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Scanning Electron Microscopy of Primary Aluminum Refined With Boric Acid

[1]***BYKOV Petr,** *Cand. of Tech. Sci., Professor, Bykov_petr@mail.ru,*
[1]***KUANDYKOV Almaz,** *doctoral student, azeka200892@mail.ru,*
[2]***CHAIKIN Vladimir,** *Dr. of Tech. Sci., Professor, sro_ral@mail.ru,*
[1]***ZHUNUSOV Ablai,** *Cand. of Tech. Sci., Professor, zhunusov_ab@mail.ru,*
[1]***SUYUNDIKOV Merhat,** *Cand. of Tech. Sci., Professor, suyundikovm@mail.ru,*
[1]*NCJSC «Toraighyrov University», Kazakhstan, Pavlodar, Lomov Street, 64,*
[2]*Nosov Magnitogorsk State Technical University, Russia, Magnitogorsk, Lenin Avenue, 38,*
**corresponding author.*

Abstract. *In the work, using scanning electron microscopy, samples of raw aluminum obtained using a complex technology of refining by flux refining with boric acid (H_3BO_3) in a ladle with further filtration were studied. In recent years, there has been a tendency to involve in the production of lower quality sources of raw materials for the production of anodes, which leads to an increase in impurities in aluminum. This is mainly due to the production of heavy oil (which includes asphaltene compounds). In asphaltenes, metal impurities (Fe, Si, V, Ni, etc.) are concentrated, which, during coking, turn into coke, and then into aluminum. An undesirable impurity in primary aluminum is vanadium, which reduces the electrical conductivity of the metal at a concentration of about 2 ppm. The authors of the work carried out experimental studies on the filtration refining of aluminum melt after flux treatment with boric acid (H_3BO_3). Detailed studies on a JEOL scanning electron microscope with an INCA Energy microanalysis system showed a decrease in the content of vanadium impurities in the process of flux refining with boric acid and further filtration through a grain filter.*

Keywords: *aluminum, metallic impurities, refining, vanadium, scanning electron microscopy, metallurgy, filtration, non-ferrous metallurgy, flux, purification.*

Introduction

In recent years, an aluminum cluster has formed in Northeast Kazakhstan, including the production of alumina, aluminum, and engineering products [1]. The scientists of Kazakhstan are developing their own technologies for the further development of the aluminum cluster [2-10].

In recent years, there has been a tendency to involve in the production of lower quality sources of raw materials for the production of anodes, which leads to an increase in impurities in aluminum. This is mainly due to the production of heavy oil (which includes asphaltene compounds). Metal impurities (Fe, Si, V, Ni, etc.) are concentrated in asphaltenes, which pass into coke and then into aluminum during coking [11, 12]. An undesirable impurity in primary aluminum is vanadium, which reduces the electrical conductivity of the metal at a concentration of about 2 ppm [12].

It is known [13] that the main sources of vanadium in primary aluminum are (g/ton Al): alumina (up to 24%), anode mass (up to 33%), cryolite (2%). The amount of vanadium impurities can reach 29.0 g/ton Al.

In a number of works [14-15], methods for refining primary aluminum from vanadium impurities have been proposed, which have not found wide industrial application.

In [14], the authors studied the technology of refining primary aluminum from vanadium impurities with a boron-containing alloy Al-B outside the electrolysis bath.

Studies have shown [13]:

- decrease in the content of vanadium by an average of 78% in the bulk of the metal, with an increase in its content in volume up to 5-10% of the ladle capacity;
- averaging the content of vanadium in the volume of the bucket with stirring;
- conversion of a significant amount of vanadium into an intermetallic compound;
- difficulty in separating vanadium and refined aluminum intermetallic compounds in a ladle using traditional methods (settling for 4-7 hours).

The authors in [12] carried out comparative experimental studies on the refining of raw aluminum with boric acid (H_3BO_3) and Al-B ligature, which showed similar results, the advantage of using boric acid (H_3BO_3) in the refining of raw aluminum was also substantiated, due to its availability. and cheap compared to Al-B ligature.

Thus, the solution of the issue of successful removal of vanadium intermetallic compounds from already refined aluminum is of great importance for improving the quality of primary aluminum.

Field Study and Results

In this work, experimental studies were carried out on the filtration refining of aluminum melt after flux treatment with boric acid (H_3BO_3).

