MICROSTRUCTURE EVOLUTION OF AZ31 MAGNESIUM ALLOY DURING TWIST CHANNEL ANGULAR PRESSING

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Microstructure evolution of hot extruded AZ31 magnesium alloy during twist channel angular pressing was investigated. The grains were refined significantly after one to four passes at 200°C. Microstructure evolution after TCAP processing was investigated using optical microscopy, transmission electron microscopy and electron backscatter diffraction. The results show effect of TCAP process on the achieving the UFG structure with average grain size below 1 µm.

KEYWORDS

Severe Plastic Deformation, ECAP, TCAP, Mg alloys, Microstructure

1 INTRODUCTION

UFG materials have recently found increasing application in a wide range of industries [Cep 2016, Sajdlerova 2015, Necas 2019, Cada 1996, Cada 2003, Sternadelova 2019]. The thesis deals with increasing the efficiency of Severe Plastic Deformation (SPD) [Rusz 2003] of Equal Channel Angular Pressing (ECAP) [Valiev 2006] technology using the new progressive geometry of the segment tool in AZ31 alloys. These materials are currently used for their production mainly by an extreme plastic deformation [Rusz 2014, Valicek 2012, Maziarz 2022]. For the formation of these deformations, several well-known so-called "top-down" methods are used, which are based on the increasing density of dislocations during plastic deformation and the formation of a fine-grained or nanocrystalline structure. These methods include the equilateral rectangular channel extrusion (ECAP) method. The effectiveness of the new geometry of ECAP is then verified by evaluating the microstructure using an optical microscope and a TEM transmission electron microscope [Kvackaj 2012, Dutkiewicz 2006] and EBSD.

2 ECAP TOOL GEOMETRY

Tools with a built-in helix connect practically the ECAP method and the TE (twist extrusion) method. The main goal of using the helix created in this way is to simulate the back pressure and thus increase the extrusion force. This force ensures the creation of intense shearing deformation, which leads to higher grain refinement and better homogeneity of the achieved structure.

The tools are made of two segments, in which a channel with a square cross section with dimensions of 15×15 mm and a helix with a pitch of 10° and 30° is made. Both parts are pressed in a bandage and fixed to the hydraulic press. A diagram of both tools with built-in helixes is shown in Fig. 1 and 2.

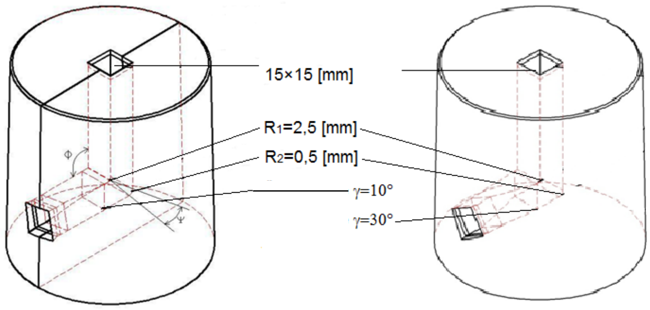

Figure 1. ECAP tool geometry diagram with 10° and 30° helix

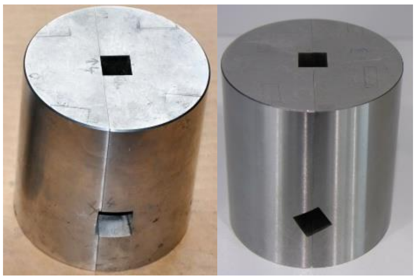

Figure 2. ECAP helix tools: (a) 10°, (b) 30°

Due to the high thermal and compressive stress of the tools, both ECAP tools were hot-pressed into special bandages of circular cross-section. This bandage is further fixed to the base plates with four guide pins using four screws made of high-strength steel. A pusher is subsequently fixed at the top of the base plates. This entire system is fixed to the given hydraulic press. For both tools, special extrusions were made of tool steel with exact dimensions within the necessary tolerances. The extruder is fixed by means of a centering washer and a screw to the upper base plate. The ends of the extrusions are equipped with a special facet that prevents the reverse flow of the test material.

