Effects of twin-twin interactions and deformation bands on the nucleation of recrystallization in AZ31 magnesium alloy

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HIGHLIGHTS

• Recrystallization around twin-twin interactions results in weakening texture in annealed cryogenically-rolled samples.
• Twin-twin interactions are prone to recrystallization due to high kernel average misorientation values.
• Recrystallization around deformation bands results in strengthening texture in annealed room-temperature-rolled samples.

GRAPHICAL ABSTRACT

ABSTRACT

Investigating recrystallization is essential to optimize the microstructure including texture weakening and grain refinement in the rolling of magnesium alloys, thus to improve the mechanical properties of magnesium sheets for industrial applications. This research has gained an in-depth understanding of the effects of deformation bands and twin-twin interactions on recrystallization, which will potentially lead to improved manufacturing processes and mechanical properties of magnesium alloys. To study their individual effects, the recrystallization mechanisms of the room-temperature (RT)-rolled and liquid-nitrogen-temperature (LNT)-rolled samples during the annealing process were analysed with the quasi-in-situ electron backscatter diffraction method, respectively.

It is found that recrystallization mainly occurred in deformation bands in the RT-rolled sample, which enhanced the initially formed texture, due to oriented and inhomogeneous grain growth. However, it is of great interest to see that the recrystallized sites were mainly located around the (1012)-(0112) twin-twin interactions with high kernel average misorientation (KAM) values in the LNT-rolled sample, resulting in rather weaker texture, finer grain size and more homogeneous microstructure, because of the randomized orientations of recrystallized grains and uniform grain growth, while almost no recrystallization was observed around the single tension twin variant.

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1. Introduction

Recrystallization occurs in the thermo-mechanical processing of magnesium alloys and has significant effects on their microstructural and mechanical behaviours [1–4]. Recrystallization is usually defined as the formation of a new grain structure by the formation and migration of high angle grain boundaries driven by the stored energy of deformation [3,5]. However, strong texture and coarse grains are usually formed in recrystallized magnesium alloys, resulting in poor material ductility and strength [6,7]. The reason for forming strong texture is that basal planes are easily oriented towards the sheet surface in the rolling process [8], while the coarse grains generally form at high temperature due to recrystallization and oriented grain growth [9,10].

Although extensive studies on recrystallization in magnesium (Mg) alloys have been conducted [2,11], the effects of different twin types and twin-twin interactions on recrystallization of magnesium alloys remain unclear. Deformation twins are found to act as recrystallization nucleation sites, and twinning recrystallization can usually weaken texture [2,12–14]. According to the research on pure Mg single crystal under c-axis compression, it was found that the recrystallized grains inherited the orientation of their twin host and showed different orientations from the initial matrix [15]. However, tension twins (TTWs), the most common twin type in magnesium alloys, are generally difficult to recrystallize [14]. TTWs can be formed on any one of six (10T2) planes in grains, and would impinge on one another to form three types of twin-twin interactions, including (10T2)- (0T12), (10T2)- (0T12) and (10T2)- (0T12) [16]. Differing from a tension twin on its own, these twin-twin interactions would create dense and fine twin boundaries, resulting in twinning-induced grain refinement and blocking the dislocation slip [17]. High dislocation density could be introduced, contributing to the occurrence of recrystallization around twin-twin interactions. However, very few studies were conducted on the recrystallization in twin-twin interactions, and the twin-twin interactions induced texture evolution is still unclear.

The cryogenic rolling (cryo-rolling) process would be a novel way to generate abundant twins for the recrystallization, as twinning would be predominately formed at cryogenic temperature [18,19]. It is recently found that the cryogenic deformation is an effective way to generate extensive twinning in titanium [20–23], and enhance the mechanical properties and microstructures after the annealing in Al-Mg alloy [24] and stainless steel [25], due to attained finer grain size. Since magnesium has a similar hexagonal closed packed (HCP) structure, it is expected to form abundant twins at low temperature [26]. Recrystallization around the formed twins would enable a systematic investigation on the microstructure and texture evolution of twin recrystallization.

Besides twin recrystallization, recrystallization in magnesium alloys also occurs in deformation bands including the deformed areas near grain boundaries, as they contain intensive plastic deformation [1,3,10,27], but the recrystallization in these sites may not contribute to weakening texture in pure Mg or AZ31 Mg alloys [28]. It was found that the anisotropic grain boundary properties (i.e. low angle grain boundaries have lower energy and mobility compared to high angle grain boundaries) could lead to the texture strengthening during grain growth in AZ31 Mg alloys [10]. However, the texture and microstructure behaviours during the nucleation stage of recrystallization in deformation bands are still unclear and need further investigations.

In this study, the cryo-rolling and subsequent annealing approach are conducted to investigate the texture evolution during recrystallization in twins and twin-twin interactions. A room-temperature (RT)-rolling with the same subsequent annealing condition is also conducted to investigate the recrystallization mechanisms in deformation bands. The microstructure evolution in the annealing process is studied using the quasi-in-situ EBSD method (i.e. trace the microstructure change at the same region of interest).

2. Experimental programme

2.1. Material and experimental tests

The as-received material in this research is cast AZ31 alloys (Mg-Al-Zn alloys class) provided by Magnesium Elektron, and its chemical compositions are shown in Table 1. Fig. 1 shows the schematic processing of heat treatment (T4 temper), rolling at RT and LNT, and subsequent annealing. Note that the rolling-plates with dimensions of 100 mm × 35 mm × 10 mm were machined from the T4-temper plates using an electro-discharged machine (EDM).

