Effects of Pre-Strain on the Evolution of Microstructure and Strain Hardening of Extruded Az31 Magnesium Alloy

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Pre-compression 3% and pre-stretch 3% subsequent annealing at 200ºC for 2h are conducted on AZ31 magnesium alloys, then inverse tensile and compressive deformation are carried out at room temperature, respectively. During inverse tension 3% deformation on 1st pre-compression samples, detwinning behavior happens; after 2nd pre-compression 3%, the volume fraction of {10-12} extension twins decreases comparing with 1st pre-compression. Due to the interaction of dislocation and induced twinning lamellas, strain hardening rate (θ) increases on 1st and 2nd pre-compression samples. {10-12} tensile twinning is restrained during inverse compressive deformation by pre-stretch process. Owing to the decreasing amount of twins, the texture strengthening in compressive deformation weakens. So the slop of stage III in strain hardening rate sustaining reduces after 1st pre-stretch 3% and 2nd pre-stretch 3% deformation during inverse compression deformation.

Keywords: *AZ31 magnesium alloy, microstructure, pre-strain, strain hardening*

1. Introduction

Due to advantages of high specific strength, low density and so on, magnesium alloys have been attracted by a large number of industrial sectors[1,2]. However, the basal plane only provides two independent slipping systems owing to its hexagonal close packed (HCP) structure which cannot fit Von-Misses criterion with five slipping systems[3,4]. At this time, twinning plays an important role to coordinate the deformation. Twinning is a polar deformation mechanism which is active only when the load is on specific direction of c-axis of Mg grains (compression perpendicular to c-axis or tension parallel to c-axis)[5,6]. Currently, the effects of pre-twinning induced by pre-strain on the properties of magnesium alloys are widely investigated.

Song et al.[7] cold pre-rolled on AZ31 magnesium plates along transverse direction for a small thickness reduction, the strength improved and yield asymmetry was weakened. Xu et al.[8] conducted multidirectional pre-compression along three directions of magnesium blocks, the tension-compression asymmetry reduced. Lou et al.[9] carried out dynamic plastic pre-deformation along the rolling direction at a smaller degree, the ductility improved largely. Ozaki et al.[10] indicated that pre-compression along extrusion direction to induce {10-12} extension twins, fatigue life of magnesium alloys got improved due to twinning-detwinning behavior during cycling deformation. Xin et al.[11] reported that maximum thickness reduction per pass of Mg alloys sheet during rolling at 300 °C increased after pre-extension twinning. While, Zhang et al.[12] pre-stretched on Mg alloy sheet and subsequent annealing, the formability enhanced greatly after pre-strain process. Besides, pre-strain plays also an important role on the strain hardening of magnesium alloys. Sarker et al.[13] indicated the strain hardening rate of AM30 magnesium alloys increased with a positive slope after pre-compression along extrusion direction to induce {10-12} extension twinning during stage B. Wu et al.[14] introduced the excess twinned grains through pre-compression along the rolling direction, the rapid strain hardening reduced after the pre-twinning process.

Thus, pre-strain can be regarded as an effective way to modify the microstructures and properties (strength[7], yield asymmetry[8], ductility[9], fatigue life[10], and formability[11,12] et al.) of Mg alloys. Besides, it has an important effect on the strain hardening during deformation[13,14]. About the strain hardening, pre-compression process is mainly focused; however, the effects of pre-stretch have rarely investigated. While it is much more significant to make clear how the pre-strain affects the strain hardening behavior, no matter pre-compression but also pre-stretch, especially during cycling deformation. Therefore, the effects of pre-compression and pre-stretch on the evolution of microstructure and strain hardening of Mg alloys have been systemic studied in this paper.

