RETRACTED: Effect of Final Rolling Temperature on Microstructures and Mechanical Properties of AZ31 Alloy Sheets Prepared by Equal Channel Angular Rolling and Continuous Bending

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Abstract: The effects of final rolling temperature on the microstructures, texture and mechanical properties of AZ31 Mg alloy sheets prepared by equal channel angular rolling and continuous bending (ECAR-CB) were investigated. Extension twins {10–12} could be observed in the ECAR-CB deformed sheets. The increase in the number of {10–12} extension twins with increasing final rolling temperature might be attributed to the larger grain size and faster grain boundary migration. For all the ECAR-CB sheets at different final rolling temperatures, the deformation texture contains a basal texture component and a prismatic texture component, whereas the annealing recrystallization texture becomes a non-basal (pyramidal) texture with double peaks tilting away from normal direction (ND) to rolling direction (RD). With increasing final rolling temperature, the tilted angle of double peaks of annealing recrystallization non-basal texture increases. In addition, the plasticity and formability of ECAR-CB-A (ECAR-CB and then annealing) AZ31 Mg alloy sheets at room temperature can be improved by increasing the final rolling temperature.

Keywords: magnesium alloy; ECAR-CB; final rolling temperature; extension twins; texture

1. Introduction

Magnesium (Mg) alloys have received great attention because of outstanding properties such as low density, high specific strength and damping capacity; thus, they have potential applications in aerospace and automotive industries [1–5]. It is well known that the hexagonal close packed (HCP) crystal structure for Mg alloys can only supply a limited number of available slip systems at room temperature. Moreover, for wrought magnesium alloy sheets, a strong basal texture can usually be generated after the traditional rolling process, which further degrades their stretch formability at room temperature [6–10]. As a result, the wide applications of Mg alloys, especially for the wrought sheets, have been restricted by their poor room temperature ductility and formability. Weakening or eliminating the basal texture has been considered as an effective way to improve the formability of wrought magnesium alloy sheets at room temperature.

To date, many plastic deformation technologies have been developed to tailor the texture of wrought magnesium alloy sheets for improving their formability at room temperature [11–21]. For instance, Suh et al. [11] revealed that the cold formability of AZ31 Mg alloy sheets could be enhanced by weakening the basal texture of rolled AZ31 Mg alloy sheets using equal channel angular
pressing (ECAP). Cheng et al. [13] and Kim et al. [14] introduced shear deformation by equal channel angular rolling (ECAR) to weaken the basal texture of rolled AZ31 Mg alloy sheets, which improved their drawability at room temperature. Yanagida et al. [21] reported that weakening basal texture by the repeated roll bending and subsequent low temperature annealing is effective in increasing the room temperature formability of AZ31 magnesium alloy sheets. However, these processes are mainly focused on the weakening of basal texture, seldom concerning the elimination of basal texture for AZ31 Mg alloy sheets which, in practice, are better in the improvement of their room temperature formability.

Recently, a new plastic deformation method, i.e., equal channel angular rolling and continuous bending (ECAR-CB), was proposed and confirmed to be a more effective and manageable way to eliminate basal texture and then significantly improve the formability of AZ31 Mg alloy sheet at room temperature [22]. As known, deformation temperature has an important influence on the microstructures and mechanical properties of Mg alloys [23–25]. Nevertheless, how final rolling temperature influences the microstructures and mechanical properties of ECAR-CB Mg alloy sheets, a better understanding of which is virtually important and helpful in the research and development of ECAR-CB method, has not yet been clarified. In this study, we investigated the effects of final rolling temperature on the microstructures, texture and mechanical properties of AZ31 Mg alloy sheets prepared by the ECAR-CB method. The influencing mechanism was also discussed.

