Structural Modification of Hypereutectic Al-16.5mass%Si Alloy by Thermo-Mechanical Treatment with ECAP

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Abstract Evolution of the microstructure and mechanical properties of the hypereutectic Al-16.5mass%Si-3.77mass%Cu alloy by treatment in the liquid state by magnetohydrodynamic (MHD) and hydrodynamic (HD) methods, followed by processing in the solid state by equal channel angular pressing (ECAP) method and thermal treatment has been investigated. This alloy has in initial state a very low value of plasticity at room temperature. Optical microscopy technique was employed in order to determine the evolution of the microstructure after different operating conditions of ECAP and thermal treatments. It was demonstrated that it is possible to significantly improve mechanical properties of this alloy by means of combining a low number of ECAP passes after an adequate combination of MHD+HD processing and thermal treatments.

Keywords: Aluminum Alloy, ECAP, thermal treatment, microstructure, mechanical properties

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1. Introduction

Among the Al-Si alloys the hypereutectic alloys are assessed as the most suitable material for automotive piston, since they have the higher wear resistance and heat resistance due to a high amount of Si. The drawbacks, however, are their low plasticity and strength. It is known that when the Si content is higher than that of the eutectic alloy, much larger polygonal primary Si particles (\approx 50 µm) are present in cast Al-Si alloys. Such coarse primary crystals are the main cause of low plasticity of the hypereutectic alloys.

Dispersing of the silicon crystals provides a possibility for improvement of both technological and functional properties of the alloys. Methods are known to refine the solid alloy structure due to treatment of the melt by acoustic fields, use of different modifying reagents, increase of the cooling rate at solidification, powder technology, melt punching and plastic deformation of the billets.

Particularly, severe plastic deformation (SPD) processing of alloys by equal-channel angular pressing

(ECAP) could be used. The rotary-die equal-channel angular pressing (RD-ECAP) was proposed for refining primary crystals of Al-23mass%Si alloy [1]. Before RD-ECAP processing, the alloy melt was modified and casted into a metal mold, the castings were heat-treated. After 32 RD-ECAP passes at high temperatures of 673 K and 623 K most of the large primary silicon particles were broken, the value of the impact toughness was 18 times higher than that of the as-cast alloy. The drawback of RD-ECAP technique is lesser strain of the billet end parts as compared with one of the billet main part that is cause of different degree of the alloy structure transformation in compared parts.

In order to develop advanced performance materials, a complex technology that involves induction melting of metals, manufacturing of rapidly solidified powders, cold compacting of powders in an Al can with following degassing, hot extrusion of workpieces and cold ECAP was used for Al-20wt%Si alloy [2]. The sizes of the primary Si particles were about 10 μ m in powders, about 5 μ m in extrudates and \leq 3 μ m after ECAP. Enhanced compressive strength up to 350 MPa after ECAP was achieved. The drawback of this technology is the use of many operations with low productivity of some of them.

The billets of Al-18%Si were processed by ECAP at temperature 623-723 K [3]. The initial casts were performed by the convert casting with acoustic fields treatment. After ECAP the average size of primary Si crystals was decreased to 20 μ m from 30 μ m in initial cast material. The ultimate tensile strength was increased to δ =220 MPa, the elongation to failure was increased to δ =15% from σ_b =190 MPa, δ =3% in initial cast material. The drawbacks of this ECAP method are complicated procedure and low productivity: the billet was extruded six times and every time the billet was manually rotated 90° around longitudinal axis before next charging in container.

An approach to the improvement of Al-Si alloys structure by means of combining the new methods of melt processing followed by SPD of billets by hydroextrusion or high pressure torsion methods was developed by authors [4]. Rational modes and parameters of the melt processing into the magnetodynamic installation (MDI) have been developed. It has been found that the complex magnetohydrodynamic (MHD) and hydrodynamic (HD) processing of liquid Al-Si alloys into the MDI allows to realize multiple and cyclic thermal and forced action on the melt. Such processing destroys micro-inhomogeneities in the liquid metal and further refines and enhances properties of solid alloys. Such physical modification of melts can be more promising than the well-known methods of liquid alloy treatment by reagents. Moreover, cooling of the melt at pouring into the mould and special heat treatment additionally improves the casting quality. Effectiveness of combining the new methods of melt processing followed by SPD of billets by hydroextrusion or high pressure torsion methods has been demonstrated.

