Microstructure and mechanical properties of a Zn-0.5Cu alloy processed by high-pressure torsion

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Abstract

The microstructure, texture and mechanical properties of a quasi-single-phase Zn-0.5Cu (wt. %) alloy processed by high-pressure torsion (HPT) for up to 10 turns were investigated using electron backscatter diffraction (EBSD), Vickers hardness measurements and uniaxial tensile tests. The results show that during torsional straining there is dynamic recrystallization, subgrain refinement, a dissolution of ε – Zn_4Cu precipitates and solid-solution strengthening. Monotonic deformation develops a strong $\{0001\}\langle 11\bar{2}0\rangle$ local texture instead of the characteristic basal fiber texture. Sharp texture and misorientation angles for all grain boundaries of < 30° causes significantly higher yield stress and ultimate tensile stress compared to processing of the alloy by equal-channel angular pressing.

Keywords: grain refinement; high-pressure torsion; severe plastic deformation; ultrafine grains; zinc alloys

1. Introduction

Zinc alloys are promising biodegradable structural materials [1,2], and recent research has focused on the manufacturing, processing and characterization of new Zn-based alloys. Zinc alloys are generally processed using conventional hot rolling or extrusion sometimes followed by cold rolling [3–6] and the application of severe plastic deformation (SPD) processing methods has received less attention. Recently, equal channel angular pressing (ECAP) and hydrostatic extrusion have been used to produce high-alloyed high-strength Zn-based alloys [7,8] while for low-alloyed Zn-alloys the same processing methods decrease the strength and may activate room-temperature (RT) superplasticity [9–11].

Very few reports are at present available on the processing of Zn and its alloys using high-pressure torsion (HPT), although some results are available analyzing the effect of HPT on the microstructure and mechanical properties of pure Zn [12–14]. The relatively low melting temperature (T_M) of Zn causes very high dislocation annihilation and relatively easy recovery and recrystallization during and immediately after processing [15,16]. Thus, no substantial strengthening effect was recorded and steady-state grain sizes were measured from 5.2 to 19 μm. Only one dual-phase eutectoid Zn-22Al alloy has been investigated after HPT processing, and then the presence of a second phase leads to significant grain refinement to ~350 nm after 5 turns under a pressure of 6.0 GPa [17–21]. In contrast to pure zinc, the dual-phase Zn alloy exhibits a strain softening behavior [22]. Besides the conventional alloys, Zn-Mg hybrids produced by HPT processing have been recently reported [23,24]. Diffusional bonding allowed the joining of Zn/Mg/Zn layers into a single compact disc of Zn-Mg material; however, visible inhomogeneity in the cross-sections leads to an ambiguous determination of chemical and phase compositions considering the entire disc volume. Thus, microstructure and mechanical properties significantly vary depending on the amount of Zn, Mg or Zn-Mg phases.

The present research was conducted to provide the first experiments on a low-alloyed quasi-single-phase Zn-based alloy processing by HPT. For this investigation, a Zn-0.5Cu (wt. %) alloy was selected because earlier research using ECAP showed that this alloy provides a capability of exhibiting RT superplasticity and significant grain refinement by comparison with pure Zn [9,11]. The investigation was undertaken with two main objectives. First, to systematically analyze the evolution of microstructure and texture after processing by HPT through different numbers of turns. Second, to determine the effect of microstructural changes on the mechanical properties after processing by HPT.

2. Materials and methods

The alloy was prepared from high purity (> 99.995 wt. %) zinc and high-purity Cu-40%Zn brass (< 0.007 wt. of impurities) through melting in a graphite crucible at 650°C in air, homogenizing for 30 minutes and subsequently casting into a cylindrical steel mold. The cast billets were annealed at 400°C for 4 hours to homogenize the chemical composition and subsequently water-cooled. The chemical composition of the material was analyzed using a Fischerscope® XDV-SDD X-ray fluorescence spectrometer and the results showed 0.50 ± 0.03 wt. % Cu with Pb, Fe and Cd content below the 0.005 wt. % detection limit. HPT samples with a diameter of 9.8 mm and thickness of approximately 0.8 mm were machined, ground and polished to remove any deformed layer.

