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Influence of High-Temperature Treatment of Melt on the Composition and Structure of Aluminum Alloy

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Abstract

The aim of the current study was to examine the structure of an alloy treated at various temperatures up to 2,000–2,100 °C. Among research techniques for studying alloy structure there were the electron and optical microstructure, X-ray structure, and spectral analysis, and for studying the developed furnace geometric parameters the authors employed mathematical modeling method. The research was performed using aluminum smelting gas-fired furnaces and electric arc furnaces. The objects of the study were aluminum alloys of the brand AK7p and AK6, as well as hydrogen and aluminum oxide in the melt. For determining the hydrogen content in the aluminum alloy, the vacuum extraction method was selected. Authors have established that treatment of molten aluminum alloy in contact with carbon melt at high temperatures of 2,000–2,100 °C has resulted in facilitating reduction of hydrogen and aluminum oxide content in the melt by 40–43% and 50–58%, respectively, which is important because hydrogen and aluminum oxide adversely affect the structure and properties of the alloy. Such treatment contributes to the formation of the extremely fine-grained microstructure of aluminum alloy.

Keywords: Innovative foundry technologies and materials, Heat treatment, Aluminum alloy structure, Fine-grained microstructure, Gas-fired and electric arc furnaces

1. Introduction

Development of the aluminum alloy castings manufacturing was accompanied by research and development [1–4], performed by Russian and foreign scientists [5–7].

Aluminum alloys thermal and thermochemical treatment is a known and widely developed topic. Processing is performed both in solid [8], liquid, [9] and in combined forms [10]. At the same time, a serious impact on the structure and properties of the alloys

has been noted [11]. Many studies have been made in the GNU VIAM [12] and their contribution to science is well known [13].

The work most relevant to the topic of this article dwells upon the melt processing being performed at a temperature of 1,200 °C [14]. It is known from a review of the literature that non-metallic inclusions that result from the interaction of the components of the alloys with oxygen and water vapor, have a significant effect on the properties of aluminum alloys. The formation of insoluble nonmetallic inclusions, mainly aluminum oxide Al₂O₃, is caused by high activity of aluminum. Non-metallic inclusions reduce castability, corrosion resistance and impermeability of alloys, as

well as worsen the mechanical and technological properties of aluminum alloys [14].

It is known that aluminum oxide and hydrogen interact. The adsorption of hydrogen by oxides of aluminum under the influence of chemical bonding forces begins on active centers, which are specific parts of the oxide Al_2O_3 surface, formed in the process of its dehydration [14].

The paper [14] studied the effect of thermo-temporary (high temperature) modes of the melt processing on the gas content in AK7ch alloy. The hydrogen content was determined by vacuum heating. The hydrogen content in the alloy depending on the melt heating temperature was as follows (average values of five specimens from each melt): 800 °C – 0.61 cm³/100 g, 900 °C – 0.69 cm³/100 g, 1,000 °C – 0.79 cm³/100 g, 1,100 °C – 0.79 cm³/100 g, 1,200 °C – 0.80 cm³/100 g. The data shows that upon heating the melt from 800 to 1,000 °C there is an increase of gas content in the alloys. However, upon heating the melt from 1,000 °C and up to 1,200 °C the hydrogen content in the alloy becomes sufficiently stable.

These results can be explained by the fact that a transition occurs when heated up to 1,000 °C and above. Oxide particles lose their activity with respect to hydrogen, complexes $(\text{Al}_2\text{O}_3)_x\text{H}$ are not formed and the melt stops being intensively saturated with hydrogen ceases.

Undoubtedly, more research is necessary to obtain a better understanding of the effect of high-temperature treatment (HTT) on the quality of the melt. However, the perspective of applying HTT for smelting cast aluminum alloys with desired properties, using low-grade raw materials, becomes obvious.

In our research, we have increased the temperature level up to 2,000 °C.

Relevance of the work is based on the fact that aluminum is the most abundant metal in the earth's crust and is on the third place after oxygen and silicon in its reserves, and 350 times more than the reserves of copper, lead, chromium, tin, and zinc combined. At the same time, deepening of the globalization process, intensification of the process of global warming make the application of aluminum alloys instead of ineffective alloys of iron or steel very topical in countries such as the USA, Japan, the UK, Germany, Ukraine, Russia and Uzbekistan.