The primary chemical composition of raw aluminum is presented in table 1.

Experimental studies were carried out as follows. First, raw aluminum was melted in a laboratory induction furnace, H_3BO_3 was introduced at a temperature of 850°C at the rate of 1.2-2 kg/t of raw aluminum, then the melt was held for 15 minutes and the chemical composition of refined raw aluminum was determined on a DFS optical emission spectrometer –500. It was found that the content of vanadium in the melt decreased to 0.00565%. Vanadium in the metal is in the form of intermetallic compounds VB2.

At the second stage of laboratory experiments, filtration refining of raw aluminum treated with boric acid through a granular filter was carried out.

The layout of the experimental setup was similar to that in [15] and consisted of a filter unit 1, filter grains 2, and mold 3; in the lower part, there was an opening for the outflow of the filtered metal, which was covered with a refractory mesh.

Ash from Ekibastuz coal, consisting mainly of SiO_2, Al_2O_3, was used as a material for the manufacture of filter grains.

The variable parameters in experimental studies were: filter grain heating temperature (°C), filter grain size (mm), filter layer height (mm).

As a result of the experiments, filtered aluminum samples were obtained, which were analyzed using a scanning electron microscope (SEM).

As a result of the experiments, filtered aluminum samples were obtained, which were further analyzed on a JEOL scanning electron microscope with an INCA Energy microanalysis system.

Based on the results of electron microscopy, photographs of inclusions were obtained, the chemical composition of samples of the initial raw aluminum, metal samples after H_3BO_3 flux treatment and after filtration was determined.

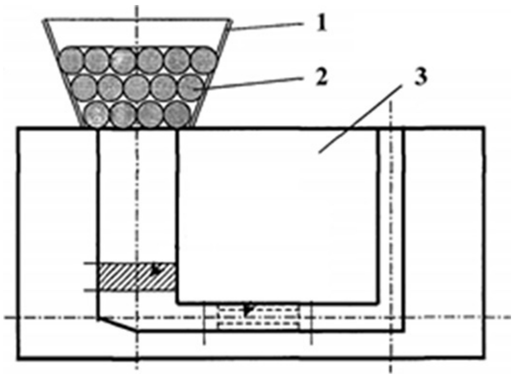
Figure 3 and Table 3 show the results of scanning electron microscopy (SEM) of the original sample of raw aluminum.

Figure 4 and Table 4 show the results of scanning electron microscopy (SEM) of a raw aluminum sample after flux refining.

Figure 5 and Table 5 show the results of scanning

Table 1 – Chemical composition of raw aluminum, %

Al	Si	Fe	Cu	Mn	Mg	Ni	Cr	Ti	V
96,1299	3,2557	0,4105	0,0071	0,0032	0,0239	0,0115	0,001	0,0323	0,0132



a

b

1 – filter block; 2 – filter grains; 3 – metal form

Figure 1 – Scheme (a) and top view (b) of the installation for studying the filtration of an aluminum alloy [15]

Table 2 – Filter options

Heating temperature, °C	Layer Fill Height, mm	Filter grain size, mm
300	20	15
400	30	20
500	40	25

electron microscopy (SEM) of a raw aluminum sample after filtration refining.

Figure 2 – Samples of filtered raw aluminum

The discussion of the results

The authors studied the process of complex refining of raw aluminum from vanadium using boric acid (H_3BO_3) in a ladle with further filtration refining.

Laboratory studies have shown a decrease in the content of vanadium in the melt after flux refining from 0.0132% to 0.00565% (on average by 42.8%) and almost complete removal of vanadium according to the results of filtration.

Detailed studies on a JEOL scanning electron microscope with an INCA Energy microanalysis system showed the following results:

- the presence of vanadium in the form of inclusions in certain areas of the raw aluminum sample before refining with a vanadium content of 0.53% (Figure 3, spectrum 1);