The processing by HPT was conducted at room temperature using quasi-constrained conditions where there is a small outflow of material around the periphery of the disk during processing [25]. For all samples, the applied pressure was 6.0 GPa with a constant rotation speed of 1 rpm. Care was taken to ensure there was no slippage during processing [26] and the rotation speed suggests a minor temperature rise of ~10°C [27]. A set of disks was processed through total turns of $N = 0$ (only compression for 60 seconds), 1/2, 1, 2, 5 and 10.

One disk sample after each number of turns was prepared for detailed microstructural observation using a scanning electron microscope (SEM). To avoid any heating induced changes in the microstructure, specimens were cold-mounted in epoxy resin and then grinding and polishing were conducted using water-resistant abrasive papers up to #2000 and water-free diamond suspensions with grain sizes of 3 and 1 μm. The final step was low-angle Ar^+ ion polishing for 15 minutes at 4° and 4 kV using a Hitachi IM4000Plus Ion Milling System. Ion polishing was used to remove the deformed surface layer and thus significantly increase the EBSD pattern quality. SEM-EBSD observations were conducted at a position located ~2.5 mm from the centre of the HPT disk using an FEI Versa 3D SEM equipped with a field-emission gun and EDAX OIM TSL EBSD collecting system. The step size was set at least 10 times smaller than the observed subgrain structure. One iteration of the grain dilation cleanup procedure was performed. Grains and subgrains were defined as a set of at least five measurement points, surrounded by a continuous grain boundary segment with a misorientation of at least 15° and 3°, respectively. Using the EBSD data it was possible to calculate the grain size, grain boundary misorientation angle distribution and the texture as pole figures (PF). The EBSD data analysis including texture analysis was performed using the MTEX Toolbox in Matlab [28].

The mechanical properties after HPT processing were evaluated in two ways as illustrated schematically in Fig. 1. First, the HPT disk was mechanically polished to a thickness of approximately 0.6 mm and then two miniature tensile specimens were cut using wire electro-discharge machining (WEDM) to give gauge lengths and widths of 1.5 and 0.7 mm, respectively. Tensile testing was conducted at RT using an INSTRON 5966 universal testing machine with 1 kN load cell. Tests were performed at the three strain rates of 10^{-2}, 10^{-3} and 10^{-4} s^{-1}. The tensile curves were used to measure the yield stress (YS), ultimate tensile stress (UTS) and elongation to failure (E_T). Additionally, the strain rate sensitivity parameter *m* was calculated based on the slope of the double-logarithmic plot of flow stress at 15% of elongation against strain rate. Second, the SEM samples were used for Vickers hardness (Hv) measurements at specific points on a line across the diameter of each disk (see Fig. 1). The distances between indents were set at 0.5 mm for all samples and Hv was determined as the average of three measurements recorded at the same distance from the centre using a load of 2.95 N (HV0.3) with a holding time of 10 s.

3. Results

The effect of torsional straining on microstructure during HPT processing depends on the distance from the disk center and the number of rotations. Fig. 2 shows EBSD maps at positions of ~2.5 mm from the centres of the disks after a) 0, b) 1/2, c) 1, d) 2, e) 5, and f) 10 turns, respectively. After compression (Fig 2a), there are a significant number of twins both inside larger grains and within the primary twins. After 1/2 turn (Fig. 2b), it is apparent that the severe torsional straining causes significant grain refinement but the microstructure is inhomogenous with coarse equiaxed grains of ~20 μm separated by regions of fine-grained, deformed grains. Further straining to 1 turn (Fig. 2c) produces an equiaxed structure with a small number of twins and with the grain size reduced to ~9.9 μm. Increasing to 2 turns, there is additional grain refinement (Fig. 2d) to ~7.9 μm. The measured subgrain size decreases from ~5.1 to ~3.9 μm after 1 and 2 turns, respectively. After 5 and 10 turns (Fig 2e,f) the grain size increases to ~29 and ~31 μm but the subgrain size continues to decrease to a saturated value of ~1.9 μm.

