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# Influence of processing routes on structure and residual stress in aluminium-magnesium alloy after ECAP

**T. Tański [a], P. Snopiński [a,*], P. Nuckowski [a], T. Jung [a], W. Kwaśny [a], T. Linek [b]**

[a] Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

[b] ENVO sp. z o.o., ul. Radomska 76, 27-200 Starachowice, Poland

* Corresponding e-mail address: przemyslaw.snopinski@polsl.pl

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## ABSTRACT

**Purpose:** The main goal of present study is try to find the influence of severe plastic deformation using the ECAP (equal channel angular pressing) process and different processing routes on structure formation, properties and lattice strain of EN AC 51100 alloy.

**Design/methodology/approach:** The cold ECAP behaviour was determined using three different processing routes A, $B_c$ and C and four repetitive pressings. The comparison between three types of processing routes has been established based on microstructure observation, grain size measurements and X-ray diffraction analysis.

**Findings:** It was found that the structure after three types of mechanical treatments is strongly deformed, and the processing route has a significant impact on shape and grain size. It was also found that direction of shearing patterns that appears after deformation have influence on residual stress in ECAPed material. The use of equal channel angular pressing method has also an influence on hardness of investigated alloy which increase more than 90% after four repetitive pressings.

**Research limitations/implications:** Current study presents the investigation results which was carried out on samples, not on final products.

**Practical implications:** Current research is moving towards to develop high strength materials with increased mechanical properties and fine microstructure which are known as ultra-fine-grained materials, compared to well-known common materials.

**Originality/value:** This paper presents the results of structure and properties investigation including X-ray analysis of severely deformed AlMg3 alloy by ECAP process.

**Keywords:** Aluminium alloys; SPD; ECAP; Structure analysis; Stress measurement; X-ray analysis

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## MATERIALS

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## 1. Introduction

In accordance to the current market demand for lightweight and reliable structures an important role plays the aluminium alloys, belongs to the group of structure materials characterized by series of high properties such as good strength and corrosion resistant. Aluminium alloys that the biggest advantage is high strength to density ratio are increasingly used where the main requirement is mass reduction. Over the last decade, their use in the automotive industry increased several times [1]. However, despite their good mechanical properties these alloys still are not equal to materials such as steel whose usage is very high. Thus, it becomes necessary to develop new treatments or new alloys with improved mechanical properties. Over the years, scientist have devoted a lot of work in order to optimize the heat or mechanical treatment to increase properties of large group light metals [2-6, 9-11]. However, the well known processes of mechanical and heat treatment often result in a ductility decrease of the material which in many applications is undesirable, and sometimes unacceptable. These restrictions result in an increased interest in methods of severe plastic deformation due to the potential to achieve large grain refinement from micrometre to nanometre range. There are many methods of SPD [7-8] but the most promising method, focusing attention of scientist over the past decade has become the equal channel angular pressing. In this method a sample with circular, square or rectangular cross section is exposed to an intensive plastic deformation by repeatedly pressing through a die consisting two channels. The sample is deformed by the action of shear forces in the area of the bend of the channel. The principle of ECAP proses is shown in Fig. 1.

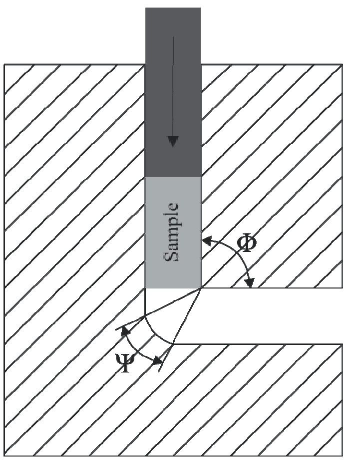


Fig. 1. Principle of equal channel angular pressing process [13]

The die consists two channels whose curvature is described by two angles defining the inner and outer curvature. The sample is made to fill tightly the channel where pressing is proceeded. The advantage of the process is the ability to control the structure produced by changing the orientation of the sample between consecutive passes. The nomenclature indicates four different processing routes of deformation respectively called A, $B_a$, $B_c$ and C. By using different processing routes of deformation it is possible to control the angle at which intersect the shear bands and thus the structure and texture of the material should be different for particular deformation scheme [12-19].

## 2. Materials and experimental procedure

The investigation have been carried out on casting aluminium alloy in initial state ENAC-AlMg3 – 51100. Because of it's very good properties such as good corrosion resistant which is caused by increased Mg content found an wide range of applications. The chemical composition of investigated material is shown in Table 1.

