MICROSTRUCTURES, MECHANICAL PROPERTIES INGOT AlSi7Fe1 AFTER BLOWING OXYGEN THROUGH MELT

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Abstract
The new technology of producing ingot of AlSi7Fe1 high-strength is described. This new technology consists in saturation of melt with hydrogen, with further blowing with oxygen. Studied the microstructure, phase composition and mechanical properties of ingot after blowing oxygen of melt and ingot obtained with the traditional method. Have suggested that in liquid aluminum alloy AlSi7Fe1 because of blowing with oxygen arise refractory particles Al_2O_3. These particles Al_2O_3 further in crystallization serve as a modifier of the microstructure of ingot. Mostly observed modifications of eutectic phases. Thus saturation of melt with hydrogen, with further blowing with oxygen provides an increased tensile strength of ingot AlSi7Fe1.

Keywords: aluminum alloys, modification, melt, blowing oxygen, microstructure, strength, particles Al_2O_3

1 Introduction

Alloy AlSi7Fe1 is widely used in industry due to a combination of good castability and mechanical performances. Modification of microstructure, aimed at improving mechanical performance, has received continued research attention in this paper.

The technology of modifying of the microstructure of ingot silumins by blowing oxygen of melt describe in the first time. Initially, under industrial conditions, it was found that casting properties liquid alloy AlSi12Fe1.5 significantly improved after blowing oxygen. Duration of blowing was 3 hours. However, it did not lead to slogging of the melt. On the contrary, melt acquired high fluidity and produced castings had a smooth surface. Ultimate tensile strength of this ingot exceeds the ultimate tensile strength of the initial aluminum alloy. While it was found that initial aluminum alloy was heavily contaminated with hydrocarbons. It has been suggested this effect is due to hydrogen saturation melt, because the hydrocarbons are decomposed into carbon and hydrogen, the melt is saturated with hydrogen to limit of solubility, but carbon practically does not interact with components of the melt.

For laboratory research, we chose alloy AlSi7Fe1, most commonly used in industry due to its good casting properties. Iron in silumins may present inclusions of intermetallics Al_5SiFe in forms plate, significantly reducing mechanical properties and corrosion resistance of castings [1-3]. However, getting such a "dirty" alloy is justified due to economic reasons, since secondary metal is used to produce it. Therefore, industrial alloy AlSi7, containing up to 1% of iron, it is of interest for research. The existence of fragile crystals of silicon and Al_5FeSi in microstructure causes the low strength of metal [1-3]. The microstructure of alloy AlSi7Fe1 in cast state comprises phases Si and Al_5SiFe as a component of eutectic (α-Al+Al_5SiFe+Si). In order to

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increase the strength of alloy AlSi7Fe1, one can modify the melt. Modifiers envelop silicon crystals, and intermetallics Al_5FeSi hamper their growth, which increases the mechanical strength of alloy AlSi7Fe1.

Earlier studied AlSi7 alloy modification by introducing particles TiC, Al_2O_3, TiN [4-5]. Finely dispersed refractory particles, which create a suspension in the melt, simultaneously nucleating centers and the surfactants. The quality of ingot at modification depends on the distribution of dispersed phase in the melt. Is required provide the dispersion of particles and uniform distribution of particles in the melt. Method Blowing oxygen melt AlSi7Fe1 solves these two problems at same time.

Modification of aluminum alloys by introducing Al_2O_3 particles also was described for metal foam and metal matrix composites [6-12]. Formation of oxide shell even under argon cannot be avoided due to residual oxygen traces in gas. Solids inclusion can influence foam stability through their wetting behavior, their shape, and their distribution in the melt (network formation, clustering or segregation). A certain concentration of particles in an aluminum melt is necessary for producing stable foam by a collection of bubbles formed by any blowing gas. Stabilization of bubbles is due to segregation of particles to the bubble surfaces. If aluminum melt does not contain micrometer-sized particles, such particles can be created in-situ by bubbling air through the melt [8]. Foam stability can be increased by using Al_2O_3 instead of SiC, because of increased cell wall thickness. Certain oxygen partial pressure for foaming gas is required for stability of bubble and oxide shell formed on cell walls.

