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Filtration Refining of Primary Aluminum from Impurities of Vanadium Intermetallides

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Abstract – *In the last decade, there has been a tendency to use lower-quality raw materials for the production of anodes in the electrolytic production of aluminum. This is mainly due to the extraction of heavy oil (which includes asphaltene compounds). Asphaltenes concentrate metal impurities (Fe, Si, Ti, V, etc.), which during coking are converted into coke, and then into aluminum. One of the impurities in primary aluminum, which reduces the electrical conductivity of the metal at a concentration of about 2 ppm, is vanadium. This work has studied the process of filtration refining of aluminum melt, after flux treatment of the melt in a casting ladle with boric acid (H_3BO_3), in order to clean it from vanadium diboride VB_2 formed during flux refining. Thermodynamic and experimental studies of the process of filtration refining of liquid raw aluminum after flux treatment with boric acid (H_3BO_3) from intermetallic compounds VB_2 formed during refining in a bulk volume filter made of Ekibastuz coal ash with a binder in the form of lignosulfonate have showed the possibility of reducing the vanadium content in aluminum melt.* ***Copyright © 2024 Praise Worthy Prize S.r.l. - All rights reserved.***

***Keywords**: Aluminum, Filtration, Refining, Vanadium, Vanadium Intermetallics*

Nomenclature

ΔH Change in enthalpy [kJ/mol]
ΔS Change in entropy [J/(mol C)]
ΔG Difference in Gibbs free energy [kJ/mol]

I. Introduction

Aluminum remains one of the leading structural materials in mechanical engineering, construction, electrical engineering and other industries, which leads to the presence of research and the constant development of various technologies for the manufacture of structural products from aluminum alloys [1]-[6]. In recent decades, the problem of reducing the quality of ores and other raw materials for the production of base metals has been increasing in the world, which leads to the need to find solutions for the integrated use of raw materials and options for processing the resulting waste [7]-[10]. In recent years, there has been a tendency to involve lower-quality sources of raw materials for anode production in the electrolysis production of aluminum [11]. This is mainly due to the extraction of heavy oil (which includes asphaltene compounds). Asphaltenes concentrate metal impurities (Fe, Si, Ti, V, etc.), which, during coking turn into coke and then into aluminum. One of the impurities in primary aluminum, which reduces the electrical conductivity of the metal at a concentration of about 2 ppm, is vanadium [11].

It is known from [11] that the materials shown in Table I are sources of vanadium and other impurities in aluminum during electrolysis.

TABLE I
SOURCES OF VANADIUM AND OTHER IMPURITIES IN ALUMINUM IN ELECTROLYTIC PRODUCTION ARE PRESENTED IN GRAMS/TONNE AL

Source of income	Element, gr/t					
	Si	Fe	Ti	V	Zn	Ga
Alumina	123	348	67	24	60	131
Anode mass	173	227	3	33	1	2
Cryolite	19	31	1	2	-	-
Structural elements, tool	200	223	-	-	-	-
Amount of receipts	515	829	71	29	61	133
Transformed into aluminum	473	451	25	20	48	65

In Kazakhstan, local coke from UPNK-PV LLC (Pavlodar, Republic of Kazakhstan) with an increased content of vanadium impurities is partially used for the production of baked anodes for aluminum electrolyzers.

[11]-[13] have investigated the processes of reducing the vanadium content in primary aluminum by treating it with boron. The kinetics of chemical reactions with the formation of VB_2 compounds during refining with Al-B-based ligatures is characterized by a low rate due to the formation of an insoluble ring of reaction products consisting of vanadium diboride (VB_2). The thickness of this ring increases from 1 μm to 20 μm as the melt holding time increases to 720 minutes [12]. In [13], boric acid has been added to the anode mass during the production of baked anodes, and during aluminum electrolysis, boron has passed into the aluminum melt and has contributed to a decrease in the vanadium content in the melt. This method is characterized by the disadvantages of the previous method. In the practice of aluminum production, methods of feeding boric acid (H_3BO_3) during the pouring of aluminum in the foundry department directly into the molds of the casting machine are also known. However,

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this method has showed an insignificant decrease in the vanadium content in aluminum (by 4 - 6 ppm). Based on the above, it can be concluded that further research is needed to develop methods for separating the impurities formed during flux refining of aluminum melt with boric acid (H_3BO_3) from the resulting vanadium diboride VB_2.

