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Achieving enhanced mechanical properties in Mg-Gd-Y-Zn-Mn alloy by altering dynamic recrystallization behavior via pre-ageing treatment

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Abstract: The effects of pre-ageing treatment on the microstructure and mechanical properties of the Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn (wt.%) alloy were investigated. Microstructural analysis indicated that the ageing treatment before extrusion led to the formation of dense prismatic β' and basal γ' precipitates in heat treated alloys. The presence of these precipitates and their solution obstructed the dynamic recrystallization process during hot extrusion. The lamellar long-period stacking ordered (LPSO) phases restrained the recrystallization through forming the kink band and releasing the stress concentration, and the fine β-Mg5(Gd, Y) particles suppressed the recrystallization by the particle pinning effect. The block-shaped LPSO phases and coarse β-Mg5(Gd, Y) particles promoted the recrystallization following the particle stimulated nucleation (PSN) mechanism. The combined effects led to the formation of the bimodal microstructure, which shows fine recrystallized grains with random grain orientation and deformed grains with strong fiber texture. The bimodal microstructure

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with lower recrystallization fraction provides the alloy higher strength and lower
ductility. With solid-solution and pre-ageing treatments, the as-extruded alloy shows
the best strength-ductility balance with an ultimate tensile strength (UTS) of 455 MPa,
tensile yield strength (TYS) of 382 MPa and elongation to failure (EL) of 11.0%. The
outstanding mechanical properties are mainly attributed to the bimodal microstructure,
strong fiber texture, β-Mg5(Gd, Y) particles, lamellar and block-shaped LPSO phases.

**Keywords:** Mg-Gd-Y-Zn-Mn alloy; Heat treatments; Morphology; Long-period
stacking ordered phase; Mechanical properties.

**1. Introduction**

Magnesium alloys containing rare-earth (RE) and transition-metal (TM) elements
have aroused wide attention as lightweight structural materials because of their good
combined properties of mechanical properties, creep resistance, corrosion resistance
and damping capacities [1-5]. To acquire high strength and favorable ductility, the
LPSO-strengthening magnesium alloys have been designed by adding the Gd/Y and Zn
elements [6-8]. As the LPSO phase exhibits a stacking ordered and chemically ordered
structure, its formation is related to the co-segregation of RE and Zn atoms, which
endow the LPSO phase a higher hardness and Young’s modulus [9]. The dispersed
distribution of LPSO phases can improve the critical resolved shear stress (CRSS) of
the basal plane and facilitate the activation of non-basal slip. As a hard phase, the
aligned LPSO phase can also strengthen the alloy by short-fiber strengthening and
prevent failure through retarding the propagation of microcracks [1]. Recently, a LPSO-
strengthening Mg-8.2Gd-3.8Y-1.0Zn-0.4Zr (wt.%) alloy with bimodal microstructure
has been fabricated by hot extrusion followed by forced-air cooling treatment [10]. The optimized strength-ductility balance of this alloy is not only related to the LPSO phase but also affected by its bimodal grain structure. The typical bimodal microstructure consists of untextured recrystallized grains as a soft region and hot-worked grains as a hard region. In fact, the analogously bimodal microstructures also have been observed in Al-Mg [11], Cu-Ag [12] and low carbon steels [13], and exert favorable strengthening as well as toughening. Accordingly, through the thermo-mechanical processing to regulate the dynamic recrystallization behavior and acquire an appropriate bimodal microstructure should be an effective way to improve mechanical properties of alloys.

It has been confirmed that the coarse particles and their clusters can promote the dynamic recrystallization via PSN, while the fine particles dispersion can retard it by the particle pinning effect [14-17]. Yu et al. [17] developed a high strength Mg-11Gd-4.5Y-1Nd-1.5Zn-0.5Zr alloy by pre-ageing and hot extrusion process, and reported that the dense coarse Mg₃RE particles formed by pre-ageing treatment promote the transformation of low-angle sub-grain boundaries to high-angle grain boundaries. In ZM61 magnesium, ageing before extrusion results in the formation of dispersive MgZn₂ particles, which promotes the dynamic recrystallization during subsequent extrusion and generates a completely recrystallized microstructure [16]. Therefore, the particles are usually introduced into alloys prior to hot plastic deformation, which is used to control the grain size and texture of wrought alloys via altering the dynamic recrystallization behavior.
However, research rarely focuses on acquiring a bimodal microstructure based on the particle effects in magnesium alloys. In the present study, dense prismatic $\beta'$ and basal $\gamma'$ precipitates were introduced in a Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn (wt.%) alloy through ageing treatment before extrusion to produce a bimodal microstructure. Room-temperature mechanical properties of the as-extruded alloy were improved remarkably. The recrystallization behavior and strengthening mechanism were systematically discussed.