Studied the influence of Al_2O_3 nanoparticles on mechanical properties and the microstructure of alloy AlSi7 [11]. The addition of Al_2O_3 nanoparticles led to increased yield strength, ultimate strength, and ductility in the cast state. Improved mechanical properties of the ingot were determined by grain size and modifying eutectic phase. Nanocomposites AlSi20Cu4.5+Al_2O_3 also showed high values of strength and ductility [12].

2 Experimental Materials and Methods

Melting of AlSi7Fe1 was performed in a resistance furnace in an alundum crucible. The melt was overheated up to the temperature of 700°C. The melt was saturated with hydrogen by using titanium hydride, which significantly exceeds solubility limit. Titanium hydride was packed into aluminum foil; then it was mixed in the melt via the plunger. Thereafter melt was blown with oxygen for 1 hour. The voluminous flow rate of oxygen was measured by flow rate meter RMF-0.63 GUZ (2.5% error). Tubers for blowing were made of glass capillaries with D= 2 mm. The melt was poured into a sand mold. After solidification and cooling of castings samples for metallographic examination and mechanical tests were taken. The elemental composition of samples was determined by emission spectral analysis using "Optima 4300DV" (**Table 1**).

Phase analysis of samples conducted using X-ray diffractometer "Bruker D8 Advance". Metallographic studies were performed using an optical microscope Neophot 2 and a scanning electron microscope JSM-5900 LV with function EDS-analysis. Particle size was measured using an image analyzer SIAMS 600 (1% error). We are determined the volume fraction of eutectics, and the dendrites parameter is. Microhardness was measured using an instrument PMT-3 with a load of 0.2 N.

Microhardness to method Brinell was measured using the ball of 5 mm at a load of 250 kg. Produced material was also subjected to tensile test via Instron 3382 Universal at the room temperature. Samples for mechanical tests were made according to Standard for Tension Testing of Metallic Materials (ASTM E8/E8M).

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Table 1 Chemical analysis of samples, wt.%

Element	P	S	Al	Si	Mn	Fe	Cu	Zn
Wt.[%]	<0.02	0.086	89.466	6.788	0.376	1.078	0.733	0.656
Element	Mg	Cr	Ni	Ti	Zr	Sn	V	B
Wt.[%]	0.544	0.016	0.048	0.154	0.011	0.019	0.005	0.013

3 Results and Discussion

The microstructure of initial AlSi7Fe1 alloy is shown in **Fig. 1**. Microstructure consists of a primary dendrites α-Al (solid solution based on aluminium) and complex multicomponent eutectics. Crystals of plate-needle-like morphology $FeSiAl_5$ is part of eutectic (α-Al + $FeSiAl_5$). Crystals Fe_2SiAl_8 have the form of Chinese characters; silicon grows in the form of plates. In addition, in space between dendrites, there are binary eutectic colonies containing crystals Fe_2SiAl_8 and $FeSiAl_5$. It should be noted that iron is predominantly in $FeSiAl_5$-phase lamellar and acicular morphology. Crystals $FeSiAl_5$ has a negative influence on mechanical properties of ingots, as, during heat treatment, it retains its shape and determines a low level of ductility.

The microstructure of alloy AlSi7Fe1 obtained after the purged with oxygen melt is shown in **Fig. 2**. The microstructure of sample identical to initial microstructure and it is composed of dendrite α-Al and multi-component eutectic complex of same composition that is the initial sample.

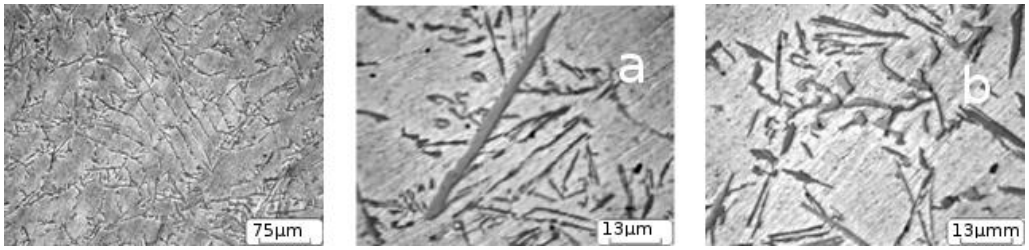

Fig. 1 Microstructure of AlSi7Fe1initial alloy a - $FeSiAl_5$in form of thin plates; b - Fe_2SiAl_8 skeleton shape

