## Article

# Investigating the Microstructure, Crystallographic Texture and Mechanical Behavior of Hot-Rolled Pure Mg and Mg-2Al-1Zn-1Ca Alloy 

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Citation: Chaudry, U.M.; Hamad, K.; Jun, T.-S. Investigating the Microstructure, Crystallographic Texture and Mechanical Behavior of Hot-Rolled Pure Mg and
Mg-2Al-1Zn-1Ca Alloy. Crystals 2022, 12, 1330. https://doi.org/10.3390/ cryst12101330

Academic Editor: Christiane Scharf

Received: 26 August 2022
Accepted: 17 September 2022
Published: 21 September 2022
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#### Abstract

In this study, the microstructure, crystallographic texture and the mechanical performance of hot-rolled pure Mg and $\mathrm{Mg}-2 \mathrm{Al}-1 \mathrm{Zn}-1 \mathrm{Ca}$ (herein inferred as AZX 211 ) were thoroughly investigated. The results showed that the designed AZX211 alloy exhibited an exceptional strength/ductility synergy where an almost $40 \%$ increase in ductility was received for AZX211. The microstructural characterization revealed the grain refinement in the AZX211, where the grain size was reduced by more than $50 \%(24.5 \mu \mathrm{~m}, 10 \mu \mathrm{~m}$ for the pure Mg and the AZX 211 , respectively). Moreover, a discernible number of precipitates were dispersed in the AZX211, which were confirmed to be (Mg, $\mathrm{Al})_{2} \mathrm{Ca}$. The pure Mg showed a conventional strong basal texture while a significantly weakened split basal texture was received for the AZX211. The fraction of basal-oriented grains was $21 \%$ for the pure Mg and $5 \%$ for the AZX211. The significant texture weakening for the AZX211 can be attributed to the precipitation and co-segregation that triggered the preferential evolution of the non-basal grains while impeding the growth of the basal grains. This was also confirmed by the crystal orientation and the pseudo-rocking curves. The higher ductility of the AZX211 was explained based on the texture softening and Schmid factor for the basal and non-basal slip systems.


Keywords: magnesium; AZ alloys; grain refinement; Texture; EBSD

## 1. Introduction

Magnesium and alloys (especially the AZ series) have been widely utilized in the transportation sector owing to its high specific stiffness and low density [1]. However, the key bottleneck to the diverse applications lies in its inferior ductility/formability and strong anisotropy at room temperature [2-5]. The availability of the limited number of slip systems to accommodate the shear strain during deformation poses challenging barriers in the path of highly ductile Mg. Moreover, the emergence of the strong basal texture ([0001]/ /normal direction) during primary processing (rolling or extrusion) is also liable for the intrinsic brittleness in Mg due to the low resolved shear stress based on Schmid's law [6-8]. Hence, tremendous efforts have been devoted to overcome these issues, which has consequently hampered the cost-effective production of Mg sheets. During the last decade, grain refinement has attracted more attention for enhancing the strength and ductility of Mg alloys in view of the Hall-Petch relationship ( $\sigma_{\mathrm{y}}=\sigma_{0}+\mathrm{kd}^{\mathrm{n}}$ ) [9,10]. In addition, many strategies were developed to enhance the mechanical performance of Mg by weaken the strong basal texture (e.g., alloying, severe plastic deformation, secondary processing) [11-13]. Precipitation and solute segregation have also reported to be an efficient strategy to tweak the basal texture during recrystallization. Hence, common approaches being utilized are intelligent alloying, microstructure optimization and texture modification [14-16].

