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Effect of equal channel angular pressing combined with heat treatment on structure and properties of AlMg3 aluminium alloy

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ABSTRACT

Purpose: The main goal of present study is to investigate the connection of the effect of the heat treatment with severe plastic deformation using the ECAP (equal channel angular pressing) process and by application of precipitation treatment.

Design/methodology/approach: Precipitation treatment was used to increase workability and mechanical properties of the investigated alloy. Two periods of solution treatment time were applied. An aging behaviour of solution treated was studied The cold ECAP behaviour was determined using processing route Bc and the material was subjected to ECAP process up to six pressings.

Findings: It was found that precipitation treatment increases the mechanical properties and workability of EN AC 51100 aluminium alloy. It was also found that the structure during severe plastic deformation is refined by interactions of shearing and slip bands. The application of ECAP method increases also mechanical properties of the tested alloy.

Research limitations/implications: Current study presents the investigation results that 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 with micrometer size of structure materials.

Originality/value: This paper present the results of the structure investigation of AlMg3 alloy in initial state and after precipitation treatment using light and electron microscopy and includes the study of changes in mechanical properties in the tested alloy subjected to different types of treatments.

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ANALYSIS AND MODELING

1. Introduction

It is well known that there is relationship between grain size and properties of the materials. The grain refinement causes a significant increase in strength in bulk metallic materials due to the Hall–Petch relationship which connects the mechanical properties and microstructural features such as the grain size. This relationship illustrates the potential for increasing the mechanical strength by decreasing the size of grains and subgrains and this has been main driving force in the expansion of ultrafine grained (UFG) and nanostructured (NS) materials. From the practical point of view, materials with refine structure are most readily manufactured in bulk form by subjecting samples to severe plastic deformation (SPD) processes. Over the last few decades, severe plastic deformation techniques have been extensively utilized to fabricate bulk (NS) materials that exhibits an unusual properties which are very attractive for diverse structural and functional applications [4]. Ultrafine grained metals and its alloys made by severe plastic deformation techniques exhibits in fact excellent mechanical properties which is a result of significant grain refinement connected with dislocation strengthening. A great number of technologies are now commercially available for the SPD cold and hot working of metals (HPT- High Pressure Torsion, ARB – Accumulative Roll Bonding, TE – Twist Extrusion, CONFORM – Continuos Extrusion Forming, CEC – Cyclic Extrusion Forming, RCS – Repetitive Corrugation and Strenghtening, CGP – Constrained Groove Pressing, HE – Hydrostatic Extrusion) but in the recent years the most attention had gained an equal channel angular pressing method (ECAP). The equal-channel angular pressing method is at present one of the most promising and commonly used of all Severe Plastic Deformation processes . In this technique, a sample with circular, square or rectangular cross section is pressed through a die consisting of two channels. These channels have identical cross-section and intersects at a specific angle of (Φ). The work sample to be processed is prepared to fit tightly the die channel and has to be well lubricated in order to minimalize friction coefficient. The specimen is inserted to the first channel of a die and then pressed through the second channel (with a constant speed) using a plunger. Ideally, severe deformation process occurs by simple shear in the area of intesection of two channels. As the work sample is pressed through this area, new regions of sample are exposed to shearing strain providing a homogeneous shear deformation of the specimen apart from its end regions, which result in strain accumulation and grain/subgrain refinement [1-17].

The work hardening effect via ECAP method of ultrafine grained Al-Mg alloys was a subject of study in terms of both their microstructural and mechanical properties of many scientists. Recent works have shown that increased content of Mg in SPD aluminium alloys changes their properties such as the work hardening rate, dynamic strain aging effect, thermal stability, dislocation generation, grain refinement and thus the mechanical strength and plasticity [4]. Generally, an increased Mg content leads to the stacking fault energy decrease, normally resulting in low recovery rates and therefore finer grains after the severe plastic deformation process. When the Mg content reaches 4 % the material exhibits lower workability at ambient temperature witch result in cracking and failure occurring during ECAP. However, refining the grain size of light alloys such as Al-Mg is particular interest because the yield stress evidently increases when grain size starts to decrease, leading to an increase in the strength to density ratio, which is a desirable property when material is applicated in the automotive or aerospace industry [3-5].

