MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ULTRA-FINE GRAINED AZ31 ALLOY PROCESSED BY ECAP

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Abstract

Commercial magnesium alloy AZ31 was processed by equal channel angular pressing preceded by hot extrusion (EX-ECAP). Microstructure evolution with strain due to ECAP was investigated by light and transmission electron microscopy. ECAP processing resulted in continuous grain fragmentation, significant grain refinement and the formation of dislocations. Dislocation structure changes in individual specimens after different number of ECAP passes were investigated by positron annihilation spectroscopy (PAS). Substantial increase of dislocation density occurred during the first two passes of ECAP and was followed by the decline manifesting the dynamic recovery at higher strains. The microstructure and defect structure evolution correlates well with mechanical properties as determined by a detail inspection of microhardness variations in individual ECAPed specimens.

Keywords: Magnesium alloys; ECAP; dislocation density; positron annihilation spectroscopy

1 Introduction

Due to low density, magnesium alloys are very attractive materials for structural components in automotive, aerospace and other transport industries. Nevertheless, the use of magnesium alloys in more complex applications is limited because of the problems associated with poor corrosion and creep resistance and above all the low ductility. The limited ductility is a consequence of the hexagonal structure providing the lack of independent slip systems and the large difference in the values of the critical resolved shear stress in different slip systems. Moreover, the strong deformation textures and stress anisotropy in magnesium alloys reduces significantly the variety of possible industrial applications.

It is well-known that the properties of magnesium alloys can be improved by refining the grain size to the submicrocrystalline or even nanocrystalline level. In the last two decades, a variety of new techniques have been proposed for the production of the ultra-fine grain (UFG) structure in materials. The common feature of all these techniques is the imposition of large straining and consequent introduction of very high density of lattice defects in the material resulting in exceptional grain refinement. Since these procedures introduce severe plastic deformation (SPD) to bulk solids, it became convenient to describe all of them as SPD processing. Several processes of SPD are now available but only three of them receiving the most attention at present time, in particular equal channel angular pressing (ECAP), accumulative roll-bonding and high-pressure torsion (HPT) [1-5].

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The objective of this work is to provide a detail analysis of microstructure and dislocation density evolution in the magnesium alloy AZ31 subjected to ECAP and to correlate the evolution with mechanical properties.

2 Experimental Materials and Methods

Commercial AZ31 alloy with a nominal composition of Mg-3wt. % Al-wt.1% Zn in the initial as cast condition was used in this investigation. Prior to ECAP the as received material was hot extruded at the temperature of T=350°C using the extrusion ratio of ER=22. Equal channel angular pressing was performed at 180°C following route B_c. This two-step technique of severe plastic deformation is referred to as EX-ECAP [6] and proved to be effective in grain refinement of many materials [7]. A series of billets of the cross section of 10x10 mm after 1, 2, 4, 8 and 12 passes was processed by ECAP.

Specimens for light and transmission electron microscopy (TEM) observations of the ECAP-generated microstructure were taken from the middle part of the billet perpendicular to the pressing direction. Specimens for TEM were first mechanically polished and finally ion-polished using a Gatan PIPS^{TM} ion mill at 4 kV at a very low incidence angle of 2-4°. TEM investigations were performed with a Jeol 2000FX electron microscope operated at 200 kV.

A $^{22}Na_2CO_3$ positron source (~1.5 MBq) deposited on a 2 μm thick Mylar foil was used in positron lifetime measurements. The source was always sandwiched between two identical samples of the studied alloy. Positron lifetime measurements were carried out using a fast-fast spectrometer [8] with a time resolution of 150 ps (FWHM ^{22}Na). At least 10^7 positron annihilation events were accumulated in each positron lifetime spectrum which was subsequently decomposed into individual exponential components by a maximum likelihood procedure [9].

Vickers microhardness HV0.1 (F=100 g, dwell time t=10 s) was measured on a semi-automatic Wolpert tester allowing automatic indentation. The variations of microhardness along the different surfaces of ECAP billets were inspected by making 3 lines of indents; one in the centre and other two 2.5 mm from the edge of each plane of the billet (X, Y and Z plane) [10].

3 Results and Discussion

3.1 Microstructure evolution

Microstructure changes in specimens subjected to EX-ECAP were observed by light (LM) and transmission electron microscopy (TEM). The microstructure of AZ31 alloy in the initial condition is shown in **Fig. 1a**. It consists of almost equiaxed grains with the average size of approximately 150-300 μm. Hot extrusion resulted in significant grain refinement and the formation of bimodal structure consisting of large grains of 50–100 μm mixed with relatively fine grains of 2–5 μm, see **Fig. 1b**.

