**Abnormal grain growth in a Zn-0.8Ag alloy after processing by high-pressure torsion**

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**Abstract**

Abnormal grain growth (AGG) in a Zn-0.8Ag (wt%) alloy, produced through the application of high-pressure torsion (HPT), was systematically investigated using scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), high-resolution transmission electron microscopy (HR-TEM) and microhardness testing. The HPT-deformed alloy exhibits AGG at room temperature without any additional heat treatment. Analysis by EBSD revealed oriented grain nucleation in a $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ direction from the initial $(0001)$ fibre texture which agrees with the maximum energy release model. New grains were oriented according to the minimal Young’s modulus direction (c-axis), parallel to the shearing direction. The strain-induced dissolution of nanocrystalline Zn3Ag precipitates was identified as the main driving force for AGG in this alloy. The strains necessary for the initiation and termination of AGG were determined as ~4.0 and ~5.0, respectively. The increase in solid-solution strengthening caused an increase in hardness from ~47 HK in the fine-grained centre to ~84 HK in the coarse-grained region. A Hall-Petch investigation revealed grain refinement softening below a grain size of 23 µm. These results provide the first comprehensive description of AGG in metallic materials processed by a severe plastic deformation method at room temperature.

**Keywords**

Abnormal grain growth; High-pressure torsion; Severe plastic deformation; Texture; Zinc alloys.

1. **Introduction**

 Abnormal grain growth (AGG) takes place in many polycrystalline materials after primary recrystallisation is complete. Unlike in primary recrystallisation where grain growth is uniform, the occurrence of AGG produces an inhomogeneous microstructure. The presence of second-phase nanometric particles is a crucial factor enabling AGG. Thus, small particles interact with grain boundaries (GB) via the Zener mechanism which impedes normal grain growth. In almost all cases of AGG, the dissolution of second-phase precipitates leads to a destabilisation of Zener-pinned GB [1–3]. In highly textured, fine-grained polycrystalline materials, the AGG of coarse grains at the expense of small equiaxed, recrystallised grains was observed for thin films [4–6] and also for bulk materials [7–11]. The main driving forces in a material with a strong recrystallisation texture tend to be both high GB density and high GB misorientations [4,12–14]. A dynamic version of AGG was observed in pure Mo during slow plastic straining at an elevated temperature where the dynamic AGG was initiated at a lower temperature compared to static annealing and produced centimetre lengths of single crystals [15,16].

High-pressure torsion (HPT) is an severe plastic deformation (SPD) method that provides the capability of imposing a large amount of deformation at significant strain rates [17]. In HPT, deformation is applied through torsional shearing of a thin disk held between two anvils. Thus, using a typical sample size of *h* = 0.7 mm and *r* = 10 mm, it is possible to apply a shearing strain of up to 80 during 1 turn which typically takes between 30 and 120 seconds. Another essential factor in HPT is a high hydrostatic pressure of up to 10 GPa [17] which prevents the sample from cracking or fragmenting and significantly enhances the processing of brittle materials such as Ti alloys [18,19], Mg alloys [20], Zn alloys [21], martensitic steels [22] or high-entropy alloys [23].