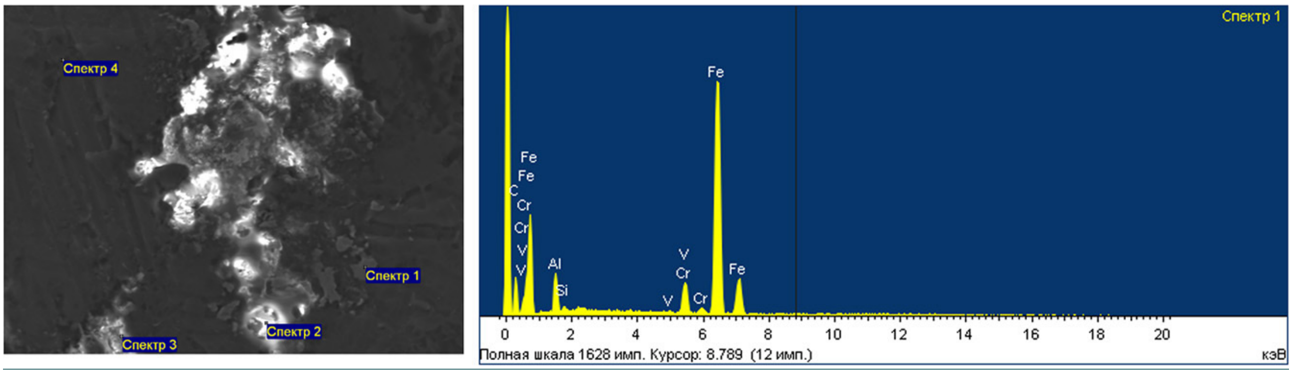


Figure 3 – The results of scanning electron microscopy of the original sample of raw aluminum (before refining)

Table 3 – Characteristics of chemical elements according to the results of scanning electron microscopy of the initial sample of raw aluminum (before refining), %

Range	In stat.	O	Al	Si	V	Cr	Fe	Total
Range 1	Yes	-	5,71	0,63	0,53	6,44	86,69	100,00
Range 2	Yes	21,03	78,97	-	-	-	-	100,00
Range 3	Yes	15,28	84,72	-	-	-	-	100,00
Range 4	Yes	30,74	69,26	-	-	-	-	100,00
Max.		30,74	84,72	0,63	0,53	6,44	86,69	
Min.		15,28	5,71	0,63	0,53	6,44	86,69	

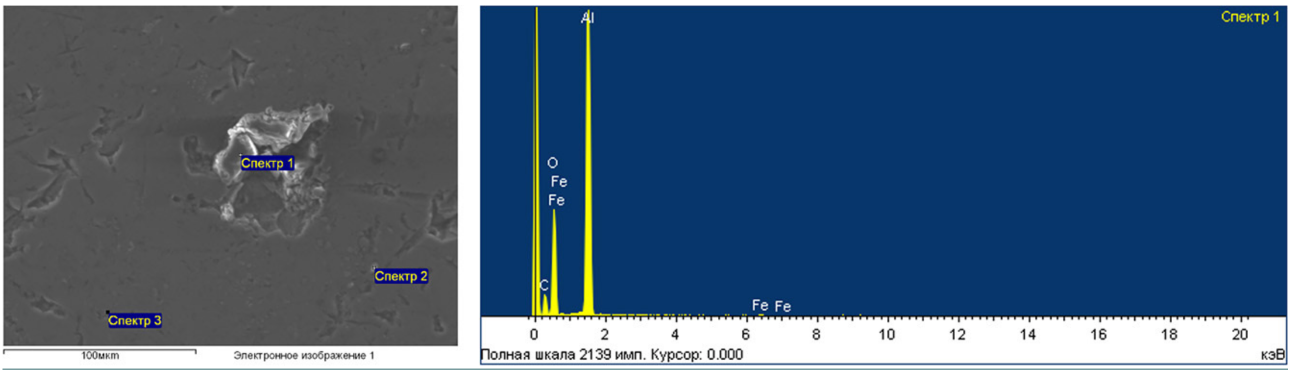


Figure 4 – The results of scanning electron microscopy of a raw aluminum sample after flux refining

Table 4 – Characteristics of chemical elements according to the results of scanning electron microscopy of a raw aluminum sample after flux refining, %

Range	In stat.	O	Al	Si	V	Cr	Fe	Total
Range 1	Yes	51,77	47,50	-	-	-	0,73	100,00
Range 2	Yes	52,21	47,79	-	-	-	-	100,00
Range 3	Yes	11,59	77,31	0,83	-	-	8,65	100,00
Max.		52,21	77,31	0,83	-	-	8,65	
Min.		11,59	47,50	0,83	-	-	0,73	

