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Metallurgy

Optimization of joint electric smelting of the Shalkiya sulfide ore and its beneficiation tailings with medium-silicon ferrosilicon production

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ABSTRACT
Flotation beneficiation of the Shalkiya deposit high-silicon sulfide ore, containing 4-6% of ∑ Pb and Zn, is ineffective, with the extraction of <80% of zinc and <60% of lead into the concentrate and the formation of up to 0.93 t of tailings per 1 ton of the ore. The paper presents the results of experimental studies on the processing of a mixture of the Shalkiya ore and its beneficiation tailings by electric smelting in the presence of coke, steel cuttings and magnetite concentrate, which acts as a sulfide oxidizer and iron supplier. The effect of coke and the iron replacement degree in magnetite concentrate with iron in steel cuttings on the silicon extraction in the alloy and its content in the alloy was studied using the method of planning experiments and their optimization. It was established that from 75 to 82.8% of silicon is extracted from the mixture into the silicon-containing alloy. The silicon content in the alloy varies from 30 to 44.2%. The formation of FeSi45 grade ferrosilicon, containing 41-47.7% of silicon, occurs during the smelting of the mixture of ore and tailings with a ratio of 1:1 in the presence of 26.8-33.0% of coke and the replacement of iron from magnetite concentrate with iron from steel cuttings from 19.4 to 97%. The sublimates formed during the electric smelting contain 25.2% of Zn and 11.7% of Pb. They are 9.4 times richer in ∑Zn and Pb compared to the base mixture.

Keywords: sulfide ore, beneficiation tailings, lead-zinc ore, electric smelting, zinc sublimate, ferrosilicon.

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Introduction

Lead-zinc ores are divided into three classes according to their lead and zinc content: rich, containing >7% ∑Pb and Zn, ordinary - ∑Pb and Zn = 4-7%, and poor - ∑Pb and Zn = 2-4%. Based on the total reserves of zinc and lead, the ores are divided into rich (2-10 million tons), medium (0.5-2 million tons), and poor (<0.5 million tons) [1]. Based on this, the Shalkiya deposit sulfide ore with the total zinc and lead reserves of 5-8 million tons and containing 4.1-6% of ∑ Zn and Pb belongs to the category of rich with ordinary content [[2], [3]]. Ores of this category are mainly processed by hydrometallurgical method: beneficiation, roasting, leaching of the resulting cinder and then producing cathode zinc [[4], [5], [6], [7]]. Even though at least 90% of zinc-containing ores are processed using the hydrometallurgical method, taking into account flotation, and all of its stages are constantly being improved (from flotation to producing cathode zinc) [[8], [9], [10]], this method is associated with the formation of beneficiation tailings, and, as a rule, with an insufficiently high degree of zinc extraction into concentrate. The latter circumstance is quite often connected with the fact that in the process of ore crushing and grinding it is not possible to obtain relatively homogeneous grains of ore minerals, which differ in their properties from the minerals of gangue; existing technologies and reagent regimes do not allow effectively process such ores and obtain required indicators [[11], [12], [13], [14], [15], [16], [17], [18], [19], [20], [21], [22], [23], [24]].

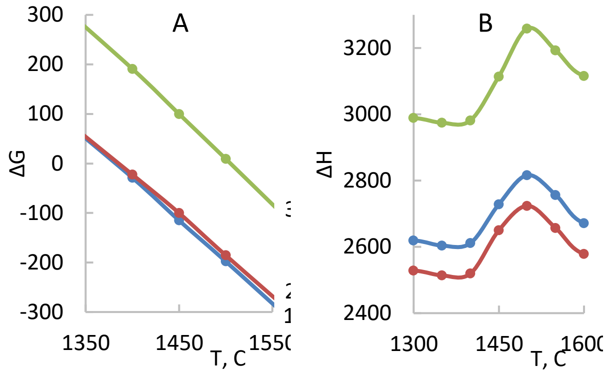
For this reason, the flotation beneficiation of the Shalkiya ore is characterized by low indicators. Thus, 70-77% of Zn (on average 73.5%) is extracted into the zinc concentrate with a content of 56% of Zn, and 49-56% of Pb (on average 52.5%) is extracted into the lead concentrate with a content of 45% of Pb. With an average content of 3.8% of Zn in the ore, the zinc concentrate mass formed from 1 ton of the ore is 1·0.038·0.735/0.56=0.0498 tons. With a concentration of 1.3% of Pb in the ore, the lead concentrate mass is 1·0.013·0.525/0.45=0.015 tons. The beneficiation tailings mass per 1 ton of the ore is 1-(0.0498+0.015)=0.93 tons. The number of tailings during the 8 years of the Shalkiya ore beneficiation is approximately 3 million tons. Therefore, the development of new, low-waste technologies for processing the Shalkiya ore and its beneficiation tailings is currently relevant. A distinctive feature of the Shalkiya ore and its tailings is a significant (40-60%) content of SiO_2 [[25], [26]]. Based on this, modern technology for processing the ore and tailings should provide for high zinc and lead extraction, and production of commercial silicon-containing products. By this, the authors of this paper propose a joint electrothermal processing of the Shalkiya ore and tailings with the extraction of zinc and lead into the gas phase and the production of silicon-containing ferroalloys. To obtain gaseous zinc, lead and silicon ferroalloy, the authors [27] propose the electric smelting of the mixture of ore and tailings in the presence of Fe_3O_4 with the implementation of the following reactions:

