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Satbayev University

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[1]M.Auezov South Kazakhstan university, Shymkent, Kazakhstan;
[2]National Center on Complex Processing of Mineral Raw Materials of the Republic of Kazakhstan, Almaty, Kazakhstan;
[3]Satbayev University, Almaty, Kazakhstan.
E-mail: aigerim_nurpeis@mail.ru

THERMODYNAMIC PREDICTION AND EXPERIMENTAL PRODUCTION OF SILICON ALLOYS FROM TAILINGS LEACHING OF OXIDIZED COPPER ORE ALMALY

Nurpeisova Aigerim Muratbaevna — Master of Technical Sciences, doctoral student of the departmentof Metallurgy of M. Auezov University of South Kazakhstan. Shymkent, Kazakhstan
E-mail: aigerim_nurpeis@mail.ru, https://orcid.org/0000-0001-8494-7872;
Shevko Viktor Mikhailovich — Doctor of Technical Sciences. Professor. Head of the Department of Metallurgy of M.Auezov South Kazakhstan University. Shymkent, Kazakhstan
E-mail: shevkovm@mail.ru, https://orcid.org/0000-0002-9814-6248;
Aitkulov Dosmurat Kyzylbievich — Doctor of Technical Sciences. Professor. Director of the National Research Center for Integrated Processing of Mineral Raw Materials of the Republic of Kazakhstan, Almaty, Kazakhstan
E-mail: aitkulov_dk@mail.ru, https://orcid.org/0000-0003-2571-6710;
Joldassov Aidos Abdisalimovich — Master of technical science, Satbayev University, Almaty, Kazakhstan
E-mail: jaydos@mail.ru, https://orcid.org/0000-0002-4740-2352.

Abstract. The article presents the results of studies on the production of silicon-containing ferroalloys from the tailings of sulfuric acid leaching of oxidized copper ore from the Almaly deposit. The studies were carried out by the method of thermodynamic modeling using the HSC — 6.0 rototable second-order planning complex (Box – Hunter plan) and electric charge melting in an arc single-electrode furnace. The influence of temperature, carbon, and charge composition on the production of siliceous alloys was determined. It was found that under equilibrium conditions from the tailings in the temperature range 1903–in the presence of 52–66 % carbon and 24 % iron, it is possible to form ferrosillicoaluminium grade FeSi45A10 at 1753–1875^{0}C and 34-66% carbon ferrosillium grade FeSi45, and in the temperature range 1796–1950^{0}C, 34–66 % carbon Fe-Si-Al ligature containing 2,0–7,5 Al and 45,9–47,5 % Si. During electric melting of charges containing 52–54 % tailings, 25–28 % coke and 20-21% steel chips of ferrosillium grade FeSi45 (41,3 % Siand 1,3 % Al) and ferrosillicoaluminium grade FeSi45A10 (43,4 % Siand 7,4 % Al) with extractions to the alloy up to 79,8 % Siand 50,8 % Al.

Keywords: oxidized copper ore, leaching tailings, thermodynamic prediction, electric melting, ferrosilicon, ferrosilicoaluminium, Fe-Si-Al ligature

© В.М. Шевко[1], А.М. Нурпеисова[1*], Д.К. Айткулов[2], А.А. Жолдасов[3], 2023
[1]М. Әуезоватындағы Оңтүстік Қазақстан университеті, Шымкент, Қазақстан;
[2]Қазақстан Республикасының минералдық шикізатты кешенді қайта өңдеу жөніндегі ұлттық орталығы, Алматы, Қазақстан;
[3]Сәтбаев университеті, Алматы, Казахстан.
E-mail: aigerim_nurpeis@mail.ru

АЛМАЛЫ ТОТЫҚҚАН МЫС КЕНІН ШАЙМАЛАУ ҚАЛДЫҚТАРЫНАН КРЕМНИЙЛІ ҚОРЫТПАЛАРДЫ ТӘЖІРИБЕЛІ АЛУ ЖӘНЕ ТЕРМОДИНАМИКАЛЫҚ БОЛЖАУ

Нурпейсова Айгерім Муратбаевна — Техника ғылымдарының магистрі, М. Әуезов атындағы Оңтүстік Қазақстан университетінің Металлургия кафедрасының докторанты. Шымкент, Қазақстан
E-mail: aigerim_nurpeis@mail.ru, https://orcid.org/0000-0001-8494-7872;
Шевко Виктор Михаилович — Техника ғылымдарының докторы, профессор, Оңтүстік Қазақстан университетінің Металлургия кафедрасының меңгерушісі. М. Әуезов. Шымкент, Қазақстан
E-mail: shevkovm@mail.ru, https://orcid.org/0000-0002-9814-6248;
Айткулов Досмурат Кызылбаевич — Техника ғылымдарының докторы, профессор, Қазақстан Республикасы Минералдық шикізатты кешенді қайта өңдеудің Ұлттық зерттеу орталығының директоры, Алматы, Қазақстан

E-mail: aitkulov_dk@mail.ru, https://orcid.org/0000-0003-2571-6710;
Жолдасов Айдос Абдисалимович — Техника ғылымдарының магистрі. Сәтбаев университеті, Алматы, Қазақстан
E-mail: jaydos@mail.ru, https://orcid.org/0000-0002-4740-2352.

