LITHIUM CONTAINING AZ31 MAGNESIUM ALLOY PROCESSED BY ECAP

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Abstract

In this article, a hot extruded (EX) AZ31 magnesium alloys with a lithium content of 7.5 wt.% and 15 wt.% were subjected to severe plastic deformation using Equal Channel Angular Pressing (ECAP) at 250 °C. In order to investigate changes in the structure and substructure, light microscopy and scanning microscopy were used. The influence of severe plastic deformation was determined by hardness measurement using the Vickers method. The results were compared with typical hot extruded alloy AZ31.

Keywords: Magnesium alloy AZ31, ECAP, hardness, SEM, EBSD

1. INTRODUCTION

Magnesium - lithium alloys belong to the group of light materials with a very small density of approximately 1.35-1.45 g/cm^3, which makes these alloys a popular construction material. At the same time, these alloys are characterized by relatively low values of the strength characteristic (tensile strength, yield point, hardness).

Lithium additive has a substantial impact on the increase of the magnesium plasticity, which crystallizes in the hcp lattice α, as opposed to lithium, which crystallizes in the bcc lattice β. Lithium thus increases the ability of magnesium and its alloys to deform plastically. According to the binary diagram Mg-Li (**Figure 1 a**) the transition between the α and β phases is conditioned by the minimum of 11 wt.% Li (30 at.%). The optimal share of the phases α + β ranges between 6.5-10 wt.% (15-30 at.%). **Figure 1 b** indicates the impact of the amount of lithium on the density of the Mg-Li alloy. The amount of lithium also has a major impact on the reached plasticity of the given alloy; an alloy of Mg-Li in as cast state, which showed an extension by as much as 60% during tensile test [1]. It is known that aluminum has an impact on consolidation of the basic Mg matrix and the preservation of good plastic properties thanks to the crystallization of these elements in the bcc lattice [1 - 3].

One possibility of preparation of structural materials characterized by a low specific mass with simultaneous increased strength and preservation of good plasticity of Mg - Li-based alloys is the application of the method using severe plastic deformation through ECAP (Equal Channel Angular Pressing). This method uses the effect of a high plastic deformation for refining the structure of the deformed material.

ECAP is currently a popular manner to modify microstructure through refinement of the grain, as during the pressing no change of the cross-section of the formed sample takes place and the energy of deformation is accumulated inside the formed material. This leads to the start of a grain refinement processes. As it can be seen in the scheme of the ECAP (**Figure 2**), the sample is during the pressing being deformed through shearing, which appears in the place of the transition between the vertical and horizontal channel at an angle of 90°. Increased density of accumulated dislocations in the material leads to the creation of sub-grains, and thus to the disintegration of the former grain. As the sample did not change its cross-section after the transition, this process for the achievement of a high level of deformation, and thus final refinement of the microstructure, can be "freely" repeated [4 - 5].

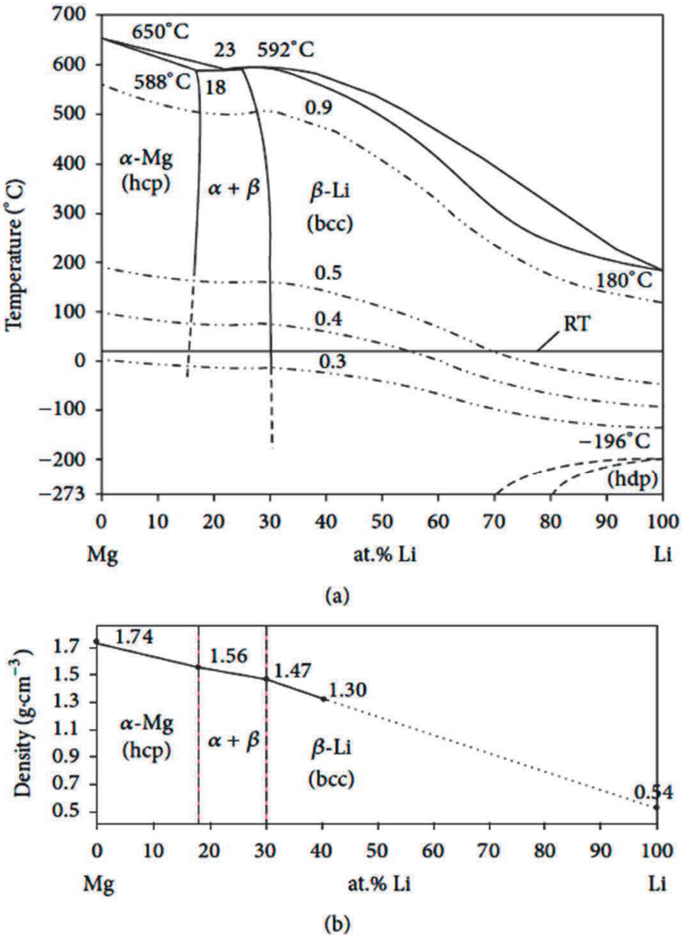


Figure 1 Phase equilibrium diagram for Mg-Li alloys **(a)** and change in density **(b)** as a function of the changing lithium content in Mg-Li alloy [1]