Fig. 3 shows the (0001), $(10\bar{1}0)$ and $(11\bar{2}0)$ PF of the HPT-processed samples obtained from the EBSD data. After compression (Fig. 3a), the number of grains in the observation area was not high enough in order to produce a statistically significant texture. After 1/2 rotation (Fig. 3b), a relatively weak and slightly tilted basal fiber texture developed. Further torsional shearing through 1 and 2 turns (Fig. 3c and d) promoted a basal fiber texture with a small $\{0001\}\langle 11\bar{2}0\rangle$ texture contribution visible as peaks parallel to the shearing direction in the $(11\bar{2}0)$ PF. Increasing the number of turns to 5 and 10 gave a very sharp texture consisting of two components: basal fiber and $\{0001\}\langle 11\bar{2}0\rangle$ texture [29]. The texture was analyzed to calculate the volume fractions of the two components. Volume fractions were obtained as a ratio of the orientations in the orientation distribution function (ODF) within the misorientation tolerance from specified reference orientation. For the texture components measured, a misorientation tolerance of 5 degrees was used. In the case of the basal fiber the reference was specified by the crystal direction ⟨c⟩ parallel to the sample normal direction (Z). For the $\{0001\}\langle 11\bar{2}0\rangle$ component, the reference orientation was determined by finding the modal orientation (peak) of a given ODF [30]. The modal orientation coincided with the six-fold symmetry of maxima in the prismatic pole figures after HPT with N>0. In the case of pure compression N=0, no modal orientation within the misorientation tolerance from the $\{0001\}\langle 11\bar{2}0\rangle$ orientation was found. The contribution of each component in the texture is presented in the Figure 4. Up to 2 turns, the fraction of the basal fiber component reaches about 5 % but after 5 it increases significantly to ~25 %.

Further deformation produces no change in the basal fiber component. The $\{0001\}\langle 11\bar{2}0\rangle$ component changes from 0 % at N = 0 to ~40 % after 2 turns and continuously increases up to ~55 % after 10 turns. The sharp texture formed during continuous shearing up to N = 10 gives a very high PF intensity of up to 95.

The correlated misorientation distributions were calculated using the MTEX toolbox [28] in Matlab based on collected EBSD data and the results are presented in Figure 5. In the coarse-grained microstructure after pure compression (Fig. 5a), the preferred misorientation angle is ~86 ± 3° which is related to the $\{10\bar{1}2\}\langle 11\bar{2}0\rangle$ twinning [31]. Applying the torsional straining for 1/2 turn (Fig. 5b) causes significant changes in the boundary misorientation distribution such that the 86 ± 3° twinning peak is almost eliminated, and the contribution of low-angle grain boundaries (LAGB) increased compared to the initial state. After 1 turn (Fig. 5c), the maximum shifted to 30° and the frequency of boundaries below this angle was about 4 times higher than for higher angles. Further straining promoted the formation of boundaries with misorientation angles < 30°. In Fig. 5e after 5 rotations there are two types of boundaries at 0 ÷ 30° and 86 ± 3° which correspond to c-axis rotation [32] and twinning [33], respectively. Further straining for 10 turns (Fig. 5f) increases the contribution of boundaries with misorientation angles < 2.5° but without any other changes. In Fig. 6, the density of LAGB (3 ÷ 15°) and high-angle grain boundaries (HAGB) (> 15°) is initially low for the coarse-grained microstructure but torsional straining significantly increases the HAGB density to a saturation level at 1.3 μm/μm^2 after 1 and more turns. The LAGB density steadily increases as the strain increases and at 10 turns the LAGB density is double that of the HAGB density.

The values of the Vickers microhardness measured on the surface of the disk are presented in Fig. 7a where the lower dashed line at Hv = 47 is for the hardness in the initial annealed condition and the upper dashed line at Hv = 61 is the hardness after compression: for simplification, the average hardness value at each point was calculated. It is readily apparent that this material exhibits strain-softening as expected for low melting temperature alloys [15]. The pure compression increased the hardness to 61 Hv and subsequent torsional straining for low numbers of turns gave slight additional strengthening up to 69 Hv at low radii. At higher numbers of turns there was a tendency towards homogenization. Thus, after 5 turns the hardness was higher only in the disk centre and after 10 turns the hardness values were nearly homogeneous across the diameter. The overall hardness evolution during HPT is plotted against the equivalent strain in Fig. 7b where the equivalent strain (ε_{eq}) was estimated from

$$\varepsilon_{eq} = \frac{2\pi N r}{h\sqrt{3}} \tag{1}$$

where: N is the number of turns, r is the radius and h is the thickness of the disk equal to 0.7 mm [34]. Thus, a low equivalent strain of ~1 strengthens the alloy above the pure compression effect but further straining decreases the hardness to Hv ≈ 44 at equivalent strain above ~50 and the final hardness is then lower than in the annealed state.

