Microstructure of AZ31 and AZ61 Mg alloys prepared by rolling and ECAP

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Abstract

Microstructure and mechanical properties of AZ31 and AZ61 magnesium alloys after rolling and then subjected to equal channel angular pressing (ECAP) were investigated. Tensile tests were carried out at room temperature. The values of microhardness, the ultimate tensile strength and the elongation to failure were determined.

Key words: magnesium alloy, mechanical properties, microstructure, rolling, forging

1. Introduction

Magnesium alloys with their low density have a great potential for many applications. They have a relatively high strength to density ratio. However, they possess low ductility at room temperature, in most cases only a few percent, and small strength at elevated temperatures [1]. The deformation behaviour of Mg alloys depends on alloying elements (composition), temperature and grain size. Trojanová and co--workers have shown that the mechanical properties of magnesium alloys are significantly influenced by the testing temperature [2-7] leading to a decrease in the strength, by reinforcement and/or grain reinforcement [8–13] leading to an increase in the strength. In some cases, dynamic strain ageing associated with solute atoms is observed in some magnesium alloys, if deformed at certain temperatures [14–16].

Magnesium wrought alloys with their improved mechanical properties may find high engineering applications. Recently, it has been shown that the mechanical properties of AZ31 magnesium alloy sheets, with the strain axis parallel to the rolling direction, are strongly influenced by the test temperature [17, 18]. A preheating temperature may influence the yield stress and the maximum stress of AZ31 magnesium alloys prepared by hot rolling [19]. The texture and the microstructure of rolled AZ31 sheets in the stress--relieved (H24) temper are also influenced by annealing [20]. The grain refinement may be processed by rolling, forging and extrusion.

The aim of the present work is to study the microstructure AZ31 and AZ61 Mg alloys after rolling and equal channel angular pressing (ECAP) followed after rolling. The ultimate tensile strength of samples after rolling and after ECAP will be compared.

2. Experimental procedure

Modified commercial magnesium alloys AZ31 (nominal chemical composition: Mg-2.96Al-0.23Zn-0.09Mn in mass %) and AZ61 (nominal composition: Mg--5.9Al-0.49Zn-0.15Mn in mass %) were studied in the present work. Samples were rolled at a temperature of 380 °C. The rolling rate was about 366 mm/s and the strain rate varied between 3.9 and 8.9 s⁻¹. The samples of a prism shape had the dimensions 150 \times $15 \times 10 \text{ mm}^3$. After each rolling pass, a part of the sample was cut away for the microstructure investigations. Some samples after rolling were subjected to ECAP. The samples for ECAP had dimensions 60 \times $8 \times 8 \text{ mm}^3$. The ECAP processing consisted of two stages: the samples went for 4 passes at a temperature of 250° C using route B_C followed by 1 pass at a temperature of 180°C. Tensile specimens with 40 mm in gauge length, 5 mm width and with a thickness of 2 mm were machined. Tensile tests were carried out at

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a constant cross head speed using an INOVA 250 universal testing machine. The tests were conducted at the room temperature (20 °C) at an initial strain rate of $4.25 \times 10^{-3} \text{ s}^{-1}$. The temperature was controlled to within ± 2 °C. The tensile direction was parallel to the rolling direction. Details of the samples preparation are described elsewhere [21, 22].

Vickers microhardness (HV) was measured on the polished surfaces along and perpendicular to the longest sample axis under a load of 100 g for 15 s. Each data point was the mean value of at least ten indentations. Microstructural features were examined by light microscopy (Olympus IX 70). Metallographic specimens were cut from the samples, mechanically ground on progressively finer grade of SiC impregnated paper and then mechanically polished. Specimens were etched in a solution 5 ml acetic acid, 6 g picric acid, 100 ml ethanol and 10 ml H_2O . Specimens for transmission electron microscopy (TEM) were prepared by cutting slices with a diameter of 3 mm and a thickness of 1.2 mm. The final step of the foil preparation was electropolishing in a solution (LiCl + Mg)perchlorat + Methanol + Butoxy-ethanol) in the temperature range from $-45 \,^{\circ}$ C to $-50 \,^{\circ}$ C. Substructures of the specimens were examined using a JEOL 2000 FX transmission electron microscope operating at 200 kV with electron diffracted X-rays (EDX) system for the phase composition analysis.

3. Experimental results and discussion

The variation of microhardness of AZ61 after one pass of rolling as a function of the distance along the longest specimen axis (parallel to the rolling direction) is shown in Fig. 1. The microhardness variation of AZ61 after one rolling pass followed by ECAP is given in Figs. 2 and 3. Differences in the microhardness values measured along and perpendicular to the longest specimen may be seen from Figs. 2 and 3. It can be seen that microhardness of the ECAP processing specimen of AZ61 alloy is higher than that of the AZ61 specimen before ECAP. The microhardness measurements indicate that the microstructure is not homogeneous. An increase in the microhardness values after ECAP is caused by an increase in the dislocation density due to ECAP processing and/or a refinement of grains. The variation of microhardness of AZ31 alloy after one pass of rolling is shown in Fig. 4.

Optical micrograph in Fig. 5 shows a typical microstructure of AZ61 specimens after one pass of rolling. The microstructural observations revealed inhomogeneous grain size distribution, which corresponds to the microhardness variations along the longest sample axis. Grain sizes vary in the range of 15–30 μ m. There are grains with precipitates or very small grains (~ 0.5 μ m) observed at grain boundaries. Twins are present



Fig. 1. Microhardness of AZ61 alloy after one rolling pass measured along the longest specimen axis.



