

Microstructural evaluation of an Al-Mg alloy after ECAP and post ECAP processing

P. Snopiński

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

E-mail: przemyslaw.snopinski@polsl.pl

Abstract. This work focuses on the effect of pre-deformation treatment and post-deformation aging on the microstructure, especially the precipitation kinetics of the Al-Mg aluminium alloy. The UFG material was produced by equal channel angular pressing which resulted in dramatic increase of mechanical strength. Important anomalies resulting from the severe plastic deformation appeared in the dilatometric curves, which must be influenced by the recrystallization and the precipitation processes. Furthermore, the acceleration effect of SPD processing on precipitation nucleation was confirmed.

1 Introduction

The Al-Mg alloys are suitable for marine applications due to their excellent properties such as low density, ease fabrication, mechanical strength and most notable resistance to corrosion. 5xxx series aluminium alloys are generally classified as a non-heat treatable. In contrast with other Al alloys, strength is achieved primarily by a solid solution strengthening, work or dispersion hardening. However, the Al-Mg alloys can actually form several precipitates and thus can be classified as heat treatable to some degree [1-4].

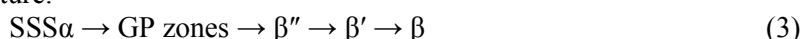
The precipitation behaviour of Al-Mg alloys has been a subject of intensive studies for a few decades. Scientists efforts were focused on the manner in which stable or metastable precipitates nucleate from a supersaturated solid solution. The first proposal of a possible precipitation sequence in Al-Mg alloys was suggested by Kelly et al.[5]:



They suggested that two precipitate phases can form: 1) intermediate β' -phase and 2) the equilibrium β – phase (Al_3Mg_2). Both phases has a similar chemical composition but different crystal structure. Further work presented by Pollard [6] showed that previous precipitation sequence lack of formation of Guinier-Preston zones and thus, he suggested a modified sequence:



He has explained his results in such a way that during ageing $<50^\circ\text{C}$, the formation of GP zones take place, which results in an increase of mechanical strength. Other scientists [7,8] reported the second type of GP zones can form β'' – phase. The culmination of researches discussed above by other scientists in this field [9-11] have yielded the following precipitation sequence that is commonly reported in the literature:



The increase in the aging temperature above 100°C led to a dissolution of GP zones and the β'' precipitates, thus allowing formation the intermediate (metastable and semi-coherent) β' phase with



a hexagonal structure. Such type of precipitates are formed by a nucleation and growth on the structural defects of the matrix [9-13].

Recently, there has been a growing interest in controlling the precipitation microstructures of aluminium alloys and thereby obtaining a desirable combination of strengthening from both grain refinement and precipitation hardening. One of the most promising methods for obtaining an increased strength as well as an increased ductility is a low-temperature ECAP processing and subsequent short heat treatment. Using this combination, strengthening processes such as the formation of very fine particles as well as softening recovery processes occurs simultaneously.

The understanding of the precipitation behaviour at the early stages of artificial ageing of SPD treated Al-Mg alloy appears to be important for clarifying the changes in the mechanical strength during ageing. The purpose of this paper is to investigate the microstructure of Al-Mg alloy subjected to ageing treatment after severe plastic deformation process.

2 Material and research methodology

The investigation has been carried out on commercial aluminium alloy—appointed by the standard EN 1706:2010 – EN AC 51100. The chemical composition of the alloys is given in Table 1.

Table 1. Chemical composition of the EN AC 51100 alloy

Element	Mg	Si	Fe	Cu	Al
Mass/%	2.86	0.07	0.07	0.01	Bal

The experiment described in this article was preceded by a selection of the most beneficial heat treatment conditions. The results were published in the previous articles [14,15]. The second stage of the experiment was to study the influence of the ECAP strain on the precipitation kinetics in an AlMg3 alloy. Cylindrical samples of 20 mm in diameter and 70 mm in length were ECAP processed with a constant ram speed of 20 mm/min using a die with an internal angle (Φ) of 120°. In order to decrease friction between ECAP sample and die molybdenum sulfide (MoS₂) was used. Solution treated samples were then subjected to the ECAP process (4 passes, route Bc - the rotation of the sample was precisely checked between consecutive passes) and finally artificially aged at 160 °C.