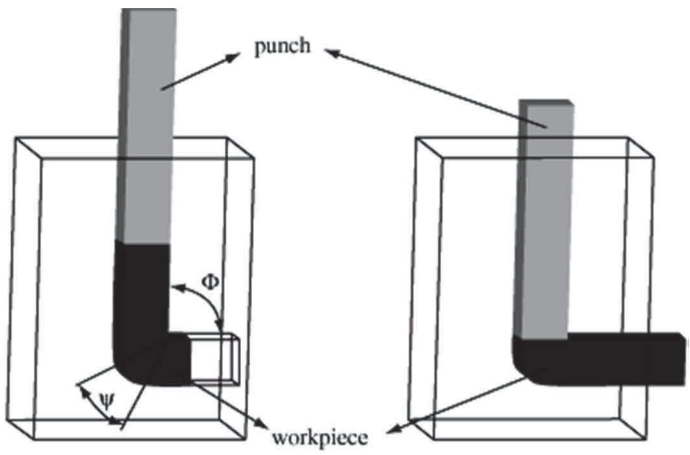


Figure 2 Principle of Equal Channel Angular Pressing method [4]

Using the validity of the Hall-Petch relation (1), it is possible to quantify the impact of the size of the grain on the reached properties of strength of any material processed by the method of severe plastic deformation.

$$\sigma_y = \sigma_o + k / \sqrt{d} \tag{1}$$

Where σ_y present yield stress, σ_o stress for overcoming of Peierls-Nabarr friction stress of lattice, *k* is constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations and *d* present the average grain size.

The aim of this paper is to define the impact of the amount of lithium (7.5 and 15 wt.%) in the magnesium alloy AZ31 on the reached refinement of microstructure and on the hardness of these alloys after forming, using the ECAP method. Microstructure analysis is carried out with an optical microscope NEOPHOT 2, and the deformed microstructure is analyzed with the electron microscope Inspect F50 (method SEM).

2. INVESTIGATION PROCEDURE

2.1. Material

The experimental materials were provided in a hot-extruded state (at the temperature of 430 °C) from bars of 40 mm in diameter to a beam of 20 x 20 mm. Extrusion in the default state has a substantial impact on the reached refinement of the microstructure prior to the refining process applying the ECAP method, impact on the reduction of the necessary number of forming passes ECAP [6 - 7] can thus be expected.

Table 1 Chemical composition of investigated alloys (wt.%)

Material	Li	Al	Zn	Mn	Mg
AZ31	-	2.96	0.20	0.10	Rest
AZ31 + Li	7.50	2.80	0.61	0.40	Rest
AZ31 + Li	15.00	2.90	0.45	0.20	Rest

2.2. ECAP process

Forming of experimental magnesium alloys with the use of ECAP method was realized in the laboratory of the Department of Mechanical Technology, Faculty of Mechanical Engineering, VSB - Technical University of Ostrava at the newly renovated DP2000 hydraulic press (**Figure 3**). The ECAP processing was realized at the temperature 250 °C. At this temperature, the total number of 3 passes was applied in dependence on the evolution of extrusion.

Figure 3 Workplace of VSB - TU Ostrava for materials forming by ECAP method

2.3. Hardness and microstructural analysis

The materials processed by ECAP were then divided into individual series for the manufacture of specimens for mechanical testing (hardness measurement on the tester HPO 250) and metallographic analysis. The samples for metallographic analysis on the light optical microscope NEOPHOT 2 were prepared in a usual manner. In the first stage the samples were polished with the use of Al_2O_3 based polish suspension. In the second stage, the polishing was performed with the use of very fine velvet cloth with short fibers. Diamond powder with grain size 1 μm was used as the polishing material. Diamond was applied by spraying and cloth was regularly wetted by the alcohol-based liquid.

Detailed studies of experimental materials after ECAP were realized on the scanning microscope (HR-SEM) Inspect F50. The SEM sample was prepared by melting, mechanical polishing and etching.