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2. Experimental Procedure

As-extruded AZ31 magnesium bars with a diameter of 16mm are used as the initial materials. And a diameter and length with 12mm×100mm billets are cut from the bars for pre-strain process. Firstly, the billets are 1st pre-compression by 3% along extrusion direction (ED). While subsequent annealing process is carried out at 200ºC for 2h to remove the dislocations. After that, reversed tension 3% is conducted. Then 2nd pre-compression is taken again for 3% degree. At last, the tensile samples are wire-cutting from as-received, 1st pre-compression and 2nd pre-compression billets to tensile fracture. The dimensions of tensile specimens are with nominal gage of 6mm×36mm. In order to avoid bending-buckling of Mg billet during pre-compression, a special die is used to hold the samples, as shown in section view Figure 1. Two separate holders are used to provide side stress during so as to prevent lateral strain.
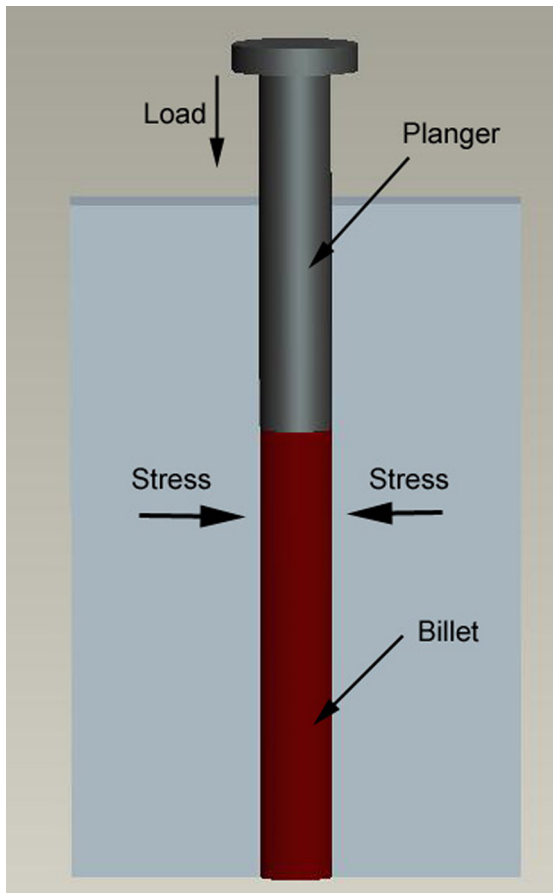

Figure 1. The section view of pre-compression die on Mg alloy billets

The same dimensions with 12mm×100mm billets are used for pre-stretch process. Firstly, the billets are 1st pre-stretch 3% along ED. After annealing at 200ºC for 2h, pre-compression 3% is taken out. Then 2nd pre-stretch 3% is conducted. At the end, the compressed specimens are taken from as-received, 1st pre-stretched and 2nd pre-stretched billets. The compressive specimens are with the height and diameter with 15mm×10mm. Tensile and compressive tests are carried out on a CMT6305-300KN electronic universal testing machine at room temperature. The strain rate was set at $10^{-3}s^{-1}$.

The microstructure is observed by optical microscopy and electron backscatter diffraction (EBSD). The EBSD test is conducted on ED-TD plane of the Mg specimens by a Zeiss EVO 50 SEM and the data were processed by INCA OXFORD crystal software.

3. Results and Discussions

Microstructure and (0002) pole figure of as-extruded AZ31 Mg alloys are shown in Figure 2. It can be seen that equiaxial grains distribute and there are no twins emerging in the microstructure. The grain size is about 15.5µm. Besides, (0002) pole figure of Mg billet expresses a typical extruded texture and basal plane of grains are parallel to extrusion direction.

True strain vs stress curves of various pre-strained specimens during inverse tension and compression are shown in Figure 3. It can be seen that as-extruded Mg expresses a typical tensile curve; however, it shows a hollow during yield stage after pre-compression 3% process. Sarker et al.[15] indicated that detwinning would be active during inverse tension on pre-compressed AM30 magnesium alloys. Detwinning behavior is a similar deformation mechanism which will result in tensile curves emerging depression. For pre-stretched Mg billets, all of the specimens express typical compressive curves. This indicates that pre-stretch process has no obvious effects on the deformation mechanism during compression.

