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Full Length Article

Quasi in-situ EBSD analysis of twinning-detwinning and slip behaviors in textured AZ31 magnesium alloy subjected to compressive-tensile loading

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Abstract

Twinning and detwinning behavior, together with slip behavior, are studied in a textured AZ31 magnesium alloy under compressive and tensile strains along the rolling direction (RD) after each interrupted mechanical test via quasi in-situ electron backscattered diffraction technique. The results show that twinning firstly takes place under the compressive strain along the RD. With the increasing compressive strain, $\{10\bar{1}2\}$ tensile twins firstly nucleate, then propagate, and finally thicken. While under a reversed tensile strain along the RD, detwinning occurs. No nucleation happens during detwinning. Thus, tensile twins can detwin at lower tensile strain, followed by thinning, shortening, and vanishing. Slips are also activated to accommodate the plastic deformation. In the matrix, prismatic slip can only dominate at relatively high strains. Otherwise, basal slip dominates. While in the twins, prismatic slip can activate at lower strains, which is ascribed to the texture reorientation.

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Keywords: Magnesium alloy; Twinning; Detwinning; Prismatic slip; quasi in-situ EBSD.

1. Introduction

Twinning and detwinning play an important role in plastic deformation for hcp structure materials [1, 2], as hexagonal materials cannot offer enough numbers of slip systems during deformation [3–6]. The most common and easily activated twinning mode in magnesium alloys is $\{10\bar{1}2\}$ tensile twinning at room temperature [7–10]. Tensile twinning can only be activated under c-axis extension [11–13]. Consequently, it should exhibit an asymmetric deformation behavior on a textured magnesium alloy, i.e., rolled or extruded material, with a lower yield strength under compression and a higher yield strength under tension [14–17].

Generally, detwinning of the $\{10\bar{1}2\}$ tensile twins can occur under two loading conditions: (1) reloading along the opposite

direction after the pre-loading; and (2) re-compression along the orthogonal direction after the pre-compression along the RD [18–22]. Therefore, twinning and detwinning usually can be observed when cyclic loadings are applied. With a limited number of fatigue cycles, the deformation was mainly dominated by twinning/detwinning mode [23, 24]. Furthermore, the fracture morphology was controlled by the repeated twinning-detwinning process [25]. The twinning/detwinning under different strain paths can lead to different mechanical responses [26, 27]. Pre-compressive deformation usually caused a drop in the tensile yield strength [28, 29]. Previous research reported that the activation of detwinning increased the work hardening rate [23, 30]. It was also found that not only twinning-slip interaction but twinning-detwinning activity could result in the Bauschinger effect, due to the stress relaxation during the detwinning process [31–33].

The main explanation of the twinning-detwinning behavior is that the process is controlled by the migration of twin boundaries (TBs) [34–36]. Compared to the twinning nucle-

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ation process, the propagation process needs much lower energy, i.e., $CRSS_{nucleation} > CRSS_{propagation}$ [37]. Moreover, the process of detwinning does not need nucleation [38, 39]. As a result, detwinning can take place under a stress even lower than the CRSS for twin growth, which is attributed to the internal stress in the grain matrix generated during twinning. It was reported that CRSS values for {1012} tensile twinning and detwinning in wrought ZK60A alloys are 15 MPa and 6 MPa, respectively [40].

Besides the twinning and detwinning mechanisms, there should be the activations of slips to accommodate the plastic deformation. The different activations of slips, twinning, and detwinning during the loading path change should be responsible for the asymmetric deformation behaviors [41, 42]. Several simulation results suggested that during the loading path change process, the activities of the slip systems changed both in the matrix and the twins due to the texture reorientations [43–45]. Hama et al. [44] suggested that during the reloading process under tension, there was a transfer from the detwinning-dominated mode to the slip-dominated mode as the tensile strain increased. Wang et al. [45] found increased activation of the prismatic slip system during the reversed shear loading under cyclic shear. However, there is a lack of experimental study on slip behaviors during the loading path change process.

To date, the twinning-detwinning phenomenon was observed to take place under cyclic loading or different strain paths. However, there is still a lack of study focusing on the twinning-detwinning behaviors, associated with the twinning/detwinning-slip interactions during the loading path change in rolled AZ31 magnesium alloy. The purpose of this paper is to understand the evolution of twinning/detwinning, as well as the dislocation slips, subjected to two cycles of compressive-tensile loadings along the RD in a textured AZ31 magnesium alloy sheet. The microstructure and texture evolutions were investigated by the quasi in-situ EBSD technique.

