THE EFFECT OF ECAP AND SUBSEQUENT POST-ECAP ANNEALING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AlSi7Mg0.3 ALLOY

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Abstract
The effect of equal channel angular pressing (ECAP) and post-ECAP heat treatment on microstructure and mechanical properties of AlSi7Mg0.3 alloy was investigated. The pre-ECAP heat treatment (annealing at 550 °C for 4 hours, water quenching and annealing at 250 °C for 5 hours) was effective in ductility improvement of as-cast alloy state. Repetitive ECAP of the heat treated alloy state at room temperature caused fragmentation and redistribution of eutectic Si-particles and an increase in alloy strength. The post-ECAP annealing for 2 hours at temperature range from 100 to 300 °C initiated gradual decrease in alloy strength and microhardness due to the recovery or recrystallization of solid solution, without noticeable ductility improvement of analyzed alloy.

Keywords: aluminium alloys, ECAP, annealing, microstructure, mechanical properties

1 Introduction

Castings made from commercial hypoeutectic foundry AlSi7Mg0.3 alloy are widely used in the automotive industry mainly on the score of their excellent castability, good mechanical properties at increased temperatures, high corrosion and wear resistance, low density and low thermal expansion. Low toughness and ductility of these castings limit their wider applications in practice. Mechanical properties of AlSi7Mg0.3 alloy castings are mainly determined by their chemical composition, character of their as-cast microstructure and existence of intermetallic phase particles and as-cast defects in alloy structure [1,2].

Mechanical properties of AlSi7Mg0.3 alloy castings can be improved by the optimization of their structure parameters, most frequently by application of the heat treatment. The low density and good wear resistance of this alloy opens new possibilities of its special applications. Recently, the foundry and PM alloys are processed by methods of the severe plastic deformations (SPD) [3]. The most widely used SPD technique for processing of AlSiMg alloys is the equal channel angular pressing (ECAP) because it is most efficient method for homogenization of the alloys as-cast microstructure and forming of the ultra-fine solid solution grains with high dislocation density. These micro- and substructure changes resulted in a simultaneous improvement of both strength and ductility of processed alloys [4-12]. However, before ECAP processing at room temperature, the AlSi7Mg0.3 alloy was generally heat treated

in order to increase its ductility [11-13]. Applications of severely deformed alloys is limited by thermal stability of its ultra-fine grained structure, because an alloys exposure at the higher temperature than the critical one causes the decrease in alloy strength, the solid solution grains growth and the decrease in its dislocation density due to solid solution recrystallization [8].
Taking into account a possibility of improving the properties by the morphology changes of AlSi7Mg0.3 alloy microstructure and its thermal stability, the effect of ECAP technique and post-ECAP annealing on microstructure and mechanical properties were evaluated in this paper.

2 Experimental materials and procedures

The experimental material was castings of AlSi7Mg0.3 alloy, which chemical composition is given in **Table 1**. The optimal character of as-cast structure of this hypoeutectic alloy was achieved by the modification of strontium on the content of 0.01 wt. % and by the inoculation with AlTi5B1 alloy on the content of 0.15 wt. % of titanium. The experimental casting samples of this alloy were cast using the technology of gravitational pouring into metallic moulds with optimized cooling. The cylindrical samples (10 mm in diameter and 100 mm long) were annealed at temperature of 550 °C for 4 hours, subsequently water quenched and artificially aged at temperature of 250 °C for 5 hours before their processing by ECAP technique. The pre-ECAP heat treated (HT) alloy samples were severely deformed at room temperature in the ECAP die ($\Phi = 90°$, $\Psi = 37°$) using route A (without specimen rotation between each pass). The repetitive specimen passing by this technique (**Fig. 1**) is the most effective in refinement and homogenization of this type alloy structure [9,10,14]. The cylindrical specimens were 4 times passed through ECAP die. After SPD, the deformed samples were annealed for 2 hours in the temperature range from 100 to 300 °C.

Table 1 Chemical composition of AlSi7Mg0.3 alloy [wt. %]

Si	Fe	Cu	Mn	Mg	Zn	Sr	Ti	Al
7.25	0.09	0.01	0.08	0.417	0.01	0.0121	0.19	bal.