$$ZnS + Fe_3O_4 + 8C + 3SiO_2 = Zn_g + 3FeSi + SO_2 + 8CO; \quad (1)$$
$$PbS + Fe_3O_4 + C + 3SiO_2 = Pb_g + 3FeSi + SO_2 + 8CO; \quad (2)$$
$$FeS_2 + Fe_3O_4 + 6C + 3SiO_2 = 3FeSi + 2SO_2 + 6CO. \quad (3)$$

The effect of temperature on ΔG^0 and ΔH^0 of reactions 1-3 is shown in Figure 1 (the calculation was performed using the Equilibrium Compositions module of the HCS-10 software package [28]).

It can be seen that as the temperature increases, the reactions' equilibrium shifts to the right. The dependences of ΔG^0=f(T) for the reactions 1, 2, and 3, respectively, have the form:

$$\Delta G^0=2228.1-1.619T; \quad (4)$$
$$\Delta G^0=2166.1-1.569T; \quad (5)$$
$$\Delta G^0=2592.6-1.727T. \quad (6)$$



The numbers on the lines are the numbers of the reactions

Figure 1- Effect of temperature on ΔG^0 (a) and ΔH^0 (в) for reduction of Zn, Pb and Fe from sulfides

Based on the equations 4-6, it was determined (by ΔG^0=0) that the first reaction becomes possible at a temperature of >1376.10C, the second at a temperature of >1380.60C, and the third at a temperature of >1501.20C. All the reactions under consideration are classified as endothermic. A more energy-consuming reaction is the interaction of pyrite with quartzite, Fe_3O_4 and carbon. The observed maximum on the dependence ΔH^0=f(T) is associated with the melting of FeSi. Earlier [[27], [29]] the authors published the results of a complete thermodynamic analysis of the formation of silicon-containing ferroalloys from the mixture of the Shalkiya ore and its beneficiation tailings and exploratory (preliminary) electrothermal smelting of silicon-containing alloys from the mixture.

This paper presents the results of experimental studies on the optimization of obtaining silicon-containing alloys from the mixture of the Shalkiya ore and its beneficiation tailings.

Experimental part

The electric smelting of the mixture of the Shalkiya ore and its beneficiation tailings (SHOT) with a mass ratio of 1:1 was carried out using a single-electrode arc furnace with a maximum power of 15 kV·A. The melting was carried out in a graphite crucible with a height of 12 cm and an internal diameter of 8 cm. The setup diagram and the experimental methodology were described by the authors earlier in [29].

The resulting alloy and sublimate were analyzed by SEM microscopy (a scanning electron microscope JSM-6490LV), gravimetric method. The content of lead and zinc was determined by the polarographic method according to [30]. The content of Si in the alloy ($C_{Si(alloy)}$) was determined by its density (D, g/cm^3) by the formula (7):

$$C_{Si(alloy)}=252.405-101.848D+18.209D^2-1.243D^3 \quad (7)$$

The silicon extraction degree in the alloy ($\alpha_{(Si(alloy))}$) was determined by the ratio of the silicon mass in the alloy to the silicon mass in the charge. The studies were carried out by the method of mathematical planning of experiments using the second-order rotatable plans (Box-Hunter plan) [[31], [32]]. Based on the obtained experimental data, the regression equations were found for the effect of the coke amount (C, % of the SHOT mass) and the iron replacement degree in the magnetite concentrate with iron in the steel cuttings (γ, %) on $\alpha_{(Si(alloy)}$ and $C_{Si(alloy)}$. The adequacy of the regression equations was checked by the Fisher's criterion, the significance of the equation coefficients was checked by the Student's criterion. The experimental error did not exceed 5%. Based on the regression equations, 3D and planar images of the coke and γ effect on $\alpha_{(Si(alloy)}$ and $C_{Si(alloy)}$ were constructed.