The microstructure of the alloy was characterized by light optical microscope Axio Observer Image Analyser. The examinations of thin foils were made on the high-resolution transmission electron microscope JEM 3010UHR from JEOL, at the accelerating voltage of 300 kV. TEM specimens were prepared by cutting thin plates from the material. The specimens were ground down to foils with a maximum thickness of 80 µm before 3 mm diameter discs were punched from the specimens. The disks were further thinned by ion milling method with the Precision Ion Polishing System (PIPS™), used the ion milling device model 691 supplied by Gatan until one or more holes appeared. The ion milling was done with argon ions, accelerated by a voltage of 15 kV.

Static tensile test and microhardness were performed to investigate the precipitation kinetics of the artificially aged samples. Vickers microhardness (HV) was measured on cross-section plain designated as y-plane by imposing a load of 300 g for 15 s. Tensile tests were performed at room temperature using a universal tensile testing machine ZWICK Z/100.

The dilatometric analysis was performed using a TA Instruments Dilatometer under a protective atmosphere of pure argon. Thermal cycles consist of heating until 440 °C (10°C/min), followed by holding of 10 min at this temperature and a cooling with the same rate until room temperature.

3 Results

3.1 Structure

The microstructure of the solution treated sample prior ECAP consists of well-defined grains of different sizes as shown in Fig. 1a. The average grain size measured using the line interception method is $\sim 330 \mu\text{m}$.

As follows from the Fig. 1a during solution treatment, most of the Mg was brought into the α -Al solid solution. The ECAP process has changed the original coarse-grained microstructure of the AlMg3 alloy (Fig. 1b-c). Comparing to the initial microstructure, the ECAP processed samples grains are stretched, some areas of different crystallographic orientation – shear bands forms during ECAP shear deformation. It is evident that these shear bands nucleate on a grain boundary, inducing large offsets on it. Artificial ageing has a minor influence on the microstructure - no visible evidence of new grain nuclei (Fig. 1c).

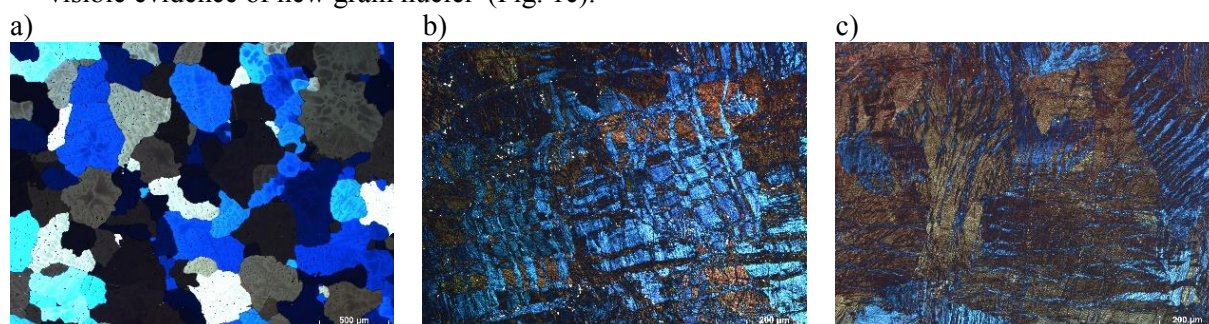


Figure 1. Structure of the EN AC 51100 alloy a) solution treated state, b) subjected to 4 ECAP passes, c) subjected to 4 ECAP passes and aged for 30 min

It is known from a nomenclature that the hexagonal β' -phase precipitates at 150 and 200 °C, while the direct formation of the equilibrium β -phase occurs at 250 and 300 °C. In our previous work, we found that β' -precipitates are responsible for precipitation strengthening of a solution treated Al-Mg alloy [9]. However, this investigation shows also that two types of precipitates exist in the microstructure of artificially aged sample (Figs. 2-3). The first one is the hexagonal (hp24) β' -precipitate (Fig. 2) responsible for the strengthening effect, while the second one is the β equilibrium precipitate with a spherical shape (Fig. 3). The presence of the equilibrium β -phase can be explained by dissolution of β' -precipitates during artificial ageing or independent appearance of β -phase precipitates, near existing strengthening precipitates.