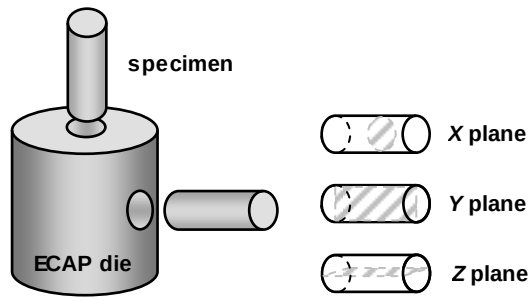

Fig.1 Scheme of ECAP technology and description of pressed samples planes

Analysis of the alloy microstructure changes was realized by evaluation of the eutectic Si-particles and precipitated Mg_2Si- and/or Si-particles morphology, their average size and number per unit area using the metallographic methods. The alloy microstructure in as-cast, pre-ECAP heat treated, ECAPed and post-ECAP annealed state was studied using the light microscope on metallographic samples prepared by grinding, polishing and etching in the etchant of 0.5 % HF in H_2O. After ECAP and post-ECAP annealing the alloy microstructure was studied in the

X plane – perpendicular plane on pressing direction, as shown in **Fig. 1**. The ECAPed alloy state substructure was studied using the transmission electron microscopy on the finally thinned foils in a solution of 33 % HNO_3 and 67 % CH_3OH at temperature of -35 °C and voltage of 16 V. The chemical nature of intermetallic phase particles that were found in alloy microstructure was evaluated by scanning electron microscope using EDX analysis. The effect of heat treatment and ECAP technology on changes of alloy mechanical properties was evaluated using the tensile test and Vickers microhardness measurement (HV0.2). The tensile test was realized according to STN EN ISO 6892-1 on short cylindrical samples (d_0 = 5 mm, l_0 = 10 mm) at the deformation rate of 2.5 x 10^{-4} s^{-1}. The tensile strength characteristics ($R_{p0.2}$ - yield strength, R_m - tensile strength) and ductility characteristics (A - percentage elongation after fracture, Z - percentage reduction of area) were determined.

3 Results and discussion

The as-cast AlSi7Mg0.3 alloy state microstructure was dendritic and heterogeneous. It consisted of α-solid solution dendrites, eutectic (α + Si) cells and intermetallic phase particles (**Fig. 2**). The eutectic Si-particles formed space skeleton and they grew from centre of eutectic cell to α-phase dendrite arms. In this state, the eutectic Si-particles have the shape of partially grouped fibres in the centre of eutectic cell and relatively coarse irregular rod-like particles in vicinity of α-phase dendrite arms [11-13,15]. The average size of eutectic Si-particles was 1.3 μm and their number per unit area was 6.3 x 10^4 mm^{-2} (**Table 2**). Intermetallic particles (**Fig. 2**, arrow marked) were present in *"*Chinese script*"* form at eutectic/α-phase dendrites interfaces. The results of EDX analysis (**Fig. 3**) in agreement with results of the works [1,2,12,15,16] indicated that these particles were complex intermetallic π($Al_8FeMg_3Si_6$)-phase particles.