In present paper, the effect of the combining MHD+HD processing of melt, thermal treatment and ECAP of the castings on structure and mechanical properties of hypereutectic Al-Si alloy is described. For the first time, the billets were deformed by ECAP method through the Stype angular die. It has been shown that it was possible to obtain hypereutectic Al-Si alloy with a significant improvement in its mechanical properties by means of combining a low number of ECAP passes with preliminary adequate thermo-mechanical treatment.

2. Materials and Methods

As subject of investigation the experimental hypereutectic Al-Si alloy was used (nominal composition were 16.5 mass% Si, 3.77 mass% Cu, 0.46 mass% Mg, 0.07 mass% Ti, 0.316 mass% Fe and the balance of aluminum). This hypereutectic alloy is similar to the commercial piston alloy A390.

MHD and HD processing of liquid Al-Si alloy were conducted as follows. Upon melting in the electric resistance furnace, the liquid alloy was poured into the MDI [4]. Thermal-forced processing was realized at 923-1073 K and the mass flow rate of the melt in the channel ranged 0.3-3.0 kg/s. Cast samples were poured into the chill mould. The pouring temperature was 1013-1023 K, the chill mould temperature was 493-508 K.

The casting samples were prepared in two states: by MHD+HD treatments of the melt (A series) or by MHD+HD treatments of the melt with a copperphosphorous alloy Cu7%P (B series). The use of Cu7%P modifier makes it possible to decrease the average size of primary Si crystals by 15-20% and to increase the density of crystals distribution in matrix [5].

Billets, of 22 mm in diameter, were extruded by ECAP at high temperature of 623-653K through the S-type angular die, as shown in Figure 1 [6]. The channel segments of S-type die were intersecting at the angles Φ =120°, each with Ψ =20°. The calibrating disk was placed behind S-type die in order to calibrate the billet for a repetitive ECAP without any additional operations of the reduction of the billet before each next pass.

Compared to conventional 90° ECAP, the S-type die provides larger cumulative shear deformation in one pass. Billet-after-billet extrusion was used. The back pressure was automatically provided for the first shear zone by the deformed material in the next shear zone. Such condition was more favorable for increase of billets' technological plasticity compared to 90°-ECAP. The false billet was extruded before the first billet of Al-Si alloy. The number of ECAP passes through the die varied from 1 to 3 with extrusion velocity 2 mm/min. The lubricant used to perform the ECAP was a composition based on graphite powder.



Figure 1. The scheme of the ECAP facility

The billets were extruded in two states: (i) with homogenization at temperature of 773 K during 6 hours before plastic deformation; (ii) without homogenization. After ECAP, a part of the samples was warmed to 773 K with follow-on quenching in water. The aging treatment of samples was made at 423-523 K during 2 hours. The marking of samples after different types of the treatment is provided in Table 1.

Table 1. The Marking of Samples

Thermo-mechanical treatment	Treatment of melt			
of the samples	MHD + HD	MHD + HD with Cu7%P		
Initial	А	В		
Initial + nECAP	An	B _n		
Initial + nECAP +	Ang	B _{ng}		
Quenching +Aging	iig	— iig		
Homogenization	AH	BH		
Homogenization + Aging	AHg	BH_{g}		
Homogenization + nECAP	AH _n	BH_n		
Homogenization + nECAP + Quenching + Aging	AH_{ng}	$\mathrm{BH}_{\mathrm{ng}}$		

The structure and properties of the alloys in the initial state, after SPD and after aging were studied using optical polarization microscopy POLAM P-34, microhardness measurements, volumetric and mechanical tensile tests. Samples were etched in HF aqueous solution and aqueous solutions of KMnO4 and NaOH before the microscopy investigation. The assessment of the particle sizes was made by automated image analyzer. More than 1000 Si particles were analyzed in every sample. Variational series were used to obtain distribution functions of the probability density of Si particle sizes. The particle average sizes and the dispersions of distribution were determined. Each of the microhardness (HV-5) values was assessed from ten random measurements throughout the cross-section of the samples. Three mechanical tensile tests of samples, 3 mm in diameter of the necking zone, were performed for each case at a velocity of 100 N/s.

3. Effect of SPD on the Alloy Microstructure

3.1. Deformation of the Samples after MHD+HD Processing of Alloy

After treatment of alloy in the magnetodynamic installation the samples of the A and B series were deformed by ECAP at elevated temperature of 623-653K. The high temperature deformation is accompanied by the processes of the dynamical aging with the heterogeneous discharge of the Al_2Cu stable phase and dynamical recrystallization. The increase of the samples density with growing number of passes indicates healing of the defects in the casting alloy (Figure 2).