2. Experimental methods

A commercial rolled AZ31 magnesium alloy with a thickness of 20 mm was used in this study. The specimen with a dog-bone shape was cut from the as-received rolled AZ31 magnesium alloy using an electrical discharge machine, with a gage length of 8 mm (RD), a thickness of 10 mm (ND), and a width of 4 mm (TD). Here, RD, ND, and TD represent rolling direction, normal direction, and transverse direction, respectively. Mechanical tests were carried out in a CMT 5150 machine. Both tensile and compression tests were processed along the RD at room temperature with a strain rate of $10^{-3} s^{-1}$. Mechanical tests were interrupted at certain deformation strains to obtain the mechanical responses and evaluate the microstructure evolution of the AZ31 magnesium specimen.

Electron back-scattered diffraction (EBSD) measurements were performed in a JOEL-JEM 7800F field emission scanning electron microscope (SEM) equipped with Aztec and HKL Channel 5 software. The scanning step size is 0.7 μm .

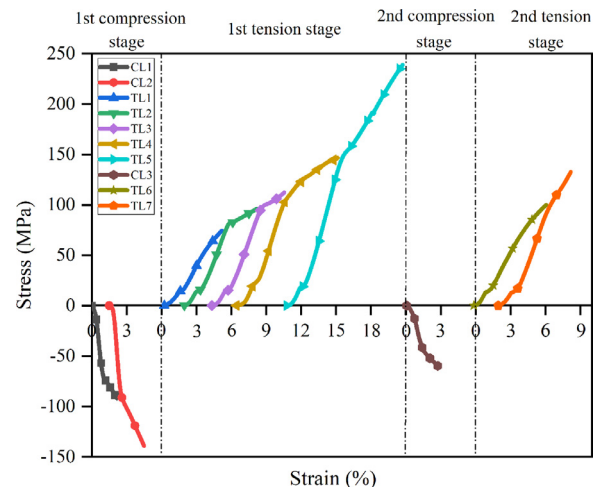


Fig. 1. Compressive and tensile stress-strain curves along the RD to different strain levels.

The deformed specimen for EBSD measurement was mechanically polished, followed by electrochemical polishing with AC2 solutions. EBSD measurements were carried out in the sample at a certain region parallel to the RD-TD plane after each deform stage.

3. Results and discussion

3.1. Mechanical behaviors

The successive deformation stress-strain curves are exhibited in Fig. 1, including compression and tension along the RD to different strain levels. The stress-strain curve above the x-axis in the diagram represents the tensile test, and the curve below the x-axis represents the compression test. The sample initially deformed under two successive compression tests (denoted as CL1 and CL2) with a pre-strain of 1.3% and 3.2%, respectively, in the first compression stage. It is observed that the compressive stress-strain curve contains a yield plateau, which indicates the tensile twinning activation during the compression test with the deformation load perpendicular to the c-axis [46, 47]. In the first tension stage, the pre-strained sample was then deformed under tensile loading to strains of 1.6%, 4.8%, 7.6%, 11%, and 14.6%, respectively. The tensile stress-strain curves (denoted as TL1, TL2, TL3, TL4, and TL5, respectively) show similar deformation behaviors that all exhibit a typical convex shape. An increment of the yield strength after each tensile deformation can be observed. This increment can result from the strain hardening effect during the deformation, where tangled dislocations prevent further dislocation movements [48]. After then, at the second compression stage, a compressive deformation with a strain of 0.9%, was conducted. A similar stress-strain curve (denoted as CL3) is plotted as those in the first compression stage, indicating the similar deformation mode activations in this deformation stage. Finally, in the second tension stage, two steps of the successive tension deformations (de-