3. INVESTIGATION RESULTS

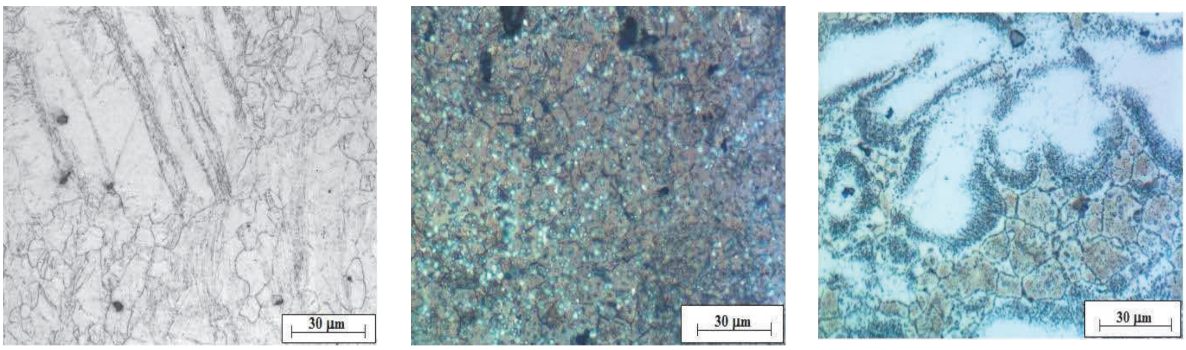
3.1. Structure

From the analysis of the microstructure of the default state of the given alloys using the optical microscope (**Figure 4**), it can be drawn that the microstructure of all types of alloys shows features characteristic for the particular chemical composition and processing. Hot-extrusion in as cast state has an impact on the refining of the originally cast grain [7].

The microstructure of the AZ31 alloy without any addition of lithium (**Figure 4a**) comprises basic matrices based on the solid solution of magnesium with additional Al and Li. In the grain boundary area, a minor intermetallic phase based on $Mg_{17}(Al, Zn)_{12}$, typical for Mg-Al-Zn alloys, can be observed. Also, the presence of Al_6Mn-based precipitates can be detected.

The microstructure of the AZ31 alloy with an additional 7.5 wt.% of lithium (**Figure 4b**) is characterized by its two-phase composition of solid lithium solution in magnesium (α), characterized by its hcp lattice and magnesium in lithium (β), characterized by its crystallographic bcc lattice. Also, there is an apparent presence of the intermetallic phase AlLi.

The alloy with 15 wt.% of lithium (**Figure 4c**) is specific by a presence of a single - phase microstructure of the solid solution.

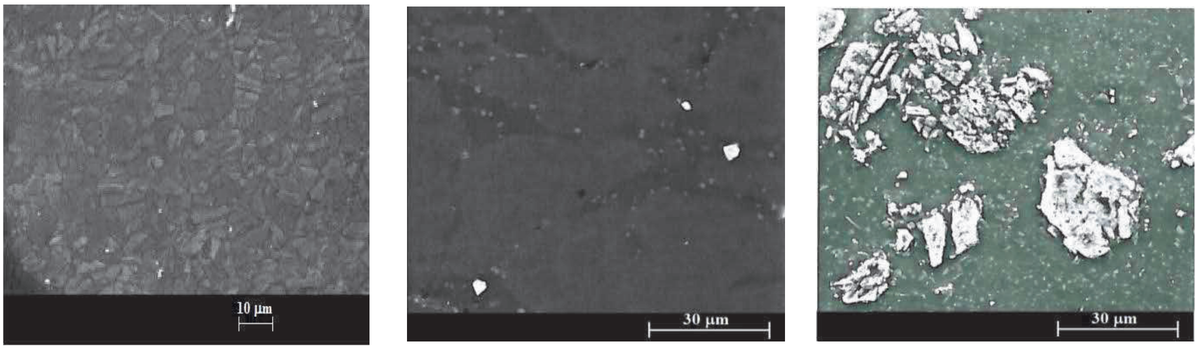


a) b) c)

Figure 4 Microstructure of alloys - initial state: AZ31 **(a)**, AZ31+7.5 wt.% Li **(b)**, AZ31+15 wt.% Li **(c)**