The detail inspection of LM and TEM micrographs indicate that the process of microstructure evolution and grain fragmentation with strain imposed by repetitive ECAP pressing is complex resulting in the formation of a bimodal grain structure with high fraction of fine grains already after the first or second pass. During further processing only coarse grains are continuously refined while the size of fine grains remains almost unchanged with increasing strain resulting in the homogeneous equiaxed structure of the average size of 800-900 nm. Similar course of grain fragmentation in material with hexagonal metals was reported by several authors [11, 12]. Bimodal distribution of grains is probably due to limited active slip systems in magnesium, therefore only favourably oriented grains are deformed and refined as first during ECAP process and areas of less deformed and larger grains are left in microstructure. Examples of grain structure after extrusion (N = 0P) and different number of ECAP passes (N = 1, 2 and 8P) are shown in TEM micrographs in **Fig. 2**.

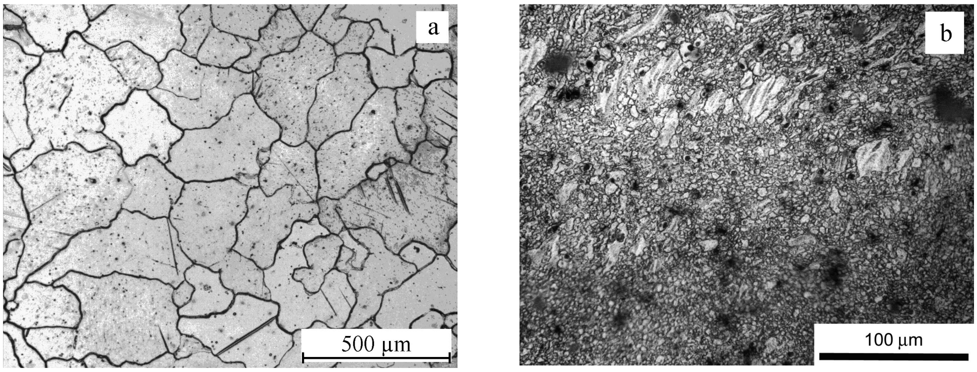

Fig. 1 Microstructure of the AZ31 alloy (a) as-cast, (b) hot extruded condition (N = 0P)

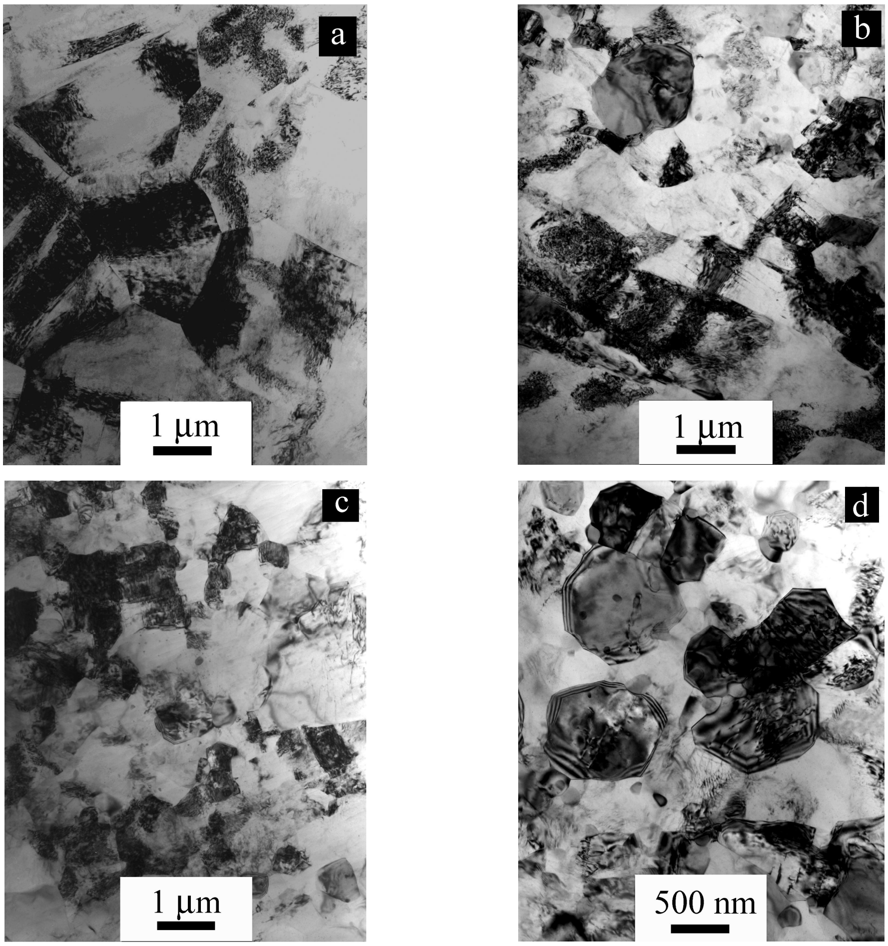

Fig. 2 Microstructure of specimens processed by ECAP (a) 0 P, (b) 1P, (c) 2P, (d) 8P

