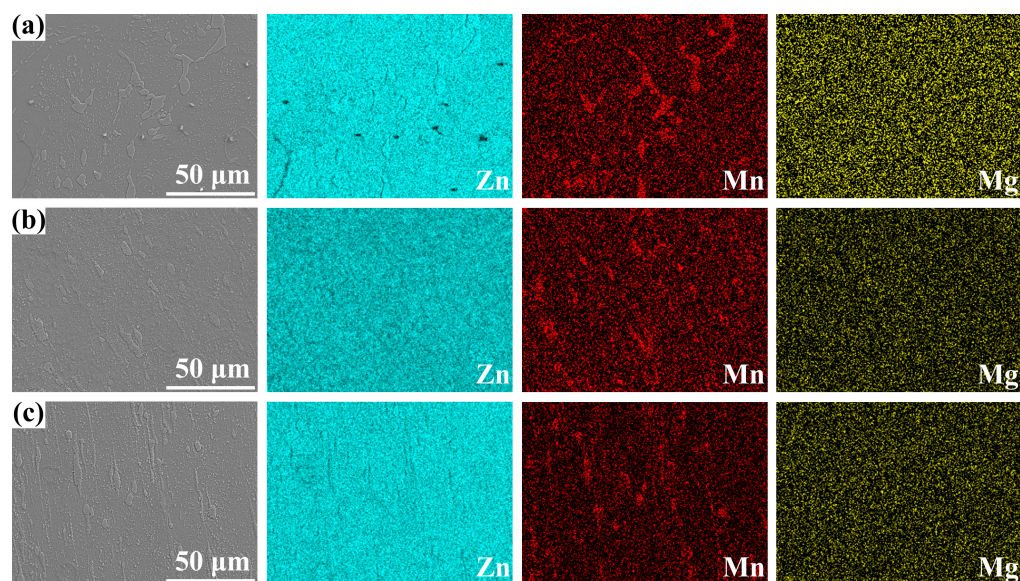


Figure 4. SEM images showing elemental distribution of (a) 4P, (b) 8P, and (c) 12P alloys.

The inverse pole figure (IPF) maps and corresponding grain-size histograms presented in Figure 5 reveal a pronounced refinement of the grain structure as the number of ECAP passes increases from 4 to 12. In the 4P alloy (Figure 5a), the microstructure is characterized by an average grain size (AGS) of 1.94 μm , with a notably broad distribution spanning approximately 0.5–7 μm . Grain refinement originates from dynamic recrystallization (DRX). The low melting point of Zn facilitates DRX even at ambient temperature [20]. The IPF map further indicates that many grains retain an elongated morphology aligned along the ED, interspersed with clusters of finer, equiaxed grains. This heterogeneous architecture suggests that DRX is only partially completed after 4 passes, leaving a substantial fraction of deformed matrix with stored dislocation substructures. This heterogeneity is corroborated by the high fraction of low-angle grain boundaries (LAGBs), which reaches 39.1% (Figure 6a,d), indicating that a substantial volume of the matrix remains in a deformed state with abundant dislocation substructures.