Table 1.
Chemical composition of ENAC AlMg3 alloy, mass fraction

| Aluminium designation | EN AC AlMg3 | |
|---|---|---|
| Chemical composition, Mass concentration, % | Mg | 2.86 |
| | Fe | 0.07 |
| | Si | 0.07 |
| | Ti | 0.01 |
| | Cu | 0.01 |
| | Al | rest |

Equal channel angular pressing was carried out using a die with an internal angle (Φ) of 120°. Rods with diameter of 20 mm and length of 80 mm were pre-machined from as cast material. First stage of experiment was to determine maximum number of passes that material could be subjected, and it was observed that after 5$^{th}$ pass first cracks appears, thus only four repetitive pressing were carried out on each samples according to the processing routes A, $B_c$ and C which is shown in Fig. 2. Samples were pressed through a die at room temperature. As a lubricant was used molybdenum sulfide ($MoS_2$).

Metallographic specimen has been taken from a location in the middle of the work-sample. They were grounded using 600 and 1200 SiC papers and polished with 6 μm, 3 μm and 1 μm diamond paste. In order to determine grain contrast as polished samples were anodized using Barker's reagent (5 ml $HBF_4$, 200 ml $H_2O$) at the voltage of 20 V for 60 s. Structure of the alloy before and after ECAP process was observed on Axio observer light microscope under polarized light.

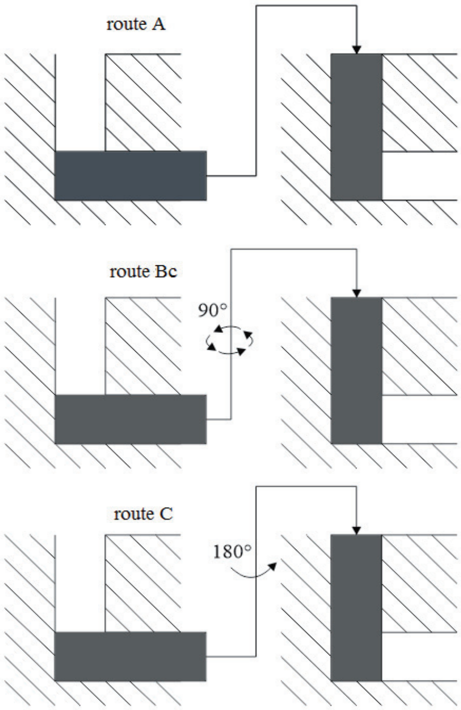


Fig. 2. Schematic illustration of processing routes used in current study [13]

X-ray diffraction analysis were carried out on PANalytical X'Pert Pro diffraction system with cobalt anode in order to determine the average lattice strain. Stress measurements were performed on longitudinal axis of samples using $\sin^2\Psi$ technique basing on the Stress X'Pert Plus software. XRD measurements were also conducted on well-polished samples.

Hardness measurement was carried out using Rockwell method with the load of 60 kg.

## 3. Results and discussion

### 3.1. Structure

According to the Al-Mg binary diagram, investigated alloy should consist consist of two phases, α-Al which is a matrix and second phase β-$Al_3Mg_2$. However the chemical composition which is given in Table 1 suggest that there is possibility of forming phases that contain Si or Cu. Table 2 presents the quantitative analysis of chemical composition performed on Scanning Electron Microscope using EDS. It has been proven the presence of $Mg_2Si$ phase however it was very hard to find phases that contain Cu. Figure 3 shows the structure image of AlMg3 alloy performed on SEM microscope with marked points that were the EDS analysis results obtained.

Table 2.
Results of quantitative analysis of chemical composition

| Point | Element | Wt% | At% |
|---|---|---|---|
| 1 | MgK | 17.52 | 19.25 |
| | AlK | 58.85 | 58.27 |
| | SiK | 23.64 | 22.49 |
| 2 | MgK | 09.44 | 10.37 |
| | AlK | 90.59 | 89.63 |
| 3 | MgK | 03.86 | 04.26 |
| | AlK | 96.14 | 95.74 |