Processing by HPT is used to produce exceptional grain refinement in metallic materials. For pure metals, the steady-state grain size is determined by the mobility of dislocations which determines the rate of dislocation annealing and it correlates with physical parameters such as the atomic bond energy and melting temperature [24–28]. For pure Zn, the minimal grain size obtained in a room temperature SPD processing was determined to be ~5.1 μm [24]. However, grain size observations in HPT-processed Zn and Zn-alloys show behaviours that differ from the steady-state grain size in other metals and alloys. In pure Zn, HPT processing for 3 and more turns under a pressure of 1 GPa caused significant grain growth such that the grain size initially decreased to ~20 μm but after 3 turns started to increase up to ~97 μm [29]. Grain growth in pure Zn (impurities content < 615 ppm) during HPT processing can be attributed to the relatively insignificant factors such as a heat generated by friction between anvils (< 10 °C) [30,31] and dissolution of impurities which lead to dynamic recovery processes and grain growth. Nevertheless, even a small addition of alloying elements significantly reduces the grain size due to dislocation-solute atom interactions which affect the dislocation mobility [32] and grain boundary pinning force on solutes atoms [33]. During HPT processing of a Zn-0.5Cu alloy, the strain increase caused grain refinement to ~8 μm but after 5 turns the grain size increased [34] so that ultimately there were grains with an average size of ~30 μm composed of equiaxed subgrains with an average size ~2 μm. The grain growth observed in the Zn-0.5Cu alloy is related to a sharp texture formation and a grain reorientation which transforms high-angle grain boundaries (HAGB) into low-angle grain boundaries (LAGB) [34].

 The present research was conducted to analyse the effect of HPT processing on the microstructure of a Zn-0.8Ag (wt. %) alloy and, for the first time, to provide a comprehensive description of the SPD-induced abnormal grain growth (SPD-AGG) in this alloy after room temperature HPT processing. Additionally, the crystallographic relation between the dynamically recrystallised grains and the new abnormal grains was identified. Hardness distribution analysis after AGG and Hall-Petch relationship present an unusual grain refinement softening effect below the critical grain size.

1. **Experimental material and procedures**

The alloy used in the present investigation was a high purity Zn-0.8Ag (wt.%) alloy. The alloy was melted in a graphite crucible, homogenised for 30 minutes at 650 °C and subsequently cast into a cylindrical steel mould. The cast ingot was annealed for 4 hours at 400 °C and then water-cooled. The chemical composition of the material was analysed using a Rigaku ZSX Primus IV wavelength dispersive X-ray fluorescence spectrometer (WD-XRF) and the results showed 0.813 ± 0.008 wt. % of Ag and < 0.005 wt. % of Cd, Cu, Fe, and Pb. The HPT disks were prepared by machining a rod of 9.8 mm diameter, cutting 1.3 mm thick disks using wire electro-discharge machining, and then grinding and polishing to a final thickness of ~0.85 mm to remove the deformed layer. The HPT processing was performed under quasi-constrained conditions with steel anvils under an applied pressure of 6.0 GPa at 1 rotation per minute [35]. A small material outflow was observed around the periphery of the disk during the HPT processing [31]. A set of disks was deformed through totals of N = 0, 1/2, 1, 2, 5 and 10 turns. After HPT processing, each sample was held for 30 seconds under the applied pressure.

For microstructural analysis, samples were cold-mounted in an epoxy resin, ground, polished using water-free diamond suspensions and finally polished using low-angle an Ar+ Hitachi IM4000Plus Ion Milling System. Microstructure and microtexture analyses were carried out using a Nikon Eclipse LV150N polarised light microscope and an FEI Versa 3D scanning electron microscope (SEM) equipped with an EDAX OIM TSL electron backscattered diffraction (EBSD) detector. For microstructure evolution, the EBSD map size and step size were set to 600 μm × 400 μm and 400 nm, respectively and all maps were collected in the shear plane at ~3 mm from the centre of the disk using constant SEM and EBSD camera parameters. Additionally, a series of EBSD maps were collected in two perpendicular directions, first in the shear plane and second in a radial view to analyse microstructure changes along the diameter after 10 turns. Maps ~1.5 mm and 3 mm from the disk centre were collected with step size equal to 400 nm, while the step size for maps in the disk centre was set to 100 nm. Based on the EBSD data, (0001), $\left(10\overbar{1}0\right)$ and $\left(11\overbar{2}0\right)$ pole figures (PF), average grain sizes were calculated using Matlab M-TEX software [36]. All PF were projected in the shear plane – shear direction format [37]. The Kikuchi pattern identification fraction exceeded 96.6% in all maps. Grains and subgrains were defined by 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. For transmission electron microscopy (TEM), foils were cut from the disk centre and outer region using a focused ion beam (FIB) installed in a dual-beam SEM FEI Quanta 3D 200i. High-resolution (HR-STEM) examination using bright field (BF-STEM) and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), electron energy loss spectroscopy (EELS) mapping and selective area electron diffractions (SAED) were performed using HR-TEM FEI Titan Cubed 2 60-300. For phase identification, SAED patterns were analysed using JEMS software.