2. Experimental Procedure

The experimental material used in this work was hot-rolled AZ31 magnesium alloy (Mg-3 wt. %Al-1 wt. %Zn-0.3 wt. %Mn) sheets with a width of 120 mm, which were preheated to 400 °C for about 20 min prior to rolling, and then rolled from 4.3 to 1.26 mm in thickness by multi-pass at 400 °C. Subsequently, the sheets were preheated at three different temperatures (i.e., final rolling temperatures of 350, 450 and 550 °C) for 1 min, rolled from 1.26 to 1.2 mm in thickness with one pass at the rolling speed of 0.4 m/s, and then pushed into the equal channel angular rolling with continuous bending (ECAR-CB) device. The ECAR-CB device is illustrated in Figure 1, and more details about the ECAR-CB device have been well described in the literature [22]. In addition, several ECAR-CB sheets were annealed at 350 °C for 1 h, which were defined as ECAR-CB-A sheets.

![Figure 1. Schematic illustration of the equal channel angular rolling and continuous bending (ECAR-CB) process: shear deformation in corner 1 and bending deformation in corners 2, 3 and 4.](image)

In this study, RD, TD and ND represent the rolling, transverse and normal directions, respectively. Microstructures of selected samples on the RD–TD plane were observed by using an optical microscope (OM, Leica Camera AG, Frankfurt, Germany) and Electron Backscattered Diffraction (EBSD). Microtextures were also characterized by using EBSD, which was conducted on a FEI NOVA 400 Zeiss Sigma field emission scanning electron microscope (Carl Zeiss Company, Oberkochen, Germany) equipped with an HKL-EBSD system using a step size of 1.5 μm. The macrotextures were measured by X-ray diffraction (XRD) using a Rigaku D/Max 2500 PC at the upper surface which was first mechanically polished (about loss of 0.1 mm in thickness).
Uniaxial tension experiments of selected AZ31 Mg alloy sheets along RD were performed at room temperature by using a SNAS tensile testing machine (MTS Systems Corporation, Eden Prairie, MN, USA) at a constant strain rate of $1.0 \times 10^{-3}$ s$^{-1}$. Uniaxial tension samples with 15 mm in gauge length and 4 $\times$ 1.2 mm in cross-section were cut from the AZ31 Mg alloy sheets by electro-discharge machining. Lankford values ($r$-value) were calculated by measuring the strains in the width ($\varepsilon_w$) and thickness ($\varepsilon_t$) directions ($r = \varepsilon_w/\varepsilon_t$) of the tensile samples with a plastic strain of 12%. Strain-hardening exponent values ($n$-value) were obtained by a method described by Luo et al. [26]. It is noted that three tensile samples were tested for each type of mechanical property. Moreover, circular blanks with 60 mm in diameter were cut from the AZ31 sheets for the Erichsen test, which were carried out using a hemispherical shape punch with a diameter of 20 mm. The punch speed and the blank holder force were about 4 mm/min and 10 kN, respectively. The average Erichsen values (IEs) were obtained from two Erichsen tests for each AZ31 sheet.

3. Results and Discussion

3.1. Microstructure and Texture Evolution

Figure 2 shows the microstructure and texture of the as-received hot-rolled (HR) AZ31 magnesium alloy sheet. The as-received HR sheet mainly consists of equiaxed grain with uniform distribution, and nearly no twins could be observed.

![Figure 2](image-url)

Figure 2. (a) Inverse pole figure and (b) (0002) pole figure of the as-received HR AZ31 Mg alloy sample.

Figure 3 shows the microstructures of the ECAR-CB sheets prepared at different final rolling temperatures. As the final rolling temperature increased from 350 to 550 °C, the average size of deformation grains progressively increased. When the final rolling temperature was 350 °C, some twins were observed in a small number of grains. With the increase of final rolling temperature, the fraction of twinned grains increased gradually.

![Figure 3](image-url)

Figure 3. Microstructures of the ECAR-CB AZ31 Mg alloy samples prepared at different final rolling temperatures: (a,b) 350 °C, (c,d) 450 °C and (e,f) 550 °C.
Figure 4 shows the [0002] pole figures of the ECAR-CB sheets prepared at different final rolling temperatures. As indicated, besides the basal (c-axis//ND) texture component, a new prismatic (c-axis//RD) texture component was also observed in the [0002] pole figures of the three ECAR-CB samples. In addition, as the final rolling temperature increased, the maximum intensity of prismatic texture component was almost constant first and then elevated, whereas that of the basal texture component decreased gradually. It suggests that increasing the final rolling temperature could weaken the basal texture of ECAR-CB sheets.