In microstructure of ingots after purged with the oxygen of melt is present dispersed porosity and nonmetallic inclusions (presumably Al_2O_3). Nonmetallic inclusions are mostly concentrated around the discontinuity of metal, some of them - on silicon crystals. Iron is present in the form of crystals Fe_2SiAl_8 and $FeSiAl_5$. In microstructure of samples after purged with oxygen epycrystals, Fe_2SiAl_8 have skeletal form, and much of structure is occupied finely differentiated eutectic colonies (Fe_2SiAl_8 + $FeSiAl_5$). It is more favorable for mechanical properties because crystals Fe_2SiAl_8 under heating (unlike from $FeSiAl_5$) capable of considerable fragmentation into many separate inclusions. Furthermore, presence and location of non-metallic inclusions suggesting their modifying effect on the morphology of eutectic phases.

Analyzing the data in **Table 2**, it can be noted that in microstructure ingot after purged with the oxygen of melt volume fraction of eutectic unchanged. Slight differences in microhardness eutectic and size of the interdendrite layer may be associated with differences in morphology of crystals Fe2SiAl8 and FeSiAl5 in multiphase eutectics. Results of metallographic study shown in **Table 3**. The parameter of dendrite (distance between the ages of the second order), which depends on crystallization rate in ingot after purged with oxygen is not changed. Despite the

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presence of porosity, the hardness of ingots after purged with oxygen above due to the presence of aluminum oxide. The main difference in the microstructure of ingots in morphology and proportion of iron-containing intermetallic phases that make up the complex eutectic. Preferential allocation of iron in the form of branched crystals Fe2SiAl8 has a positive impact on mechanical properties.

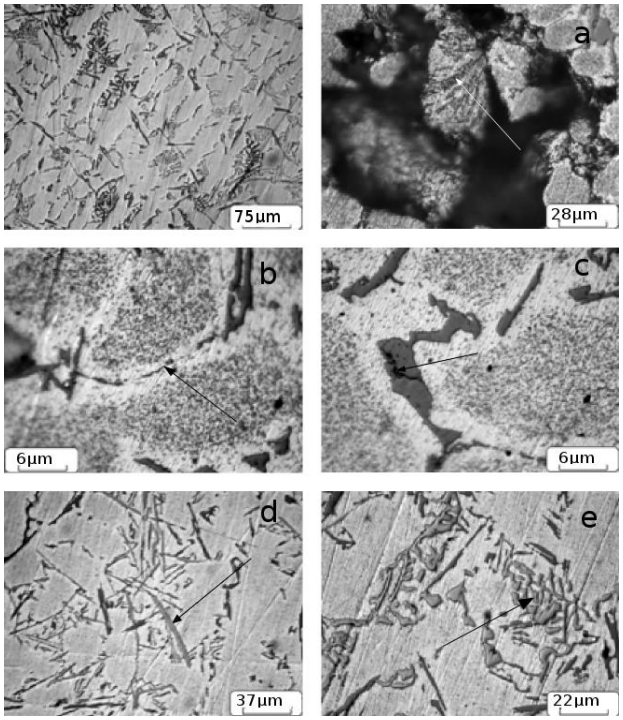

Fig. 2 Microstructure of AlSi7Fe1alloy (after melt by oxygen blowing) a - the modified a eutectic near defect (discontinuity of metal); b - pieces of skin an oxide (thikness less than 1µm) presumably Al_2O_3; c - the nonmetallic a inclusions on silicon crystals (presumably Al_2O_3); d - FeSiAl5 in the form of thin plates; e - Fe2SiAl8 in skeleton shape

Table 2 The results of metallographic measurements

Alloy	Volume fraction of eutectic, [%]	Microhardness of eutecticH$_\mu$, [MPa]	Microhardness of α-Al H$_\mu$, [MPa]	Macrohardness, HB	Dendritic parameter - Width interdendritic layer, [µm]
Initial alloy	14	1000	470	39,0	19,5 - 1,8
Alloy after blowing melt by oxygen	15	950	500	40,5	20,5 - 3,8