It has been reported previously that the addition of rare earth (RE) elements can significantly improve the performance of Mg due to texture weakening, grain refinement and activating the additional slip systems [17,18]. However, the relatively high cost and the limited resources of RE elements have encouraged researchers to explore alternate strategies to achieve this target. Calcium $(\mathrm{Ca})$ is reported to be the most attractive candidate owing to low cost, adequate solid solubility and large atomic size equivalent to RE elements [19-21]. Han et al. evaluated the compressive behavior of the $\mathrm{Mg}-0.5 \mathrm{Ca}$ at room and cryogenic temperature $\left(-150{ }^{\circ} \mathrm{C}\right)$ [22]. The results showed that the $\mathrm{Mg}-0.5 \mathrm{Ca}$ showed a significant increase in the ductility at both deformation temperatures as compared to the pure Mg , which was associated with the texture softening in the Mg-0.5Ca. Recently, Liu et al. fabricated the $\mathrm{Mg}-0.3 \mathrm{Ca}$ by low-temperature extrusion [23]. The ultrafine grained (grain size $\sim 0.7 \mu \mathrm{~m}$ ) binary alloy displayed excellent strength/ductility synergy (yield, tensile strength and elongation of $361 \mathrm{Mpa}, 370 \mathrm{Mpa}$ and $10.6 \%$, respectively). The exceptional increase was mainly due to the solute strengthening effect of the Ca segregation at the grain boundaries, which acted as an energy barrier to the dislocation emission from the grain boundaries resulting in the increased flow stress for the dislocation nucleation. Several studies also investigated the effect of Ca in the tertiary alloys, especially the AZ series. The AZ31-0.5Ca has demonstrated excellent mechanical performance, which was attributed to the weakening of the strong basal texture, the grain refinement due to precipitation and decreasing the critical resolved shear stress gap between the basal and non-basal slip systems [24]. Moreover, the AZ31B-0.3Ca exhibited excellent properties due to the precipitation of $\mathrm{Al}_{8} \mathrm{Mn}_{5}, \mathrm{Al}_{2} \mathrm{Ca}$ and co-segregation of $\mathrm{Al}, \mathrm{Zn}$ and Ca [25]. Elemental cosegregation hampered the basal grains' growth and accelerated the preferential growth of the non-basal grains with random orientations. Several literatures are available on the Ca-based AZ31 alloys, but studies on the effect of Ca on the AZ21 alloys are scarce. Hence the purpose of this study was to investigate the effect of multiple solute additions on the microstructural and textural characteristics of pure Mg .

## 2. Experimental

The hot-rolled pure Mg and the $\mathrm{Mg}-2 \mathrm{Al}-1 \mathrm{Zn}-1 \mathrm{Ca}$ (AZX211) alloy sheets used in this work were provided by POSCO (Pohang Iron and Steel Co., Ltd., South Korea). The homogenization treatment ( $400^{\circ} \mathrm{C}$ for 12 h ) was performed on the multi-pass hot rolled $\left(300^{\circ} \mathrm{C}\right)$ cast strips with 1 mm thickness. The rolling schedule included three passes of $30 \%$ reduction with a rolling speed of 5 rpm . For microstructure characterization, optical microscopy (OM, Carl Zeiss, Oberkochen, Germany), scanning electron microscopy (SEM, SU-5000 Hitachi, Tokyo, Japan), integrated with energy-dispersive X-ray spectroscopy and electron backscattered diffraction (EBSD), were utilized. The samples were cut from a rolling transverse direction (RD-TD plane). The samples were prepared by the conventional metallographic procedures and were etched with acetic picral solution consisting of 4.2 g picric acid, 10 mL acetic acid, 10 mL distilled water and 70 mL ethanol $[26,27]$. For identifying the intermetallic particles dispersed in the matrix, energy-dispersive X-ray spectroscopy (EDX, JSM-7800 F, JEOL, Tokyo, Japan) was employed. Moreover, EBSD was utilized to investigate the crystallographic orientation information of the as-received samples. A cross-sectional polisher (IM4000, Hitachi, Tokyo, Japan) was used to prepare the samples for the EBSD. The samples were analyzed with the map size of $1000 \times 1000 \mu^{2}$ with the step size of $1.5 \mu \mathrm{~m}$. The received EBSD crystallographic data were analyzed by TSL OIM (v8.6) software (Silicon Valley, CA, USA). For mechanical characterization, the dog-bone samples with a gauge length of 25 mm were sectioned along the rolling direction (RD) from the rolled plates of both materials via electrical discharge machine (EDM) (Seoul, South Korea), and room temperature tensile tests were performed on the universal testing machine (UTM, RB 301 UNITECH-T, R\&B) at the strain rate of $10^{-3} \mathrm{~s}^{-1}$ based on the ASTM-E8 standard.