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3.2 Dislocation density

Severe plastic deformation is known to impose large plastic strains into the material and to create the high density of lattice defects. Positron annihilation spectroscopy (PAS) proved to be a unique technique to investigate the type and the concentration of lattice defects in SPD materials by inspecting the lifetimes of positrons ejected to the material. Any lattice defect is in fact an open volume defect and as such acts as a trap for positrons whose lifetime (time before they annihilate with electrons) is longer than that of positrons in defect free lattice (so called free positrons). Accordingly, positron lifetime spectra can be fitted by several exponential functions with lifetimes τ_i corresponding to different kinds of lattice defects. The intensity of each component (I) represents the concentration of the respective kind of lattice defect. The numerical processing of lifetime spectra obtained from specimens after different number of ECAP passes showed that they can be well fitted by two exponential components. The shorter component with the lifetime $\tau_1 < \tau_B = 225$ ps [13], where τ_B represents the lifetime of free positrons in pure Mg, corresponds to the lifetime of free positrons in AZ31 while the longer component $\tau_2 \approx 260$ ps represents positrons trapped at dislocations [14]. As no other components were found in PAS lifetime spectra one can conclude that the dislocations are the only lattice defects present in specimen after ECAP.

The dependence of positron lifetimes τ_1, τ_2 on the number of ECAP passes is plotted in **Fig. 3a**. The lifetime τ_2 of positrons trapped at dislocations was found to be almost independent of the number of ECAP passes confirming that the nature of positron traps does not change with strain. **Fig. 3b** shows the development of the intensity I_2 of positrons trapped at dislocations on the number of ECAP passes. Note, that strong dislocation component with the intensity exceeding 20 % was detected already in the extruded alloy. It proves that high density of dislocations was introduced into the alloy already during extrusion process before ECAP. The intensity I_2 of positrons trapped at dislocations first increases with increasing number of ECAP passes up to N=2. Further ECAP processing (N>2) causes a gradual decrease of I_2. Note that we have observed similar dependence of the intensity of dislocation component on strain (number of passes) also in other Mg alloys (AZ80, ZK60) [15].

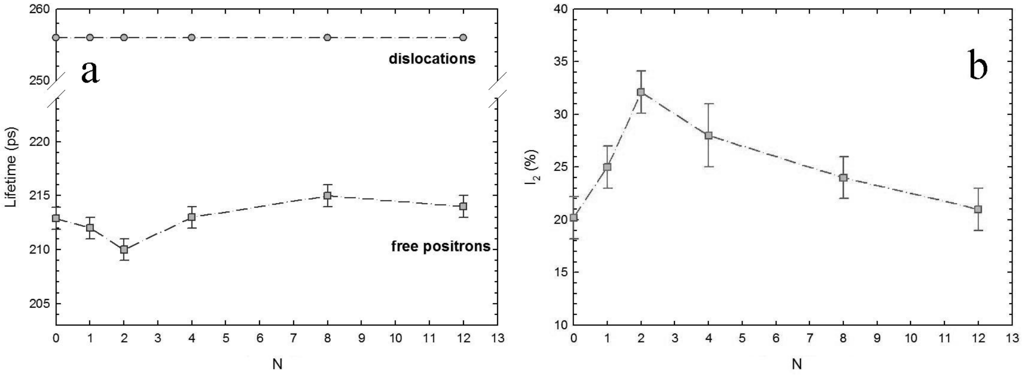

Fig. 3 PAS results in ECAPed AZ31 specimens (a) lifetimes, (b) intensity of the dislocation component I_2

The two-state simple trapping model [16] yields the mean dislocation density ρ_D from the known values of lifetimes τ_1, τ_2 and the intensity of the dislocation component I_2 according to the following formula:

$$\rho_D = \frac{1}{\nu_D} I_2 \left(\frac{1}{\tau_1} - \frac{1}{\tau_2} \right), \qquad (1)$$

where: $\nu_D = 1 \times 10^5$ [$m^2 s^{-1}$] is the positron trapping rate to dislocations [16].

