Thermal stability and microstructure evolution of ultra-fine grained Al-Mg alloy

P. Snopiński, T. Tański

Silesian University of Technology, Akademicka 2A st., Poland

E-mail: przemyslaw.snopinski@polsl.pl

Abstract. The Al-Mg alloys are widely used in the automotive and marine industry due to their excellent properties such as corrosion resistance, good weldability and intermediate mechanical properties along with their low price. Magnesium has a high solubility in the aluminium ~17.35 wt. %. Its addition increases the work-hardening ability, thus an essential work hardening can be expected in those alloys. Refining the grain size of the aluminium-magnesium alloys to the ultra-fine (<1 μm) and even nanometric scale using severe plastic deformation processes become the most effective strategy to increase their mechanical strength, whereas the post plastic deformation treatments are strategies to increase ductility. Experiments were conducted to evaluate the mechanical behaviour of an AlMg5 alloy after ECAP processing and subjected to the post-ECAP annealing treatment. The initial grain size was ~270 μm and was reduced to ~20 μm by ECAP with post-ECAP annealing at 350 °C for 30 min. The annealing treatment led to some sort of bimodal grain mixture with the larger grains embedded in the original UFG structure that provide an excellent combination of strength and ductility.

1 Introduction

The aluminium-magnesium alloys are most widely used due to their excellent corrosion resistance, high ductility, low cost, low density and single phase structure (without precipitates). There are many attempts to improve the mechanical properties of Al-Mg alloys [1-5]. Grain refinement down to nanometric scale becomes the most effective strategy to increase their specific strength. Bulk nanostructured materials have motivated considerable attention due to their unusual mechanical and physical properties, e.g., very high strength in comparison with their coarse-grained alloys. Nanostructured materials are very promising for functional and structural applications, especially where its high strength to density ratio improves Energy efficiency. Such type of materials has also the possibility to significantly enhance the performance of nanomaterial-based devices. However, bulk nanostructured metallic materials have also some weakness - generally exhibit low uniform ductility, which hinders their industry applications [6-8].

Generally, the trade-off between high strength and ductility is a common material science engineering problem. These two very important properties are mutually exclusive. Therefore, since the last decades the engineers choose have been limited to strong or ductile materials, not both as it was required. Several research groups focused their efforts focused on the metallic nanomaterials with increased ductility [9-12]. There are some microstructural approaches that might improve ductility which includes: (i) bimodal grain-size distribution, (ii) post-deformation heat treatment (ageing) – generation of high fraction of nanoscale precipitates, (iii) introduction of large fraction of nanoscale twins, (iv) control of grain boundary character, but the applicability of this approaches

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

depends on the composition of the alloy. Also, the bimodal grain size approach depends on the initial alloy state (severe plastic deformation processing). Despite varying degrees of success, most of the approaches have inherent processing and/or material limitations. For example, nanoscale precipitates can only be formed in precipitation hardenable aluminium alloys. Formation of a bimodal grain structure seems to be a most promising route to obtain simultaneously: excellent mechanical strength and increased ductility [13-16].
Since now, the significant experimental attempts have been devoted to developing bimodal grain structure in metallic non-ferrous alloys that exhibit simultaneously a high strength and increased ductility [7,11,12,15]. The challenge for producing desired microstructure remains, but with recent technological advancements (such as new SPD technologies), it is becoming increasingly more achievable to control and engineer the desired grain structure in metallic materials.
This paper shows the approach to develop a bimodal grain microstructure in a AlMg5 aluminium alloy and focuses on the microstructure-mechanical properties relationship of as fabricated bimodal material, and strengthening mechanisms.

2 Material and research methodology

The investigation has been carried out on commercial aluminium alloy—appointed by the standard EN 1706:2010 – EN AC 53100. The chemical composition of the alloys is given in Table 1.

