Evolution of Microstructure and Texture During Uniaxial Compression of Cast AZ31Mg Alloy at Elevated Temperatures

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Received: 2 August 2012 / Revised: 28 September 2012 / Published online: 8 November 2012
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Abstract Isothermal compression on cast AZ31 magnesium alloy was conducted at different deformation conditions (different temperatures and strain rates) to study microstructure development and texture evolution in the alloy. The as-deformed microstructure and texture distribution were investigated using light microscopy (LM), x-ray diffraction (XRD), and electron backscattered diffraction (EBSD). The examination of the flow stress as a function of strain rate showed that grain boundary sliding was not a dominant mechanism in the cast AZ31 alloy. Occurrence of dynamic recrystallization was confirmed by the evaluation of stress–strain curves and LM. The large grain sizes resulted in a coarse pole figures by XRD. To improve the statistical robustness of the pole figures, the results from multiple sample sections were averaged. The averaged result indicated the formation of basal texture at low strains, where basal planes of most grains were perpendicular to the compression axis. EBSD results showed that the texture distribution at small strain was dominated by the orientations of deformed grains.

Keywords Cast AZ31 alloy · Microstructure · Texture · Dynamic recrystallization

Introduction

Magnesium has received significant attention for the automotive and aerospace applications in recent years due to its light weight and consequent potential to reduce both fuel consumption and green-house gas emission. To enable reliable engineering design of magnesium components, the microstructure development and deformation mechanisms under different temperatures and strain rates must be understood.

Of the various commercial magnesium alloys, the Mg–Al–Zn-type alloy, such as AZ31, has found a large number of industrial applications. The forming processes of AZ31 alloy are often conducted at elevated temperatures due to its limited room-temperature formability. The deformation behavior of AZ31 alloy at elevated temperature has been widely studied in the recent years, and can be summarized as:

- (a) There are different slip systems that can operate if the critical resolved shear stresses (CRSS) are exceeded: basal $\langle a \rangle$ slip, prismatic $\langle a \rangle$ slip, pyramidal $\langle a \rangle$ slip, and pyramidal $\langle c+a \rangle$ slip. The CRSSs of non-basal slip systems show strong temperature dependence, decreasing significantly with increasing temperature [1–3]. Hence, non-basal slip systems are more easily activated at high temperature than at room temperature [4].
- (b) Twinning and non-basal slip are competitive deformation mechanisms [1, 5]. Twinning plays a vital role by accommodating strain at low temperature because of the limited activity of non-basal slip systems. Although non-basal slip is easily activated at high temperature, twinning occurs at small strain to reorient grains [6, 7].
- (c) Dynamic recrystallization (DRX) in AZ31 alloy is widely observed at elevated temperatures and several

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Table 1 The compositions of AZ31 (wt.%)

Alloy	Mg	Al	Be	Cu	Fe	Mn	Pb	Si	Zn
AZ31	Bal.	2.66	<0.0005	<0.005	0.01	0.36	<0.01	0.01	0.84

Fig. 1 Location of compression samples from a section of a cast billet **Fig. 2** Sketch of the “masked” sample for XRD measurement **Fig. 3** Measured σ - ε curves from the uniaxial compression testing of the inner samples under different temperatures and strain rates. **a** 623 K and **b** 673 K **Fig. 4** Measured σ - ε curves from the uniaxial compression testing of the outer samples under different temperatures and strain rates. **a** 623 K and **b** 673 K **Fig. 5** Critical grain sizes d_s and d_t as a function of Z and the possible deformation mechanisms for the compression testing in this study (the black symbols represent the billet inner sample grain size of 181 μm , and the red symbols the billet outer sample grain size of 135 μm)

mechanisms have been proposed. Discontinuous DRX (DDR_X) [8–10] is the conventional recrystallization phenomenon characterized as nucleation by high angle grain boundary bulging. Continuous DRX (CDR_X) [11–14] is identified by an increase in misorientation from the center to the edge of grain

Fig. 6 Microstructure of the casting billet. **a** Inner and **b** outer regions

Fig. 7 Calculated strain rate sensitivity exponent m for the two compression temperatures at different strain rates

and subgrain development near the boundary. Rotation DRX (RDRX) [4, 15, 16] is a variation of CDRX where new grains form near grain boundaries as a consequence of intergranular strain incompatibilities. Regardless of the recrystallization mechanisms, the necklace structure where recrystallization occurs primarily at grain boundaries is frequently reported.