Figure 4 shows the tensile/compressive yield strength (TYS/CYS) and peak strength (TPS/CPS) on various pre-strained specimens. TYS decreases from 132.3MPa to 97.6MPa after 1st pre-compression comparing with as-extruded specimens. However, it increases to 117.4MPa after 2nd pre-compression 3% process which should be related to the strain hardening behaviors. For TPS, it increases from 323.3MPa to 345.2MPa and 341.1MPa after 1st and 2nd pre-compression, respectively. The peak strength after 1st and 2nd pre-compression 3% is almost consistent. Figure 4 (b) shows the compressive yield (CYS) and peak strength (CPS) on various pre-stretched specimens. CYS increases on both 1st and 2nd pre-stretched Mg alloys. Comparing with as-extruded samples, it increases from 108.2MPa to 137.3MPa and 145.5MPa. CPS decreases from 349.2MPa (as-extrusion) to 337.8MPa (1st prs) and 342.4MPa (2nd prs), respectively. The changes are relative smaller which is similar with TPS on pre-compressed specimens. This phenomenon indicates pre-strain deformation just has an
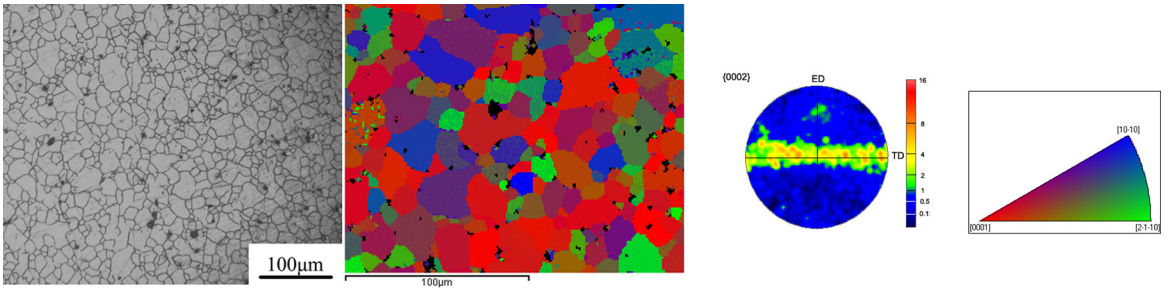
Figure 2. Microstructure and (0002) pole figure of as-extruded AZ31 magnesium alloy
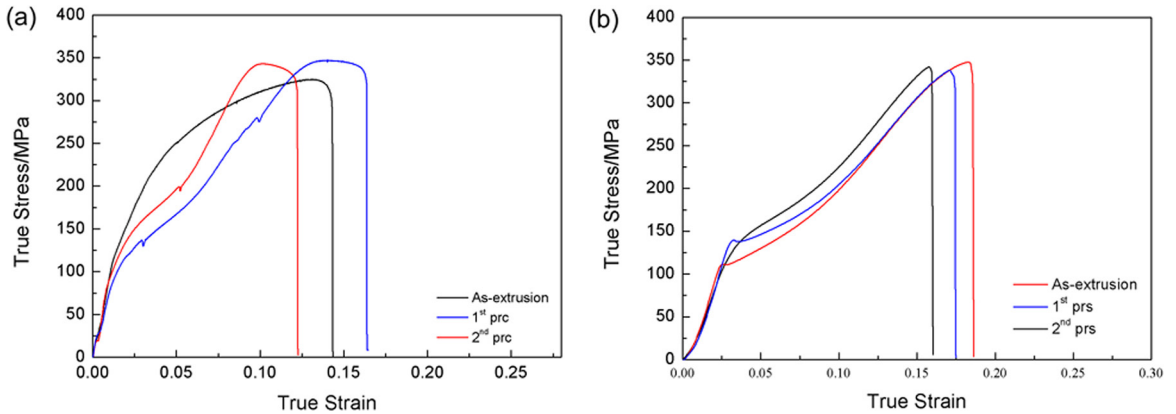

