

SUPERPLASTIC DEFORMATION OF TWO PHASE MgLiAl ALLOY AFTER TCAP PRESSING

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Abstract

Magnesium based alloy containing 9 wt. % Li, 1,5 wt. % Al, composed of $\alpha + \beta$ (hcp + bcc) phases was cast under argon atmosphere and extruded at 350°C as bars of cross section 12x12 mm. Twist Channel Angular Pressing TCAP deformation was applied at a temperature of 160°C using B_C rotation of samples in the following passes. TCAP tool consisted of helical part in horizontal area of the channel with an angle of lead $\gamma = 30^\circ$ to simulate back pressure and thus to increase the extrusion force. The initial grain size of the hexagonal α phase estimated at 30 μm decreased in the following passes down to 6 μm and that of bcc β phase decreased after TCAP from initial 12 μm , down to 5 μm . TEM studies after TCAP passes showed higher dislocation density in the β region than in the α phase. Crystallographic relationship $(001) \alpha \parallel (110) \beta$ indicates parallel positioning of slip planes of both phases due to activation of slip. Electron diffraction indicates increase of grain misorientation with a number of TCAP passes. Stress/strain curves measured at temperature of 200°C show superplastic forming after 1st and 3rd TCAP passes with better superplastic properties with increasing number of passes. Values of strain rate sensitivity coefficient m were calculated at 0.31 after 1 TCAP pass using flow stress values from step strain increase curves. The sample after 3 TCAP passes showed increase of m value up to 0.47 for the strain rate range $10^{-5} - 5 \times 10^{-4} \text{ s}^{-1}$. Obtained values are slightly lower than those reported in the literature, however increase of a number of TCAP passes has a positive effect on superplastic properties due to fine grains and increase of their misorientation

Keywords: MgLiAl alloys, superplastic forming, TEM

1 Introduction

Severe plastic deformation using ECAP causes substantial grain refinement and therefore improves mechanical properties of magnesium base and other metals [1-4]. Mg-Li alloys show

improved formability through activation of non-basal slip systems [5] particularly prismatic slip as confirmed by texture studies [6]. The addition of Li hinders the activation of $\{1012\}\langle 1011\rangle$ tension twinning, however the addition of aluminum decrease the resistance to this phenomena [7]. Slightly higher addition of Al up to 4 % causes decrease the yield stress with increasing temperature what was attributed to activation of cross slip at elevated temperatures [8]. Mg 8 %Li alloy shows excellent superplastic properties after equal channel angular pressing at 473K and the strain rate 10^{-4} s^{-1} with the strain rate sensitivity coefficient oscillating between 0.4-0.6 [1]. ECAP processing at higher Li content with β bcc structure have shown substantial refinement of grains down to 200 nm and existence of MgLiAl_2 precipitates due to Al addition [6]. Superplastic deformation was also observed in two phase Mg 8% Li 2% Zn alloy after grain refining using rolling with rather nonhomogeneous grain distribution from 1-2 μm to several μm [9]. Nevertheless it allowed to obtain tensile elongation from 100-400 % at temperatures 423-523K even at moderate grain refinement. It was confirmed in rolled Mg 9% Li 1,8% Al 1,6% Zn alloy where also above 500% superplastic deformation was observed in two phase $\alpha + \beta$ alloys [10]. Authors suggested that between 473-523K a partial dynamic recrystallization takes place, while at higher temperatures a complete recrystallization occurs. Even extruded alloys have shown superplastic deformation of Mg8% Li alloy [11]. It was shown that the amount and size of cavitation increase when the strain rate decreases what is explained by diffusion of vacancies. Very effective method of grain refinement of high pressure torsion HPT was applied to Mg 8% Li alloy, leading to grain refinement of average size 500 nm. Superplastic deformation was observed at temperature of 323K or higher and at higher strain rates between 10^{-3} and 10^{-2} s^{-1} , higher than reported so far [1, 2-5, 10]. From the microstructure studies performed after superplastic deformation of two phase alloys reported in [12] it was found that grain boundary sliding (GBS) is the dominant deformation mechanism. Twist Channel Angular Pressing (TCAP) method was chosen since in [13] a high effectiveness of the TCAP process is confirmed by the lower number of passes in comparison with the classical ECAP.