Results and Discussion

The process was optimized by superimposing horizontal images $\alpha_{(Si(alloy)}$ =f(K, γ) and $C_{Si(alloy)}$= f(K, γ).

The chemical composition of the Shalkiya ore and its beneficiation tailings is shown in Table 1.

Table 1- Chemical composition of the Shalkiya ore and its beneficiation tailings, %

Component of the mixture	**Content, %**									
	ZnS	***PbS***	***FeS_2***	***SiO_2***	***$CaCO_3$***	***$MgCO_3$***	***Al_2O_3***	***Na_2O***	***K_2O***	***Fe_2O_3***
Ore	5.2	1.0	4.0	50.2	19.5	10.1	6.6	0.3	0.4	2.5
Tailings	2.5	0.8	3.1	60.1	17.06	8.9	2.2	0.5	0.6	3.2
Mixture	3.8	0.9	3.4	55.3	18.41	9.5	4.5	0.4	0.5	2.8

The ore and coke were used in 1-1.5 cm fractions. The beneficiation tailings were pelletized together with bentonite clay to 1-1.5 cm and dried at 150-160°C for 30 minutes. The magnetite concentrate obtained from the copper ore flotation tailings at the Balkhash plant was used as a raw material containing Fe_3O_4 and the following, by mass %: 86.0 of Fe_3O_4; 10.76 of SiO_2; 2.0 of CaO; 1.4 of Al_2O_3; 0.3 of MnO; 0.3 of K_2O; 0.2 of Na_2O; 0.4 of MgO; 0.017 of ZnO; 0.1 of PbO. The ash content in the coke was 13.0%, volatiles – 1.1%, moisture – 0.8%, S – 0.7%, and C – 84.5%. The steel cuttings contained 97.31% of Fe, 1.8% of C, 0.4% of Si, 0.2% of Mn, and 0.1% of Al.

Table 2 shows the matrix of the experiments and their results.

Table 2- Matrix of the experiments on the electric smelting of the Shalkiya ore and tailings mixture and their results

#	***Variables***				***Output parameter***			
	Coded		***Natural***		***$\alpha_{(Si(alloy)}$, %***		***$C_{(Si(alloy)}$, %***	
	X_1	***X_2***	***C, %***	***γ, %***	***Exp.***	***Calc.***	***Exp.***	***Calc.***
1	+	-	31.5	15	73.4	72.97	39.2	39.08
2	+	+	31.5	85	49.0	78.86	42	42.09
3	-	-	24.5	15	58.1	58.06	34.58	34.57
4	-	+	24.5	85	73.5	73.76	37.5	38.09
5	1.414	0	33	50	82.0	82.36	44.4	44.52
6	-1.414	0	23	50	68.4	68.21	39.1	35.51
7	0	1.414	28	100	74.3	74.17	38.1	37.71
8	0	-1.414	28	0	58.6	58.9	33.2	33.11
9	0	0	28	50	78.0	77.0	41.3	41.48
10	0	0	28	50	76.5	77.0	42	41.48
11	0	0	28	50	76.0	77.0	41.1	41.48
12	0	0	28	50	77.5	77.0	41.8	41.48
13	0	0	28	50	77.0	77.0	41.2	41.48

Based on the data in Table 2, the following regression equations were obtained:

$$\alpha_{(Si(alloy)} =-64.204+1.141\,\gamma+63.428\cdot10^{-1}\cdot C-42.694\cdot10^{-4}\gamma^2-69.877\cdot10^{-3}\cdot C^2-0.02\cdot C\cdot\gamma; \quad (8)$$

$$C_{Si(alloy)}=15.6+32.276\cdot10^{-2}\gamma-58.36\cdot C-24.767\cdot10^{-5}\,\gamma^2-10.204\cdot10^{-4}C\cdot\gamma. \quad (9)$$

Then, using the equations 8-9, 3D and planar images of $\alpha_{(Si(alloy)}=f(C,\ \gamma)$ and $C_{Si(alloy)}=f(C,\ \gamma)$ were constructed, shown in Figures 2-3.