In Figure 7, the hardness decreases with increasing distance from the sample centre but becomes reasonably constant at 10 turns. In practice, the overall hardness behaviour may be explained based on the dissolution of ε – Zn_4Cu precipitates. It is already established that high pressure torsion promotes precipitate dissolution at high strains [35,36]. Thus, in the present material small ε precipitates were observed in the disk centre causing solid-solution strengthening (Fig. 8a) but they were dissolved in the outer regions of the sample (Fig 8b). This shows that the high imposed strain causes a single-phase microstructure similar to that obtained by annealing and this is consistent with the hardness measurements.

A tensile test was conducted to investigate the effect of HPT processing on the mechanical properties at room temperature at various strain rates. The results for the YS, UTS and E_T for strain rates from 10^{-4} to 10^{-2} s^{-1} are shown in Fig. 9 where the equivalent strain was calculated using r = 2.5 mm and h = 0.7 mm. The YS and UTS behavior can be divided between high strain rate (10^{-2} s^{-1}) when straining up to 5 turns results in an increase in mechanical properties and lower strain rates (10^{-4} – 10^{-3} s^{-1}) when there is a significant decrease in YS and UTS. Further straining up to 10 turns increases both the YS and UTS slightly at all measured strain rates by comparison to samples after 5 turns. Moreover, the strain rate sensitivity (m) tends to increase with increasing strain (see Fig. 10). After low straining, up to 1 turn, m was measured as ~0.08 and ~0.13 at strain rates in the range of $10^{-2} \div 10^{-3}$ s^{-1} and $10^{-3} \div 10^{-4}$ s^{-1}, respectively. Torsional straining was constantly increasing the strain rate sensitivity up to m = 0.16 and m = 0.30 for 10 turns at the above mentioned ranges of strain rates. Fig. 9c presents the effect of torsional strain on E_T at various strain rates. In general, torsional straining leads to an enhancement in elongation with the most significant effect occurring at 10^{-4} s^{-1} when the elongation changed from ~100 % for 1/2 turn to 285 % for 10 turns. Nevertheless, even at a strain rate of 10^{-2} s^{-1}, the measured elongations (65 % to 109 %) are high compared to other Zn-based alloys. The observed trends in all three plots suggest that 10 turns were not sufficient to reach a saturation in the YS, UTS and E_T.

4. Discussion

4.1 Microstructure development during HPT processing

The microstructure results (Fig. 2) show that during HPT processing the initially deformed grains and twins recrystallize, producing highly oriented non-deformed grains. Earlier studies presented similar results for pure Zn [13], Cu [37] and Al [38] and many single-phase alloys [39] where lack of obstacles allows the formation of recrystallized, equiaxed grains. Based on the current results, it is assumed that twinning occurs during the initial straining and the subsequent continuous shearing introduces a high density of dislocations leading to dynamic recrystallization (DRX) of the highly-deformed coarse grains [40]. In the present investigation, the small Cu addition tends to hinder the DRX. Thus, twins were observed even after 1 turn but continuous grain refinement was observed up to 2 turns which suggests grain growth was hindered compared to pure Zn. Further straining for 5 and 10 turns gave substantial grain growth. The main difference between the present observations and grain growth in pure Zn is the formation of a substructure in the Zn-0.5Cu alloy. Severe torsional straining for 5 and 10 turns transformed a collection of small grains separated by HAGBs into coarse grains composed of subgrains of the same size as the initial small grains. The subgrain size was measured as about 2.1 and 1.9 µm after 5 and 10 turns, respectively. In an earlier study, the grain size in the present Zn-0.5Cu alloy after ECAP at RT was measured as ~2.1 µm [9]. The grain size measurements suggest a steady-state grain size in Zn-0.5Cu alloys after the severe plastic deformation of ~2 µm but the present research shows the subgrain formation is an essential part of the microstructure development which causes strong textures.

In texture development during HPT processing for hexagonal close-packed (HCP) materials, basal slip is the preferred slip system and thus basal planes orient parallel to the shearing plane and produce typical basal fiber textures [13,23,41]. The observed texture changes in Zn-0.5Cu alloy are similar to those for pure Zn [13]. An initial random texture after 1/2 turn presents a relatively weak fiber texture with an intensity of approximately 10 (Fig. 3). Further straining enhances the intensity of the observed texture. Rotation for 5 and 10 times causes the appearance of two components: a basal fiber and $\{0001\}\langle 11\bar{2}0\rangle$.