Nowadays, along with obtaining high-quality products made of aluminum alloys, an increase of economic efficiency is relevant. A number of scientific research is conducted in this direction, in particular, conducting research in the following areas is one of the priorities: increasing the share of aluminum alloys in the industry, due to their mechanical, physical and performance properties; development of smelting technology, which prevents the harmful effects of unwanted factors; development of technology, which provides for resource and energy saving. Therefore, conducting research on the development of scientific and technical solutions in the formation of a high-quality structure of during smelting of aluminum alloys is of a great scientific and practical importance.

Research aimed at obtaining a high-quality aluminum alloy structure is conducted in many leading scientific centers of the world, including NIST (National Institute of Standards and Technology, the USA), Kobe Steel, LTD and the University of Tsukuba (Japan), in TU Wien and the Austrian Research Institute (Austria), CEGEDUR (France), the University of Technology in

London (the UK), EUROALLIAGES Association of European ferro-alloy producers (Belgium), at the Kyiv Polytechnic Institute (Ukraine), at the Polytechnic Institute of the Penza State University and the All-Russia Institute of Light Alloys (Russia) and the Tashkent State Technical University (Uzbekistan) [5–17].

As a result of the worldwide research on the high-quality aluminum alloy structure, the following research results were obtained. For reducing the hydrogen content in aluminum alloys there have been developed: a secondary treatment technology and modifiers' structure (NIST, the USA), an HTT technology (Kobe Steel and the University of Tsukuba, Japan), a melting technology and shaft-reverberatory gas furnace design (CEGEDUR, France). Also, a technology for recovering 30% of aluminum from its oxides (TU Wien and the Austrian Research Institute, Austria); a technology for reducing the oxide inclusions in the structure of aluminum alloys and technology of aluminum reduction from its oxides (the University of Technology in London, the UK, and EUROALLIAGES, Belgium) have been developed. In addition, the Polytechnic Institute of the Penza State University and the All-Russia Institute of Light Alloys (Russia) have proposed a gas furnace design and a technology for preventing the introduction of harmful gaseous inclusions in aluminum alloys [18].

The leading scientists of the world have developed various smelting technologies of aluminum alloys. A number of smelter designs for obtaining high-quality structure have been developed.

J. Boin, M. Bertram, H. Puga, J. Barbosa, A. Kalemann have devoted their research to the technology of reduction of aluminum from its oxides to produce high quality structures, and have developed a number of constructions of furnaces for melting aluminum alloys [19–23].

The scientists from the CIS countries have conducted an important research on the formation of aluminum alloys structure (V. A. Grachev, D. Andreev, A. Danilkin, V. Gogin, A. A. Grigorieva, L. F. Vjugin, V. A. Gutov, O. S. Eremin, A. N. Zadiranov). They have improved the design of gas melting units to make the melt quality better; they have increased the energy conversion efficiency of gas furnaces from 30% to 60% [2–4, 24–27].

The technology of high-temperature laser machining of aluminum alloys to reduce the quantitative hydrogen content in the melt was developed (S. Sasaba, A. Matsunava, S. Katayama). A flux composition was developed that helps to prevent diffusion of hydrogen into the melt (V. N. Simonov, L. F. Vjugin, V. A. Gutov, O. Eremin, A. N. Zadiranov). Researchers from Uzbekistan have developed a two-chamber structure of gas furnace bath and the mechanism of introducing iron and lead into aluminum alloys during smelting (A. O. Shazimov, F. S. Abdullayev, J. N. Mansurov) [28–31].

Despite the great advance in the research on aluminum alloys smelting, yet there are many problems to be solved. For instance, a technology of melting aluminum alloys which provides high-quality formation of structures without additional secondary treatment hasn't been yet developed. All studies have been conducted mainly at the temperatures of aluminum melting; whereas flux processing has been carried out only on the surface of the liquid bath. The studies mainly involved research on the physical and chemical processes between oxide inclusions and furnace atmosphere, the processes of hydrogen and oxide inclusions diffusion due to violation of the integrity of the oxide

film on the liquid bath. These studies haven't considered changes in the nature of the impact of carbon at various temperatures. No research on hydrogen and aluminum oxide diffusion in a carbon medium has been performed. The deoxidizing effect of carbon at high temperatures has not been taken into account. Development of HTT during melting, preventing the natural saturation of the melt with gas and oxide inclusions, with a focus on high-quality structure is an actual scientific and practical task.

2. Approach, work methodology, materials and equipment for research

The objects of the study were aluminum alloys of the brands AK7p and AK6, as well as hydrogen and aluminum oxide in the melt.