Knoop microhardness (HK) measurements of the HPT-processed sample after 10 turns were performed across a diameter using a Wilson-Hardness Tukon 2500 universal hardness tester under a constant load of 100 gf held for 10 s. Each reported value represents the average of 6 indentations all made at an equal distance from the centre of the disk. Additionally, to obtain the relationship between grain size and hardness in a Zn-0.8Ag alloy a set of Knoop hardness measurements were performed for cold-rolled samples subsequently annealed at various temperatures. For the Hall-Petch relationship, the Knoop hardness values were recalculated to the Vickers hardness scale (HV) using equations presented in [38] and then to the yield stress using HV divided by Tabor factor of 3 [39]. The yield stress results were correlated with the average grain size measured by EBSD analysis performed in the same way as described above.

1. **Experimental results**

Microstructures of the investigated samples are presented in the form of inverse pole figure IPF maps in Fig. 1. After pure compression (N = 0), the alloy exhibits a coarse-grained microstructure with a significant number of twins introduced within the grains (Fig. 1a). Torsional straining with N = ½ and N = 1 turn causes significant grain refinement and the development of an inhomogeneous microstructure with a bimodal grain size distribution (Fig. 1b and c). Increasing N to 2 turns leads to the development of an unexpected coarse-grained recrystallised microstructure (Fig. 1d). Figs 1e and 1f present the effect of further torsional straining through 5 and 10 turns, respectively, revealing a further increase in grain size Most of the observed grains exhibit an atypical orientation compared to the expected basal fibre texture [29] with $the \left〈11\overbar{2}0\right〉$ a-direction perpendicular to the sample surface. In all microstructures in Figs 1b-f, either dynamic or static recrystallisation has taken place during or after plastic deformation.

The unexpected microstructure and texture development observed after N ≥ 2 turns requires a detailed analysis. The EBSD-IPF maps of the sample after N = 10 (Fig. 2) reveal that the centre of the disk sample (Fig. 2a,d) consists of small equiaxed subgrains with an average size of ~3 μm. Almost all grains exhibit the <0001> *c*-axis perpendicular to the shearing plane. At ~1.5 mm from the disk centre (Fig. 2b,e), there is a bimodal microstructure with large recrystallised grains between regions composed of small grains. Furthermore, the large grains tend to grow at the expense of the small grains. At 3 mm from the disk centre (i.e. 2 mm from the edge), there is complete recrystallisation with an average grain size value of ~355 μm (Fig. 2c). The radius where the bimodal and coarse-grained microstructures occurred was measured in all HPT samples, and the values of the total strain *ε* corresponding to these radii were calculated from equation (1)[40]. In all samples, the total strain *ε* required for initiation of the SPD-AGG was ~4.0, and for full recrystallisation, it was ~5.0:

$ε=\frac{2}{\sqrt{3}}ln\left[\left(1+\frac{γ^{2}}{4}\right)^{{1}/{2}}+\frac{γ}{2}\right]$ (1)

$γ=\frac{2πNr}{h}$ (2)

where: γ is the shear strain, *h* is the disk thickness, *r* is the radius, and *N* is the number of turns.