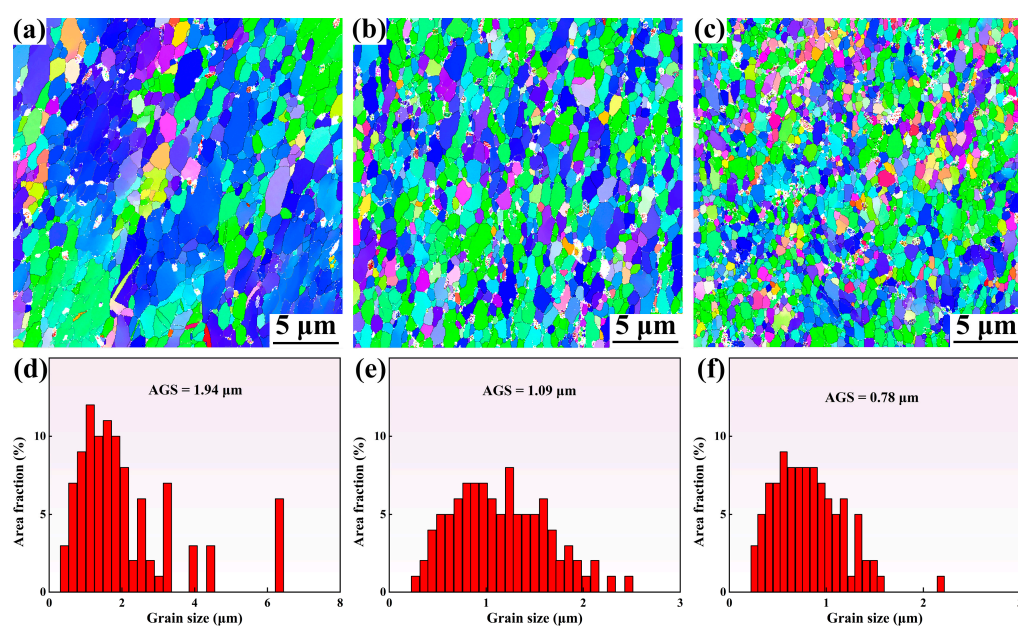


Figure 5. IPF maps of (a) 4P, (b) 8P, and (c) 12P alloys; grain size histograms of (d) 4P, (e) 8P, and (f) 12P alloys.

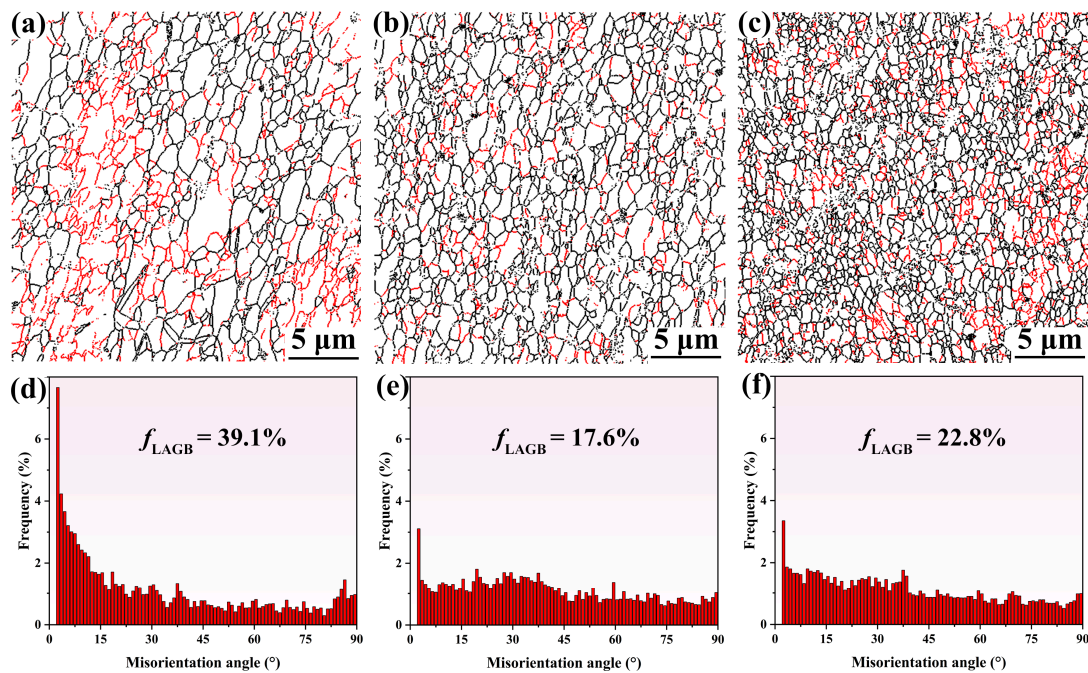


Figure 6. GB maps of (a) 4P, (b) 8P, and (c) 12P alloys with black line denoting HAGB and red line representing LAGB; misorientation angle histograms of (d) 4P, (e) 8P, and (f) 12P alloys.

Upon increasing the deformation to 8 passes (Figure 5b), the AGS decreases markedly to 1.09 μm, and the grain-size distribution narrows considerably (Figure 5e). Concomitantly, the LAGB fraction drops to 17.6% (Figure 6b,e), the lowest value among the three processing states. The corresponding IPF map displays a markedly more homogeneous and equiaxed grain morphology with a high density of high-angle grain boundaries (HAGBs). These observations collectively signify that DRX has proceeded to an advanced stage by 8P, whereby geometrically necessary dislocations (GNDs) reorganize into subgrain boundaries that progressively accumulate misorientation and ultimately transform into HAGBs [23,51,52].