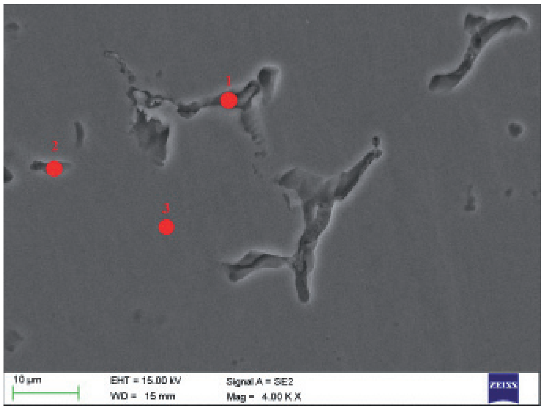


Fig. 3. Structure image of AlMg3 alloy performed on scanning electron microscope

In order to determine grain size and their distribution standard metallography was also realized. Table 3. presents results of grain size measurement which was performed using line intersection method. An initial state alloy has an average grain size about 330 µm. After ECAP process as it was expected grain size is decreasing. Highest grain refinement was achieved for route $B_c$ which allows obtaining highly deformed structures as it was described by Segal et al [14]. Figure 4 presents histogram of grain size distribution measurements. As it could be seen most grains have an average size in range of 100 to 500 µm. Comparison with the structure image that is presented in Fig. 5 allows judging that alloy structure is bimodal, which means that few large grains are surrounded by smaller grains. The histogram analysis confirms that conclusion because there are visible some large grains with a size from 800 to 1200 µm and many grains that has smaller diameter. Similar dependence is observable after ECAP process. Figure 6 shows grain size distribution of AlMg3 alloy processed using route A. There is still small amount of large grains that are surrounded by smaller refined grains and as it could be observed that proportion between large and small grains is changing. The volume share of smaller grains increase which is observable for all samples that is shown in Fig. 7.

Table 3.
Results of grain size measurements

| Condition | Average grain size |
|---|---|
| As cast | ~330 µm |
| ECAPed (Route A) | ~255 µm |
| ECAPed (Route $B_c$) | ~231 µm |
| ECAPed (Route C) | ~271 µm |

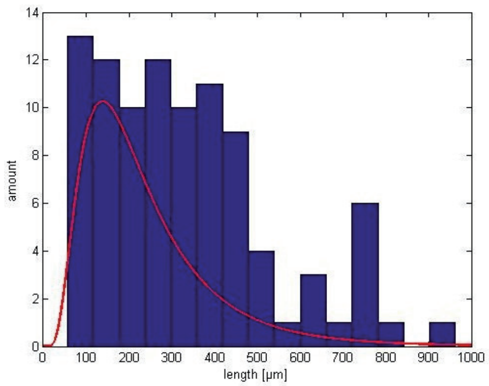


Fig. 4. Histogram of grain size distribution of AlMg3 alloy in initial state

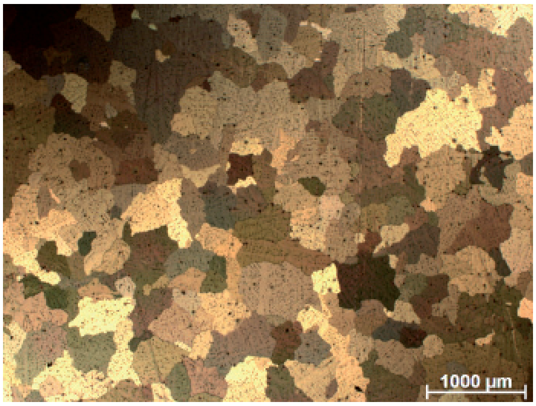


Fig. (5). Polarized light image of AlMg3 alloy in initial state (magnification 25x)

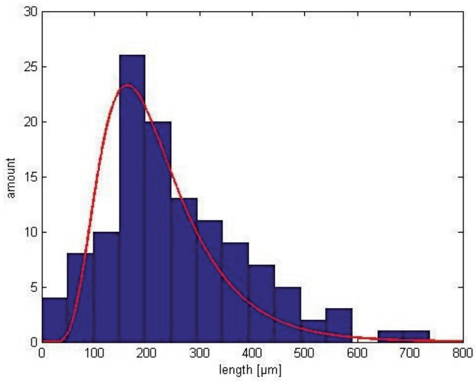


Fig. 6. Histogram of grain size distribution of AlMg3 alloy processed using route A

