Effect of Ca concentration on Microstructure formation behaviors in AZ61 magnesium alloy

K Kim¹ D Han² and K Kim¹²

¹ Department of Metallurgical Engineering, Pukyong National University, 45 Yongso-ro, Nam-gu, Busan 48513, Republic of Korea
² Marine Convergence Design Co-work, Pukyong National University, 45 Yongso-ro, Nam-gu, Busan 48513, Republic of Korea

mrppeng@pknu.ac.kr

Abstract. Magnesium alloys have been paid attention as lightweight materials in various industrial fields. However, their use is limited by poor formability at room temperature which was caused by the limited number of slip systems. The texture control plays an important role in plastic workability of magnesium. Addition of Ca as alloying element is known to improve the general corrosion resistance and mechanical integrity of magnesium alloys in chloride environment. In order to investigate the Ca concentration on microstructure formation behaviors, Ca addition on AZ magnesium alloy were experimentally investigated by high-temperature uniaxial compression. Uniaxial compression test was conducted at 673K and 723K with a strain rate of 5.0×10⁻²s⁻¹. The working softening was observed and the main component of texture and the sharpness of basal texture in two kinds of specimens varies depending on the deformation conditions.

1. Introduction

Magnesium alloys have been paid attention as lightweight materials in various industrial fields. However, their use is limited by room temperature poor formability, which was caused by the limited number of slip systems. In order to improve poor formability, it is important to understand the texture control in magnesium [1-2].

The researchers investigated the characteristics of texture formation behavior of AZ80 magnesium alloy during high-temperature deformation. They reported the main component and its sharpness of texture was varied depending on deformation condition [3]. Also, Park et al. have studied the effects of the solute element and its concentration on the texture formation behavior of AZ system magnesium alloys with different aluminum solute concentration. It was found that the main components and its sharpness of texture varied depending on deformation conditions and Al concentrations. Especially, basal texture was developed with an increasing of Al concentration and these texture were stable for deformation of that study [4].

Addition of Ca as alloying element has been known to improve the corrosion resistance and mechanical integrity of magnesium alloys in chloride environment [5-6]. The authors reported that AZ system magnesium alloys were formed second- phase by adding element Ca and these second-phases play an important role to improve their corrosion resistance and the mechanical properties. However, the study of the crystallographic microstructure change according to the solute content in AZ system magnesium alloy is insufficient.
In order to investigate the effect of Ca concentration on microstructure formation behaviors, AZ61 magnesium alloy and Ca addition on AZ61 magnesium alloy were experimentally investigated by high-temperature uniaxial compression. Uniaxial compression test is conducted at 673K and 723K with a strain rate of $5.0 \times 10^{-2}$ s$^{-1}$ and the microstructure is observed by the optical microscope.

2. Experimental procedure

The materials used in this study are a commercial AZ61 (Mg-6.0%Al-1%Zn wt. %) and AZX611 (Mg-6.0%Al-1%Zn-1%Ca wt. %) magnesium alloys. Table 1 shows chemical composition of AZ61, AZX611.

|         | Al    | Zn    | Ca    | Mn    | Mg    |
|---------|-------|-------|-------|-------|-------|
| AZ61    | 5.5-6.5 | 0.5-1.5 | -     | 0.15-0.4 | Bal.  |
| AZX611  | 5.8-7.2 | 0.4-1.5 | 1.0   | 0.15-0.5 | Bal.  |

These starting materials were hot-rolled at 673K with a rolling reduction of 30% and then machined out in such a way that the compression plane was parallel to rolling plane for uniaxial compression tests. The experimental specimens were annealed at 723K for 1h to produce a homogeneous microstructure. Figure 1 shows crystallographic microstructure in two kinds of specimens before high-temperature deformation. Figure 1 (a) shows initial microstructure of AZ61 and Figure 1 (b) shows initial microstructure of AZX611. As-cast Mg alloys formed second-phase particles such as Al-Ca compound and Mg$\text{17Al}_{12}$ around the grain boundaries. Depending on the types of second-phase particles, these second-phase particles disappear or exist after annealing. Figure 1 (a) shows removed second-phase particles in microstructure after annealing. Whereas, Figure 1 (b) shows second-phase particles exist on grain boundary after annealing. A recent study showed that Mg$\text{17Al}_{12}$ disappeared on annealing at temperature 673K to 723K. However, Al-Ca compound has high-melting point, which means that hardly dissolution. AZX611 shows uniform grain size and smaller microstructure size than AZ61 [8]. That means that microstructure formation was affected by second-phase particles.

![Figure 1](image_url)

**Figure 1.** The optical micrographs in two kinds of specimens before deformation: (a) AZ61 magnesium alloy, (b) AZX611 magnesium alloy
Figure 2. True stress - true strain curves for the deformation at 723K under a strain rate of $5.0 \times 10^{-2} \text{s}^{-1}$ up to a strain of -0.4.

Uniaxial compression tests were carried out at temperature 673K and 723K under a strain rate of $5.0 \times 10^{-2} \text{s}^{-1}$ up to a strain of -1.0. The direction of compression was marked after the rolling direction, and all specimens compressed same direction. These specimens were immediately quenched in water after deformation to prevent changes in the microstructure. The preparation of specimens is mid-plane by polishing. The surfaces were finished using as silica suspension. Moreover, electro-polishing carried out at 2% perchloric acid and 98% pure ethanol. After electropolishing immediately wash ethanol to prevent etching.

