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Microstructure and mechanical properties of cryorolled AZ31 magnesium alloy sheets with different initial textures

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Abstract: AZ31 magnesium alloy sheets with different strong textures were cryorolled at the liquid-nitrogen temperature to the strain of 4% and 8%. The microstructure and texture of the rolled sheets were investigated via scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and X-ray diffraction (XRD). The mechanical properties of the sheets were tested through in-plane uniaxial tensile tests at ambient temperature. The tensile stress was exerted in the rolling direction (RD) and transverse directions (TD). The microstructural and textural evolutions of the alloy during cryorolling were investigated. Due to active twining during rolling, the initial texture significantly influenced the microstructural and textural evolutions of the rolled sheets. A $\{10\bar{1}2\}$ extension twin was found as the dominated twin-type in the cryorolled samples. After cryogenic rolling, the ductility of the samples decreased while the strength increased. Twinning also played an important role in explaining the mechanical differences between the rolled samples with different initial textures. The samples were significantly strengthened by the high stored energy accumulated from cryorolling.

Keywords: magnesium alloy; cryorolling; microstructure; mechanical properties; twinning

1. Introduction

There is an increasing interest in the use of wrought magnesium alloys as alternative lightweight materials for transportation applications. Several efforts have focused on improving the mechanical properties of the alloys. A key issue in improving the mechanical properties of the alloy involves controlling the microstructure and texture of the alloys [1–21]. Deformation mechanisms such as dislocation slip and twinning for the wrought alloys strongly affect microstructural and textural evolutions [16–20]. With respect to hexagonal closed-packed (HCP) metals, dislocation activity is always limited to *a*-slips [22], and hence twinning is very important. Twinning can accommodate the strain in *c* direction. Especially at a low deformation temperature, twinning plays a critical role during deformation when the dislocation slips are suppressed. Several types of twins are possible in wrought magnesium alloys. The most common twin is the $\{10\bar{1}2\}$ extension twin. Researchers investigating cold rolled magnesium alloys have also reported the $\{10\bar{1}1\}$ contraction twin and $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twin [21,23–24]. Twining can affect the microstructure of the deformed alloys by separating mother grains and influencing the texture by rotating the crystal orientation of the mother grain. Various types of twinning are possible in deformed magnesium alloys. The type of twinning depends on the crystal orientation of the mother grain. The extension along the *c*-axes of grains results in $\{10\bar{1}2\}$ extension twinning, while the contraction along the *c*-axes leads to contraction twinning $\{10\bar{1}1\}$ [22]. $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twins are usually found in the cold rolled sheets [21,23–24]. The dependence between the crystal orientation and twinning makes the initial texture important for microstructural and textural evolutions of the magnesium alloys.

A number of investigations were conducted on the microstructure, texture, mechanical properties, and deformation mechanism of the alloys. Extant research focused on the deformation mechanisms at various deformation temperatures [22,24–26]. Previous studies also discussed the effect

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of texture [19–20,27–30]. However, very few studies have focused on the effects of cryogenic temperature and on the deformation at a relatively high strain rate such as rolling. In this study a magnesium alloy AZ31 sheet was rolled at the temperature of liquid nitrogen (77 K) to different strains. The microstructural and textural evolutions of the magnesium alloy AZ31 during cryogenic rolling were investigated. The mechanical properties of the cryorolled sheets were examined. Additionally, the textures of the initial sheets were different. Thus, the influence of the initial texture was also discussed.

2. Experimental

The as-supplied material was a commercially available hot rolled AZ31 sheet (thickness 6 mm). The as-supplied material was annealed at 300°C for 40 min in an air furnace and then air cooled. Initial sheets were cut as shown in Fig. 1 to obtain different initial textures. Prior to rolling the sheets were placed in a vessel filled with liquid nitrogen for 30 min in order to achieve a uniform temperature. Initial_A and Initial_B were the initial samples with different textures. Then, the sheets were transferred to a rolling mill, rolled immediately to the reduction of 4% (samples CRA and CRB), and then rolled to the final reduction of 8% (samples CRA_f and CRB_f). Liquid nitrogen cooling followed this. Rolling conditions were chosen to ensure homogeneous deformation with a roll gap ratio $l/d \approx 1$ (l is the arc contact length; d is the average sheet thickness before and after rolling). The rolling strain rate was approximately 3 s^{-1}.