Figure 3. True strain vs stress curves of various pre-strain specimens: (a) tension, (b) compression
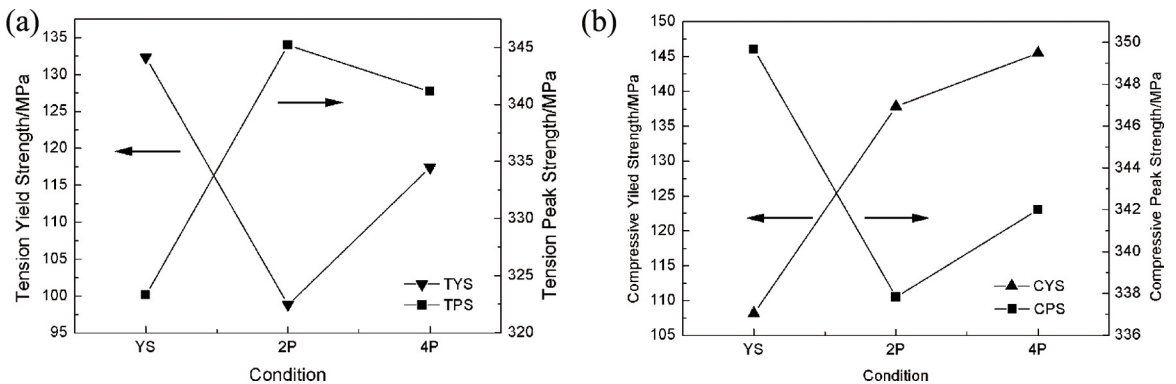

Figure 4. Tensile/compressive yield strength and peak strength on various pre-strained specimens

important effect on the yield stage. In order to make clear the evolution of mechanical properties, microstructures on various pre-strain deformations are observed.

Evolution of microstructures during 1st and 2nd pre-compression Mg specimens is shown in Figure 5. It can be seen that a large volume fraction of twins generates after pre-compression 3% and annealing at 200ºC for 3h. The type of the twins can be recognized as {10-12} tensile twins which leads to grains rotates from TD to ED about 86°. Thus, a ED // texture is generated, as shown in Figure 5 (e). The twins can be existed after annealing which is consistent with previous research. Xin et al.[16] reported that the dislocations could be removed and {10-12} twinning variants induced