- (d) Grain boundary sliding (GBS) is an important deformation mechanism at elevated temperatures, especially in the fine-grained AZ31 alloy [17–19].
- (e) Strong texture distribution is formed during different deformation processes, e.g., tension [4], compression [3], rolling [20], and extrusion [21].

Most of the aforementioned research is concentrated on wrought AZ31 alloy (e.g., extruded rods, rolled sheets). The research on deformation mechanisms of cast AZ31 alloy is very limited. Compared with wrought AZ31, cast billets have larger grains and more random grain orientations [22]. Previous research on AZ31 alloy [23–26] revealed that the initial microstructure had significant influence on the deformation behavior. In this article, we described the microstructure evolution of cast AZ31 under uniaxial compression at different temperatures and strain rates. The stress–strain curves, microstructure development, and texture evolution were discussed.

Experimental Procedure

The material used in this study is cast AZ31 alloy; the alloy composition is listed in Table 1. The diameter of the direct chill (DC) cast billet is $\varnothing 100$ mm. To study the influence of sample locations on mechanical properties, cylindrical samples ($\varnothing 10$ mm $\times$ 15 mm) were machined from both the inner and the outer regions of the casting billet (Fig. 1). Isothermal compression was performed on a Gleeble 3500 mechanical testing system under different deformation temperatures (623 and 673 K) and strain rates (0.01, 0.1, and 1.0 s⁻¹), to the final strain of 1.0. To investigate the development of microstructure at various stages of deformation, compression tests at a strain rate of 0.1 s⁻¹ were terminated at selected strains, ranging from 0.2 to 1.0 at interval of 0.2. After completion of compression tests, the samples were immediately unloaded from the Gleeble and then quenched into water to arrest grain growth during cooling.

Specimens for light microscopy (LM) were mounted in epoxy resin, ground with 1200 grit SiC paper, mechanically polished using 6, 3, and 1 μ m diamond paste and 0.3, 0.05 μ m alumina solution. Specimens were then etched for 5 s in acetic picral (10 mL acetic, 10 mL distilled water, 70 mL picral).

The x-ray diffraction (XRD) texture measurement was conducted on a four-circle diffractometer equipped with a Cu tube, and Si(Li) detector with a “thin-film” collimator. Specimens for XRD were polished with 2000 grit SiC paper, followed by mechanically polishing with 3 μ m diamond paste. The observation plane was perpendicular to the compression axis. The pole figures were collected in

Fig. 8 Microstructure evolution with the increasing strain (temp. = 623 K, $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$). **a–d** Cross sections and **e–h** longitudinal sections. **a, e** $\varepsilon = 0.2$, **b, f** $\varepsilon = 0.4$, **c, g** $\varepsilon = 0.6$, and **d, h** $\varepsilon = 0.8$

5° increments: $0^\circ\text{--}\chi\text{--}80^\circ$, $0^\circ\text{--}\phi\text{--}355^\circ$. Background intensities were obtained by interpolating from points on either side of each reflection and net intensities were normalized to intensities obtained from a powder sample of Mg. To avoid introducing experimental noise and small deviations from the true random powder intensity, the pole intensities from the powder sample were first azimuthally averaged to produce a better estimation of the true defocusing and geometric effects. To avoid distortion of the pole intensities from irregularly shaped samples, all measurements (including from the random Mg powder) were confined to a 5 mm diameter region on the sample face using a mask made from Ag paint (Fig. 2). Pole figures were plotted using the software program popLA [27]. In the current research, the normal directions of pole figures are parallel to the compression axis.

Specimens for electron backscattered diffraction (EBSD) were polished with 2000 grit SiC paper, followed by mechanically polishing with 3 μm diamond paste and electro-polishing in AC2 electrolyte at 20 V for the final stage. Polished specimens were loaded into a scanning electron microscope equipped with a backscatter diffraction

stage. Diffraction at each point was used to automatically determine the crystallite orientation and the collection of orientations for the area sampled was used to calculate the orientation distribution.