2. Experimental Procedures

2.1. Sample preparation

Experimental Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn (wt.%) alloy with 87 mm in diameter prepared by semi-continuous casting was used in this study. The actual composition of the casting billet was measured by inductively coupled plasma (PE ICP-OES). Different fabrication procedures are summarized in Table 1. The billets with 150 mm in length were homogenized at 500 °C for 8 h and 525 °C for 4 h followed by annealing to 400 °C within 10 h or quenching in water (90 ± 5 °C), respectively. Then the as-annealed and as-quenched billets were pre-aged at 225 °C for 110 h and machined into round bars with 82 mm in diameter and 140 mm in height. Subsequently, the round bars were preheated at 420 °C for 30 min prior to the hot extrusion which was carried out using an XJ-500T horizontal extruder at 420 °C with extrusion ration of ~11:1 at a ram speed of 0.3 mm/s.

2.2 Materials characterization

The thermal behavior of the as-cast alloy was determined by the Differential
Scanning Calorimetry (DSC) from Netzsch (449F3). The DSC samples were heated to 650 °C followed by cooling to 50 °C at a rate of 10 °C/min under an Ar atmosphere. DSC results are shown in Supplementary Fig. 1.

The microstructures of the samples were characterized by an optical microscope (OM, OLYMPUS OLS4000), a scanning electron microscope (SEM, TESCAN VEGA3 LMH SEM) equipped with a backscatter electron (SEM-BSE) detector, another scanning electron microscope (SEM, JEOL JSM-7800F FEG SEM) equipped with a HKL-Electron backscatter diffraction (EBSD) system and a transmission electron microscope (TEM, FEI Tecnai G2 F20). Samples for OM observation were ground with SiC papers and mechanically polished followed by etching with an acetic-piceral solution of 3 g picric acid, 10 ml acetic acid and 14 ml ethanol. Samples for SEM tests were ground on SiC papers and electrochemically polished in the AC2 electrolyte at 20 V and -25 °C for 100 s. The EBSD data was analyzed with Channel 5 software. Thin foils for TEM observation and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) with 0.06 mm in thickness were punched into discs with 3 mm in diameter and prepared by a precision ion polishing system (Gatan 691).

2.3. Tensile test

For evaluating the mechanical properties, the bone-shape tensile samples with a gauge length of 30 mm and a diameter of 5 mm were machined from the as-extruded rods. Tensile tests with a tensile direction parallel to the extrusion direction (ED) were conducted on a Shimadzu CMT-5105 material testing machine at ambient temperature
and an initial strain rate of 0.001 s⁻¹. The tests were repeated three times for each as-extruded bar.

3. Results

3.1. Microstructure of heat treated samples

Fig. 1 displays the OM micrographs of heat treated Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn (wt.%) alloys. All the heat treated alloys exhibit equiaxed grains with irregular shapes. The average grain sizes of the as-annealed and as-quenched samples are estimated to be approximately 208.6 µm and 123.6 µm, respectively. After pre-ageing at 225 °C for 110 h, obvious grain growth is observed with the average grain sizes of 233.2 µm and 144.1 µm, respectively. The SEM-BSE micrographs of heat treated alloys are illustrated in Fig. 2. It can be seen that the heat treated microstructures of the four samples are all composed of α-Mg matrix, Mg₅(Gd, Y) particles, block-shaped and lamellar LPSO phases. The bright particles with irregular shapes generally distribute around the grain boundaries or present inside the grains, indicating that they are the so-called β-Mg₅(Gd, Y) phase. The block-shaped LPSO phases primarily distribute around the grain boundaries. Some of the block-shaped LPSO phases are formed by the accumulation of lamellar structures. The lamellar LPSO phases appear in the grain interiors or precipitate from the block-shaped LPSO phases and grain boundaries to the grain interiors. Compared with the as-annealed and as-quenched samples, the pre-aged B and D samples possess dense relatively small precipitates, which cannot be distinguished distinctly in OM and SEM-BSE micrographs.