3. Result and discussion

Figure 3. Microstructure of specimen observed by Optical Microscopy. The deformation condition was up to the strain of -0.4 with the strain rate of $5.0 \times 10^{-2} \text{s}^{-1}$ and temperature at 723K. (a) AZ61 magnesium alloy. (b) AZX611 magnesium alloy.
3.1. Behavior of deformation

Figure 2 shows the true stress - true strain curves for the deformation at 723K under a strain rate of \(5.0 \times 10^{-2} \text{s}^{-1}\) up to a strain of -0.4 in AZ61 and AZX611 magnesium alloys. The flow stress of AZX611 was lower than that of AZ61, due to initial grain size and composition. All specimen observed work softening such as the deformation behaviour of AZ80 reported by Kim et al [3].

3.2. Behavior of microstructure

Figure 3 (a) and (b) show the grain structure at compression plane after deformation at 723K under a strain rate of \(5.0 \times 10^{-2} \text{s}^{-1}\) up to the strain of -0.4. As reported on the deformation behavior of AZ magnesium alloys, small grains and coarse grains simultaneously existed as shown in Fig. 3 (a). However, Figure 3 (b) shows different microstructure formation behavior. It was found that the second-phase particles and fine grains are mostly distributed in the microstructure and second-phase particles are formed around fine grains.

Figure 4 (a) and (b) show the grain structure at compression plane after deformation at 723K under a strain rate of \(5.0 \times 10^{-2} \text{s}^{-1}\) up to the strain of -0.7. The microstructure formation behaviors in Fig.5 show similar formation behaviors as shown in Fig.4. Also, the grain size decrease with increasing of a strain in two kinds of specimens.

Figure 5 shows the grain structure at compression plane after deformation at 723K under a strain rate of \(5.0 \times 10^{-2} \text{s}^{-1}\) up to the strain of -1.0. After deformation, it shows different microstructure formation behaviors at figure 5 (a) and (b). Figure 5 (a) shows fine and uniform grain microstructure, and it was found confirmed that grain size of AZ61 magnesium alloy decreases with increasing of a strain. This means that dynamic recrystallization occurred as reported by Kim et al [3].

Figure 5 (b) shows mixed fine grain and large grain microstructure. Moreover, as compared with figure 5 (a), fine grain was smaller, and second-phase particles are surrounding fine grains. As reported in recent study, second-phase particles stimulates nucleation and recrystallization [8]. That is, Ca addition affected on dynamic recrystallization of AZ magnesium alloys and different formation mechanisms.

Figure 4. Microstructure of specimen observed by OM measurement. The deformation condition was up to the strain of -0.7 with the strain rate of \(5.0 \times 10^{-2} \text{s}^{-1}\) and temperature at 723K. (a) AZ61 magnesium alloy. (b) AZX611 magnesium alloy.
Consequently, microstructure formation behavior of AZ61 magnesium alloy is formed and developed by dynamic recrystallization as reported Park et al [4], whereas AZX611 magnesium alloys are affected by particle stimulated nucleation (PSN) in this study.

Figure 6 shows the variation of mean grain size with true strain in two kinds of specimens. White symbol is AZ61 magnesium alloy and black symbol is AZX611 magnesium alloy. The grain size was decreasing with true stain increase. That means dynamic recrystallization was occurred in AZ61 and AZX611 magnesium alloy. However, it is necessary to study that Al-Ca compounds affect dynamic recrystallization.

4. Conclusion

In order to investigate the effect of additional Ca on microstructure formation behaviour of AZ61 and AZX611 magnesium alloy during high-temperature deformation, AZ61 and AZX611 magnesium alloy were experimentally studied by uniaxial compression. The major results are as follows.
(1) Dynamic recrystallization occurs at AZ61 and AZX611 magnesium alloys in deformation conditions of this study.
(2) Two kinds of specimens shows different formation behaviors.

Acknowledgments
This work was supported by the Marine Convergence Design Co-work of Busan Korea 21 PLUS and the National Research Foundation of Korea grant funded by the Korea government (No.2017R1C1B5076690)

References
[1] Mordike B and Ebert T 2001 Mater. Sci. Eng. A 302 37
[2] Roberts C S 1960 Magnesium and its alloys (New York: Wiley)
[3] Kim K, Okayasu K and Fukutomi H 2015 Mater. Trans. 56 17
[4] Park M, Park H, Choi J and Kim K 2016 Mater. Sci. Forum 879 1449
[5] Beak S, Kim H, Jeong H, Sohn S, Shin H, Choi K, Lee K, Lee J, Yim C, You H, Ha H and Park S 2016 Corrosion Science 112 44
[6] Hakamada M, Watazu A, Saito N and Iwasaki H 2010 Mater. Sci. Eng. A 527 7143
[7] Xie B, Zhang B, Ning Y and Fu Y W 2019 Alloy and Compounds 786 25
[8] Xu S W, Kamado S and Honma T 2010 Scripta. Met. 63 293