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A Study on Microstructure and Mechanical Properties of AZ31 Magnesium Alloy after Constrained Groove Pressing

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Abstract. This study the AZ31 magnesium alloy trips of the dimension $60 \times 60 \times 3 mm$ was used for constrained groove pressing (CGP) after 4 cycles under elevated temperature condition. Microstructure and mechanical properties were analyzed for a material with ultra-fine grained structure prepared by CGP of AZ31 magnesium alloy. Analysis of the scanning electron microscope (SEM) micrographs observations showed significant drop of the grain size from (50 \div 60) μm to (4 \div 6) μm after four CGP cycles. The minimal grain size of (1 \div 1.5) μm was detected in the regions of the highest deformation. Moreover, the tensile test along the *x* and *z* directions showed excellent high strength (325 MPa) and also plastic elongation of 35 %. It is shown that the CGP technique is a good tool for the grain refinement and improving mechanical properties of magnesium alloys.

Keywords: severe plastic deformation; constrained groove pressing; magnesium alloy; microstructure; SEM; mechanical properties.

1. Introduction

Due to its significantly high durability, ultrafine grained (UFG) materials based on metals, light alloys have been strongly developed for applications in the automotive and aircraft industries to make finishing parts. The evolution of microstructure to improve the mechanical properties of metals such as hardness and ductility in plastic deformation was extensively studied several decades ago [1-3]. It has been confirmed that deformation at room temperature (or warm) makes the average particle size of metals drop significantly. Severe plastic deformation (SPD), developed in the 1970s by Russian scientists is an effective method for creating ultrafine grained structure materials.[4] During the process of the SPD method, large hydrostatic pressure is used to reduce lattice defects of metals. Severe plastic deformation method is defined as the process of forming metal with very high deformation can reduce particles during processing. The particle size of the material after being fabricated by this method is usually less than 1 μm , depending on which SPD technique is used. These methods include: Equal Channel Angular Pressing (ECAP, Mul-ECAP) [5-10], High Pressure Torsion (HPT) [11].

A severe plastic deformation method with great potential for the production of ultrafine materials has been developed as a CGP [12,13]. In 2001, Zhu undertook a SPD method based on the method of squeezing a flat metal plate with a cyclic groove die and then restoring the original planar shape to the flat die. Repetition of this process repeatedly causes large plastic deformation to accumulate in the sample without significantly altering the initial size (Figure 1). One of the main benefits of the CGP technique is the improvement of mechanical properties without any change in the size of the material.

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In this study the AZ31 magnesium alloy was used for CGP. The investigation of the strain homogeneity along the material was done by the tensile test. The SEM were evolution observation of microstructure after CGP.



Figure 1. Schematic representation of CGP process





Figure 2. Designed grooved die (a), flat die (b)



The designed grooved die (a) and flat die (b) are given in Figure 2. The sheets are subjected to total number of 16 passes (four cycles) of CGP and further processing could not be continued beyond due to cracking of plate. Every CGP cycle consists of 4 passes and 9 steps (Figure 3). In the 1st pass (1-2), a specimen is placed in the die with slight gap which is equal to the sheet thickness (1) and groove pressing begins (2). In the 2nd pass (3-4), flat pressing occurred (3); after that the specimen is rotated by 180 ° around the z direction and the groove pressing is repeated (4). In the 3rd pass (5-6), flat pressing occurred and followed by a move of t to right (5) and then a groove pressing (6). In the 4th pass (7-8), flat pressing occurred (7); after that the specimen is rotated by 180 ° around the *x* direction and moves to left and the groove pressing is repeated (8). At the end, the specimen is flattened again and rotated by 180 ° around the *z* direction (9) and the groove pressing of the next cycle is started (1). The specimen has been heated to 315 °C together with the dies during CGP. The maximum pressing load is 100 tons.

The AZ31 magnesium strips of the dimension $60 \times 60 \times 3 \text{ mm}$ anneal at 320 °C for 2 h and then furnace cooled have been used as an input material. The microstructure micrograph and the chemical composition (measured on an X-ray) of AZ31 magnesium alloys are showed in Figure 4 and Table 1. The change of microstructure after deformation associated with the CGP processing at 315 °C has been investigated by the Scanning Electron Microscope (SEM). Grinding of the test specimens has been conducted with SiC papers of different grades (80, 100, 200, 400, 600, 800 and 1000) using automatic grinding machine. Then ground samples are polished with fine diamond paste of 1 μm . All samples have been kept in ethanol between the different polishing steps to avoid the hydrate reaction. Etching has been done at room temperature in $(10 \div 40) s$ in a mixture of ethylene glycol, acetic acid and nitric acid. The mechanical properties in different directions (x and z) of specimens are measured using tensile test, which is performed at room temperature with universal testing machine at cross head speed of 2 *mm/min*.

The tensile specimens with the gauge dimensions of 20 mm in length, 5 mm in width, and 2.8 mm in thickness are prepared using wire-cutting machine.