2.1 ECAP induction tool heating

To improve the technological process and reduce the deformation resistance of magnesium alloys, ECAP tools are heated to the desired extrusion temperature using a special heating sleeve. This heating sleeve is fixed around the tool and its temperature is regulated by the compact JUMO dTROM 304 controller with thermocouple. Fig. 3

Figure 3. Induction tool heating

2.2 Induction material heating

Due to the poor formability of magnesium and magnesium alloys at ambient temperature (approx. 23 °C), the material selected for experimental verification must be preheated to a suitable forming temperature. The M20VA electric induction furnace is used for this purpose in the laboratory of the Department of Mechanical Technology. Fig. 4

Figure 4. Electric induction furnace M20VA

2.3 Lubricant

As a lubricant to reduce material friction in the extrusion tool, a special metal-based assembly paste NICRO Thermocup 1200 was chosen. With its unique composition, this lubricant protects all separable joints against seizure. Contains chemically pure aluminum, zinc and steel particles that perfectly fill and compensate for surface unevenness, creating a highly durable, separating layer with superior lubricating effects. Its properties include a completely unique coefficient of friction, which is 0.1. It has good pressure resistance, so it is an ideal lubricant for sliding surfaces with heavy mechanical loads, reliably operates in aggressive environments and is usable in the temperature range from -180 °C to +1200 °C at pressures up to 220 MPa.

3 EXPERIMENTAL PART

Experimental verification of the new geometry of the ECAP helix tool was carried out on a series of samples of AZ31 magnesium alloys, where four material samples were evaluated from each series after each individual pass. Between each passage, the sample was rotated by 90°. As part of the laboratory execution of the process, the test parameters were recorded and the results obtained on samples after the 4th pass, 3rd pass, 2nd pass and 1st pass through the ECAP tool with a 30° helix were evaluated. The evaluation of the achieved results is described in the following chapters. Following previous experience dealing with the influence of extrusion rate on the resulting structure and homogeneity of aluminum alloy material, the basic extrusion rate was determined to be 40 mm/min (approx. 0.7 mm/s). The temperature of the extruded material and the temperature of the ECAP helix tool were set at 200 °C. This temperature was achieved for magnesium alloys using an electric induction furnace and for the ECAP tool using a special induction heating cuff, see above.

3.1 Magnesium alloy AZ31

To carry out the experimental verification of the ECAP tool with a built-in 30° helix, the initial material state was used. The gravitationally cast ingot was pushed forward at a temperature of 430 °C in a protective atmosphere of argon into bars of 40x40x1000 mm. Samples for extrusion using the ECAP method with the modified geometry of the extrusion tool were subsequently prepared from these bars. An overview of the chemical composition of magnesium alloy AZ31 is given in table 1.

Chem. comp.	Al	Zn	Mn	Cu	Si	Fe	Mg
[%]	2,970	0,760	0,250	0,002	0,020	0,001	rest

Tab 1. Chemical composition of magnesium alloy AZ31

3.2 Metallographic evaluation of structure using optical microscopy

The evaluation of structural characteristics after individual passes, ECAP was carried out using the NEOPHOT 2 optical microscope. The evaluation of the resulting structures of magnesium alloys should be based on knowledge of the structures of the initial state. (Fig. 5) The microstructure consists of a basic solid solution of aluminum and zinc, dissolved in a magnesium matrix. Probably, this is the occurrence of a small amount of the $Mg_{17}Al_{12}$ phase, which is primarily excreted along the grain boundaries. From the resulting metallurgical evaluation of the AZ31 alloy after the 1st pass through the ECAP tool (Fig. 6) a softening of the grain and a partial improvement in the homogeneity of the structure compared to the structure before the ECAP method are noticeable (Fig. 5). With the increase in the number of sample passes with the new ECAP tool, the refinement of the grain of the alloy together with the homogeneity of the structure escalates to the 4th pass, where a fine and homogeneous structure is achieved (Fig. 6-9).