In order to further identify the twin types in the deformation structure, the EBSD results of the ECAR-CB AZ31 Mg alloy sample prepared at 350 °C are shown in Figure 5. In the grain boundary (GB) map, [10–12] extension twin boundary, [10–11] compression twin boundary, high-angle grain boundaries (HABs) with the misorientation angle greater than 10° and low-angle grain boundary (LAB) with the misorientation angle ranging from 2° to 10° are marked by red, green, black and gray lines, respectively. As indicated, most of the twins in the ECAR-CB AZ31 Mg alloy sample prepared at 350 °C are identified as [10–12] extension twins. Several twin lamellae are nearly parallel with each other in one matrix grain which is subdivided into a few separated parts, as shown in Figure 5a. Moreover, there are many low-angle grain boundaries (LAGBs) which might be related to the dislocation slip. It is well known that [10–12] extension twinning could rotate the basal plane about 86° between the twin and the un-twinned matrix grain, while dislocation slip can only result in a negligible orientation change [27]. Therefore, formation of the prismatic (c-axis//RD) texture component for the ECAR-CB AZ31 Mg alloy sheets might be attributed to the generation of [10–12] extension twins.

Figure 5. EBSD maps of the ECAR-CB AZ31 Mg alloy sample at final rolling temperature of 350 °C: (a) inverse pole figure (IPF) map and (b) grain boundaries (GB) map.
In fact, many [10−12] extension twins could also be observed in the ECAR-CB sample prepared at a final rolling temperature of 550 °C, as shown in Figure 6. However, the fraction of extension twins at 550 °C might be different from that at 350 °C. Table 1 shows the calculated volume fractions of extension twins for the various ECAR-CB samples prepared at 350 and 550 °C. For an easier calculation, it was assumed that the volume fraction of twins was identical to their area fraction. Three different regions of each ECAR-CB sample were used in the calculation of average area fraction. The calculated results showed that the volume fraction of the [10−12] twinning for the ECAR-CB samples at 550 °C was about 55.2%, which was more than two times of that (26.3%) at 350 °C. Again, it is suggested that, for the ECAR-CB Mg alloy sheets, increasing the final rolling temperature can increase the fraction of [10−12] extension twins. This observation could contribute to the decrease in the maximum density of the basal (c-axis/ND) texture component. In addition, the larger fraction of [10−12] extension twins for the ECAR-CB Mg alloy sheets at higher final rolling temperature might be related to the larger grain size and quicker growth of extension twins. It has been reported that the stress to activate [10−12] extension twins increases dramatically with decreasing grain size [28]. As mentioned above, the grain size of ECAR-CB Mg alloy sheets increases with increasing final rolling temperature. Therefore, the activation of [10−12] extension twins at a relatively high final rolling temperature (e.g., 550 °C) might be easier than that at a relatively low rolling temperature (e.g., 350 °C). On the other hand, the growth of twins is generally accompanied by migration of the twin boundary, which is usually through the glide-shuffle of twin dislocations and the climb-shuffle of interface dislocations [29]. It is well known that the motion of dislocations can be accelerated by increasing temperature [30]. As a result, the growth of [10−12] extension twins might be faster at relatively high rolling temperature (e.g., 550 °C).

Figure 6. EBSD maps of the ECAR-CB AZ31 Mg alloy sample at final rolling temperature of 550 °C: (a) inverse pole figure (IPF) map and (b) grain boundaries (GB) map.