It follows from Figure 2 that the minimum silicon extraction degree in the alloy is 45%, and the maximum is 82.8%. $\alpha_{(Si(alloy)}$ from 75 to 82.8% occurs in the presence of 15.0-33% of coke and 14.8-99.6% of γ, $\alpha_{(Si(alloy)}$ from 80 up to 82.3% occurs in the presence of 22.6-33% of coke and 31.5-81.0% of γ. At a constant value of γ, an increase in the amount of coke increases the silicon extraction in the alloy. The silicon concentration in the alloy varies from 30 to 44.2%. Moreover, the formation of FeSi45 grade ferrosilicon (41-44.7% of Si) occurs in the presence of 26.8-33% of coke and 19.4-97.2% of γ. At a constant value of γ, an increase in the amount of coke increases the silicon concentration in the alloy. The low silicon extraction degree in the alloy in the absence of steel cuttings is associated with the foam formation during the magnetite reduction.

I

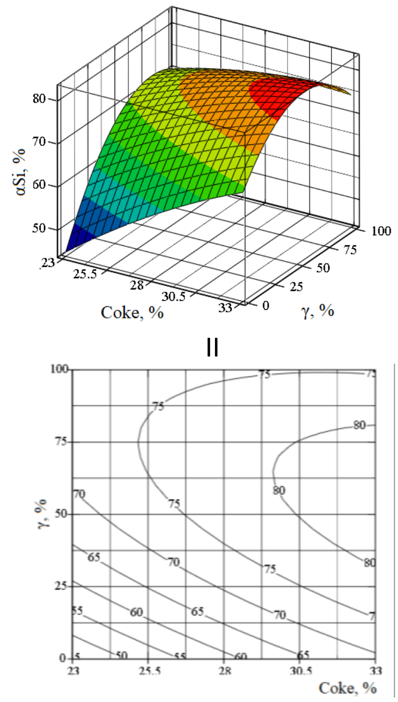


Image: I – 3D, II – planar

Figure 2- Effect of coke and iron replacement degree in the magnetite concentrate with iron in the steel cuttings on the silicon extraction degree in the alloy

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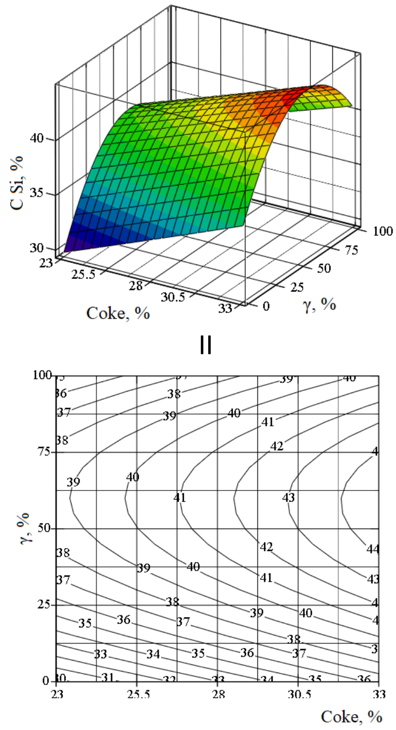


Image: I – 3D, II – planar

Figure 3- Effect of coke and iron replacement degree in the magnetite concentrate with iron in the steel cuttings on the silicon concentration in the alloy

Figure 4 shows a combined image picture of the dependence $\alpha_{(Si(alloy)}$=f(C, γ) and $C_{Si(alloy)}$=f(C, γ). Table 3 shows values of technological parameters in two areas, in the first area $C_{Si(alloy)}$=41-44.7% and $\alpha_{(Si(alloy)}$=75.1-82.6%, in the second area $\alpha_{(Si(alloy)}$=80.0-82.6% and $C_{Si(alloy)}$=42.6-44.7%.