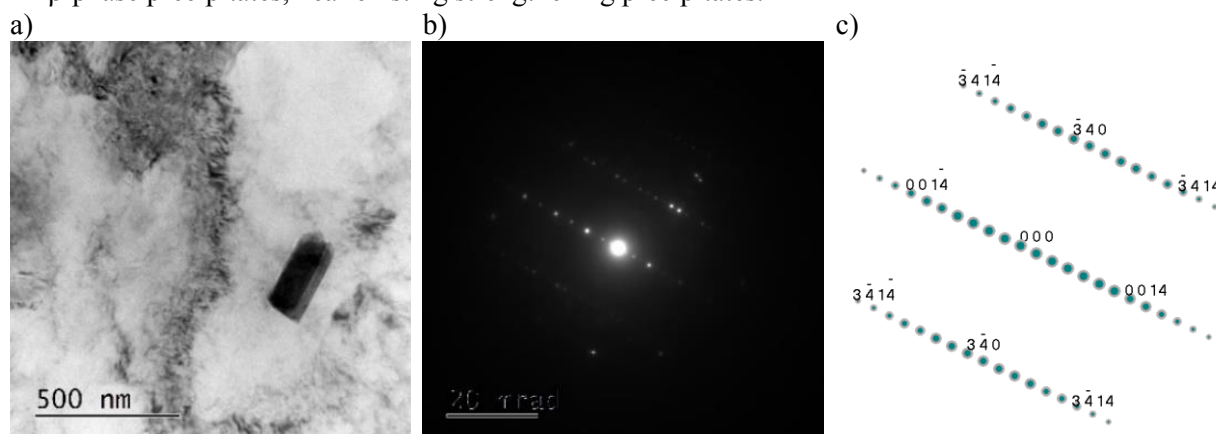


Figure 2. Representative TEM image of solution treated – ECAPed and aged sample showing β' - Al_3Mg_2 hexagonal precipitate, (b) diffraction pattern, (c) solved diffraction pattern

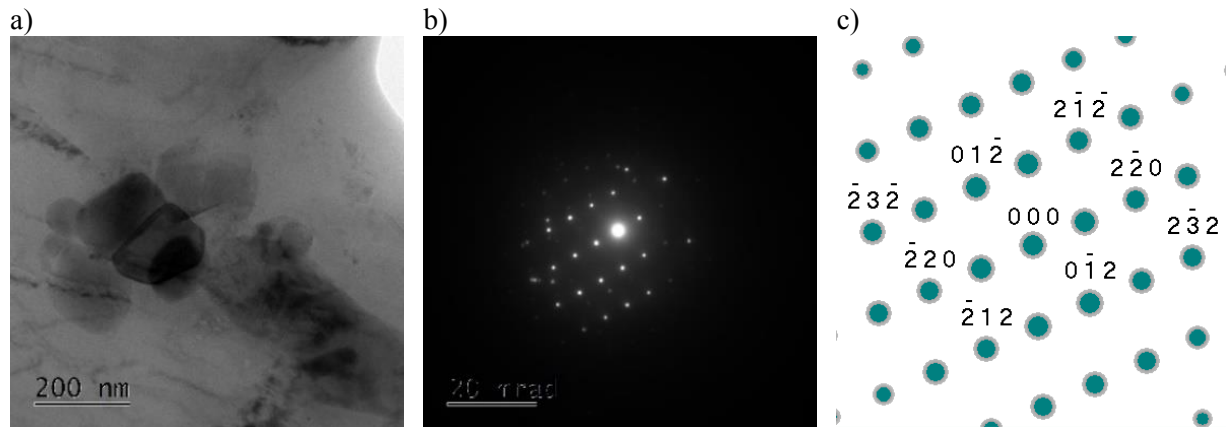


Figure 3. Representative TEM image of solution treated – ECAPed and aged sample showing β - Al_3Mg_2 fcc equilibrium precipitates, (b) diffraction pattern, (c) solved diffraction pattern