The equipment included: Brinell and Rockwell hardness testing machines, pendulum impact testing machine, tensile testing machine, NERIS grinding-and-polishing machines, optical microscopes MIM-8, MMU-3, scanning electron microscope REM-200, X-ray diffractometer DRON-2.0.

In studying the structure of the alloy, electron and optical microstructure, X-ray structure and spectral analysis were used, and when determining the geometric parameters of the developed furnace, mathematical modeling method was used. Analysis of the aluminum samples' porosity was carried out in accordance with Standard Specification GOST 1583-93 by means of software-hardware complex for the aluminum alloys ingots' porosity determination on the basis of the SIAMS700 analyser [32].

For the study of hydrogen and aluminum oxide content in the aluminium alloys AK7p and AK6, aluminum smelting gas furnaces and electric arc furnaces were selected, and Angara-2 AE-installation for determination of hydrogen content in aluminum casting alloys was used [33].

In order to control the heating temperature of the charge and to determine the heating temperature influences the charge and the hydrogen content of oxide inclusions during the experimental work of this dissertation, the shaft portion (1) of the gas shaft-reverberatory furnace has an installed platform (9) for heating the charge (13). This platform loads the batch heated to a predetermined temperature into the furnace shaft (7) by the loading device (8) and the hinges (10). The molten metal passes through the transition channels (15), formed in the lower part of the refractory walls (5), into the superheater chamber (4), which prevents the destruction of the integrity of the oxide film formed on the surface of the liquid melt and the diffusion of gaseous impurities generated as a result of combustion of the gas in the burners (2). For the sampling of the molten aluminium, the working chamber (1) is divided by another refractory partition (3) and transition channels (14). In the first stage of the experiment, the charge loaded into the shaft of the furnace was heated to the temperature of 20 °C, 100 °C, 200 °C, 400 °C and 500 °C. For obtaining objective results, experiments were carried out 5 times and 5–7 samples were selected for each of these 5 experiments.

The smelting furnace that was used in the experiments is shown in Figure 1.

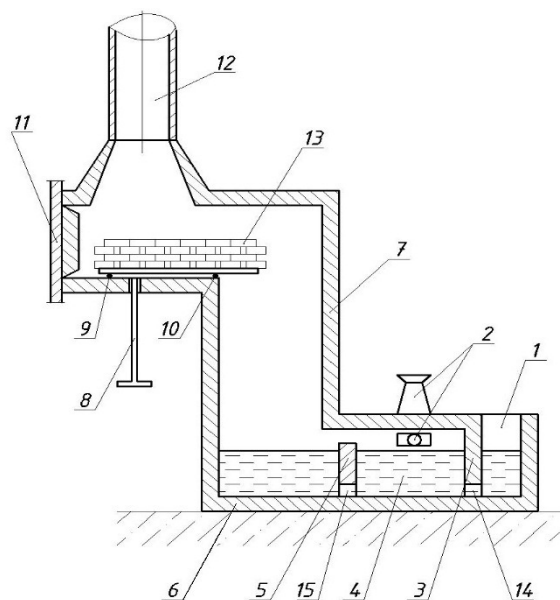


Fig. 1. Experimental gas-fired shaft-reverberatory furnace: 1 – sampling chamber; 2 – gas burners; 3, 5 – fireproof partitions; 4 – superheater chamber; 6 – hearth; 7 – furnace shaft; 8 – loading device; 9 – batch heating pad; 10 – hinge mechanism; 11 – batch charging opening; 12 – exhaust pipe (into the atmosphere); 13 – charge; 14, 15 – transition channels

As a method of determining the hydrogen content in the aluminum alloy, the vacuum extraction method was selected, where the air evacuating to a vacuum of 10^{-2} mm Hg was carried out by a backing vacuum pump, and pumping down to a residual vacuum in the system of 10^{-6} – 10^{-7} mm Hg was carried out by a diffusion pump. The change in pressure in the system was measured by a McLeod gauge. For analysis, the samples were heated to a temperature of 30–40 °C below the solidus line. Gases emitted from the sample were pumped into the analytical part of the apparatus by means of a diffusion pump. Control of the vacuum was carried out by a vacuum gauge BT-3. The hydrogen content in the sample was determined by a formula taking into account the change in pressure during the experiment, the volume of the analytical part of the apparatus, the atmospheric pressure, ambient temperature and the change in pressure of the McLeod gauge. Gas porosity of the alloy was determined using a porosity scale with pore number comparison conducted at three different squares of 1 mm² with the scale of porosity and a reference table. The content of the amount of aluminum oxide was measured by the photometric method and the concentration peaks method. In the first method, a purified sample weighing 1.0 g is placed in a 50 cm³ flask with the reaction solution. The reactionary portion is the following: 65 cm³ of ethyl acetate, 2.0 g of potassium bromide and 7 cm³ of bromine. The solution is prepared at room temperature. After dissolution of the sample, the reaction solution is added to the flask in an amount of 5 cm³, and is dissolved within 10–15 minutes at a temperature of 45–50 °C. The absorbance of the filtered solution was determined using a flame photometer – FPM. Aluminum oxide weight was deduced from