For further observation, the STEM micrographs and corresponding selected area
electron diffraction (SAED) patterns of the second phases in as-quenched and pre-aged D samples are shown in Fig. 3. The $\beta$-Mg$_5$(Gd, Y), (Gd, Y)H$_2$ and LPSO phases are detected in both samples. The Mg$_5$(Gd, Y) particles with face-centered cubic (fcc) crystal structure should be the residue from the as-cast alloy (see Supplementary Fig. 2) after the heat treatment, which have been widely observed in as-cast Mg-RE alloys [18-20]. A small number of bright particles with rectangular shape can be identified and the rectangular shape particles consist with the (Gd, Y)H$_2$ precipitates, which are typically observed near the region with high Gd and Y content. It has been confirmed that the hydrogen in rectangular RE-H particles observed in Mg-RE alloys typically comes from the external sources [21, 22], which suggests that the (Gd, Y)H$_2$ particles in the studied alloy is inevitably formed during sample preparation. Thus, further discussion about it is outside the scope of this article. The SAED patterns suggest that both the block-shaped and lamellar LPSO phases have a 14H structure. The lamellar LPSO phase in as-quenched sample ranges from ~11 nm to ~110 nm in thickness and from ~105 nm to ~447 nm in interlamellar spacing. After pre-ageing treatment, a great number of metastable $\beta'$ and $\gamma'$ precipitates generate as lenticular particles on the prismatic plane and lamellar plates on the basal plane, respectively. The size of prismatic $\beta'$ particles observed from the [0001] direction ranges from ~78 nm to ~260 nm, while the thickness of basal $\gamma'$ plates is invariable of a single unit cell height [23]. The interlamellar spacing of $\gamma'$ precipitates ranges from several nanometers to ~170 nm, which is much narrower than that of lamellar LPSO phases.

3.2. Microstructure and texture of as-extruded samples
Fig. 4 shows the EBSD IPF maps of as-extruded samples observed on longitudinal sections. The unindexed regions (indicated as black) with elongated morphology in ED correspond to the locations of coarse second phases in Fig. 4a-d. The microstructures are highly heterogeneous and remarkably refined after hot extrusion via dynamic recrystallization (DRX). All the four as-extruded samples show a typical bimodal microstructure which consists of fine DRXed grains and coarse unDRXed grains. In the EBSD analysis, the DRXed grains are defined as the grains with in-grain misorientation angle spread lower than 2°. The unDRXed grains are shown in Fig. 4e-h, and the colour variation indicates the presence of the substructures with dense dislocations and subgrain boundaries. As a consequence, the DRX ratios of as-extruded A and C samples are estimated to be about 86% and 61%, while the DRX ratios of as-extruded B and D samples are measured to be about 55% and 22%, respectively. The average grain sizes of DRXed grains in as-extruded A and C samples ($d_{a,DRX}=4.8 \pm 1.6 \mu m, d_{c,DRX}=3.1 \pm 0.9 \mu m$) are slightly larger than those of as-extruded B and D samples ($d_{b,DRX}=1.7 \pm 0.5 \mu m, d_{d,DRX}=1.5 \pm 0.4 \mu m$). These results suggest that the DRX is dramatically obstructed by the dense $\beta'$ and $\gamma'$ precipitates formed during the pre-ageing process. The dynamic recrystallization behavior has been discussed in the section 4.1.

Fig. 5 displays the SEM-BSE images of the as-extruded samples observed on transverse and longitudinal sections, and Fig. 6 shows the local magnified images of Fig. 5 (marked by red dashed boxes). It can be identified that the block-shaped LPSO, lamellar LPSO and Mg$_5$(Gd, Y) phases should also be the primary second phases, which are attributed to their high thermostability and/or dynamic precipitation. Different from
the LPSO phases observed in heat treated samples, the block-shaped and lamellar LPSO phases in as-extruded samples are crushed and exhibit an alignment with ED. Many kink bands in LPSO phases are observed. Combined with the EBSD IPF maps, the block-shaped LPSO phases are generally surrounded by the fine DRXed grains in obedience to the PSN mechanism [14, 24]. However, contrary to this, the kinked lamellar LPSO phases are usually found in the unDRXed grains. Bright particles marked by yellow circles in Fig. 6 are Mg$_3$(Gd, Y) phases. As the number density of Mg$_3$(Gd, Y) particles is much higher than that in heat treated samples, their presence is mainly related to the dynamic precipitation rather than the inheritance.