Observations by SEM in the fine-grained region revealed precipitates with a size of a few nanometers grouped in clusters that were randomly located at the grain boundaries (Fig. 2d). These precipitates were not observed in the outer region of the disk (Fig. 2e). Additional precipitates analyses were undertaken utilising HR-STEM imaging, SAED and EELS. Fig. 3a-c shows nano-sized precipitates with sizes ranging from ~15 to ~50 nm located at the GB. SAED patterns presented in Fig. 3e,f indicate the Zn matrix and the Zn3Ag phase, respectively. Additionally, the HR-STEM image shown in Fig. 3d reveals a coherent GB between the Zn matrix and a Zn3Ag particle. Fig. 4 presents HR-STEM analysis performed in the coarse-grained outer zone. Fig. 4a shows a GB pinned on a relatively large and undissolved Zn3Ag particle apart from which no other precipitates were observed. More detailed Ag and Zn distributions are presented on EELS maps (Fig. 4b and c). The Ag content is visibly higher in the Zn3Ag particle, while in the matrix and at the GB the Ag distribution is uniform. No GB segregation or clustering were detected.

Texture changes along the diameter were investigated using pole figures calculated from the EBSD data corresponding to the IPF maps in Fig. 2a-c. In the disk centre (Fig. 5a), the relatively low applied strain gave a sharp basal fibre texture characteristic of hexagonal close-packed materials after HPT [29]. At ~1.5 mm from the disk centre (Fig. 5b), an increase in torsional strain caused the appearance of the $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ texture component which hitherto has not been reported for HCP materials processed by HPT. A further increase in strain led to the development of a sharp $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ texture in the observed area (Fig. 5c).

Fig. 6 shows the Knoop microhardness changes across the diameter of the disk after 10 turns together with the corresponding microstructure image. The lower dashed line at 60 HK represents the average microhardness of the undeformed sample in the pre-HPT condition while the lower and upper thin dashed lines denote the initial microhardness standard deviation. For samples deformed up to 10 turns as in Fig. 6, the microhardness increases with the distance from the centre to the edge and therefore with increasing strain. For low radii, the microhardness of ~47 HK is lower than for the initial material which indicates the occurrence of strain-softening as expected for low melting temperature alloys [26,34]. At higher strains, a simultaneous increase in grain size and microhardness is observed. Specifically, the microhardness increased from ~54 to ~73 HK between 0.8 and 1.8 mm from the disc centre. A further increase in strain led to a hardness saturation at ~84 HK. The last measured point at *r* = 4.8 mm tends to be too close to the disk edge and therefore this measured value has a significant error. It is readily apparent that the observed correlation between grain size and microhardness is the opposite of the trend anticipated by the Hall-Petch relationship [39,41–43].

Fig. 7 presents the relationship between grain size and the yield stress for the investigated alloy after cold rolling and subsequent annealing. Such material processing route leads to a fully recrystallized, strain-free microstructure with grain size from 3.6 μm to 300 μm. Initially, grain refinement provided strengthening with hardening coefficient *k* equal to 219 MPa∙μm-0.5, however, for the grain size below 23 μm a significant softening occurred.

1. **Discussion**

It is reasonable to anticipate that the observed SPD-induced abnormal grain growth is caused by an interaction between the two separate phenomena of oriented grain nucleation and preferred grain growth. These two processes are examined in the following sections.

* 1. *Oriented grain nucleation*

As presented in Fig. 2b, the microstructure shows an almost perpendicular orientation of new grains compared to the primary grains. In practice, the measured orientation of these new grains after HPT processing is unusual. Thus, it is usual for HPT to produce a stable basal fibre texture (the B fibre according to [37]) with some twins having a $\left〈10\overbar{1}0\right〉$ direction perpendicular to the shearing plane [29,34]. By contrast, the observed $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ texture cannot form directly from the deformation system because it requires shearing only in the *c-*direction which is not allowed in HCP crystals. The appearance of this texture component is explained by post-deformation oriented grain nucleation which is consistent with the strain–energy release maximisation model [44,45].

