Original Article

Evolution of mechanical properties of LAE442 magnesium alloy processed by extrusion and ECAP

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1. Introduction

The microstructure development of the magnesium alloys after thermomechanical processing through ECAP has been investigated over the last decade using different parameters (channel die geometry, temperature, cross-head velocity). The selection of the investigated alloys was usually limited to the most widespread industrial alloys, such as AZ31, AZ61 and AZ91 [1–3]. Moreover, pure magnesium is often being investigated in order to assess the mechanisms occurring during the ECAP, which affect the final microstructure [4]. ECAP was found to have major effect on the microstructure, especially grain size. Severe plastic deformation (SPD) introduced to the material results in a dynamic recrystallization and a substantial grain refinement. Although it was proposed that a substantial grain refinement severely affects mechanical properties according to the Hall–Petch law, the specific texture formation after ECAP has major influence on the mechanical properties as well. This might lead to a significant softening of the processed material [5,6].

The presented work is focused on the investigation of the LAE442 magnesium alloy. This alloy was lately considered by several authors as promising material for biodegradable magnesium implants [7–9]. Moreover, the in vivo tests showed superior corrosion properties of the extruded state of this alloy compared to the as cast state. This implies a beneficial (positive) effect of lower grain size on corrosion properties [10]. However, the biocompatibility and biodegradability are not the only important parameters of the implant material. The mechanical properties are very important parameters as well. The volume of the implant is conditioned (determined)

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by the stiffness of the material. As mentioned above, ECAP was found to have major influence on the mechanical properties of magnesium alloys. In this work, preliminary results of the influence of processing through ECAP on the mechanical properties of the LAE442 magnesium alloy are presented.

2. Experimental procedures

The magnesium alloy LAE442 (Mg-4%Li-3.6%Al-2.4%RE, in wt. %) was extruded at the temperature of \( T = 350 \degree C \) with the extrusion ratio of \( \text{ER} = 22 \). The billets for ECAP, with the initial dimensions of 10 mm x 10 mm x 100 mm, were machined from the extruded bar. The billets underwent severe plastic deformation through ECAP to a maximum equivalent strain of 12 [11] by twelve passes (12P) following route Bc. The angle \( \theta \) between two intersecting channels and the corner angle \( \Psi \) were 90\(^\circ\) and 0\(^\circ\), respectively. Molybdenum disulfide grease was used as a lubricant. The processing was performed between 185–230 \degree C and 5–10 mm/min.

The microstructure after ECAP processing was studied by a scanning electron microscope (SEM) equipped with EBSD. The specimens were cut from the billet with the investigated surface perpendicular to the processing direction. The samples were mechanically polished with the grain size decreasing to 0.25 \( \mu \)m and afterwards ion polished using the Gatan PIPS™ ion mill. The scanning electron microscope FEI Quanta with EDAX EBSD camera was used for microstructure observation.

Tensile deformation tests were performed using the INSTRON 5882 deformation machine at the room temperature with a initial strain rate of \( 10^{-3} \text{ s}^{-1} \). The bone shaped samples were machined from the billets with the deformation direction parallel to the extrusion and ECAP directions and afterwards cut to 1 mm thick plates.

3. Experimental results

Microstructural evolution of the investigated alloy was observed by SEM equipped with an EBSD camera. The average grain size after the extrusion was \( \sim 21 \mu \)m. Intensive plastic deformation introduced to the material by ECAP resulted in a substantial grain refinement as shown in Fig. 1. The first pass through ECAP caused a decrease of the average grain size to \( \sim 11 \mu \)m. The average grain size after the second and the fourth pass decreased in both cases almost to one-half and was \( \sim 6 \mu \)m and \( \sim 2.6 \mu \)m, respectively. Further thermomechanical processing only led to a limited grain refinement as compared to the previous ones. The average grain size in the 8P sample was \( \sim 1.9 \mu \)m and after twelve passes through ECAP, it was \( \sim 1.5 \mu \)m.