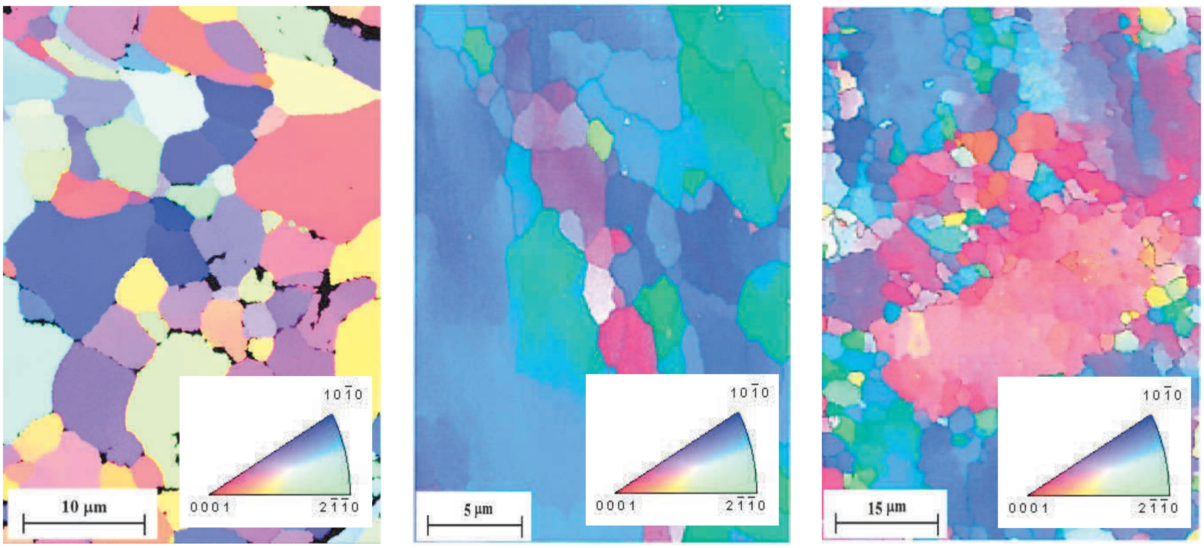
3.2. Structure after ECAP process

Based on the analysis of the microstructure carried out with the electron microscope SEM (**Figure 5**), conclusions can be drawn, which correspond with the used literature [7-10]. It can be said that after the third forming operation applying the ECAP method, the microstructure of the alloy AZ31 + 7.5 wt.% Li was composed of a rough phase of magnesium and LiAl. The alloy containing 15 wt.% of lithium comprises a relatively fine phase of magnesium and Li_2MgAl.



a) b) c)

Figure 5 SEM analysis of the tested alloys after 3 ECAP passes: AZ31 **(a)**, AZ31+7.5 wt.% Li **(b)**, AZ31+15 wt.% Li **(c)**



a) b) c)

Figure 6 EBSD of the tested alloys after 3 ECAP passes: AZ31 **(a)**, AZ31+7.5 wt.% Li **(b)**, AZ31+15 wt.% Li **(c)**

Results of the EBSD analysis of crystallographic orientation of the analyzed alloys after the third pass using the ECAP method, presented in **Figures 6a - 6c**, show refinement of the original microstructure and a potentially significant role of dynamic recrystallization in the course of the ECAP process [11]. The microstructure of the analyzed alloys appears to be bimodal, showing the existence of minuscule grains, as well as an area with a presence of coarse, non-refined part of the structure. The map of crystallographic orientation of grains, acquired by the EBSD method, may not clearly indicate the presence of grains/sub-grains with a very small angle of disorientation (angles below 1°).

The average size of the grain in the analyzed alloys is presented in **Table 2**. The AZ31 alloy without any additional lithium, after pressing in the default state, has an average grain size of 18.01 µm. Alloys, which contain lithium, have typically a grain size of 28.4 µm in the alloys with 7.5 wt.% of Li, and 31.2 µm with 15% of Li. The development of the grain size after the first forming operation by ECAP clearly shows that the original grain is disintegrated into small sub-grains. After the third ECAP operation, a microstructure was achieved, which consisted of very small grains with heterogeneous distribution after the analyzed cross-section of the metallographic sample. The results of the measuring of the average grain size of all three analyzed alloys containing lithium show a higher effect of refinement of the original grain, which can be explained by the impact of the bcc lattice that enabled an activation of a higher number of slide systems, and thus a higher level of refinement of the original grain in comparison to the alloy crystallizing in the hcp lattice.

Table 2 Average grain size (by EBSD) of the analyzed alloys (μm)

Material	Initial state	1st ECAP pass	3rd ECAP pass
AZ31	18.0	6.4	4.2
AZ31 + 7.5 wt.% Li	28.4	5.1	2.3
AZ31 + 15 wt.% Li	31.2	8.9	3.5

3.3. Hardness evaluation

In order to determinate the structural homogeneity, hardness measurements were performed in the middle of the specimen. The Vickers hardness (HV) was measured on a cross-section plane, by imposing a load of 10 kg for 20 s.