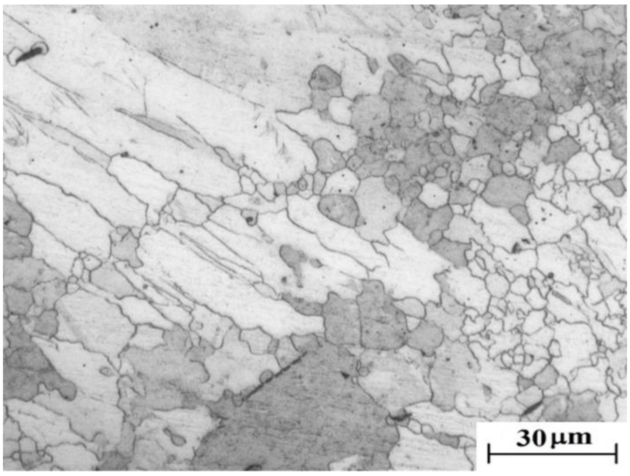

FIgure 5. Magnesium alloy AZ31 – initial state

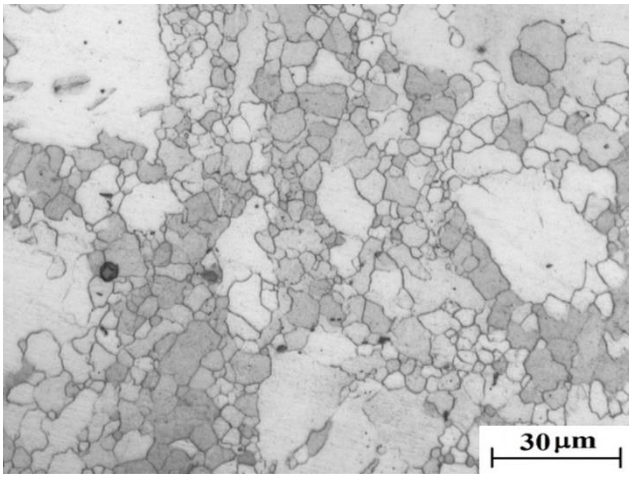

Figure 6. Structure of magnesium alloy AZ31 (after hot extrusion) after the 1st pass

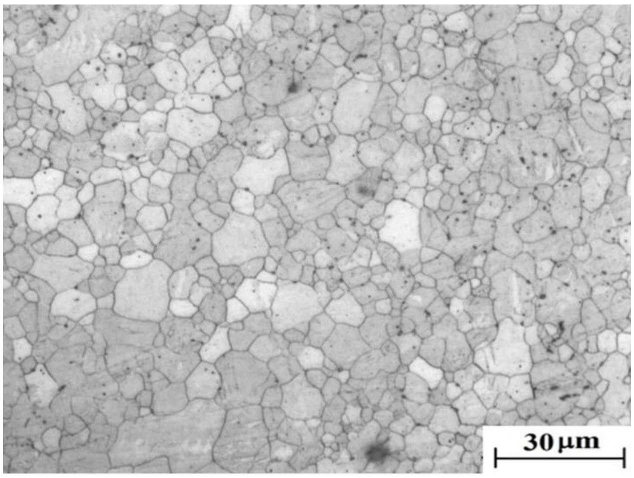

Figure 7. Structure of magnesium alloy AZ31 (after hot extrusion) after the 2nd pass

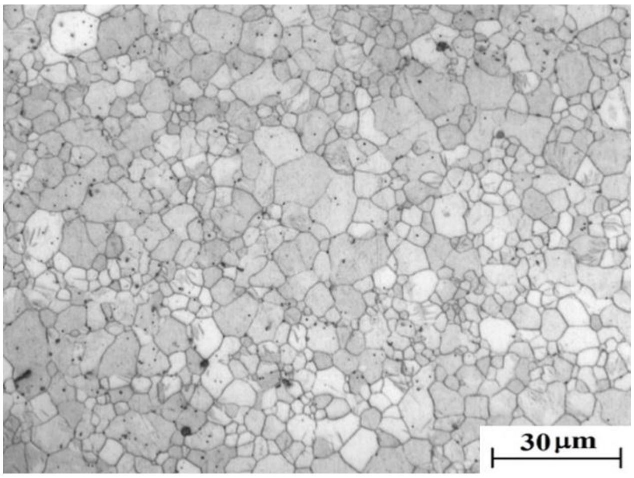

Figure 8. Structure of magnesium alloy AZ31 (after hot extrusion) after the 3st pass

