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The microstructure and mechanical properties of Mg-Gd-Y-Zn-Zr system and Mg-Gd-Y-Zr system alloys by RUE deformation

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Abstract

This study investigates the effects of long-period stacking order (LPSO) phases on the dynamic recrystallization (DRX) process. By comparing Mg-Gd-Y-Zn-Zr (GWZ) alloy and Mg-Gd-Y-Zr (GW) alloy deformed by 4 passes of Isothermal Reciprocating Upsetting-Extrusion (RUE), the initial alloy grain sizes are 113.4 μm and 88.2 μm, respectively, after 4 passes, the grain size becomes 3.5 μm and 4.8 μm, and the grain refinement of GWZ is greater. After 1 pass, the DRX volume fraction of GWZ alloy is 83% and 15%, which is related to the LPSO phases contained in the GWZ alloy. The texture strength of the GWZ alloy ranges from 8.5 of 1 pass to 2.2 of 4 passes, while the GW alloy is from 5.9 of 1 pass to 2.8 of 4 passes, mainly due to the DRX grain volume fraction. The tensile test results at room temperature (RT) showed that the ultimate tensile strength (UTS) and tensile yield strength (TYS) of GWZ alloy are higher than that of GW alloy, and the elongation is lower than that of GW alloy.

1. Introduction

As the lightest metal structural material at present, magnesium alloy has a series of advantages such as small specific gravity, specific strength, high specific rigidity, good machinability and easy to recycle, and is widely used in aerospace, transportation, and other fields [1–4]. However, the applicability of Mg alloy has been limited [5–10] due to the HCP (hexagonal close-packed) structure and poor deformability, so Mg-RE alloys with enhanced deformability and weakened mechanical anisotropy have been in increasingly more attention [11–15]. Mg-Gd-Y-Zr alloy stands out among Mg-RE alloys due to its excellent high-temperature strength, reasonable ductility, and ideal creep resistance [16–19]. Jing Li et al [20] studied the Mg-8Gd-3Y-0.5Zr alloy having a ultimate tensile strength (UTS) of 293 MPa, a tensile yield strength (TYS) of 210 MPa, and an elongation (EL) of 12% at room temperature (RT). In recent years, researchers have added Zn element to the Mg-Gd-Y-Zr alloy. Under the appropriate addition amount and process conditions, a long period ordered structure (LPSO) can be generated inside the alloy. This structure enables the alloy to exhibit excellent room temperature yield strength and good elongation [21–32]. Duxiu Zhang et al [33] studied the Mg-6Gd-3Y-Zn-0.4Zr alloy containing LPSO phase. After hot compression deformation, the UTS at room temperature was 232.5 MPa, the TYS was 232.5 MPa, and the EL was 9.9%. However, so far, the relevant research has only a single analysis of the microstructure and mechanical properties of Mg-Gd-Y-Zr alloy or Mg-Gd-Y-Zn-Zr alloy. No scholar has intuitively compared the two alloys quantitatively. There are few studies on the influence of LPSO on the microstructure and mechanical properties of alloys, and there is no strong evidence for the influence of LPSO on the DRX process and texture changes of alloys.

In this study, the Mg-8Gd-3Y-2Zn-0.5Zr alloy and Mg-8Gd-3Y-0.5Zr alloy are subjected to isothermal RUE deformation. Through a series of microstructure characterization and mechanical property tests, the microstructure and mechanical properties of Mg-Gd-Y-Zn-Zr and Mg-Gd-Y-Zr alloys by RUE deformation was studied. It provides some ideas for the future industrial selection of alloys and alloy deformation methods.

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2. Material and methods

The experimental alloy used semi-continuous casting Mg-8Gd-3Y-2Zn-0.5Zr alloy (hereinafter referred to as GWZ alloy) and Mg-8Gd-3Y-0.5Zr alloy (hereinafter referred to as GW alloy), then homogenized at 520 °C for 16 h. Cylindrical specimens with 50 mm in diameter and 230 mm in height for further RUE experiments. Figure 1 shows a schematic diagram of RUE. Then, the cylindrical samples were upset into round rods with the diameter of 70 mm, and then the ejector rod is taken out, and the upsetting samples were positively squeezed. After the extrusion, the diameter changed back to 50 mm, which completes a single pass of deformation. Then repeat until four passes of deformation are completed.