The rolling tests were conducted on a DRM C130 powered rolling mill at a rolling speed of 4.9 m/min, at RT and LNT, respectively. In the LNT-rolling process, AZ31 samples were firstly immersed in liquid nitrogen for approximately 10 min until no obvious volatilization of liquid nitrogen. The same nominal thickness reduction for rolling tests was applied for all materials, and the final thickness reduction of these rolling tests at RT and LNT was measured as ~9%. After the rolling process, two samples were cut from a similar location of the RT-rolled and LNT-rolled samples, respectively. These samples were annealed at 300 °C for 5, 10, 70, 110 min to study the recrystallization nucleation. For brevity, the IPF images and pole figures for 5 and 110 min were shown in Supplementary Fig. S1.

2.2. Microstructure characterization

The sample surface preparation for EBSD analysis was conducted with progressive mechanical polishing using P4000 SiC grinding papers and OPS suspension. The EBSD characterization was made on the Rolling Direction (RD) × Normal Direction (ND) planes. For the annealed samples, the same region of interest at the corner of the surface was traced by EBSD during the annealing process. To capture the microstructure of rolled samples, a 2505 μm × 1887 μm EBSD map with 3 μm step size at ×50 magnification was obtained using a Bruker e’-FlashHR detector and Quantax Esprit 2.1 system. A 25 keV voltage was applied in a Hitachi SEM. For the annealed samples, the step size of EBSD scanning was set as 8 μm. The step size of 8 μm is sufficient to capture the essential microstructure information for these samples, while the finer step size of 3 μm was selected for the rolled samples in order to obtain the detailed information about twinning and deformation bands. The EBSD pattern indexing rate is about 90–95%, which meets the requirement to obtain high-quality images. The number of grains in the annealed samples is about 1000–1500 grains, which could ensure a statistical EBSD analysis. During the annealing process, these samples were repeatedly characterized and annealed from SEM to a preheated Lenton furnace. Note that water quenching was undertaken for these two samples to retain the annealing microstructure. Gentle OPS polishing was also conducted to remove the thin oxide formed at the sample surface.

The acquired EBSD data were analysed using the software of HKL CHANNEL 5. Different twin boundaries were detected and highlighted in terms of a specific misorientation axis and angles. The kernel average misorientation (KAM) values caused by geometrically necessary dislocations (GNDs) were calculated using the average misorientation value between the probed pixel and its surrounding eight pixels, and cations (GNDs) were calculated using the average misorientation (KAM) values caused by geometrically necessary dislocations (GNDs).

| Table 1 Chemical compositions of AZ31. |
|---------------------------------------|
| Mg | Fe | Mn | Zn | Ti | Al | Ca | Ni | Si | Cu |
|---|---|---|---|---|---|---|---|---|---|
| wt% | Bal | 0.004 | 0.32 | 0.95 | <0.3 | 3.1 | <0.005 | 0.0006 | 0.02 | <0.0005 |
the intragranular misorientation distribution within grains, i.e. low intragranular misorientation is expected in those newly recrystallized grains (low GNDs) [14]. The texture of the recrystallized grains in these samples was analysed using the pole figure, because the pole figure is a typical statistical method to represent the positions and intensities of specific crystallographic orientations of the grains and widely used to present the texture of magnesium alloys [2,31].

3. Experimental results

3.1. The microstructure of RT-rolled and LNT-rolled samples

Fig. 2 (a) and (c) show the Inverse Pole Figure (IPF) images of RT-rolled and LNT-rolled AZ31 samples, respectively. According to the band contrast (BC) image superimposed by various twin boundaries in Fig. 2 (b), abundant deformation bands were observed in the RT-rolled sample. Some twins including {1012} tension twins (TTWs) and {1011}-{1012} double twins were also observed. This is in good agreement with the results reported in cold rolled pure Mg and AZ31 Mg alloys [28].

Compared to the RT-rolled AZ31 sample, fewer deformation bands and more twin boundaries were observed in the LNT-rolled AZ31 sample, as shown in Fig. 2 (b) and (d). The main twin types in the LNT-rolled sample are {1012} TTWs and {1012}-(0112) twin-twin interactions. It is worth noting that abundant twin boundaries are interacted with each other to form twin-twin interactions. Compared to TTWs, the twin-twin interactions show finer structures. Different dislocation structures would form for subsequent recrystallization, so distinct recrystallization behaviours would be expected in TTWs and twin-twin interactions.

3.2. Microstructure evolution during annealing

In order to systematically study the microstructure evolution during the annealing process, the quasi-in-situ EBSD tests are conducted for both RT-rolled and LNT-rolled samples. Fig. 3 plots IPF images of the RT-rolled and LNT-rolled samples, and their corresponding samples in the annealing process at 300 °C for 10 and 70 min, respectively. The recrystallized grains are highlighted in a bright colour, and the unrecrystallized grains are not plotted. The corresponding (0001) pole figures from recrystallized grains are plotted in the subsets.