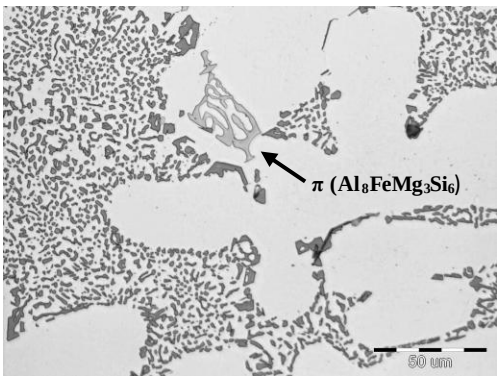

Fig.2 The microstructure of as-cast AlSi7Mg0.3 alloy state

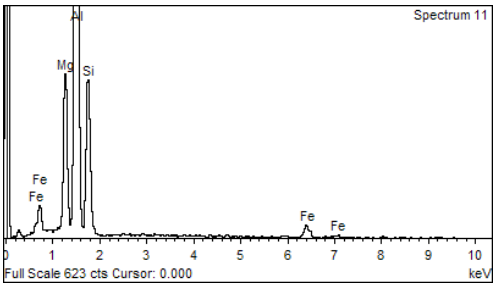

Fig.3 The EDX spectrum of intermetallic π($Al_8FeMg_3Si_6$)-phase particle

The pre-ECAP heat treatment consisted of the solution annealing (550 °C for 4 hours) and artificial aging (250 °C for 5 hours) was applied to optimize of AlSi7Mg0.3 alloy microstructure and to improve its plasticity. During solution annealing of as-cast alloy state, the spheroidization and coarsening of eutectic Si-particles occurred (**Fig. 4**). The average size of eutectic Si-particles was increased to 3.1 μm and their number per unit area was decreased to 0.9 x 10^4 mm^{-2}; as it is presented in **Table 2**. The intermetallic π-phase particles of irregular shape were dissolved during annealing and plate-like intermetallic manganese-enriched iron silicides $Al_{15}(FeMn)_3Si_2$-

phase particles were formed (**Fig. 4**, arrow marked), that is confirmed by EDX analysis (**Fig. 5**). The intermetallic π-phase and $Al_{15}(FeMn)_3Si_2$-phase particles are usually found in AlSi7Mg0.3 as-cast alloy microstructure [16].

Table 2 Average size and number per unit area of eutectic Si-particles measured for states of AlSi7Mg0.3 alloy

alloy state	average size [μm]	number per unit area [μm]
as-cast	1.3	6.3 x 10^4
pre-ECAP heat treated	2.9	1.0 x 10^4
ECAPed (route A, N = 4, T = 25 °C) [13]	2.4	1.7 x 10^4

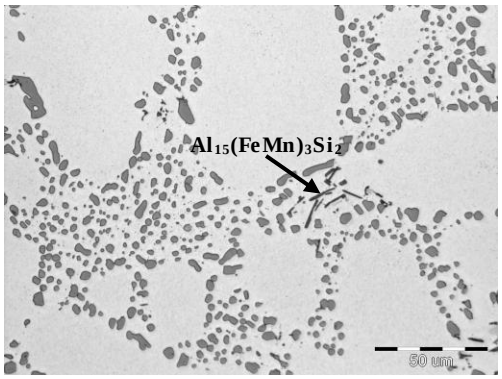

Fig.4 The microstructure of the pre-ECAP heat-treated AlSi7Mg0.3 alloy state

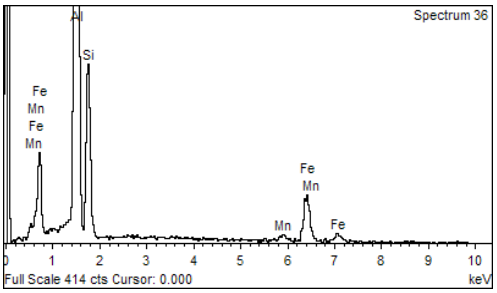

Fig.5 The EDX spectrum of intermetallic $Al_{15}(FeMn)_3Si_2$-phase particle

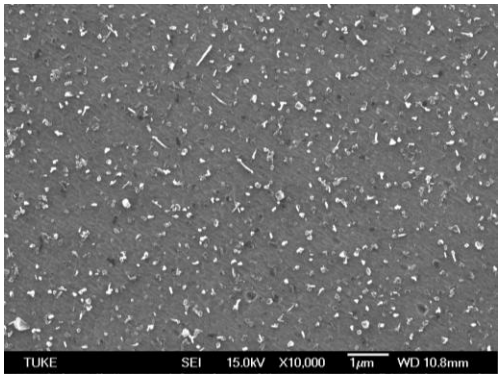

Fig.6 The Mg_2Si-phase and/or Si-particles situated in α-solid solution of the pre-ECAP heat-treated AlSi7Mg0.3 alloy state

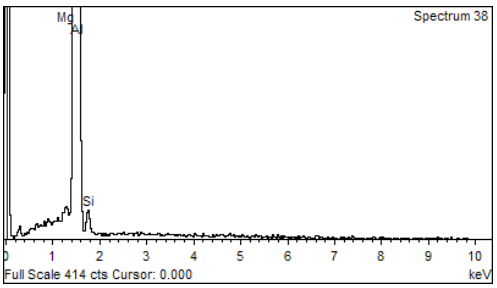

Fig.7 The EDX spectrum of Mg_2Si-phase and/or Si- particles

During application of the artificial aging of analyzed alloy, the incoherent rod-like Mg_2Si-phase and/or Si-particles precipitated uniformly from α-solid solution (**Fig. 6**). The EDX spectrum of these particles is shown in **Fig. 7**. These particles with an average size of 110 nm and a number per unit area of 10.1 x 10^6 mm^{-2} were homogeneously distributed in α-solid solution.