Table 1. Chemical composition of the EN AC 51300 alloy (AlMg5)

Element	Mg	Si	Fe	Cu	Ti	Al
Mass/%	5.55	0.08	0.07	0.01	0.01	Bal

The work samples were subjected to the heat treatment prior plastic deformation. ECAP process was performed using a hydraulic press at room temperature via route Bc. Samples were pressed through an ECAP die with an abrupt internal angle of 120° up to two passes. The samples were processed through a die with a constant ram speed of 2 mm/s. To obtain a bimodal grain size distribution annealing was applied on the SPD processed samples. Microstructure observations of the AlMg5 alloy samples were carried out using light optical microscope Axio Observer A1 on the cross-section plane of the work specimens.
X-ray diffraction analysis were carried out on PANalytical X'Pert Pro diffraction system with cobalt anode (Kα = 1.789 Å) in order to determine the average dislocation density using a Williamson-Hall method.
The universal tensile testing machine ZWICK Z/100 was used to evaluate tensile properties. Tensile tests were performed at room temperature under a strain rate of $6.7 \times 10^{-4}\ s^{-1}$. The hardness was measured using Automatic Rockwell hardness tester ZWICK ZHR 4150 under a load of 60 kg. To obtain precise results, ten hardness measurements were taken on each sample.

3 Results

3.1 Structure

Optical microscopy images showing the microstructure of solution treated AlMg5 alloy are presented in Fig. 1a-b. As seen, the started material has an equiaxed coarse grain structure with an average size of about 270 μm. As follows from the Fig. 1a after solution heat treatment, most of the Mg was brought into the α-Al solid solution.

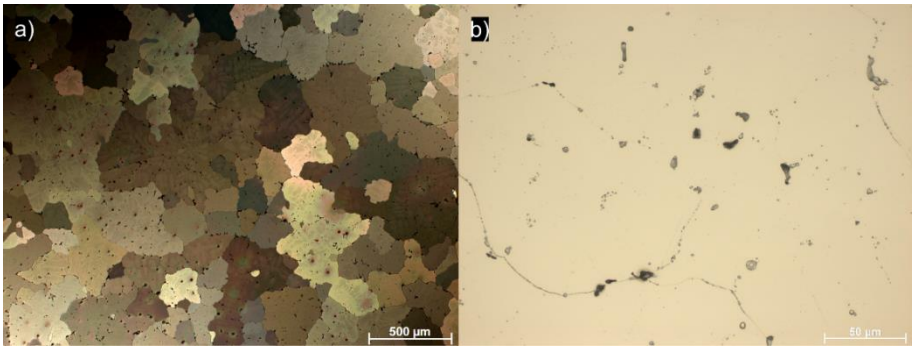


Figure 1. Structure of the EN AC 51300 alloy in the initial state a) polarized light image, b) bright field image