One of the most effective methods for removing suspended impurities in melts is filtration refining [14], [15]. The analysis of [14]-[16], which are works devoted to the thermodynamic foundations of the filtration process, shows that the efficiency of separating inclusions varies significantly depending on the filter material and the filtration scheme. Recent studies [14]-[16] have proposed a Reaction-Crystallization Mechanism (RCM) for melt filtration. The RCM scheme includes the following stages: delivery of the impurity to be removed to the filter surface and the component that binds it, their adsorption on the filter surface and a chemical reaction between them, as well as the release of a non-metallic or phase that can accumulate on the surface or be removed by absorption into the filter pores.

The article contains the following sections: materials and methods of research, results and discussion, and conclusions. The section "Materials and methods" shows the chemical composition of the object of study and methods for its determination. The main results are presented as data on chemical composition measurements, as well as thermodynamic modeling.

II. Materials and Methods

The authors have conducted a study of the process of filtration refining of aluminum melt, after flux treatment of the melt in a casting ladle with boric acid (H_3BO_3), in order to clean it from vanadium diboride VB_2 formed during flux refining. The study has been conducted in two stages:

- Thermodynamic modeling of the process of interaction of the filter material with vanadium diboride VB_2 during filtration refining;
- Experimental studies of filtration refining of aluminum treated with boric acid (H_3BO_3) in a casting ladle of aluminum melt.

Before conducting the experiments, flux refining of raw aluminum with boric acid has been carried out. The chemical composition of raw aluminum is presented in Table II. Boric acid (H_3BO_3) has been used as a refining flux. Raw aluminum with an increased vanadium content, taken from individual electrolyzers operating on baked anodes made of petroleum coke with an increased vanadium content (manufactured by UPNK-PV LLC), has been melted in a GW-MF-25 crucible induction furnace.

Raw aluminum has been provided by Kazakhstan Electrolysis Plant JSC.

TABLE II
AVERAGE CHEMICAL COMPOSITION OF RAW ALUMINUM, %

Mass fraction of elements, %									
Si	Fe	Cu	Mn	Mg	Ni	Cr	Ti	V	Al
3,2557	0,4105	0,0071	0,0032	0,0239	0,0115	0,001	0,0323	0,0132	main

Boric acid has been introduced below the metal level at a temperature of 850 °C. The temperature has been measured with an immersion thermocouple (chromel-alumel, type K). Boric acid powder has been preliminarily wrapped in aluminum foil and heated to 165 °C. The mass of boric acid has been taken at the rate of 1.2 - 2 kg per ton of raw aluminum. This method of loading flux into the melt has ensured high reactivity of raw aluminum and flux. As a result of the chemical reaction, bubbling on the surface of the molten metal has been visually observed.

Then the metal has been held for 15 minutes, and subsequently poured into a ladle heated to 750 °C. The processing time has been selected based on the calculation of minimizing the transportation time of the vacuum ladle from the aluminum electrolysis shop to the foundry department. The chemical composition of raw aluminum samples at all stages of the experiments has been measured using a DFS-500 optical emission spectrometer. After the aluminum melt has been released from the induction furnace, the metal has been sent for filtration refining. A bulk volume filter with granules of 15 - 25 mm in size has been selected for filtration refining. Ash from Ekibastuz coal, consisting mainly of oxides of various metals (Tables III and IV), has been chosen as the material for the manufacture of the bulk filter. Ligosulfonate has been used as a binder.