In the present work binary Al-Mg alloy in heat treated state was subjected to the severe plastic deformation via Equal Channel Angular Pressing Method at room temperature. Microstructure evolution was comparatively investigated by light and electron microscopy. In order to examine the influence of cold working on mechanical properties of the alloy hardness measurement was performed.

2. Material and experimental procedure

The investigation was carried out on casting aluminium alloy ENAC-AlMg3 – 51100. The most important characteristic of this alloy is corrosion resistance, including exposure to seawater and marine atmospheres. Another advantage of this material is possibility to perform the heat treatment, which allows obtaining material with greater strength and increased workability through the precipitation of β phase. The chemical composition of investigated material is given in Table 1.

Table 1.
Chemical composition of EN AC 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

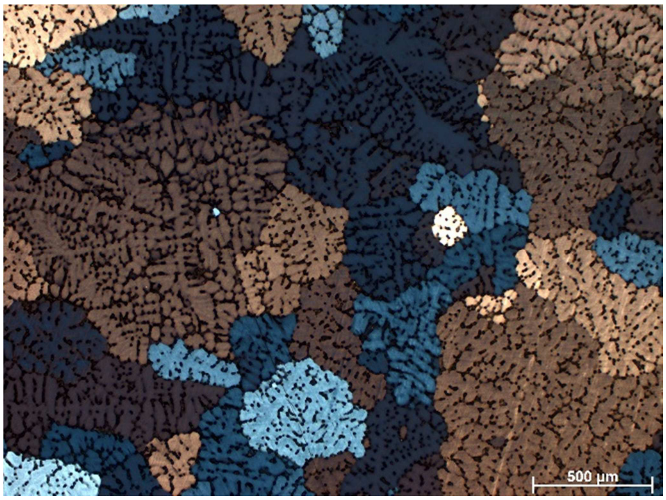
Equal channel angular pressing was carried out on the heat treated samples using a die with an internal angle (Φ) of 120°. Rods with diameter of 20 mm and length of 80 mm were premachined from an ingot and then subjected to solution treatment and artificial aging. The first stage of experiment was to determine most beneficial heat treatment conditions. In the second stage of investigation maximal number of pressings that material can be subjected was checked. It was found that after 5th pass some crack's on the surface of the sample appears, however in the heat treated samples similar phenomena occurs after 7th pass. All samples were pressed through a die at room temperature. As a lubricant molybdenum sulfide (MoS_2) was used. Microstructural characterisation of the alloy was carried out by optical and scanning electron microscopy (SEM) on the choosen planes of the samples that were polished using standard metallographic techniques (mechanical grinding and polishing with diamond pastes) and then anodized using Barker's reagent (5 ml HBF_4 48% in 200 ml H_2O, under a current of 20V through 90s). To reveal dendritic segregation in the initial state material, samples were etched using Weck's reagent (4g $KMnO_4$, 1g NaOH, 100 ml H_2O) until the etched surface becomes coloured. The microstructure of the alloy before and after ECAP was characterized using light optical microscope Axio Observer Image Analyser and the structure was observed using bright field and under polarized light. Chemical composition microanalysis was prepared on the scanning electron microscope ZEISS Supra using Energy-dispersive X-ray spectroscopy. Vickers microhardness (H_v) was measured on cross-section plain designated as y-plane by imposing a load of 300 g for 15 s using Vickers hardness tester Future-Tech FM-ARS.