In the present paper superplastic deformation characteristics of two phase $\alpha + \beta$ MgLi9Al1.5 was investigated. The changes of microstructure and effect of TCAP deformation were examined in alloys of $\alpha + \beta$ phase composition. A small aluminum addition was applied to MgLi alloys in order to increase their mechanical properties as reported in [14].

2 Experimental Procedure

Magnesium based alloy of composition 9 wt. % Li, 1,5 wt. % Al composed of $\alpha + \beta$ (hcp + bcc) phases were cast under argon atmosphere and extruded at 350°C to obtain bars of cross section 12x12 mm for TCAP deformation. TCAP tool; consists of helical part in horizontal area of the channel with an angle of lead $\gamma = 30^\circ$. The basic aim of use of helix was to simulate back pressure and thus in increase an extrusion force. Up to 3 TCAP passes, there was applied using B_C rotation of samples in the following passes with measurement of the pressure force during the process. The hardness of samples was tested using using a Zwick ZHU 250 instrument using Vickers method and the tensile tests were performed using Instron 6025 - testing machine at room and elevated temperatures using samples cut out after ECAP using electro spark machine of thickness 2 mm and testing length 18 mm. The structure and composition was studied using Philips CM20 transmission electron microscopes. Thin samples of hot pressed or TCAPed alloys were cut by electro spark, then dimpled and electropolished in electrolyte consisting of 750 ml AR grade methanol, 150 ml butoxyethanol, 16.74 g magnesium perchlorate and 7.95 g lithium

chloride and finally dimpled using Gatan dimpler and ion beam thinned using Leica EM RES101 ion beam thinner. X-ray diffraction was performed using a Philips PW 1710 diffractometer with Co K α radiation.

3 Results and discussion

Fig. 1a shows optical micrographs from alloy after extrusion at 350°C and after TCAP pass at 160°C. Microstructure of extruded alloy shows elongated particles of hexagonal α phase visible as bright and darker bcc β grains. In extruded alloy α lamellar grains are placed in the extrusion direction and the grain size of the α phase was estimated at about 20 μm and that of β phase near 12 μm . As one can see in **Fig. 1b** after TCAP the lamellae are much finer and strictly set parallel to the pressing direction. The lamellae are often broken and finer after TCAP pressing at 160°C. The grain size however cannot be estimated from this microstructure and TEM studies were performed. **Fig. 2** shows graphs presenting stress versus displacement relation during TCAP pressing of investigated alloy at 160°C after 1-3 passes. One can see that stress initially increases up to about 370 MPa and then stabilize near 200 MPa. The graphs for the following passes have a similar character with a little lower stabilization stress between 130 and 145 MPa.

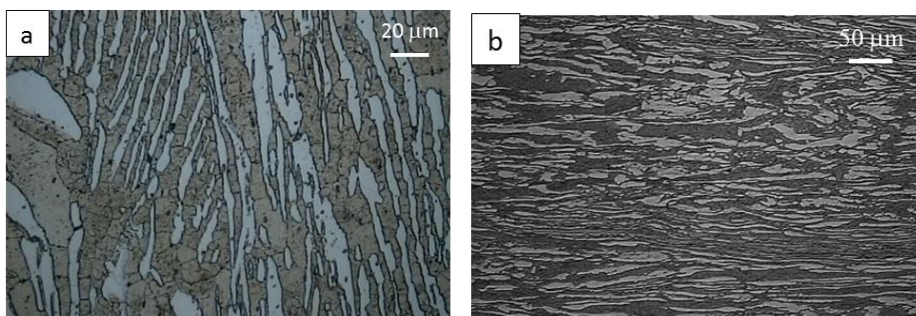


Fig. 1 (a) Optical microstructure of alloy MgLi9Al1,5 extruded at 350°C and (b) The alloy after 1 TCAP pass

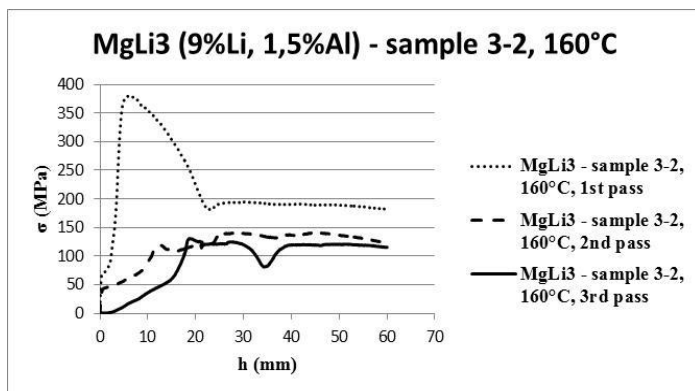


Fig. 2 Stress versus displacement relationship during 1-3 TCAP passes of extruded alloy MgLi9Al1,5 at 160°C