III. Results and Discussion

At the first stage of the work, a thermodynamic analysis of the possibility of chemical interaction of vanadium diboride VB_2, formed during flux refining of primary aluminum with minerals present in the ash of Ekibastuz coal (Table II) has been carried out.

Fig. 1. General view of a bulk granular filter

TABLE III
AVERAGE CHEMICAL COMPOSITION OF RAW ALUMINUM AFTER FLUX REFINING WITH BORIC ACID, %

SiO_2	Al_2O_3	SO_2	FeO, Fe_2O_3	MgO	TiO_2	CaO	K_2O	BaO	MnO	V_2O_5
51,3	23,2	2,67	4,47	2,3	1,01	4,35	0,78	0,17	0,11	0,01

TABLE IV
AVERAGE CHEMICAL COMPOSITION OF RAW ALUMINUM AFTER FLUX REFINING WITH BORIC ACID, %

Glass phase	Amorphized clay substance	Iron oxides	Feldspar, quartz, pyroxene	Corundum, mullite, cristobalite	Calcite	Carbonaceous particles
30	25	9	10	7	8	11

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For thermodynamic analysis, the HSC Chemistry 9.0 software from Outotec Technologies has been used. The studied temperature range has corresponded to the production conditions of JSC "Kazakhstan Electrolysis Plant" and has been within 650 - 950 °C. The pressure has been taken within 102.39 kPa. Of all the chemical compounds presented in Table III, in this temperature range, a reaction is possible only with iron oxides (FeO):

$$2FeO + VB_2 = 2FeB + VO_2 \quad (1)$$

Table V and Figure 2 show the calculation of the Gibbs energy in the temperature range from 650 to 950 °C for formula (1). From the data in Figure 2, it can be noted that the calculated values of ΔG for the chemical reaction $2FeO + VB_2 = 2FeB + VO_2$ in the range of operating temperatures of the electrolysis and casting of primary aluminum are within the range of - 92.973 - 99.574 kJ/mol, and with a decrease in temperature from 950 °C to 650 °C the value of ΔG decreases, which increases the probability of the reaction of iron oxides and vanadium diboride. Further, as known from [19]-[23], at a temperature of 500 - 600 °C, VO_2 is converted into V_2O_5 according to the formula:

$$4VO_2 + O_2 = 2V_2O_5 \quad (2)$$

Thus, thermodynamic analysis shows the possibility of implementing the reaction-crystallization mechanism of aluminum filtration by a filter from Ekibastuz coal ash due to the chemical interaction of vanadium diboride impurities on the surface of iron oxides included in the composition of Ekibastuz coal ash. At the second stage of the work, experimental studies of filtration refining of aluminum treated with boric acid (H_3BO_3) from vanadium diborides have been carried out. For the experiments, three metal samples have been taken from metal smelting with flux refining with boric acid. The chemical composition of aluminum before filtration refining is given in Table VI.

The study has been conducted on a setup that has included a filter unit 1, made in the form of a steel cup with a diameter of 12 cm and a height of 20 cm, covered with a refractory material. At the bottom of the cup, there has been a hole with a diameter of 6 cm, through which the filtered alloy has flowed out. A fiberglass mesh with a cell size of 2.5 × 2.5 mm has been used to close the hole.