3. Results

3.1. Structure

The representative grain structure of the AlMg3 alloy in the as-received state is presented in Figure 1a. Micrograph presents the dendrites of aluminium solid solution as the primary phase, with a eutectic mixture filling the interdendritic spaces. The second phase can be an intermetallic compounds that contain aluminium and one or more alloying elements. It is known that in polycrystalline materials the individual grains usually have a random crystallographic orientation with respect to one another, and the grain structure is randomly oriented which also represents the polarized light image of initial state alloy. Because of fact that alloy consist of more than one two alloings elements this system should be characterized as ternary which is composed of Al-Mg-Si, thus it is expected that the starting microstructure consist of three phases α-Al (bright matrix), Al_3Mg_2 (bright precipitations) and Mg_2Si (grey precipitation) which forms near grain boundaries in the form of irregular shaped particles (Fig. 1b). Those particles are mainly in a size of few micrometers. An EDS quantitative (Table 1) and (Fig. 2) analysis confirms the presence of main alloying elements in the investigated alloy. Structure observation prepared on scanning electron microscope and quantitative analysis confirms the presence of eutectic phase α+β (α-Al and Al_3Mg_2), and small amount of Mg_2Si intermetallic phase. Precipitation treatment leads to the disappearance of the dendritic structure and has no significant impact on the grain size of the alloy and their morphology (Fig. 3a). It is clear that during the precipitation treatment process from supersaturated solid solution, precipitates the hardening secondary phase β-Al_3Mg_2 that is uniformly distributed in the matrix of the alloy (mainly in the interdendritic space). In addition it can be observed that after precipitation treatment some grey irregular shaped particles appears (Fig. 3b). The results of the EDS quantitative analysis presented in (Table 3) and (Fig. 4) and the analysis of stoichiometric composition suggests that, the second phase can be Al_3Fe which forms into elongated blades or star-shaped clusters when is the eutectic phase or Al_6Fe that forms fine lamellar eutectic and is characterized by fine particle size [22]. It is expected that the β-Al_3Mg_2 hardening secondary phase has size of >1µm thus it's presence should be confirmed using higher magnification by application of TEM microscopy [18-21]. Figures 5a-f illustrates the evolution of microstructure (on the X – extrusion direction and Y – normal direction plane) of an alloy subjected to ECAP process after precipitation treatment up to 6 passes using route B_c. As it could be observed individual grains are still relatively large and are inclined by shearing bands. It is apparent that the microstructure is refined by the interaction of shear bands, slip bands and their increase in number (Fig. 6a-b). It may be also observed that at the beginning of the deformation process, when the sample is pressed only 2 times using route B_c the grains are very similar to that in the original precipitation treated material. It is also apparently that, not every grain is inclined by shearing or slip bands, shearing occurs only in the grains that are oriented in the direction of shearing. When to the material greater amount of strain is introduced it is harder to distinguish individual grains, thus even after four passes it is hard to observe the grain boundaries, however each grain is inclined by shearing or slip bands. The highest amount of strain (after six passes) result in obtaining most homogenous microstructure of all investigated samples in which grains are heavily inclined by shearing and slip bands. It leads to the conclusion that interaction of shearing and slip bands causes substructure (subgrains) refinement. The presence of shearing and slip bands can be well distinguished only when material is subjected to small amounts of strains which is presented on the Figs. 6a and b.

a)



b)

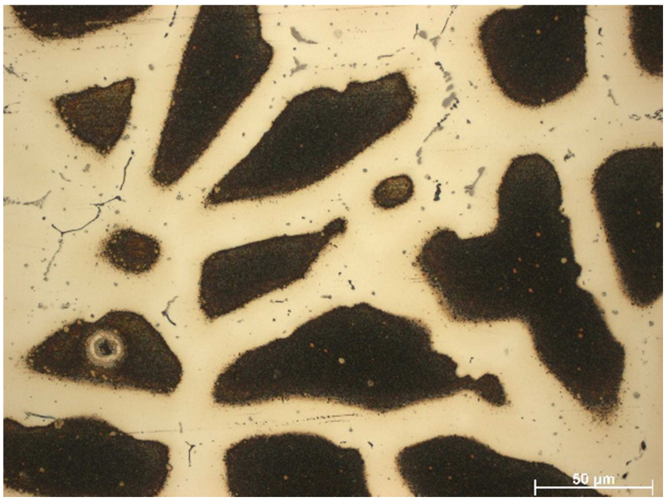


Fig. 1. Structure of AlMg3 in initial state (as cast) a) polarized light (Barker's reagent), b) bright field (Weck's reagent)