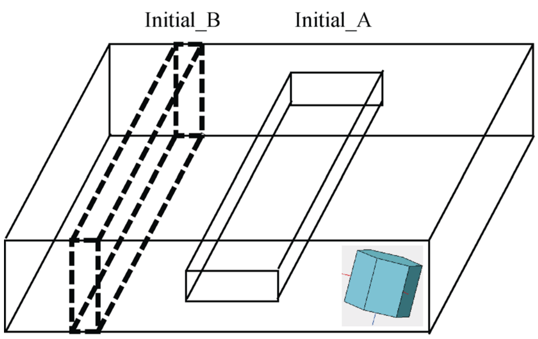

Fig. 1. Illustration of the two types of machined samples for rolling.

All the samples for the microstructural investigation were machined from the center part of the sheets. The surface of the transverse section was inspected in all the cases. Microstructural observations were carried out using a Zeiss supra 55 scanning electron microscope equipped with electron back-scatter diffraction (EBSD) detector. After standard metallographic polishing, the samples were electrolytically polished for 60 s at 20 V at room temperature using Struers ACII polishing solution, and then etched immediately in an acetic picral solution (5 g picric acid + 70 mL ethanol + 10 mL water) for 7 s. Samples were grounded to the mid-rolling plane. The texture analysis was conducted by a Siemens D5000 X-ray diffractometer with Cu K_α radiation at 40 kV. In-plane uni-axial tensile tests were carried out using a universal material testing machine with an electronic extensometer at room temperature at a constant strain rate of 10^{-3} s^{-1}. According to the ASTM standard E8, quarter-sized dog-bone-shaped specimens were cut from the center layer of the sheet. The dog-bone-shaped specimens were 1 mm in thickness and 10 mm in gage length. The tensile axes were parallel to the rolling direction (RD) and transverse direction (TD) of the sheets.

3. Results and discussion

The textures of the samples are illustrated in Fig. 2. The initial sheets had different strong textures. They were cut from the same hot rolled magnesium alloy AZ31 sheet, which had a typical strong basal texture [21]. The samples of series A (Initial_A, CRA and CRA_f) had similar textures, while the textures of the series B samples (Initial_B, CRB and CRB_f) were different. As the strain increased, an increasing number of {0001} poles rotated from the transverse direction (TD) to the normal direction (ND). As it will be shown later, this was due to the active twinning during cryorolling of the samples of series B. The textural change was slight at the strain of 4% while the change was obvious at the strain of 8%. That could be because of the dislocation slips at the early deformation stage. Sample CRA_f cracked at the strain reduction of 8%. Therefore, the strain of 8% was considered as the failure strain in this study. The textural evolution of the AZ31 alloy sheets at the cryogenic temperature was similar to that at the room temperature [31]. However, the failure strain reduction was smaller than that at the room temperature, even though the strain rate in this study was relatively moderate. This indicated the inhomogeneous strain distribution and strain concentration of the cryorolled samples.