Figure 8 shows the results of the hardness measurements of investigated alloys in the initial state (hot extruded-EX) and after severe plastic deformation using the ECAP method. It can be observed that the specimens are characterized by a homogeneous hardness distribution. It should also be noted that the use of intensive plastic deformation results in a hardness increase of approx. 21% (AZ31), 31% (AZ31 + 7.5 wt.% Li) and 51% (AZ31 + 15 wt.% Li).

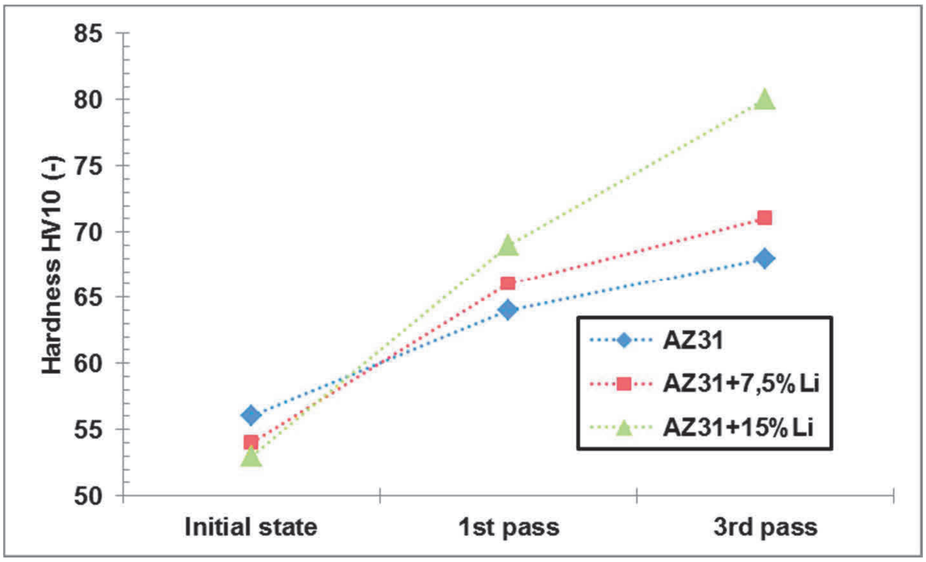


Figure 8 Hardness HV10 investigation of AZ31 **(blue line)**, AZ31+7.5 wt.% Li **(red line)** and AZ31+15 wt.% Li **(green line)**

4. CONCLUSIONS

The paper analyses the impact of the forming method ECAP on the reached refinement of the microstructure and a corresponding change of the strength of alloys AZ31, AZ31 + 7.5 wt.% Li, and AZ31 + 15 wt.% Li, which were pressed at a temperature of 430 °C in the default state. The initial pressing has an impact on a substantial refinement of the initial microstructure when the average grain size after casting is typically approximately 100 μm.

The microstructure of the alloy AZ31 without additional lithium is characterized by a solid solution of aluminum in magnesium and by the presence of intermetallic phase $Mg_{17}(Al, Zn)_{12}$, Mn-Al-based precipitates are also present in the microstructure. Alloys containing lithium are characterized by the existence of both phases (α + β), which have a considerable impact on the increase of plastic properties of these alloys, and thus on a potentially higher refinement of the structure. By using electron microscopy in the analysis of the samples with lithium, the existence of precipitates in the area of grain boundaries was proved, which were identified as AlLi (in the alloy with 7.5 wt.% of Li) and Li_2MgAl in the alloy with additional 15% of lithium, while crystallographic parameters of the individual phases of AZ31 + Li alloys were identical.

The above-mentioned results clearly show a positive impact of the ECAP method on refining of the grain size in all types of the tested alloys. During the first pass, a substantial refinement of the grain was achieved, and after the third passage, the average grain size was even smaller. However, a fine-grained microstructure composed of sub-grains separated by high-angle boundaries (grains with an average size of below 1 μm and disorientation angle higher than 12°) was not achieved. We have reached a relatively fine-grained microstructure with an average disorientation angle of approximately 10°. It is most probable that the selected pressing temperature with the ECAP of 250°C plays a major role in this case. An impact of softening processes can be presumed, namely dynamic recrystallization in the course of the ECAP forming process.

The amount of lithium has a positive impact on the increase of plastic properties of the AZ31 alloy, compared to the alloy without added lithium. An impact of the amount of lithium on the potential reduction of the pressing temperature of the ECAP method thus increased the efficiency of the refinement of the grain through the accumulation of dislocations in the microstructure and their following configuration and creation of sub-grains can be expected, therefore these studies shall continue in the following experimental works.

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