Effect of pre-twinning on microstructure and texture evolution in room and cryogenically deformed Mg-0.5Ca alloy

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Abstract Introducing \{10-12\} tension twin lamellas has proved to be the most effective strategy to enhance the performance of Mg alloys. Twins can provide the preferential nucleation sites for recrystallization, hence twinning induced grain refinement can result in higher strength material based on Hall-Petch Effect. In addition, twinning can also modify the crystallographic c-axis distribution in wrought Mg alloys resulting in lower plastic anisotropy. Accordingly, in the present work, the twinning evolution in the Mg-0.5Ca (wt.%) alloy at room and cryogenic temperature was critically investigated. The samples were subjected to pre-compression (5%) at room temperature and -150 °C at the strain rate of 10⁻³ s⁻¹. The microstructural and textural characterization was carried out using OM, SEM and EBSD to emphasize the twinning evolution during compression at room and low temperatures. The local lattice distortion and the localized deformation in terms of Kernel average misorientation (KAM) was also studied. The results revealed the significant grain refinement (13.7 μm, 5.61 μm and 6.02 μm of uncompressed and RT, -150 °C pre-compressed, respectively), where slightly enhanced twinning fraction in the sample compressed at RT was observed. Finally, remarkable increase in the yield strength was noticed in the pre-twinned samples, which was attributed to the twinning-induced grain refinement based on the Hall-Petch relationship.

1 Introduction

Owning to their low density, high specific strength and exceptional recyclability, Magnesium (Mg) and its alloys have been considered as promising candidate for weight reduction in aerospace and automotive industries [1]. Unfortunately, the diverse application of Mg alloys has been impeded due to its low intrinsic ductility at room temperature (RT), which can be attributed to the availability of limited slip systems during plastic deformation, owning to its hexagonal close packed (HCP) structure. As per von mises criteria, minimum of five slip systems needs to be available to favor the uniform plastic deformation in polycrystalline material. However, the plastic deformation in Mg is only governed by the basal $<a>$ \{0001\}.

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<10-20> dislocation slip and tensile twinning {10-12} <10-11> due to their low critical resolved shear stress (CRSS) [2]. Moreover, the strong basal texture evolved after the primary processing (e.g., rolling, extrusion) is also considered as a bottleneck resulting in plastic anisotropy.

Introducing {10-12} <10-11> tension twin lamellas in the microstructure of Mg alloys has been suggested to be a simple yet very effective approach to enhance the RT strength and formability of Mg alloys [3]. Moreover, twinning can alter the texture by the reorientation of numerous grains by 86° to the parent grain due to the stress energy and changes the crystal orientation. {10-12} <10-11> tension twins can be operated at the early stages of deformation due to low CRSS (2-5 MPa). Previous studies have revealed that the twinning activity is strongly dependent on the loading direction, deformation temperature, strain rate, total strain and initial characteristics of the material (grain size, texture, second phase particle) [4]. The microstructural evolution, texture randomization and the final mechanical properties can be attributed to the twinning volume fraction, twinning patterns and twinning type. The major outcomes of the role of twinning in the deformation of Mg alloys can be summarized as: (i) accommodating plastic strain in association of stress relaxation effect; (ii) refinement hardening due to grain refinement by the twin boundaries based on the Hall-Petch relationship; (iii) twinning induced texture weakening due to the reorientation of parent grain; and (iv) twin boundaries strengthening effect [5]. Song et al have concluded that the {10-12} twin boundaries can enhance the yield strength of AZ31 magnesium alloy by refinement hardening [6]. AZ31 plate was pre-rolled to 3% and annealed at 200 °C for 6h to remove the dislocations but retain the twinned structure. The tension and compression yield strength of pre-rolled alloy was increased by 34 MPa and 49 MPa, respectively, as compared to the initial plate. In addition, the twinned structure and texture is reported to enhance the mechanical properties of Mg alloys during severe plastic deformation in which multidirectional stress states are induced [7]. In summary, the performance of Mg alloys can be significantly enhanced by introducing twins in the microstructure. The twin characteristics including, twin morphology, twin variant selection, total twin volume fraction and twin-induced texture are suggested to be function of stress state and/or deformation temperature. Hence it is important to understand the twinning evolution under the specific conditions.