TEM analyses of different regions of the as-extruded D sample are exhibited in Fig. 7. In the STEM images on the DRXed region (Fig. 7(a, b)), the lamellar LPSO phase with a straight appearance inside the grains and pile-up dislocations with bulging characteristic around the grain boundaries can be observed (electron beam (EB) //[$\overline{2}\overline{1}0\overline{1}]_{Mg}$). The metastable $\beta'$ and $\gamma'$ precipitates observed in pre-aged samples are almost eliminated after extrusion. The straight LPSO phase in the as-extruded D sample is confirmed to have a 14H structure with thicknesses in the range from ~8 nm to ~26 nm, which is much narrower than that in pre-aged samples. Therefore, the lamellar LPSO phase in DRXed grains should form via dynamic precipitation during hot extrusion. The straight morphology is related to the negligible internal strain in DRXed grains. In the bright-field TEM image on the unDRXed region (Fig. 7d), the kinked LPSO phases and pile-up dislocations around lamellar LPSO phases can be observed with the EB/[$\overline{2}\overline{1}0\overline{1}]_{Mg}$. As a dominate deformation mode in LPSO phase, kink bands
of lamellar LPSO phases in the observed unDRXed grain exhibit a kinking degree about 8.2°. A number of dislocations are piled up between the adjacent lamellar LPSO phase, which suggests the formation of subgrain boundaries in unDRXed grains during hot extrusion. As the bright-field TEM image and SAED patterns shown in Fig. 7(e, f), it can be further confirmed that the β-Mg$_5$(Gd, Y) particles (F$\bar{4}$3m) have a fcc crystal structure with $a$=2.23 nm. The β-Mg$_5$(Gd, Y) particles show various sizes in diameter ranging from $\sim$12 nm to $\sim$383 nm. Around the coarse β-Mg$_5$(Gd, Y) particle, a DRXed grain and an unDRXed grain can be identified. The contrast variation in the unDRXed grain implies that the presence of misorientation gradient surrounding the coarse β-Mg$_5$(Gd, Y) particles, which is an indispensable evidence for PSN.

The inverse pole figures of entire grains, DRXed regions and unDRXed regions are presented in Fig. 8. All the as-extruded samples exhibit a bimodal texture, which consists of [10$\bar{1}$0]$_{\text{Mg}}$ and [0001]$_{\text{Mg}}$ texture components. The former texture component with [10$\bar{1}$0]$_{\text{Mg}}$ axis parallel to the ED is a typical fiber texture which is commonly observed in magnesium alloys [25], while the latter with [0001]$_{\text{Mg}}$ axis parallel to ED is an unusual texture which showed a wider spread of grain orientations. The maximum texture intensity of all samples is primarily dominated by the texture for unDRXed regions, while the unusual texture component is mainly affected by the texture for DRXed regions. The maximum texture intensity of as-extruded B and D samples is much higher than that of A and B samples, especially in the [10$\bar{1}$0]$_{\text{Mg}}$ texture component, which is attributed to the lower DRX ratio in the former two samples. The weak unusual texture component is also observed in other magnesium
alloys with high RE content, and its formation should be related to solute segregation and drag effect of Gd/Y atoms during recrystallization [26-28].

Fig. 9 shows the (0001)<11̅20> and (1̅100)<11̅20> Schmid factors (SF) distribution maps and histograms for different regions of the as-extruded alloys when the tensile stress is applied along the ED. In general, the average SF (m̄) for basal slip is much lower than that for prismatic slip, especially for the as-extruded B and D samples. The higher recrystallization fraction endows the sample with a lower m̄_{All} for basal slip but the higher m̄_{All} for prismatic slip. The as-extruded D sample exhibits the lowest m̄_{All} for basal slip but the highest m̄_{All} for prismatic slip. The m̄_{DRX} for basal slip is lower than that for m̄_{unDRX}, while the m̄_{DRX} for prismatic slip is higher than m̄_{unDRX}. It suggests that the basal slip for most unDRXed grains displays a hard orientation and is difficult to be activated. The weakened texture component for DRXed regions results in the relatively random distributions of both (0001)<11̅20> and (1̅100)<11̅20> SFs.

### 3.3. Mechanical properties of the as-extruded samples

Fig. 10 shows the tensile nominal stress-strain curves of the as-extruded samples tested along ED at ambient temperature. The as-extruded B and D samples show higher UTS and TYS, and slightly lower EL than A and C samples. The as-extruded D sample possesses the best tensile properties with UTS of 455 MPa, TYS of 382 MPa and EL of 11.0%. The data of tensile properties of various Mg-Gd-Zn and Mg-Y-Zn alloys, which were prepared by traditional casting, homogenization and hot extrusion, are summarized in Table 2. Samples of this work, in contrast, exhibit an outstanding
strength-ductility balance. The underlying strengthening mechanism of as-extruded samples will be discussed in section 4.2.