## 3. Results and Discussion

Figure 1 illustrates the EBSD inverse pole figure (IPF) map and the grain size distribution of both samples. As seen from Figure 1a,b, both samples exhibited an equiaxed twin-free microstructure that resulted from the dynamic recrystallization mechanism during the rolling process. The pure Mg showed a heterogeneous microstructure where some of the grains with a much coarser grain size were also detected. On the other hand, the AZX211 has a homogeneous microstructure with a fine grain size with random orientations. The average grain size of 24.5 and $10.1 \mu \mathrm{~m}$ was noticed for the pure Mg and the AZX211, respectively (Figure 1c). It has been already established that the addition of Ca can effectively refine the grain size of Mg due to the formation of the thermally stable intermetallic particles $\mathrm{Al}_{2} \mathrm{Ca}, \mathrm{Mg}_{2} \mathrm{Ca},(\mathrm{Mg}, \mathrm{Al})_{2} \mathrm{Ca}$ [28]. The grain refinement in the AZX211 can be anticipated to result from the pinning imposed by the intermetallic particles on the grain boundaries to restrict the grain growth. The kernel average misorientation (KAM) maps and geometrically necessary dislocation (GNDs) maps for both samples are provided in Figure 2. It is already known that the KAM values account for the localized deformation/local lattice distortion. The AZX211 showed higher values for KAM and GNDs density $\left(0.74,160 \times 10^{12} \mathrm{~m}^{-2}\right)$ as compared to the pure $\mathrm{Mg}\left(0.43,66 \times 10^{12} \mathrm{~m}^{-2}\right)$, which can be attributed to the back stresses generated due to the pinning effect imposed by the intermetallic particles.


Figure 1. Inverse pole maps (IPF) of (a) pure Mg and (b) AZX211 and (c) grain size distribution of both the samples.

To detect and identify the intermetallic precipitates dispersed in the AZX211, the SEM and elemental analysis through the EDS measurement is provided in Figure 3. As shown in Figure 3a, the significant number of the disconnected network of particles with different sizes was observed in the microstructure. The EDS analysis confirmed that these particles are composed of $\mathrm{Mg}, \mathrm{Al}$ and Ca , which is persistent with the already published literature (Table 1).

Table 1. EDS elemental analysis of phase.

| Elements | Concentration | wt. $\%$ | Atomic. $\%$ |
| :--- | :--- | :--- | :--- |
| $\mathbf{M g}$ | 28.12 | 47.22 | 53.02 |
| $\mathbf{A l}$ | 10.93 | 33.37 | 33.76 |
| $\mathbf{C a}$ | 8.83 | 19.41 | 13.22 |



Figure 2. Kernel average misorientation (KAM) and geometrically necessary dislocation maps for (a) pure Mg and (b) AZX211.

Moreover, no $\beta-\mathrm{Mg}_{17}-\mathrm{Al}_{12}$ phase was detected, indicating that this phase was completely dissolved by the homogenization treatment; in addition, the co-segregation of Al , Zn and Ca was also witnessed, as shown in Figure 3d-f.

It is already established that the intermetallic particles and co-segregation can restrict the grain boundary mobility during recrystallization, leading to grain refinement and also potentially triggering the preferential grain growth of the non-basal grains resulting in random oriented grains, which can be seen in Figure 1b [28]. Moreover, the preferential growth of the non-basal grains contributes strongly to the texture modification; hence, it would be interesting to see the texture characteristics of both samples.