the ratio to the weight of its sample. In order to obtain samples, aluminum alloys smelted in gas silo-reflective and graphite electric arc furnaces were used. To control the temperature of the batch loaded, the liquid melt in the vessel of the furnace and electric arc furnace processing temperatures, cromel-alumel thermoelectric pyrometers (TCA) were used. Samples of aluminum alloys were selected at the temperature equal to a loaded batch at 20 °C, 100 °C, 200 °C, 400 °C and 500 °C, during processing by graphite electrodes in the melting and superheater chambers, with the use of a protective flux and without it. Electric arc treatment was carried out in the melting and superheater chambers of the furnace designed by the authors and described above. Five samples were selected for each method. When sampling, foundry ladles were used in order to prevent further oxidation. The teapot spout ladles for aluminum alloys sampling were developed by the All-Russia Institute of Light Alloys VILS (Russian Federation) [34].

The microstructure of the samples was studied by metallographic microscope MIM-8 and electron microscope REM-200. Microsections were prepared by the traditional method. Mass spectrometry and spectroscopic analysis methods were used to determine the content of impurities in aluminum alloys.

To determine the quality of aluminum alloys, Harrington desirability function was used. At the same time, indicators of each function were given as the functions of private desirability, and the overall desirability was calculated as the geometric mean of the general desirability functions. Hydrogen and oxide inclusions in aluminum alloy, tensile strength, hardness, toughness and elongation were accepted as desirability.

Table 2.

Chemical composition of the flux

No.	Chemical composition of the flux by %						
	C	CaO	NaCl	CaF ₂	SiO ₂	Fe ₂ O ₃ + FeO	Others
1	10.0	43.0	10.0	24.0	5.0	4.0	4.0
2	15.0	40.0	10.0	20.0	5.0	7.0	3.0
3	20.0	35.0	15.0	15.0	10.0	4.0	1.0
4	30.0	30.0	10.0	20.0	4.0	5.0	1.0
5	35.0	20.0	15.0	15.0	5.0	8.0	2.0
6	35.0	40.0	5.0	5.0	5.0	6.0	4.0
7	40.0	25.0	10.0	10.0	5.0	5.0	5.0
8	40.0	20.0	10.0	15.0	5.0	6.0	4.0
9	45.0	15.0	20.0	15.0	2.0	2.0	1.0
10	45.0	45.0	4.0	3.0	1.0	1.0	1.0
11	50.0	30.0	10.0	5.0	3.0	1.0	1.0
12	50.0	10.0	10.0	5.0	15.0	7.0	3.0

Three samples were selected for each flux composition. In order to obtain objective research findings the surface of the liquid bath was cleaned after each batch of flux. The second stage of the experimental studies was performed using an electric arc

3. Results

The aim of our study was to examine the structure of a resultant alloy treated at various temperatures up to 2,000–2,100 °C. We have studied the structure of the alloys during their heat treatment at different temperatures.

Four flux compositions with different carbon content were applied to determine the effects of integrity of the oxide film on the melt surface as well as the effects of the composition of the protective flux on diffusion of non-metallic inclusions in the melt.

In the first case, the heating of the batch to 400 °C showed minor changes in the structure. However, at the temperature ranging from 400 °C to 500 °C, the hydrogen and aluminum oxide content in the melt sharply decreased.

This can be explained by the removal of adsorbed moisture from the surface of the charge. Hydrogen and aluminum oxide content in the melt charge after heating to aforementioned temperatures are shown in the Table 1.

The chemical composition of the flux is represented in the Table 2.

Table 1.