Аннотация. Мақалада Алмалы кен орнының күкірт қышқылды сілтісіздендіру қалдықтарынан тотыққан мыс кенін алу жөніндегі зерттеулердің нәтижелері келтіріледі.Зерттеулер HSC – 6,0 екінші ретті рототабельді жоспарлау кешенін (бокс – Хантер жоспары) және доғалы бір электродты пештегі зарядты электрмен балқыту арқылы термодинамикалық модельдеу әдісімен жүргізілді.Кремний қорытпаларын алуға температураның, көміртектің, зарядтың құрамының әсері анықталды. Тепе-теңдік жағдайында 1903-1950^{0}С температура диапазонындағы қалдықтардан 52–66 % көміртегі және 24 % темір болған кезде ФС45А10 маркалы ферросилликоалюминий 1753–1875^{0}С және 34–66 % ФС45 маркалы көміртегі ферросиллийінің пайда болуы мүмкін екендігі, ал температура аймағында 1796-1950^{0}С, 34–66 % Fe-Si-Al көміртегі құрамы бар лигатура 2.0–7.5 Al және 45.9–47.5 % Si болатыны анықталды. Құрамында 52–54 % қалдықтар, 25–28 % кокс және 20–21 % болат жаңқалары ФС45 (41,3 % Si және 1,3% Al) маркалы ферросиллиций және ФС45А10 (43,4 % Si және 7,4 % Al) маркалы ферросилликоалюминий бар шикіқұрамды электрмен балқыту кезінде қорытпаға 79,8 % Si және 50,8 % Al дейін бөлінеді.
Түйін сөздер: тотыққан кен, термодинамикалық моделдеу, электрлі балқыту, ферросилиций, ферросиликоалюминий

© В.М. Шевко[1], А.М. Нурпеисова[1*], Д.К. Айткулов[2], А.А. Жолдасов[3], 2023
[1]Южно-Казахстанский университет имени М. Ауэзова, Шымкент, Казахстан;
[2]Национальный центр по комплексной переработке минерального сырья Республики Казахстан, Алматы, Казахстан;
[3]Сатбаев университеті, Алматы, Казахстан.
E-mail: aigerim_nurpeis@mail.ru

ТЕРМОДИНАМИЧЕСКОЕ ПРОГНОЗИРОВАНИЕ И ЭКСПЕРИМЕНТАЛЬНОЕ ПОЛУЧЕНИЕ КРЕМНИЕВЫХ СПЛАВОВ ИЗ ХВОСТОВ ВЫЩЕЛАЧИВАНИЯ ОКИСЛЕННОЙ МЕДНОЙ РУДЫ АЛМАЛЫ

Нурпейсова Айгерім Муратбаевна — магистр технических наук, докторант кафедры металлургии Южно-Казахстанского университета имени М. Ауэзова. Шымкент, Казахстан
E-mail address: aigerim_nurpeis@mail.ru, https://orcid.org/0000-0001-8494-7872;
Шевко Виктор Михаилович — доктор технических наук, профессор, заведующий кафедрой металлургии Южно-Казахстанского университета им. М.Ауэзова. Шымкент, Казахстан
E-mail address: shevkovm@mail.ru, https://orcid.org/0000-0002-9814-6248;
Айткулов Досмурат Кызылбаевич — доктор технических наук, профессор, директор Национального исследовательского центра комплексной переработки минерального сырья Республики Казахстан, Алматы, Казахстан
E-mail address: aitkulov_dk@mail.ru, https://orcid.org/0000-0003-2571-6710;
Жолдасов Айдос Абдисалимович — Магистр технических наук, университет Сатбаева, Алматы, Казахстан
Email address: jaydos@mail.ru, https://orcid.org/0000-0002-4740-2352.

Аннотация. В статье приводятся результаты исследований по получению кремнийсодержащих ферросплавов из хвостов сернокислотного выщелачивания окисленной медной руды месторождения Алмалы. Исследования проводились методом термодинамического моделирования с использованием комплекса HSC — 6,0 рототoтабельного планирования второго порядка (план Бокса – Хантера) и электроплавкой шихта в дуговой одноэлектродной печи. Определялось влияние температатуры, углерода, состава шихты на получение кремнистых сплавов. Было найдено, что в равновесных условиях из хвостов в температурном интервале 1903–1950^{0}С в присутствии 52–66 % углерода и 24 % железа возможно образование ферросилликоалюминия марки ФС45А10 при 1753–1875^{0}С и 34–66 % углерода ферросиллиций марки ФС45, а в температурном области 1796–1950^{0}С, 34–66 % углерода Fe-Si–Al лигатура с содержанием 2,0–7,5 Al и 45,9–47,5 % Si. При электроплавке шихт, содержащих 52–54 % хвостов, 25–28 % кокса и 20–21 % стальной стружки, получен ферросиллиций марки ФС45 (41,3 % Si и 1,3% Al) и ферросилликоалюминий марки ФС45А10 (43,4 % Si и 7,4 % Al) при извлечении в сплав до 79,8 % Si и 50,8 % Al.
Ключевые слова: окисленная медная руда, хвосты выщелачивания, термодинамическое прогнозирование, электроплавка, ферросилиций, ферросиликоалюминий, Fe-Si-Al лигатура