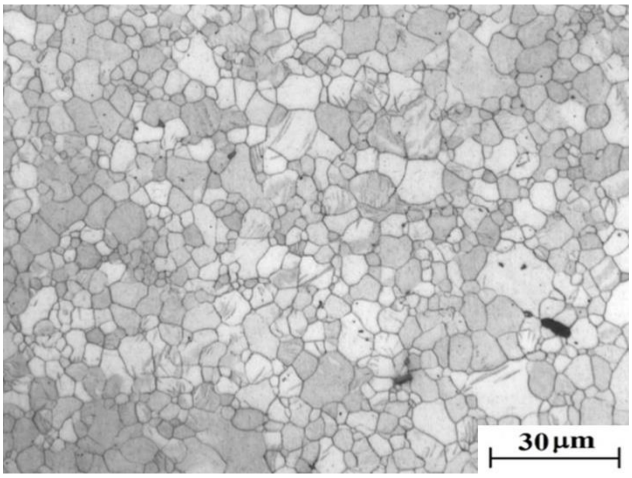

Figure 9. Structure of magnesium alloy AZ31 (after hot extrusion) after the 4th pass

3.3 Metallographic evaluation of structure using transmission electron microscopy

To verify the microstructure and grain size inside the material, AZ31 alloys were selected initial state and after the 4th ECAP passes. On selected samples of AZ31 alloy, TEM analysis was performed using the Philips CM20 transmission electron microscope.

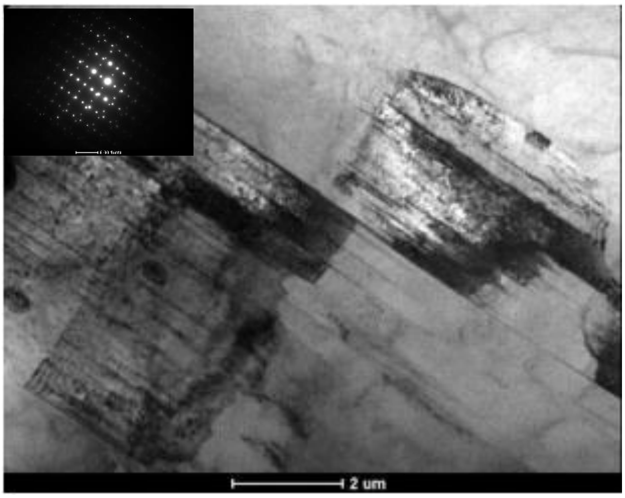

Figure 10. TEM analysis of AZ31 alloy before ECAP - longitudinal section, scale 2 µm

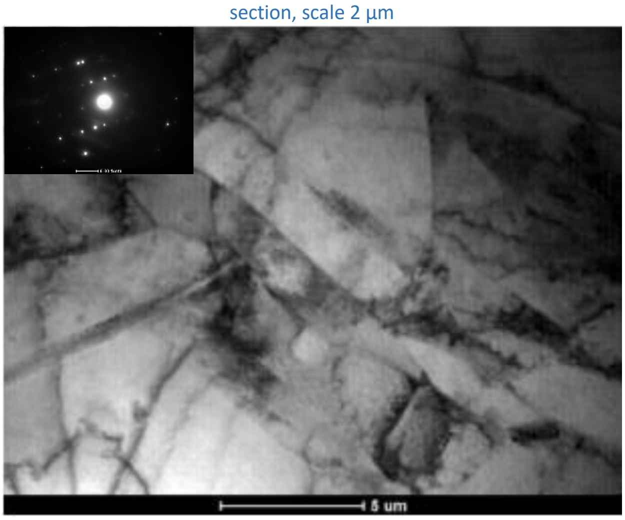

Figure 11. TEM analysis of AZ31 alloy before ECAP - cross section, scale 5 µm

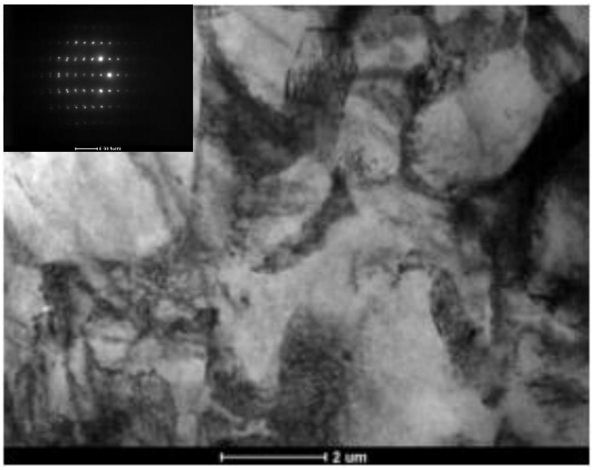

Figure 12. TEM analysis of AZ31 after 4th pass ECAP - longitudinal section, scale 2 µm