Microstructures of the samples are illustrated in Fig. 3. Twinning was found to be very active in the samples of series B. The initial material was an equiaxed structured sheet with a mean grain size of approximately 30 μm. No twins were found in the initial sheet. Lens-shaped twins were observed in the cryorolled sample. Larger grains contained more twins. Considerably more twins were found in the

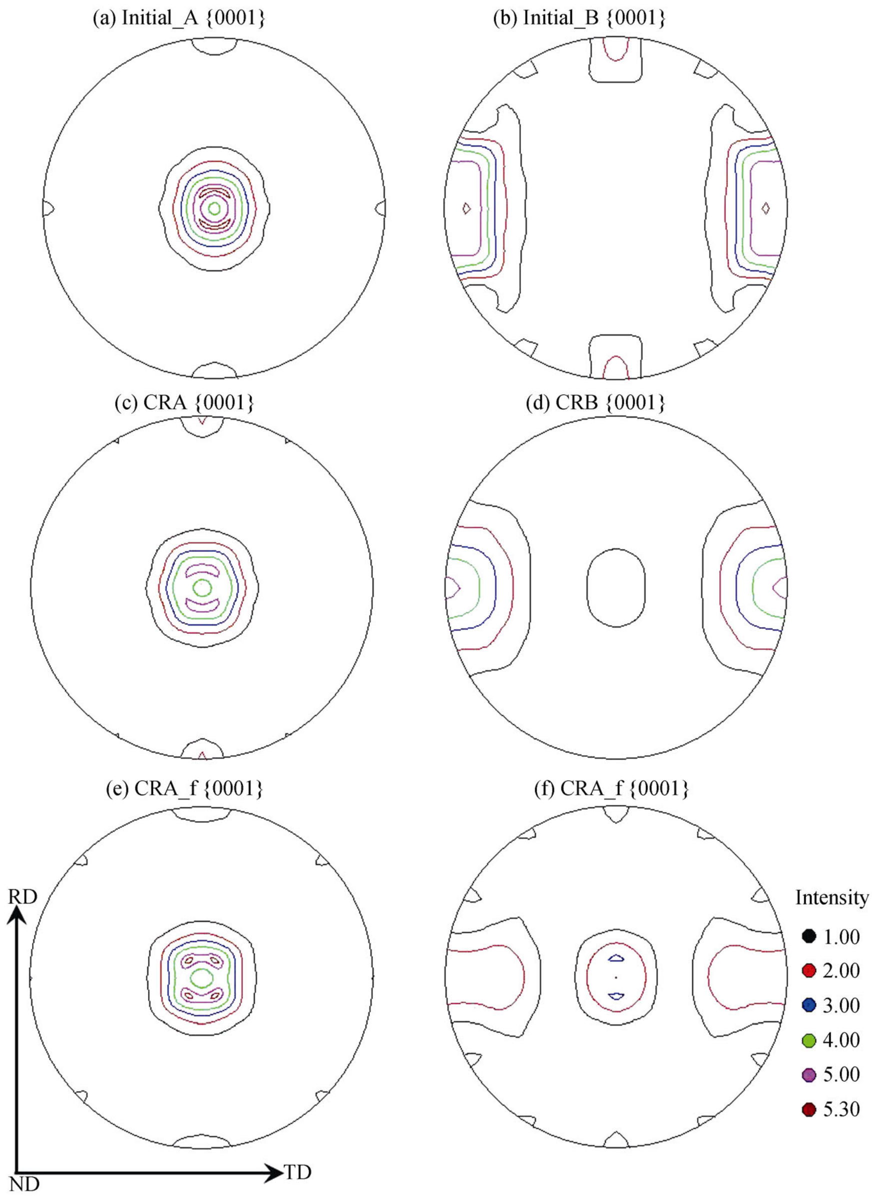

Fig. 2. Textures of the (a) Initial_A, (b) Initial_B, (c) CRA, (d) CRB, (e) CRA_f, and (f) CRB_f samples (RD: rolling direction; TD: transverse direction; ND: normal direction).

rolled samples of series B than that in the rolled sample of series A. Also, the twins in the rolled samples of series B were significantly wider and larger. These results indicated that twinning played an important role and was significantly active in the samples of series B during the cryogenic rolling. Micro-cracks could be seen in the CRA_f sample (as shown in the Fig. 3(d)). The micro-cracks caused by strain concentration could directly cause the early failure of the sample.