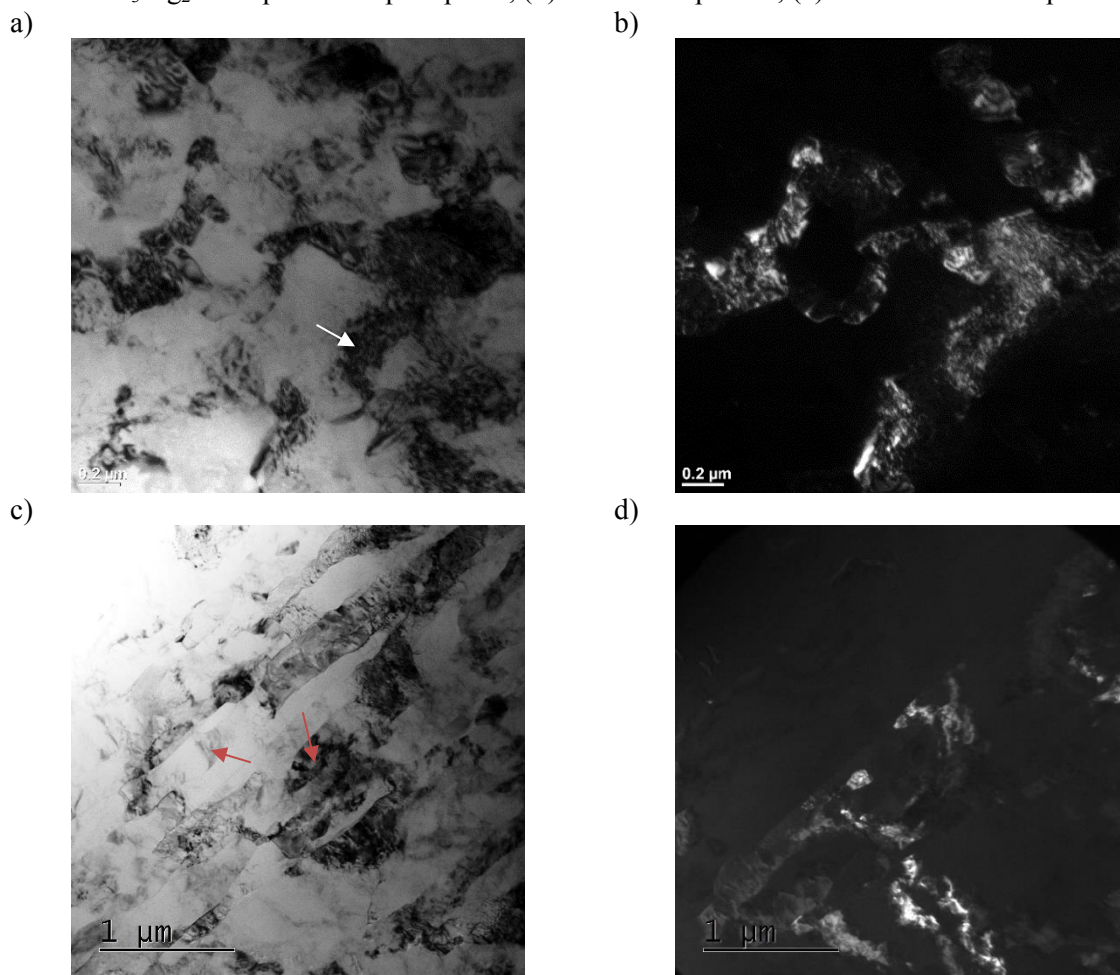


Figure 4. Representative bright and dark field TEM images a) and b) solution treated and ECAPed sample, c) and d) solution treated ECAPed and aged at 160 °C for 2h sample

TEM bright and dark field micrographs of the as-ECAP processed sample are shown in Figs. 4a-b. After four ECAP passes dislocation structure was formed (white arrow). Some equiaxial subgrains are also visible in the substructure. Artificial aging led to visible changes in the microstructure. Parallel bands with well-defined subgrains (red arrows) are also visible. There is also a noticeable dislocation density decrease (as a result of recovery and recrystallization processes).

3.2 Dilatometric measurement

Figure 5a-b shows, respectively, the variation of the ratio $\Delta L/L_0$ with a derivative of heating curve as a function of temperature. Some anomalies composed of a weak change in expansion in temperature ranges 150-190 °C and 330-350 °C are visible in (Fig. 5b), while the shape of the dilatometric curve of solution treated sample looks quite smooth (Fig. 5a). This first anomaly can be a result of the β' -phase precipitation. The precipitation process seems to be accelerated by the pre-deformation using ECAP procedure. The observation of the dilatometric curve of the ECAP processed sample reveals only some anomalies in the temperature range 390-400 °C, which can be a result of the recrystallization processes that taking place in a deformed sample.