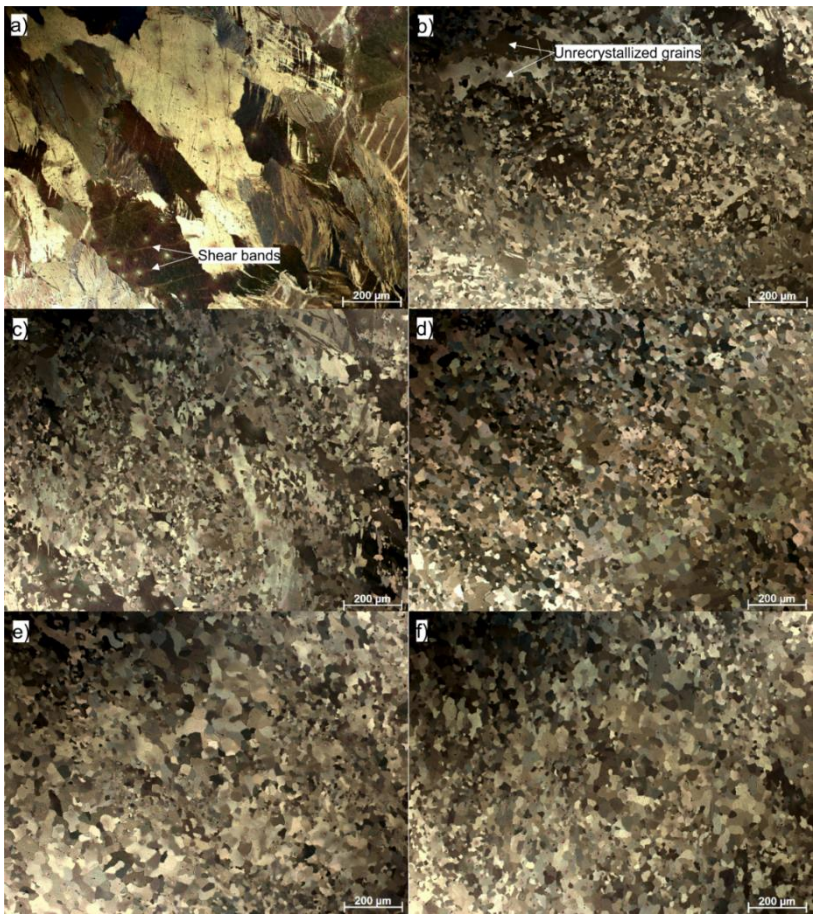


Figure 2. Structure of the EN AC 51300 alloy a) ECAP2x, b) ECAP2x + annealing 30 min, c) ECAP2x + annealing 1h, d) ECAP2x + annealing 2h, e) ECAP2x + annealing 4h, f) ECAP2x + annealing 8h

The ECAP process has changed the original coarse-grained microstructure of the AlMg5 alloy (Fig. 2a). The microstructure of the sample subjected to 2 ECAP passes consist of the elongated grains. The grains become fragmented through the mutual interactions of shear and microshear bands. In the structure of the post-ECAP annealed sample, a large number of fine-grains accompanied by a considerable fraction of micrometre-sized unrecrystallized grains (>400 µm) can be observed. This indicates that a bimodal grain structure was developed (Fig. 2b). After annealing at 350 °C for 1 h, the microstructure is still quite heterogeneous (Fig. 2c), which consists of fine-grained grains with larger grains >200 µm. This bimodal grain structure indicates straightforward that discontinuous

recrystallization occurred locally. Obviously, to facilitate full recrystallization to occur at this annealing temperature (350 ºC), annealing time should be prolonged. It is revealed that when the annealing time increases up to 2h, full recrystallization occurs and the average grain size is about ~23 μm (Fig. 2d). With further post-ECAP annealing, there is a slight increase in grain size to 28 μm ~30 μm after 4 and 8 h (Figs. 2e-f), while the microstructure become homogenous.
To investigate the grain structure and size of as deformed sample TEM was employed. In the Fig. 3 it can be seen that dislocation substructures and elongated sub-grains with a width of about 0.6 μm are formed after ECAP.

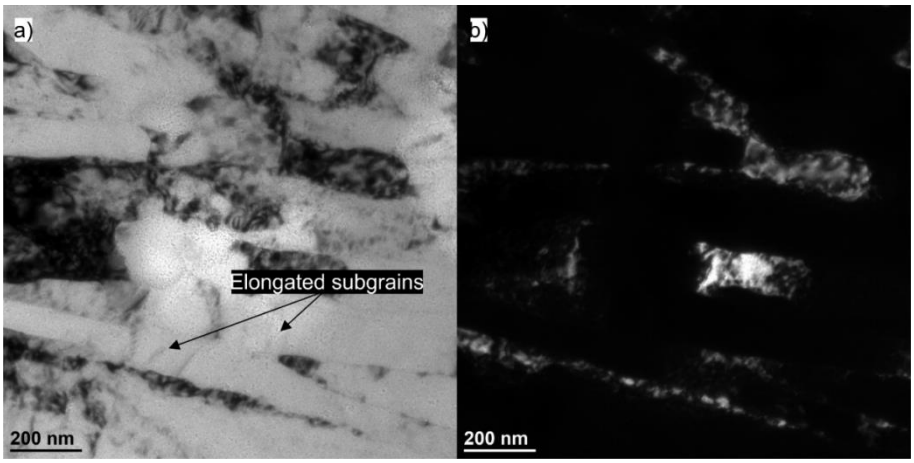


Figure 3. Representative TEM image of solution treated – ECAPed sample (a) bright field image, (b) dark field image

3.2 Mechanical properties

Tensile test curves of the as ECAPed and post-ECAP annealed samples are shown in Fig. 4. Corresponding mechanical properties obtained from Fig. 4 are summarized in Table 2. It is clearly seen that ECAP process leads to increase of mechanical strength (tensile/yield strength and hardness increase) and to significant drop of elongation to failure. Further processing result in a mechanical properties drop; whereas ductility increases significantly. Additionally, flow serrations during tensile deformation of as ECAPed and post-ECAP annealed samples are clearly seen on the tensile test curves. In the as ECAP processed sample A type of flow serrations are observed; whereas in the post-ECAP annealed samples, the combination of A, B and D types of flow serrations takes place.