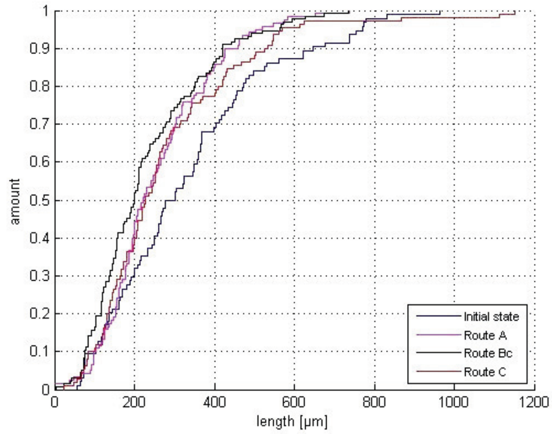


Fig. 7. Empirical grain size distribution function for AlMg3 alloy in initial state and after ECAP process

Figure 8 presents the longitudinal section image of AlMg3 alloy pressed without rotation between consecutives passes. As it could be observed grains are elongated in one direction but they are still relatively large. There is also visible that individual grains are inclined at angle of 30° to pressing direction which correspond to the die channel angle 120°. Figure 9 presents polarized light image of the same alloy processed using route $B_c$. Individual grains are strongly deformed but it could be also observed that are smaller in comparison to as cast state. The shear bands after this route are diversified oriented in individual grains. The last longitudinal section image shown in Fig. 10 is after ECAP process using route C. Even in that case there is only small change in grain size is observable and the microstructure looks at least deformed comparing to the others processing routes. Grains are quite good visible and inclined by shearing bands.

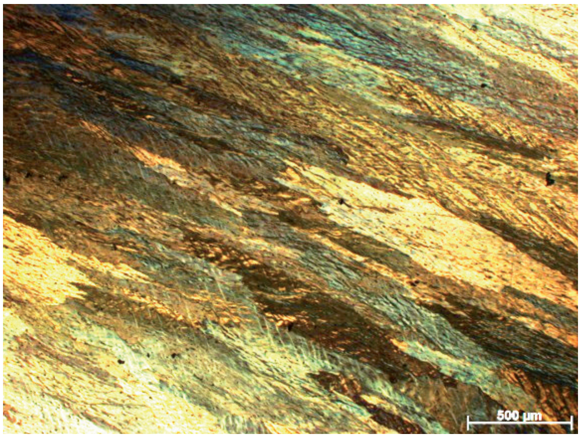


Fig. 8. Longitudinal section image of AlMg3 alloy after 4 passes, route A (magnification 50x, polarized light)

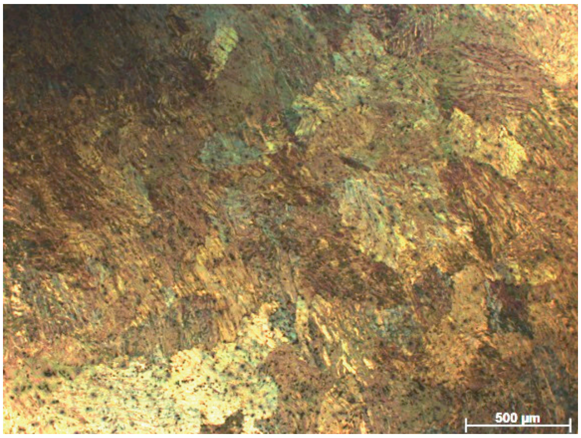


Fig. 9. Longitudinal section image of AlMg3 alloy after 4 passes, route $B_c$ (magnification 50x, polarized light)

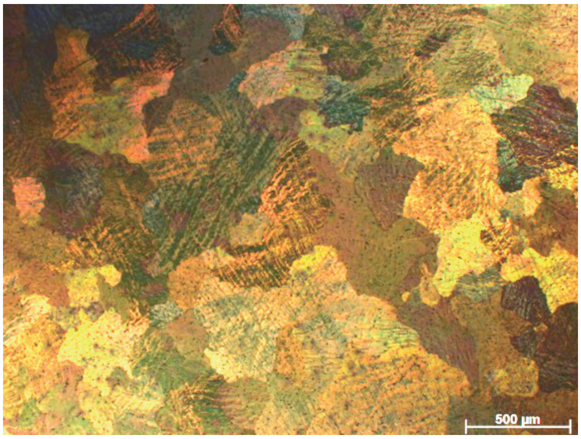


Fig. 10. Longitudinal section image of AlMg3 alloy after 4 passes, route C (magnification 50x, polarized light)