Figure 4 provides the texture in terms of the pole figure (PF), the inverse pole figure (IPF) and the orientation distribution function (ODF) of both samples. The pure Mg showed the typical basal oriented (c-axes//ND) texture, where most of the grains were oriented along the (0001) with the basal intensity of 20.78 mrd . On the other hand, the AZX211 showed broadening along the RD and the split basal texture along the TD with a significant lower intensity of the basal poles ( 4.90 mrd ). Moreover, the non-basal texture components in the AZX211 can be rationalized by the ODF intensity distribution along $\varphi_{1}$ and $\Phi$ in the reduced Euler space ( $\varphi_{2}: 0^{\circ}, \varphi_{1}: 0-90^{\circ}$ and $\Phi: 0-90^{\circ}$ ). As can be clearly seen, the AZX211 showed a much weaker basal intensity along $\varphi_{1}$ and more broadening of the basal poles along $\Phi$.


Figure 3. (a) SEM showing the precipitates distributed in the matrix, (b) OM, (c) EDS and (d-f) elemental mapping of $\mathrm{Al}, \mathrm{Ca}$, and Zn distributed in the AZX211.


Figure 4. (a) Texture characteristics including pole figure (PF), inverse pole figure (IPF), orientation distribution function (ODF) and schematics of texture distribution in (a) pure Mg and (b) AZX211.

In addition, the crystal plasticity simulation has revealed that the broadening of the basal pole towards the RD is related to the activation of the pyramidal <c+a> slip system, while the spread towards the TD is associated with the orientation changes of the matrix grains due to twinning in addition to the enhanced activity of prismatic <a> slip [29]. As explained above, the texture plays an important role in accommodating the strain during deformation, and texture weakening usually results in superior mechanical properties in Mg alloys in accordance with the Schmid factor law. Pei et al. thoroughly investigated the synergetic effect of Ca alloying on microstructure and texture evolution during recrystallization and grain growth of the AZ31B [28]. The results revealed that the precipitates and co-segregation of $\mathrm{Al}, \mathrm{Zn}$ and Ca retarded the recrystallization nucleation due to the strong interaction of Ca atoms and the vacancies resulting in the pinning of the basal-oriented grains during recrystallization leading to texture weakening.

In order to examine how texture evolves during primary processing, the IPF of the partitioned basal grains, the crystal orientation and the pseudo-rocking curves of the pure Mg and the AZX211 are provided in Figure 5. The partitioned maps of the basal-oriented grains revealed the evolution of low fraction of the basal-oriented grains in the AZX211 (5\%) as compared to the pure Mg ( $21 \%$ ) (Figure 5a,b). Moreover, the pure Mg showed substantial fraction of grains with their c-axis tilt 10-20 away from ND, while the AZX211 displayed a much broader distribution of crystal orientations to the higher tilt angles, as can be seen from Figure 5c. Similar results were confirmed from the pseudo-rocking curve provided in Figure 5d, where more fraction of grains were oriented up to $20^{\circ}$ to the perfect $<0001>$ crystallographic orientation in the pure Mg , while on the other hand the AZX211 showed much broader distribution randomized texture. These results are consistent with the IPF maps provided in Figure 1b, where more evolution of the randomized grains was witnessed for the AZX211 and also with the PF provided in Figure 4b showing the split basal texture with reduced intensity. The less evolution of the basal grains and the high fraction of the grains with randomized orientations in the AZX211 can be attributed to the pinning of the grain boundary mobility by the precipitates and elemental co-segregation restricting the growth of the basal-oriented grains, consequently weakening the texture. Zeng et al. studied the texture evolution during the static recrystallization in the $\mathrm{Mg}-\mathrm{Zn}$, $\mathrm{Mg}-\mathrm{Ca}$ and $\mathrm{Mg}-\mathrm{Zn}-\mathrm{Ca}$ alloys using quasi-in situ EBSD [30]. In binary alloys, the weak recrystallization texture was formed at the early stages of the recrystallization, which was gradually replaced by the strong basal texture due to the preferential growth of the recrystallized grains. However, the ternary alloy showed the randomly oriented recrystallized grains, which were attributed to the solute segregation effect of Zn and Ca on the high energy grain boundaries, enhancing the solute drag effect leading to the uniform growth of the recrystallized grains with random orientations, which is the case in this study.