After 12 passes (Figure 5c), the AGS is further refined to 0.78 μm, and the histogram exhibits a tight, unimodal distribution centred in the submicrometre regime (Figure 5f). Interestingly, however, the LAGB fraction exhibits a modest rebound to 22.8% (Figure 6c,f). This slight increase relative to the 8P state suggests that, although the overall grain structure remains ultrafine and equiaxed, the additional cumulative strain introduces fresh dislocation networks and subgrain walls within the already recrystallized grains. Such a phenomenon is consistent with the operation of competing mechanisms at high strain levels: while DRX continues to consume dislocations and generate HAGBs, the ever-increasing imposed strain simultaneously nucleates new LAGBs inside the refined grains before they can fully convert to HAGBs.

Kernel average misorientation (KAM) mapping and the associated local misorientation angle histograms (Figure 7) provide quantitative insight into the spatial distribution of plastic strain and the evolution of GND densities across the ECAP-processed microstructures. The average KAM value registers 0.35° for the 4P alloy, decreases to a minimum of 0.31° at 8P, and subsequently rebounds to 0.34° after 12 passes. This non-monotonic trajectory mirrors the evolution of LAGB fraction (Figure 6), corroborating a transition from dislocation-dominated deformation to advanced DRX, followed by a regime of renewed dislocation accumulation within the UFG matrix.

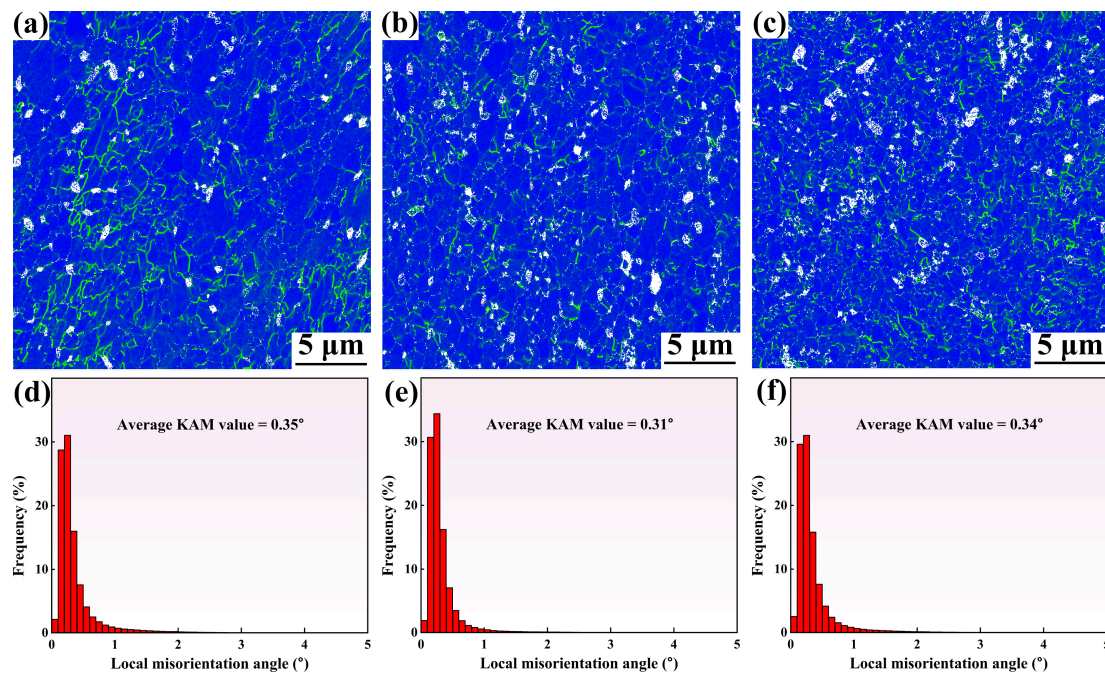


Figure 7. KAM maps of (a) 4P, (b) 8P, and (c) 12P alloys; local misorientation angle histograms of (d) 4P, (e) 8P, and (f) 12P alloys.