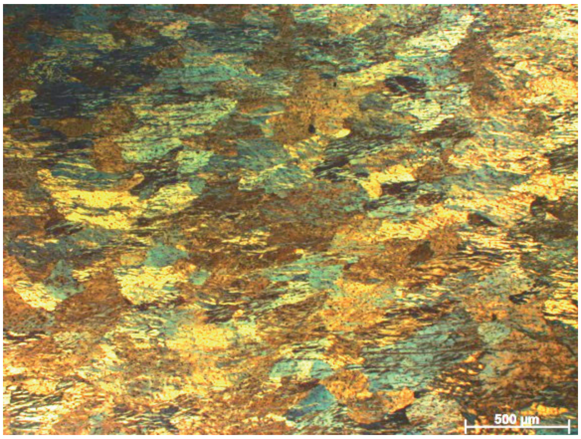


Fig. 11. Cross-section image of AlMg3 alloy after 4 pressing, route A (magnification 50x, polarized light)

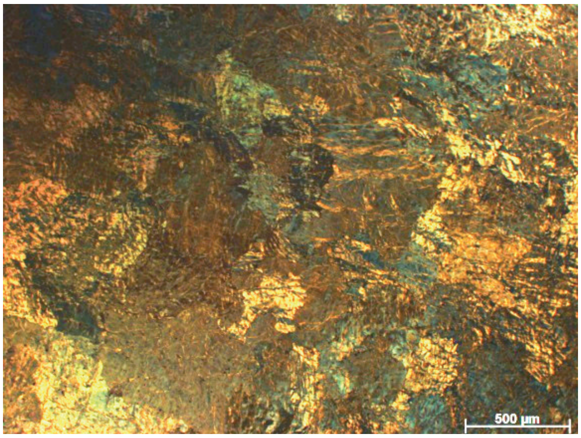


Fig. 12. Cross-section image of AlMg3 alloy after 4 pressing, route $B_c$ (magnification 50x, polarized light)

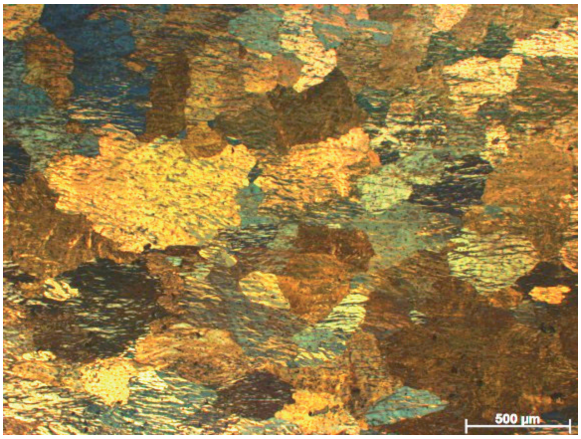


Fig. 13 Cross-section image of AlMg3 alloy after 4 pressing, route C (magnification 50x, polarized light)

The cross-section images of alloy processed by routes A, $B_c$ and C are correspondingly shown in Figs. 11, 12 and 13. It could be observed that the lowest grain size has an alloy after 4 passes using route A and $B_c$. Highest grain size and lower deformation can be observed for alloy that was processed using route C which correspond to 180° rotation between each pass.

## 3.2. XRD stress measurements

Figure 14 shows diffraction patterns of investigated alloy processed using three different processing routes. Using X-ray phase analysis it was found the presence of primary Al (α-Al) phase which is an alloy matrix as confirmed by metallographic study. X-ray phase analysis did not confirmed the presence of another phases like $\beta$-$Al_3Mg_2$ or $Mg_2Si$ which may indicate that, mass concentration of these precipitations is under detection limit of this method.