by pre-compression along TD on magnesium alloys would be kept after annealing at 200ºC for 6h. However, the twins disappear after inverse tension 3%, as shown in Figure 5 (b). As well known, {10-12} twinning is a polar mechanism. It can be active only when the tensile load is parallel or compressive load is perpendicular to c-axis of grains[17,18]. However, Wu et al.[14] and Sarker et al.[15] reported that detwinning behaviors would happen when the inverse tensile load was carried out on compressed Mg samples. Due to detwinning, {10-12} tensile twins induced by pre-compression disappear after inverse tension 3%. So detwinning dominates the yield stage during inverse tensile deformation on 1st pre-compression specimens. While, the critical resolved shear stresses (CRSS)
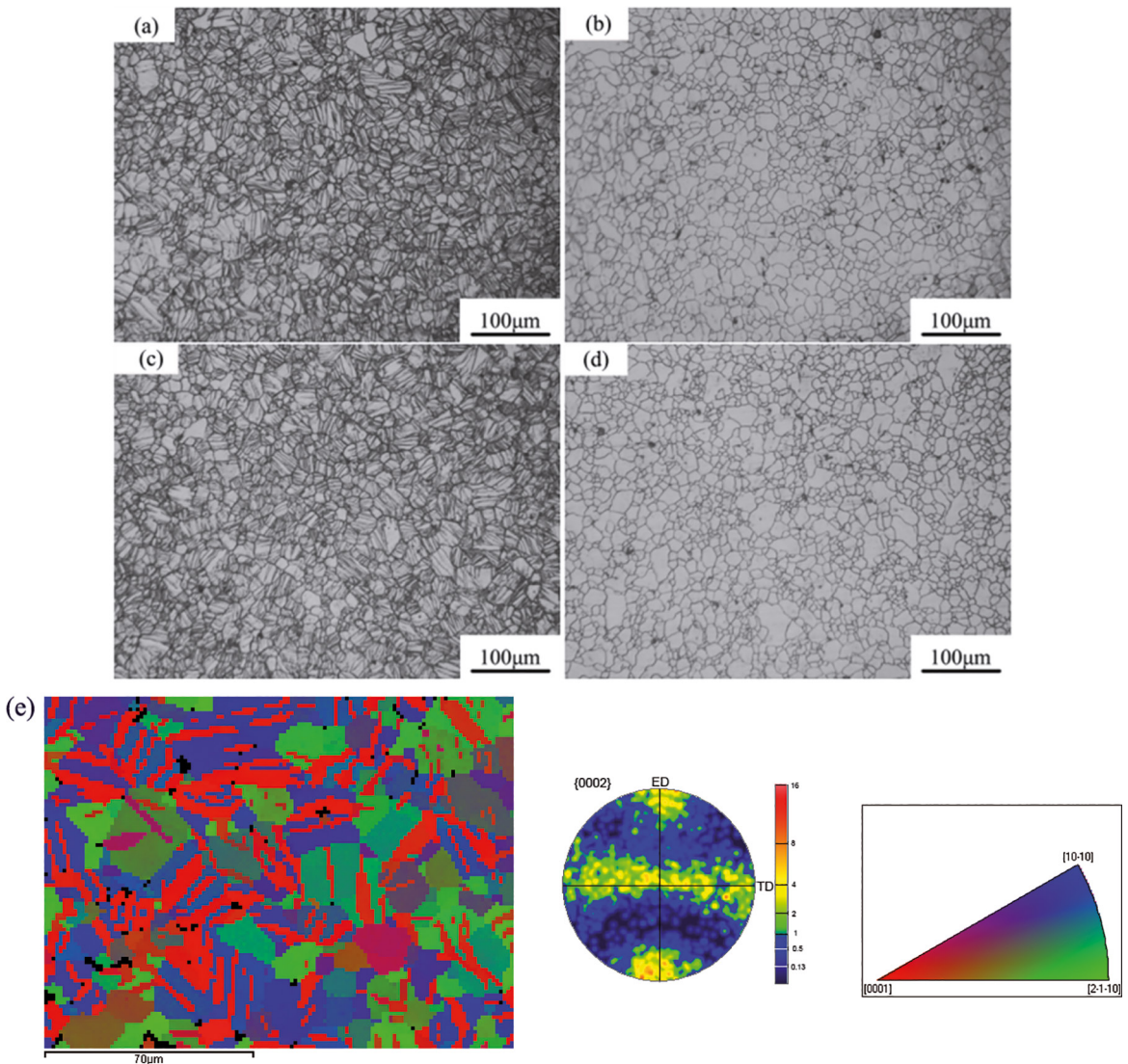
Figure 5. Evolution of microstructures during pre-compression on AZ31 Mg alloy billets: (a) 1st prc3%, (b) 1st inverse tension 3%, (c) 2nd prc 3%, (d) tensile to fracture, (e) EBSD maps of 1st prc3% specimens.

of detwinning is much smaller than prismatic slips[19]. Thus, the yield strength decreases than as-extruded Mg billets.

During 2nd pre-compression 3%, {10-12} tensile twins are generated again and detwinning also happens after inverse tension to fracture on 2nd pre-compressed Mg specimens. Equiaxed grains distribute in the microstructure. While the grain size of 1st inverse tension 3% and 2nd inverse tension to fracture specimens are 14.5μm and 15.1μm. This concludes that detwinning has little effect on evolution of grain size. Thus, cycling deformation like fatigue process of magnesium alloys can be regarded as a twinning-detwinning mechanism alternately dominates the deformation. Due to the rotation 86°of grains induced by {10-12} tensile twins, a softening effect are applied during inverse tensile deformation stage. So the fatigue life can be enhanced by pre-twinning. However, comparing microstructures of 1st with 2nd pre-compression 3% specimens, it seems that the volume fraction of twins decreases. Sheng et al.[20] reported that the c/a axial ratio would be decreased after tension for a smaller degree which led to the distance of tangential strain of {10-12} twins increasing. And the resistance of twins would increase as well. So the volume fraction of {10-12} tensile twins on inverse tension 3% Mg specimens decreases during 2nd pre-compression deformation. Due to the smaller amount of tensile twins, the softening effect is weakened at the same time. And the strain hardening will play a more important role than softening effect induced by twins as the cycling deformation continuing. From Figure 4 (a), TYS of 2nd pre-compression is larger than 1st prc samples, which may be related to this transformation. The strain hardening will be investigated in following parts.