The grain size of the hexagonal α phase decreased after the 1st TCAP pass at 160 °C from about 30 μm down to 6 μm and that of β from about 12 μm , down to 5 μm . **Fig. 3** shows TEM

micrograph taken from the alloy after 3 TCAP passes, where non-homogeneity of the grain size from exceeding $4\ \mu\text{m}$ down to $1\ \mu\text{m}$ was observed. The diffraction pattern shows misorientation between individual grains manifested by diffused reflections toward Debye-Scherrer rings. There is also few degrees rotation from the crystallographic relationship between bcc and hexagonal phases $[1\bar{1}0]\ \beta\ \text{bcc} \parallel [0001]\ \alpha(\text{Mg})$ and $(110)\ \beta\ \text{bcc} \parallel (10\bar{1}0)\ \alpha$ similarly like observed in [15] where deviation of $0,6^\circ$ from the Burgers relationship was reported. The refinement of mixed α/β grains near grain boundaries was also observed in regions adjacent to α hexagonal grain what is illustrated in Fig. 4.

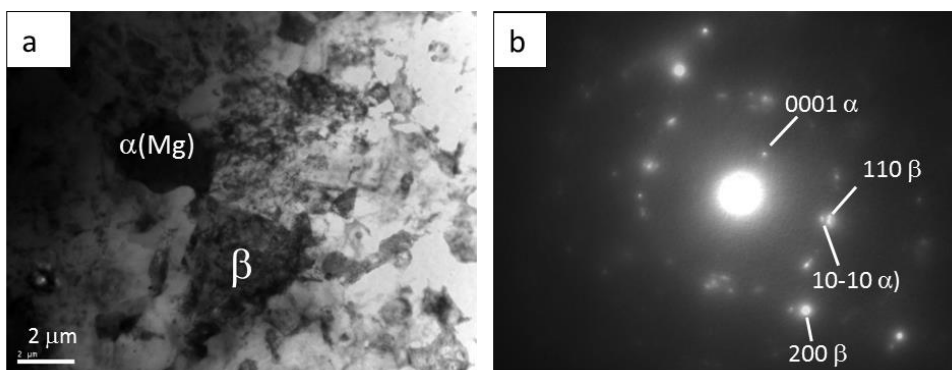


Fig. 3 TEM micrograph of the alloy 3 after 3 TCAP passes.(b) Selected Area Diffraction Pattern SADP from the area of two darker larger grains visible in (a)

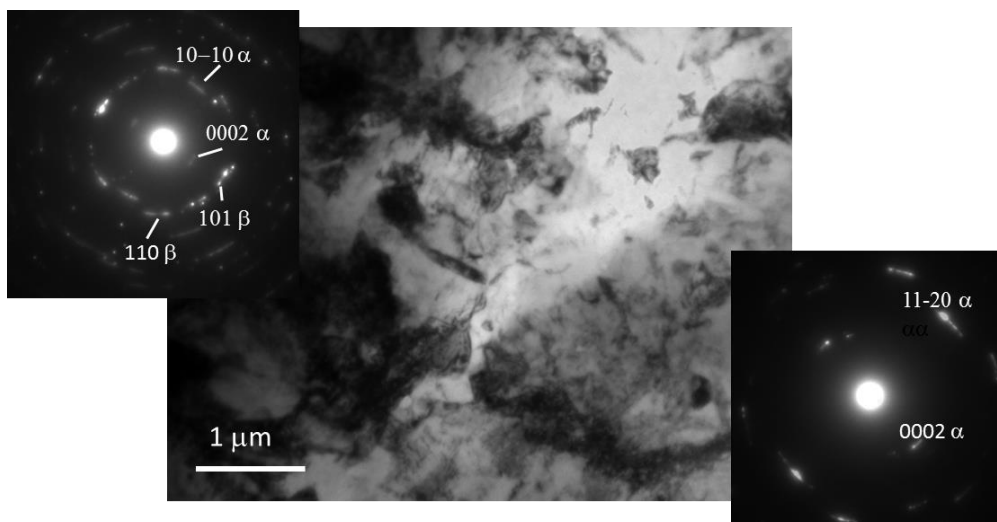


Fig. 4 TEM micrograph taken from the alloy 3 after 3 TCAP passes and tensile tested at $200\ ^\circ\text{C}$. SADP placed in the upper left corner indicate presence of the mixture of α and β phases and that shown in the lower right corner hexagonal α phase