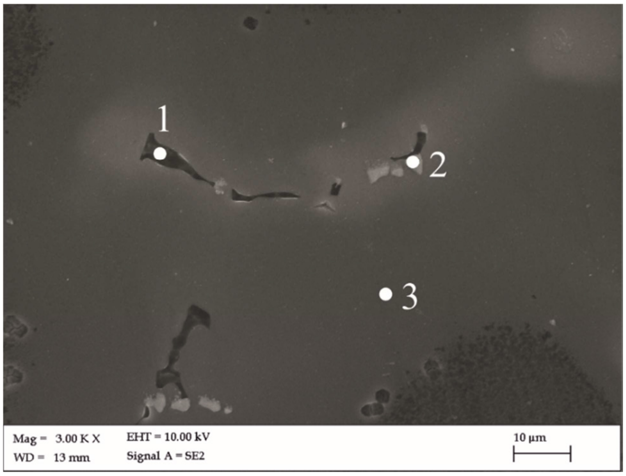


Fig. 2. SEM micrograph of the AlMg3 alloy in initial state and EDS spectra of corresponding points

a) b)

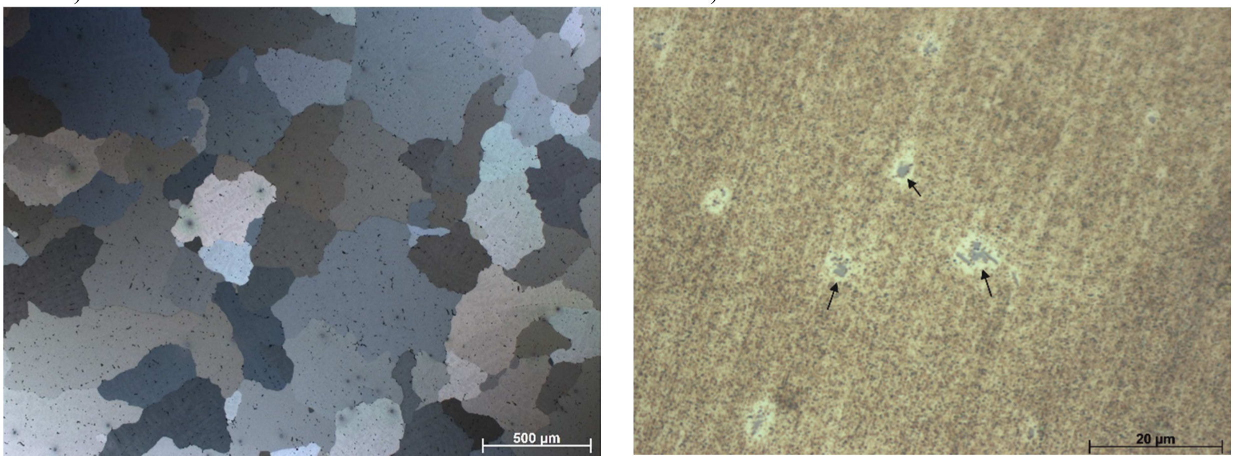


Fig. 3. Structure of the AlMg3 alloy after artificial ageing a) polarized light (Barker's reagent), b) bright field (Weck's reagent)