During torsional straining, HCP crystals deform through the $\{0001\}\langle 11\bar{2}0\rangle$ slip system and the basal fiber component is due to a reorientation of the $\{0001\}$ planes in the shearing plane while the $\{0001\}\langle 11\bar{2}0\rangle$ component is also implemented by shearing. However, the nature of this phenomenon is the correlation of a shearing direction in HPT and $\langle 11\bar{2}0\rangle$ a-direction in HCP crystals. In the present Zn-0.5Cu alloy, the basal fiber component develops in two stages (Fig. 4). At first, a weak (0001) fiber texture component develops due to the ongoing grain refinement. Further straining, without any grain size changes, significantly increases the contribution of basal fiber in the total texture. Moreover, the steady increase in the $\{0001\}\langle 11\bar{2}0\rangle$ texture contribution occurred because of the tendency for recovery and recrystallization at RT, which allows a constant deformation in the basal slip system with simultaneous dislocation annihilation and without any changes in the active slip systems.

The opposite effects were observed recently in cast pure Mg after HPT [41] where initially there was a $\{0001\}\langle 10\bar{1}0\rangle$ texture which transformed into basal fiber as the strain increased. In pure Zn [13], a characteristic basal fiber was observed using X-ray diffraction measurements in the disk center. However, the local $\{0001\}\langle 11\bar{2}0\rangle$ texture component may also occur. A highly oriented (0001) texture corresponds to almost parallel c-axis in HCP crystals, which in combination with 6-fold symmetry produces a high contribution of LAGBs. Grain boundary misorientation plots (Fig. 5) present changes of the misorientation angle distribution during torsional straining. A large number of twin boundaries are present in the coarse-grained microstructure and grain reorientation during DRX constantly increases the numbers of grain boundaries with misorientation angles < 30°. After 5 and 10 turns, most of the grain boundaries have a misorientation angle < 30° which can be explained based on texture results and a geometrical analysis presented schematically in Figure 11. Thus, severe plastic deformation produces a sharp (0001) fiber texture so that all basal planes are located in a shearing plane reducing the degrees of freedom to a rotation around the c-axis. In this configuration, the maximum GB misorientation angles match the angle between the $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ planes, which in six-fold symmetry equals 30°. The development of the $\{0001\}\langle 11\bar{2}0\rangle$ texture component (Fig. 6) reduces the degrees of freedom to oscillations around the c-axis which produce mainly LAGBs.

The observed exceptional GB misorientation angle distribution in the Zn-0.5Cu alloy after HPT processing stands in contrast to typical results after SPD processing which produces mostly HAGBs especially during HPT [42,43]. In an earlier study, the Zn-0.5Cu alloy after ECAP processing presented a characteristic misorientation distribution with a high contribution of HAGBs which is superimposed in Figure 5f. The observed difference can be explained based on the monotonic and complex strain paths imposed by HPT and ECAP processing, respectively.

4.2 Influence of torsional straining on the hardness

Figure 7a presents hardness distributions across the sample diameter. The initial low hardness after annealing is typical for low-alloyed recrystallized Zn alloys. Pure compression introduces significant hardening caused by a high number of twins which are known to effectively strengthen HCP materials [44]. Low torsional straining for 1/2 turn causes additional deformation and increases the hardness at the point of 2 mm from the disk centre. Increasing straining in the outer parts of the disk gives DRX and the hardness consequently decreases by comparison with the compressed state. Increasing torsional straining reduces the strengthened area and the hardness remains the same as in the initial state only in the central point after 2 turns. After 5 and 10 turns, high strain causes complete DRX throughout the sample.

Figure 7b presents the hardness distribution versus equivalent strain. The low strain increases the hardness up to 69 Hv while a strain of approximately 20 reduces the hardness to the annealed level. Further straining causes a decrease in hardness to about 44 Hv. Earlier results showed a decrease in hardness from approximately 38 Hv for annealed pure Zn to 33 Hv after 5 turns at 1 GPa [13] while in this study, the initial straining significantly strengthened the alloy and further straining led to a hardness decrease below the annealed hardness value.