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We conducted SEM study of microstructure and EDS-test for AlSi7Fe1 alloy after blowing oxygen of melt. EDS-test or X-ray microanalysis (combination of identification chemical data of each phase with mapping) a simple method for phases identification in case of Al-Si alloys [13]. X-ray phase analysis together with that showed presence in microstructureof following phases: α-Al, Si, $FeSiAl_5$, Mg_2Si, $Al_6(Fe,Mn)$. Results of studyof microstructure and X-ray microanalysis of the AlSi7Fe1 alloy after blowing oxygen shown in **Fig. 3**. Microstructure AlSi7Fe1 alloy it contains crystallites of Al_5SiFe, which be included in eutectic (α-Al+Al_5SiFe+Si) (**Fig. 3**). Crystals of silicon and Al_5FeSi uniform distribution of in microstructure of cast sample. Agglomeration of silicon particles and crystallites of Al_5SiFe are observed. In microstructure also there are observed particles $Al_6(Fe,Mn)$.

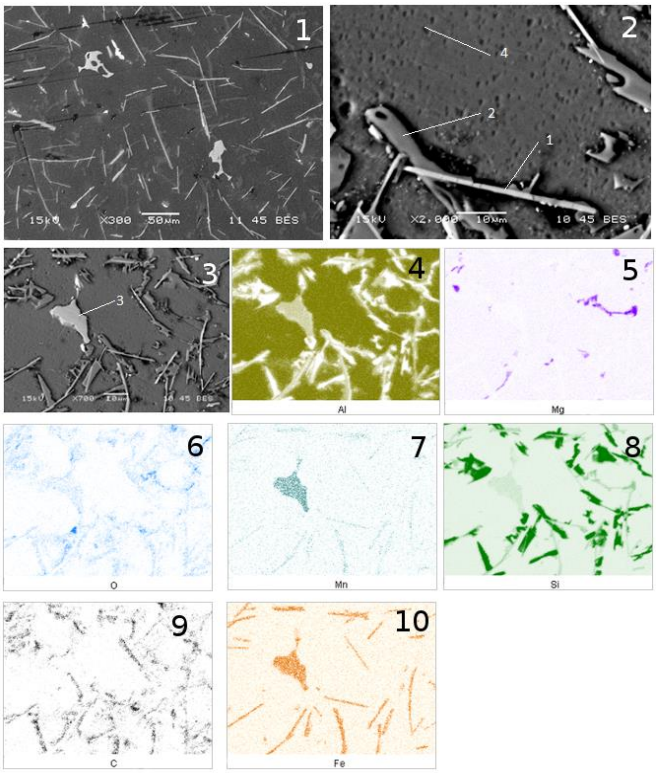

Fig. 1 Microstructure(pictures 1-3) andelement analysis (pictures 4-10) for cast alloy AlSi7

Initial alloy AlSi7Fe1 and alloy after blowing melt by oxygen was subjected to tensiletest at room temperature. Samples of the alloy after blowing melt by oxygen show tensile strength 175 MPa, the yield strength 172 MPa. Ductility (relative narrowing of) of the sample was 0% (**Table 3**). Samples of initial alloy AlSi7Fe1 show tensile strength 120 MPa, ductility (relative narrowing of) of the sample were 3% (**Table 3**). Results obtained for alloy after blowing are higher than ultimate tensile strength for alloy AlSi7 – 164 MPa according to Hatch [14].

We offer a description of processes occurring in AlSi7Fe1 liquid alloy by blowing it with oxygen. TiH_2 was added to liquid alloy AlSi7Fe1 before blowing with oxygen. At 680°C, hydrogen dissociates from TiH_2 in the form of bubbles gas in liquid aluminum: $TiH_2(s) \rightarrow Ti(s)+H_2(g)$. Hydrogen dissolved in pure molten aluminum to 0.69 cm^3/100 g (at the melting

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temperature) in accordance with [14], the residue of hydrogen in molecular form is burned. Burning hydrogen locally increases the temperature of the molten aluminum and creates conditions for conversion of Al_2O_3 in volatile suboxide AlO. Thus part of oxide is removed frommelt and melt refining occurs. The remaining portion of the oxide film on the surface of oxygen bubbles is divided into pieces and modifies melt.