One can see rather low density of dislocations in the hexagonal α phase due to more extensive deformation of softer β phase. What is characteristic α and β phases mix at the interfacial area most probably due to partial grain boundary sliding. Similarly like in the case of the diffraction

in **Fig. 3** there exist identical crystallographic orientation of α and β phases $\{110\} \beta \parallel \{01\bar{1}0\} \alpha$ and $\langle 110 \beta \parallel \{0001\} \alpha$. There are not many TEM studies of superplastic deformation of two phase MgLi alloys. In [14] it was found that the grains keep the characteristics of equiaxed ones after large deformation, indicating that grain boundary sliding (GBS) is a dominant deformation mechanism. However, presented microstructures in [14] show fine and also large grains, what was not commented and the microstructures were similar like that observed in the present paper. After High Pressure Torsion (HPT) process the microstructure consisted of small grains with an average grain size of 500 nm what was attributed to the effect of solute atoms on the mobility of dislocations [12]. Nature of diffused reflections along Debye Scherrer ring visible in SADP in **Fig. 4** suggests presence of low angle grain boundaries consisting of dislocations. Consequently a deviation from crystallographic relationship of both types of α and β phases due to presence of grain misorientation is observed. **Fig. 5** shows a plot of stress versus strain curves of samples from the investigated alloy after 1 and 3 TCAP passes measured at 200°C. One can see that the flow stress is lower for 1 TCAP pass than after 3 TCAP passes. It may result from growth of the grain size of α and β phases in the following passes. The elongation is also slightly higher for

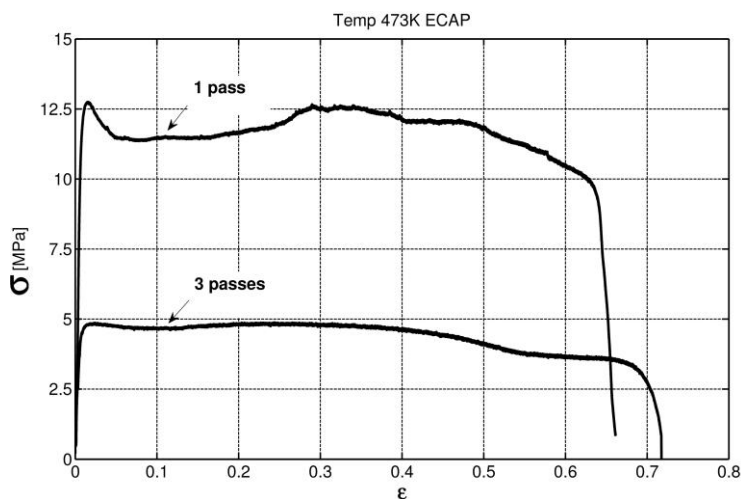


Fig. 5 Tensile curves of investigated alloy after 1 and 3 TCAP passes at 200°C

the sample tested after 1 TCAP pass. **Fig. 6** shows a plot of stress versus strain curves of the investigated alloy after 1 TCAP and 3 TCAP passes performed at temperatures 220° and 200°C at progressively increasing crosshead velocities from 10^{-4} - 10^{-2} s^{-1} . One can see that after changing cross head velocities the stress stabilizes at different values depending on tensile deformation rate. This method was applied after that reported in [16] studying superplastic titanium alloys. One can measure flow stress σ at different strain rates $\dot{\epsilon}$ and then calculate the strain rate sensitivity coefficient $m = d(\ln\sigma)/d(\ln\dot{\epsilon})$. This method is particularly convenient for experiments like ECAP where the amount of material particularly after larger number of passes is small and one can determine the m value from one tensile experiment. **Fig. 7** shows a graph of stress versus strain rates plots in logarithmic scale from step strain rate change tests shown in **Fig. 6**. Calculated values of strain rate sensitivity coefficient m are marked in all plots. One can see that in the case of two phase alloy after 1 TCAP pass the m values calculated using values in **Fig. 7** are equal to 0.31 within strain rates $10^{-4} - 5 \times 10^{-2} \text{ s}^{-1}$. The alloy sample after 3 TCAP

passes shows that value of m calculated from the step rate change in **Fig. 6** is equal to 0.47 for the strain rate range $10^{-5} - 5 \times 10^{-4} \text{ s}^{-1}$ accompanied by lower yield stress value. Obtained m values are slightly lower than those reported in the literature [9, 10] what may be caused by close to industrial conditions of casting and extruding.