Introduction

According to the National Center for Integrated Processing of Mineral Raw Materials at the end of the last century, over the past 20–25 years, the share of hard-to-enrich ores has increased by≈3 times, accounting for 45 % of the total mass of enriched mineral raw materials (Poly`vyany`j al 1995: 18). This category includes oxidized and mixed copper ores, whose reserves in Kazakhstan amount to at least 1 billion tons (Zharmenov et al., 2019: 630). For the processing of such a category of ores, their preliminary sulfidation with various substances is proposed (Mitrofanov et al., 1984: 216; Bekturganov et al., 1989: 211; Shevko et al., 2014: 287; Zharmenov et al., 2015: 1; Ory`ngozhin et al., 2020: 4) and then (after sulfidation) use for flotation enrichment. A combined chloride-electrothermal method of processing oxidized and mixed ores to produce ferrosilicon and cathode copper is described in the literature (Kushakova et al., 2010: 31; Robinson et al., 2003: 24). In hydrometallurgy technology, leaching processes are characterized by a significant variety of raw materials (Zharmenov et al., 2015: 1; Ory`ngozhin et al., 2020: 4; Kushakova et al., 2010: 31). Heap leaching, in particular, is used to extract copper from oxidized and mixed ores (Kushakova et al., 2010: 31; Robinson et al., 2003:2 4). However, the most widespread technology for this category of ores is "Heap leaching –SX-EW" (Robinson et al., 2003: 24; ProcessSX/EW//MiningMag 1994: 256; Jenkins et al., Cobre'99 conference, Jenkins 1964: 1094). According to the data of the International Copper Study Group (International Copper Study Group ICSG), the share of commercial copper obtained by this method in 2016 amounted to 16 % of the total world production (Zharmenov et al., 2019: 630). In Kazakhstan, the "Heap leaching –SX-EW" technology has been implemented since 2008 at the Konurad, Almaly, Aktogay, Ayak-Kojan and a number of other fields (Zharmenov et al., 2019: 630) , Kushakova et al., 2013: 343). In 2019, this technology produced ≈ 50 thousand tons of copper in Kazakhstan (≈ 12 % of the total amount of copper produced) (Zharmenov et al., 2019: 630). Despite the rather high extraction of copper from the ore into a copper solution (80–82 %), this method involves the extraction of only copper from the ore. At the same time, the non-metallic component (98–98.5 % of the ore mass) is not used. Therefore, the degree of integrated use of the raw materials of the "Heap leaching –SX-EW" technology is low.

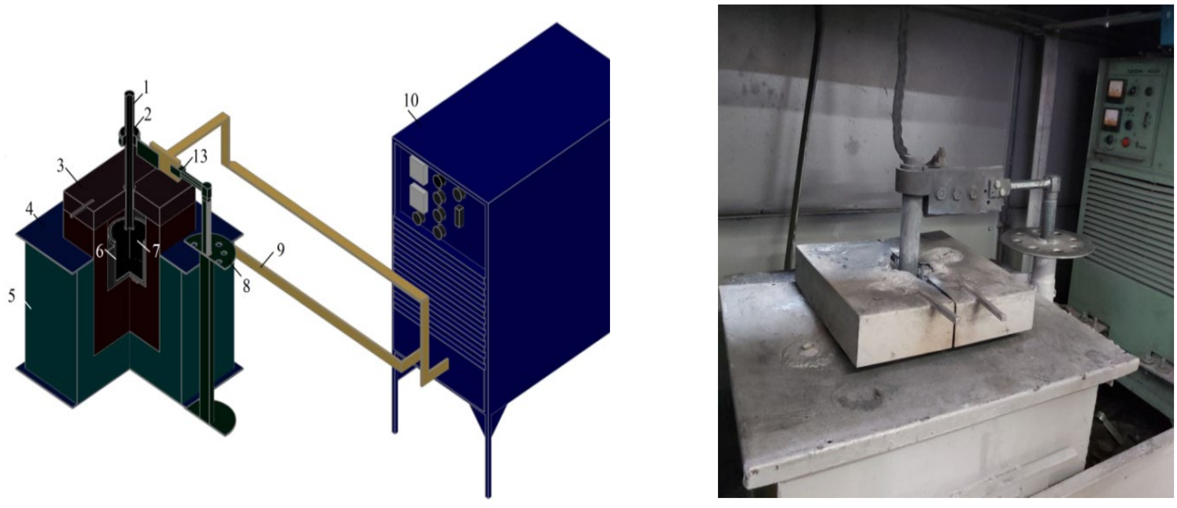
This paper presents the results of studies on the production of ferroalloys from the leaching tailings (TL) of oxidized ores of the Almaly deposit, which contained mass.%: 73,4 % SiO_2, 16.3 % Al_2O_3, 3.1 K_2O, 2.4 % CaO. 1.1 % MgO. 21 % Fe_2O_3, 0.07 Cu. Coke mass content, %: 86,4 C, 4,3 SiO_2, 1.3 CaO, 0.5 MgO, 1.9 Al_2O_3, 2.0 Fe_2O_3, 0.7 S, 1.0 H_2O, 1.2 other, and steel chips 98,7 % Fe, 0.2 %C, 1.1 % other(Si, Mn, Al)