4. Discussion

4.1. Dynamic recrystallization behavior

The EBSD IPF maps (Fig. 4) show that DRX primarily takes place at grain boundaries. Some bulging grain boundaries between DRXed and unDRXed grains can be identified and the bulging characteristic is commonly associated with strain-induced boundary migration [29]. The size of these bulges is on the same scale with the adjacent DRXed grains. This conforms that the conventional discontinuous DRX (DDRX) mechanism took part in the dynamic recrystallization process. Furthermore, as the prerequisite condition for continuous DRX (CDRX), the multiple slip systems in Mg-RE alloys can be activated due to the decreased CRSS of non-basal slip during hot plastic deformation [30, 31]. The CDRX should also be the main mechanism for the recrystallization process, which is related to the transformation from low-angle grain boundaries gradually to the high-angle grain boundaries via recovery, dislocation climb and cross slip of multiple slip systems [32].

All the four samples prior to hot extrusion contain β-Mg₃(Gd, Y), block-shaped LPSO and lamellar LPSO phases. The pre-aged B and D samples additionally contain dense prismatic β′ and basal γ′ precipitates. The second-phase particles with various sizes, fractions and distributions are expected to significantly affect the dynamic recrystallization behavior during subsequent hot extrusion.

Some DRXed grains are observed at phase boundaries between the matrix and
block-shaped LPSO phases (Fig. 4). The hard block-shaped LPSO phases easily lead
to the frequent occurrence of high-stress concentration near the Mg/LPSO phase
interface during hot extrusion [1]. Therefore, the block-shaped LPSO phases usually
contribute to the recrystallization via the PSN mechanism. Some coarse β-Mg5(Gd, Y)
particles observed at grain boundary (Fig. 7e), analogously, promote the
recrystallization by the PSN mechanism, which was proved by the misorientation
gradient in adjacent unDRXed grains [14]. Contrary to this, the kinked lamellar LPSO
phases observed in coarse unDRXed grains (Fig. 8d) suggest a retarding effect on the
recrystallization. Although the dense dislocations trapped by lamellar LPSO phases are
caused by the high-stress concentration which is regarded as a prerequisite for
recrystallization, the kink deformation of the lamellar LPSO phase can effectively
coordinate the strain and release the distortion energy. Accordingly, the residual storage
energy is insufficient to support further recrystallization.

In the pre-aged samples, the dense prismatic β' and basal γ' precipitates are in the
size range which is expected to exert a particle pinning effect during hot extrusion (Fig.
3) [15]. The fraction and average grain size of DRXed grains in as-extruded B and D
samples are much lower and smaller than that in A and C samples. Compared with the
as-annealed and as-quenched samples with non-β' and non-γ' precipitates, the
recrystallization kinetics of pre-aged samples is observably retarded by dense β' and γ'
precipitates. In addition, the β' and γ' precipitates are subject to the dynamic solution
and almost eliminated due to the high temperature and pressure caused by hot extrusion.
The severe plastic deformation leads to a number of subgrain boundaries and a high
dislocation density in the vicinity of metastable β' and γ' precipitates [33, 34]. The formation of such microdefects promotes the diffusion and dissolution of RE and Zn into the matrix. Redissolving RE and Zn atoms are inclined to segregate at grain boundaries and slow the recrystallization kinetics by solute drag [35, 36]. Besides, the fine β-Mgs(Gd, Y) particles can also effectively pin the grain boundaries and prevent the growth of DRXed grains [37].

4.2 Strengthening mechanism

The strengthening of the as-extruded alloys is primarily attributed to microstructure characteristics: (i) the fine DRXed grains dragged by segregate solute and pinned by second-phase particles, (ii) the coarse unDRXed grains with strong [10\bar{1}0]_\text{Mg} fiber texture and dense substructures, (iii) the second-phase particles containing β-Mgs(Gd, Y), lamellar LPSO and block-shaped LPSO phases.

The presence of dense β' and γ' precipitates caused finer DRXed grains than the precipitate-free alloys after hot extrusion. Their grain boundary strengthening should follow the Hall-Petch relationship: \( \Delta \sigma_{\text{GB}} = k \cdot d^{1/2} \) [38]. \( k \) is a constant which has been determined to be approximately 164 MPa\(^{1/2} \) [39]. \( d \) is the average grain size. If all the alloys were fully recrystallized and kept the current DRXed grain size, the grain boundary strengthening of the four as-extruded samples should be \( \sim 75 \) MPa, \( \sim 126 \) MPa, \( \sim 93 \) MPa and \( \sim 134 \) MPa, respectively. Whereas, compared with the fine DRXed grain boundaries, grain boundary strengthening of coarse unDRXed regions is negligible (Fig. 4). Hence, the accurate grain boundary strengthening should be the estimated data multiplying the area fraction of the DRXed regions [40] and is expected to be \( \sim 64 \) MPa,
~69 MPa, ~57 MPa and ~29 MPa, respectively. Although the as-extruded sample D shows the highest strength, it exhibits the weakest grain boundary strengthening due to its low recrystallization fraction.