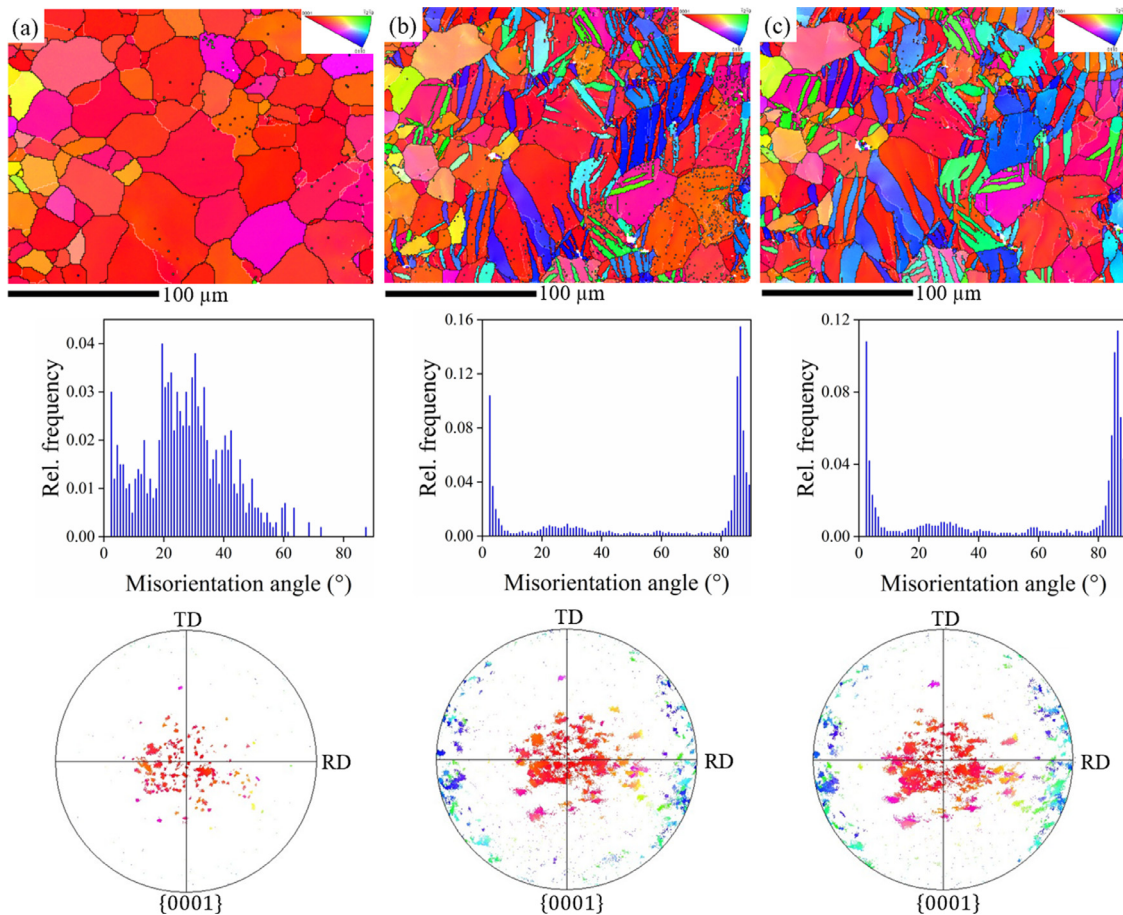


Fig. 2. EBSD inverse pole figure (IPF) maps, misorientation angle distributions, and $\{0001\}$ pole figures for the rolled AZ31 magnesium alloy sample: (a) the initial state; (b) pre-compressed to 1.3% along the RD; (c) pre-compressed to 3.2% along the RD.

noted as TL6 and TL7, respectively) were carried out. The tensile strains can be clarified as 1.6% and 4%, respectively.

3.2. Microstructure evaluation after each deform stage

Figure 2 presents the IPF maps, the misorientation angle profiles, and the $\{0001\}$ pole figures to illustrate the initial-state microstructure and the microstructure evolutions of the sample compressed to the pre-strain of 1.3% (CL1) and 3.2% (CL2), respectively. Figure 2a shows the initial microstructure and texture of the textured AZ31 magnesium alloy. The sample exhibits an equiaxed grain structure with the grain size ranging from ~ 5 to ~ 50 μm in the IPF map. No twins are detected in the initial microstructure. The boundary misorientation angle distribution ranges from 10 to 60° , which indicates that the predominant boundaries in the initial-state sample are high angle grain boundaries (HAGBs). In this work, HAGBs are defined as grain boundaries with a misorientation angle greater than 15° and low angle grain boundaries (LAGBs) contain a misorientation angle from 2 to 15° . The $\{0001\}$ pole figure in Fig. 2a reveals that the initial state sample has a typical basal texture that the c-axis of the unit cell is parallel to the ND. Figure 2b shows the microstructure of the sample at 1.3% pre-compressive strain along the RD.

Many twins newly appeared in the matrix grains as can be seen in the IPF map. After CL1, two peaks, ranging from 2 to 15° and 80 – 90° , generate in the misorientation angle distribution histogram. The peaks ranging from 2 to 15° result from the generation of the LAGBs during compression. While the peaks ranging from 80 to 90° are attributed to the activation of the twin lamellae, which is consistent with the result in the IPF map in Fig. 2b. Furthermore, it indicates that the active twins are of $\{10\bar{1}2\}$ type, as the misorientation angle tilts $\sim 86.3^\circ$. It is known that $\{10\bar{1}2\}$ tensile twins are easily activated under compressive strain perpendicular to or under tensile strain parallel to the c-axis of the HCP lattice [49]. An increase of the twinning component and a decrease of the basal component can be observed from the $\{0001\}$ pole figure in Fig. 2b. Figure 2c shows the microstructure of the sample at 3.2% compression strain. Twinning took place in nearly all matrix grains with a soft orientation. The existing $\{10\bar{1}2\}$ tensile twins continued propagating, thickening, and further coalesced at the CL2 stage, leading to the disappearance of some twin boundaries. Thus, the peaks of the grain boundary misorientation angle ranging from 80 to 90° , as can be seen, decrease slightly due to the coalescence of the existing twin lamellae. While the texture changed little under CL2, as shown in the pole figure in Fig. 2b.