In the present study, accordingly, the evolution of {10-12} <10-11> tension twinning in the Mg-0.5Ca alloy was systematically studied. Mg-0.5Ca alloy was pre-compressed (5%) along rolling direction at the strain rate of 10^{-3} s^{-1} at RT and cryogenic temperature (CT) to induced tension twins in the microstructure. A detailed microstructural and textural comparison of twinned structure produced at RT and CT is carried out. Finally, the activation of tension twin variants and its effect on the mechanical properties was thoroughly investigated.

2 Materials & Methods

Hot-rolled and consequently homogenized plates of Mg-0.5Ca (wt.%) was used in the present study. For the characterization, the samples were cut from the plate by electro-discharge machine (EDM). Samples were prepared using standard metallography techniques. Optical microscope (OM) with polarized filter (Carl Zeiss, Axio Scope.A1) was used to characterize the microstructure. Scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX, JSM-7800F, JEOL). was utilized for the detection and compositional analysis of the presence of any second phase particles in the matrix. The uniaxial compression tests at RT and CT (-150 °C) were carried out using universal testing machine (UTM, RB 301 UNITECH-T, R&B) at the strain rate of 10^{-3} s^{-1}. The cryogenic tests were performed in
a chamber circulating the liquid nitrogen. To investigate the twinning evolution at both the temperatures, samples were compressed to 5% at both the temperatures. From now on, the samples pre-compressed at RT and CT will be named as PC-RT and PC-CT, respectively. Finally, SEM equipped with electron backscattered diffraction (EBSD) system (VelocityTM Super, EDAX) was used to obtain the detailed crystallographic information of as-received, PC-RT and PC-CT samples. OIM Analysis v8.6 software was utilized to analyze the EBSD scanned data.

3 Results and Discussion

Fig. 1 (a) shows the OM image of the initial microstructure of Mg-0.5Ca alloy under the polarized light filter. The distribution of homogeneous and equiaxed grains can be observed. SEM micrograph shown in Fig. 1 (b) revealed the noticeable amount of white precipitates, which were distributed throughout the Mg matrix. These particles were determined to be consist of Mg and Ca as illustrated by EDS mapping (see Fig. 1 (c) and (d)). This SEM/EDS analysis is well consistent with the previous works, which suggest the formation of Mg$_2$Ca intermetallic particles in the Mg-Ca alloys [8].

![Fig. 1. Optical (a) and SEM micrograph with EDS spectra (b) of as received Mg-0.5Ca, (c) and (d) EDS elemental mapping for Mg and Ca, respectively.](image)

The microstructure of as-received Mg-0.5Ca alloy was characterized by EBSD to further investigate the crystallographic orientation and texture characteristics. The image quality (IQ) and inverse pole figure (IPF) maps were found to be consistent with the OM, where equiaxed twin-free microstructure was observed (see Fig. 2 (a)). Few black precipitates (as shown by the yellow arrows in the IQ map) were dispersed in the microstructure, which are anticipated as Mg$_2$Ca precipitates. A typical rolling type texture with maximum intensity 9.89 mrd can be witnessed from Fig. 2 (b). Mostly, the grains were found to be aligned parallel to ND with few peaks at 15°-30° along TD. The grain size distribution of as-received Mg-0.5Ca is presented in Fig. 2 (c). The average grain size was found to be 13.7 µm with few very coarse grains of 70 µm size also noticed. Furthermore, Fig. 2 (d) provides the misorientation angle distribution analysis of Mg-0.5Ca alloy prior to deformation. Two interesting information can be witnessed: the high fraction of very low angle grain boundary
(VLAGB) (2°-5°) and the low fraction of tension twins, which is well consistent with the OM and IPF map. The appearance of high fraction of VLAGB can be attributed to the dislocation boundary accumulation due to non-homogeneous deformation of the Mg matrix that surrounds the Mg_2Ca precipitates [9]. To investigate the twinning behavior in Mg-0.5Ca alloy and the effect of temperature on the twinning evolution, the samples were pre-compressed to 5% at RT and CT.

The microstructure and texture of PC-RT and PC-CT can be seen in Figs. 3(a) and (b), respectively. An extensive twinning behavior was observed as a result of pre-compression at both the temperatures as presented by the IPF and IQ map of PC-RT and PC-CT.