In order to analyze the twins in further detail, a statistical study of the twins based on the microstructural observations was conducted. The results are illustrated in Fig. 4. In the figure, gray bars indicate the proportion of the twinned grains (grains containing twins), solid circles represent the average number of twins per hundred grains, and the solid star indicates the average number of twins per grain. Evidently the samples of series B contained more grains with textures in favor of extension twinning. Furthermore, more grains experienced twinning in the series B samples than in the series A samples. Approximately 65% and 53% of the grains contained twins in the CRB and CRB_f samples, respectively. The percentages of the grains with twins for the CRA and CRA_f samples were only 23% and 36%, respectively. There were more twins in the samples of series B than in the samples of series A at the same strain. However, there was not much difference in the average numbers of twins per grain between the two series of the samples. That

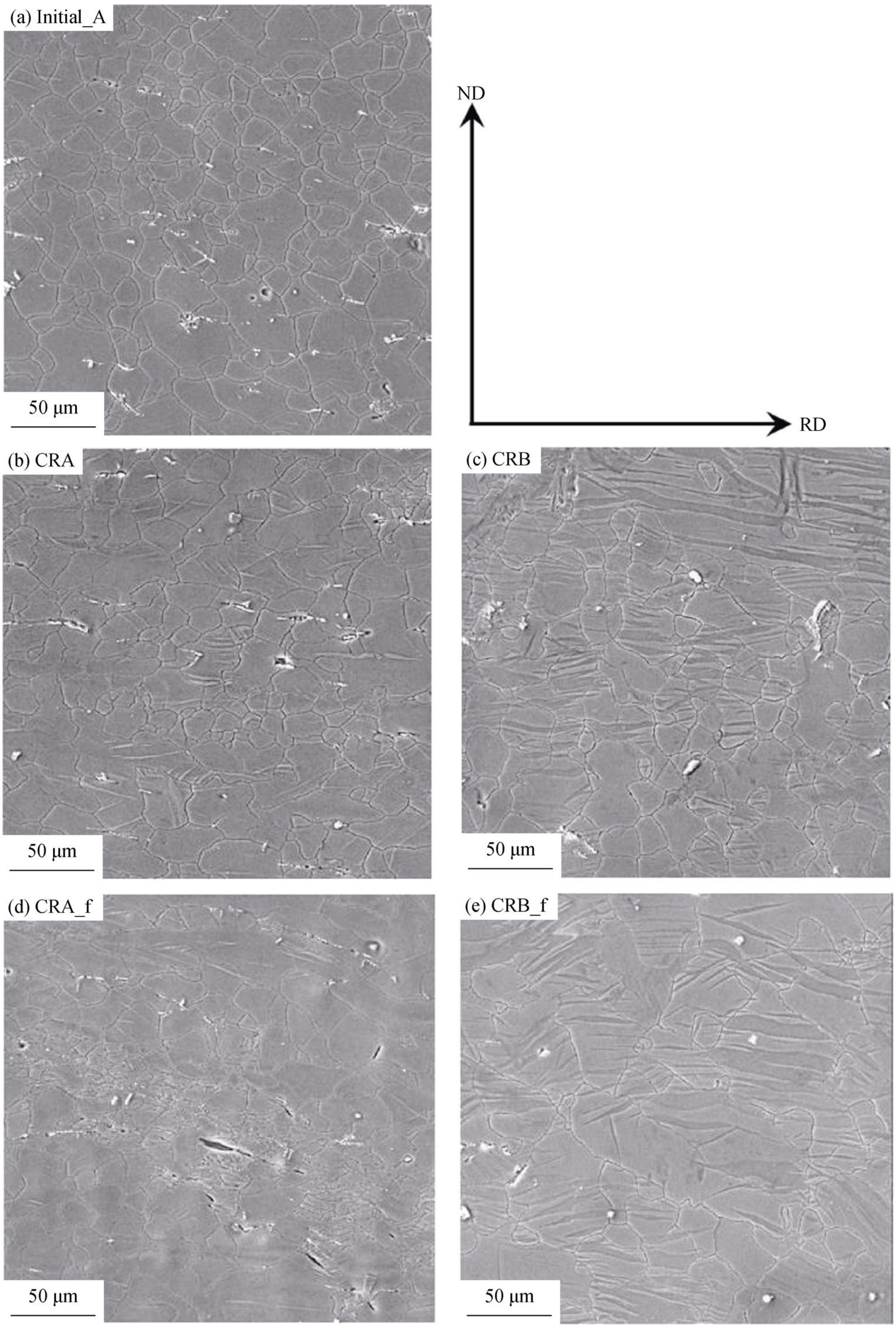

Fig. 3. Microstructures of the (a) Initial_A, (b) CRA, (c) CRB, (d) CRA_f, and (e) CRB_f samples (ND: the normal direction; RD: the rolling direction).