In this model, a correlation between the highest dislocation density and the strain direction in deformed grains with a minimal Young’s modulus direction in dislocation-free new grains is suggested. This means that the minimal Young’s modulus direction in the new grain nuclei lies parallel to the maximum stress direction in the primary grains and a common rotation axis exists between these two orientations. High residual stresses are rarely observed in Zn and its alloys because of a tendency for rapid dislocation recovery at room temperature [26,46,47]. However, in this alloy, the interaction between Ag solute and the dislocations may effectively hinder dislocation mobility and thereby reduce the dislocation annihilation compared to the pure Zn [46]. Fig. 8 shows schematically the Zn crystal orientation with respect to the HPT geometry. For grains in the fine-grained microstructure, the *c*-directions are oriented perpendicular to the shearing plane and the *a­-*directions are oriented parallel to the shearing direction, whereas in the coarse-grained regions the *c­*-direction is oriented parallel to the shearing direction and the *a­*-direction is oriented perpendicular to the shearing plane. Following earlier analyses [44,45], the direction of the maximum stress is parallel to the Burgers vector of the activated slip system. Based on the measured local texture orientation in the fine-grained region and texture analysis presented in [34], it is evident that the basal $\left\{0001\right\}\left〈11\overbar{2}0\right〉$ slip system is the most dominant slip system during HPT processing and other slip systems play a negligible role. This means that in the Zn alloys the $\left〈11\overbar{2}0\right〉$ direction appears to be the maximum stress direction [48] while the direction of the minimal Young’s modulus (approximately 36 GPa) is the $\left〈0001\right〉$ direction [49]. Therefore the maximum stress $\left〈11\overbar{2}0\right〉$ *a-*­­direction (Fig. 3d) is parallel to the shearing direction in the deformed grains. During nucleation of new grains, the minimum Young’s modulus $\left〈0001\right〉$ *c­-*direction is parallel to the *a­*-direction in the primary grains and therefore the $\left〈0001\right〉$ direction of new grains is parallel to the shearing direction. Additionally, the $\left〈10\overbar{1}0\right〉$ direction was measured as a rotation axis between the pre- and post-AGG orientations.

* 1. *Preferred grain growth*

Coherent Zn3Ag precipitates are present both prior to HPT processing and in locations that have experienced low strains (Fig. 3d) and these precipitates will generate lattice elastic strain energy and induce GB Zener-pinning so that they play a significant role in controlling any grain growth at elevated homologous temperatures [2,8,11,13]. It is well-established that coherent precipitates produce a stronger Zener-pinning effect than incoherent particles and hence the coherent Zn3Ag precipitates in the present alloy are an effective obstacle for GB movement [50]. In this investigation, an increase in torsional straining led to nano-sized precipitate dissolution in regions subjected to high strain (Fig. 2i and Fig. 4). Therefore, based on the microstructural evolution, it appears that the applied total strain of ~4.0 causes second-phase dissolution and removes obstacles for GB movement so that consequently there is a grain growth. Additionally, post-deformation HR-TEM analysis (Fig. 4) shows the uniform Ag distribution both in the matrix and at the GB indicating no noticeable solute drag effect [3].

The mechanism for dissolution of the precipitates at high strain is related to the repeated cutting of these coherent fine precipitates by moving dislocations which generate additional precipitate-matrix interfaces and thus increase the effective Gibbs free energy of the precipitate relative to the Zn-rich phase. At very high strains, this causes the precipitate to become thermodynamically unstable and to dissolve. This mechanism will be more prevalent where the initial size of the precipitates is small because these smaller precipitates will contain a relatively high contribution of interfacial energy to their total free energy. Consistent with this mechanism, it has been established that SPD provides the capability for producing a non-equilibrium supersaturated solid-solution [51–54].