Figure 2. Effect of the ECAP Passes Number on the Alloy Density in A and B States

After deformation, the morphology of the eutectic Si particles was significantly changed: their acicular shapes were changed to the spherical ones as a result of the faster development of the coagulation processes (Figure 3a and Figure 3b). The primary Si particles were crushed. The primary Si and the redundant eutectic particles were more uniformly distributed in the matrix.

The size distribution functions of Si particles were plotted for B, B_3 and AH_3 states of alloy (Figure 4a). The functions were bimodal and were approximated by two Gaussian curves. The example of doublet division for the

B initial state is shown on Figure 4b. The component S_1 correspondents to the eutectic particles, the component S_2 – to primary Si particles.



Figure 3. Microscopy of the alloy in different states: a) initial state of B series sample, b) B series sample after 3 passes of ECAP, c) A series sample after homogenization, d) A series sample after homogenization and 3 passes of ECAP



Figure 4. Distribution curves of the probability density of Si particles on sizes: a) distribution dependences from the thermo-mechanical regime of alloy treatment; b) division of doublet curve for the B initial state on components of S_1 (for eutectic Si particles) and S_2 (for primary Si particles)

The characteristics of the doublet components: average sizes (d₁ and d₂), distribution dispersions (Δ_1 and Δ_2), ratios of curve areas S₁/S₂, which are proportional ratios of part volumes of eutectic and primary particles, were determined for B, B₃ and AH₃ states (Table 2).

Table 2. Parameters of the Distribution Functions of the Si Particles on Sizes

Sample	d1, µm	$\Delta 1, \mu m$	d2, µm	Δ2, μm	S1/S2
В	20	11	33	20	2
B3	9	3.3	18	12	3.7
AH3	13	6	21	17	2

It has been identified that the most dispersed state was achieved in the B_3 case, namely after 3 ECAP of alloy, which was processed by MHD+HD with Cu7%P method. In this case there are most narrow distributions (dispersion Δ_1 =3.3 µm) at the least volume fraction of primary Si particles and the least average values of sizes of eutectic and primary Si particles, which indicated forming of quasi-eutectic state of the alloy.

3.2. Deformation after MHD+HD Processing Followed by Homogenization of the Alloy

For the purpose of Cu liquation elimination in alloy the continuous high-temperature annealing of the samples of the A series and B series was made at temperature of 773 K during 6 hours. Coalescence and coagulation of the particles were going on during the homogenization. As a result of the coalescence process the alloy structure changed into coarser state, in consequence of the coagulation process the spheroidizing of the eutectic particles occured (Figure 3c).

The homogeneous structure with the dispersed Si particles that was similar in morphology to quasi-eutectic state was formed by ECAP (Figure 3d). But, after analysis of the distribution curves of the particles on sizes for two quasi-eutectics, which were formed after the deformation of the cast alloy B_3 or homogenization alloy AH_3 (Table 2), it has been established that quasi-eutectic B_3 was more disperse and homogeneous.



Figure 5. Effect of Number of ECAP Passes on the Maximum Sizes of Primary Si Particles

It is necessary to mention that in the case of the homogenization of alloy before ECAP the sensitivity of alloy to number of ECAP passes was practically absent: after deformation the particle size distributions were similar for AH_1 , AH_2 and AH_3 . Same was observed for the dependence of maximum sizes of primary Si particles on

the number of ECAP passes that was different from alloy state BH (Figure 5).

A considerable grain refinement has been observed after ECAP deformation of the billets in any alloy state. In AH state the non-deformed material average grain size was found to be 45 μ m; after being processed by 3 ECAP passes the average grain size decreased approximately down to 15 μ m. Si particles were of substantially spherical form and were located at the grain boundaries (Figure 6b).



Figure 6. Effect of ECAP on the grain structure of alloy in AH state: a) initial AH state; b) after deformation AH₃ state

4. Effect of the Aging on the Strengthening of Alloys

After hot deformation the alloys in AH and BH states lost the capability to strengthening by aging as a result of disintegration of solid solution and discharge of Al₂Cu non-strengthening stable phase. To create a supersaturated solid solution of Cu and to restore strengthening capability, the alloys were quenched in water from the temperature of 773 K.



Figure 7. Effect of the Number of ECAP Passes on the Alloy Hardness after the Isochronal Aging at Temperature of 423-523K

The isochronal dependences of the alloy hardness in the temperature interval of 423-523K at aging time of 2 hours were created for the quenched samples of AH_n and BH_n states with n=1,2,3 (Figure 7).