is, twinning was more active in the samples of series B due to the activation of twining in more grains. The initial texture of the Initial_B sample was in favor of $\{10\bar{1}2\}$ extension twinning. The Initial_A sample had a texture in favor of $\{10\bar{1}1\}$ contraction twinning or then evolved to $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twinning. However, the $\{10\bar{1}1\}$ contraction twinning had a higher shear value γ and higher critical resolved shear stress (CRSS) value than the $\{10\bar{1}2\}$ extension twinning [16,19–20,22]. Therefore, it was considerably more difficult for the $\{10\bar{1}1\}$ contraction twinning and $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twinning to occur when compared to the $\{10\bar{1}2\}$ extension twinning. Our investigations further revealed that neither $\{10\bar{1}1\}$ contraction twins nor $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twins were found in the samples. This could be a reason why fewer twins were found in the rolled samples of series A. The relative low rolling strain rate used in this study could be another reason for the suppression of contraction twinning. It should be noted that although the sample CRB_f was at a greater reduction, there were fewer twins and fewer twinned grains in the sample CRB. That could be because of the fast growth of the extension twins. With the increase in the strain, the extension twin grain boundary could migrate and even consume the mother grain completely [27].

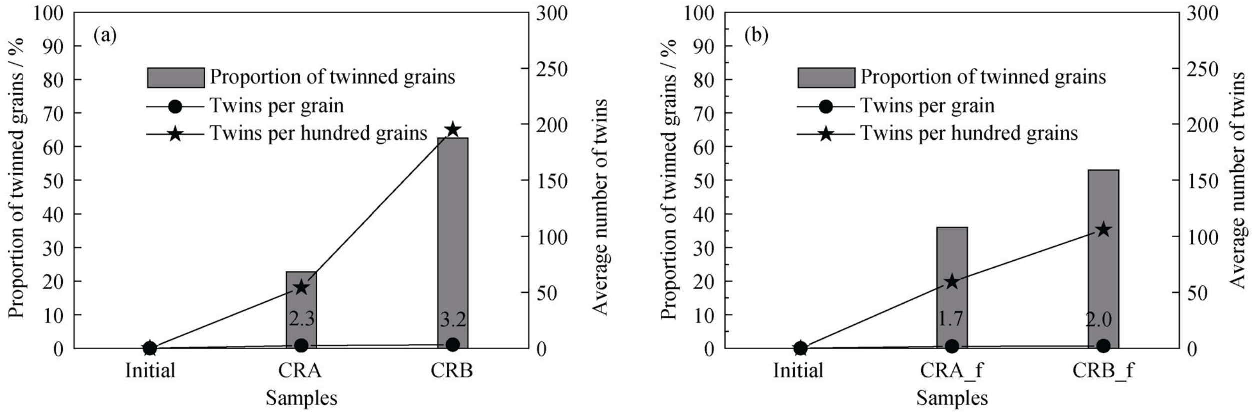

Fig. 4. Statistical results of the observed twins in the samples.