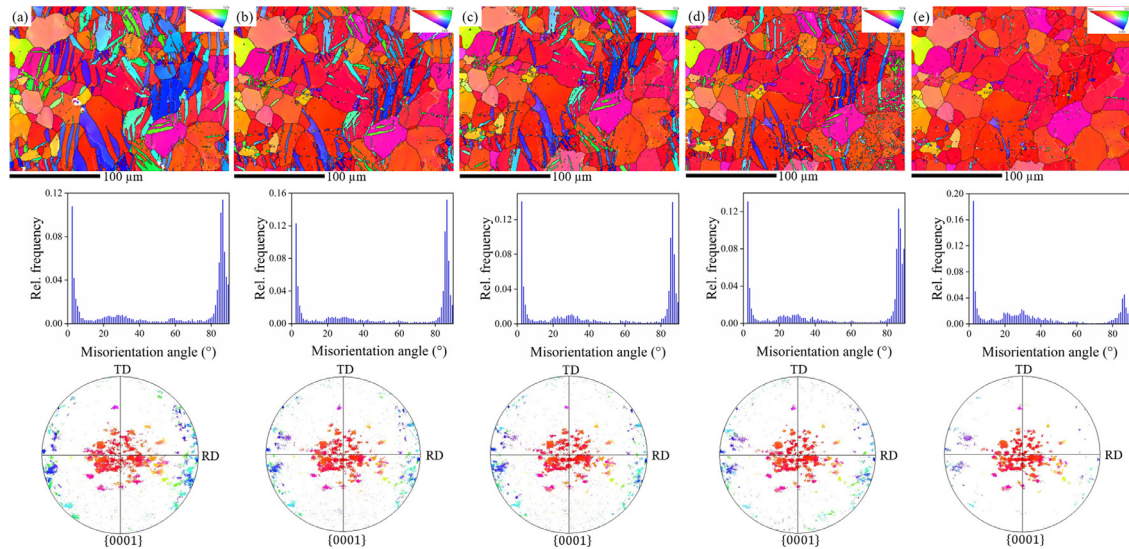


Fig. 3. EBSD inverse pole figure (IPF) maps, misorientation angle distributions, and $\{0001\}$ pole figures of the AZ31 magnesium alloy under reversed tension loading: (a) reversely tension to 1.6%; (b) reversely tension to 4.8%; (c) reversely tension to 7.6%; (d) reversely tension to 11%; (e) reversely tension to 14.6%.

Figure 3 exhibits the IPF maps, the misorientation angle distributions, and the $\{0001\}$ pole figures for the pre-strained sample deformed after successive reversed tensile loadings along the RD. The detwinning phenomenon is observed during the deformation process. In Fig. 3a, when a 1.6% tensile strain (TL1) was applied to the pre-compressed sample, no significant change in the microstructure can be observed. Moreover, when the applied strain increased to 4.8% (TL2), nearly all $\{10\bar{1}2\}$ tensile twins became thinner, as seen in the IPF map in Fig. 3b. Additionally, some extremely thin twin lamellae disappeared. In the misorientation angle distribution profile, the peaks ranging from 80 to 90° increase, since the coalesced twins started to disperse, and new twin boundaries were generated. As can be observed in the $\{0001\}$ pole figure (Fig. 3b) for the sample at TL2, the basal pole also tilts slightly back to the ND. In Fig. 3c and d, as the tensile strain increased from 7.6% (TL3) to 11% (TL4), twin lamellae continued to become thinner and shorter. Finally, only several twin lamellae vanished. The distributions of the grain boundary misorientation angle and the textures after TL3 and TL4 do not change too much. In Fig. 3e, when the reversed tensile strain was up to 14.6% (TL5), nearly all the $\{10\bar{1}2\}$ tensile twins vanished, but only several extremely thin twin lamellae remained. The remaining thin twin lamellae may result from the incomplete reversed tensile deformation. From the grain boundary misorientation angle and the textures after TL3 and TL4 do not change too much. In Fig. 3e, when the reversed tensile strain was up to 14.6% (TL5), nearly all the $\{10\bar{1}2\}$ tensile twins vanished, but only several extremely thin twin lamellae remained. The remaining thin twin lamellae may result from the incomplete reversed tensile deformation. From the grain boundary misorientation angle histogram, the peaks ranging from 80 to 90° decrease drastically, consistent with the microstructure observation that almost fully detwinning occurred in all matrix grains. While the peaks ranging from 2 to 15° increase. It is suggested that twin boundaries are replaced by LAGBs during detwinning [50]. As seen from the $\{0001\}$ pole figure in Fig. 3e, the basal pole tilts back to the ND. More specifically, the reversed tensile loading along RD renders c-axis re-aligning perpendicular to the ND, while the pre-formed tensile twins from compression disappear. As