The observed strain softening in Zn was attributed earlier to dynamic recovery and recrystallization [12,34]. The main factor affecting this behavior in Zn-alloys is the relatively high homologous temperature (~0.43) [15]. The controlling factor for recrystallization is dislocation mobility. In HCP materials, cross-slip controlled by the stacking fault energy is suppressed and therefore dislocation climb controlled by the homologous temperature is the primary mechanism for softening in Zn alloys [12]. In this study, the alloying addition is the main reason for hindering the dislocation movement and recrystallization process. The present results suggest a transitional behavior between softening with recovery where the peak hardness is at a low strain and weakening without peak hardness where the hardness at high strain is lower than after annealing [34]. Additionally, non-equilibrium precipitate dissolutions increase the hardness in the outer regions of the disk after 10 turns (Fig. 8), thereby suggesting that solid-solution strengthening is more effective than precipitate strengthening by the relatively coarse Cu-rich precipitates. The opposite behaviour was observed in Zn-Mg hybrids, where an increase in strain during HPT processing caused formation of Zn-Mg phases and significant hardness improvement [24]. Post-deformation annealing of HPT-processed Zn-Mg hybrids resulted in a further increase in hardness caused by precipitation of nano-size particles inside supersaturated grains.

4.3 The effect of HPT on the tensile properties

HPT processing significantly reduces the grain size and causes texture sharpening, and both have a significant effect on the mechanical properties of the material. The subgrain refinement observed in this study leads to an increase in the elongation to failure to 285 % and the strain rate sensitivity to 0.30. Even though these measured values are high, the results do not confirm the advent of true superplasticity where the elongation is >400% and $m \approx 0.5$ [45]. An increasing number of turns causes significant differences in YS, UTS, and E_T between the various strain rates and a steady increase in the *m*-parameter value in Fig. 10. Such behavior represents the activation of non-slip deformation in Zn-0.5Cu alloys after HPT and its increasing contribution to the total deformation. This behaviour stands in contrast to the observed grain growth shown in Fig. 2. However, the constant decrease in subgrain size observed after HPT can be responsible for changes in the mechanical properties. In an earlier study, room temperature superplasticity was achieved in the Zn-0.5Cu alloy after ECAP [9,11] where the elongation to failure was measured as ~510 % and the strain rate sensitivity was ~0.31. As mentioned in section 4.1, the average grain size in the earlier study and the average subgrain size in this investigation were very similar and equal to ~2.0 μm. Apparent similarities in the steady-state subgrain size and the mechanical behavior at various strain rates, both after HPT and ECAP, indicate the occurrence of the same deformation mechanisms in both situations. However, in the HPT material, the non-slip deformation mechanism plays a minor role.

Grain refinement is required to activate non-slip deformation modes but it cannot play a role in the observed differences between the Zn-0.5Cu material after HPT and earlier studies using ECAP since the grain sizes are identical. The two main differences appear to be the texture and the grain boundary misorientation distribution. Figure 3 presents the texture evolution during HPT processing where the sharp basal texture is formed after at least 1 turn whereas the texture after the ECAP process is tilted by approximately 45° in both the X and Y directions [9]. It should be noted that the tensile testing direction was oriented differently during these two experiments. After HPT, the tensile direction is parallel to the shearing direction (vertically in Fig. 3) whereas after ECAP the tensile direction is parallel to the pressing direction Z (perpendicularly to XY plane). The occurrence of a sharp basal texture after HPT processing results in a low value of the Schmidt factor (SF) for the Zn basal slip system [46] whereas after ECAP the SF for this slip system reaches mostly maximum values. Such geometrical crystal orientations imply that a higher shear stress is needed to initiate deformation and that is consistent with the measured mechanical properties after HPT and ECAP processing. At the higher strain rate of 10^{-2} s^{-1}, the strong texture changes the YS from 86 to 160 MPa and the UTS from 175 to 240 MPa for ECAP and HPT, respectively. Similar behaviour was also observed at the low strain rate of 10^{-4} s^{-1} when the YS reached 17 and 47 MPa after ECAP and HPT, respectively. At the lower strain rate, the high contribution of the texture-independent non-slip deformation mode is expected, and therefore, the texture effect should be less pronounced.