Fig. 4 shows the dependence of the mean dislocation density ρ_D on the number of ECAP passes calculated from the Eq. 1. For low strains the dislocation density was found to increase with increasing the number of passes. In the specimen N=2 it reaches the maximum and then declines continuously for higher number of passes (N>2). The decline of ρ_D for higher strains (N>2) indicates the onset of recovery processes. The most probable recovery mechanisms are the mutual annihilation of dislocations and/or their rearrangement into lower energy structures with increasing strain imposed to the material by ECAP [2, 10].

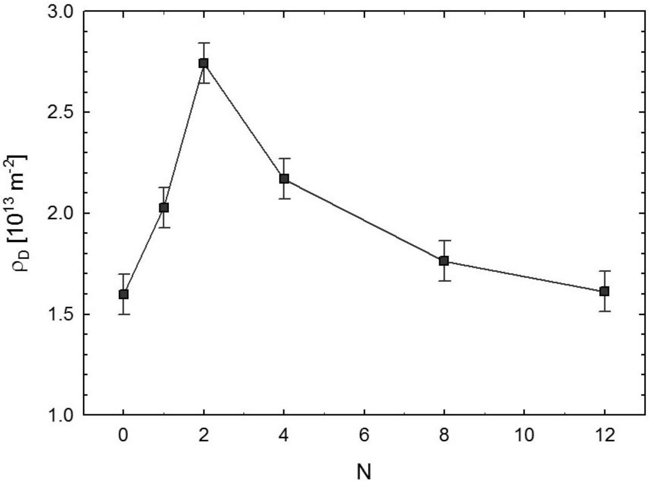

Fig. 4 The development of dislocation density in AZ31 alloy subjected to different number of ECAP passes

3.3 Microhardness

The Vickers microhardness HV0.1 of samples after extrusion and ECAP is schematically shown in **Fig. 5**. HV was measured on all three reference planes (X, Y and Z) [10] to inspect the possible influence of texture on mechanical properties. Unlike the texture investigated in our previous study and found to be different in different billets planes [17], the HV values obtained at the respective planes differ within the statistical error only. Therefore only the values measured on the plane perpendicular to the extrusion direction (plane X) are plotted in **Fig. 5**. The HV value corresponding to 0 passes belongs to the sample in the as extruded condition (N = 0). The microhardness of the as cast alloy is approximately equal to 58 (not shown in **Fig.5**). We can see that the microhardness increases up to the fourth ECAP pass and then declines continuously with increasing strain indicating again the operation of recovery processes at higher strains imposed by ECAP. The microhardness evolution with strain corresponds well with that of dislocation density (cf. **Fig. 4** and **Fig. 5)**. However, the maximum of HV is shifted to higher strains indicating the complex character of strain hardening and softening.

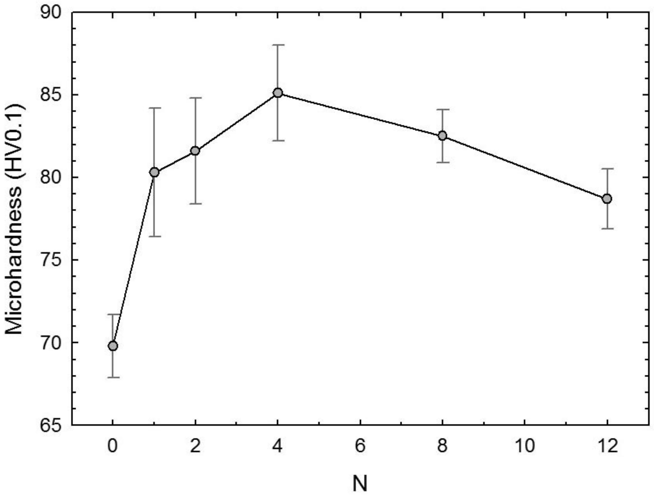

Fig. 5 The dependence of microhardness on the number of ECAP passes N

4 Conclusions

Microstructure and lattice defect evolution in commercial AZ31 alloy processed by equal channel angular pressing was investigated and correlated with mechanical properties. The following conclusions may be drawn from this investigation:

- ECAP pressing resulted in strong grain refinement and bimodal structure,
- further ECAP pressing caused the fragmentation of coarse grains while the fine grains were refined only slightly,
- homogeneous distribution of almost equiaxed grains of the average size of 900 nm with equilibrium high-angle boundaries was observed only for high strains $\varepsilon \approx 8$,
- dislocation density was found to increase with increasing strain up to $\varepsilon \approx 2$; for higher strain it declined continuously up to the value close to that of extruded specimen,
- the influence of microstructure on mechanical properties is rather complex controlled by dislocation density, texture and grain size evolution with strain due to ECAP. Grain refinement controls the strengthening only for small strains ($\varepsilon \leq 2$), for larger strains softening processes (recovery and texture evolution) become important.

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