Materials and basic methods

The research was carried out by the method of thermodynamic modeling using the HSC — 6.0 software package based on the Gibbs minimum energy principle (Roin, 2022). Based on the algorithm developed at M. Auezov SKU, we calculated the equilibrium degree of the distribution of elements and the concentration of elements in the alloy (Shevko et al., 2019: 1501). The method of thermodynamic modeling was combined with rotatable planning of the second plan (the Box-Hunter plan) (Axnazarova et al., 1985: 327) with the derivation of regression equations (Inkov et al., 2003) by constructing volumetric and planar images of the behavior of Si, Al and metals in an alloy (Ochkov al., 2009: 512).

Electric melting of TL was carried out on the installation shown in Figure 1. The installation consisted of a single-electrode single-phase electric furnace, a furnace transformer TDJF-1002 with adjustable power, a short network and current and voltage monitoring devices.

III



I-Installation sketch, II-photo of an electric furnace

1 - the electrode; 2- clip; 3-cap; 4 - lining; 5 - furnace casing; 6 - coke; 7 - crucible; 8-the mechanism of moving the electrode, 9 - the tire; 10- transformer

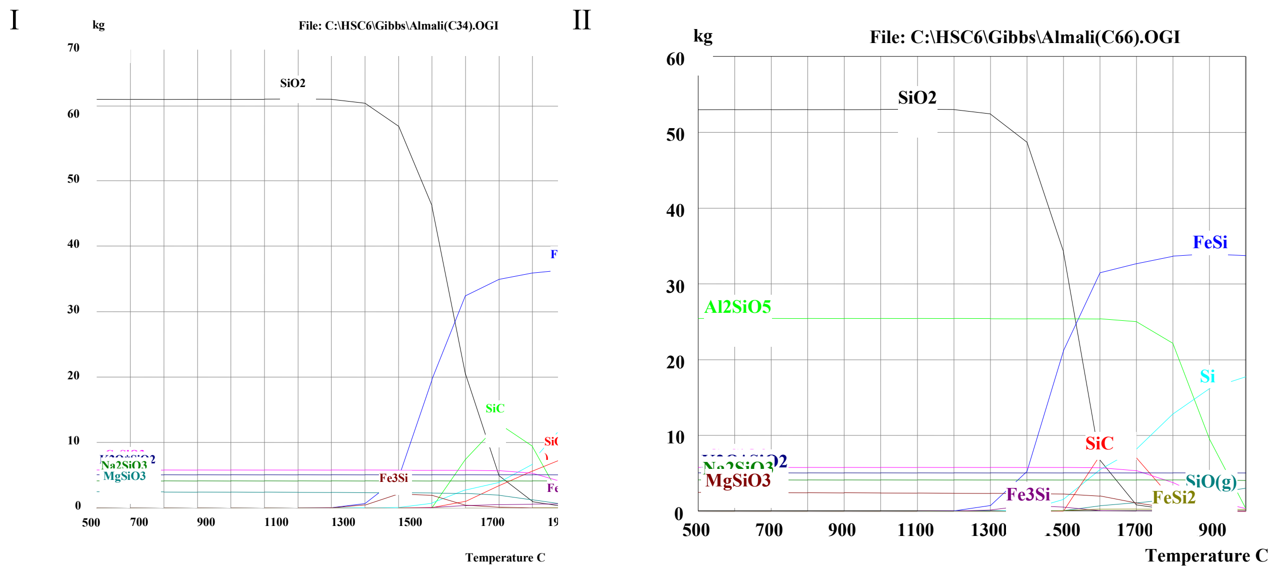
Figure 1- installation for electric melting of copper oxide ore leaching tails

The lining of the electric furnace is chromomagnesite, the base is carbon graphite. Graphite electrode with a diameter of –7 cm. The melting was carried out in a graphite crucible (with an internal diameter of 9 cm and a height of 2 cm). The crucible was installed on a graphite plate. The space between the crucible and the lining was filled with graphite carbon-graphite dust. The lid of the furnace, made of refractory material, was sliding. The vertical movement of the electrode was carried out by a mechanical screw mechanism. The maximum movement of the electrode was 50 cm. The short network-the section from the furnace to the transformer, was made of aluminum tires (1.5x4.5cm). The lower tire was connected to the carbon-graphite block using three copper studs.the upper busbar was connected to the graphite electrode using a flexible copper cable. Rated power of the furnace transformer to 56. The voltage and current on the low side were controlled by a voltmeter and an ammeter of the brands respectively TENGEN 42L6 GB/T7676–1998, CHNT 4226 (China) (accuracy class 1.5) Before melting, the leaching tails were granulated with 2–3 % bentonite clay, dried at150–160 ^{0}C, and then fired at 600–700 ^{0}C. The fired pellets had a diameter of 1–1.5 cm. Coke was also crushed to a fraction of 1–1.5 cm. The size of the steel chips did not exceed 1 cm. The furnace was heated for 3–3.5 hours by an arc at a current of 400–500A and a voltage of 40–50 V. Then the first batch of charge was loaded (600–700 g). It was melted for 5–7 minutes. After that, 2 more portions of the charge were loaded with an interval of 6–8 minutes. The total duration of the melting was 35–45 minutes. During melting, the current strength was 500–600 A. the voltage is 20–30V. After melting, the crucible was cooled in the furnace for 3–4 hours, removed from the furnace, cooled for another 3–3.5 hours in the air. Then the crucible was broken. The contents of the crucible were sorted into alloy and residue.