Besides particle dissolution, the strong texture and high GB density in the fine-grained region act as AGG driving forces. Fig. 2a presents the fine-grained microstructure with a high density of GB. High energy stored in GB is a driving force for both normal and abnormal grain growth [1]. Additionally, sharp texture in which almost all GB have a misorientation angle below 30° tends to act as an additional factor promoting AGG. It is well known that in such microstructures AGG occurs relatively easily [4,12,13]. The new nuclei with the $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ orientation are separated by high angle GB measured approximately at 80° ÷ 90° and this presents much higher mobility than LAGB [55,56]. Based on this, these new grains grow towards a high-energy, fine-grained region much faster than others producing initially bimodal microstructure, and finally, a fully recrystallised coarse-grained material [55]. Additionally, both the temperature increase during HPT processing as well as the high hydrostatic pressure can act as factors stimulating AGG. Based on the relationships developed using finite element modelling [30,31], the temperature rise of the present alloy during HPT, including the friction between the anvils, is estimated as ~10 K. This temperature rise is too small to have any significant impact on the grain growth and stability of the precipitates. However, earlier studies examined the effect of the high hydrostatic pressure on the deformation behaviour of polycrystalline materials [57–60], and it was shown that, under high hydrostatic pressure, GB migration is enhanced and the activation energy for the GB migration process attains a similar level to self-diffusion and GB diffusion [59]. It is worth emphasizing again that the observed preferred grain nucleation and subsequent growth occur under an applied pressure when the rotation has stopped.

* 1. *The effect of abnormal grain growth on hardening*

Fig. 6 shows there is an increase in microhardness with increasing grain size as a consequence of the increasingly severe strain. Apparently, the Zn3Ag dissolution produces both a matrix supersaturation which causes solid solution strengthening and AGG. In the fine-grained disk centre, a low hardness of ~47 HK was measured, which is consistent with the dynamic recrystallisation characteristics for low melting temperature alloys [26,61]. Between 0.8 and 2.8 mm from the disk centre, a significant increase in hardness was observed. Moreover, in the same region, there is a transition from a fine- to a coarse-grained microstructure. A further increase in strain leads to a hardness saturation at ~84 HK. The observed tendency could be divided into two separated phenomena. First, the increase in hardness associated with solid-solution strengthening, grain boundary strengthening and the hardness anisotropy. The precipitation hardening may be ignored, both in the disk centre and in the AGG region, because second-phase precipitates located at the GB in the disk centre do not act as dislocation obstacles within grains whilst the AGG region exhibits a lack of precipitates. Second, the grain refinement softening related to the GBS [62] and the absorption of dislocation pile-ups by grain boundaries during dynamic recovery (DRV) [63]. It follows therefore that a comprehensive analysis of hardening should include the above-mentioned effects.

The solid solution strengthening effect in Zn-Ag alloys has never been quantitatively investigated, but an evaluation of this effect may be based on the hardness measurements reported earlier [46] and the hardness measured for Zn-Ag alloys with Ag additions from 0 to 3 wt. % (not presented in this work). In the annealed and supersaturated coarse-grained alloys, the solid solution strengthening effect may be presented as [64,65]:

 $∆σ\_{sol}= k\_{sol}^{{1}/{n}}∙c^{n}$ (3)

where *ksol* is the solid solution strengthening coefficient calculated of 6.6 MPa∙(at.%)-1/2, *c* is element content in at. % and *n* is a constant equal to 0.5 [66]. The measured solid solution strengthening coefficient of Ag is lower than reported earlier where the coefficients for Al and Mg were equal to 9.1 and 11.8 MPa∙(at.%)-1/2, respectively [67]. to the maximum solubility of Ag in Zn at room temperature estimated from the binary phase diagram [68], the Ag content in the matrix equals 0.3 wt.% (0.18 at.%) at the disk centre and 0.8 wt.% (0.49 at. %) in the supersaturated AGG region which generate Δ*σ­sol­* equal to ~18.5 MPa and ~30.5 MPa, respectively.