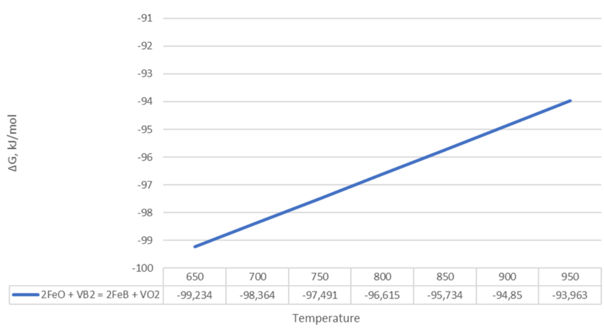
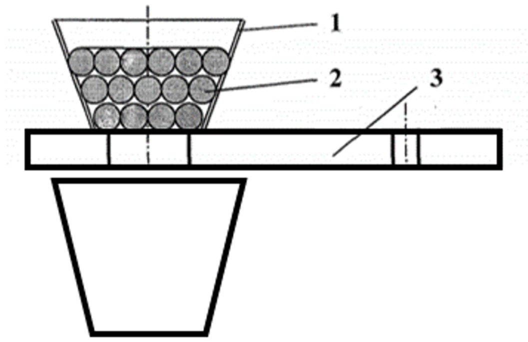


Fig. 2. Calculation of the Gibbs energy for the chemical reaction $2FeO + VB_2 = 2FeB + VO_2$ in the temperature range from 650 to 950 °C



1 - filter block; 2 - filter grains; 3 – damper

Fig. 3. Schematic diagram of the setup for studying the filtration of aluminum alloy

The calculation of the installation has been carried out according to the generally accepted calculation method for gating-feeding systems, by taking into account the features associated with the installation of filters [20].

The calculated dimensions of the gating-feeding system are: area of the filter block Fф = 0,009 m^2, filter block diameter dф = 0,12 m. These dimensions have been adopted for the design of the laboratory setup. For the experiments, filter grains 2 have been placed in filter block 1 above damper 3 (Figure 3). In order to prevent the floating of filter grains made of Ekibastuz coal ash (ash density is 2100 kg/m^3), steel grinding balls with a diameter of 40 mm coated with refractory material (Ekibastuz coal ash with a binder in the form of lignosulfonate) with a density of 7800 kg/m^3 have been placed on top. Before using the filter, it has been heated to a temperature of 400 - 500 °C. The raw aluminum melt previously treated with boric acid at a temperature of 720-760 °C has been poured into the filter block, while a constant metallostatic pressure of 60 mm has been maintained. At the same time, the duration of metal retention above the filter and the filtration time have been measured. Next, the chemical composition of aluminum has been determined by using a DFS-500 optical emission microscope (Table VII).

According to the data in Table VII, it can be noted that the vanadium content in the filtered metal samples has significantly decreased to values of 0.0003 - 0.0012%.

The obtained samples of filtered aluminum have been subjected to microstructural analysis on a JSM-6390 LV electron microscope from JEOL ltd. Figure 5 and Table VIII show the results of scanning electron microscopy (SEM) of a raw aluminum sample after filtration refining.

Fig. 4. Samples of filtered raw aluminum

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TABLE V
GIBBS ENERGY IN THE TEMPERATURE RANGE FROM 650 TO 950 °C

Element	Parameter	650 °C	700 °C	750 °C	800 °C	850 °C	900 °C	950 °C
	H	-115,27	-115,31	-115,38	-115,46	-115,54	-115,63	-115,73
	S	-17,37	-17,42	-17,49	-17,56	-17,64	-17,71	-17,79
2FeO + VB2 = 2FeB + VO2	G	-99,23	-98,36	-97,49	-96,61	-95,73	-94,85	-93,96
	Log K	5,61	5,28	4,97	4,70	4,45	4,45	4,01
	H	-115,27	-115,32	-115,38	-115,46	-115,54	-115,63	-115,73

TABLE VI
AVERAGE CHEMICAL COMPOSITION OF RAW ALUMINUM AFTER FLUX REFINING WITH BORIC ACID, %

№ of melt	Si	Fe	Cu	Mn	Mg	Ni	Cr	Ti	V	B
1	0,3883	0,4105	0,0070	0,0016	0,0205	0,0114	0,0010	0,0258	0,0055	0,0021
2	0,1116	0,4095	0,0021	0,0017	0,0106	0,0114	0,0009	0,0166	0,0058	0,0041
3	1,2573	0,4105	0,0069	0,0029	0,0164	0,0153	0,0010	0,0036	0,0069	0,0057