The elemental composition of the alloy was determined by SEM analysis using a scanning electron microscope of the brand JSM-6490LV (Japan). The degree of extraction of silicon and aluminum in the alloy was determined by the ratio of the mass of the metal in the alloy to the mass of the metal in the charge.

Results

Figure 2 shows the effect of temperature on the equilibrium quantitative (kg) distribution of silicon and aluminum in the TL (100 kg) Fe (24 kg) – C (34–66 kg).



1 – 34 % C, 2 – 66 % C

Figure 2 - influence of temperature and carbon on the equilibrium (kg) quantitative distribution of silicon-containing substances

It can be seen that in the technological field 1200-2000 ^{0}Cthe main products of the interaction of SiO_2 tails with carbon are FeSi, Fe_3Si_2, Si, SiC and SiO_g. Increasing the amount of carbon does not lead to a significant change in the formation of FeSi (Table 1), but increases the formation of silicon and reduces the formation of SiC and SiO_g.

Table 1 – Influence of temperature and carbon on the quantitative (kg) distribution of silicon-containing substances

Substance	Carbon,%	Temperature^{0}C

		1300	1400	1500	1600	1700	1800	1900	2000
FeSi	34	0,67	4,62	19,61	32,43	34,94	35,90	36,38	36,06
	66	0,75	5,24	21,19	31,46	32,66	33,65	33,96	33,76
Si	34	0,01	0,08	0,74	2,75	3,90	6,66	12,55	12,38
	66	0,01	0,17	1,56	5,47	8,21	12,85	16,20	17,76
SiC	34	0,00	0,00	0,00	7,41	12,84	9,41	0,21	0,00
	66	0,00	0,00	0,00	7,34	7,05	1,49	0,00	0,00
SiOg	34	0,00	0,00	0,07	1,02	3,42	5,66	7,73	9,53
	66	0,00	0,00	0,08	0,71	1,12	1,61	2,37	3,05
Fe_3Si	34	0.47	2.15	1.95	0.43	0.13	0.01	0.00	0.00
	66	0.16	0.74	0.55	0.10	0.02	0.00	0.00	0.00

A noticeable formation ofFeSiis noted atT >1300^{0}C, Si – > 1400^{0}C,SiO_g and SiC > 1500^{0}C. Aluminum in the system under consideration begins to recover at a temperature of more than 1700^{0}C. With an increase in carbon, the amount of aluminum formed increases markedly (Table 2)

Table 2 – Influence of temperature and carbon on the quantitative (kg.0) formation of aluminum

Carbon, %	Temperature ^{0}C			
	1700	1800	1900	2000
34	0,02	0,17	1,23	3,75
66	0,02	0,41	3,59	7,80

Further studies were carried out by the method of rotatable planning of the second order (the Box-Hunter plan). The temperature and the amount of carbon (Carbon, % of the weight of the enrichment tailings) were used as independent factors. Optimization parameters are the degree of extraction of silicon and aluminum into the alloy $\alpha_{Si(alloy)}$ and $\alpha_{Al(alloy)}$, % concentration of silicon and aluminum in the alloy — $C_{Si(alloy)}$ and $C_{Al(alloy).}$Table 3 shows the plan and results of the study.

Table 3 – Plan of research and their results for obtaining ferroalloy from copper ore leaching tailings

№	Variables				Technological parameters			
	Coded view		Natural form					
	X_1	X_2	T, ^{0}C	carbon, %	$\alpha_{Si(alloy)}$, %	$\alpha_{Al(alloy)}$, %	$C_{Si(alloy)}$,%	$C_{Al(alloy)}$,%
1	+	+	1921	61,3	88,0	48,3	47,5	7,5
2	+	-	1921	38,7	74,8	26,2	46,3	4,3
3	-	+	1779	61,3	67,6	3,9	44,6	0,4
4	-	-	1779	38,7	56,1	2,0	40,8	0,3
5	1,414	0	1950	50	84,2	52,3	46,4	7,2
6	-1,414	0	1750	50	51,8	3,2	40,8	0,2
7	0	1,414	1850	66	79,1	22,4	48,0	4,5
8	0	-1,414	1850	34	64,8	5,5	43,9	1,3
9	0	0	1850	50	70,0	11,9	46,6	1,7
10	0	0	1850	50	69,7	12,1	47,8	1,6
11	0	0	1850	50	69,5	12,3	46,8	1,9
12	0	0	1850	50	71,1	12,1	47,5	1,8
13	0	0	1850	50	71,3	12,5	47,9	1,5