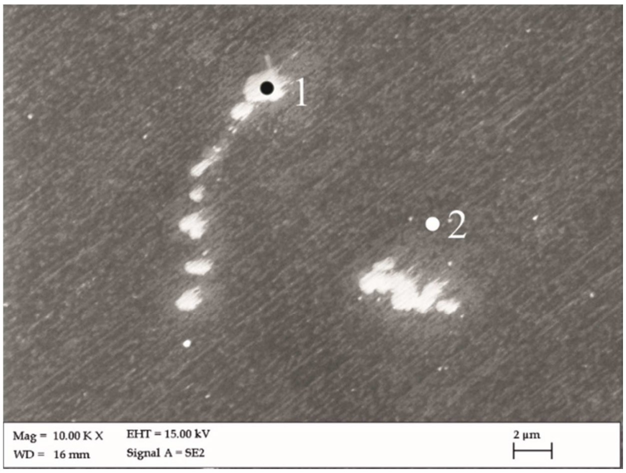


Fig. 4. SEM micrograph of the AlMg3 alloy in initial state and EDS spectra of corresponding points

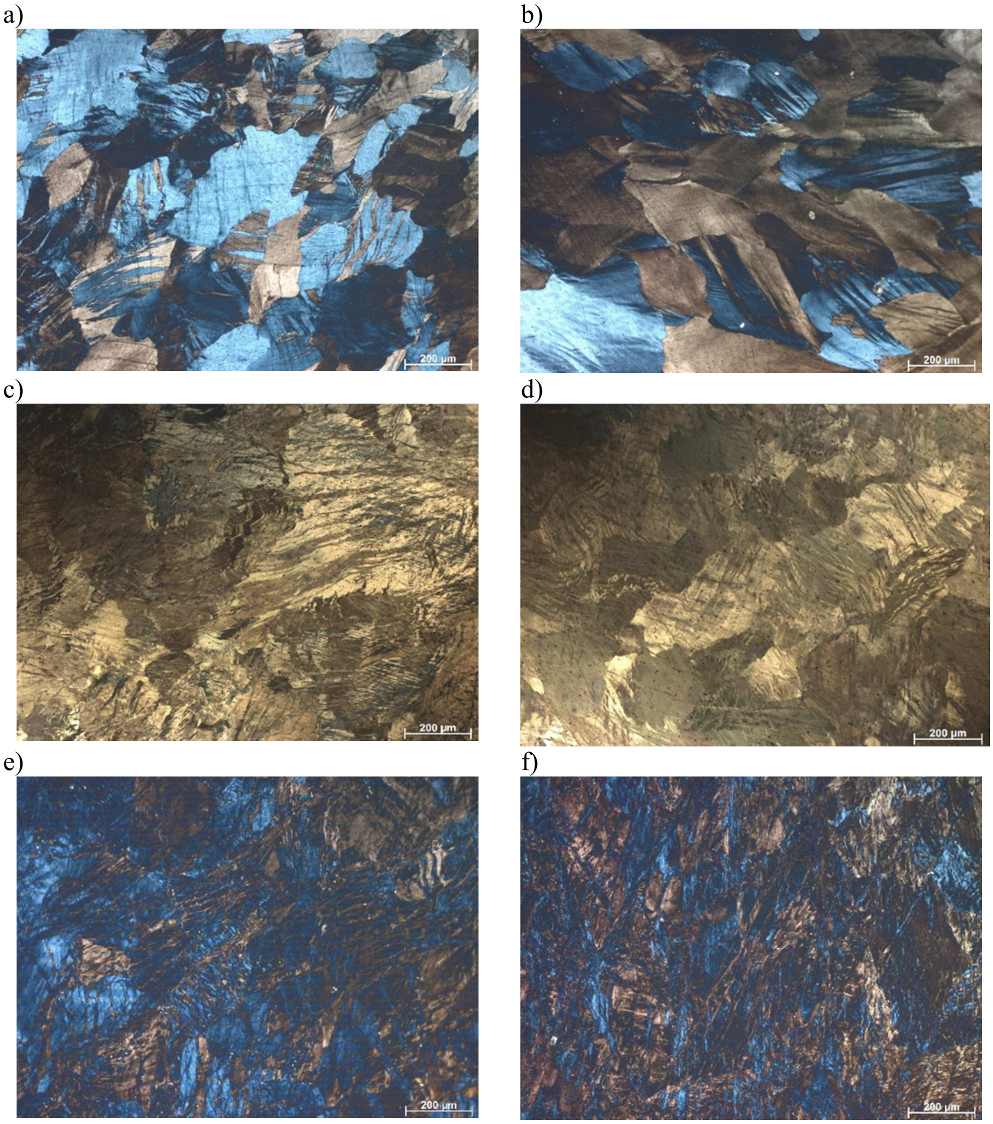


Fig. 5. Structure of the AlMg3 alloy after heat trearment and ECAP process a) 2 pass (X-plane), b) 2 pass (Y-plane), c) 4 pass (X-plane), d) 4 pass (Y-plane), e) 6 pass (X-plane), f) 6 pass (Y-plane)

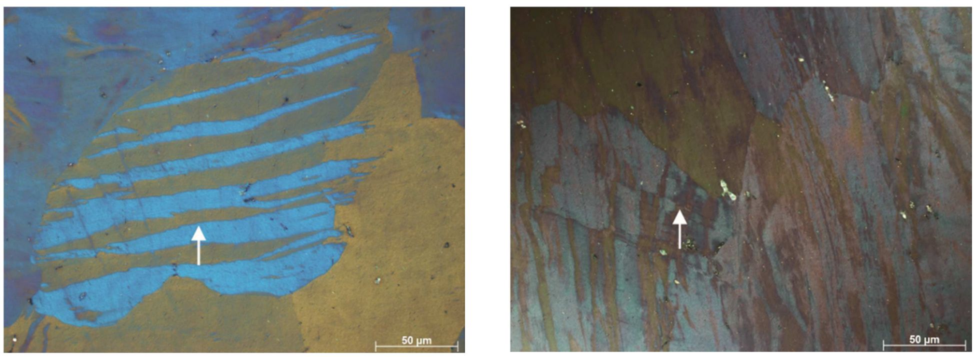


Fig. 6 Microstructures of AlMg3 alloy after ECAP process showing a) shearing bands, b) slip lines

Table 2.
Results of pointwise chemical composition analysis (from figure 2)

Point	Element	The mass concentration of main elements (%)	
		Weight (%)	Atomic (%)
1	MgK	16.01	17.56
	AlK	69.30	68.49
	SiK	14.69	13.95
2	MgK	08.46	09.30
	AlK	91.54	90.70
3	MgK	02.52	02.79
	AlK	97.48	97.21

Table 3.
Results of pointwise chemical composition analysis (from figure 4)

Point	Element	The mass concentration of main elements (%)	
		Weight (%)	Atomic (%)
1	AlK	64.32	78.87
	FeK	35.68	21.13
2	MgK	04.27	04.72
	AlK	95.73	95.28

3.2. Structure

To find the most beneficial heat treatment conditions two periods of solution treatment time were applied. In order to investigate the aging potential of supersaturated solid solution of AlMg3 aluminium alloy, hardness measurements were performed after three different periods of artificial aging (4, 8 and 12 hours respectively). It was found that even after 4h of aging, hardness increased rapidly (Fig. 7). Further aging,produce only small increase in material properties. This increase in hardness is a results of the β-Al_3Mg_2 phase apperance due to the precipitation from supersaturated solid solution. Nevertheless, for the further investigation, (ECAP), as the most beneficial condition, sample after 16 hours of the heat treatment was selected (8h of solution treatment and 8h of artificial aging). It can be observed (Fig. 8) that after severe plastic deformation process, with increasing equivalent strain, mechanical properties also increases. The values of the quivalent strain after N passes, ε_N, may be written in a general form of the relationship:

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)+\Psi cosec\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right] \quad (1)$$

It is known that increase in material properties of the severely deformed materials is caused by increase in dislocation density in as processed material. In current investigation, increase in hardness is a sum of the heat treatment and SPD process, which result in obtaining more than 200% grater hardness, in comparison to material in initial state ~ 45 Hv (as cast).