Processing of the heat treated AlSi7Mg0.3 alloy by ECAP technique homogenized its heterogeneous microstructure. A redistribution and fragmentation of the eutectic Si-particles were occurred (**Fig. 8**). The average size of eutectic Si-particles was decreased to 2.4 μm by reason of coarse particles fragmentation and their number per unit area was increased to 1.7 x 10^4 mm^{-2} [13]; as it is presented in **Table 2**. Homogenization of AlSi7Mg0.3 and AlSi7 alloys microstructures by ECAP technology were also founded by authors [4-12]. The application of severe plastic deformation had the same effect on the intermetallic $Al_{15}(FeMn)_3Si_2$-phase particles and the rod-like Mg_2Si-phase and/or Si-particles that were formed during applied pre-ECAP heat treatment of analyzed alloy. The average size of rod-like Mg_2Si-phase and/or Si- particles decreased from 110 nm to 71 nm and their number per unit area increase from 10.1 to 21.4 x 10^6 mm^{-2} (**Fig. 9**) in comparison with pre-ECAP heat treated alloy state.

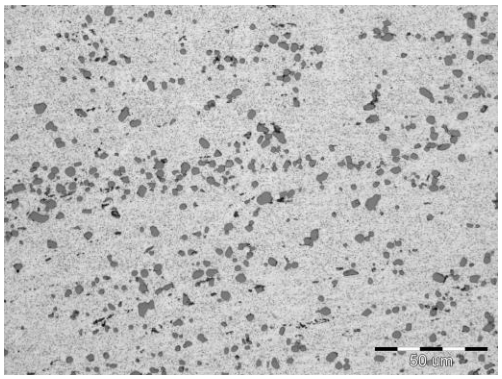

Fig.8 The microstructure of AlSi7Mg0.3 alloy after ECAP processing

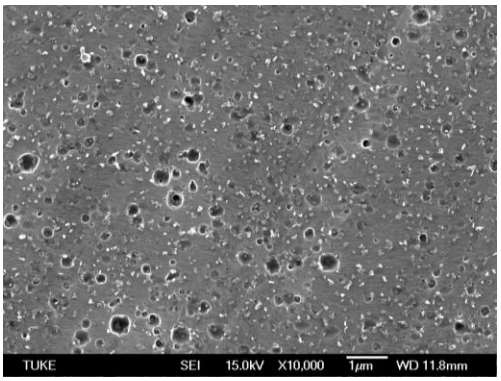

Fig.9 The Mg_2Si-phase and/or Si-particles distributed in α-solid solution of ECAPed AlSi7Mg0.3

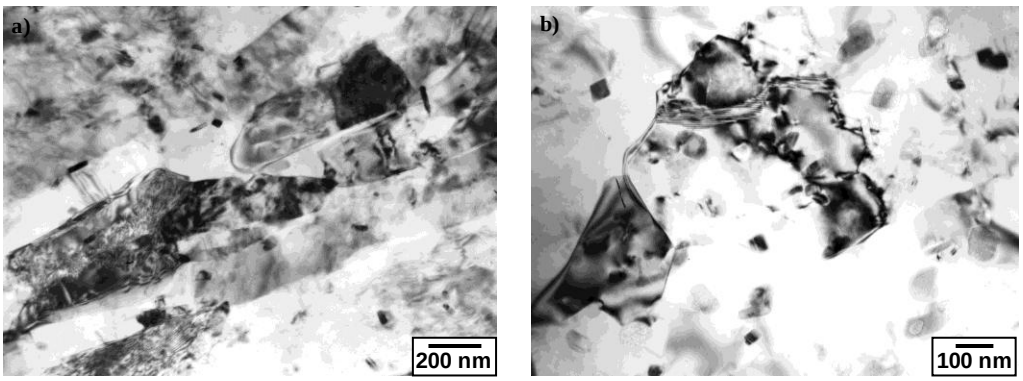

Fig.10 The substructure of α-solid solution and Mg_2Si-phase and/or Si-particles distributed in α-solid solution of AlSi7Mg0.3 alloy after ECAP processing

Figs. 10a and **10b** show AlSi7Mg0.3 alloy substructures after processing by ECAP technique analyzed by transmission electron microscopy. The grains and/or subgrains of α-solid solution have elongated shape, curved or wavy boundaries and high dislocation density both within the subgrains and in the zone of the subgrains boundaries. The average size of formed α-solid solution grains and/or subgrains is 230 nm in width and 610 nm in length.
From the comparison of ECAPed (**Fig. 8**) and post-ECAP annealed alloy state microstructure (**Fig. 11a-c**) is obvious, that the size and distribution of eutectic Si-particles and intermetallic $Al_{15}(FeMn)_3Si_2$-phase particles in solid solution were not notably changed during applied post-ECAP annealing.