Figure 6 shows the evolution of microstructures during pre-stretch deformation. After 1st pre-stretch 3% and annealing at 200℃ for 2h, the grains are elongated with a size of 14.8μm. It rotates from TD to ED about 10°and still keeps a basal fiber texture. When 1st compression 3% is conducted along ED, a large amount of {10-12} twins are generated. So tensile twinning is still the dominate deformation mechanism. Then detwinning behavior happens and {10-12} tensile twins disappear during 2nd pre-stretch 3%. The grain size is about 15.7μm which keeps almost consistent with as-extruded and 1st pre-stretch specimens. At last, 2nd inverse compression is conducted and twins emerge again. However, comparing with microstructure of 1st compressed specimens, the volume fraction of twins decreases obviously. This may be related to the drop of c/a axial ratio by pre-stretch. Due to the increasing distance of tangential strain of {10-12} tensile twins, the amount of twins decreases. So the

compressive yield strength improved and CYS is larger on 2nd than 1st pre-stretched Mg specimens. From the evolution of microstructures, it can state that the cycling deformation is dominated by twinning-detwinning behaviors. And the strain hardening and softening effect induced by twins play a competitive role during the deformation alternately. At first, the softening effects play an important role while the strain hardening plays much more important role as the deformation continuing. Based on previous analysis, it can be concluded that pre-compression does benefits on the cycling deformation than pre-stretch process due to softening effects induced by twins at first stage.