Based on Table 3 , the following adequate regression equations are obtained:

$$\alpha_{Si(alloy)} = -694,91-0,7T-1,458C+1,5\cdot10^{-5}\cdot T^2+1,03\cdot10^{-2} C^2\ 4,98\cdot10^{-5}\cdot T\cdot C: \quad (1)$$

$$\alpha_{Al(alloy)} =+5248,03-5,6\cdot T-11,69\cdot C+1,49\cdot10^{-4}\cdot T^2+5,1\cdot10^{-3}\cdot C^2+6,3\cdot T\cdot C: \quad (2)$$

$$C_{Si(alloy)} = -1358,05+1,43\cdot T+2,14\cdot C-3,6\cdot10^{-4}\cdot T^2-5,3\cdot10^{-3}\cdot C^2-8,1\cdot10^{-4}\cdot T\cdot C. \quad (3)$$

$$C_{Al(alloy)} = 668,069-0,702\cdot T-2,1\cdot C+1,8\cdot10^{-4}\cdot T^2+4,35\cdot10^{-3}\cdot C^2+9,49\cdot10^{-4}\cdot T\cdot C \quad (4)$$

The adequacy of the equations was determined by the Fisher criterion (F), the values of which are shown in Table (4)

Table 4 – Values of the Fisher criterion

Fisher 's Criterion	Equations			
	$\alpha_{Si(alloy)}$=f(т,c)	$\alpha_{Al(alloy)}$= f(т,c)	$C_{Si(alloy)}$ = f(т, c)	$C_{Al(alloy).}$ = f(т,c)
Calculated	6,476	4,856	1,054	6,029
Tabular	6,59	6,59	6,59	6,59

Forallequations, the$F_{criterionistabular}$>$F_{\text{is the calculation criterion}}$. Hence all the equations are adequate.

Discussion

Volumetric and planar images of the dependences of technological parameters on the temperature of the amount of carbon are shown in Figure 3. It can be seen that a high degree of silicon extraction into the alloy (≥80 %) is noted atT>1850 ^{0}C. More than 50 % of aluminum is extracted into the alloy only at 1915 ^{0}C. The concentration of silicon in the alloy varies from 37 % (at1750 ^{0}C) to 48 % (T=1850-1905 ^{0}C), aluminum from 5 % (at 1865 ^{0}C) to 11 % (at 1950 ^{0}C)

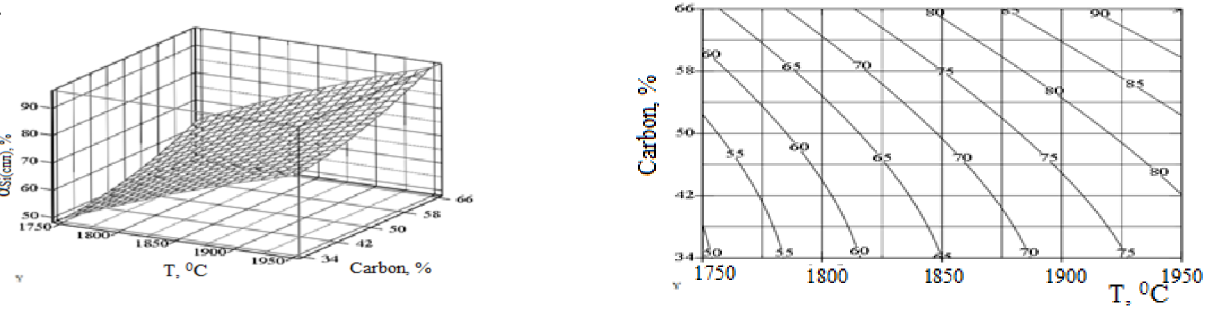
Figure 4 shows the combined information on the effect of temperature and carbon on the technological parameters of obtaining ferroalloy from the leaching tailings of oxidized copper ores.

When constructing the diagram, it was assumed that the minimum degree of transition of silicon to the alloy should be more than 65 %, the concentration of Siin ferrosilicon -41–47 %, the concentration of aluminum in ferrosillicoaluminium-≥7,5 %, and silicon in it ≥47,5 %. Figure 4 shows that in the temperature range of1750–1950 ^{0}C and 34-66 % carbon, ferrosilicon grade FS45, ferrosilicoaluminium and Fe-Si-Al ligature can be formed from the leaching tails. More over, the maximum extraction of silicon into the alloy is 95.0 % and aluminum 69.8 % (point e). The maximum silicon content in the alloy –48.6 % is noted at 1878^{0}C and 59,3 % carbon (point M), and aluminum – 11.0%, – at1950 ^{0}C and 66 % carbon (point e).