reported in the previous study, the mechanism of detwinning can be considered as the reversed process of twinning [35, 51]. It is known that the twinning propagation is controlled by the movement of TBs [52]. TBs consist of coherent twin boundaries (CTBs), basal-prismatic (BPs), and prismatic-basal (PBs) facets. The evolution of twinning/detwinning then can be considered as the glide or climb of twinning dislocations along these interfaces, resulting in the migration of TBs. Consequently, the residual twins become thinner and shorter.

Figure 4 shows the IPF maps, the distribution of misorientation angle, and the $\{0001\}$ pole figures for the sample under a repeated process of compression and tension. In Fig. 4a, under re-compression with a 0.9% strain (CL3), a limited number of thin twin lamellae nucleated. While there is no significant growth of the existing tensile twins. In the misorientation angle distribution histogram, the consistent results can be observed that the peaks ranging from 80 to 90° increase slightly. It is also found that re-twinning at the CL3 stage takes place at the same locations as that in the pre-compression stage. A similar phenomenon of the second-generation twin was observed in pure Mg under cyclic loading via in-situ TEM [53]. It might be explained that twinning prefers to nucleate at specific sites. Figure 5b shows the detwinning phenomenon under the reversed 1.6% tensile strain (TL6). Due to the limited tensile strain, only several thin twins became narrower. No significant change can be observed neither from the grain boundary misorientation angle distribution nor from the $\{0001\}$ pole figure. As the tensile strain increased to 4% (TL7), most of the residual twin lamellae vanished, as can be observed from the IPF map in Fig. 4c. At the same time, in the misorientation angle distribution histogram, the peaks ranging from 80 to 90° decrease to nearly zero, while the fraction of the LAGBs increase. The $\{0001\}$ pole figure shows a basal texture with only a small fraction of the c-axis distributed away from the ND.