After the annealing process at 300 °C for 10 min in Fig. 3 (b), most new grains in the RT-rolled sample are nucleated around the deformation bands including the deformed areas near grain boundaries, and the texture of recrystallized grains in the annealed RT-rolled sample is strong and close to the centre of pole figure. However, in Fig. 3 (e), the recrystallized grains in the annealed LNT-rolled sample are located around (1012)-(0112) twin-twin interactions, resulting in much weaker and more scattered texture than that in the annealed RT-rolled sample.

In Fig. 3 (c) and (f), after the annealing process at 300 °C for 70 min, more new grains nucleate and the grain growth also occurs in these two samples. The deformation bands including the deformed areas near grain boundaries in the RT-rolled sample are almost consumed by the recrystallized grains, while the twin boundaries in the LNT-rolled sample, especially boundaries of twin-twin interactions, are consumed by the recrystallized grains. It is worth noting that the microstructure in the annealed LNT-rolled sample becomes more homogenious than that in the annealed RT-rolled sample. The texture of recrystallized grains in the annealed RT-rolled sample becomes stronger and concentrates in the centre of pole figure, resulting in a typical basal texture. However, the texture of recrystallized grains in the annealed LNT-rolled sample in Fig. 3 (f) becomes weaker and more scattered, and several separate texture peaks are situated away from the centre of basal texture. Therefore, recrystallization around twin boundaries, especially twin-twin interactions, can weaken texture and avoid forming strong basal texture in the annealing process of the LNT-rolled sample.

3.3. Texture evolution and its mechanisms during annealing

Four typical zones in RT-rolled and LNT-rolled samples are selected to investigate the texture behaviours during the annealing process, respectively, as shown in Fig. 4. The recrystallized grains are highlighted in a bright colour, and the unrecrystallized grains are plotted in dark colour. The corresponding (0001) pole figures of recrystallized grains in different zones are plotted in the subsets.

The main deformation structures in the RT-rolled sample are deformation bands, which provide the nucleation sites for recrystallization.
As shown in Fig. 4(a), many deformation bands are distributed in this zone. At 10 min, most grains are recrystallized around the deformation bands, the texture distribution of the recrystallized grains in this sample is relatively scattered (Fig. 4(b) and (g)). However, at 70 min, oriented grain growth occurs and basal grains shown as blue ones grow faster than non-basal grains, resulting in the concentration of the texture in the centre of the pole figure (Fig. 4(c) and (h)). This is in good agreement with the result reported in Ref [28], in which the strong basal texture is formed after hot rolling in AZ31.

As shown in Fig. 4(d), for another zone in the RT-rolled sample, some black deformation bands are located around grain boundaries, which generally result from high dislocation density in these areas. Recrystallization would tend to occur around these grain boundaries with high dislocation density. At 10 min, in Fig. 4(e), the recrystallized grains are mainly nucleated along grain boundaries, and some basal grains shown as green ones are larger than non-basal grains, which contribute to the strengthening of the central part in the pole figure. After 70 min, more basal grains grow, and the central part of the pole figure becomes stronger (Fig. 4(f) and (h)). This result indicates that the preferential basal grain growth around deformation bands near grain boundaries contributes to the basal texture strengthening.

Texture evolution analysis is also conducted for the LNT-rolled sample during the annealing process. As shown in Fig. 4(i), the main deformation structure of the LNT-rolled sample in this zone is \((10\overline{1}2)-(01\overline{1}2)\) twin-twin interactions. At 10 min, in Fig. 4(j), many new grains with various orientations are nucleated around the boundaries of \((10\overline{1}2)-(01\overline{1}2)\) twin-twin interactions, and these recrystallized grains represent different orientations from the matrix, which contribute to the formation of weak texture. At 70 min, more grains are generated around the twin boundaries, and show a homogeneous distribution and uniform grain growth, resulting in a scattered and weak texture (Fig. 4(k) and (p)).

Fig. 4(l) shows another typical zone in the LNT-rolled sample, \((10\overline{1}2)-(01\overline{1}2)\) twin-twin interactions and TTWs are both located in this grain. According to Fig. 4(l–n), the main recrystallized sites are the boundaries of \((10\overline{1}2)-(01\overline{1}2)\) twin-twin interactions, very few grains recrystallize around TTWs. The texture in this zone is scattered and weak, as shown in Fig. 4(n) and (p), because more non-basal grains with various orientations are nucleated and grow uniformly. Therefore, unlike texture strengthening in the annealed RT-rolled sample, the texture of the LNT-rolled sample becomes much weaker during the annealing process, because the recrystallization with randomized orientations and uniform grain growth occurs around the boundaries of \((10\overline{1}2)-(01\overline{1}2)\) twin-twin interactions.

### 3.4. Deformation structures in RT-rolled and LNT-rolled samples

Fig. 5 plots microstructural analysis of the four typical zones in RT-rolled and LNT-rolled samples to investigate their main deformation structures, as these deformation structures including deformation bands and twin-twin interactions could result in significant influences on recrystallization. In Fig. 5(a) and (b), the Zone A and Zone B in the RT-rolled sample are plotted to show TTWs and deformation bands, respectively. In Fig. 5(a), a grain with \((10\overline{1}2)\) TTWs is plotted in Zone A. Twin variant A1 shows a misorientation of ~86° from matrix M, according to its pole figure and misorientation distribution map. In Fig. 5(b), some deformation bands are distributed and interacted in the matrix M. Few \((10\overline{1}1)-(10\overline{1}2)\) DTWs, such as twin variant B1, are located in these deformation bands, the misorientation of these twin boundaries
is ~38°. These microstructural behaviours are in good agreement with the results in the research on cold-rolled Mg alloys [28].