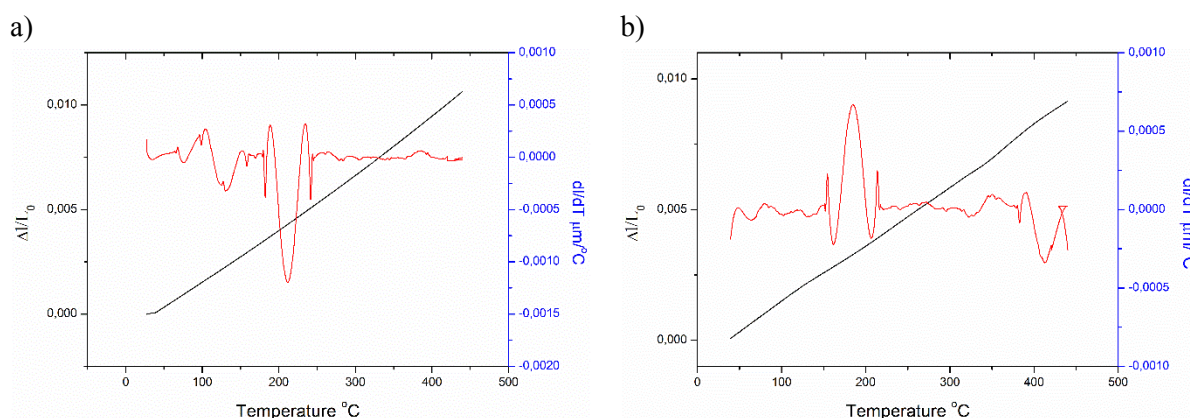


Figure 5. Dilatometric curves of an Al-Mg alloy a) solution treated and aged for 2h at 160 °C, b) solution treated and ECAPed,

3.3 Mechanical properties

Table 2 summarizes the results of the mechanical properties examinations. It is clearly visible that ECAP processing has an influence on the kinetics of precipitation process. The hardness and tensile strength increase slightly in the early stages of the post-ECAP ageing process, while further ageing led to mechanical properties decrease. The observed phenomenon – increase in the mechanical properties is due the precipitation of the hexagonal β' -phase. With an increase in ageing time the amount of equilibrium precipitates increases, thus the mechanical properties decrease. This is in good agreement with the TEM study in the previous paragraph where the presence of two type of precipitates in the sample after 2 hours of artificial ageing was observed.

Table 2. Summarized results of the mechanical properties examination

Aging time, [h]	0	0.5	1	2	4
Hardness, HV	112.3	115	112.6	111.1	111.2
Tensile strength, [MPa]	352	360	354	347	348

4. Conclusions

The whole results presented in this work reflect in particular the effect of the ageing time on the precipitation in Al-3%Mg alloy.

In this article, we found that during an artificial ageing process of the pre-deformed Al-Mg alloy two types of precipitates exists in the microstructure. The first one responsible for precipitation strengthening, that is semicoherent with the Al matrix β' -phase and the second equilibrium β phase precipitates.

The results of the mechanical properties tests confirmed a beneficial influence of the pre-deformation treatment on the precipitation kinetics of AlMg3 alloy. We found that after 30 min of artificial ageing the mechanical properties slightly increases, while further ageing has a negligible influence on the mechanical strength. The thermal expansion coefficients changes versus temperature showed the anomalies, that can be related to the formation of the phases during the artificial ageing process.

References

- [1] Bensaada S, Bouziane M T, Mohammedi F 2011 Effect of the temperature on the mechanism of the precipitation in Al-8% mass.Mg alloy, *Mater Lett.* **65** 2829-2832,
- [2] Nebti S, Hamana D and Cizeron G 1995 Calorimetric study of pre-precipitation and precipitation in Al-Mg alloy *Acta metall. Mater.* **43** 3583
- [3] Hamana D, Boucheur M and Derafa A 1998 Effect of plastic deformation on the formation and dissolution of transition phases in Al-12 wt.% Mg alloy *Mater Chem Phys* **57** p 99
- [4] 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
- [5] Kelly A, Nicholson RB, Thomas G. 1936 *Electron Microscopy and Strength of Crystals*: John Wiley & Sons, Inc.,
- [6] Pollard W 1965 *Journal of the Institute of Metals* **93** 339
- [7] Starink M J and Zahra A M 1997 Low temperature of Al-Mg alloys *Phil. Mag.* **76** 701
- [8] Starink M J and Zahra A M 1996 Precipitation kinetics of an Al-15%Mg alloys *Mater Sci Forum* **217** 795
- [9] 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
- [10] Hamana D, Baziz L and Boucheur M 2004 Kinetics and mechanism of formation of transformation of metastable β' -phase in Al-Mg alloys *Mater Chem Phys* **84** 112
- [11] Jie J C, Wang H W, Zou C M, Wei Z J and Li T J 2014 Precipitation in Al-Mg solid solution prepared by solidification under high pressure *Mater Charact* **87** 19
- [12] Slabanja M and Wahnstrom G 2005 Kinetic Monte Carlo study of Al-Mg precipitation *Acta Mater* **53** 3721
- [13] Yukawa H, Murata Y, Morinaga M, Takahashi Y and Yoshida H 1995 Heterogeneous distributions of magnesium atoms near the precipitate in Al-Mg based alloys *Acta metall. Mater.* **43** 681
- [14] 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
- [15] 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