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Study of Dynamic Precipitation during Severe Plastic Deformation of As-cast AZ91 Alloy and Its Influence on Microstructure and Mechanical Properties

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1- Introduction

Magnesium and its alloys as light metals with a high specific strength have attracted much attention in various industries. However, the poor workability of these alloys due to insufficient independent slip systems at room temperature limits their usage. Severe plastic deformation (SPD) techniques are widely used to produce the fine grain structures and to improve the workability and mechanical properties of the material. ECAP as the most frequently used SPD technique is able to impose a high shear strain to the material without any change in the cross-sectional area. As the ECAP of Mg alloys at low temperatures usually leads to the formation of deep cracks, the process is carried out at elevated temperatures. Increasing the die angle, reducing the pressing speed, conducting the process at high temperatures, applying a back pressure and performing a preliminary step prior to ECAP are the main attempts used to improve the workability of magnesium alloys in ECAP process.

In the case of AZ91 alloy, the presence of the $Mg_{17}Al_{12}$ (β) precipitates in the microstructure can strongly affect the microstructure evolution, grain refinement mechanism and mechanical properties. It has been reported that during ECAP of AZ91, grain refinement and precipitation can take place simultaneously. Yuan et al. confirmed the presence of these precipitates after ECAP as well. They indicated that a subsequent aging treatment with formation of higher volume fraction of fine precipitates could improve the material properties. It was also reported a change in both grain size and precipitates morphology. In addition to the grain refinement, precipitation hardening can be another mechanism for strengthening of this alloy. Only few studies can be found in the literature focusing on the development of ECAP processing of cast AZ91 alloy. Since the cast AZ91 alloy used in current study contains very coarse grains with network-like precipitates in its structure, one-step ECAP of cast AZ91 alloy is not feasible. Thus, the present work has been undertaken to develop a two-step ECAP process for as-cast AZ91 to control the grain growth and achieve a fine structure without cracking. The microstructure development during ECAP of the alloy has been studied in this work. The effect of precipitation on grain refinement and final mechanical properties was also investigated.

2- Experimental

The as-cast AZ91 magnesium alloy with the chemical composition of (9.2 wt. % Al, 0.8 wt. % Zn, 0.2 wt. % Mn, 0.09 wt. % Si and 0.03 wt. % Cu) was used in this study. The cylindrical ECAP samples with the diameter of 9.9 mm and the length of 65 mm were prepared. To homogenize the dendritic microstructure of the as-cast AZ91 and remove the precipitates, all samples were heated at 420°C for 24h under protective atmosphere followed by water quenching. An ECAP die was designed and manufactured from AISI D6 tool steel as shown in 0. In order to obtain a uniform temperature within the samples, four electrical heating elements were properly inserted into the die. The temperature of the channel was measured using a K-type thermocouple. The angle between two intersecting channels (φ) and the outer arc of curvature (ψ) were 90° and 20°, respectively. Both channels had a circular cross section with a diameter of 10 mm. This design of the die provides an equivalent imposed strain (ε_{eq}) about 1 for each pass. To maximize the effect of ECAP on grain refinement and also minimize the possible cracking of the samples, the first ECAP pass for all samples was conducted at 350°C and the next passes followed at lower temperature of 290°C.

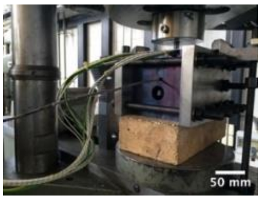

Fig. 1 The photograph of ECAP die used in present study

ECAP was conducted up to eight passes using route B_C, in which samples were rotated around the longitudinal axis by 90° clockwise after each ECAP pass. MoS_2 was used as lubricant. The constant ram speed was set to 3 mm/min. After ECAP, the samples were cut in cross sectional plane perpendicular to the pressing direction, ground and polished and then etched using an acetic-picric solution. The microstructural analysis of the samples was performed by polarized optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy dispersive X-ray spectroscopy (EDX). Tensile tests were carried out at room temperature by Zwick/Roell universal testing machine. Average grain size measurements were carried out using the intercept method.

3- Results and Discussion

0a illustrates the microstructure of the as-cast AZ91 alloy. A typical dendritic structure with the coarse grains that consists of the Mg-α matrix, $Mg_{17}Al_{12}$-β precipitates, eutectic β-phases and Al_8Mn_5 precipitates. The homogenization of the sample resulted in dissolving

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the majority of β-precipitates and eutectic β-phases (0b). The Al_8Mn_5 precipitates were not removed by this homogenization treatment. The average grain size of the as-cast and homogenized alloy was measured to be about (1300±200) μm and (758±150) μm respectively.