Fig. 5 (c) and (d) plot the microstructure of the LNT-rolled sample in Zone C and D, and the main twin types in the LNT-rolled sample are TTWs and (1012)-(0112) twin-twin interactions. In Fig. 5 (c), two twin variants C1 and C2 consume the most parts of the matrix M and interact with each other to form (1012)-(0112) twin-twin interactions, which also contribute to the formation of fine structure. According to the pole figure, twin variant C1 and C2 are scattered and show different orientations from its matrix M. A high peak is observed near the misorientation of ~60° in the misorientation distribution map, indicating that abundant (1012)-(0112) twin-twin interactions are formed in this zone.

In Fig. 5 (d), the two TTW variants D1 and D2 are interacted to form (1012)-(0112) twin-twin interactions in matrix M. Similar to the twin interactions in Fig. 5 (c), these two variants are located away from the centre of pole figure and result in a scattered distribution, according to its pole figure. The two peaks with specific misorientations of ~86° and ~60° are both high, indicating that abundant boundaries of TTWs and (1012)-(0112) twin-twin interactions are presented in this zone.

3.5. Recrystallization in different deformation structures

The main deformation structure in the LNT-rolled sample is twinning including TTWs, (1012)-(0112) twin-twin interactions. Therefore, the static recrystallization sites in this sample are mainly located around these twin boundaries. As shown in Fig. 6 (a) and (b), few recrystallized grains are observed around the TTWs, which is in agreement with the result in Ref [14, 32]. It is worth noting that very few recrystallized grains are also observed around the TTWs in the annealed RT-rolled
Fig. 4. IPF images and their corresponding (0001) pole figures of recrystallized grains in the typical zones of RT-rolled and LNT-rolled samples during the annealing process at 300 °C for (a, d, i, l) 0, (b, e, j, m) 10, (c, f, k, n) 70 min, respectively. The (0001) pole figures of all recrystallized grains in Fig. 3 of RT-rolled and LNT-rolled samples during the annealing process at 300 °C for (g, o) 10 and (h, p) 70 min, respectively. The unit of the colour bar is mud.
Fig. 5. Typical zones in Fig. 2 (a, c) for the RT-rolled and LNT-rolled samples, respectively: (a) Zone A, (b) Zone B in the RT-rolled sample, and (c) Zone C, (d) Zone D in the LNT-rolled sample, where the first row presents the IPF images in these zones; the second row shows their corresponding pole figures, and the hexagonal prisms were superimposed in these pole figures to present their orientations; the third row gives their misorientation distribution maps.

Fig. 6. (a, b) IPF images of deformed and annealed grain A at 300 °C for 70 min, and (e, f) their corresponding KAM maps, respectively; (c, d) IPF images of deformed and annealed grain B at 300 °C for 70 min, and (g, h) their corresponding KAM maps, respectively.
sample, as shown in Fig. 2(b) and Fig. 3(c). Fig. 6(e) shows the kernel average misorientation (KAM) map, which can reflect the GND density distributions \([20,29,33]\). For example, the KAM value in these TTWs is low, indicating that its GND density is low and the stored strain energy is not sufficient to activate recrystallization in this area. In addition, in Fig. 6(e) and (f), some sites with high KAM value are located in the matrix of grains, but no recrystallized grains are observed in these sites. Similar results were also shown in the RT-rolled sample, according to Fig. 3 and Supplementary Fig. S2.

As shown in Fig. 6(c) and (d), it is also rare to observe the recrystallization in these TTWs, as the KAM value is low in these twins (Fig. 6(g)). However, some recrystallized grains are observed in the boundaries of twin-twin interactions which show high KAM value, according to Fig. 6(g) and (h). In Fig. 6(f) and (h), the KAM value in these recrystallized grains is low, confirming that few dislocations exist in newly formed grains. The average KAM value of recrystallized grains in the annealed grain A and grain B is 0.61° and 0.55°, which is much lower than the average KAM value of 1.28° and 1.36° in the deformed grain A and grain B, respectively.

Another typical deformation structure in the LNT-rolled sample is (1012)-(0112) twin-twin interactions, according to Fig. 2(d). As shown in Fig. 7(a) and (b), abundant recrystallized grains are formed around these boundaries of twin-twin interactions in grain C, resulting in significant grain refinement. In Fig. 7(e), the KAM value around the twin-twin interactions is high, which would provide adequate stored energy to initiate recrystallization. The average KAM value in the deformed grain C containing twin-twin interactions is 1.68° and higher than the average KAM value of 1.28° in the deformed grain A containing the single tension twin variant. Similar to twin-twin interactions, the high KAM value was also observed in the double twins [14], which contributed to the active recrystallization.

As shown in Fig. 7(c), both TTWs and twin-twin interactions are observed in grain D. The recrystallized grains are mainly located around the boundaries of twin-twin interactions rather than the boundaries of TTWs, which could be attributed to higher KAM values around the boundaries of twin-twin interactions than that around TTWs in Fig. 7(g).