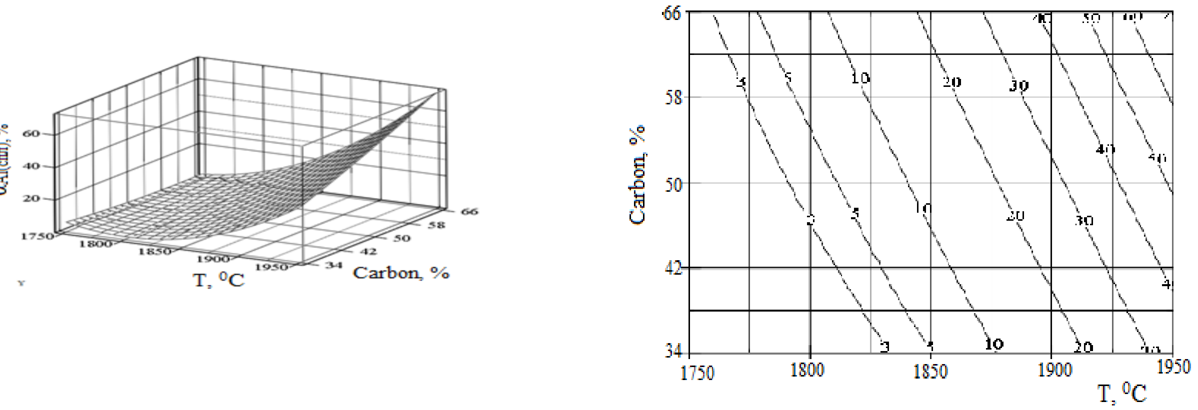
Tables 5 and 6 provide information on the technological parameters for the formation of ferroalloys of various types.

I II

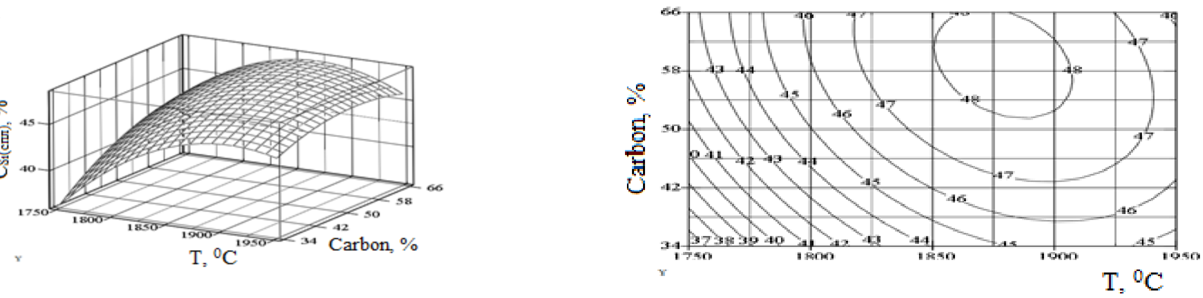
A

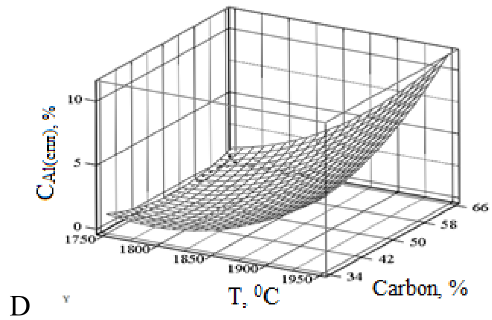


B

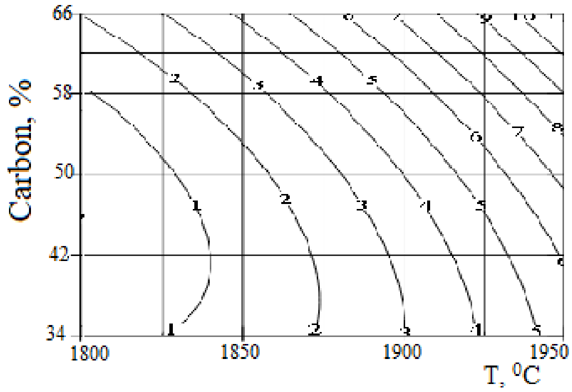


C





D



I – volume image, II – planar image
The numbers on the lines are the value of the parameter, %

Figure 3 – graphical representation of the effect of temperature and amount of carbon on the degree of extraction of silicon in alloy (A), aluminum in alloy (B), silicon content in alloy (C), aluminum in alloy (D)

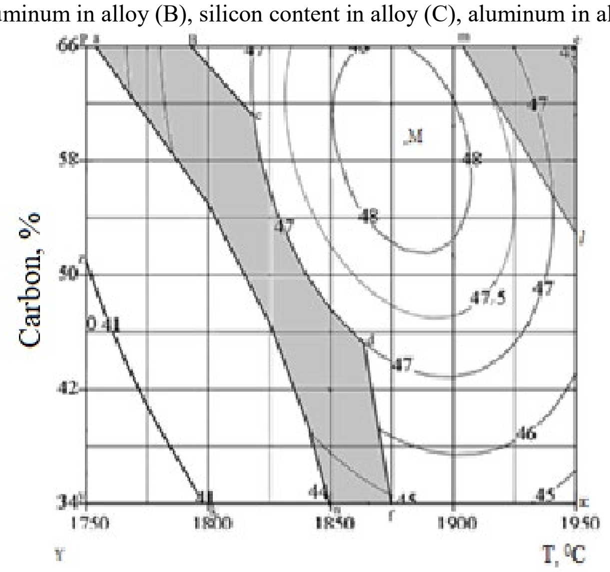


Figure 4-combined information on the influence of temperature and carbon on the technological parameters of obtaining ferroalloy from tailings

Lines ab - $\alpha_{Si alloy}$ = 65 %, dfand bc - $C_{Al alloy}$ = 2 %, ml - $C_{Al alloy}$ = 7,5 %, Cd - $e_{S alloy}$ = 47 %, zk - $C_{Si alloy}$= 41 %

Table 5 – Values of technological parameters in the boundary areas of Figure X.

