INFLUENCE OF PROCESSING ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MAGNESIUM ALLOY AZ91
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Resume
This paper deals with differences of mechanical characteristics and fracture surface morphology of AZ91 magnesium alloy in extruded state and after subsequent equal channel angular pressing (ECAP). According to the results, the tensile properties were not controlled by grain size only as values of the tensile strength and 0.2 proof stress were similar for both alloys despite having average grain size 15.9 μm for the extruded alloy and 1.2 μm for the ECAPed alloy. In contrast, microhardness seemed to be dependent solely on the grain size. Fractographic analysis has shown changes in the damage mode from quasi-cleavage fracture in extruded state to rather ductile fracture with dimple morphology in exECAPed state during tensile loading.

1. Introduction
As one of the lightest metallic structural materials, magnesium and its alloys are promising materials in various applications in industries where weight reduction is demanded. Application of magnesium alloys is still limited because of problems connected with its limited ductility and relatively low strength [1]. Mechanical properties of magnesium alloys in the as-cast state are often insufficient for many applications, while other processing methods have limitations due to HCP (hexagonal close packed) crystal lattice of the magnesium alloy matrix. Primarily, a processing method such as cold working is problematic and there is a risk of crack formation (caused by a lack of independent active slip systems at room temperature). Due to this fact, processing at elevated temperature is used to avoid possible cracking [2].

It is well known that the grain size is a very important microstructural factor affecting mechanical behavior [3]. Wrought magnesium alloys are in the focus of designers thanks to better mechanical behavior than cast Mg alloys. Grain refinement via working leads to significant mechanical properties improvement of the alloys, but for some specific application further enhancement of properties usually connected with more pronounced grain refinement is required [4].

An effective way to mechanical properties enhancement is to use one of the severe plastic deformation (SPD) methods, especially equal channel angular pressing (ECAP) due to its good potential for control of microstructural evolution [5, 6]. Processing of magnesium alloys via SPD methods is usually conducted at or above 150 °C [7 - 9].
As a consequence of different methods of the semi-product preparation following into the different resulting microstructure, the fracture mechanism of magnesium alloys vary from brittle to partly ductile (quasi-cleavage) strongly depending on the state of material and on the correlation between tensile and extrusion axis [10 - 15].

In this paper, the microstructure and its influence on tensile properties, microhardness and fracture mode of AZ91 magnesium alloy in the as-extruded state and after ECAP were analyzed.

2. Material and experimental methods

AZ91E magnesium alloy in extruded initial state with chemical composition given in Table 1 was used in this work. This alloy was subjected to ECAP process (exECAP) in the laboratory of Politecnico di Milano, Italy, using four passes through ECAP die (the angle between channels Φ was 110°) at temperature 200 °C using route BC; ECAP direction was identical to the extrusion direction. Samples for metallographic analysis were prepared by conventional mechanical grinding and polishing procedures. Microstructural analyses were performed using light optical microscope Zeiss Axio Observer Z1.m. For more detailed microstructural study of the sample exhibited significant grain refinement and the final microstructure was almost unimodal with the average grain size of 1.2 μm with only few large grains (~10 μm) remaining in the material volume (Fig. 1b). An increase of Mg17Al12 particles amount and its refinement and redistribution was observed after the ECAP process (Fig. 1c - white particles), whereas this process of Mg17Al12 particles precipitation during ECA is typical for analyzed alloy [16, 17].

According to the tensile tests results (Fig. 2, Table 2), σ0.2 proof stress and ultimate tensile strength achieved for material in extruded state was 260 MPa and 366 MPa, respectively, elongation at the break was 15.7%. All these values were slightly lower for material in the exECAPed state (σ0.2 proof stress - 251 MPa, ultimate tensile strength - 359 MPa, elongation at the break - 11.9%) even though the average grain size was smaller. The microhardness of the material exhibited opposite tendency with values of 73 HV 0.1 for extruded material and 106 HV 0.1 for exECAPed material (see Table 2).