According to Fig. 7(c) and (d), TTWs are profuse and show coarse structure, while twin-twin interactions are fine and dense. The dislocation structures in TTWs and twin-twin interactions are also different. Although there are few sites with high KAM values in TTWs, these sites are scattered and away from the twin boundaries. However, abundant sites with high KAM values are distributed around the twin-twin interactions, and close to the twin boundaries and interacting sites. After the annealing process, very few recrystallized grains are found around the twin boundaries of TTWs, while abundant recrystallized grains are formed near the boundaries of twin-twin interactions.

Recrystallization in the annealed RT-rolled sample mainly occurs around deformation bands including the deformed areas near grain boundaries (Fig. 3). Two typical zones are selected to gain an in-depth analysis of their recrystallization mechanisms. In Fig. 8(a) and (e), deformation bands are observed in Zone E, and the KAM value in these areas is high, resulting in active recrystallization and large recrystallized area in Fig. 8(b) and (f). Similarly, the recrystallized grains in Zone F are situated along grain boundaries, and the recrystallized area is small, as shown in Fig. 8(c) and (d). This would be attributed to the high KAM value around deformation bands including the areas near grain boundaries (Fig. 8(g) and (h)), which could promote the recrystallization in these sites. The average KAM value in deformed Zone E and Zone F is 1.22° and 1.04°, which is both higher than the average KAM value of 0.52° and 0.53° in recrystallized grains of these two zones after annealing, respectively.

As shown in Fig. 8(e) and (g), the sites with high KAM values are close to the deformation bands, especially grain boundaries, while the KAM value is low inside the grains. After the annealing process, the recrystallized grains are located near these boundaries, and very few recrystallized grains are observed inside the original grain. This result indicates that high KAM values near these boundaries are essential to activate recrystallization.

### 3.6. Grain size evolution during annealing

The grain size information of the RT-rolled and the LNT-rolled samples during the annealing process is summarized in Fig. 9. In Fig. 9(a), the average grain size decreases with a diminishing rate during the...
annealing process. At the initial stage, the recrystallization starts to occur, resulting in a fast decrease of the average grain size, and then the decrease of the average grain size approaches stable. The average grain size of the LNT-rolled sample is smaller than that of the RT-rolled sample in the annealing process, indicating that the grain refinement in recrystallization of the LNT-rolled sample is more effective than that of the RT-rolled sample.

The grain size distribution of the RT-rolled and LNT-rolled samples in the annealing process at 110 min is plotted in Fig. 9(b). Compared to the annealed RT-rolled sample, the grain size distribution of the annealed LNT-rolled sample, especially the small grains below the average grain size, is more homogenous, and more fine grains are observed. The fine and homogenous microstructure in the annealed LNT-rolled sample could be attributed to its active recrystallization and uniform grain growth around the twin boundaries and twin-twin interactions. In addition, compared with deformation bands in the RT-rolled sample, twin boundaries in the LNT-rolled sample are distributed more homogenously, resulting in more active twin recrystallization and the formation of more homogenous microstructure.

4. Discussion

4.1. Illustration of deformation and recrystallization mechanisms in the RT-rolled and LNT-rolled samples

Fig. 10 (a) and (b) plot the schematic of deformation mechanisms for AZ31 magnesium alloy in the RT-rolling and LNT-rolling processes, based on the experimental observations. As shown in Fig. 10 (a), initial samples show homogenous microstructure with low dislocation density. After the rolling process, the main deformation microstructures in
the RT-rolled sample are deformation bands and some twins including \(\{10\bar{1}2\}\) TTWs and \(\{10\bar{1}1\}\) DTWs, this is consistent with the results reported in cold rolled Mg alloys samples [28]. However, more twins including \(\{10\bar{1}2\}\)-\(\{01\bar{1}2\}\) twin-twin interactions are generated in the LNT-rolled sample. This is attributed to the effects of temperature on twinning and dislocation slips. In general, the critical resolved shear stress (CRSS) for twinning is independent on temperature while the CRSS for dislocation slips increases with decreasing temperature [22,34]. Therefore, the activity of dislocation slips would be limited at cryogenic temperature, resulting in more twins to accommodate the plastic strain. On the other hand, limited dislocation slips cannot effectively relieve the strain misfit between neighbour grains at cryogenic temperature, which would result in low deformation compatibility and high local stress near grain boundaries [23]. The high local stress near grain boundaries would promote the twin nucleation, and then these twins would grow and interact with each other to form abundant twin-twin interactions.

Fig. 10 (c–e) plots the schematic of microstructure evolution in the subsequent annealing process. In the initial stage, few static recrystallized (SRXed) grains are mainly nucleated around the deformation bands including the deformed areas near grain boundaries in the RT-rolled sample, and some SRXed grains are nucleated around the twin boundaries of the LNT-rolled sample, as shown in Fig. 10 (c). With increasing annealing time, in Fig. 10 (d), more SRXed grains in the RT-rolled sample are nucleated around the deformation bands, and oriented grain growth also occurs, resulting in strengthening texture. However, most recrystallized grains in the LNT-rolled sample are generated around the twin boundaries, especially \(\{10\bar{1}2\}\)-\(\{01\bar{1}2\}\) twin-twin interactions, which contribute to weakening texture. In addition, due to more homogenous distribution of twin boundaries than deformation bands, the microstructure of the LNT-rolled sample during the annealing process is more homogenous and finer than that of the RT-rolled sample. Finally, the nucleation and growth of SRXed grains become stable, as shown in Figs. 9 and 10 (e).