Further investigations were carried out by EBSD mapping to distinguish the twinning types as there were various twining types in the magnesium alloy. The restructured microstructure is shown in Fig. 5. The results revealed that $\{10\bar{1}2\}$ extension twins dominate in all the cryorolled samples. Few constriction twins and double twins were observed. The misorientation angles between the different types of twins and the mother grains were different (misorientation between the $\{10\bar{1}2\}$ extension twin and the mother grain was approximately 86° $\langle 11\bar{2}0\rangle$, i.e. the misorientation of the twin grain boundary is 86° around $\langle 11\bar{2}0\rangle$). Additionally, the frequency of low angle grain boundaries (LAGB) (<5°) indicated the activity of dislocation slips and stored energy of the sample. Hence, the misorientation angle distribution should be investigated further. Fig. 6 illustrates the results of the misorientation angle distribution of the samples. The high LAGB frequency of the rolled samples indicated the active dislocation slips during cryorolling and the high stored energy of the rolled samples. The significantly high stored energy of the sample CRA_f could be one of the main reasons for the failure. Considerably higher numbers of twins were found in the rolled samples of series B. Therefore, the twin grain boundaries (TGBs) frequency of the CRB and CRB_f samples was higher than that of the CRA and CRA_f samples. The difference between the microstructural evolutions of the AZ31 sheets rolled at cryogenic and room temperature was small. Also, no special twin was found in the sample rolled at the cryogenic temperature. However, twinning was more active when the temperature was decreased.

Finally, the mechanical properties of the rolled samples CRA and CRB were tested. The samples CRA_f and CRB_f were not considered because they were at the failure strain. The results are illustrated in Fig. 7. Uniaxial tension tests were performed at the room temperature along the rolling direction (RD) and transverse direction (TD) for each sample. The grains at a distance of {0001} poles from ND were in a soft orientation for activating dislocation slips at a relative low stress [16,20]. Therefore, given the anisotropic basal texture of the sheets, the strength in the RD was higher than that in the TD. Conversely, the Initial_B and CRB samples were stronger in the TD. The flow curves of the sheets in the TD were S-shaped [19,22,27], indicating the active extension twinning during the tension test in TD. The high intensity in the TD for the textures of the samples was the reason for the following phenomena. Specifically, several grains in these two samples have the *c*-axes parallel to the extension axis. This makes it very easy for the $\{10\bar{1}2\}$ extension twinning to take place. The fracture elongation values of both the rolled samples decreased significantly and the strength values of the samples increased when compared with the initial samples. This could be because of the high stored energy of the sheets accumulated during the cryogenic rolling. The twins also significantly contributed to this phenomenon. The twins in the rolled samples separated and refined the grains, thereby changing the crystal orientation and affecting the texture. Also, the pre-existing $\{10\bar{1}2\}$ extension twins in the cryorolled samples could cause the early failure by concentrating local strain on the boundaries or at the triple junctions [14,16–18,20]. The strain accommodation due to active twinning was also beneficial to the ductility during tension [19,22,25]. Twinning had an important influence on the work hardening of the samples.