In the 4P state, the KAM map (Figure 7a) exhibits a heterogeneous distribution of elevated misorientation, with numerous high-KAM filaments and clusters pervading the microstructure. These regions correspond to areas of intense dislocation tangles and incipient subgrain boundaries within the partially recrystallized matrix, consistent with the high LAGB fraction (39.1%). The relatively high average KAM of 0.35° indicates that the cumulative strain has generated substantial GND densities, yet the thermal and mechanical driving forces at this stage remain insufficient to fully consume these defects via boundary migration and annihilation.

Upon increasing the pass number to 8, the average KAM drops to 0.31° (Figure 7b), and the spatial distribution becomes markedly more homogeneous. The histogram reveals a sharper peak concentrated at low misorientation angles, signifying that most grains have undergone extensive DRX and that the stored dislocation energy has been substantially relieved. This minimum in KAM coincides with the lowest LAGB fraction (17.6%) and the most equiaxed grain morphology. This indicates that at this intermediate strain level, the rate of dislocation annihilation and HAGB conversion during DRX exceeds the rate of new dislocation generation by the imposed shear.

At 12 passes, the average KAM exhibits a discernible rebound to 0.34° (Figure 7c), despite the finest AGS ($0.78\ \mu\text{m}$) and a predominantly HAGB-dominated structure. Several interconnected factors account for this behavior. First, the extreme cumulative strain continuously nucleates new dislocations within the already refined grains, and the diminished mean free path for dislocation motion in the submicrometre regime promotes rapid accumulation at intragranular obstacles and in the vicinity of grain boundaries. Second, the increased grain boundary area in the UFG microstructure elevates compatibility stresses at triple junctions and HAGBs, necessitating the storage of additional GNDs to accommodate strain gradients. Third, the modest increase in LAGB fraction (to 22.8%) at 12P reflects the formation of fresh dislocation substructures that have not yet been fully assimilated into HAGBs by DRX. Consequently, the 12P microstructure represents a dynamic equilibrium wherein recrystallization continues to operate but is counterbalanced by strain-induced dis-

location generation, a phenomenon characteristic of high-strain severe plastic deformation in metals with low stacking-fault energy.

The {0001} pole figures (PFs) presented in Figure 8 reveal a distinct two-stage evolution of crystallographic texture as the ECAP pass number increases. In the 4P alloy (Figure 8a), a strong tilted-basal fiber texture is established, with the $\langle 0001 \rangle$ poles concentrated in a locus inclined approximately $20\text{--}30^\circ$ from the ED, and the maximum pole density reaches 15.1 multiples of uniform distribution (MUD). This texture component is characteristic of ECAP-processed Zn alloys and arises from the cooperative activation of basal $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip [53,54].

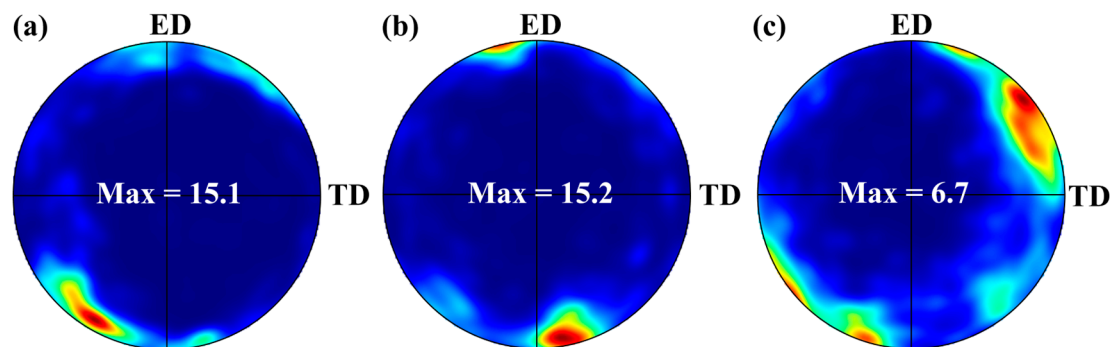


Figure 8. {0001} pole figures of (a) 4P, (b) 8P, and (c) 12P alloys.