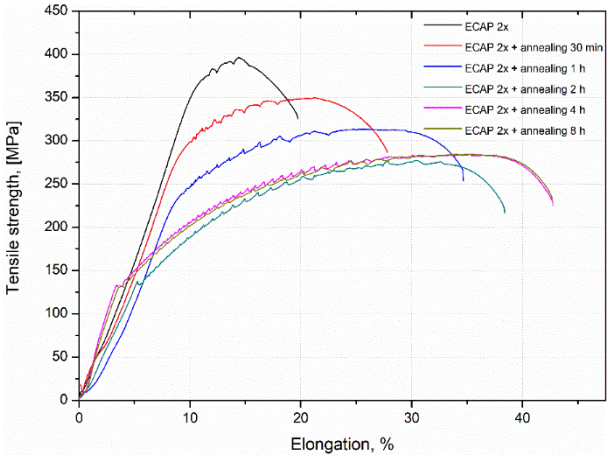


Figure 4. Representative tensile test curves of the AlMg5 alloy subjected to ECAP and post-ECAP annealing

Table 2. Summarized results of the mechanical properties examination

Annealing time, [h]	0	0.5	1	2	4	8
Hardness, HRF	93	78	76	70	72	70
Tensile elongation, %	7	14.7	24	30	33	32
Yield strength, [MPa]	335	275	230	124	124	124
Tensile strength, [MPa]	395	350	312	278	282	283

3.3 Analysis of strengthening mechanism

ECAP processing significatly affects the grain structure and the mechanical properties. The strengthening mechanisms are discussed in this section to investigate the relationship between the obtained microstructure and yield strength. Taking into account that different strengthening mechanisms act independently, thus having additive contributions, the overall YS of the AlMg5 alloy can be expressed as follows:

$$\sigma_{0.2} = \sigma_0 + \sigma_{ss} + \sigma_{gb} + \sigma_d \quad (1)$$

Where $\sigma_{0.2}$ is the yield strength, σ_0 is the flow stress of un-deformed pure Al (35 MPa), σ_{ss} is the solid solution strengthening, σ_{gb} is the grain boundary strengthening – due the grain refinement, σ_d is the dislocation strengthening.

Solid solution strengthening (σ_{ss}) is controlled by the magnesium solute content, which can be expressed as follows:

$$\sigma_{ss} = HC^n \quad (2)$$

where C is the concentration of an element in solid solution and n and H are constants equal to 1.19 and 13.3 Mpa/(wt.%). Substituting C=5.55 wt.% and the values of constants, the value finally estimates σ_{ss} = 102 MPa.

The most important and most visible from the microstructural point of view consequence of ECAP process is the grain refinement. The obtained refined microstructure shows improved YS which increase may be explained by Hall-Petch equation as below:

$$\sigma_{gb} = k_y d^{-0.5} \quad (3)$$

Where: k_y is the Hall-Petch coefficient – 0.13, D is an average grain size obtained by TEM measurement ~ 0.6 μm. The calculated contribution to the Yield strength reaches the value of σ_{gb}=165 MPa.

In the work-hardened materials dislocations mutually interacts and impede their motion. Therefore, increasing the dislocation density, the yield strength increases. The importance of dislocation strengthening may be expressed as follows:

$$\sigma_d = M\alpha Gb\rho^{1/2} \quad (4)$$

Where σ_d is the strength increment from dislocation strengthening, α is a constant 0.24, G is the shear modulus 24 GPa, b is the length of burgers vector of dislocations 0.286 nm for Aluminium, M is a Taylor factor (M=3 for untextured polycrystalline materials) and ρ is a dislocation density calculated using a Williamson-Hall method XRD $\rho = 0.205*10^{14} m^{-2}$. The overall contribution due the stored dislocations reaches value of σ_d=26 MPa.

Based on the calculations presented above the overall YS reaches value 328 MPa, whereas the measured YS value is ~335 MPa.