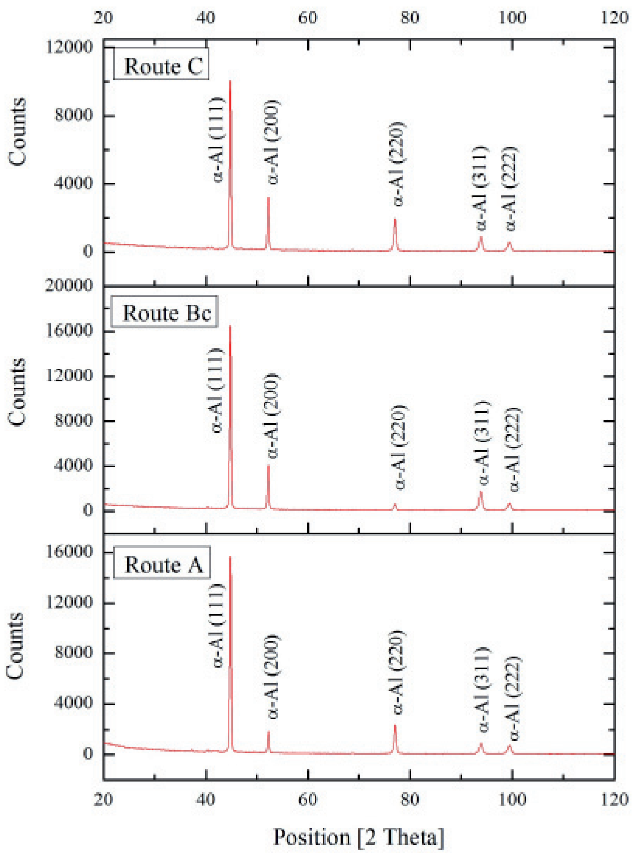


Fig. 14. Diffractograms of AlMg3 alloy after Equal Channel Angular Pressing processed with different processing routes

Figures 15 to 17 presents results of stress measurements and linear dependence of d (311) upon $\sin^2\Psi$ corresponding to the pressing direction measurements. Summarized results of residual stress examination are presented in Table 4. Measurements indicates that in two cases of deformation (route A and $B_c$) there are only tension stresses presented. Highest value of residual stress measurement is observed for the processing route A which may be related to evolution of grain shape during deformation process. Individual grains are arranged in the same direction which probably increase stress that appears in the lattice. In case of deformation route $B_c$ sample rotation between passes causes different arrangement of shear bands which reduces stress. Plastic deformation using route C result in occurrence also compressive stresses. This phenomena could be related to configuration of shearing patterns between consecutive pressings which arrange in only one direction using route C and that results in occurrence of compressive strains.

Table 4.
Statement of the stress in analysed AlMg3 alloy

| Processing rote | Stress in pressing direction, MPa | Stress in a cross direction to the pressing direction, MPa |
|---|---|---|
| A | 56.4 ±5.4 | 67.2 ±7.5 |
| $B_c$ | 16.6 ±4.0 | 46.8 ±3.2 |
| C | -24.7 ±5.0 | 39.9 ±4.1 |

## 3.3. Hardness

Results of hardness measurements are shown in Table 5. As it could be seen there is large increase in hardness for all ECAP processing routes about 95% in comparison to initial state material. The difference between hardness value of processed samples is indiscernible, which leads to conclusion that each processing route allows introducing similar amount of plastic strain into pressed material and the structure formation or internal stresses have negligible influence on hardness.

Table 5.
Results of hardness measurements of initial state AlMg3 alloy and after ECAP process using different processing routes

| Condition | Hardness, HRF |
|---|---|
| Initial state | 47.1 |
| ECAPed route A | 92.7 |
| ECAPed route $B_c$ | 93.3 |
| ECAPed route C | 92.5 |

a) b)

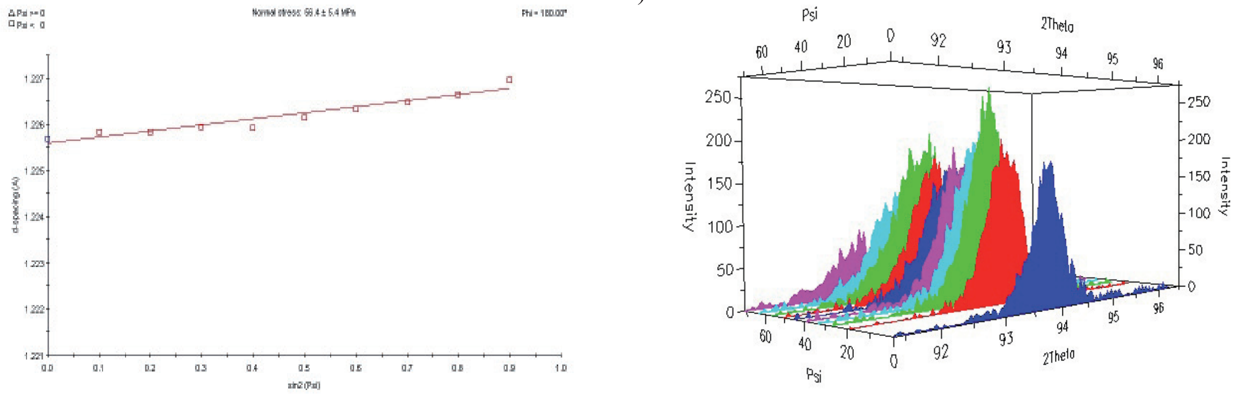


Fig. 15. Results of residual stress measurement: a) linear dependence of d (311) upon $\sin^2\Psi$, b) Changes in the diffraction line position of (311) (Route A)

a) b)