The strong [10\overline{1}0]_\text{Mg} fiber texture component (Fig. 4 and 8) implies that the c-axis of most coarse unDRXed grains is perpendicular to the ED. This kind of crystallographic orientation leads to a distinct low SF for basal slip and high difficulty to activate their basal slip, which is normally easy to be activated due to its relatively low CRSS. Although the CRSS ratio of basal slip to prismatic slip is relatively low, the average SF for prismatic slip is quite high (Fig. 9) and the hard-to-activate prismatic dislocations slip should be the dominant deformation mode. A similar phenomenon has been confirmed in unDRXed grains in Mg-8.2Gd-3.8Y-1Zn-0.4Zr alloy by the in situ SEM-EBSD tensile tests along the [10\overline{1}0]_\text{Mg} direction [10]. In addition, acting as stress concentrators, the dense substructures with accumulated dislocations and subgrain boundaries inside unDRXed grains contribute to impede the dislocation slips but exerts an adverse effect on the ductility. Therefore, the higher fraction of unDRXed regions in the as-extruded B and D samples give the alloy higher strength but lower elongation.

The second-phase particles affect not only the dynamic recrystallization behavior but also the strengthening mechanism. Dynamically precipitated fine \(\beta\)-Mg\(_5\)(Gd, Y) particles and lamellar LPSO phases play a role in dispersion strengthening via the Smith-Zener pinning effect. Whereas the \(\beta\)-Mg\(_5\)(Gd, Y) particles, especially in coarse particles, have been proved to be the main crack source and provide a path for crack
extension along grain boundaries [41]. The basal plates, lamellar LPSO phase, are known to be more effective in increasing the activation energy of prismatic slip than the basal slip [42]. In the strongly textured unDRXed regions, the activation energy of the dominant deformation mode, prismatic slip, can be further improved. The thinner thickness and narrower interlamellar spacing for the lamellar LPSO phase are more effective in hindering the dislocation motion and improving the strength. Differently, the block-shaped LPSO grains aligned along the ED are a too large to directly affect the motion of dislocation via the pinning effect rather than acting as the reinforcement exert short-fiber strengthening effectively [1]. It has been reported that the CRSS of prismatic slip (~290 MPa) is approximately 13 times higher than basal slip (~23 MPa) in LPSO grain [43], since the basal slip is perceived as its dominant deformation mode [44]. Nevertheless, the SF of basal slip is very small due to the plate-like interface of the aligned LPSO phase being approximately parallel to the basal plane of matrix and LPSO grain. The activation of basal slip is forcefully restricted in LPSO grain. The short-fiber strengthening mechanism is similar to the reinforcement in composite materials [1].

5. Conclusion

In summary, this study investigated the influence of pre-ageing treatment on the microstructure and mechanical properties of the Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn (wt.%) alloy with outstanding strength-ductility balance. The following conclusions can be made:

(1) Pre-ageing treatment at 225 °C for 110 h generates dense prismatic β’ and basal
\( \gamma' \) precipitates inside grains in the heat treated alloy before hot extrusion. These precipitates are subject to the dynamic solution and totally eliminated during hot extrusion.

(2) The dynamic recrystallization process is dramatically obstructed by the dense precipitates (\( \beta' \) and \( \gamma' \)) and the recrystallization kinetics is effectively slowed by the solution of precipitates and segregation of solute elements (Gd, Y and Zn) at grain boundaries.

(3) The block-shaped LPSO phase and coarse \( \beta\text{-Mg}_5(Gd, Y) \) phase promote recrystallization via PSN, while the lamellar LPSO phase restrains it via forming kink bands and releasing the stress concentration. The fine \( \beta\text{-Mg}_5(Gd, Y) \) particles suppress the recrystallization by the particle pinning effect.

(4) The as-extruded D sample exhibits the best strength-ductility balance with UTS of 455 MPa, TYS of 382 MPa and EL of 11.0%. The outstanding mechanical properties result from the bimodal microstructure, strong \([10\overline{1}0]\text{Mg}\) fiber texture, \( \beta\text{-Mg}_5(Gd, Y) \) particles, lamellar and block-shaped LPSO phases.