Points in the drawing I	T, ⁰C	carbon,%	$\alpha_{Si(alloy)}$, %	$\alpha_{Al(alloy)}$, %	$C_{Si(alloy)}$, %	$C_{Al(alloy)}$, %
a	1753	66,0	65,0	2,0	43,0	0,9
b	1796	68,0	68,2	1,3	45,9	2,0
c	1817,0	71,1	71,1	12,4	47,0	2,0
d	1860,3	71,3	71,3	13,5	47,0	1,8
f	1875	68,0	68,0	9,9	44,9	2,0
n	1850	60,0	65,0	5,0	44,0	1,5
m	1903	66.0	88.6	42.6	47.5	7.5
e	1950	66.0	95.0	69.8	45.9	11.0
l	1950	52.4	85.2	51.3	46.7	7.5
k	1800	34.0	57.4	2.1	41.0	0.1
z	1750	51.0	54.3	20.1	41.0	0.1
x	1950	34,0	77,4	34,3	45,2	5,3

Table 6 – Characteristics of ferroalloys in technological fields Figures T

The area in the figure 3	*mel*	*bmlxfdc*	*abcdfn*	*kzpan*	*kzy*
Characteristics of ferroalloys	Ferrosilicoaluminium grade FeSi45 A 1	Fe-Si-Al ligature with 2-2,75% Al content	Ferrosilicon FeSi45	Ferrosilicon Grade FeSi45 Extraction Si<65%)	Substandard ferrosilicon (Si<41%)

It follows from Tables 5 and 6 that to obtain ferrosilicoaluminium grade FS45A10, a temperature of1903–1950 ⁰Cand 52,4-66,0 % carbon is required (while the extraction of Si and Al into the alloy is 85.2–

95.0 % and 42.6–69.8 %, respectively. In the *abcdfn region* (at1753–1875^{0}C and 34–66 % carbon), ferrosilicon of the FS45 brand is formed with the extraction of 60.0–71.3 % silicon into it. In the *kzpan region*, ferrosilicon of the FS45 grade is also formed (at 1750–1850^{0}C and 34–54,3 % carbon). However, the degree of silicon extraction into the alloy is only 51–60 %. A large field in Figure 4 occupies the *bmlxfdc region*, in which a Fe-Si-Al ligature is formed with a content of 2–7.5 % Al and 44–48,6 % Si. Electric melting of the leaching tails was carried out with two charge compositions (Table 7).

Table 7 - Charge compositions for electric tailings melting

Charge №	Composition of the charge, %		
	Leachingtails	Coke	Steel shavings
1	54	25	21
2	52	28	20


I II

Figure 5-shows samples of the production of ferroalloys

I Alloy melting charge No. II Alloy melting charge No.

Figure 5 - photos of ferroalloys smelted from the leaching tailings of oxidized ore from the Almaly deposit

The ferroalloy obtained during the melting of the first charge contained 41,3 % Si, 1,3 % Al, 1.0 % C, 54.9 % Fe, 1,5 % (∑Mn, Al, Ca, Ti, Cz). According to [GOST 1415–93. Ferrosilicij 2011], this alloy can be attributed to ferrosilicon grade FeSi45. Ferroalloy, the second melting contained 43,4 % Si, 7,4 % Al, 47,3 % Fe, 0,7 %Mn, 0,3 % Ti, 0.3 % Ca, 0.9 % C, 0.3 % others. According to (Ferrosilikoalyuminij 2013), it belongs to ferrosilicoaluminium grade FeSi45A10.

The degree of silicon extraction into the alloy from the first charge was 75.8 % and from the second 79.8 %. Aluminum from the first charge turned into an alloy by 15.9 % and from the second 50.8 %.

Conclusion

Based on the results obtained for the production of ferroalloys from the tailings of sulfuric acid leaching of the oxidized copper ore of Almaly , the following conclusions can be drawn

1. Under equilibrium conditions, the formation of ferrosillicoaluminium grade FeSi45A10 is possible from tailings in the temperature range1903–1950^{0}C in the presence of 52–66 % carbon and 24 % iron. At 1753–1875^{0}C and 34–66 % carbon, ferrosillium grade FeSi45 is formed, and in the temperature range 1796–1950^{0}C, 34–66 % carbon Fe-Si-Al ligature with a content of 2.0–7.5 Al and 45.9–47.5 % Si.

2. For electric melting of charges containing 52–54 % tailings, 25–28 % coke and 20–21 % steel chips. The resulting ferrosillium has a grade of FeSi45 (41.3 % Si and 1.3 % Al), and ferrosillicoaluminium has a grade of FeSi45A10 (43.4 % Si and 7.4 % Al).75,8–79,8 % Si and 70–50,8 % Al are extracted into the alloy.

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