Table 1. Calculated area fractions of [10−12] extension twins for the ECAR-CB samples prepared at 350 and 550 °C.

| Final Rolling Temperature | Region 1 | Region 2 | Region 3 | Average Value |
|--------------------------|----------|----------|----------|---------------|
| 350 °C                   | 26.2%    | 25.7%    | 27.1%    | 26.3%         |
| 550 °C                   | 55.1%    | 53.7%    | 56.9%    | 55.2%         |

Figure 7 shows the recrystallization microstructures of the ECAR-CB-A samples prepared at different final rolling temperatures. It indicates that all the ECAR-CB-A samples were almost completely recrystallized during the annealing process, and twins were nearly absent. In addition, the ECAR-CB-A samples had little difference in the average grain size, which was about 15.2, 14.6 and 14.9 μm at final rolling temperatures of 350, 450 and 550 °C, respectively.
The yield strength (YS) decreased from 82 to 74 MPa, the ultimate tensile strength (UTS) decreased slightly from 225 to 220 MPa, the Lankford value (r-value) decreased from 0.62 to 0.43, while the n-value increased from 0.40 to 0.47 and the fracture elongation (FE) increased slightly from 23.5% to 26.1%.

Figure 7. (a–c) Inverse pole figure (IPF) maps and (d–f) grain boundaries (GB) maps of the ECAR-CB-A AZ31 Mg alloy samples prepared at different final rolling temperatures: (a,d) 350 °C, (b,e) 450 °C and (c,f) 550 °C.

Figure 8 shows the (0002) pole figures of ECAR-CB-A samples prepared at different final rolling temperatures. For all the ECAR-CB-A samples, the prismatic (c-axis/RD) texture and basal (c-axis/ND) texture components disappeared, while a non-basal recrystallization texture with double peaks tilting from ND towards RD (i.e., pyramidal recrystallization texture) was identified. For the ECAR-CB-A samples at 350 °C, the tilting angle (θ) was about 29°, and the contour lines of pyramidal recrystallization texture were not separate. Furthermore, the tilting angle increased with the increasing final rolling temperature. For the ECAR-CB-A samples at 550 °C, the tilting angle (θ) increased to about 40°, and contour lines of the pyramidal texture had been completely separated. However, the underlying mechanism for the static recrystallization mechanism of twins in the ECAR-CB AZ31 Mg alloys and its effect on texture evolution need to be revealed by further works.

Figure 8. Pole figures (0002) of the ECAR-CB-A AZ31 Mg alloy samples prepared at different final rolling temperatures: (a) 350 °C, (b) 450 °C and (c) 550 °C.

3.2. Mechanical Properties

The mechanical properties of the ECAR-CB-A samples at different final rolling temperatures are summarized in Table 2. As the final rolling temperature increased from 350 to 550 °C, the yield strength (YS) decreased from 82 to 74 MPa, the ultimate tensile strength (UTS) decreased slightly from 225 to 220 MPa, the Lankford value (r-value) decreased from 0.62 to 0.43, while the n-value increased from 0.40 to 0.47 and the fracture elongation (FE) increased slightly from 23.5% to 26.1%.

| Sample | YS (MPa) | UTS (MPa) | FE (%) | r-value | n-value |
|--------|----------|-----------|--------|---------|---------|
| 350 °C | 82 ± 2   | 225 ± 10  | 23.5 ± 1.5 | 0.40 ± 0.02 | 0.62 ± 0.02 |
| 450 °C | 79 ± 6   | 223 ± 12  | 24.6 ± 0.9  | 0.43 ± 0.01 | 0.58 ± 0.03 |
| 550 °C | 74 ± 5   | 220 ± 10  | 26.1 ± 2.1  | 0.47 ± 0.01 | 0.43 ± 0.01 |