The experiment was carried out on a 6300kN press, the speed was 1 mm s⁻¹, and the deformation temperature was 420 °C for each pass. Before each pass of the experiment, the mold and sample should be heated to 450 °C and kept for two hours. Oil-based graphite was used as a lubricant to reduce the friction between the sample and the mold. The experiment was carried out 4 passes in total, and the samples were cooled to room temperature at the end of each pass. Taken samples for observation from the center of the head of the deformed bar, the observation direction is parallel to the extrusion direction (ED).

Optical microscope (OM, Zeiss), scanning electron microscope (SEM, SU-5000, Hitachi Co.) and electron backscatter diffraction (EBSD, Hitachi SU5000 FE-SEM) were used to analyze the microstructure of different samples. Before OM observation, first sanded with sandpaper and mechanically polished, and then etched in a solution of 1 g picric acid, 14 ml alcohol, 2 ml acetic acid and 2 ml distilled water. Scanning electron microscope observation is equipped with backscattered electron (BSE) mode, field emission gun and energy dispersive X-ray spectroscopy (EDS) detector. The EBSD observation was performed on Hitachi SU5000 FE-SEM, the working distance was 15 mm, and the working voltage was 20kv. The samples for SEM and EBSD observation were prepared by sandpaper and mechanical polishing. Before the EBSD observation, there isidual stress layer was removed by using a Leica EM Res 102 ion thinning instrument operating at 6.5 kV for 35 min X-ray diffraction (XRD, Smart Lab SE,) under the conditions of 40 kV and 40 mA The Cu Ka radiation was analyzed to analyze

3. Results

3.1. Initial state microstructure

The optical microstructures (OM) of the two alloys in the initial state were observed, as shown in figure 2. It can been seen from figure 2(a) that the block-shaped phases are distributed at the grain boundary in a network shape, the lamellar phases are distributed on the matrix. It can be identified that the matrix phase is α-Mg, and the block-shaped and lamellar phases are both Mg12(Gd,Y)Zn, two different forms of 14H LPSO [33]. GW alloy does not contain LPSO phase because it does not contain Zn element. Only the α-Mg matrix phase can be

![Figure 1. Schematic illustrations of RUE process.](image1)

![Figure 2. OM images of initial state (a) GWZ alloy and (b) GW alloy.](image2)
observed (figure 2(b)). The picture shows that both alloys are mainly composed of equiaxed grains, the initial grain size of GWZ alloy is about 113.37\(\mu\)m, and the initial grain size of GW alloy is about 88.23\(\mu\)m. The initial grain size of GW alloy is relatively smaller.

### 3.2. Evolution of microstructure after RUE deformation

Figure 3 shows the OM micrographs of the two alloys after RUE in different passes. Figure 4 is the XRD pattern of the alloys after RUE deformation. During the RUE deformation of the alloy from 1 pass to 4 passes, no new phases are formed, but the content changes (figure 4). The content of precipitated phases increases with the increase of the deformation passes. After 1 pass, the grains of the two alloys have been refined (figures 3(a) and (e)). Typical bimodal microstructures can be found at the original coarse grain boundaries. The coarse deformed grains are elongated along the ED, and chain-like DRXed grains appear at the trigeminal grain boundaries (GBs) of the coarse grains. It shows that DDRX [34] occurred after RUE 1 pass. The GBs have a great hindrance to the movement of dislocations, hence it is easy to cause stress concentration and severe deformation in GBs. A few particles phases are precipitated at the DRXed GBs, which indicates that the alloy is dynamically precipitated during the RUE process. These particles have a pinning effect on the sliding of the GBs, which can inhibit the growth of DRX grains during the heating and thermal deformation between passes [35]. The lamellar LPSO phases in the GWZ alloy have also changed after a single deformation. The lamellar LPSO phases inside the original coarse grains are significantly reduced compared to the initial state, and some of them are bent at a small angle, and the size of the block-shaped phases are also larger than the initial state (figure 3(a)).