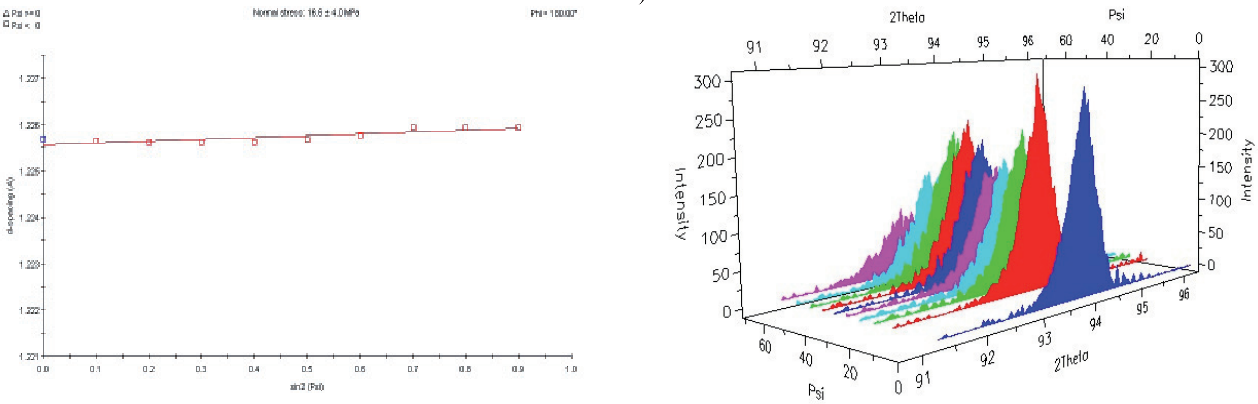


Fig. 16. Results of residual stress measurement: a) linear dependence of d (311) upon $\sin^2\Psi$, b) Changes in the diffraction line position of (311) (Route $B_c$)

a) b)

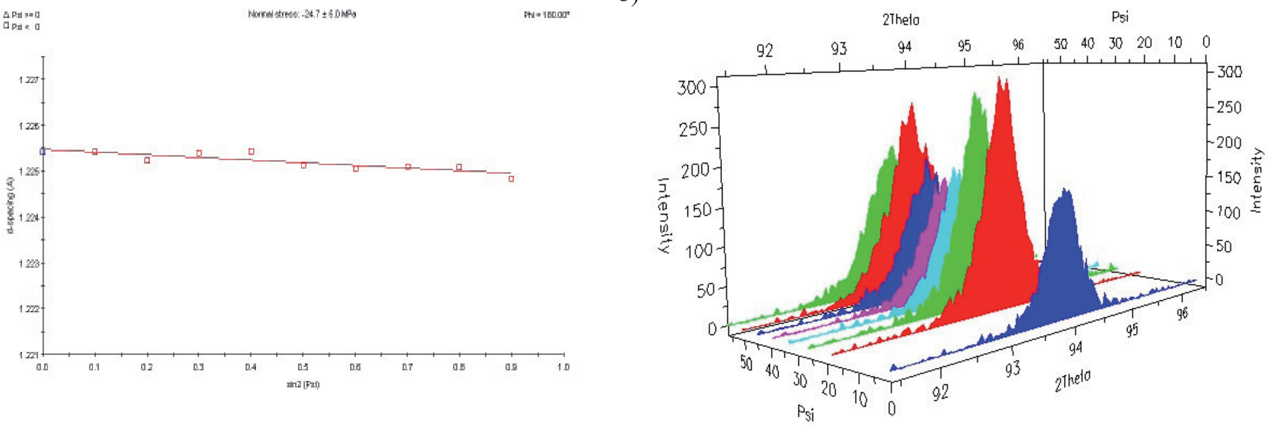


Fig. 17. Results of residual stress measurement: a) linear dependence of d (311) upon $\sin^2\Psi$, b) Changes in the diffraction line position of (311) (Route C)

## 4. Conclusions

Equal channel angular pressing was applied to obtain grain refinement of AlMg3 casting alloy. It was found that this severe plastic deformation process have an influence on structure formation and that there is possibility to obtaining significant grain refinement in aluminium casting alloys.

XRD measurements confirms the presence of tension residual stresses in two cases but there is also found that route C leads to compressive residual stresses which could be related with direction of shearing patterns in material.