Figure 7 (a) indicates strain hardening rate ($\theta=d\sigma/d\varepsilon$) vs flow stress ($\sigma-\sigma_{0.2}$) of various pre-compressed specimens during inverse tension test. It can be seen hardening rate (θ) decreases linearly with stress on as-extruded Mg specimens. However, θ keeps the same trend at initial stage on 1st and
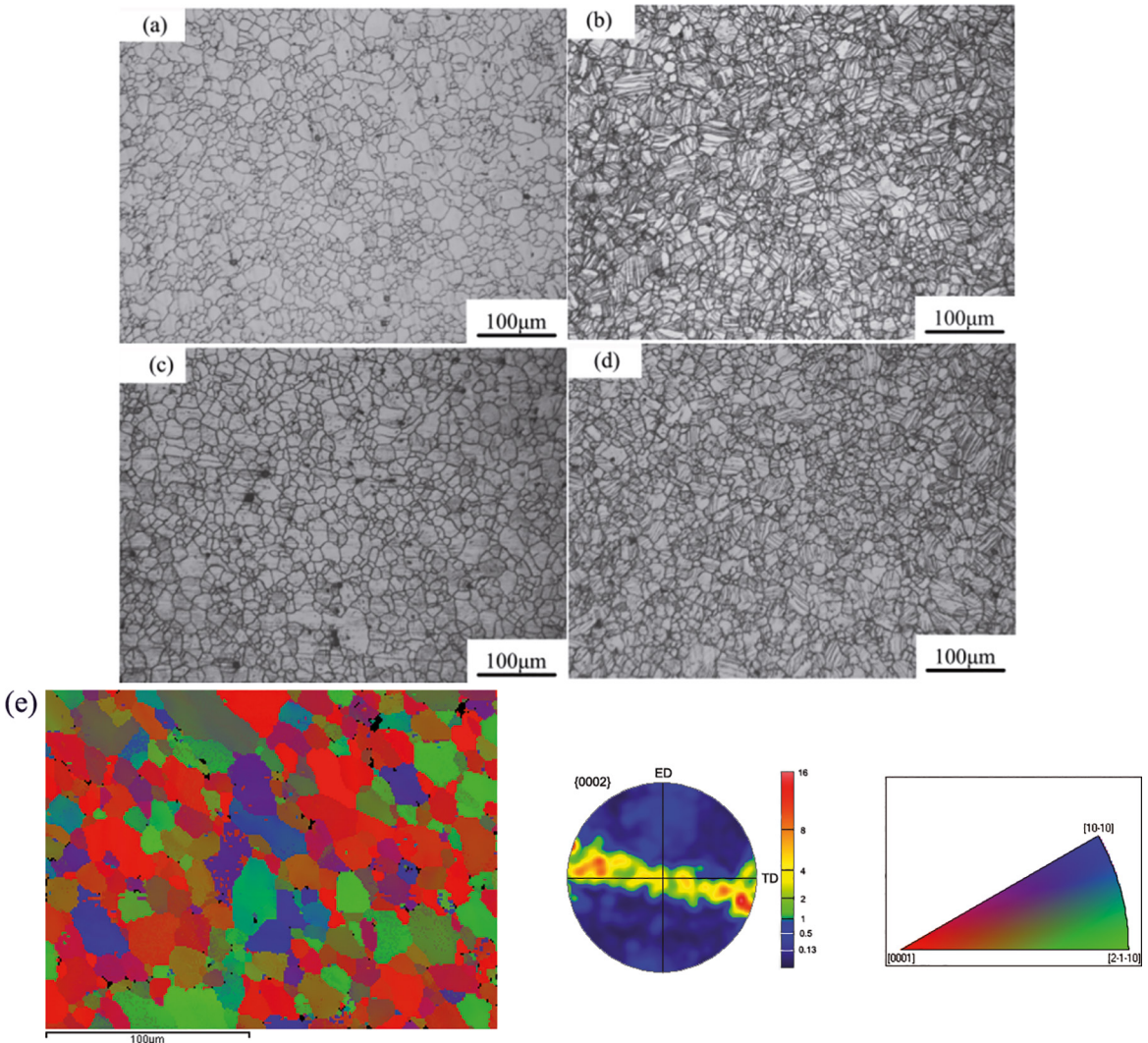

Figure 6. Evolution of microstructures during pre-stretch on AZ31 Mg alloy billets: (a) 1st prs3%, (b) 1st inverse compression 3%, (c) 2nd prs 3%, (d) compression to fracture, (e) EBSD maps of 1st prcs% specimens.
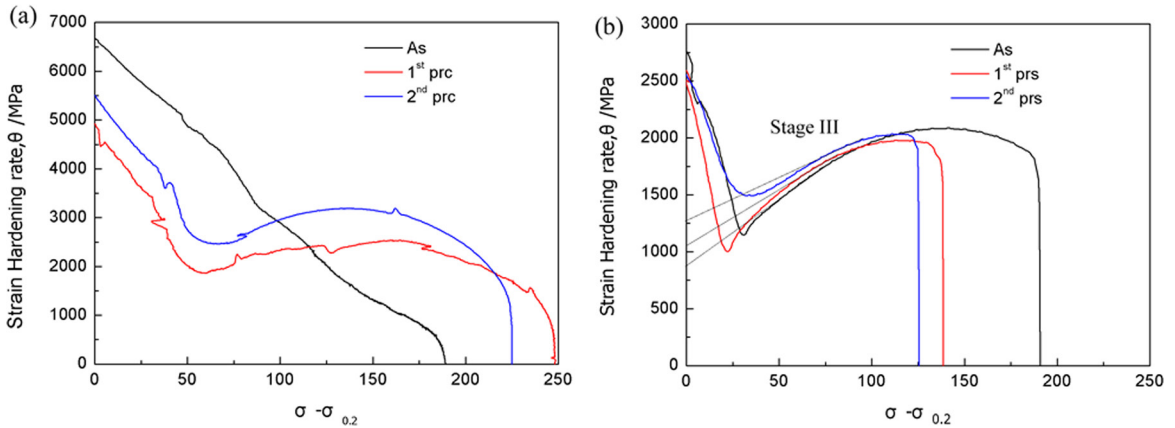
Figure 7. The strain hardening rate of various pre-strained Mg specimens: (a) tension, (b) compression.