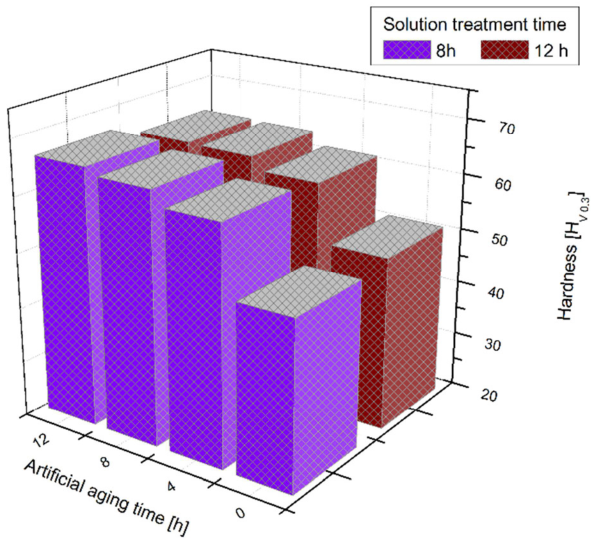


Fig. 7. Effect of heat treatment on the hardness of AlMg3 alloy

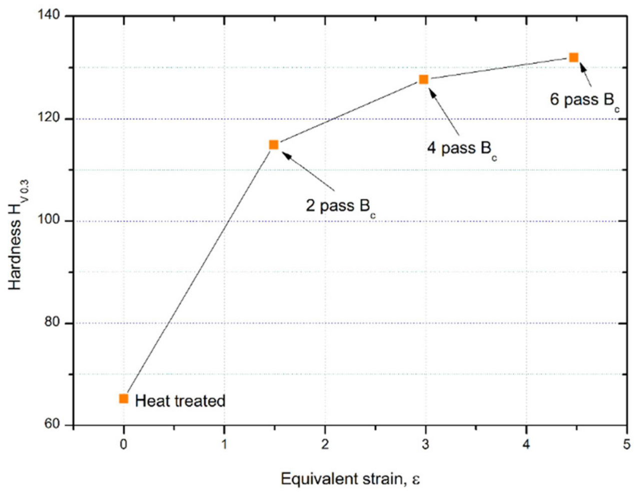


Fig. 8. Variation of the hardness Hv, with the equivalent strain and number of passes

4. Conclusions

A device for the Equal Channel Angular pressing has been tested at room temperature on the samples of heat treated AlMg3 aluminium alloy. It was found that the mechanical properties and the workability of the EN AC 51100 aluminium alloy can be increased by combining of the heat treatment with severe plastic deformation process. This increase in hardness after heat treatment is related with the precipitation of the β-Al_3Mg_2 phase from supersaturated solid solution of the aluminium alloy. The severe plastic deformation process using ECAP method result in the deformation of the structure of the heat treated material, grains become elongated in the shearing direction, depending on the observation plane and number of pressings. This deformation of structure causes apperance of the slip and shearing bands that refine substructure (subgrains). Decrease in the grain/subgrain size connected with increased amount of accumlated strain and defects in structure result in increased material properties of investigated alloy.

References

[1] M. Zha, Y. Li, R.H. Mathiesen, R. Bjorge, H.J. Roven, Achieve high ductility and strength in an Al-Mg alloy by severe plastic deformation combined with inter-pass annealing, Materials Science and Engineering A 598 (2014) 141-146.

[2] A.Vinogradov, S. Nagasaki, V. Platan, K. Kitagawa, M. Kawazoe, Fatigue properties of 5056 Al-Mg alloys produced by equal-channel angular pressing, NanoStructured Materials, 11/7 (1999) 925-934.

[3] G. Khalaj, M. J. Khalaj, A. Nazari, Microstructure and hot deformation of AlMg6 alloy produced by equal-channel angular pressing, Materials Science and Engineering A 542 (2012) 15-20.

[4] M. Liu, T. Jiang, X. Xie, Q. Liu, X. Li, H. J. Roven, Microstructure evolution and dislocation configurations in nanostructured Al.-Mg alloys processed by high pressure torsion, Transactions of Nonferrous Metals Society of China, 24 (2014) 3848-3857.