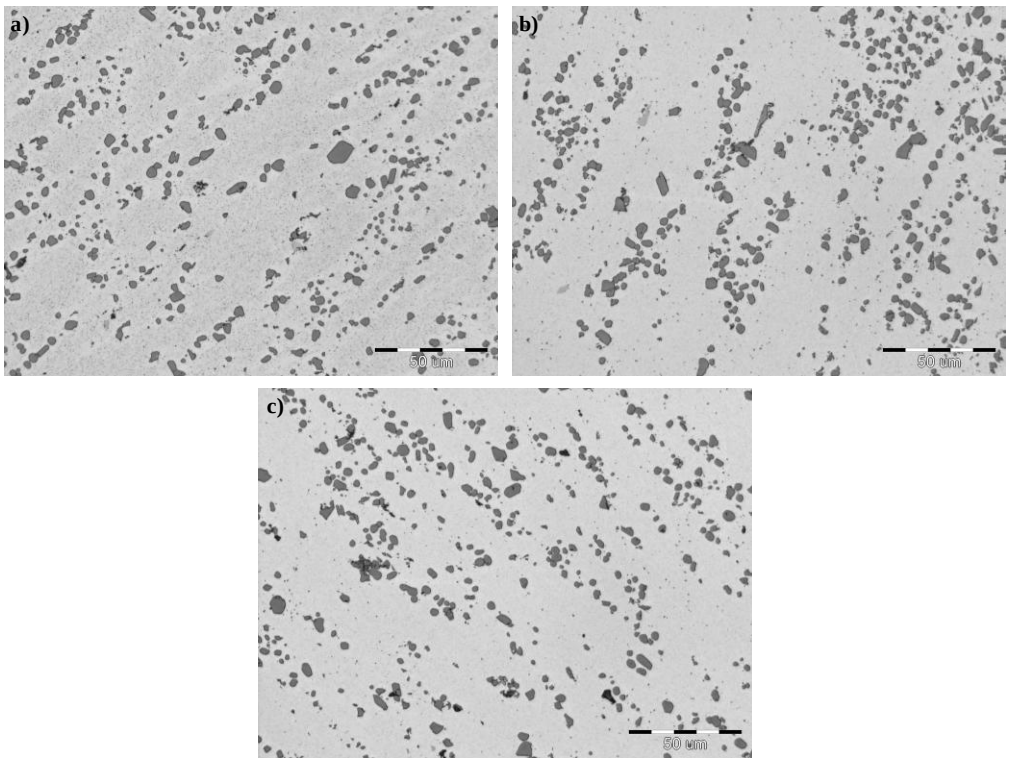

Fig.11 The microstructures of AlSi7Mg0.3 alloy after the post-ECAP annealing at 150°C (a), at 250°C (b) and at 350°C (c)

Effect of the pre- and post-ECAP heat treatment and the SPD of AlSi7Mg0.3 alloy realized by ECAP technique on its mechanical properties is evident from tensile stress-strain curves, shown in **Fig. 12**, and obtained values of microhardness, tensile strength characteristics and characteristics of ductility presented in **Table 3**. The application of alloy pre-ECAP heat treatment for as-cast alloy state leaded only to a significant increase in percentage elongation after fracture (A) from 7.1 % to 18.9 %, but the alloy strength was not affected markedly. The obtained ductility improvement is a necessary for application of severe plastic deformation at room temperature. It is the result of homogenous precipitation of fine dispersed Mg_2Si-phase and/or Si-particles from α-solid solution and spheroidization of eutectic Si-particles during pre-ECAP heat treatment. Repetitive ECAP of pre-ECAP heat-treated alloy state leaded to a

significant increase in alloy microhardness HV0.2 from 70.3 to 92.7, a large increase in alloy proof strength ($R_{p0.2}$) from 132 MPa to 281 MPa and tensile strength (R_m) from 183 MPa to 291 MPa, but the alloy percentage elongation after fracture was only slightly decreased. The strain and boundary hardening of alloy α-solid solution is the main effect of the AlSi alloys ECAP processing at room temperature [7].