Figure 6 provides the image quality maps superimposed by the grain boundaries $\left(0^{\circ}-90^{\circ}\right)$ and misorientation distribution profile for both samples. The misorientation between $0^{\circ}-20^{\circ}$ is regarded as the low angle grain boundary (LAGB), while misorientation larger than $20^{\circ}$ is considered as the high angle grain boundary (HAGB).

Moreover, the grain boundaries with the misorientation angle $\left(80^{\circ}-90^{\circ}\right)$ are related to the $\{10-12\}$ tension twinning. The AZX211 showed higher evolution of the low angle grain boundaries (LAGBs) $\left(0^{\circ}-20^{\circ}\right)$ where the total length was measured to be 5.35 cm and 21.12 cm for the pure Mg and the AZX211, respectively (Figure 5a,b), which was also confirmed by the misorientation distribution profile in Figure $6 \mathrm{c}, \mathrm{d}$. The higher evolution of LAGBs in the AZX211 can be associated with the inhomogeneous deformation of the matrix surrounding the intermetallic particles $(\mathrm{Mg}, \mathrm{Al})_{2} \mathrm{Ca}$ during the primary processing. In addition, the misorientation angle distribution confirmed the twin (tension twins $\sim 86^{\circ}$, compression twins $56^{\circ}$, double twins $38^{\circ}$ ) free microstructure, which was consistent with the IPF maps provided in Figure 1. It is well known that the grain boundary misorientation has been reported to affect the deformation transfer; for instance, if the slip plane in the neighborhood grains share a common intersection line at the grain boundary with the collinear Burger vectors, then the dislocation in one grain can pass unpinned through the
grain boundary to the neighboring grain [31]. Moreover, the grain boundaries below $25^{\circ}$ can allow the direct transmission of the twin while the HAGBs poses strong barriers [32].


Figure 5. Partitioned IPF maps for basal-oriented grains in (a) pure Mg , (b) AZX211, (c) crystal direction and (d) pseudo rocking curves for both the samples.

To elucidate the effect of the grain refinement and the texture weakening on the mechanical properties of both samples, uniaxial room temperature tensile tests were carried out. Figure 7a provides the representative true stress-true strain curve of the pure Mg and the AZX211 at the strain rate of $10^{-3} \mathrm{~s}^{-1}$ along the RD. The pure Mg showed slightly higher yield strength (YS) ( $110 \pm 1.4 \mathrm{MPa}$ ) and ultimate tensile strength (UTS) $(262 \pm 2.3 \mathrm{MPa})$ as compared to the AZX211 ( $102 \pm 1.6 \mathrm{MPa}$ (YS) and $251 \pm 1.9 \mathrm{MPa}$ (UTS)) while a higher elongation to failure was recorded for the AZX211. An exceptional $50 \%$ increase in the ductility was noticed for the AZX211 ( $16.4 \% \pm 0.24$ and $10.9 \% \pm 0.17$ for the AZX211 and the pure Mg , respectively). The superior ductility received for the AZX211 can be attributed to the texture weakening as discussed in the above section. Moreover, to understand the ductility effect, the work-hardening rate $(\theta)$ from the true stress-strain curve was evaluated and displayed in Figure 7 b , which could be quantified as the negative slope of the curve, where the lower values correspond to the higher work-hardening capability. The workhardening rate can be divided into three distinct stages: in stage A, a sharp decrease in the
work-hardening rate was witnessed for both the materials as the strain was increased. In stage $B$, the decrease in the strain-hardening rate was slightly slowed down and eventually in stage $C$, the work hardening rate decreased again. As seen from the Figure 7b, the AZX211 showed the lower strain-hardening behavior as compared to the pure Mg , where the strain-hardening exponent (n) was evaluated to be 0.39 and 0.24 for the pure Mg and the AZX211, respectively. Usually, the grain refinement results in the superior YS according to the Hall-Petch relation $\left(\sigma_{y}=\sigma_{0}+\mathrm{kd}^{\mathrm{n}}\right)$; however, in the present study, the AZX211 showed a slightly lower yield strength despite having a lower grain size as compared to the pure Mg . The yield strength has also been reported to depend on the Schmid factor (SF) of the basal slip $\left(\mathrm{m}_{\mathrm{s}}\right)$ described in [33]:

$$
\sigma_{\mathrm{s}}=\tau / \mathrm{m}_{\mathrm{s}}
$$

where $\tau$ is the critical resolved shear stress for the basal slip. It can be inferred from the equation that the high SF for the basal slip can lead to inferior ductility as more soft grains can accommodate the deformation at the earlier stages. The SF for the basal and non-basal (prismatic <a>, pyramidal <a> and pyramidal <c+a>) slips for both the materials are shown in Figure $6 \mathrm{c}, \mathrm{d}$. The AZX211 has the higher SF for the basal as compared to the pure Mg, which was the reason behind the slight decrease in the YS. Moreover, the higher elongation of the AZX211 can be attributed to the higher SF of non-basal slip system, where the deformation can be accommodated by the prismatic $<a>$ and pyramidal $<c+a>$ slip system.


Figure 6. Image quality maps superimposed with the grain boundaries rotational angles for (a) pure Mg , (b) AZX211, (c) and (d) Misorientation distribution profiles for both samples.


Figure 7. (a) True stress-strain curves (Inset is tensile sample dimensions) and (b) strain-hardening curves for pure Mg and AZX211, Schmid factor of basal and non-basal slip systems of (c) pure Mg and (d) AZX211.

## 4. Conclusions

In this study, microstructure, crystallographic texture and mechanical performance of the pure Mg and the AZX211 alloy were critically investigated. Both materials showed a twin-free equiaxed microstructure, while the grain refinement was witnessed in the AZX211 ( $24.5 \mu \mathrm{~m}$ and $10.1 \mu \mathrm{~m}$ for pure Mg and the AZX211, respectively). Moreover, the pure Mg showed the strong basal texture with a texture intensity of 20.78 mrd , while on the other hand the AZX211 had the split basal texture with a weak intensity of 4.90 mrd . The texture weakening in the AZX211 was attributed to the presence of the intermetallic particles in the matrix, which restricted the preferential growth of the basal-oriented grains resulting in the randomized texture. Furthermore, the crystal orientation and pseudo-rocking curve confirmed the texture modification where the pure Mg showed the maximum distribution within $20^{\circ}$ away from the perfect basal orientation, while the AZX211 showed more the uniform distribution for the higher tilt angles. The uniaxial tensile tests revealed a $\sim 40 \%$ increase in elongation to failure for the AZX211 as compared to the pure Mg , which was attributed to the substantial texture weakening and high SF for the non-basal slip system.

Author Contributions: Conceptualization, U.M.C., K.H. and T.-S.J.; methodology, U.M.C.; formal analysis, U.M.C.; investigation, U.M.C. and T.-S.J.; resources, K.H. and T.-S.J.; data curation, U.M.C.; writing-original draft preparation, U.M.C.; writing-review and editing, T.-S.J.; supervision, K.H. and T.-S.J.; funding acquisition, T.-S.J. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korean government (MSIT) (No. 2020R1C1C1004434).

Institutional Review Board Statement: Not Applicable.
Informed Consent Statement: Not Applicable.

Data Availability Statement: Data are contained within the article.
Conflicts of Interest: The authors declare no conflict of interest.

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