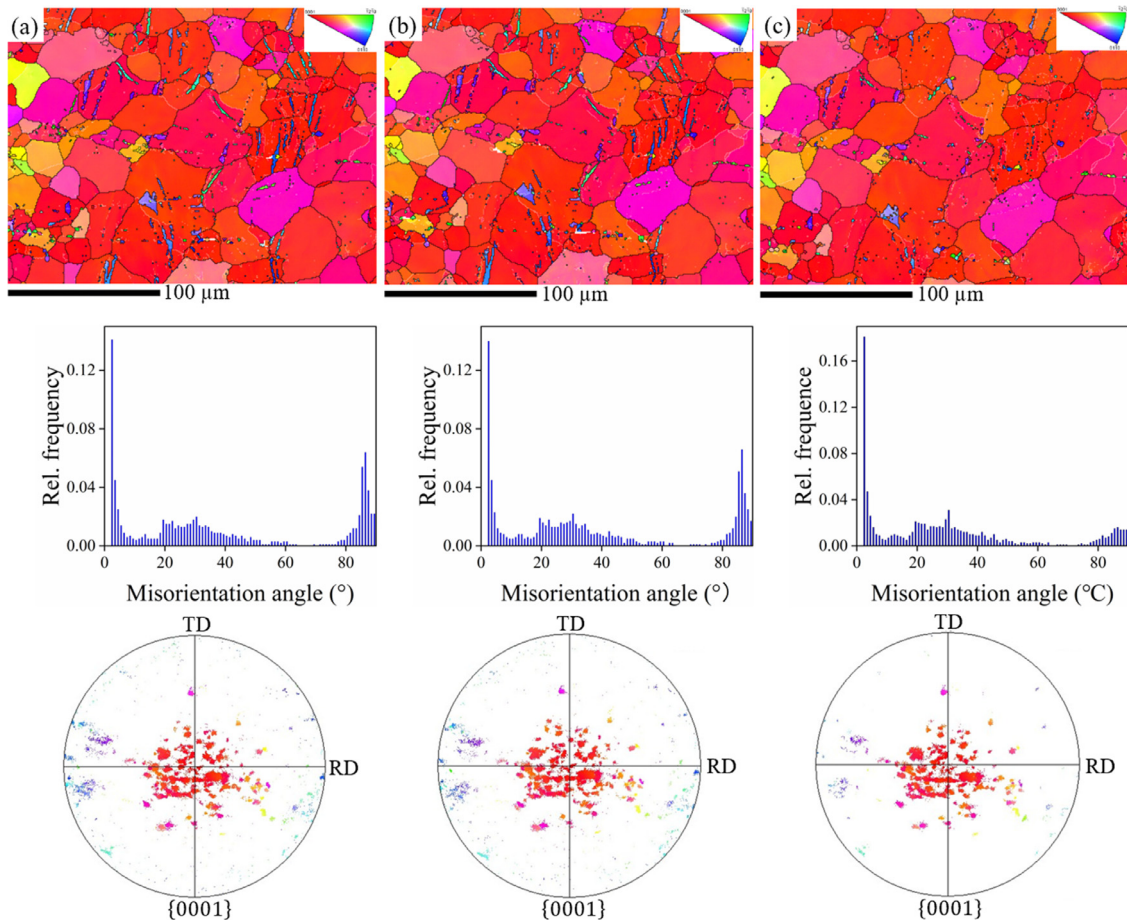


Fig. 4. EBSD inverse pole figure (IPF) maps, misorientation angle distributions, and {0001} pole figures of the AZ31 magnesium alloy under repeated compression and tension loading: (a) compression to 0.9%; (b) reversely tension to 1.6%; (c) reversely tension to 4%.

To study the interactions between twinning and slip, the in-grain misorientation axis (IGMA) analysis, associated with the Schmid factor (SF) analysis was applied. Theoretically, the activation of slips can cause lattice rotations, which in turn can be detected by electron backscattered diffraction. The detailed in-grain misorientation axis distribution method is introduced by Chun et al. [54, 55]. For each slip system in magnesium, they tilt around a certain axis (also named as Taylor axis). Namely, the $\{0002\}\langle 11\bar{2}0\rangle$ basal slip, the $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ prismatic slip, and the $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ pyramidal slip have the $\langle 0\bar{1}10\rangle$, $\langle 0001\rangle$, and $\langle 1\bar{1}00\rangle$ Taylor axis, respectively. Therefore, the identifications of the slip types both in the matrix and the twins can be studied via the IGMA method. The IGMA distributions during the twinning and detwinning process in four selected twinned/untwined grains are calculated. The misorientation angle concerned in this work is from 0.5° to 2° . And to obtain accurate results, only matrix and twins with more than 400 measured points are chosen. Figure 5 shows the IGMA distribution of four selected grains in rolled AZ31 magnesium alloy under repeated compression and tension loadings along the RD. The selected matrix grains and twins are marked as M1~M4 and T2~T4, respectively. The loading condition and the maximum/minimum intensity

are given at the top-left corner of each figure. Table 1 shows the SF calculations for grains illustrated in Fig. 5.

Figure 5a shows the IGMA distributions in an untwinned grain (marked as M1). The IGMA distributions are mainly concentrated around $\langle uv\bar{t}0\rangle$ directions. As mentioned above, the $\langle uv\bar{t}0\rangle$ -type rotation results from the activation of $\langle a\rangle$ type basal and $\langle c+a\rangle$ type pyramidal slips. Considering the high CRSS for $\langle c+a\rangle$ type pyramidal slip and the relatively low Schmid factor (SF) values shown in Table 1, the activation of $\langle c+a\rangle$ type dislocations is difficult. It can be assumed that in M1 during the repeated compression and tension loadings, the dominant slip mode is always the $\langle a\rangle$ type basal slip. Figure 5b exhibits the IGMA distributions in a twinned grain, where the matrix is marked as M2, and the twin is marked as T2. Similarly, during the pre-compression process (CL1 and CL2), the axis distributions in the matrix M2 are concentrated around $\langle uv\bar{t}0\rangle$ directions. Under the reversed tension along the RD, from TL1 to TL4, the IGMA distribution is focused around the $\langle uv\bar{t}0\rangle$ axis. As the tensile strain increased to 14.6% (shown in TL5) during the detwinning process, the maximum intensity of the IGMA in the matrix rotates around the $\langle 0001\rangle$ axis. It is proposed that $\langle a\rangle$ type prismatic slips are more favorable at the TL5 stage. Consistently, the SF value