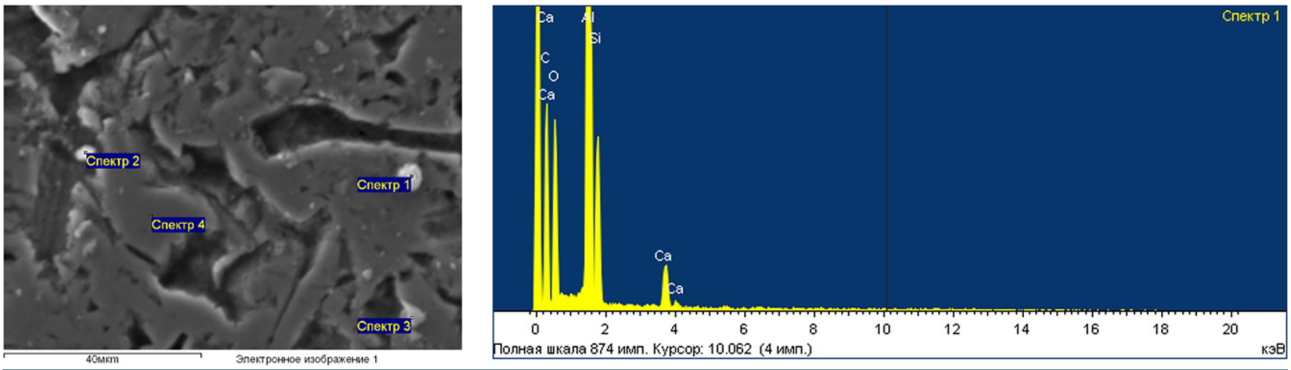


Figure 5 – The results of scanning electron microscopy of a raw aluminum sample after filtration refining

Table 5 – Characteristics of chemical elements according to the results of scanning electron microscopy of a raw aluminum sample after filtration refining, %

Range	In stat.	O	Al	Si	V	Ca	Fe	Total
Range 1	Yes	35,45	49,41	11,70	-	3,44	-	100,00
Range 2	Yes	48,96	25,13	6,51	-	19,40	-	100,00
Range 3	Yes	7,33	23,59	69,08	-	-	-	100,00
Range 4	Yes	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	

- no inclusions of vanadium were found on the samples after flux refining and filtration (Figures 4, 5).

The degree of vanadium removal depending on the filter parameters is as follows: with an increase in the height of the filter layer and a decrease in grain size, the degree of vanadium removal increases, and the metal temperature within the specified limits does not, in principle, affect the degree of vanadium removal.

Thus, it can be concluded that the complex technology for refining raw aluminum from vanadium using boric acid (H_3BO_3) in a ladle with further filtration makes it possible to remove vanadium from raw aluminum.

Conclusions

1) An integrated technology for refining raw aluminum from vanadium using boric acid (H_3BO_3) in a ladle with further filtration was experimentally studied, which showed the possibility of almost complete removal of vanadium.

2) Scanning electron microscopy of the original raw aluminum, metal samples after H_3BO_3 flux treatment and after filtration showed:

- the presence of vanadium in the form of inclusions in certain areas of the raw aluminum sample before refining with a vanadium content of 0.53%;

- no inclusions of vanadium were found on the samples after flux refining and filtration.

3) The degree of vanadium removal depending on the filter parameters is as follows: with an increase in the height of the filter layer and a decrease in grain size, the degree of vanadium removal increases, the temperature of the metal within the specified limits does not in principle affect the degree of vanadium removal.

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Бор қышқылымен тазартылған бастапқы алюминийдің растрлық электронды микроскопиясы

[1]***БЫКОВ Петр Олегович,** т.ғ.к., профессор, Bykov_petr@mail.ru,*

[1]***КУАНДЫКОВ Алмаз Болатович,** докторант, azeka200892@mail.ru,*

[2]***ЧАЙКИН Владимир Андреевич,** т.ғ.д., профессор, sro_ral@mail.ru,*

[1]***ЖУНУСОВ Аблай Каиртасович,** т.ғ.к., профессор, zhunusov_ab@mail.ru,*

[1]***СУЮНДИКОВ Мерхат Мадениевич,** т.ғ.к., профессор, suyundikovm@mail.ru,*

[1]*«Торайғыров университеті» КеАҚ, Қазақстан, Павлодар, Ломов көшесі, 64,*

[2]*Носов атындағы Магнитогорск мемлекеттік техникалық университеті, Ресей, Магнитогорск, Ленин даңғылы, 38,*