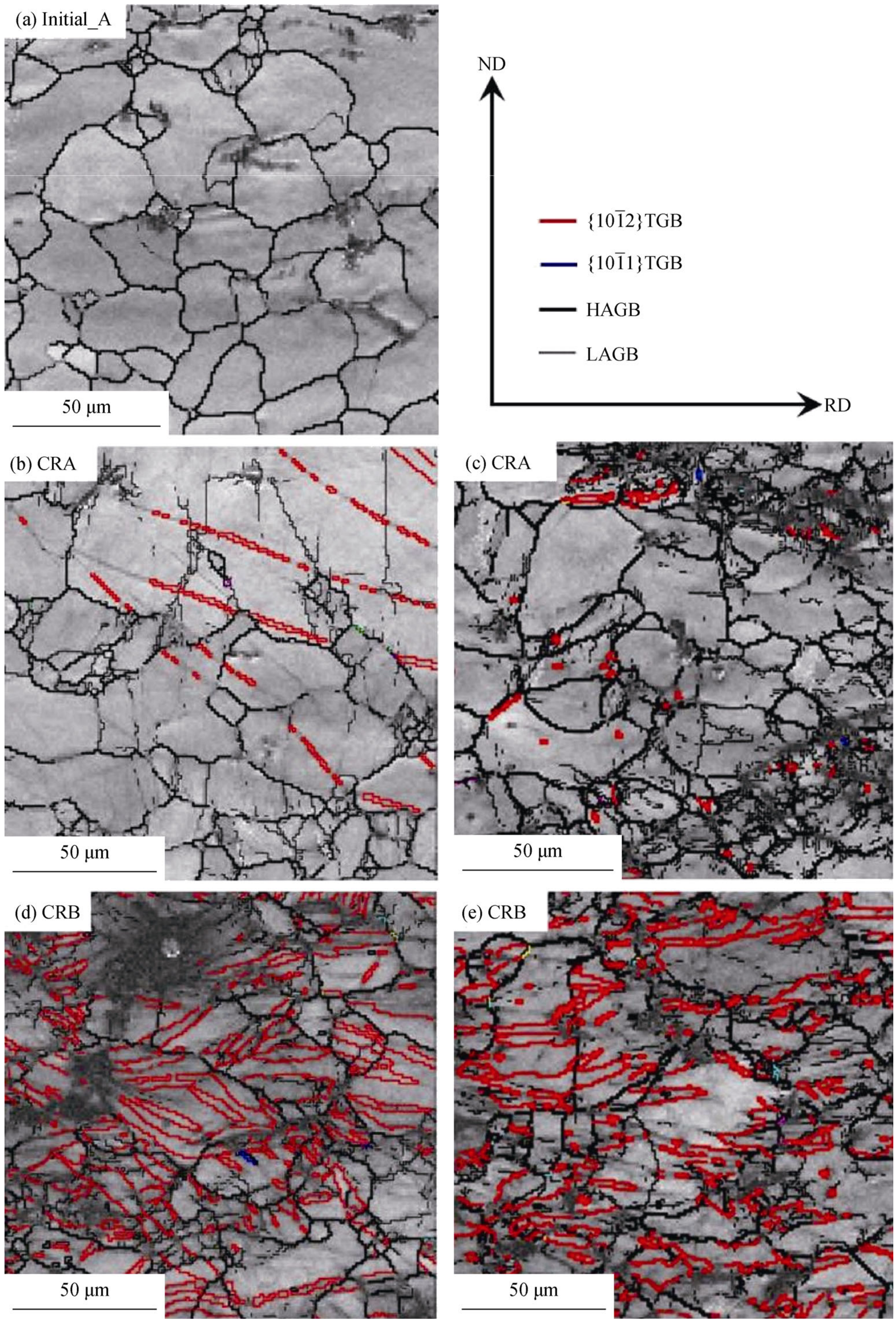

Fig. 5. Restructured microstructures of the (a) Initial, (b) CRA, (c) CRB, (d) CRA_f, and (e) CRB_f samples (TGB: twin grain boundary; HAGB: >15° high angle grain boundary; LAGB: <2° low angle grain boundary).