[5] M . Zha, Y. Li, R. H. Mathiesen, et al., Microstructure, hardness evolution and thermal stability of binary Al.-7Mg alloys processed by ECAP with intermediate annealing, Transactions of Nonferrous Metals Society of China 24 (2014) 2301-2306.

[6] M. Komarasamy, R. S. Mishra, Serration behaviour and shear band characteristics during tensile deformation of an ultrafine-grained 5024 Al. alloys, Materials Science & Engineering A 616 (2014) 189-195.

[7] M. Liu, H. J. Roven, X. Liu, M. Murashikin, R. Z. Valiev, T. Ungar, L. Balogh, Special nanostructures in Al-Mg alloys subjected to high pressure torsion, Transactions of Nonferrous Metals Society of China, 20 (2010) 2051-2056.

[8] P. Bazarnik, Y. Huang, M. Lewandowska, T.G. Langdon, Structural impact on the Hall-Petch relationship in an Al-5Mg alloy processed by high-pressure torsion, Materials Science & Engineering A, 626 (2015) 9-15.

[9] R. Valiev. Nanostructuring of metals by severe plastic deformation for advanced properties, Nature Materials, 3/8 (2004) 511-516.

[10] 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

[11] R.Z. Valiev, R.K. Islamagaliev, I.V. Alexandrov, Bulk nanostructured materials from severe plastic deformation, Progress Material Science, 45/2 (2000) 103-189.

[12] X. Suavage, N. Enikeev, R. Valiev, Y. Nasedkina, M. Murashkin, Atomic-scale analysis of the segregation and precipitation mechanism in a severely deformed Al-Mg alloys, Acta Materialia 72 (2014) 125-136.

[13] H.J. Roven, M.P. Liu, M. Murashkin, R.Z. Valiev, et al., Nanostructures and microhardness in Al and Al-Mg alloys subjected to SPD, Materials Science Forum, 604/605 (2009) 179-185.

[14] S. Rusz, K. Malanik, Refining of structure of the alloys AlMn1Cu with use of multiple severe plastic deformation, Journal of Achievements in Materials and Manufacturing Engineering 27/2 (2008).

[15] S. Rusz, K. Malanik, J. Dutkiewicz, L. Cizek, I. Skotnicova, J. Hluchnik, Influence of change of direction of deformation at ECAP technology on achieved UFG in AlMN1Cu alloy, Journal of Achievements in Materials and Manufacturing Engineering 35/1 (2009).

[16] T. Tański, P. Snopiński, P. Nuckowski, T. Jung, W. Kwaśny, T. Linek, Influence of processing routes on structure and residual stress in aluminium-magnesium alloy after ECAP, Journal of Achievements in Materials and Manufacturing Engineering 63/1 (2014).

[17] T. Tański, M. Król, P. Snopiński, M. Roszak, S. Rusz, P. Palcek, Thermal, structure and phases analysis of

the aluminium ENAC-AlMg5Si2Mn alloys, Materials Engineering 22/2 2015.

[18] M.J. Starink, A.-M. Zahra, *β′* and *β* precipitation in an Al–Mg alloy studied by DSC and TEM, Acta Materialia 46/10 (1998) 3381-3397.

[19] Y. Zhao, M. Polyakov, M. Mecklenburg, M. Kassner, A. Hodge, The role of grain boundary plane orientation in β phase precipitation of an Al-Mg alloy, Scripta Materialia 89 (2014).

[20] X. Sauvage, N. Enikeev, R. Valiev, Y. Nasedkina, M. Murashkin, Atomic-scale analysis of the segregation and precipitation mechanisms in a severely deformed Al-Mg alloy, Acta Materialia 72 (2014).

[21] J. Jie, H. Wang, C. Zou, Z. Wei, T. Li, Precipitation in Al-Mg solid solution prepared by solidification under high pressure, Materials Characterization 87 (2014).

[22] F. Vander Voort, ASM Handbook of Metallography and Microstructures, ASM International, 9, 2004.