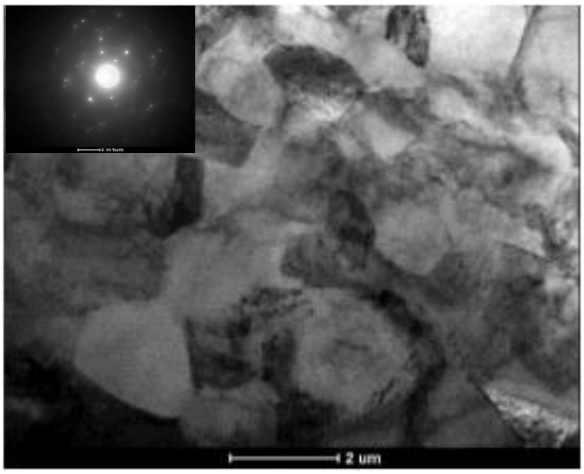

Figure 13. TEM analysis of AZ31 after 4th pass ECAP - cross section, scale 2 µm

Fig. 10, 11 shows TEM analyses of magnesium alloy AZ31 (Fig 10) longitudinal section, (Fig. 11) cross section at baseline. The grain size of AZ31 alloy prior to etrusion with the ECAP tool was measured in the range of 40–60 µm. Small intermetallic inclusions were also observed, occurring mainly along the grain boundaries. Figure 10 and 11 shows the initiation of high-density shear belts. Between these bands there are fine particles of intermetalic phases (either $Mg_{17}Al_{12}$ or Al_6Mn).

The structure of the AZ31 alloy after the 4th pass through the ECAP tool with a 30° helix is shown in fig. 13, 14. The achieved structure shows refinement with an average grain size of 1–2 µm. Small intermetallic inclusions are also observed, especially along the grain boundaries, which partially indicate the possibility of dynamic recrystallization.

3.4 Evaluation using EBSD

EBSD data was collected using high-resolution 3D FEI FEGSEM Quanta. Figures 14 and 15 show images from the initial state, both transverse (Fig. 14) and longitudinal (Fig. 15) status. Figures 16 and 17 show images of 4 ECAP passes, both transverse (Fig. 16) and longitudinal (Fig. 17) status.