Result and Discussion

Stress–Strain Curves

Figure 3 shows the measured $\sigma\text{--}\varepsilon$ curves from the uniaxial compression testing of the inner samples under different temperatures and strain rates. Flow stress increases as the temperature decreases under the same strain rates; flow stress also increases as the strain rate increases under the same temperature. Two metallurgical processes influence the flow stress: work hardening and work softening. Work hardening is induced by the increase of dislocation density resulting from plastic deformation. Two softening mechanisms, dynamic recovery (DRV) and DRX can alleviate dislocations and decrease flow stress. In DRV, the flow stress initially increases with strain before reaching a

Fig. 9 Microstructure evolution with the increasing strain (temp. = 673 K, $\dot{\epsilon} = 0.1 \text{ s}^{-1}$). **a–d** Cross sections and **e–h** longitudinal sections. **a, e** $\epsilon = 0.2$, **b, f** $\epsilon = 0.4$, **c, g** $\epsilon = 0.6$, and **d, h** $\epsilon = 0.8$

Fig. 10 Microstructure at the strain of 1.0 (temp. = 623 K). **a–c** Cross sections and **d–f** longitudinal sections. **a, d** $\dot{\epsilon} = 1.0 \text{ s}^{-1}$, **b, e** $\dot{\epsilon} = 0.1 \text{ s}^{-1}$, **c, f** $\dot{\epsilon} = 0.01 \text{ s}^{-1}$

Fig. 11 Microstructure at the strain of 1.0 (temp. = 673 K). **a–c** Cross sections and **d–f** longitudinal sections. **a, d** $\dot{\epsilon} = 1.0 \text{ s}^{-1}$, **b, e** $\dot{\epsilon} = 0.1 \text{ s}^{-1}$, **c, f** $\dot{\epsilon} = 0.01 \text{ s}^{-1}$

Fig. 12 Measured X_{DRX} at the strain of 1.0 under different deformation conditions (A 673 K, $\dot{\epsilon} = 0.01 \text{ s}^{-1}$; B 623 K, $\dot{\epsilon} = 0.01 \text{ s}^{-1}$; C 673 K, $\dot{\epsilon} = 0.1 \text{ s}^{-1}$; D 623 K, $\dot{\epsilon} = 0.1 \text{ s}^{-1}$; E 673 K, $\dot{\epsilon} = 1.0 \text{ s}^{-1}$; F 623 K, $\dot{\epsilon} = 1.0 \text{ s}^{-1}$)

constant value when the balance between work hardening and DRV is established. However, DRV is difficult to activate in magnesium alloys because of their low stacking fault energies (60–78 mJ/m² for pure magnesium [28]). In DRX, the flow stress also increases at the beginning of deformation and then decreases after reaching a peak stress when the equilibrium between the hardening and the softening [29] is realized. As shown in Fig. 3, most stress–strain curves exhibit a peak at the early stage of deformation, followed by a decrease in stress. Therefore, DRX should be an important softening mechanism for cast AZ31 alloy under the deformation conditions in this study.

The measured σ – ϵ curves from the uniaxial compression testing of the outer samples under different temperatures and strain rates (Fig. 4) show no significant difference from the inner samples. Previous research showed that the

stress–strain curves from the compression of AZ31 alloy were sensitive to the initial grain size, especially when that grain size favored a specific deformation mechanism [30]. Barnett et al. [1] characterized the flow stress as slip-dominated when the initial grain size is smaller than d_s , twinning dominated when the initial grain size is larger than d_t and a transition region bounded by these two grain sizes. Empirical models were established to estimate the grain size d_s and d_t

$$d_t = \left(\frac{0.15 \ln Z - 12.2}{73 - 3.8 \ln Z} \right)^2 \quad (1)$$

$$d_s = \frac{d_t}{3}, \quad (2)$$

where Z is the Zener–Hollomon parameter and can be expressed as

$$Z = \dot{\epsilon} \exp(Q/RT) \quad (3)$$

where $\dot{\epsilon}$ is the strain rate, R the gas constant, T the temperature (K), and Q the activation energy (estimated as 135 kJ/mol) [1]. The critical grain sizes of d_s and d_t are plotted as a function of Z in Fig. 5 based on Eqs 1 and 2. To determine the deformation mechanisms of the compression testing in this study, the initial grain sizes of the billet both at inner and outer locations were measured.

The microstructures of the casting billet (Fig. 6) show an average grain size of 181 μm for the inner region and 135 μm the outer region. For both the regions, the average grain size is large compared with those of wrought AZ31 alloy, which are usually between 5 and 50 μm [1, 9]. The billet grain sizes are

Fig. 13 (0002) pole figure of the sample strained to 0.2 after four polishing preparations

plotted in Fig. 5 with respect to parameter Z from different testing conditions. As shown in Fig. 5, the samples taken from the inner and outer regions of the billet have similar deformation mechanisms under the same compression condition, which can lead to similar σ - ϵ curves. As the sample location has no significant effect on the mechanical properties, the results from the outer part of the cast billet were employed for further investigation in this study.