Hydrogen and aluminum oxide content in the melt charge during heating up to 500 °C

No.	Temperature of the loaded batch, °C	Hydrogen content, cm ³ /100 g	Aluminum oxide content, %
1	20	0.60-0.62	10-12
2	100	0.55-0.60	8-10
3	200	0.52-0.55	7-8
4	400	0.40-0.42	5-6
5	500	0.33-0.35	4-5

furnace graphite electrode at high temperatures (above 1,828 °C) at which carbon activity increased dramatically.

In this mechanism, the graphite electrodes with a diameter of 76 mm and a TC-500 welding transformer were used. Considering

flow characteristics of aluminum, the upper and lower electrodes were installed on the sides of the mechanism.

At the second stage of research, the flux was charged into a preheated oven chamber, where it melted. After having reached the temperature of liquid flux of 800–850 °C, the batch was loaded. The composition of the obtained alloy is shown in Table 3.

The aluminum charge melting process lasted during 10–12 minutes. To improve processing efficiency, heat treatment was performed by the electric arc for 10 minutes. It took 40–45 minutes from the furnace launching to the complete melt processing. According to the findings, it can be concluded that the treatment of molten aluminum with carbon at high temperatures (2,000–2,100 °C) facilitates the removal of up to 40–43% of hydrogen and up to 55–58% of aluminum oxide from, negatively affecting the structure, from the melt. Moreover, the presence of carbon and chlorine in the flux improves processing efficiency by 35–40%. The main recovery portion (60–65%) is performed by carbon at high temperatures during electric arc processing with graphite electrode.

Table 3.
Composition of the obtained alloy

Flux No.	Hydrogen content, cm ³ /100 g	Aluminum oxide content, %
1	0.34-0.36	4-5
2	0.30-0.32	2-3
3	0.28-0.30	2-3
4	0.34-0.36	4-5
5	0.32-0.34	3-4
6	0.28-0.30	2-3
7	0.42-0.44	6-7
8	0.40-0.42	5-6
9	0.36-0.38	3-4
10	0.37-0.39	3-4
11	0.34-0.36	3-4
12	0.32-0.34	2-3

Thus, there has been developed a scientific and technical solution of forming a high-quality structure of aluminum alloys during the melting preventing the natural diffusion of hydrogen and oxygen, which form gas pores and oxide inclusions in the melt structure.

According to the research, for obtaining high-quality structure aluminum alloys during melting, it is necessary to produce an electric arc treatment of the melt in a gas-fired furnace bath at a temperature of 1,900–2,100 °C in a carbon medium. The arc formed by using graphite electrodes is of a local character, i.e. being effective only within the space between the electrodes. In order to ensure processing of the entire mass of metal, it is necessary to maintain the temperature of the melt at the expense of additional thermal energy expenditure. Therefore, it is effective to use the construction of the shaft-reverberatory gas-fired furnace with an electric arc melt processing with a melting bath of a new design. At the same time, for melting the charge and maintaining it at a predetermined temperature, the heat of combustion products of natural gas is used.

The processing of the liquid melt by graphite electrodes at 2,000–2,100 °C provided the displacement of hydrogen and

aluminum oxide, diffused into the structure. The application of the developed technology ensured the displacement of up to 40–43% hydrogen and 55–58% of aluminum oxide from the melt structure. This allowed obtaining a high-quality structure of aluminum alloys with a minimum content of negatively affecting hydrogen and aluminum (Figure 2).

The obtained findings have made it possible to create a highly efficient technology of aluminum alloys smelting and a gas-electric-arc furnace design.

4. Conclusions

On the basis of conducted research, the following conclusions have been made:

1. It has been determined that the treatment of molten aluminum alloy in contact with the carbon melt at high temperatures of about 2,000 °C facilitates removal of up to 40–43% of hydrogen and 50–58% of aluminum oxide, and contributes to the formation of a fine-grained aluminum alloy microstructure.
2. There has been developed a new composition of a protective layer (flux) to the surface of the molten bath during the melting of aluminum alloys, which has allowed reducing the diffusion of non-metallic inclusions into the melt by 15–22%.
3. An aluminum melt HTT technology has been developed where the aluminum melt is processed at the temperature of 2,000–2,100 °C, which allows reducing the hydrogen content and oxide inclusions in the resultant melt, and obtaining a more fine-grained cast structure (grain size before the HTT $d = 10.1\text{--}10.4\ \mu\text{m}$, after the HTT $d = 3.7\text{--}4.2\ \mu\text{m}$).

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