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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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Table 1 Heat treatments performed on the Mg-Gd-Y-Zn-Mn alloys.

Table 2 Tensile properties of the as-extruded samples tested along ED at ambient temperature.

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Fig. 1. Optical micrographs of heat treated alloys: (a) as-annealed sample, (b) pre-aged B sample, (c) as-quenched sample and (d) pre-aged D sample.

Fig. 2. SEM-BSE micrographs of heat treated alloys: (a, e) as-annealed sample, (b, f) pre-aged B sample, (c, g) as-quenched sample and (d, h) pre-aged D sample. Note, the orange, green and red arrows or dashed circles indicate the RE particles, lamellar and block-shaped LPSO phase, respectively.

Fig. 3. STEM observations of heat treated alloys and corresponding SAED patterns: (a, b, c) as-quenched sample, (d, e, f) pre-aged D sample.

Fig. 4. EBSD IPF maps of as-extruded samples on longitudinal sections: (a, e) as-extruded A sample, (b, f) as-extruded B sample, (c, g) as-extruded C sample and (d, h) as-extruded D sample.

Fig. 5. SEM-BSE micrographs of the as-extruded samples observed on (a, c, e, g) transverse sections and (b, d, f, h) longitudinal sections, respectively. (a, b) as-extruded A sample, (c, d) as-extruded B sample, (e, f) as-extruded C sample and (g, h) as-extruded D sample, respectively. Note, the green and blue arrows indicate the lamellar and block-shaped LPSO phase, respectively.
**Fig. 6.** Higher resolution micrographs of the regions indicated in Fig. 5. Note, orange arrows indicate kink bands of the LPSO phase. The green and yellow circles indicate the cracked LPSO and Mg$_5$(Gd, Y) phases, respectively.

**Fig. 7.** TEM micrographs of as-extruded D sample: (a, b) STEM images on the DRXed region and (c) corresponding SAED patterns; (d) bright-field TEM image on the unDRXed region; (e) bright-field TEM image of Mg$_5$(Gd, Y) phase and (f) corresponding SAED patterns.

**Fig. 8.** EBSD IPF taken from different regions of the as-extruded alloys on longitudinal sections: (a) as-extruded A sample, (b) as-extruded B sample, (c) as-extruded C sample and (d) as-extruded D sample.

**Fig. 9.** Schmid factor distribution maps and histograms of the as-extruded alloys when the tensile stress is applied along the ED: (a, e) as-extruded A sample, (b, f) as-extruded B sample, (c, g) as-extruded C sample and (d, h) as-extruded D sample.

**Fig. 10.** Tensile properties of the as-extruded samples tested along ED at ambient temperature.
<table>
<thead>
<tr>
<th>Designation</th>
<th>Thermal Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-annealed</td>
<td>1. Semi-continuous casting</td>
</tr>
<tr>
<td></td>
<td>2. 500 °C × 8 h + 525 °C × 5 h + annealing to 400 °C within 10 h</td>
</tr>
<tr>
<td>Pre-aged B</td>
<td>1. As-annealed sample</td>
</tr>
<tr>
<td></td>
<td>2. 225 °C × 110 h</td>
</tr>
<tr>
<td>As-quenched</td>
<td>1. As-cast sample</td>
</tr>
<tr>
<td></td>
<td>2. 500 °C × 8 h + 525 °C × 5 h + quenching</td>
</tr>
<tr>
<td>Pre-aged D</td>
<td>1. As-quenched sample</td>
</tr>
<tr>
<td></td>
<td>2. 225 °C × 110 h</td>
</tr>
<tr>
<td>As-extruded A</td>
<td>1. As-annealed sample</td>
</tr>
<tr>
<td></td>
<td>2. Hot extrusion (420 °C, 11:1, 0.3 mm/s)</td>
</tr>
<tr>
<td>As-extruded B</td>
<td>1. A + Pre-aged sample</td>
</tr>
<tr>
<td></td>
<td>2. Hot extrusion (420 °C, 11:1, 0.3 mm/s)</td>
</tr>
<tr>
<td>As-extruded C</td>
<td>1. As-quenched sample</td>
</tr>
<tr>
<td></td>
<td>2. Hot extrusion (420 °C, 11:1, 0.3 mm/s)</td>
</tr>
<tr>
<td>As-extruded D</td>
<td>1. Q + Pre-aged sample</td>
</tr>
<tr>
<td></td>
<td>2. Hot extrusion (420 °C, 11:1, 0.3 mm/s)</td>
</tr>
</tbody>
</table>
Table 2  Tensile properties of various as-extruded Mg-RE-Zn alloys at ambient temperature.