Figure 4. Initial microstructure of the AZ31 samples Table 1. AZ31 chemical composition

ELEMENT	WEIGHT %
MG	95.55
AL	2.88
SI	0.07
CA	0.18
MN	0.35
FE	0.12
NI	0.13
CU	0.09
ZN	0.63

3. Results and Discussions

3.1. Microstructure

The SEM analysing in flat top (undeformed area 1, Figure 5a) showed the microstructure after first pressing consisted of elongated and/or equiaxed subgrains maybe segmented by dislocations. Extensive twinning found also in almost (1, 2 and 3) areas of the specimen implied that twinning is prevalent mechanism of deformation at one complete CGP cycle [12]. The presence of subgrain microstructure is an evidence that this region has also undergone quite a large amount of plastic deformation and it couldnot be denoted as an "undeformed" area, Figure 5 (a). The mechanical processing process consists of a complete CGP cycle leading to a microstructure that represents heterogeneity in the x direction. Areas with different grain sizes were observed. The grain size is reduced down to 20 µm in the deformation regions. Observation over wide microstructure areas (flat area 1 and sheared area 2) suggests a role of dislocations in formation of the substructure already observed during the first pressing. Magnesium alloy AZ31 is a polycrystalline material in which the orientation of the grains is different and between them are grain boundaries, whose the distorted atomic order obstructs the dislocation motion. Under the external forces (shear stress), because of difference in grain orientation, the slip starts only in a few well-oriented grains. The remaining grains only elastically deform and rotate to ensure the compatibility (or continuity) of the deformation. The continuous increase in external force and the relative rotation of the grains may gradually activate many other slip planes. When most of grains plastically deformed, the permanent strain of the sample will be important. Like stresses, the number of slip planes is also dependent on the deformation temperature. High temperatures enhance the dislocations motion and facilitate the rotation of the non-deformable grains, contributing to the increased number of activated slip systems.



Figure 5. SEM micrographs of sample areas: (b) –area 1, (c) - area 3 and (d) – area 2 Dynamic recrystallization takes place in the temperature range that magnesium is usually processed by CGP ($150 \div 327 \ ^{\circ}$ C) [13-16]. Refinement in magnesium alloy occurs in the way that new grains are seeded at the grain boundaries and twins of the old grains. This leads to a necklace distribution of fine grainss around coarse grains. Therefore, a multimodal distribution of grain size is expected at the early deformation stage of magnesium alloy.

This can be seen in Figure 6, where fine grained and coarse particles are interleaved. This multimodal distribution of grain sizes will be replaced by a homogeneous distribution of fine grains when the new fine grains consume the cores of the initial coarse grains. This is observed after 4 cycles of CGP in the AZ31 alloy in Figure 6d,e. Heating at 315 °C for each cycle of the CGP process dissolved the β -particles and, thus, no secondary phases inside the grains were observed. Because the material exhibits rather inhomogeneous complex microstructure containing also large elongated grains, the refinement of the microstructure by CGP technique was made.

Figure 6a,b,c,d shows the (yz) face of the AZ31 sample. The material exhibits inhomogeneous microstructure along the cross-section (yz face) with pronounced columnar structure. There are different grain structures from coarse (a) to intermediate (b), fine (c), and ultra-fine grains (d) after 1, 2, 3 and 4 cycles of CGP respectively. It also shows a possible difference in size of the newly formed grains in relation to amount of strain imposed. Namely, the Figure 6a,b corresponds to the 1, 2 cycles of CGP (low strain) while the Figure 6c,d to 3, 4 cycles (high strain). That multi-modal distribution of grain sizes are observed when comparing the large size ($60 \ \mu m$) of initial grains with the small size ($4 \div 6$) μm of the new grains and the minimum grain size ($1 \div 1.5$) μm . A homogeneous distribution of fine grains is

readily obtained in the early cycle of CGP if the initial grain size is sufficiently small to be totally consumed by the new grains.

In the (xz) face (Figure 6e) the larger columnar grains with $(10 \div 15) \mu m$ of length are observed, while smaller grains have a size of about 2 μm .



Figure 6. Microstructure evolution of AZ31 sheet after 1 (a), 2 (b), 3 (c) and 4 (d,e) cycles of CGP

3.2. Mechanical Property



Figure 7. Tensile curves of the initial and CGPed AZ31

It's known from the Hall–Petch relation that the grain size has a direct influence on the mechanical properties of metals [17]. The analysis of the tensile test results along the x and z directions show a significant difference for the initial and CGPed samples (Figure 7). The initial alloy exhibits lower strength (235 MPa) and strain (12 %), while for the CGPed material tensile test values along the x and z directions are always higher. When the strength remains more and less 325 MPa with 24 % of strain (for x direction) and increases to 35 % (for z direction) after CGPed 4 cycles. This finding is matched with HV increase in AZ31 alloy after two cycles of CGP [18] and would be a good evidence of the microstructure evolution during CGP process resulted in grain refinement as discussed above.

4. Conclusion

The AZ31 alloy was used for the preparation of the fine-grained material with improved mechanical properties. The magnesium alloy AZ31 is the CGPed after 4 cycles under elevated temperature condition. The main results are obtained as follows.

The microstructure evolution in the CGP process and its effect on deformed behavior of AZ31 alloy has been established. Analysis of the SEM micrographs confirmed a grain refinement with distinct twinning caused by the dynamic recovery and recrystallization processes. The grain size reduced from $(50 \div 60)$ μm down to $(4 \div 6) \mu m$. The minimal grain size of $(1\div 1.5) \mu m$ was detected in the regions of the highest deformation.

The mechanical properties of AZ31 magnesium alloys showed high strength (325 MPa) and elongation (35 %) after four cycles. Because the CGP groove and flat pressing have been performed within the same container combined with the multi-cycles, the CGP technique can be more suitable method for forming as well as improving the mechanical properties of CGPed AZ31 magnesium alloys.

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