TABLE VII
CHEMICAL COMPOSITION OF ALUMINUM AFTER FILTRATION REFINING, %

№ of melt	Si	Fe	Cu	Mn	Mg	Ni	Cr	Ti	V	B
1.3	0,3883	0,2881	0,3195	0,0069	0,0013	0,0165	0,0112	0,0011	0,0243	0,0003
2.3	0,1116	0,1013	0,4014	0,0019	0,0012	0,0096	0,0111	0,0009	0,0156	0,0009
3.3	1,2573	1,1974	0,3093	0,0066	0,0023	0,0069	0,0149	0,0009	0,0027	0,0012

According to the data in Figure 5 and Table VIII, vanadium has not been observed in the samples of filtered metal. Later, a chemical analysis of the spent filter grains has been carried out (Figure 6). The studies have been carried out on a portable X-ray fluorescence analyzer Prospector 2. Metal beads of metal have been preliminarily separated from the grains. As shown in Table IX, the content of V_2O_5 oxide in the filter grains has been 0.1%. This indicates the process of filtering the metal from vanadium diboride impurities, most likely on the surface of iron oxides, which are part of the Ekibastuz coal ash.

TABLE VIII
CHARACTERISTICS OF CHEMICAL ELEMENTS BASED ON THE RESULTS OF SCANNING ELECTRON MICROSCOPY OF A SAMPLE OF RAW ALUMINUM AFTER FILTRATION REFINING, %

Spectrum	O	Al	Si	V	Ca	Fe	Summary
Spectrum 1	35,45	49,41	11,70	-	3,44	-	100,00
Spectrum 2	48,96	25,13	6,51	-	19,40	-	100,00
Spectrum 3	7,33	23,59	69,08	-	-	-	100,00
Spectrum 4	6,27	91,18	1,74	-	-	0,81	100,00
Max.	48,96	91,18	69,08	-	19,40	0,81	
Min.	6,27	23,59	1,74	-	3,44	0,81	

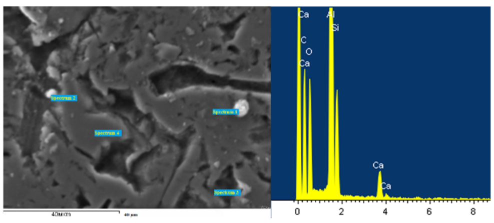

Fig. 5. Results of scanning electron microscopy of a raw aluminum sample after filtration refining

Fig. 6. Spent grains of a bulk filter

TABLE IX
CHEMICAL COMPOSITION OF SPENT FILTER GRAINS, %

SiO_2	Al_2O_3	SO2	Fe_2O3	MgO	TiO2	CaO	K_2O	BaO	MnO	V_2O_5
63.5	28,1	2,66	2,24	1,3	0,87	0,64	0,41	0,14	0,06	0,1

IV. Conclusion

Based on the results of the conducted research, the following conclusions can be made. Thermodynamic modeling with HSC Chemistry 9.0 has showed that among the minerals of Ekibastuz coal ash, only iron oxides can chemically interact with vanadium diboride (VB_2). This allows for the implementation of reaction-crystallization filtration of metal from VB_2 impurities on their surface.

An experiment on filtration purification of aluminum after treatment with boric acid showed a decrease in the vanadium content to 0.0003-0.0012%. Electron microscopy has confirmed the absence of vanadium in the purified metal, and the analysis of the filter surface has revealed the presence of vanadium oxide (V_2O_5), which confirms the efficiency of filtration. Further ways to improve this technology may include reducing the melt processing time by combining two stages (metal purification using a refining agent and filtration). The essence of the method will consist in processing the aluminum melt by creating an active surface on the filter granules in order to ensure the best implementation of the reaction-crystallization filtration mechanism.

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