Thermal Stability of Ultra-fine Grained AZ31 Magnesium Alloy Processed by Severe Plastic Deformation

J. Vrátná and M. Janeček
Charles University Prague, Faculty of Mathematics and Physics, Prague, Czech Republic.

Abstract. The microstructure of ultra-fine grained (UFG) magnesium alloy AZ31 and its development with temperature were investigated. UFG specimens were prepared by a combined two-step severe plastic deformation process: the extrusion (EX) and the equal-channel angular pressing (ECAP). This combined process leads to microstructure refinement and enhanced microhardness. The microstructure evolution and mechanical properties were studied by light and scanning electron microscopy and microhardness measurements. Specimens were annealed for 1 hour at temperatures of 150, 200, 250, 300, 350, 400, 450, 500 °C. No changes were observed after annealing at 150 °C. The coarsening of a fine-grained structure at higher temperatures was accompanied by a gradual decrease of the microhardness. Microstructure of the specimens annealed at 450 °C and 500 °C is very non-homogeneous due to the secondary recrystallization.

Introduction
Magnesium is one of the lightest metals used in constructional alloys. Replacing aluminium and steel by magnesium in the same volumes will result in a weight saving of around 33 % and 77 %, respectively. The significant weight saving ability offers potential for industrial production of automotive or aircraft components. Some of the magnesium alloys are also biocompatible and applicable in medicine, e.g. temporary screw or biodegradable stents.

The fact that the strength increases with a reduction of grain size (Hall-Petch equation [Hall, 1951; Petch, 1953]) is the reason of the interest in production of ultra-fine grained (UFG) materials. The UFG materials are characterized not only by very small grain sizes (in the submicrometer or even nanometer range [Valiev and Langdon, 2006; Valiev et al, 2006; Horita et al., 2001]) but by homogeneous and equiaxed microstructure and a high fraction of high-angle grain boundaries as well [Valiev and Estrin et al., 2006]. Nowadays, the most common procedures for the fabrication of ultra-fine grained materials are severe plastic deformation (SPD) processes. The equal-channel angular pressing (ECAP), known also as the equal-channel angular extrusion (ECAE), is currently the most developed SPD procedure. Due to their hexagonal lattice of magnesium and its alloys, ECAP process requires several parameter optimization [Gottstein et al., 2005]. Temperature is one of the most important conditions [Furui et al., 2007]. At room temperature, the basal slip is the mostly activated slip system in magnesium [Mordike et al., 2001]. Because of fewer number of slip systems in hexagonal closed packed lattice, basal slip does not offer five independent slip systems which are required for uniform deformation according to von-Mises criterion [von Mises, 1928]. At elevated temperatures, the critical shear stresses for prismatic and pyramidal slip systems reduce significantly and the twinning contribution, which is very important at room temperature, becomes less crucial [Máthis et al., 2006; Trojanová et al., 2007].

The objective of this work is to investigate thermal stability of ultra-fine grained magnesium alloy AZ31 processed by two-step process designated as EX-ECAP (extrusion and ECAP in consequence). Thermal stability of UFG structure is very important to its application in many branches of industry, especially due to potential superplastic properties of ultra-fine grained magnesium.

Experimental material and procedures
Commercial AZ31 magnesium alloy with the chemical composition given in Table 1 was used in this investigation. The extruded material was prepared from as-cast state by extrusion at 350 °C with the extrusion ratio of 22. The ECAP conditions needed to be optimized to obtain compact specimens without surface cracking. These optimal conditions were found: pressing temperature of 180 °C, pressing speed of 50 mm.min⁻¹, MoSi lubricant. The billets with dimensions of 10 × 10 × 100 mm were ECAPed through a 90° die via route Bc for 4 passes.
Table 1. Chemical composition of the investigated alloy.

<table>
<thead>
<tr>
<th>Chemical element</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Si</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition [wt. %]</td>
<td>3.14</td>
<td>2.52</td>
<td>0.75</td>
<td>0.022</td>
<td>bal.</td>
</tr>
</tbody>
</table>

ECAPed billets were cut into slices, annealed and water quenched. Isochronal annealing for 1 hour was carried out in 50 °C steps from 150 °C to 500 °C.

Development of mechanical properties was investigated by microhardness measurements. The microhardness was measured by a Vickers hardness tester Leco M-400-A. A load of 100 g and the indentation time of 10 s were used in the experiments.

The microstructure was observed using the light microscope Olympus IX70. The specimens were molded into polypropylene and prepared by mechanical grinding, polishing and etching (dilute solution of picric acid). Microstructure of the samples with very small grain sizes was observed by scanning electron microscope (SEM) Quanta 200 FEG.

Results and discussion

The microhardness of all annealed samples was measured on the plane perpendicular to the pressing direction. The indenter was applied minimally ten times in each test. Results are shown in Fig. 1. The microhardness, as shown, does not change for annealing temperature up to 150 °C and then declines markedly up to the temperature of 500 °C.

The microstructure of all annealed specimens is shown in Fig. 2a–i. Only the planes perpendicular to the pressing direction were observed. The microstructure of the unannealed sample and the samples annealed at 150 and 200 °C is so fine that the light microscopy is not suitable for its observation. Etching of these specimens is problematic; the etchant creates wave formations on the sample surface, see Fig. 2a–c. The UFG structure of the specimen after annealing at 150 °C is illustrated in Fig. 3. The microstructure consists of larger grains of the size of few µm and plenty of very fine grains smaller than 1 µm around the larger ones.

The microstructure of the remaining annealed samples is shown in Fig. 2d–i. One can see that the grains are equiaxed and the grain sizes increase with increasing annealing temperature. The grain sizes were established statistically and the results are demonstrated in Fig. 4.

The non-homogeneous microstructure of the samples annealed at temperatures 450 °C and 500 °C is probably caused by the secondary recrystallization (discontinuous grain growth). Extruded and ECAPed AZ31 alloy contains Mg17Al12 precipitates in substitutional solid solution of aluminum in magnesium. Discontinuous grain growth occurs during annealing close to the solvus temperature. During such as annealing process, precipitates locally dissolve and initiate grain growth, while the neighbouring granular microstructure remains stabilized by the precipitates [Gottstein, 2004].

![Figure 1](image-url)

**Figure 1.** The dependence of the microhardness of extruded specimens after 4 ECAP passes on the temperature of isochronal annealing.
Conclusion

The thermal stability of ultra-fine grained magnesium alloy AZ31 processed by extrusion and consequently by equal-channel angular pressing was investigated in the presented paper. This combined process led to the microstructure refinement and enhanced microhardness. Specimens were isochronally annealed for 1 hour at temperatures from 150 °C to 500 °C in 50 °C steps. The results obtained by this investigation can be summarized as follows:

- The microstructure of the initial extruded and ECAPed material after 4 passes is very fine.
- The value of microhardness does not change for annealing temperature up to 150 °C and then declines markedly up to the temperature of 500 °C.
- Microstructure evolution is observed only above the temperature of 250 °C. Microstructure of the specimens annealed at 400 °C and higher temperatures is very non-homogeneous.
- Secondary recrystallization is the softening mechanism in the investigated material at the annealing temperatures between 450–500 °C.

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Figure 3. The microstructure of the specimen annealed at 150 °C (SEM).

Figure 4. Average grain sizes of the samples after isochronal annealing.

References