Upon increasing the pass number to 8 (Figure 8b), the maximum texture intensity registers 15.2 MUD, virtually identical to that of the 4P alloy. This reflects that the texture type dictated by ECAP and the limited HCP slip systems has already been established by the early deformation stage. The intermediate cumulative strains primarily refine the grain size and convert LAGBs into HAGBs without substantially reorienting the dominant texture fiber. Indeed, the 8P alloy exhibits the lowest LAGB fraction (17.6%) and the minimum KAM value (0.31°), indicating the most advanced state of continuous dynamic recrystallization (CDRX). During CDRX, subgrains gradually rotate and absorb dislocations to transform into new grains, yet these nascent grains retain crystallographic orientations closely related to their parent grains, thereby preserving the overall texture fabric [18].

Upon further deformation to 12 passes (Figure 8c), the maximum pole density precipitously declines to 6.7 MUD. In addition, the basal poles become markedly more dispersed, forming a broad annular distribution rather than a tightly focused cluster. This dramatic texture attenuation is attributed to the confluence of three synergistic mechanisms. ECAP subjects the material to shear on multiple intersecting planes due to the 90° rotation of the billet between successive passes. This multi-axial shear effect becomes particularly pronounced at high pass numbers, where the superposition of shear on orthogonal planes overwhelms the single-slip-system-dominated texture development observed at lower strains [21]. Moreover, as the AGS approaches the submicrometre threshold ($0.78\text{ }\mu\text{m}$ at 12P), grain-boundary-mediated deformation mechanisms (including grain rotation and grain boundary sliding) become increasingly operative. These mechanisms do not rely on the activation of specific crystallographic slip systems and therefore contribute to the dispersion of preferred orientations [21]. The elevated grain boundary area in the UFG microstructure elevates compatibility stresses at triple junctions, further promoting orientation gradients that weaken the macroscopic texture intensity.

4. Discussion

The tensile strength of the Zn-0.5Mn-0.2Mg alloy exhibits a monotonic upward trend as the ECAP pass number increases from 4 to 12. Grain-boundary strengthening (σ_{HP})

constitutes the principal contributor to the elevated strength across all processing states. It is quantified through the Hall–Petch relationship [55]

$$\sigma_{\text{HP}} = \sigma_0 + kd^{-0.5} \quad (1)$$

where σ_0 ($\sigma_0 = 20$ MPa [56]) denotes lattice friction stress, k ($k = 219$ MPa $\cdot\mu\text{m}^{0.5}$ [57]) represents the Hall–Petch slope, and d is AGS. The AGS diminishes continuously from 1.94 μm (4P) to 1.09 μm (8P) and further to 0.78 μm (12P), as shown in Figure 5. Based on Equation (1) and parameters adopted from the literature [56,57], the grain-boundary contribution to YS escalates from approximately 177 MPa (4P) to 230 MPa (8P) and 268 MPa (12P). Such an upward trend confirms that grain-boundary strengthening dominates the overall strength enhancement of the present Zn-Mn-Mg alloy. The adopted Hall–Petch slope is sourced from an HPT-processed Zn-0.8Ag alloy [57]. This constant may overestimate grain-boundary strengthening for submicron grains in the present Zn-0.5Mn-0.2Mg alloy. At room temperature, grain-boundary mediated deformation may occur in ultrafine-grained Zn systems [33,34], weakening the Hall–Petch relationship below 1 μm , as highlighted in Ref. [20]. Therefore, the calculation here is a semi-quantitative estimate rather than an exact prediction.

The contribution of dislocation strengthening (σ_d) was assessed qualitatively. According to the Bailey–Hirsch relation [58]

$$\sigma_d = M\alpha Gb\sqrt{\rho} \quad (2)$$

where M , α , G , b , and ρ represent Taylor factor, a constant, shear modulus, Burgers vector, and GND density, respectively. σ_d increases monotonically with the square root of the dislocation density. A quantitative evaluation is not attempted here because well-established Taylor factors and dislocation-interaction coefficients specific to Zn alloys are not available. Importantly, M , α , G and b are material constants identical for the three processing states and therefore cannot influence the relative comparison: the trend of σ_d is governed exclusively by ρ , which is reflected in the measured KAM values ($0.35^\circ \rightarrow 0.31^\circ \rightarrow 0.34^\circ$, Figure 7). The KAM minimum at 8P and its modest rebound at 12P accordingly indicate a dislocation-strengthening contribution that is moderate at 4P, weakest at 8P and slightly recovered at 12P, consistent with the evolution of the LAGB fraction ($39.1\% \rightarrow 17.6\% \rightarrow 22.8\%$).