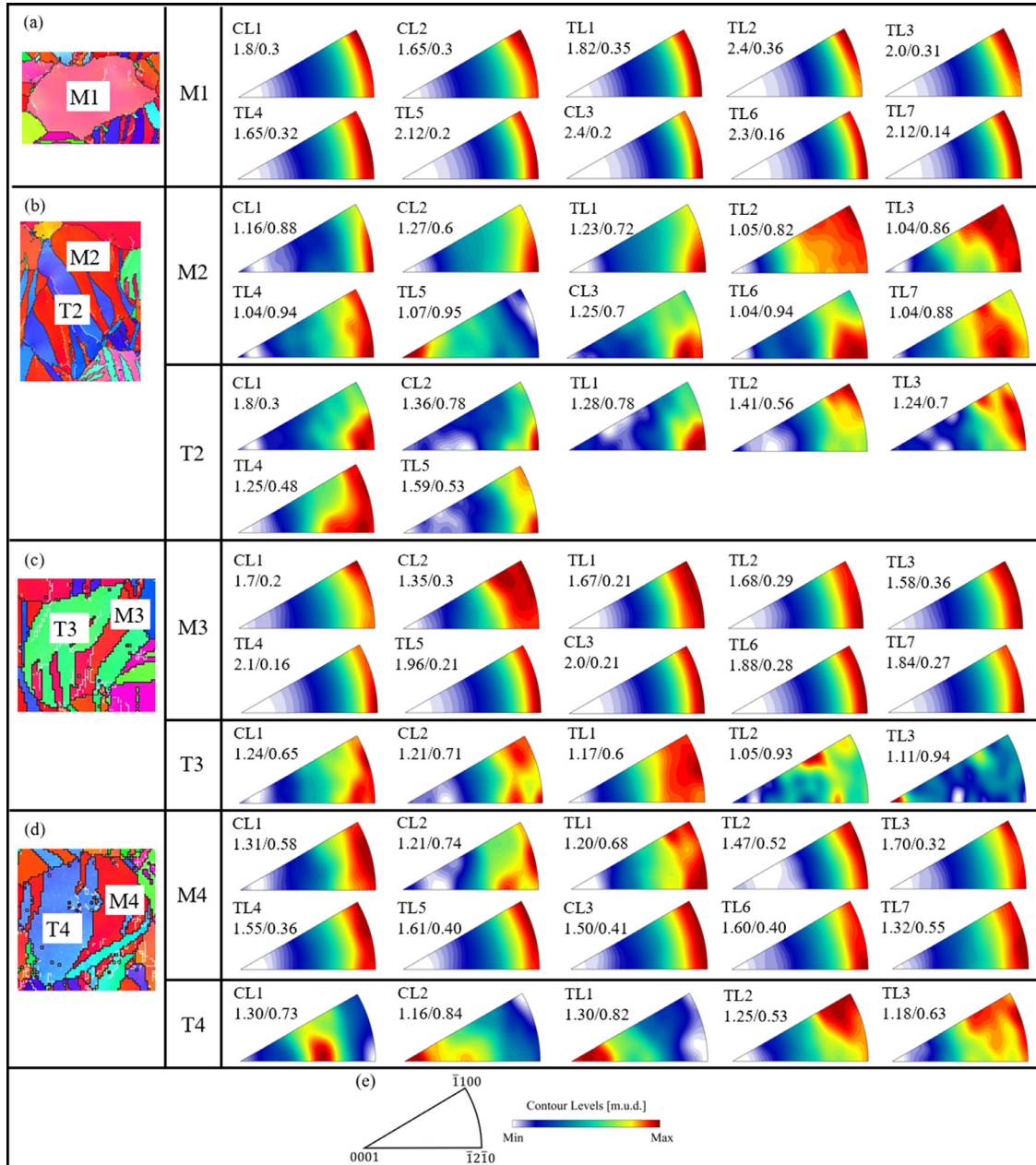


Fig. 5. The in-grain misorientation axis (IGMA) distributions of selected grains in AZ31 magnesium alloy under repeated compression and tension loadings. The maximum/minimum intensities of the IGMA distributions are given at the top of each figure.

Table 1
Schmid factors for grains illustrated in Fig. 5.