To investigate whether GBS plays an important role in the deformation of cast AZ31 alloy, the following equation [31] was employed:

$$\sigma = K \dot{\epsilon}^m \quad (4)$$

where σ is the true flow stress, $\dot{\epsilon}$ is the true strain rate, m is the strain rate sensitivity exponent, and K is a constant incorporating the microstructure and temperature dependence of the flow stress. The variation of flow stress with strain rate is plotted in Fig. 7. The flow stress was determined at a fixed strain of 1.0. The slope m in Fig. 7 is about 0.1 for both temperatures. This value is much smaller than that reported in a previous research on extruded AZ31 alloy [32], where m was about 0.5. When the strain rate sensitivity exponent m reaches about 0.5, the materials deform

principally by GBS. A smaller value of m suggests the climb-controlled dislocation slip or twinning dominated deformation [33]. Thus, as distinct from fine-grained wrought AZ31 alloy, GBS is not the dominant mechanism in cast AZ31 alloy under the current testing conditions.

Microstructure Evolution

Figure 8 shows the microstructure evolution with increasing strain during compression testing at 623 K and 0.1 s^{-1} . Deformation twinning can be seen at strains as small as 0.2. The large grain is subdivided by twins and few dynamic recrystallized grains are observed at the grain boundary. As the strain is increased to 0.4, the necklace-type structure starts to form along the grain boundaries. Twinning-related DRX is apparent in Fig. 8(b) and as the strain is further increased to 0.6 and 0.8, a large amount of dynamic recrystallized grains appears. The necklace bands formed at earlier stages are broadened by the extensive DRX. Figure 9 shows the microstructure evolution with increasing strain during compression testing at 673 K and 0.1 s^{-1} . The microstructure evolution of samples strained at 673 K

Fig. 14 Texture distribution with multiple scans (temp. = 673 K, $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$). **a** $\varepsilon = 0$, 38 scans; **b** $\varepsilon = 0.2$, 26 scans; **c** $\varepsilon = 0.4$, 18 scans; **d** $\varepsilon = 0.6$, 15 scans; **e** $\varepsilon = 0.8$, 12 scans

shows similar characteristics as the samples strained at 623 K; however, the occurrence of twinning is much lower at low strain and the dynamic recrystallized grains are bigger at 673 K.

Grain growth is not significant as the strain increases at both temperatures. Sakai and Jonas [34] suggested that in the necklace structure of DRX, concurrent deformation caused work hardening to take place within the recrystallized grains. Therefore, the driving force for grain growth was gradually reduced and became ineffective when d_{DRX} reached the stable size, allowing new similar-size grains to form.

Figure 10 shows the microstructures at a strain of 1.0 for compression testing at 623 K but with different strain rates. The percentage of DRX (X_{DRX}) decreases with increasing strain rate. Higher strain rates provide less time for DRX to occur, and thus X_{DRX} is significantly suppressed by increasing the strain rate. Intensive shear localization is observed in Fig. 10(a) and (d), suggesting the formability is

restricted at high strain rates. The material is far from being completely recrystallized even at the lowest strain rate of 0.01 s^{-1} . The microstructures at the strain of 1.0 for compression testing at 673 K but with different strain rates are shown in Fig. 11. Shear localization is not significant at 673 K, but large grains are still visible, suggesting the microstructure is not fully recrystallized.

As is seen from Figs. 8 to 11, X_{DRX} increases with the strain during compression testing at the same temperatures and strain rates in this study. The original grains are almost completely surrounded by recrystallized grains at large strains. However, Beer and Barnett [9] suggest that further deformation may not lead to significant increase of X_{DRX} , which can be attributed to the deformation being accommodated in the recrystallized material. To study the influence of deformation conditions on X_{DRX} , quantitative metallographic measurements were conducted on samples after compression testing to a strain of 1.0 at different

Fig. 15 Azimuthally averaged (0002) and (10 $\bar{1}$ 0) pole figures determined from the multi-layer average of pole figures

temperatures and strain rates. A large area (50 mm²) was measured to ensure good statistical accuracy. By plotting the measured X_{DRX} against the Z parameter (Fig. 12), it can be seen that X_{DRX} increases with the increasing temperature for the same strain rate. For example, point A has a higher percentage of DRX than B when the deformation temperature is increased from 623 to 673 K. X_{DRX} also increases with decreasing strain rate at the same temperature. For example, point C has a higher percentage of DRX than E when the deformation strain rate decreases from 1.0 to 0.1 s⁻¹. A low strain rate provides more time and high temperature provides high boundary mobility for nucleation and grain growth [22].