As the deformation passes increase, the proportion of DRXed grains continues to increase as the coarse grains are continuously consumed, and the microstructure distribution becomes more uniform (figures 3(b), (f), (c), (g)). The lamellar LPSO phases in the GWZ alloy are gradually broken, and its existence is no longer visible by the fourth pass. It may be that the phase disappears after deformation, or it may be too small to be mixed with DRX grains after crushing. The block-shaped LPSO phases are also decomposed and broken during the deformation process, and its size has changed from 20 \(\mu\)m of 1 pass to 11 \(\mu\)m of 4 passes. Although some of the original coarse grains are still retained after RUE 4 passes, many grains are refined into equiaxed grains and the microstructure has changed, as shown in the figures 3(d), (k). With the increase of processing passes, the tendency of grain refinement weakens.
Figure 5 shows the backscattered electron (BSE) images of two RUEed alloys, in which the contrast between the matrix and DRXed grains is darker, while the contrast between the block-shaped phase and the precipitated phase is brighter [36]. The phases contained in the alloy change morphologically or quantitatively as the RUE passes increases. The deformation of the lamellar LPSO is marked with an elliptical blue dashed line in the figure, the block-shaped LPSO is marked with a rectangular green dashed line, and the DRX crystal grain is marked with an elliptical yellow solid line.

In the GWZ alloy (figures 5(a)–(d)), after 1 pass, it is found that the direction of LPSO phases tends to be distributed along the ED. A small amount of lamellar LPSO phases is bent at a small angle, and some have not changed significantly. Compared with the initial state, the block-shaped phases are reduced, and a small part is broken, and the small block-shaped phase after crushing is streamlined along the ED direction. As indicated by the yellow ellipse in the figure, some fine DRX grains appeared at the original grain boundaries, and some were formed around the kinking banks of the lamellar LPSO phase. The large misorientation formed by the kinks and the sufficient local plastic strain around them create favorable conditions for the formation of DRX. There are also some DRX grains around the bulk phases. Because the elastic modulus and hardness of the LPSO phase are much higher than that of the α-Mg matrix [31, 37–39], the stress concentrated at the α-Mg/LPSO interface due to the incongruous deformation between the LPSO phase and matrix during RUE process, causes a mass of intensive stress round the LPSO phases, which can promote the emergency of DRXed grains, and LPSO can provide an ideal location for the nucleation of DRXed grains by means of the particle stimulated nucleation (PSN) [40–42] mechanism. Excluding grain refinement, there are many dynamic precipitations around DRXed grains, which are β phases (Mg5 (Gd, Y, Zn)) [43]. These β phases also inhibit grain boundary migration and hinder the growth of grains through the PSN mechanism.

As the RUE passes increase, the block-shaped LPSO will be twisted and broken one after another. At 4 passes, the block-shaped LPSO phase is very small, and its existence is almost invisible. However, the proportion of the block-shaped LPSO phase is getting larger and larger, and some of them are decomposed and gradually become lamellar. The area where DRX occurs also expands. At 4 passes, two areas can be seen: one is uniformly distributed DRX grains, and the other is a block-shaped phase that is streamlined along the ED direction.
In the GW alloy (figures 5(e), (f), (g)), the most significant change is that the precipitation phase increases as the RUE passes increases. Simultaneously, it can be seen from figures 3(e), (f), (g), and (h) that Mg5(Gd, Y) phases primarily precipitate at the DRXed GBs. The reason is that as the strain increases, the stress at the grain boundary is concentrated, the dislocation density increases, and the lattice distortion is serious, which provides the necessary channels and energy for the dissolution of solid solution atoms, that is, strain-induced precipitation. The precipitated Mg5(Gd, Y) phase accelerates the formation of DRX through the PSN mechanism, and further promotes the grain refinement of the alloy.

Figure 6 shows grain orientation distribution and grain size change diagram of the microstructure of two alloys with 4 passes RUE. The same or similar colors indicate the same or similar crystal grain orientations, and the black part indicates the LPSO phase. Although the alloy composition is different, the grains are refined a lot after the 4 passes of RUE. At the same time, it can be seen from the color distribution in figure 6 that the orientation distribution of the two alloys becomes more random and diversified as the passes increase. No twins appear in the grains, suggesting that LPSO phase and high temperature can inhibit the appearance of twins.

The grain size of GWZ alloy is refined from 8.35 μm of 1 pass to 3.5 μm of 4 passes, and the volume fraction of DRX grains increases from 83% to 95%. The grain size of GW alloy is refined from 42.8 μm of 1 pass to 4.83 μm of 4 passes, and the volume fraction of DRX increases from 15% to 94%. GWZ alloy is 5 times thinner than GW alloy after RUE 1 pass, because compared to GW alloy, GWZ alloy contains LPSO phase, which promotes the DRX process. The grain refinement during RUE can draw the following conclusions: as the RUE passes increases, the grains become finer, and the standard deviation of the grain size becomes smaller and smaller, which proves that RUE can refine grains uniformly. The coarse grains are broken, and DRX appears at the new grain boundary. Because when the coarse grains are broken, dislocations often accumulate at the grain boundaries, and then stress concentration occurs, and DRX occurs.