The contribution of second-phase strengthening is negligible. The MnZn_{13} and $\text{Mg}_2\text{Zn}_{11}$ particles remain predominantly in the micrometre size regime even after 12 passes. According to the Orowan–Ashby criterion, such coarse dispersoids exert negligible direct strengthening because their interparticle spacing is too large to effectively pin gliding dislocations. Therefore, second-phase strengthening arising from MnZn_{13} and $\text{Mg}_2\text{Zn}_{11}$ particles can be neglected, consistent with previous studies [13,48]. Their principal metallurgical role instead lies in promoting particle-stimulated nucleation (PSN) of dynamic recrystallization.

The EL of the present alloy exhibits a monotonic increase with rising ECAP pass number, increasing from 12% (4P) through 17% (8P) to a remarkable 41% (12P). The limited ductility observed for the 4P sample can be rationalized from three microstructural perspectives. First, the alloy develops a strong crystallographic texture with an intensity of 15.1 MUD, which is well-known to suppress plastic deformation capacity in hexagonal close-packed (HCP) Zn alloys and thus deteriorate ductility. Second, the 4P sample delivers the highest average KAM value (0.35°), indicative of a larger GND density within the deformed matrix. The high residual dislocation density leaves limited spare capacity for further dislocation accumulation during tensile loading, which restricts uniform plastic elongation. Third, according to the established literature on Zn-based biodegradable

alloys [49,59], coarse intermetallic particles (such as MnZn_{13} and $\text{Mg}_2\text{Zn}_{11}$) may act as preferential crack-initiation sites under tensile stress, which can accelerate premature fracture and impair ductility. For the 8P alloy, no obvious texture evolution is detected, whereas the average KAM value decreases to 0.31° and the coarse intermetallic particles are fragmented by intense shear strain imposed during ECAP. The combined effects of reduced residual dislocation density and refined second-phase particles contribute to the moderate rise in elongation to 17%. The most prominent microstructural transformation takes place upon further deformation to 12 passes, accompanied by a sharp surge of elongation up to 41%. After 12 ECAP passes, the crystallographic texture is markedly dispersed, and its peak intensity drops significantly to 6.7 MUD. Weakened texture increases the Schmid factor for pyramidal $\langle c+a \rangle$ slip, thereby facilitating the activation of this critical non-basal deformation mode and improving the plastic compatibility of the Zn matrix. Meanwhile, coarse second-phase particles are further broken down into finer fragments, which reduces the risk of crack nucleation at particle–matrix interfaces. Given the submicrometre grain size of $0.78\text{ }\mu\text{m}$, grain boundary sliding may also make some contribution to the large room-temperature elongation. Consequently, the 12-pass Zn-0.5Mn-0.2Mg alloy achieves an excellent elongation of 41%.

5. Conclusions

The present study systematically investigated the influence of ECAP pass number (4, 8, and 12) on the microstructural evolution and mechanical properties of a Zn-0.5Mn-0.2Mg alloy. Progressive accumulation of shear strain monotonically refined the grain structure, reducing the AGS from $1.94\text{ }\mu\text{m}$ (4P) to $1.09\text{ }\mu\text{m}$ (8P) and $0.78\text{ }\mu\text{m}$ (12P). Concurrently, the KAM value exhibited a non-monotonic trajectory, decreasing from 0.35° (4P) to a minimum of 0.31° (8P) before rebounding to 0.34° (12P). The tilted basal texture remained strong and stable at approximately 15.1–15.2 MUD after 4 and 8 passes, but weakened precipitously to 6.7 MUD at 12 passes. Further experiments with higher ECAP passes are recommended to validate this transition and examine the saturation of grain refinement. Both YS and UTS increased monotonically with pass number. Grain boundary strengthening constituted the dominant mechanism, supplemented by modest dislocation strengthening. EL improved progressively from 12% (4P) and 17% (8P) to a remarkable 41% (12P). The exceptional ductility of 12P alloy is ascribed to the synergistic effects of pronounced texture weakening and refined second-phase particles. Consequently, the 12P alloy simultaneously attained the highest strength and the highest ductility within the investigated processing window. The 12P alloy meets the rigorous mechanical requirements for load-bearing orthopedic implantation. Nevertheless, further studies concerning corrosion behaviour, degradation performance and cytocompatibility are necessary before considering this alloy for clinical orthopedic applications.

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