4.2. Recrystallization and texture evolution of the RT-rolled sample in the subsequent annealing process

The main deformation structures in the RT-rolled AZ31 Mg alloys are deformation bands, which would become the main recrystallized sites in cold-deformed AZ31 samples [35]. As shown in Fig. 8, abundant dislocations accumulate around the deformation bands including the deformed areas near grain boundaries, providing enough stored energy to induce the nucleation of recrystallization. Texture strengthening is observed during the recrystallization of the annealed RT-rolled AZ31 Mg alloys, it would be attributed to the effects of deformation bands including the deformed areas near grain boundaries. According to Fig. 4, the texture is scattered during the initial
nucleation stage, but it becomes stronger when the grain growth occurs. The strong basal texture in the annealed RT-rolled sample would be attributed to the oriented and inhomogeneous grain growth around deformation bands, i.e. basal grains grow much faster than non-basal grains near deformation bands or grain boundaries. This inhomogeneous and oriented grain growth could result from the anisotropic grain boundary energy and mobility in AZ31 alloys, i.e. low energy and mobility of low angle grain boundaries could be responsible for the texture strengthening [10]. In addition, unlike rare earth alloys [36–38], Al or Zn alloys in AZ31 alloys could not provide solute drag or particle pinning along deformation bands or grain boundaries, resulting in the preference for basal grain growth [28,39]. Although some twins including TTWs are observed in the RT-rolled sample in Fig. 3, it is rare to observe recrystallization around these twin boundaries, which make limited contributions to the texture [14,40]. Therefore, the main recrystallization sites in the RT-rolled sample are deformation bands, it is reasonable to form strong texture when recrystallization occurs in these sites.

4.3. Recrystallization and texture evolution of the LNT-rolled sample in the subsequent annealing process

For the LNT-rolled sample, abundant twins including TTWs and (10T2)-(01T2) twin-twin interactions are formed. It is rare to observe recrystallization around TTWs, because the KAM value around TTWs is low, which cannot provide sufficient stored energy to activate recrystallization, as shown in Fig. 6. The low KAM value around TTWs is attributed to the abundant slips which can transmit across TTW boundaries easily to relieve the local stress and dislocation pile-ups effectively [41]. Furthermore, TTWs are profuse and show coarse structure, as TTWs act as an effective sink of basal dislocations and could absorb the lattice dislocation in the twin boundary completely, which would contribute to twin propagation [42]. As a result, the twin shear localization and dislocation accumulation are limited [14]. However, According to Fig. 7, the dislocation slips are easily pinned and accumulated around these interacting twin boundaries of twin-twin interactions [43,44], resulting in high KAM value around these twin boundaries. Unlike profuse and coarse TTWs, twin-twin interactions are fine and dense, which could block the dislocation slips easily. As it is difficult for twins to transmit across the twin boundaries, twin propagation can be prevented by other twin boundaries [45], resulting in fine structure, instead, twin-twin boundaries form and contain boundary dislocations [44]. As a result, these abundant interacting twin boundaries with high KAM value would provide abundant sites for twin recrystallization [20]. In addition, twin-twin interactions would result in high local stress around the twin boundaries [45], and this high local stress would contribute to sufficient stored energy, which could be released during the recrystallization process.

Unlike texture strengthening in the RT-rolled sample during the annealing process, texture weakening is observed in the annealed LNT-rolled sample. This would result from the active recrystallization around twin boundaries, especially the boundaries of twin-twin interactions in the annealed LNT-rolled sample. In Fig. 4, as the annealing time increases, more recrystallized grains with randomized orientations are generated around twin-twin interactions, and the orientations of these grains are different from the matrix and away from the centre of pole figure. This could be attributed to the random orientations of twin variants in twin-twin interactions, and recrystallized grains would inherit the random orientations of these twin variants [15], resulting in weak texture. In addition, basal and non-basal grains in the twin recrystallization of the LNT-rolled sample grow more uniformly, compared to the oriented grain growth in deformation bands of the RT-rolled sample. Therefore, the random new orientations and uniform grain growth of recrystallized grains would result in weakening texture in the annealed LNT-rolled sample.

It is worth noting that the LNT-rolled sample shows more homogeneous microstructure with the finer average grain size than the RT-rolled sample during the annealing process. In the RT-rolled sample, deformation bands are the main recrystallization nucleation sites, but the density of the deformation bands is low in AZ31 rare-earth free Mg alloys [39], and they are not distributed homogeneously, especially inside grains, as shown in Fig. 2. In addition, the recrystallization and grain growth in deformation bands including the deformed areas near grain boundaries are anisotropic and inhomogeneous. Whereas, the density of twins, especially twin-twin interactions, is high in the LNT-rolled sample, and it is distributed homogeneously in each grain. More active recrystallization and homogeneous grain growth in these twin boundaries would contribute to forming more homogeneous microstructure with finer grains [46], and the homogeneous and fine microstructure could also contribute to weakening texture in the annealed LNT-rolled sample.

The weak texture and fine grains in the annealed LNT-rolled sample could result in the good material performance including high ductility and strength. As weak texture could generate grains in favourable orientations for dislocation slips, this would contribute to the high ductility [47]. Fine grains are also able to improve the ductility by activating abundant non-basal slips to accommodate the plastic deformation [48,49]. Besides, high strength was expected in the fine-grained annealed LNT-rolled sample, according to the Hall–Petch relation between grain size and strength [50].