2nd prc samples then increases as the stress increasing. This means an obvious strain hardening happens. After 1st pre-compression 3% and annealing process, the twinning variants are kept in the microstructure. Besides, during inverse tension test, the dislocation slips will be active as the strain levels increasing. Thus, the reserved twinning lamellas will become barriers to prevent dislocation movement[21]. The interaction between twins and dislocation slips leads to the increase strain hardening at this stage. As the deformation continues, detwinning behavior happens. Detwinning is a process of the thickness and volume fraction of {10-12} tensile twins reducing. So the volume fraction of {10-12} twins decreases during inverse tensile deformation. The transformation can be observed in Figure 5 (b). And due to the decreasing amount of twinning lamellas, the strain hardening rate decreases at the same time in last stage. And θ-value on 2nd prc specimens is always higher than 1st prc Mg specimens. It is mainly because the fraction of {10-12} twins during 2nd pre-compression process reduces and the induced orientation softening effects are weakened, as shown in Figure 5(c). The hardening caused by dislocation contributes more functions during tension deformation which led to the higher θ.

For inverse compression deformation, the hardening rate of stage III increases linearly with stress[22] and the slop of stage III decreases after 1st prs and 2nd prs process, as shown in Figure 7 (b). The decreased slop indicates that pre-stretch has significant impact on the slop of stage III. From Figure 6 (b) and (d), it can be seen that {10-12} tensile twins are the dominate deformation mechanism during the inverse compression yield stage. It has been reported that twinning does not only introduce additional barriers to dislocation glide, but also has a grain reorientation effect. Brown et al.[23] suggested that the initial {10-12} twins rotated grains to ED about 86°which was a hard orientation during compression deformation. The hard orientation causes texture strengthening as well. So it can be concluded that texture strengthening is the major

factor affecting the strain hardening of stage III. Besides, twinning lamellas are providing additional barriers to the accumulation of dislocations which enhances the strength effects. So as decreasing volume fraction of {10-12} tensile twins, the hardening will be weakened and the slop decreases. In previous research, pre-stretch will drop c/a axis ratio and the activation of {10-12} twinning is restrained[24]. So the volume fraction of extension twins decreases after 1st prs 3% deformation and the slop stage III decreases comparing with as-extruded samples. After 2nd prs deformation, the amount of twins reduces persistent and the slop of stage III decreases more. On the other hand, the dislocation density is also one important role on the strain hardening. However, song et al.[7] indicated that the accumulation of dislocations and dynamic recovery play minor roles on the strain hardening. So the weakening texture hardening induced by reduced twinning amount is responsible for the lower slop of stage III in 2nd prs Mg specimens.

4. Conclusions

In order to investigate effect of pre-strain on the evolution of microstructure and strain hardening of magnesium alloys, pre-compression/pre-stretch and subsequent annealing are applied on AZ31 magnesium billets. Detwinning behavior happens during inverse tension on pre-compressed samples. After 2nd pre-compression, the volume fraction of extension twins decreases. Due to the interaction of dislocation slips with twinning lamellas, the strain hardening rate (θ) increases. The orientation softening effects weakens as the amount of {10-12} twins decreases which lead to strain hardening induced by dislocation plays more impact. Thus, the strain hardening rate is higher in 2nd than 1st pre-compressed samples. However, extension twinning is sustained by pre-stretch process. The texture strengthening caused by {10-12}extension twins weakens as the volume fraction of twins, so the slop of stage III in strain hardening rate reduces in 1st and 2nd pre-stretch Mg alloys.

5. Acknowledgements

6. References

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