NO.	RD-Compression			RD-Tension		
	$\langle a \rangle$ basal	$\langle a \rangle$ prismatic	$\langle c + a \rangle$ pyramidal	$\langle a \rangle$ basal	$\langle a \rangle$ prismatic	$\langle c + a \rangle$ pyramidal
M1	0.461	0.268	0.226	0.461	0.268	0.22
M2	0.224	0.453	0.035	0.224	0.453	0.43
T2	0.338	0.063	0.48	0.338	0.063	-0.176
M3	0.217	0.467	0.007	0.217	0.307	0.486
T3	0.164	0.485	0	0.164	0.485	0.471
M4	0.287	0.451	0.051	0.287	0.451	0.463
T4	0.228	0.464	0.033	0.228	0.464	0.448

for prismatic slip in M2 is the highest, which means prismatic slip should be easily activated when the applied stress is high enough. Next, under the repeated relatively low compressive and tensile strains, the maximum intensities are around the $\langle uvw0 \rangle$ axis. The slip behaviors in the twin T2 are different from that in the matrix M2. During the whole loading process, including the twinning and the detwinning process, the lattice rotation distributions are concentrated around the $\langle uvw0 \rangle$ axis, indicating either $\langle a \rangle$ type basal or $\langle c + a \rangle$ type pyramidal slip dominates. According to the SF calculations in Table 1, during the twinning process, $\langle c + a \rangle$ type pyramidal slip possesses the highest SF value, while during the detwinning process, $\langle a \rangle$ type basal slip is more favorable. In Fig. 5c, the IGMA distributions in the matrix M3 and the twin T3 are presented. In M3, the maximum intensities of the IGMA are mostly around the $\langle uvw0 \rangle$ axis, which suggests that during the whole process, $\langle a \rangle$ type basal and $\langle c + a \rangle$ type pyramidal slips are favorable. At the CL2 stage, the IGMA distribution spread out towards the $\langle 0001 \rangle$ direction, indicating the activation of $\langle a \rangle$ type prismatic slip, which is consistent with the SF calculation shown in Table 1. During the twinning process, the IGMA distributions in the twin T3 are concentrated in the $\langle uvw0 \rangle$ direction. Considering the lowest CRSS and the second highest SF value (in Table 1), it is reasonable to say $\langle a \rangle$ type basal slips are dominant in T3. While during the detwinning process, as the tensile strain increased to 7.5%, the IGMA distribution concentrations tilt from the $\langle uvw0 \rangle$ direction to the $\langle 0001 \rangle$ direction. And the SF value for prismatic slip is the highest. Therefore, $\langle a \rangle$ type prismatic slips are activated. The twinning/detwinning operation, which re-orientates the grains with a hard orientation for slip, contributes to the prismatic slip activation. Figure 5d shows the IGMA distributions in the matrix M4 and the twin T4. Similarly, the maximum intensities of M4 are around the $\langle uvw0 \rangle$ axis during the whole deformation process. According to the SF results, both $\langle a \rangle$ type basal and $\langle c + a \rangle$ type pyramidal slips are favorable. The IGMA distribution in T4 at 1.3% pre-compressive strain is concentrated around the $\langle uvw0 \rangle$ direction. While as the pre-strain increased to 3.2%, the IGMA distribution rotates to the $\langle 0001 \rangle$ direction, suggesting that the $\langle a \rangle$ type prismatic slips dominate at this stage. At the TL1 stage ($\varepsilon=1.6\%$), prismatic slip is still preferred in T4. While as the reversed tensile strain increased to 4.8% and higher, the IGMA distribution concentration transfers to the $\langle uvw0 \rangle$ direction. Because at these stages, the twin T4 vanishes very quickly, we assume the twin size can also affect the slip activations [56].

4. Conclusions

In this work, the quasi in-situ electron backscattered diffraction technique was used to study the twinning and detwinning behaviors on a rolled AZ31 magnesium alloys under compression and tension along the RD after interrupted mechanical tests. The slip behavior analysis was conducted by the in-grain misorientation axis distribution method and Schmid factor method. Pre-compression tests revealed that $\{10\bar{1}2\}$ tensile twins nucleated at low compressive strains,

then further propagated and thickened with increasing strains. The microstructure evolution during reversed tensile tests along the RD exhibited the detwinning process. At a relatively low reversed tensile strain, the existing tensile twins started to become thinner. With the increasing reversed strain, twins continued thinning, shortening, and finally vanishing. Under repeated compressive and tensile loadings along the RD, re-twinning and re-detwinning occurred. It was also observed that re-twinning preferred to take place at the same location as in the first deformation stage. The IGMA analysis showed that the slip behaviors in the matrix and the twins were different. In the matrix, at low strains, $\langle a \rangle$ type basal slip was favorable. As the strain increase, it was hard to find $\langle a \rangle$ type prismatic slips that could dominate in the matrix. In the twins, since the texture reorientation took place, prismatic slips could be activated more easily, even at relatively low strains.

Declaration of Competing Interest

None.

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