5. Conclusions

In the present research, the RT-rolling and the LNT-rolling with subsequent annealing processes are studied with the aim to investigate the recrystallization behaviours in various deformation structures, namely, deformation bands and twinning. The grain orientation and KAM values during recrystallization are analysed using the quasi-in-situ EBSD method. The following conclusions are drawn:

1. Substantial twin boundaries, especially twin-twin interactions, in the LNT-rolled sample, provide abundant nucleation sites for recrystallization, which contribute to the formation of homogenous microstructure with weak texture and fine grains in the annealed LNT-rolled sample.

2. Texture weakening in the annealed LNT-rolled sample results from its uniform recrystallization and homogeneous grain growth around twin-twin interactions in which randomly oriented grains were generated.

3. The nucleation and growth of SRXed grains in the annealed RT-rolled sample mainly occur around the deformation bands including the deformed areas near grain boundaries, resulting in strengthening texture. Texture strengthening is attributed to the oriented and inhomogeneous grain growth around deformation bands, as basal grains grow much faster than non-basal grains.

4. Twin-twin interactions show the fine structure and high KAM values, which provide sufficient stored energy for active recrystallization, while TTWs are coarse and difficult to accumulate dislocations, resulting in fewer recrystallized grains.

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.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2020.108936.

References

[1] Z.R. Zeng, Y.M. Zhu, S.W. Xu, M.Z. Bian, C.H.J. Davies, N. Birbilis, J.F. Nie, Texture evolution during static recrystallization of cold-rolled magnesium alloys, Acta Mater. 105 (2016) 479–494.
[2] D. Guan, W.M. Rainforth, J. Gao, J. Sharp, B. Wynne, L. Ma, Individual effect of recrystallisation nucleation sites on texture weakening in a magnesium alloy: part 1- double twins, Acta Mater. 135 (2017) 14–24.
[3] F.J. Humphreys, M. Hatherly, Recrystallisation and Related Annealing Phenomena, Elsevier, 2012.
[4] K. Zhang, J.-H. Zheng, Z. Shao, C. Pruncu, M. Turski, C. Guerini, J. Jiang, Experimental investigation of the viscoplastic behaviours and microstructure evolutions of AZ31B and Elektron 717 Mg-alloys, Mater. Des. 184 (2019).
[5] R.D. Doherty, D.A. Hughes, F.J. Humphreys, J.J. Jonas, D.J. Jensen, M.E. Kassner, W.E. King, T.R. McNelley, H.J. McQueen, A.D. Rollett, Current issues in recrystallization: a review, Mater. Sci. Eng. A 238 (2) (1997) 219–274.
[6] A. Styczynski, C. Hartig, J. Bohlen, D. Letzig, Cold rolling textures in AZ31 wrought magnesium alloy, Scr. Mater. 50 (7) (2004) 943–947.
[7] H.Y. Wang, Z.P. Yu, L. Zhang, C.G. Liu, M. Zha, C. Wang, Q.C. Jiang, Achieving high strength and high ductility in magnesium alloy using hard-plate rolling (HPR) process, Sci. Rep. 5 (2015) 17100.
[8] F. Zarandi, S. Yue, Magnesium Sheet: Challenges and Opportunities, Magnesium Alloys-Design, Processing and Properties, IntTech, 2011.
[9] C. Bettles, M. Barnett, Advances in Wrought Magnesium Alloys: Fundamentals of Processing, Properties and Applications, Elsevier, 2012.
[10] J.J. Bhattacharyya, S.R. Agnew, S. Westlund, Texture enhancement during grain growth of magnesium alloy AZ31B, Acta Mater. 86 (2015) 80–94.
[11] I. Basu, T. Al-Sammam, Twin recrystallization mechanisms in magnesium–rare earth alloys, Acta Mater. 96 (2015) 111–132.
[12] X. Li, P. Yang, L.N. Wang, L. Meng, F. Cui, Orientational analysis of static recrystallization at compression twins in a magnesium alloy AZ31, Mater. Sci. Eng. A 517 (1–2) (2009) 160–169.
[13] M.R. Barnett, Twinning and the ductility of magnesium alloys Part II. “Contraction” twins, Mater. Sci. Eng. A 464 (1–2) (2007) 8–16.
[14] D. Guan, W.M. Rainforth, L. Ma, B. Wynne, J. Gao, Twin recrystallization mechanisms and exceptional contribution to texture evolution during annealing in a magnesium alloy, Acta Mater. 126 (2017) 132–144.
[15] T. Al-Sammam, K.D. Malodov, D.A. Malodov, G. Gottstein, S. Suwars, Softening and dynamic recrystallization in magnesium single crystals during c-axis compression, Acta Mater. 60 (2) (2012) 537–545.