The high Zn crystal anisotropy significantly affects both the elastic and the plastic properties. Thus, Young’s modulus varies from ~36 GPa in the *c­­-*axis to ~126 GPa in the *a­*-axis while the microhardness in thin deformed Zn coatings varies from ~0.60 GPa to ~1.54 GPa in those directions [49,69,70]. However, the effect of crystal orientation on the hardness in pure, recrystallised Zn has been investigated in both single crystal and polycrystalline samples [71] and it was reported that, for measurements deeper than ~1 μm, grains with the *c-*direction parallel to the load and perpendicularly oriented grains exhibit a hardness of ~0.58 GPa and ~0.70 GPa, respectively. An accurate relationship between the crystal orientation and hardness has never beenevaluated in Zn alloys. Therefore, the contribution from the orientation to the total hardness of not deformed grains was estimated as giving a value of *ΔHVOR* ≈ 120 MPa (using Tabor factor of 3, *ΔσOR* ≈ 40 MPa) which is higher in the AGG region than in the disk centre [69,71]. Nevertheless, twinning induced strengthening can significantly enhance the hardness asymmetry up to *ΔσOR* ≈ 310 MPa [70]. Thus, this effect could be taken into a further consideration.

In order to identify only the strengthening effect of grain size on the microhardness, the Hall-Petch relationship can be utilised [39,72,73]:

$∆σ\_{GB}= \frac{k\_{HP}}{d^{1/2}}$ (4)

where the Hall-Petch coefficient *kHP* = 219 MPa∙μm-0.5 (Fig. 7) and *d* is average grain size. The average grain sizes in the disk centre and the AGG region were ~3 μm and ~355 μm, respectively. Therefore, this should provide a grain boundary strengthening of ~126 MPa and 11 MPa in the disk centre and AGG region, respectively. However, omitting the Hall-Petch relationship of pure Zn, in the literature there are only a few analyses of grain boundary strengthening in Zn alloys. Thus, the relationship between hardness and the grain size of the Zn–0.8Ag alloy was analysed in Fig. 7. It shows the expected grain refinement strengthening down to the grain size of 23 μm with *kHP ­*= 219 MPa∙μm-0.5. The strengthening coefficient value is very close to the reported 220 MPa∙μm-0.5 for pure Zn [39]. Further grain refinement below 23 μm induced a significant softening resulting in hardness below the values obtained for the annealed, coarse-grained material. The measured value of critical grain size for a transition from strengthening to the softening regime is a few orders higher than for pure Zn [74]. Usually, such effect is observed in Zn at the nano-scale. However, similar grain refinement softening was reported for the Zn-22Al alloy [62,63,75] and was attributed to the occurrence of grain boundary sliding (GBS) [62,75]. Additionally, the low recrystallization temperature of Zn-rich solid solution and the high GB density lead to the activation of boundary-sensitive DRV often observed during GBS [63].

Pure Zn exhibits a strong tendency for static recovery and recrystallisation at room temperature. Thus the dislocation density strengthening plays a negligible role in the total hardening of Zn [46,47]. An earlier study [46] showed a significant effect of a minor Ag addition on the half-recrystallisation time and an increase in the Ag addition from 0.227 wt. % to 0.896 wt. % significantly hindered recrystallisation. For a lower Ag content, the half-recrystallisation time is ~4 hours at room temperature while for higher Ag contents an estimated temperature of ~57 °C was required to obtain the same effect at the same time. Using this approach for the current study, the matrix supersaturation results in a slightly higher dislocation density *ΔσD* in the AGG region compared to the disk centre (Fig. 2). Nevertheless, the dislocation density is reduced in both zones by continuous dynamic recrystallization in the disk centre and AGG in the outer region. Regardless of the initial dislocation density, the above-mentioned load-induced GBS and DRV during the hardness test reduce the dislocation density in the fine-grained disk centre ultimately enhancing the measured hardness disproportionally between the fine- and coarse-grained regions. In practice, calculations of the *ΔσD* hardness component is a challenging task since a *ΔσD*evaluation requires point-XRD measurements of the microstrain.

Also, following the same approach, the strengthening effect due to the interaction of segregated alloying elements and dislocations *ΔσSeg* cannot be measured in the disk centre using a hardness test. Based on the EELS analysis presented in Fig. 4, the Ag segregation is unnoticeable in the AGG region, thus the *ΔσSeg* plays a negligible role in the total hardness.