<table>
<thead>
<tr>
<th>Alloy (wt%)</th>
<th>UTS (MPa)</th>
<th>TYS (MPa)</th>
<th>EL (%)</th>
<th>Refs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn</td>
<td>402 ± 5</td>
<td>295 ± 6</td>
<td>11.0 ± 0.4</td>
<td>Present work</td>
</tr>
<tr>
<td>Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn</td>
<td>419 ± 5</td>
<td>338 ± 5</td>
<td>12.1 ± 0.4</td>
<td>Present work</td>
</tr>
<tr>
<td>Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn</td>
<td>416 ± 5</td>
<td>332 ± 6</td>
<td>15.2 ± 0.4</td>
<td>Present work</td>
</tr>
<tr>
<td>Mg-9.2Gd-4.4Y-1.0Zn-0.8Mn</td>
<td>455 ± 5</td>
<td>382 ± 5</td>
<td>11.0 ± 0.4</td>
<td>Present work</td>
</tr>
<tr>
<td>Mg-14.0Gd-2.3Zn</td>
<td>380</td>
<td>345</td>
<td>6.9</td>
<td>[45]</td>
</tr>
<tr>
<td>Mg-10.0Gd-5.7Y-1.6Zn-0.5Zr</td>
<td>461</td>
<td>419</td>
<td>3.6</td>
<td>[6]</td>
</tr>
<tr>
<td>Mg-8.2Gd-3.8Y-1.0Zn-0.4Zr</td>
<td>442</td>
<td>379</td>
<td>14.7</td>
<td>[46]</td>
</tr>
<tr>
<td>Mg-12.6Gd-1.3Y-0.9Zn-0.5Mn</td>
<td>395</td>
<td>314</td>
<td>15.2</td>
<td>[47]</td>
</tr>
<tr>
<td>Mg-11.5Gd-4.5Y-1Nd-1.5Zn-0.5Zr</td>
<td>362</td>
<td>305</td>
<td>6.2</td>
<td>[48]</td>
</tr>
<tr>
<td>Mg-7Y-5Sm-0.5Zn-0.3Zr</td>
<td>358</td>
<td>250</td>
<td>18.5</td>
<td>[19]</td>
</tr>
<tr>
<td>Mg-12Y-7Zn-0.6Zr</td>
<td>429</td>
<td>351</td>
<td>2</td>
<td>[49]</td>
</tr>
</tbody>
</table>
Fig. 1. Optical micrographs of heat treated alloys: (a) as-annealed sample, (b) pre-aged B sample, (c) as-quenched sample and (d) pre-aged D sample. The d in the text means the average grain size.
**Fig. 2.** SEM-BSE micrographs of heat treated alloys: (a, e) as-annealed sample, (b, f) pre-aged B sample, (c, g) as-quenched sample and (d, h) pre-aged D sample. Note, the orange, green and red arrows or dashed circles indicate the RE particles, lamellar and block-shaped LPSO phase, respectively.
Fig. 3. STEM observations of heat treated alloys and corresponding SAED patterns: (a, b, c) as-quenched sample, (d, e, f) pre-aged D sample.
Fig. 4. EBSD IPF maps of as-extruded samples on longitudinal sections: (a, e) as-extruded A sample, (b, f) as-extruded B sample, (c, g) as-extruded C sample and (d, h) as-extruded D sample.
Fig. 5. SEM-BSE micrographs of the as-extruded samples observed on (a, c, e, g) transverse sections and (b, d, f, h) longitudinal sections, respectively. (a, b) as-extruded A sample, (c, d) as-extruded B sample, (e, f) as-extruded C sample and (g, h) as-extruded D sample, respectively. Note, the green and blue arrows indicate the lamellar and block-shaped LPSO phase, respectively.
**Fig. 6.** Higher resolution micrographs of the regions indicated in Fig. 5. Note, orange arrows indicate kink bands of the LPSO phase. The green and yellow circles indicate the cracked LPSO and Mg₅(Gd, Y) phases, respectively.
Fig. 7. TEM micrographs of as-extruded D sample: (a, b) STEM images on the DRXed region and (c) corresponding SAED patterns; (d) bright-field TEM image on the unDRXed region; (e) bright-field TEM image of Mg5(Gd, Y) phase and (f) corresponding SAED patterns.
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Fig. 10. Tensile properties of the as-extruded samples tested along ED at ambient temperature.