Fig. 2. Microhardness of AZ61 alloy after one rolling pass followed by ECAP measured along the longest specimen axis.



Fig. 3. Microhardness of AZ61 alloy after one rolling pass followed by ECAP measured perpendicular to the longest specimen axis.

inside some grains. Shear structures throughout the grains (the deformed layers) are observed in AZ61 spe-



Fig. 4. Microhardness of AZ31 alloy after one rolling pass followed by ECAP.



Fig. 5. LOM micrograph of the AZ61 alloy after one rolling pass.



Fig. 7. LOM micrograph of the AZ31 alloy after one rolling pass.



Fig. 8. LOM micrograph of the AZ31 alloy after one rolling pass followed by ECAP.

cimens after one rolling pass followed by ECAP as seen in Fig. 6. The microstructure is heterogeneous, with small grain sizes about 5 μ m and less. Zones (layers) with the grain size of about 5 μ m alternate with zones with grains of 1 μ m. A typical microstructure of AZ31 specimens after one rolling pass is shown in Fig. 7. The microstructure is heterogeneous with the grain size varying between 5 and 50 μ m. Twins are present inside many grains. Partial recrystallization after rolling is observed. Figure 8 shows microstructure of AZ31 specimens after one rolling pass followed by ECAP. It can be seen that the microstructure is heterogeneous. Small grains (about $3-5 \,\mu\text{m}$) are arranged in deformed layers. The microstructure examination revealed heterogeneity. Grain boundaries of the alloys after rolling are well distinguishable whereas they are not clearly distinguishable in samples after ECAP. It is obvious



Fig. 6. LOM micrograph of the AZ61 alloy after one rolling pass followed by ECAP.



Fig. 9. TEM micrograph showing microstructure of the AZ61 alloy after one rolling pass at low (a) and high (b) magnification.

that the variations of microhardness are caused by microstructures that are heterogeneous. The observed shear band (layer) formation may be explained by the model proposed by Ion et al. [23]. They mentioned that the shear zones accrue as a result of rotation dynamic recrystallization. It should be mentioned that the mechanism of this rotational dynamic recrystallization is different from conventional dynamic recrystallization.

The TEM observations presented in Figs. 9–12 give significant details to the optical microscopy. Figure 9 shows the TEM of AZ61 specimens after one rolling pass. Figure 9a illustrates grains without dislocations. Subgrains are shown in Fig. 9a. Small grains in a big grain are shown in Fig. 9b. TEM micrographs of AZ61 specimens after one rolling pass followed by ECAP are presented in Fig. 10. Grain structure is visible in Fig. 10a, whereas Fig. 10b shows a high dislocation density in the grain and twins. Dynamic recrystallization takes place preferably at the site where the dislocation density is high enough to induce the re-



Fig. 10. TEM micrograph showing microstructure of the AZ61 alloy after one pass of rolling followed by ECAP at low (a) and high (b) magnification.

crystallization (Fig. 10a). Janeček et al. [24] have reported similar conclusions. TEM micrographs of AZ31 alloy specimens after one pass of rolling are shown in Fig. 11. Grains with and without dislocations are shown in Fig. 11a. A detailed view of the dislocations arranged in a grain (Fig. 11b) shows some dislocations grounded on precipitates. The rolled specimens indicate formation of new grains inside the original ones. We can assume according to Janeček et al. [25], who performed electron diffraction analysis in AZ31 rolled samples, that most of grains are aligned along the [0001] zone axis. It means that basal planes, that were oriented in the original material, rotated during rolling to the position parallel to the rolling plane. Grains with a low dislocation density and grains without dislocations are characteristic for the substructure of the AZ31 alloy after one rolling pass followed by ECAP (Fig. 12a). Few dislocations visible in the grain interior indicate the occurrence of dynamic recrystallization. Elongated grains and twins are also visible. The presence of dislocations in some recrystallized grains

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Fig. 11. TEM micrograph showing microstructure of the AZ31 alloy after one rolling pass at low (a) and high (b) magnification.

is probably a result of large strains imposed by ECAP. Figure 12b illustrates a detailed view of grain boundary sliding.

The values of the yield strength, YS, the ultimate tensile strength, UTS, the elongation to fracture, $\varepsilon_{\rm f}$, and the reduction in area, RA, are given in Table 1 for the samples subjected to rolling. The values of UTSfor AZ61 and AZ31 samples processed by one pass of rolling followed by ECAP increase to 296 MPa and 274 MPa, respectively. It can be seen that the ultimate tensile strength of the AZ61 alloy is higher than that of the AZ31 alloy for both samples after rolling and samples after rolling followed by ECAP.

4. Conclusions

The microstructure evolution of AZ61 and AZ31 magnesium alloys after rolling and after rolling followed by ECAP was investigated using light optical microscopy (LOM) and transmission electron micro-

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Fig. 12. TEM micrograph showing microstructure of the AZ31 alloy after one rolling pass followed by ECAP at low (a) and high (b) magnification.

Table 1. Mechanical properties of AZ61 and AZ31 alloys subjected to rolling

Alloy	YS (MPa)	UTS (MPa)	$\stackrel{arepsilon_{\mathrm{f}}}{(\%)}$	$RA \ (\%)$	
AZ61 AZ31	164 182	282 244	15.3 18.0	$17.2 \\ 14.8$	

scopy (TEM). The mean grain size was reduced but the microstructures were heterogeneous. TEM observations showed grains with a high density of heterogeneously distributed dislocations and grains without dislocations. Some recrystallized grains were found. The microhardness values exhibited heterogeneity as well. The values of tensile strength of the materials after rolling followed by ECAP are higher than those for materials subjected to rolling only.

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