![Fig. 2.](image)

Fig. 2. (a) Image quality map and inverse pole figure map, (b) texture analysis via pole figure and inverse pole figure maps, (c) grain size distribution and (d) misorientation angle distribution of the initial microstructure.
Fig. 3. (a) and (b) Image quality map, inverse pole figure map and texture analysis via pole figure and inverse pole figure maps, (c) grain size distribution and (d) estimated boundaries fraction of the PC-RT and PC-CT.

The twinning also resulted in the randomization of basal texture. It has been already suggested by numerous studies that the twinning can result in the randomization of the basal texture due to the re-orientation of the parent grains [10-11]. Furthermore, the twinning induced grain refinement was observed, where grain size was reduced to almost half of the original grain size of undeformed sample (Fig. 3 (c)). The average grain size was recorded to be 5.61 µm and 6.02 µm in the PC-RT and PC-CT, respectively. Fig. 3 (d) shows the estimated boundary fractions of PC-RT and PC-CT alloy. The significant fraction of tension twins was noticed in both the samples. \{10-11\} \{10-11\} tension twin fraction was recorded to be higher in PC-RT as compared to PC-CT. However, very low fraction of compression twins and double twins was observed in both the samples. Fig. 4 (a) presents the Kernel average misorientation (KAM) map and IQ map superimposed with the grain boundaries. It is well established that the KAM accounts for the localized deformation and lattice concerning the geometrically necessary dislocations [12]. The average KAM values were recorded to be 0.95° and 1.02° for the PC-RT and PC-CT, respectively.

Fig. 4. (a) KAM and IQ maps superimposed by grain boundaries and (b) various tension twin variant fractions in PC-RT and PC-CT, (c) point to point misorientation (white line in PC-CT), (d) and (e) tensile curves and hardening rate for PC-RT and PC-CT, respectively.

In addition, the IQ map of both the samples superimposed with the grain boundaries rotational angle (0°-90°) can be seen from Fig. 4(a). The pre-compression induced significant
activation of {10-12} <10-11> tension twins at both the temperatures. The total length of tension twins was recorded to be 1.15 cm and 0.98 cm in PC-RT and PC-CT, respectively. To further investigate the dominance of individual tension twin variant in both the samples, the analysis of twin variant selection was carried out (see Fig. 4(b)). It was noticed that variant 2 (012) [01-1], variant 4 (-112) [-11-1] and variant 6 (1-12) [1-1-1] was dominant in PC-RT while variant 1(102) [10-1], variant 2 (012) [01-1] and variant 5 (0-12) [1-1-1] was more activated in PC-CT sample. The point to point analysis of PC-RT (white line in IQ map) also confirmed the presence of high angle grain boundaries (HAGBs). Finally, the effect of pre-compression on RT mechanical properties was evaluated by conducting the compression test at RT. It can be seen from the Figs. 4(d) and (e) that the pre-compression leads to significant enhancement in the yield strength (Y.S) of as-received alloy by 25% (Table. 1). The remarkable improvement was attributed to the twinning induced grain refinement mechanism as based on the Hall-Petch relationship.

| Material | Y.S (MPa) | UTS (MPa) | Elongation (%) |
|----------|-----------|-----------|----------------|
| As-received | 52.5 | 365 | 8.1 |
| PC-RT | 65.2 | 309 | 6 |
| PC-CT | 66.7 | 310 | 7.5 |

4 Conclusion

In conclusion, the pre-twinning significantly enhanced the mechanical properties of Mg-0.5Ca. The formation of Mg-Ca in the Mg-0.5Ca alloy acts as a barrier to dislocation movement and dislocation accumulates around the precipitate. Microstructural characterization of PC-RT and PC-CT confirmed the twinning induced grain refinement mechanism, which leads to reduce the grain size in both the alloys. Moreover, the randomization of basal texture was also noticed as a result of pre-compression. The IQ map superimposed with boundaries revealed the profuse activation of {10-12} tension twins, which was found to be higher in PC-RT (1.15 cm) as compared to PC-CT (0.98 cm). Finally, the compression tests conducted at RT on all the samples confirmed the excellent increase in the Y.S (25% as compared to undeformed sample) of pre-compressed samples, which was attributed to the texture randomization and twinning-induced grain refinement mechanism.

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