**автор-корреспондент.*

Аңдатпа. *Жұмыста электронды растрлық микроскопия әдісімен шөміште бор қышқылымен (H_3BO_3) қождауышпен тазалаудың кешенді технологиясын қолдана отырып, одан әрі сүзгілей арқылы алынған алюминий-шикізаты үлгілері зерттелді. Соңғы жылдары анод өндірісінде сапасы төмен шикізат көздерін пайдалану, оларды өндіріске тарту үрдісі байқалады, бұл алюминийдегі қоспалардың көбеюіне әкеледі. Бұл негізінен ауыр мұнай өндіруге байланысты (оның құрамына асфальттендер қосылыстары кіреді). Металл қоспалары (Fe, Si, V, Ni және т.б.) асфальтендерде шоғырланған, олар кокстелген кезде коксқа, содан кейін алюминийге өтеді. Бастапқы алюминийдегі қажетсіз қоспа ванадий болып табылады, ол шамамен 2 ppm концентрациясында металдың электр өткізгіштігін төмендетеді. Жұмыстың авторларымен бор қышқылымен (H_3BO_3) қождауышпен өңдеуден кейін алюминий балқымасын сүзгілеп тазарту бойынша эксперименттік зерттеулер жүргізілді. INCA Energy микроанализ жүйесі бар JEOL фирмасының растрлық электронды микроскопындағы толық зерттеулер алюминий балқымасын бор қышқылымен қождауышпен тазарту және түйіршікті сүзгі арқылы одан әрі сүзу процесінде ванадий қоспаларының азаюын көрсетті.*

Кілт сөздер: *алюминий, металл қоспалары, өңдеу, ванадий, сканерлеуші электронды микроскопия, металлургия, сүзу, түсті металлургия, флюс, тазарту.*

Растровая электронная микроскопия первичного алюминия, рафинированного борной кислотой

[1]***БЫКОВ Петр Олегович,** к.т.н., профессор, Bykov_petr@mail.ru,*
[1]***КУАНДЫКОВ Алмаз Болатович,** докторант, azeka200892@mail.ru,*
[2]***ЧАЙКИН Владимир Андреевич,** д.т.н., профессор, sro_ral@mail.ru,*
[1]***ЖУНУСОВ Аблай Каиртасович,** к.т.н., профессор, zhunusov_ab@mail.ru,*
[1]***СУЮНДИКОВ Мерхат Мадениевич,** к.т.н., профессор, suyundikovm@mail.ru,*
[1]*НАО «Торайгыров университет», Казахстан, Павлодар, ул. Ломова, 64,*
[2]*Магнитогорский государственный технический университет имени Носова, Россия, Магнитогорск, пр. Ленина, 38,*

**автор-корреспондент.*

***Аннотация.** В работе методом растровой электронной микроскопии исследованы образцы алюминия-сырца, полученные с применением комплексной технологии рафинирования флюсовым рафинированием борной кислотой (H_3BO_3) в ковше с дальнейшей фильтрацией. В последние годы наметилась тенденция вовлечения в производство менее качественных источников сырья для производства анодов, что приводит к увеличению примесей в алюминии. В основном это связано с добычей тяжелой нефти (в состав которой входят соединения асфальтенов). В асфальтенах концентрируются примеси металлов (Fe, Si, V, Ni и др.), которые при коксовании переходят в кокс, а затем и в алюминий. Нежелательной примесью в первичном алюминии является ванадий, снижающий при концентрации около 2 ppm электропроводность металла. Авторами работы были проведены экспериментальные исследования по фильтрационному рафинированию расплава алюминия после флюсовой обработки борной кислотой (H_3BO_3). Детальные исследования на растровом электронном микроскопе фирмы JEOL с системой микроанализа INCA Energy показали снижение содержания примесей ванадия в процессе флюсового рафинирования борной кислотой и дальнейшей фильтрации через зерновой фильтр.*

***Ключевые слова:** алюминий, металлические примеси, переработка, ванадий, сканирующая электронная микроскопия, металлургия, фильтрация, цветная металлургия, флюс, очищение.*

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