Table 2. Mechanical properties of various ECAR-CB-A samples prepared at different final rolling temperatures.
This suggests that increasing the final rolling temperature could improve the plasticity of the ECAR-CB-A AZ31 Mg alloy sheet, which is related to the effect of texture. As mentioned above, a non-basal recrystallization texture with double peaks tilting from ND towards RD was generated in ECAR-CB-A Mg alloy samples, and the tilting angle of double peaks increased with the final rolling temperature. The average Schmid factor of basal \(<a>\) slip under uniaxial tension along RD for the ECAR-CB-A samples at different final rolling temperatures was calculated and is shown in Table 3. As indicated, the average SF of basal \(<a>\) slip under uniaxial tension increased with increasing tilting angle of double peaks non-basal texture at 350–550 °C. Furthermore, basal \(<a>\) slip had a lower critical resolved shear stress (CRSS) than other deformation mechanisms [31]. It is suggested that basal \(<a>\) slip is easier to activate under uniaxial tension along RD in the ECAR-CB-A samples with higher final rolling temperature ranging from 350 to 550 °C, which was mainly due to the larger tilting angle. Consequently, the YS of ECAR-CB-A samples, which mainly depends on the activation of basal \(<a>\) slip, decreased with increasing final rolling temperature. In addition, for the Mg alloy sample with a non-basal texture, the thickness strain could mainly be accommodated by basal \(<a>\) slip, leading to a decrease in the \(n\)-value [32]. The softening behavior of dynamic recovery can be restricted by the enhancement of basal \(<a>\) slip, leading to an increase in the \(n\)-value [33]. As a result, compared with the lower final rolling temperature (e.g., 350 °C), the ECAR-CB-A Mg alloy sample at higher final rolling temperature (e.g., 550 °C) exhibited a smaller \(r\)-value and a larger \(n\)-value owing to the easier activation of basal \(<a>\) slip under uniaxial tension along RD.

Table 3. Average Schmid factor (SF) of basal \(<a>\) slip under uniaxial tension along RD for the ECAR-CB-A samples at different final rolling temperatures.

| Sample  | 350 °C | 450 °C | 550 °C |
|---------|--------|--------|--------|
| ECAR-CB-A | 0.29   | 0.31   | 0.35   |

On the other hand, as shown in Figure 9, the Erichsen values (IE) were about 6.3, 6.4 and 7.4 mm for the ECAR-CB-A samples at final rolling temperatures of 350, 450 and 550 °C, respectively, higher than that (4.6 mm) of HR-A (HR and then annealing) samples. This indicates that the stretch formability of ECAR-CB-A AZ31 Mg alloy sheet can also be enhanced by increasing the final rolling temperature, which might be mainly attributed to the decreased YS, diminished \(r\)-value and increased \(n\)-value. Furthermore, the Erichsen value (7.4 mm) of ECAR-CB-A AZ31 Mg alloy sheet is indeed larger than the reported Erichsen values (in the range of 2.83–6.3 mm) of AZ31 sheets which underwent different deformation and subsequent annealing conditions [10,34–36].
1. For the ECAR-CB AZ31 magnesium alloy sheets, {10–12} extension twins could be observed, and the amount of {10–12} extension twins increased with increasing temperature, which might be attributed to the larger grain size and faster grain boundary migration. The deformation texture contained a basal (c-axis/ND) texture component and a prismatic (c-axis/RD) texture component, which could be attributed to the formation of extension twins.

2. For the ECAR-CB-A AZ31 magnesium alloy sheets, the grain structures at different final rolling temperatures were recrystallized completely. A non-basal recrystallization texture (pyramidal texture) with double peaks tilting from ND towards RD could be identified, and the tilting angle increased gradually with the increasing final rolling temperature.

3. Increasing the final rolling temperature could improve the plasticity and formability of ECAR-CB-A (ECAR-CB and then annealing) AZ31 Mg alloy sheets at room temperature, which might result from the increase in the tilting angle of the non-basal (pyramidal) recrystallization texture.

4. Conclusions

1. For the ECAR-CB AZ31 magnesium alloy sheets, {10–12} extension twins could be observed, and the amount of {10–12} extension twins increased with increasing temperature, which might be attributed to the larger grain size and faster grain boundary migration. The deformation texture contained a basal (c-axis/ND) texture component and a prismatic (c-axis/RD) texture component, which could be attributed to the formation of extension twins.

2. For the ECAR-CB-A AZ31 magnesium alloy sheets, the grain structures at different final rolling temperatures were recrystallized completely. A non-basal recrystallization texture (pyramidal texture) with double peaks tilting from ND towards RD could be identified, and the tilting angle increased gradually with the increasing final rolling temperature.

3. Increasing the final rolling temperature could improve the plasticity and formability of ECAR-CB-A (ECAR-CB and then annealing) AZ31 Mg alloy sheets at room temperature, which might result from the increase in the tilting angle of the non-basal (pyramidal) recrystallization texture.

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