Significant increase in hardness of material was also obtained in comparison to initial state and it was also confirmed that there is no relationship between processing route, structure formation and hardness of the material because all three processing rotes allows introducing similar amount of plastic strain into the material.

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## References

[1] T. Tański, L.A. Dobrzański, R. Maniara, Microstructures of Mg-Al-Zn and Al-Si-Cu cast alloys, Journal of Achievements in Materials and Manufacturing Engineering 38/1 (2010) 64-71.
[2] L.A. Dobrzański, T. Tański, S. Malara, M. Król, Structure and properties investigation of an magnesium alloy processed by heat treatment and laser surface treatment, Materials Science Forum 674 (2011) 11-18.
[3] L.A. Dobrzański, T. Tański, J. Trzaska, Optimalization of heat treatment conditions of magnesium cast alloys, Materials Science Forum 638 (2010) 1488-1493.
[4] T. Tański, K. Labisz, L.A. Dobrzański, Effect of Al additions and heat treatment on corrosion properties of Mg-Al based alloys, Journal of Achievements in Materials and Manufacturing Engineering 44/1 (2011) 64-72.
[5] T. Tański, K. Labisz, Surface treatment influence on properties of the heat treated light cast alloys, Proceedings of the 2nd International Conference on Recent Trends in Structural Materials COMAT, 2012, 1-6.
[6] L.A. Dobrzański, T Tański, L Čížek, Heat treatment impact on the structure of die-cast magnesium alloys, Journal of Achievements in Materials and Manufacturing Engineering 20/1-2 (2007) 431-434.
[7] W. Kwaśny, P. Nuckowski, Z. Rdzawski, W. Głuchowski, Influence of RCS process on the structure and mechanical properties of CuSn6 alloy, Archives of Materials Science and Engineering 62/2 (2013) 60-66.
[8] W. Kwaśny, P. Nuckowski, T. Jung, Z. Rdzawski, W. Głuchowski, Effect of plastic deformation on the structure and texture of CuSN6 alloy, Technical Transactions 5 (2013) 213-219.
[9] T. Tański, L.A. Dobrzański, L. Čížek, Influence of heat treatment on structure and properties of the cast magnesium alloys, Journal of Advanced Materials Research 15-17 (2007) 491-496.
[10] T. Tański, K. Labisz, K. Lukaszkowicz, Structure and properties of diamond-like carbon coatings deposited on non-ferrous alloys substrate, Solid State Phenomena 199 (2013) 170-175.
[11] L.A. Dobrzański, W. Borek, Hot-rolling of advanced high-manganese C-Mn-Si-Al steels, Materials Science Forum 706/709 (2012) 2053-2058.
[12] R.Z. Valiev, R.K Islamgaliev, I.V Alexandrov, Bulk nanostructured materials from severe plastic deformation, Progress in Materials Science 45/2 (2000) 103-189.
[13] R.Z. Valiev, T.G. Langdon, Principles of equal-channel angular pressing as a processing tool for grain refinement, Progress in Materials Science 51/7 (2006) 881-981.
[14] V.M. Segal, Engineering and commercialization of equal channel angular extrusion (ECAE), Materials Science and Engineering A 386/1-2 (2004) 269-276.
[15] C.J. Luis Pérez, D. Salcedo Pérez, I. Puertas Arbizu, Design and mechanical property analysis of ultrafine grained gears from AA5083 previously processed by equal channel angular pressing and isothermal forging, Materials and Design (in print).
[16] K.V. Ivanov, E.V. Naydenkin, Tensile behavior and deformation mechanisms of ultrafine-grained aluminum processed using equal-channel angular pressing, Materials Science and Engineering A (in print).
[17] M.H. Shaeri, M.T. Salehi, S.H. Seyyedein, M.R. Abutalebi, J.K. Park, Characterization of microstructure and deformation texture during equal channel angular pressing of Al-Zn-Mg-Cu alloy, Journal of Alloys and Compounds 576 (2013) 350-357.
[18] M. Cabibbo, Microstructure strengthening mechanisms in different equal channel angular pressed aluminum alloys, Materials Science and Engineering A 560/10 (2013) 413-432.
[19] Y.J. Chen, Y.C. Chai, H.J. Roven, S.S. Gireesh, Y.D. Yu, J. Hjelen, Microstructure and mechanical properties of Al-xMg alloys processed by room temperature ECAP, Materials Science and Engineering A545 (2012) 139-147.