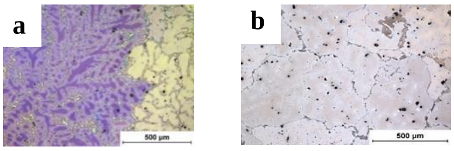

Fig. 2 Typical microstructure of (a) the as-cast and (b) homogenized AZ91 alloy

After the first ECAP pass at 350°C, a bimodal microstructure including the initial coarse grains surrounded by new fine grains can be found that resulted in formation of a necklace type structure (0a). This structure implies the occurrence of dynamic recrystallization (DRX) after the first ECAP pass.

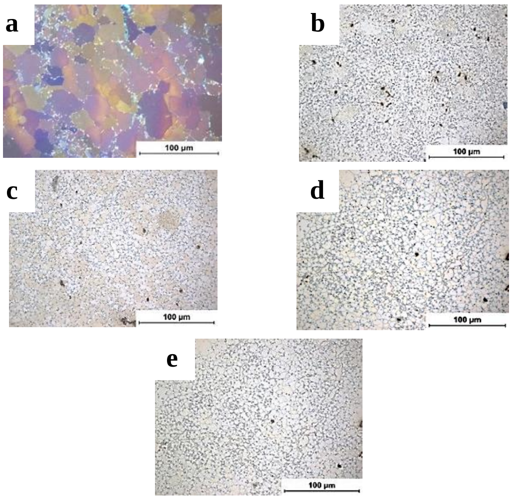

Fig. 3 Typical microstructures of the AZ91 alloy ECAPed at 290°C using route Bc after (a) one, (b) two, (c) four, (d) six and (e) eight passes

As can be observed in 0, β-precipitates appeared after the first ECAP pass in. These second phase precipitates are mainly located near the original grain boundaries and around the new grains. The presence of this spherical precipitates can be attributed to fracturing the retained large eutectic phases as well as the dynamic precipitation during the ECAP. Imposing the high strains on the material during passing in the shear zone results in dynamic precipitation in a short time. In fact, the grain boundaries act as the preferential sites for the nucleation of precipitates. Nevertheless, the high density of dislocations and defects formed during ECAP can also promote precipitating.

A significant level of grain refinement ((18±3.5) μm) has been achieved only by one ECAP pass at 350°C.

0b-e shows the typical microstructure of the AZ91 alloy after ECAP at 290°C from 2 to 8 passes using route Bc. As can be seen, increasing the strain level results in higher grain refinement and a homogeneous recrystallized structure. The average grain size was decreased to (5.5±0.9) μm and the necklace structure became wider after two passes. However, the original grains still exist in the microstructure. An almost fully recrystallized structure with equiaxed grains can be observed after six ECAP passes. Further ECAP up to eight passes resulted in a homogeneous and completely fine equiaxed grain microstructure with an average grain size of (5.0±0.2) μm. Similarly, the volume fraction of precipitates was increased by rising the strain level. An increase in ECAP passes increased the shear strain leading fracturing the large β particles into smaller ones. In addition, since recrystallization and precipitation take place simultaneously, dynamic precipitation is promoted with the formation of new grain boundaries and a high density of dislocations.

A process similar to particle stimulated nucleation of recrystallization (PSN) can occur during the deformation in the presence of larger precipitates (>1μm). The accumulation of dislocations around the precipitates results in forming the deformation zones which act as the preferred sites for nucleation of recrystallized grains. The smaller precipitates (<1μm) can also retard the grain growth by pinning the new grain boundaries and provide more grain refinement.

To evaluate the correlation between the mechanical properties and microstructure of the ECAPed AZ91 alloy, all samples were subjected to tensile tests at room temperature. According to the results, the ECAP processing at 290°C led to a remarkable improvement in strength and ductility of the alloy. As the strain is increased, the ultimate strength, yield strength and elongation to failure were enhanced. After eight passes, the ultimate strength (284 MPa) was about twofold the cast sample (147 MPa) and elongation to failure was significantly increased from 2.5% (cast condition) to 11.3% (8 passes). Increasing the yield strength with decreasing the grain size is in agreement with the Hall-Petch relation.

Ductility enhancement is attributed to decreasing the grain size and increasing the homogeneity of the structure with the pass number. Due to the occurrence of β-precipitations, in addition to grain boundary and dislocation strengthening, precipitation strengthening is also the main reason of increasing the ultimate strength in the AZ91 alloy.

4- Conclusions

In this study, applying a two-step ECAP processing resulted in a significant grain refinement from 758 μm in to 5 μm after eight passes. It can be concluded that the grain refinement is due to simultaneously DRX and dynamic precipitation phenomena. DRX mainly occurs near the precipitates with PSN mechanism. The smaller precipitates with pinning the new grain boundaries can also retard the grain growth and provide more grain refinement. The ECAP processing led to an improvement in both elongation and strength of the alloy. The significant grain refinement, a homogenous structure and the presence of the precipitates are the main reasons for ductility and strength enhancement.