[16] M.D. Nave, M.R. Barnett, Microstructures and textures of pure magnesium deformed in plane-strain compression, Scr. Mater. 51 (9) (2004) 881–885.
[17] H. El Kadiri, J. Kapil, A.L. Oppedal, L.G. Hector, S.R. Agnew, M. Cherkaoui, S.C. Vogel, M.D. Nave, M.R. Barnett, Microstructures and textures of pure magnesium deformed with increasing temperature in magnesium, Acta Mater. 63 (7) (2015) 725–730.
[18] K. Hantschke, J. Bohlen, J. Wendt, K.U. Kainer, S.B. Yi, D. Letzig, Effect of rare earth additions on microstructure and texture development of magnesium alloy sheets, Scr. Mater. 63 (3) (2015) 232–234.
[19] D.-X. Wei, Y. Koizumi, M. Nagasako, A. Chiba, L. Battaglia, M. Farnè, R. Žižek, Copper, choosing a suitable replacement for aluminium alloys, Mater. Sci. Eng. A 535 (2012) 11–18.
[20] D.-X. Wei, Y. Koizumi, M. Nagasako, A. Chiba, L. Battaglia, M. Farnè, R. Žižek, Copper, choosing a suitable replacement for aluminium alloys, Mater. Sci. Eng. A 535 (2012) 11–18.
[21] T. Al-Sammam, G. Gottstein, Dynamic recrystallization during high temperature deformation of magnesium, Mater. Sci. Eng. A 490 (1–2) (2008) 411–420.
[22] J. Jiang, T.B. Britton, A.J. Wilkinson, Evolution of dislocation density distributions in copper during tensile deformation, Acta Mater. 61 (19) (2013) 7227–7239.
[23] M. Niewczas, A. Kula, K. Noble, R.K. Mishra, Plasticity of Mg–Gd alloys between 4 K and 298 K, Philos. Mag. 96 (2) (2016) 134–165.
[24] C.W. Su, L. Lu, M.O. Lai, Recrystallization and grain growth of deformed magnesium alloy, Philos. Mag. 88 (8) (2008) 181–200.
[25] J.D. Robson, S.J. Haigh, B. Davis, D. Griffiths, Grain boundary segregation of rare-earth elements in magnesium alloys, Metall. Mater. Trans. A 47 (1) (2015) 522–530.
[26] I. Basu, K.G. Pradeep, C. Mielenz, L.A. Barrales-Mora, T. Al-Sammam, The role of atomic scale segregation in designing highly ductile magnesium alloys, Acta Mater. 116 (2016) 77–94.
[27] J.D. Robson, Effect of rare-earth additions on the texture of wrought magnesium alloys: the role of grain boundary segregation, Metall. Mater. Trans. A 45 (8) (2013) 3205–3212.
[28] D. Guan, W.M. Rainforth, J. Gao, L. Ma, B. Wynne, Individual effect of recrystallisation nucleation sites on texture weakening in a magnesium alloy: part 2- shear bands, Acta Mater. 145 (2015) 398–412.
[29] É. Martin, R.K. Mishra, J.J. Jonas, Effect of twinning on recrystallisation textures in deformed magnesium alloy AZ31, Philos. Mag. 91 (27) (2011) 3613–3626.
[30] K.D. Malodov, T. Al-Sammam, D.A. Malodov, Profuse slip transmission across twin boundaries in magnesium, Acta Mater. 124 (2017) 397–409.
[31] H. El Kadiri, C.D. Barrett, J. Wang, C.N. Tomé, Why are {1012} twins profuse in magnesium? Acta Mater. 85 (2015) 354–361.
[32] L. Jiang, J.J. Jonas, A.A. Luo, A.K. Sachdev, S. Godet, Influence of (10-12) extension twinning on the flow behavior of AZ31 mg alloy, Mater. Sci. Eng. A 445–446 (2007) 302–309.
[33] Q. Yu, J. Wang, Y. Jiang, R.J. McCabe, N. Li, C.N. Tomé, Twin–twin interactions in magnesium, Acta Mater. 77 (2014) 28–42.
[34] M. Gong, S. Xu, Y. Jiang, Y. Liu, J. Wang, Structural characteristics of (11012) non-cozone twin–twins interactions in magnesium, Acta Mater. 159 (2018) 65–76.
[35] S.Q. Zhu, H.G. Yan, X.Z. Liao, S.J. Moody, G. Sha, Y.Z. Wu, S.P. Ringer, Mechanisms for enhanced plasticity in magnesium alloys, Acta Mater. 82 (2015) 344–355.
[36] R.K. Mishra, A.K. Gupta, P.R. Rao, A.K. Sachdev, A.M. Kumar, A.A. Luo, Influence of c-axis texture and ductility of magnesium extrusions, Scr. Mater. 59 (5) (2008) 562–565.
[37] S. Sandilbes, Z. Pei, M. Frísk, L.F. Zhu, F. Wang, S. Zaefifer, D. Raabe, J. Neugebauer, Ductility improvement of Mg alloys by solid solution: Ab initio modeling, synthesis and mechanical properties, Acta Mater. 70 (2014) 92–104.
[38] B.-Y. Liu, F. Liu, L. Yang, X.-B. Zhai, L. Zhang, Y. Yang, B. Li, J. Li, E. Ma, J.-F. Nie, Z.-W. Shan, Large plasticity in magnesium mediated by pyramidal dislocations, Science 365 (6448) (2019) 73–75.
[39] Z.R. Zeng, Y.M. Zhu, R.L. Liu, S.W. Xu, C.H.J. Davies, J.F. Nie, N. Birbilis, Achieving exceptionally high strength in Mg 3Al 1Zn-0.3Mn extrusions via suppressing intergranular deformation, Acta Mater. 160 (2018) 97–108.