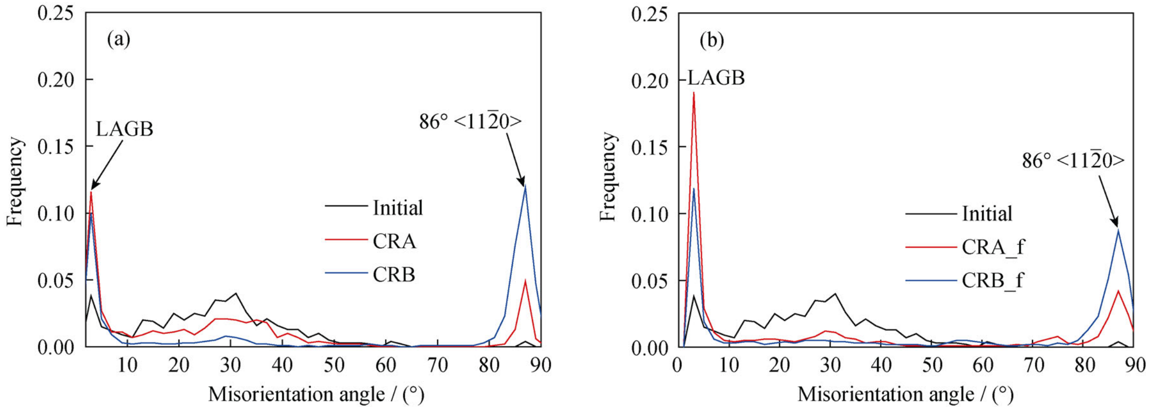

Fig. 6. Misorientation angle distribution results of the samples.

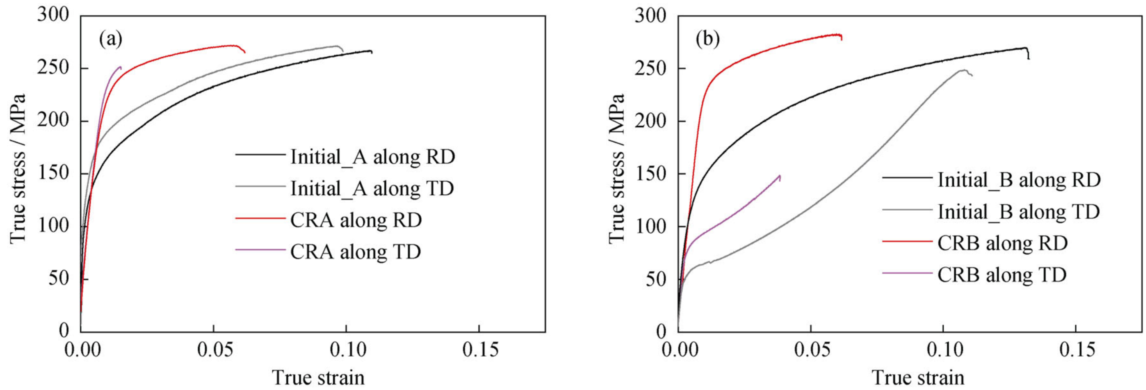

Fig. 7. Flow curves of the initial and cryorolled samples (a) series A and (b) series B.

4. Conclusions

The microstructural and textural evolutions of the cryogenic rolled AZ31 magnesium alloy sheets were investigated via SEM, EBSD and XRD. The mechanical properties of the samples were discussed. Specifically, the influence of the different initial textures was taken into consideration. The results of this study yielded the following conclusions:

(1) The initial texture of the sheet significantly influenced the twinning and microstructural and textural evolutions of magnesium alloy AZ31 sheets during cryogenic rolling. Twins were found in all the rolled samples. More twins were found in the samples of series B. The texture intensity of the samples of series B could be significantly decreased because of the active twinning during cryorolling. The textures of the samples of series A only showed a slight change.

(2) Twinning was active during the cryorolling. The $\{10\bar{1}2\}$ extension twin was the dominant twin in all the rolled samples. A few $\{10\bar{1}1\}$ contraction twins and $\{10\bar{1}1\}-\{10\bar{1}2\}$ double twins were observed in this study. There were significantly more twins observed in the sheets rolled at the cryogenic temperature when compared with the sheets rolled at room temperature. However, there were no special types of twins found in the sheets rolled at the cryogenic temperature when compared with the sheets rolled at room temperature.

(3) After cryogenic rolling, the ductility of the sheets of both sample series decreased while the strength increased. The mechanical anisotropy changed slightly for the in-plane uni-axial tension. Twinning played an important role in explaining the mechanical difference between the rolled samples of the two series. The rolled samples had high stored energy and were significantly strengthened.

Acknowledgements

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