The isochronal dependences of the alloy AH_n states were independent from the number of ECAP passes (Figure 7a). Their maximum of strengthening was higher and located closer to the lower temperatures as compared with the alloy BH_n states (Figure 7). The hardness of alloy AH_n states was about 1.68 GPa at temperature of 448 K. The maximum values of alloy BH_n states were about 1.50-1.55 GPa at temperature 473 K. The temperature shift of maximum to the lower temperature indicates a greater supersaturation of alloy matrix by Cu after quenching of AH_n samples that ensured a greater volume fraction of strengthening metastable θ' phase and more significant apparent strengthening.

5. Effect of Combined Treatment on the Mechanical Properties of Alloys

As shown above, A series alloys were processed in liquid state by MHD+HD methods, B series alloys were processed in liquid state by MHD+HD methods with Cu7%P modifier. The samples were processed in solid state by ECAP method at 623-653 K, quenching and aging treatment (Table 1). To test mechanical properties, the aging regimes were chosen for cases with maximum strengthening (Figure 7): the temperature of 448 K for 2 hours for AH₃ alloy, the temperature of 473 K for 2 hours for BH₃ alloy. The mechanical properties of alloy samples after different variants of treatment are shown in Table 3.

 Table 3. The mechanical properties of alloys after thermomechanical treatments

Alloy mark	Preliminary heat treatment	Number of ECAP passes	T, K of Quench.	T, K of Aging	σ _b , MPa	δ, %
В	-	-	-	-	252	0.94
B ₃	-	3	-	-	302	5.9
\mathbf{B}_{3g}	-	3	773	473	397	0.6
BH	Homogen.	-	-	-	102	0.6
BH ₃	Homogen.	3	-	-	219	2.9
AHg	Homogen.	-	773	448	313	1.2
AH_{3g}	Homogen.	3	773	448	459	0.6

The severe plastic deformation of billets by ECAP through the S-type die considerably increased plasticity of B alloys when treated from initial cast state, as well as in the case of homogenization before ECAP. When cast billets were deformed, the elongation to failure increased 6 times (to δ =5.9% from δ =0.94% in undeformed state). When deformed from the homogenized state, the elongation to failure increased 4.8 times (to δ =2.9% from δ =0.6% in undeformed state).

At the same time the strength increased by 20% and by 115%, correspondingly. As indicated by the microstructure study, such increase of plasticity is attributed to the forming of the quasi-eutectic (Figure 3b, Figure 3d) after ECAP. The most favorable to plasticity increase is the disperse microstructure B_3 (Figure 3b), which was formed during deformation of alloy when the melt was additionally modified by Cu7%P in MDI installation. It is

necessary to point out that this result was achieved with a low number of ECAP passes after processing of alloy in MDI installation.

Quenching and aging treatment of samples after homogenization + ECAP or after ECAP without preliminary homogenization resulted in the higher level of the strength. The highest ultimate tensile strength σ_b =459 MPa was achieved by homogenization + 3 ECAP passes + quenching at 773 K + aging treatment at 448 K. The plasticity of alloy after such treatment decreased to 0.6%. This result is explained by deposition of stable Al₂Cu phase at the grain boundaries and at the surface of Si particles during aging [7].

6. Conclusions

A method of structural modification of hypereutectic Al-Si alloys by means of combining the new MHD+HD methods of melt processing with severe plastic deformation of castings by ECAP was developed.

Use of the combined thermo-forced treatment of Al-16.5%Si alloy in magnetodynamic installation followed by ECAP of the billets at cast state or with the additional homogenization before ECAP provided considerable refinement of primary Si particles and formation of quasieutectics with particle average sizes of 10-15 μ m, as well as 5-6 times increase of plasticity.

Maximum strengthening of alloy was reached by preliminary treatment of melt in magnetodynamic installation without using Cu7%P modifier, homogenization of casings and 3 ECAP passes of billets, followed by aging. The resulting level of the ultimate tensile strength was σ_b =459 MPa, elongation to failure decreased to 0.6%.

It has been shown that it is possible to obtain hypereutectic Al-Si alloys with a significant improvement in their mechanical properties by means of combining a low number of ECAP passes with preliminary adequate thermo-mechanical treatment. For the first time it has been shown that ECAP through the S-type angular die could be successfully used to improve structure of the hypereutectic Al-Si alloy. The greater value of cumulative shear deformation of billet during one pass through the S-type die was provided as compared to 90°-ECAP. Billet-afterbillet ECAP naturally provides the back pressure for the first shear zone by the deformation material in the next second shear zone. Such condition was more favorable for increase of technological plasticity as compared to 90°-ECAP.

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