3.3. Texture evolution
Figure 7 shows the {0001} texture map of two alloys under 4 RUE passes. From a bird eye view, after RUE 1 pass, a strong basal texture appeared in the GWZ alloy and GW alloy. From (a)–(d) and (e)–(h), as the passes increase, the texture strength gradually weakens, because the cumulative strain gradually increases during the RUE deformation process, and the original coarse grains decrease, and the DRX grains increased, DRX grain
orientation is more random, so the overall texture strength is reduced. As the passes increases in figure 7, the maximum pole intensity of the GWZ alloys decreases from 8.512 to 2.207 and GW alloys from 5.971 to 2.837, respectively. After four passes of RUE deformation between the two alloys, the texture strength of the alloy is not much different. However, for the two alloys, the degree of weakening of the texture in each pass is not the same, which may be related to the change in the percentage of DRX grains. Mg alloys will produce a large number of DRXed grains during thermal deformation. DRXed grains exhibit random orientation, which can offset the deformation texture of un-DRXed (non-dynamic recrystallized) grains to a certain extent. With the continuous increase of RUE deformation passes, alloys will precipitate particle phases at the GBs, and the particle phases will have a interruptive effect to the rotation of the grains, which have a significant weakening effect on the deformation texture of the alloy.

After GWZ alloy undergoes one or two passes of RUE deformation, the maximum pole density is reduced from 8.512 to 2.968, which is about 65.13%, while the reduction of GW alloy is only 18.74%, and the difference in texture reduction is nearly 3.5 times. In addition to the PSN mechanism of Mg5Gd particles, there are also different forms of LPSO phases in GWZ alloy compared with GW alloy at the initial stage of deformation, which will induce DRX formation in different ways. The block-shaped LPSO phase is broken during the deformation process, forming a fine and dispersed phase morphology distribution inside the alloy, which will further induce the formation of DRX by the means of the PSN mechanism. During the RUE process, due to external forces, dislocation movement and grain boundary slippage are hindered, resulting in the accumulation of dislocations in the lamellar LPSO phase and the α-Mg matrix, and twisting occurs (see in figure 5(a)), release a lot of energy and induce DRX generation [45]. Therefore, after a series of RUE deformation, the existence of different LPSO phases in the GWZ alloy will induce the occurrence of DRX, resulting in the DRX ratio in the GWZ alloy being 5.5 times that of the GW alloy. In addition, the grain size of GWZ alloy is much smaller than that of GW alloy in 1, 2 passes, which is also conducive to the formation of DRXed grains. This is because there are more grain boundaries in the fine grain structure, which is more conducive to the nucleation of DRXed grains on the grain boundaries, and as the grain size decreases, the strain when the peak stress occurs is also reduced, so DRX is more likely to occur.

However, after 4 passes of deformation, the DRX volume fraction of the GW alloy and the cumulative strain gradually increase, the increase of the DRX volume fraction in the GWZ alloy becomes smaller and smaller, because there are fewer and fewer places suitable for DRX nucleation. On the contrary, GW alloy does not have a large number of LPSO phases to provide nucleation sites for DRX in the early stage of deformation, and can only rely on dislocation to slip in relatively unobstructed large grains, with a long slip distance [46], and has a lower volume fraction of DRX grains compared with GWZ alloy. However, as the cumulative strain increases, the dislocation density of grains increases significantly and piles up and rearranges, which provides favorable conditions for the formation of DRX in the later deformation. Therefore, the volume fraction of DRX in GW alloy is almost equal to that in GWZ alloy after 4 passes of RUE deformation.

3.4. The influence of DRXed grains on texture

Figure 8 describes the DRX behavior of the undRXed grain obtained from the black rectangle in the corresponding OM images (figure 6(a)), including low angle grain boundaries (LAGBs) with 2–15° and high angle grain boundaries (HAGBs) with 15–100°, as well as some new DRXed grains G1–G8 are labeled. Meanwhile, it is presented in terms of inverse pole figure coloring at the (0001) pole figure (figure 8(b)) and

![Figure 7](image_url)
inverse pole figure (figure 8(c)). It can be seen from figure 8(a) that there are plenty of LAGBs (marked by the yellow arrow), indicating a high dislocation activity exists in the GWZ alloy. It is reported that the formation of LAGBs is related to the accumulation of dislocations, and these LAGBs can continuously absorb more mobile dislocations and transform themselves into HAGBs with increasing strain, eventually transform the sub-grains into new DRXed grains, that is CDRX mechanism [47].