Table 3 Results of mechanical tests of alloy AlSi7Fe1 specimens

Alloy	Yield strength $\sigma_{0,2}$, [MPa]	Tensile strength σ_b, [MPa]	Ductility (relative elongation) δ,[%]	Ductility (relative narrowing of), [%]
Alloy after blowing melt by oxygen	172	175	0.1	0
Initial alloy	120	127	0,5	3,0

Hydrogen also in aluminum alloys is present as an interstitial solution. Hydrogen can fill cavities in molecular state and form hydrides, it can be adsorbed on finely divided inclusions of aluminum oxides inside the metal and form chemical complexes with these inclusions [15-16].
In favor of proposed description of processes occurring in liquid alloy AlSi7Fe1 at purging it with oxygen, speaks a number of experimental results obtained by other authors. The size of Al_2O_3 inclusions for dynamically formed oxide films on molten aluminum is 0.1 μm [17]. Absorption of hydrogen contributes to coagulation of oxide films by barbotage [18]. Afanas'ev V. et al. (Popova M, Prudnikov A.) received experimental data on the effect of content of diffusion-mobile hydrogen on morphological characteristics and the numerical parameters of microstructure silumins [20]. Processing hydrogen molten aluminium caused shredding crystals of silicon and increase in sizeof aluminum dendrites. Shredding and growth retardation of silicon crystals causes an aluminum oxide with adsorbed hydrogen. Furthermore, there is partial reduction hydrogen of oxides of aluminum, iron, and magnesium by the reaction: $Me_2O_3 + 6H \rightarrow 3H_2O + 2Me$.
The experiment confirmed that impurities of aluminum oxide and hydrogen in liquid AlSi7Ay1 alloy accompany each other [21-22]. Hydrogen is absorbed in interface. Therefore pore formation is observed along grain boundaries during recrystallization. The process of removing particles Al_2O_3 and hydrogen from the aluminium melt by inert gas purge have studied earlier [23]. Particles Al_2O_3 larger than the order of one micron efficiently by flotation are removed from the melt. Fine particles are distributed in the melt.
Inclusions MgO also is able to modify of microstructure alloys AlSi7 and raise strength [19]. The introduction of 0.15-0.25% MgO into silumin's resulted in a gradual increase dispersion of eutectics. Increased share of MgO 0 to 0.25%, gave a gradual increase in values of tensile strength from 147 to 152 MPa.
In the pictures 2-3 are designated: 1 - Al_5SiFe, 2 - Si , 3 -Al_6(Fe,Mn), 4 –pores.

4 Conclusions

Influence of blow down oxygen of melt on microstructure and mechanical properties of alloy AlSi7Fe1 has been studied. The technology of producing of bullion AlSi7Fe1 by the method of saturation of melt of hydrogen to addition TiH2, with further blowing with oxygen, has

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experimentally substantiated. The addition of TiH_2 doesn't lead to foaming. On contrary porosity of ingot is insignificant. We hypothesized that by at blowing of the oxygen of the liquid alloy AlSi7Fe1 produced particles Al_2O_3. Particles Al_2O_3 make modified microstructure of alloy AlSi7Fe1. Saturation of melt of hydrogen also ensures its refining. Burning hydrogen locally increases the temperature of the molten aluminum and creates the conditions for the conversion of Al_2O_3 in the volatile suboxide AlO. Thus part of the oxide is removed from the melt, and the melt refining occurs. The remaining portion of the oxide film on the surface of oxygen bubbles is divided into pieces and modifies microstructures of ingot AlSi7Fe1.

Detailed research was conducted to investigate the influence of Al_2O_3 particles on the modification of the eutectic phases where iron is present in the form of crystals Fe_2SiAl_8 and $FeSiAl_5$. The structure of ingot obtained after the oxygen blowing includes phase iron Fe_2SiAl_8 of characterized skeletal form also much of structure is occupied finely differentiated eutectic colonies (Fe_2SiAl_8 + $FeSiAl_5$), it is more favorable for mechanical properties. As a result of the ultimate tensile strength of alloy after blowing the melt by oxygen exceeds the ultimate tensile strength of the initial alloy.

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