The significant microstructural change after the processing through ECAP, especially the grain refinement, had major influence on the mechanical properties of the investigated alloy. The gradual processing through ECAP led to a continuous increase of the tensile strength up to 4P, followed by saturation. A similar evolution was also found in the case of the ultimate tensile strength, as shown in Fig. 2(a). Sharp increase of the elongation to fracture was observed after the first pass through ECAP, as shown in Fig. 2(b). After the subsequent ECAP processing, no further change in the elongation to fracture was observed. The corresponding values of the tensile tests results are presented in Table 1.

In order to examine the pronounced improvement of the elongation to fracture after the first pass through ECAP, the fracture surfaces of the samples were investigated by SEM. The

![Fig. 1](image1.png)  
**Fig. 1** - Evolution of the average grain size as a function of the increasing number of ECAP passes.

![Fig. 2](image2.png)  
**Fig. 2** - Evolution of the (a) yield and ultimate tensile strength as a function of the increasing number of ECAP passes and (b) comparison of true-strain true-stress curves of the extruded sample, the 1P sample and the 12P sample.
brittle character of the fracture characterized all states of the investigated material. However, significant difference in the character of the fracture was found. The resulting micrographs of the as-extruded sample and the 1P sample are presented in Fig. 3. Moreover, a detail of the 1P sample fracture surface is presented in Fig. 3(c).

4. Discussion

The substantial grain refinement had major influence on the yield and also the ultimate tensile strength. The evolution of the yield strength followed the grain refinement, as it rapidly increased up to four passes and then it saturated. This evolution is different from the one observed in other magnesium alloys, where significant softening of the yield strength was observed after two passes [1,2,6,12]. Therefore, different strengthening mechanisms were affecting the final mechanical properties in the LAE442 alloy. The possible explanation lies in (is based on) the texture evolution, which implies that further investigation is needed.

Moreover, investigation of the mechanical properties evolution revealed that processing through ECAP caused the elongation to fracture increase in the extruded LAE442 alloy. The first pass through ECAP led to a substantial increase of ductility, however, the subsequent grain refinement did not lead to further improvement of the elongation to fracture. This implies that the grain size itself had only limited or no impact on the resulting mechanism of the fracture. Intergranular fracture of the as-extruded sample was present mostly on the grain boundaries, when grains were pulled out from the matrix as a whole (Fig. 3(a)). On the other hand, a cleavage character of the fracture was found in the samples processed through ECAP, as depicted in Fig. 3(b) and (c). This fracture followed the crystallographic planes.

The investigation of the chemical composition of the LAE442 alloy showed that a small addition of calcium (0.1 wt.%) is present in the matrix and its concentration is below the solubility limit [13]. It was shown that segregation of calcium in the grain boundaries causes intergranular fracture in the magnesium alloys, which could substantially decrease elongation to fracture [14,15]. Moreover, the segregation tendency depends on the grain boundary type. The grain boundary excess at the high-angle grain boundaries is higher than that at the low-angle grain boundaries (LAB) [16,17]. In Ref. [1], it was shown that the first pass through ECAP led to an increase of the LAB fraction in the processed material. Therefore, a significant increase of the LAB after the first pass through ECAP led to lower segregation of calcium along the grain boundaries, and therefore higher elongation to fracture. Subsequent processing through ECAP led to decrease of the LAB fraction, but also to increase of the grain boundary volume fraction. This way, calcium could be distributed better in the material. Consequently, the ductility remained almost unchanged up to twelve passes through ECAP due to the combined effect of the LAB fraction decrease and the total grain boundary volume fraction increase. Contrary to the fact that grain boundary segregation occurs usually within few hundreds of Å [16,18], it was not possible to be observed in the scanning electron microscope.

5. Conclusions

The substantial grain refinement had a significant influence on the tensile deformation, particularly the yield strength and the ultimate strength in the LAE442 magnesium alloy. Moreover, the mechanism of the fracture changed after thermomechanical processing, which led to the improvement of the elongation to fracture. This was most probably caused by the segregation of calcium along the grain boundaries after the extrusion that was reduced by further thermomechanical processing.

Conflicts of interest

The authors declare no conflicts of interest.
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