4. Conclusions

Microstructure and mechanical properties of EN AC 51300 alloy subjected to ECAP and post-ECAP annealing treatments was comparatively investigated. The main findings described in the article can be summarized as follows:

The elongated grains were gradually replaced by the new fine equiaxed grains accompanied by a considerable fraction of micrometre-sized unrecrystallized grains forming a bimodal grain distribution.

The micrometer-sized grains in the bimodal microstructure can effectively increase the ductility of the fine-grained AlMg5 alloy.

The ECAP process increases the aluminuim yield strength and hardness up to 335 MPa. The corresponding ultimate tensile strength was increased from 398 MPa. However, the elongation was decreased to ~7% after 2 ECAP passes. The excellent strength of as ECAP processed AlMg5 alloy is due to the strain and grain size strengthening.

DSA process is observed during tensile deformation. The DSA process manifests itself in form of flow serrations on the tensile test curves

References

[1] Tanski T, Snopinski P and Borek W 2017 Strength and structure of AlMg3 alloy after ECAP and post-ECAP processing *Mater Manuf Processes* **32** 1386
[2] Tanski T, Snopinski P, Prusik K and Sroka M 2017 The effects of room temperature ECAP and subsequent aging on the structure and properties of the Al-3%Mg aluminium alloy *Mater Charact* **133** 185
[3] Krol M, Tanski T, Snopinski P and Tomiczek B 2017 Structure and properties of aluminium-magnesium casting alloys after heat treatment *J Therm Anal Calorim* **127** 299
[4] Snopiński P, Tański T and Sroka M 2017 The effect of heat treatment conditions on the structure evolution and mechanical properties of two binary Al-Mg aluminium alloys *Metalurgija* **56** 329
[5] G.E. Totten, D.S. MacKenzieHandbook of Aluminum: Physical Metallurgy and Processes Marcel Dekker, New York, USA (2003)
[6] Cepeda-Jiménez CM, García-Infanta JM, Pozuelo M, Ruano OA, Carreño F 2009 Impact toughness improvement of high-strength aluminium alloy by intrinsic and extrinsic fracture mechanisms via hot roll bonding *Scripta Mater* **61** 407
[7] Jayaganthan R, Brokmeier H-, Schwebke B, Panigrahi SK 2010 Microstructure and texture evolution in cryorolled al 7075 alloy *J Alloys Compd* **496** 183
[8] Dursun T, Soutis C 2014 Recent developments in advanced aircraft aluminium alloys *Mater Des* **56** 862
[9] Sabirov I, Murashkin MY, Valiev RZ 2013 Nanostructured aluminium alloys produced by severe plastic deformation: New horizons in development *Mater Sci Eng A* **560** 1
[10] Wang YM, Ma E 2004 Three strategies to achieve uniform tensile deformation in a nanostructured metal *Acta Mater* **52** 1699
[11] Zha M, Zhang H-, Yu Z-, Zhang X-, Meng X-, Wang H-, Jiang Q 2018 Bimodal microstructure – A feasible strategy for high-strength and ductile metallic materials *J Mater Sci Technol* **34** 257
[12] Zha M, Li Y, Mathiesen RH, Bjørge R, Roven HJ 2015 Microstructure evolution and mechanical behavior of a binary al-7Mg alloy processed by equal-channel angular pressing *Acta Mater* **84** 42
[13] Fan GJ, Choo H, Liaw PK, Lavernia EJ 2006 Plastic deformation and fracture of ultrafine-grained al-mg alloys with a bimodal grain size distribution *Acta Mater* **54** 1759
[14] Valdes-Tabernero MA, Sancho-Cadenas R, Sabirov I, Murashkin MY, Ovid'ko IA, Galvez F 2017 Effect of SPD processing on mechanical behavior and dynamic strain aging of an al-mg alloy in various deformation modes and wide strain rate range *Mater Sci Eng A* **696** 348
[15] Duan Y, Tang L, Xu G, Deng Y, Yin Z 2016 Microstructure and mechanical properties of 7005 aluminum alloy processed by room temperature ECAP and subsequent annealing *J Alloys Compd* **664** 518
[16] Tański T, Snopiński P, Hilser O 2017 Microstructure and mechanical properties of two binary al-mg alloys deformed using equal channel angular pressing *Mater Wis Werkstofftech* **48** 439