A very sharp texture induces significant changes in the grain boundary misorientation angle distribution. The distribution plots in the Zn-0.5Cu alloy after HPT and ECAP shown in Figure 5f indicate that the main difference between these two processing methods is the lack of HAGBs after HPT. It was noted earlier that the boundary misorientation angle has a significant effect on grain boundary sliding (GBS) in pure Zn and it was shown that GBS is limited for LAGBs [47]. This is consistent also with very early experiments showing that GBS cannot occur in aluminium for misorientation < 5º [48]. At this lower temperature, LAGBs effectively hinder GBS and an increase in the disorientation angle up to 30° does not significantly enhance the GBS activity. A similar effect was observed for the Zn-0.8Ag alloy after ECAP processing [10] where, regardless of texture, the isotropic mechanical behavior was observed in three orthogonal directions. The current study thus indicates that in low-alloyed quasi-single phase Zn alloys, the grain boundary misorientation distribution is a key factor controlling the GBS activity and therefore it is crucial in controlling the mechanical properties in fine-grained Zn alloys.

5. Summary and conclusions

1. The effect of high pressure torsion on a Zn-0.5Cu alloy was investigated through microstructure, texture and mechanical properties analysis. With increasing numbers of

rotations, a fine-grained microstructure with strong basal fiber texture developed. Grain refinement was observed up to 2 turns and after 5 turns there was significant grain growth occurred, whereas the subgrain size steadily decreased to a steady-state grain size of ~1.9 µm.

2. The strain rate sensitivity increases with grain growth and subgrain refinement indicating the activation of non-slip deformation mechanisms. HPT for 5 and 10 turns produces strong local $\{0001\}\langle 11\bar{2}0\rangle$ texture instead of a typical basal fiber. High torsional straining caused exceptional crystal reorientation during dynamic recrystallization with the c-axis perpendicular to the torsion plane and the slip direction tangential to the shearing direction. Such orientation limits the grain boundary misorientation angle to 30°.

3. Unusual grain boundary misorientation distributions effectively hinder grain boundary sliding after HPT by comparison with ECAP. This causes significantly enhanced mechanical properties of the HPT-processed Zn-0.5Cu material compared to the ECAP-processed alloy although there are similar grain sizes after both processing methods.

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Data Availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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Figure captions :

Figure 1 Schematic illustration of HPT disk showing the location of the tensile testing specimens, hardness measurements, and SEM observation area.

Figure 2 EBSD-IPF maps of Zn-0.5Cu alloy before and after HPT processing. a) $N = 0$ (pure compression), b) $N = 1/2$, c) $N = 1$, d) $N = 2$, e) $N = 5$, f) $N = 10$. Please pay attention to different scale bars.

Figure 3 Basal (0001), prismatic $(10\bar{1}0)$ and prismatic $(11\bar{2}0)$ pole figures of Zn-0.5Cu alloy before and after HPT processing. a) N = 0 (pure compression), b) $N = 1/2$, c) $N = 1$, d) $N = 2$, e) $N = 5$, f) $N = 10$.

Figure 4 (0001) fiber and $\{0001\}\langle 11\bar{2}0\rangle$ components contribution in total texture in Zn-0.5Cu alloy after HPT

Figure 5 Distribution of grain boundary misorientations in Zn-0.5Cu alloy before and after HPT processing. a) $N = 0$ (pure compression), b) $N = 1/2$, c) $N = 1$, d) $N = 2$, e) $N = 5$, f) $N = 10$, Mackenzie random misorientation and misorientation after 4 times ECAP using route *Bc* [9,11].

Figure 6 Low (LAGB) and high (HAGB) angle grain boundary density in Zn-0.5Cu alloy after HPT.

Figure 7 a) radial hardness distribution along the radius for all specimens b) variation of hardness with equivalent strain for the Zn-0.5Cu alloy processed by HPT for a range of turns from 0.5 to 10 turns.

Figure 8 SEM images of Zn-0.5Cu alloy processed by HPT for 10 turns, a) in the disk center, b) close to the disk edge.

Figure 9 Plots of mechanical properties versus equivalent strain at 293 K with various strain rates for HPT samples a) yield stress, b) ultimate tensile stress, c) elongation to failure.

Figure 10 Strain rate sensitivity (m) evolution after HPT processing at RT.

Figure 11 Schematic representation of grain boundary misorientation formation in sharp basal fiber texture