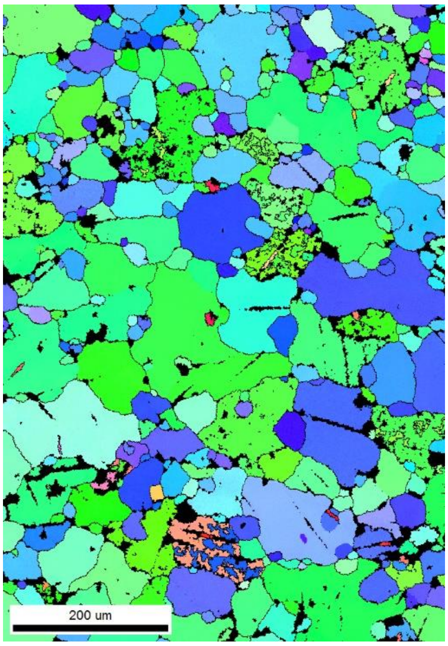

FIgure 14. IPF of AZ31 initial state – cross section

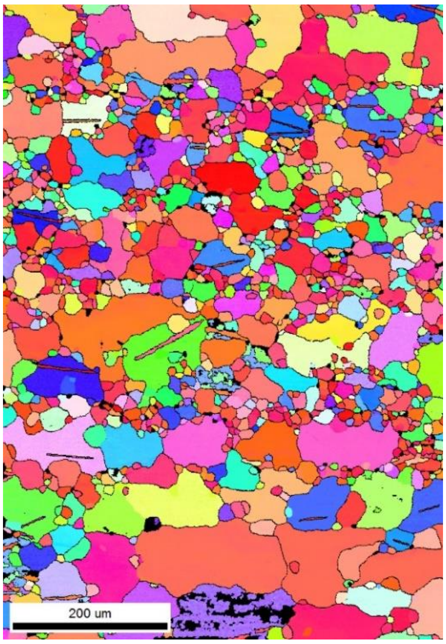

FIgure 15. IPF of AZ31 initial state – longitudinal section

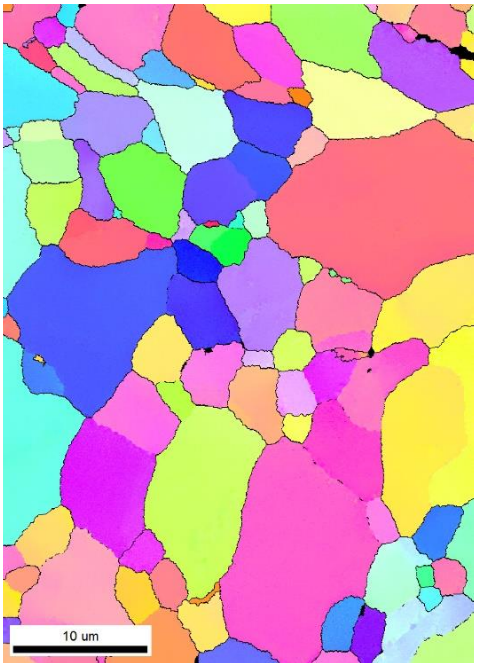

Figure 16. IPF of AZ31 after 4th pass ECAP - cross section

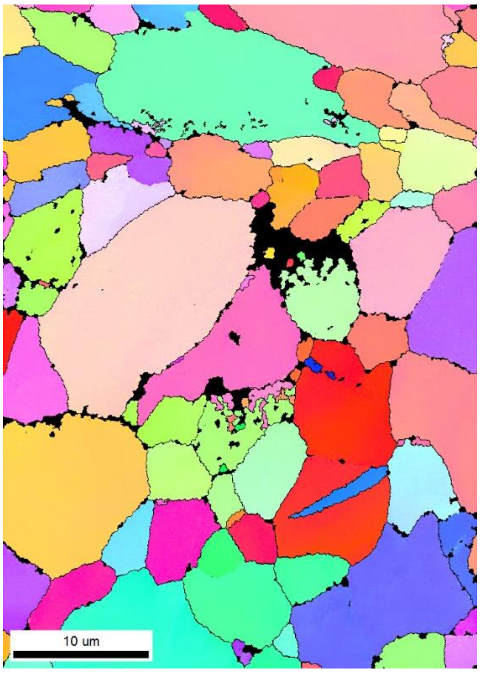

Figure 17. IPF of AZ31 after 4th pass ECAP - longitudinal section

EBSD analysis confirmed grain refinement after ECAP passes. At initial state in the transverse direction, the average grain size was 25 µm, in the longitudinal state the average grain size was 15.9 µm.

After 4th ECAP passes in the transverse direction, the average grain size was 3.5 µm, in the longitudinal state the average grain size was 3.2 µm.

4 CONCLUSIONS

From the metallographic analysis on the optical microscope, it turned out that with the increase in the number of passes with the new ECAP tool, the refinement of the grain of the alloy together with the homogeneity of the structure escalates to the 4th pass, where a fine and homogeneous structure is achieved.

From the TEM analysis, the grain size of AZ31 alloy was measured in the range of 40–60 µm before extrusion with the ECAP tool. The structure of AZ31 alloy after the 4th pass through the ECAP tool with a 30° helix shows refinement with an average grain size of 1–2 µm. Small intermetallic inclusions are also observed, especially along the grain boundaries, which partially indicate the possibility of dynamic recrystallization.

A detailed EBSD analysis confirmed the softening of the grain after ECAP passes. At initial state in the transverse direction, the average grain size was 25 µm, in the longitudinal state the average grain size was 15.9 µm.

After 4 ECAP passes in the transverse direction, the average grain size was 3.5 µm, in the longitudinal state the average grain size was 3.2 µm.

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