Texture Evolution

The formation of basal texture has been reported by other researchers when studying compression tests of wrought AZ31. The textures were measured by in situ XRD [35], EBSD [36], and neutron diffraction [37]. As shown in Figs. 8–11, large grains are visible at all strain levels in this study. This can lead to poor statistical confidence when measuring the macro-texture distribution. The sample strained to $\varepsilon = 0.2$ was chosen to study the sampling statistics of texture distribution using XRD. The sample's (0002) pole figure was measured by XRD and then re-polished and re-measured four times. Figure 13 shows

Fig. 16 Grain orientation map on the cross section of a compressed sample by EBSD (temp. = 673 K, $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$, $\varepsilon = 0.4$)

the (0002) pole figure plotted by PopLA. XRD pole figures show significant graininess, characteristic of poor sampling statistics. Hence, statistical inference based on the single preparation is not valid in the cast AZ31 alloy.

In order to obtain a better estimation of the true average texture, the intensities from a series of layers revealed by polishing were averaged. Each successive sample was prepared by removing $\sim 200 \mu\text{m}$ of material to reveal a fresh surface for XRD analysis. The procedure was repeated until the average intensity becomes stable. The expected development of basal texture is apparent in Fig. 14 and consistent with previous research on the wrought AZ31 alloy [3, 35–37]. As indicated in Fig. 14, basal texture is formed at a small strain, with the maximum intensity developing when strain reaches 0.4, and no further enhancement is observed at larger strains.

The texture evolution can also be observed from the azimuthally averaged pole figures determined from the multi-layer average of pole figures shown above. The averaged intensities for two reflections versus tilt (χ) angle are plotted in Fig. 15. The results indicate little change in texture beyond strains of 0.2.

EBSD was employed to study the deformation and recrystallization texture of specimens strained to $\varepsilon = 0.4$. Figure 16 shows the orientation map determined by EBSD. To obtain better measurement statistics, eight different areas were examined with a total data acquisition time for each map of approximately 20 h. The total number of grains detected was 6533.

As is shown in Fig. 9, recrystallization grain sizes remain almost constant with strain. Measured by the intercept method [38], the average recrystallization grain size of the sample in Fig. 9(b) is about 10 μm . This value represents an

Fig. 17 Deformed texture and recrystallization texture of compressed sample (temp. = 673 K). **a** All grains, **b** deformed grains, and **c** recrystallized grains

average of ~ 1000 recrystallized grains. On the other hand, the average grain size in the as-cast sample is about $135\ \mu\text{m}$. The recrystallized grains are much smaller than the original grains. This suggests that grains detected by EBSD can be divided into two groups based on their sizes: Grains larger

than $20\ \mu\text{m}$ are assumed to be deformed grains, and the rest grains were assumed to be recrystallization grains.

According to the criterion described above, 274 grains out of 6533 grains are indexed as deformed grains, and the rest are recrystallized grains. The EBSD pole figures for each

class of grains are shown in Fig. 17: (a) all detected grains, (b) deformed grains, and (c) recrystallized grains. The “grainy” texture apparent in the EBSD pole figures is consistent with the large grain size observed in the LM images. Although the texture distribution of the deformed grains (b) is quite similar to in the total of all grains (a), the recrystallized grains (c) show basal texture, though with a much lower intensity. This can be attributed to the fact that the deformed grains are larger and, therefore, their texture distribution is dominated by deformed grains at small strains.

Summary

In this article, the mechanical behavior of cast AZ31 Mg alloy has been studied in uniaxial compression at multiple deformation conditions. The following conclusions are drawn:

1. The sample location on the casting billet has no significant effect on the mechanical properties. The examination of the variation in flow stress as a function of strain rate shows that GBS is not the dominant mechanism in cast AZ31 alloy.
2. Occurrence of DRX is confirmed by the evaluation of stress–strain curves and LM observation.
3. The poor statistics of the coarse-grained samples is overcome by averaging many pole figures obtained with successive layer removal. XRD shows that basal texture is formed at small strains. The maximum intensity develops at strains of 0.4 and no further enhancement is observed at larger strains.
4. The texture distribution at small strains is dominated by deformed grains.

Acknowledgments The authors from Shanghai Jiao Tong University would like to acknowledge the support of the Ford University Research Program, the National Natural Science Foundation of China (No. 51175335), Ministry of Education of China (No. 311017), Shanghai Science & Technology Projects (Nos. 10QH1401400, 10520705000, 10JC1407300). The authors also appreciate the supported of the Research Fund of State Key Laboratory of China (No. MSV-MS-2010-05).

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