Taking into account all of the potential strengthening mechanisms, the measured hardness does not fit to the calculated values using equation 5, therefore the missing hardness *ΔσMiss* contributor was implemented.

$HV=C\left(σ\_{0}+∆σ\_{GB}+∆σ\_{Sol}+ ∆σ\_{OR}+∆σ\_{Miss}\right)$ (5)

where ­*C* is a Tabor factor of 3. Finally, equation 6 was used to compare the Vickers to Knoop hardness [38]:

$HK=1.1053HV-0.0134HV^{2}$ (6)

Consequently, assuming only the strengthening effects, the missing hardness *ΔσMiss* in a AGG region was measured as ~27 HK. The origin of missing strength could result from the twinning strengthening of coarse grains in an AGG region. In Fig. 1 b,c and Fig. 1 f,g some small twins are visible in the AGG grains. Due to twinning strengthening, the orientation hardness anisotropy can considerably increase. In this situation, the low twin fraction moderately enhances hardness in the range of 27 HK instead of reported earlier of 97 HK (above-mentioned 310 MPa) [70]. Following the same calculation, in the disk centre, the evaluated hardness is 24 HK higher than the measured value. The missing hardness is attributed to the grain refinement softening caused by GBS and DRV phenomenon occurring in the fine-grained region. Summarizing, the unusual hardness distribution results mainly from the hard grain orientation in the AGG zone and grain refinement softening in the disk centre.

It is important to note that SPD-AGG was observed uniquely in the Zn-Ag alloy and it appears that it was not reported in the SPD processing of Zn or any of its alloys. For example, in pure Zn after HPT [29] and in highly textured electrodeposited Zn [76] AGG in a specific orientation was not observed. This absence of AGG is probably due to the inability to store sufficient energy in order to activate nucleation where this is a direct consequence of the rapid annealing of dislocations [26,46,47]. In the Zn-0.5Cu alloy [34], a highly textured microstructure remains stable even after 10 turns and it was reported that 0.5 wt. % of Cu addition refines the microstructure of Zn alloys more effectively than 0.8 wt. % of Ag [77,78]. Additionally, the initial size of the second phase precipitates in the Zn-0.8Ag alloy is significantly smaller than in the Zn-0.5Cu alloy and, whilst the applied strains in HPT processing were sufficient to dissolve Ag-rich particles, they were not sufficient to give a dissolution of the coarser Cu-rich precipitates. Accordingly, this means that the microstructure of the Zn-0.8Ag alloy is less stable and the required strain for the activation of AGG is significantly lower than in the Zn-0.5Cu alloy.

1. **Summary and conclusions**

An investigation of the Zn-0.8Ag alloy after HPT processing shows, for the first time, an SPD-induced abnormal grain growth in the HPT-processed material without the introduction of any additional treatment. A very high total strain over ~4.0 leads to the dissolution of the GB-controlling Zn3Ag precipitates. Thus, under high-pressure conditions and with a lack of obstacles to grain boundary migration, abnormal grain growth takes place.

The main findings of this study are as follows:

* In the Zn-0.8Ag alloy after HPT processing, the total strains *ε* needed to activate and terminate SPD-AGG were determined as ~4.0 and ~5.0, respectively.
* The observed grain nucleation in the $\left\{11\overbar{2}0\right\}\left〈0001\right〉$ orientation agrees with the maximum energy release model. New grains are oriented according to the minimal Young’s modulus direction (c-axis), parallel to the shearing direction.
* A high GB density, sharp texture and Zn3Ag dissolution significantly enhance the GB mobility and become the driving force for the growth of new nuclei at the expense of the initial grains.
* The investigated alloy presents grain refinement strengthening to critical grain size 23 µm with a strengthening coefficient of *kHP* ­= 219 MPa∙μm-0.5. Below that grain size a grain refinement softening was observed.

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