It can be observed from the pole figure (figure 8(b) and inverse pole figure (figure 8(c)) that the orientation of the most DRXed grains is different from the parent grains. Only is the c-axis of some DRXed grains (G1,G5, G7) closed to TD direction, is same as the parent grains, while other DRXed grains (G2,G3,G4,G6,G8) have a more random orientation. The orientation of the most DRXed grains is random, is different from the orientation of the parent grain. It can be inferred that the extent of texture weakening is related to the fraction of DRXed grains with random orientation, showing an agreement with results of figure 7.

3.5. Mechanical properties
Figure 9 is the tensile properties of GWZ alloy and GW alloy. Figure (a) is the tensile strength, figure (b) is the yield strength, and figure (c) is the elongation. In the initial state, the UTS and TYS of GW alloy are higher than that of GWZ alloy. It is because in the initial state of the GWZ alloy, the hard block LPSO phase is distributed at the grain boundary, which easily leads to uneven stress during stretching. The stress is concentrated at the phase boundary between the α-Mg matrix and LPSO phases, and cracks tend to be induced in this place. As the RUE passes increases, the block-shaped LPSO phase is twisted, broken, and changed from the original distribution at the grain boundary to a uniformly dispersed distribution, which becomes the strengthening phase. At the same time, the Mg₅(Gd,Y,Zn) phase is conti-nuously precipitated during the deformation process, and the fine precipitated phases play a pinning role, thereby strengthening the alloy. However, there is no LPSO phase in the GW alloy, only the continuously precipitated Mg₅(Gd,Y,Zn) phase. Both alloys have strengthening phases in the deformation, so the strength increases. In the GW alloy, only the precipitated particle phase is present, and the LPSO phases does not appear, so the UTS and TYS of the GWZ alloy are higher than that of the GW alloy after deformation.

The mechanical properties of the alloy are greatly affected by the softening effect of the DRX grains and the hardening effect of the un-DRX grains. It can be obtained from figure 6 that the proportion of DRX grains in the GWZ alloy during the RUE deformation is higher than that of the GW alloy, and its average grain size is also much lower than that of the GW alloy. Therefore, the softening effect of DRX grains on the GWZ alloy is greater than that of the GW alloy, which is beneficial to the improvement of the ductility of the GWZ alloy. However, it can be observed from figure 9(c) that the ductility of the GWZ alloy during the 1–4 pass deformation process is always lower than that of the GW alloy, which should be related to the massively distributed block-shaped LPSO phase in the GWZ alloy. It shows that the block-shaped LPSO has a hardening effect on the alloy, and this hardening effect is greater than the softening effect of the DRX grains. When the deformed alloy is subjected to tensile deformation, the massively distributed block-shaped LPSO phase in the GWZ alloy will produce stress concentration, which will induce the generation of microcracks and ultimately reduce the ductility of the alloy.
4. Conclusion

1. The grains of GWZ alloy and GW alloy are refined after isothermal RUE deformation, but the degree of refinement is different. The GW alloy is refined from 8.35 $\mu$m to 3.5 $\mu$m, and the GW alloy is refined from 42.8 $\mu$m to 4.84 $\mu$m. Among the GW alloy, there are only precipitation of the granular phases promotes DRX. However in the GWZ alloy, in addition to the granular phase, there are also the LPSO phases (including the fragmentation of the bulk phases and the kinking of the lamellar phases), which creates favorable conditions for the formation of DRXed grains.

2. After RUE deformation, the two alloys have different texture strengths. The texture of GWZ alloy is lower than that of GW alloy. The reason is that the grains of GWZ alloy are relatively fine, the proportion of DRX grains is high, and the orientation distribution is random, weaken the strength of the alloy texture.

3. In the initial state, the UTS and TYS of the GWZ alloy are not as high as the GW alloy, because the bulk LPSO phase is distributed near the grain boundary, which causes cracks to initiate easily during stretching; and after the RUE deformation, the GWZ alloy is higher than the GW alloy because the uniformly dispersed block-shaped LPSO phase can strengthen the alloy. The GW alloy has only the grain precipitated phase Mg5(GdYZn) strengthened alloy.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.
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