The post-ECAP annealing of analyzed alloy in temperature range from 100 to 250 °C, caused the stepwise decrease in alloy strength and microhardness, as is shown Table 3. The values of alloy A and Z after post-ECAP annealing in this temperature range were nearly the same. The post-ECAP annealing at the highest temperature (300 °C) caused the significant decrease in microhardness of ECAPed alloy state from 92.7 to 63.2. During this post-ECAP annealing, the alloy strength and ductility were changed to the pre-ECAP heat treated alloy state ones (Table 3) due to strain hardening elimination of solid solution.

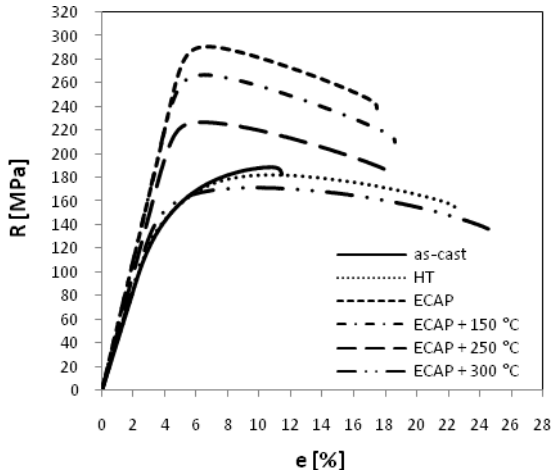

Fig.12 The stress-strain curves of AlSi7Mg0.3 alloy states

Table 3 Microhardness, proof strength, tensile strength, percentage elongation after fracture and percentage reduction of area values obtained for analyzed AlSi7Mg0.3 alloy states

alloy state	HV0.2	$R_{p0.2}$	R_m	A [%]	Z [%]
as-cast	66.4	126	189	7.1	16.2
pre-ECAP heat treated	70.3	132	183	18.9	37.4
ECAPed (route A, N = 4, T = 25	92.7	281	291	13.2	23.0
post-ECAP heat treated (100 °C / 2	87.7	262	276	14.1	25.4
post-ECAP heat treated (150 °C / 2	86.8	252	268	14.9	28.8
post-ECAP heat treated (200 °C / 2	85.4	224	242	15.0	25.1
post-ECAP heat treated (250 °C / 2	75.6	214	227	14.9	25.4
post-ECAP heat treated (300 °C / 2	63.2	141	172	21.9	35.4

4 Conclusions

The application of AlSi7Mg0.3 alloy pre-ECAP heat treatment (annealing at temperature of 550 °C for 4 hours, water quenching and annealing at temperature of 250 °C for 5 hours) leaded to modification of its as-cast microstructure. The average size of eutectic Si-particles was increased and their number per unit area was decreased on the score of partial spheroidization

and coarsening of eutectic Si-particles. The intermetallic π-phase particles were dissolved and $Al_{15}(FeMn)_3Si_2$-phase particles were formed during applied heat treatment. In addition, during application of the artificial aging of analyzed alloy, the incoherent rod-like Mg_2Si-phase and/or Si-particles precipitated uniformly from α-solid solution. These changes of as-cast microstructure leaded to improvement of alloy ductility that was required for its processing by severe plastic deformation technique at room temperature.

The ECAP of heat treated alloy state at room temperature caused fragmentation of eutectic Si-particles and incoherent Mg_2Si-phase and/or Si-particles and homogenization of their distribution in solid solution. Therefore, their average size was decreased and their number per unit area was increased. In addition, the formation of alloy solid solution ultra-fine grains and its strain hardening was achieved and it leaded to increase in alloy strength characteristics. The ductility of ECAPed alloy was higher than as-cast alloy state one.

The post-ECAP annealing of analyzed alloy did not cause the significant microstructure changes, but its strength was stepwise decreased. During annealing at the highest temperature (300 °C), the alloy strength and microhardness significantly decreased to values of the pre-ECAP heat treated alloy state level due to recrystallization of α-solid solution.

Acknowledgements

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