### Table 1

<table>
<thead>
<tr>
<th>Elements</th>
<th>Mg</th>
<th>Al</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Si</th>
<th>Zn</th>
<th>Others total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition, wt. %</td>
<td>Balance</td>
<td>8.70</td>
<td>0.001</td>
<td>0.003</td>
<td>0.20</td>
<td>0.04</td>
<td>0.67</td>
<td>&lt;0.03</td>
</tr>
</tbody>
</table>

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Fig. 1. Microstructure of experimental material. (full colour version available online)

Fig. 2. Engineering stress-strain curves at room temperature.
Fracture surfaces of the samples broken during tensile test were analyzed by means of SEM. In the case of the extruded samples, the fracture surface consisted of transgranular, quasi-cleavage fracture in which cleavage planes and tearing edges appeared (Fig. 3a, b). After 4 passes through ECAP die (exECAP state) the fracture surface of the tensile test sample slightly changed, the presence of semi-cleavage planes was rather poor and the amount of dimples was substantially higher in comparison with the extruded state. Moreover the dimples were tiny and shallow (Fig. 3c, d).

4. Discussion
According to the obtained results, the tensile properties are not solely dependent on the average grain size of the material while improving proof stress and ultimate tensile strength via ECAP is rather insignificant even if grain refinement is considerable. This phenomenon is not unusual for ECAPed alloys and according to literature data is caused by various microstructural features especially by texture weakening [15,18,19].

The decrease of elongation at the break together with a slight decrease of tensile strength

<table>
<thead>
<tr>
<th>State of material</th>
<th>Average grain size (μm)</th>
<th>0.2% proof stress (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Elongation (%)</th>
<th>Microhardness (HV 0.1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extruded</td>
<td>15.9</td>
<td>260</td>
<td>366</td>
<td>15.7</td>
<td>73</td>
</tr>
<tr>
<td>exECAP</td>
<td>1.2</td>
<td>251</td>
<td>359</td>
<td>11.9</td>
<td>106</td>
</tr>
</tbody>
</table>

Table 2

Fig. 3. Fracture surface of the specimens failed in tensile test.
also sometimes occurred during ECAP after particular number of passes [18]. In this case this behaviour could be attributed to a significant increase of amount and redistribution of Mg$_{17}$Al$_{12}$ particles in the microstructure during ECAP, these particles, located both on grain boundaries and inside grains, act as obstacles for dislocations movement and influence resulting deformation behaviour of the material. Possible cause of changes in character of strengthening during plastic deformation, which results in partly different shape of stress-strain curves during tensile tests (Fig. 2), could be also the mentioned increase of Mg$_{17}$Al$_{12}$ particles amount during ECAP process.

Contrary to the tensile properties the measured microhardness correlates strongly with the average grain size. These results are in agreement with literature data [15]. Moreover microstructural changes due to ECAP process led to fracture micromechanism change [20], nevertheless level of this change was not sufficient enough to increase the elongation at the break if compared with the extruded state [21].

5. Conclusions

Initial microstructure in extruded state was rather inhomogeneous with average grain size of 15.9 μm. A homogeneous microstructure of refined grains with average size of 1.2 μm was obtained after 4 ECAP passes.

The higher values of σ$_{0.2}$ proof stress, ultimate tensile strength and elongation were achieved by extruded alloy when compared to exECAPed state.

Alloy in exECAPed state exhibited slightly lower tensile characteristics than extruded alloy despite having smaller average grain size which was caused probably by texture modification and also possibly by Mg$_{17}$Al$_{12}$ particles precipitation during ECAP.

Slight changes in fracture micromechanism from quasi-cleavage (extruded state) to rather ductile character (exECAPed state) were observed. These changes did not lead to increase of elongation at the break during tensile test.

Microhardness increased with decreasing average grain size reaching value of 106 HV 0.1 for alloy in exECAPed state.

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Note

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References