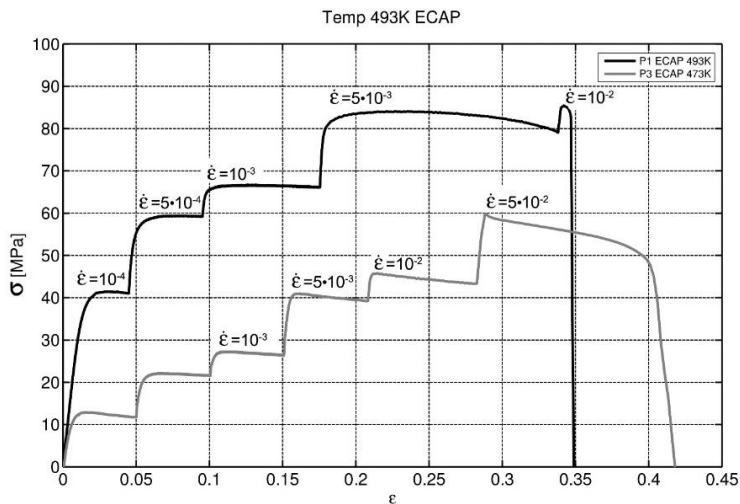


Fig. 6 Plot of stress versus strain curves of samples from investigated alloy after 1 TCAP pass and 3 TCAP passes performed at temperatures 220° and 200°C at progressively increasing crosshead velocities from 10^{-4} – 10^{-2} s^{-1}

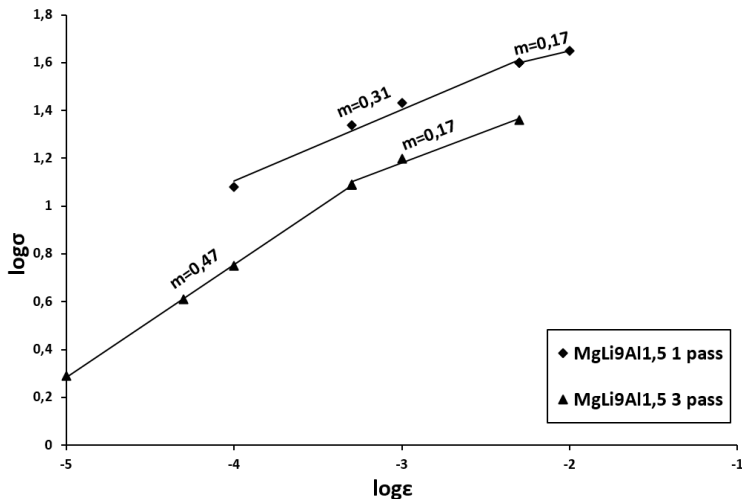


Fig. 7 Stress-strain rates plots in logarithmic scale from step strain rate tests for alloys 1 and 2 at superplastic deformation temperatures 220°C and 200°C respectively. Calculated values of strain rate sensitivity coefficient m are marked on the graph

4 Conclusions

1. Two phase $\alpha + \beta$ MgLiAl alloy possess after extrusion grain size of $30 \mu\text{m}$ of the α phase and $12 \mu\text{m}$ of β phase. Grains are refined after TCAP first pass at 160°C down to

about 5 μm of both phases. Higher dislocation density within β phase indicates that this phase is more easily deformed. Next TCAP passes cause incorporation of the α phase particles into β grains near grain boundaries probably due to grain boundary sliding.

2. Superplastic deformation was observed at 200°C for samples after 1-3 TCAP passes leading to tensile elongations above 70%. The strain rate sensitivity coefficient m measured at progressively increasing crosshead velocities from 10^{-4} - 10^{-2} s^{-1} was calculated for the investigated alloy after 3 TCAP passes at 0,47 for deformation rates below 10^{-3} s^{-1} and 0,26 for higher rates. Calculated m values after 1 TCAP pass were slightly lower.

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