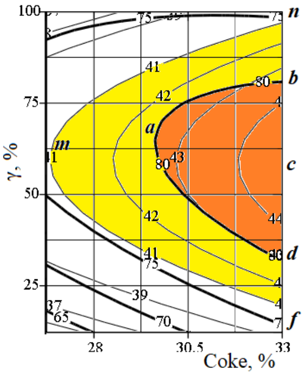


Figure 4- Combined image of the dependence $\alpha_{(Si(alloy)}$=f(C, γ) and $C_{Si(alloy)}$=f(C, γ)

Table 3- Values of technological parameters at the boundary points of two areas: 1 – with $C_{Si(alloy)}$≥91.0% and 2 – with $\alpha_{(Si(alloy)}$≥80%

Point in Figure 4	Technological parameters			
	Coke, %	***γ, %***	***$\alpha_{(Si(alloy)}$, %***	***$C_{Si(alloy)}$, %***
a	29.6	71.3	80.0	42.6
b	33.0	81.0	80.0	43.3
c	33.0	56.6	82.3	44.7
d	33.0	31.5	80.0	43.0
m	26.8	62.5	75.8	41.0
n	33.0	97.2	75.1	41.0
f	33.0	19.4	75.7	41.0

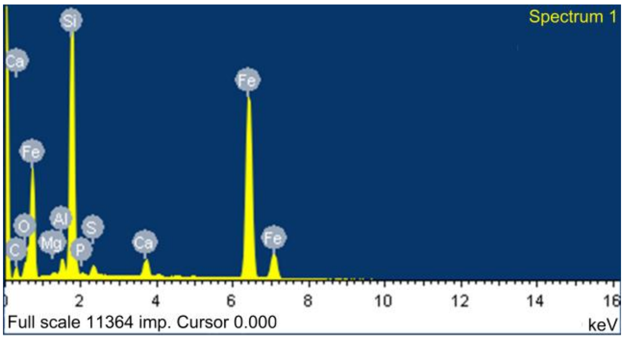
Figure 5 shows photos of ferroalloys smelted from ore and tailing mixtures with varying degrees of iron replacement in magnetite concentrate with iron in steel cuttings.

I

I – 33% of coke, γ=20.0%; II – 28.5% of coke, γ=75%

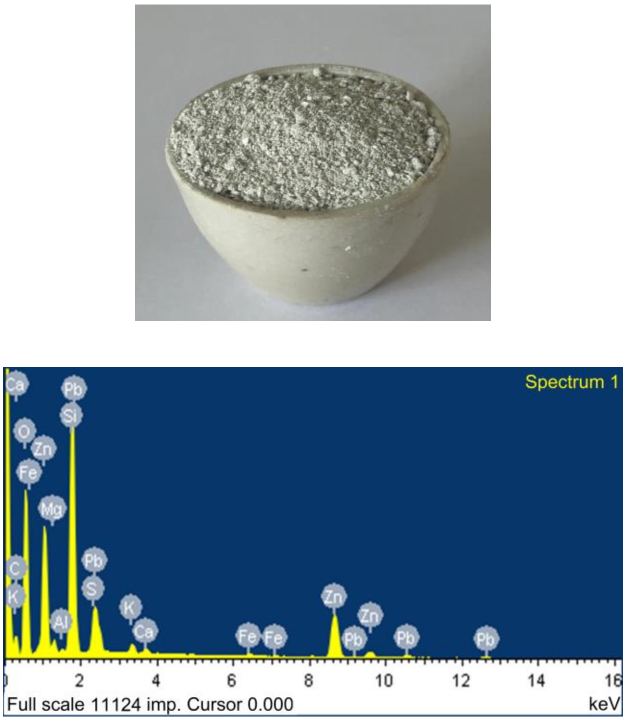
Figure 5- Photos of smelted ferroalloys

Figure 6 shows the SEM analysis of the alloy at γ=20%; Figure 7 shows the sublimates collected on the electric holder.



Element	Si	Fe	Al	Mg	P	S	Ca	O	C
Content, %	41.28	47.82	2.40	0.24	0.25	1.02	1.91	3.95	1.13

Figure 6- SEM analysis of the alloy



Element	Zn	Pb	Fe	Si	S	Ca	K	Al	Mg	C	O
Content , %	25.2	11.7	0.4	12.1	1.1	0.4	0.7	0.1	1.1	16.5	30.7

Figure 7- SEM analysis of the sublimates

The alloy containing 41.28% of silicon does not contain zinc or lead. In terms of silicon content, the alloy can be classified as FeSi45 grade ferrosilicon [33]. In the sublimates, into which 98.8% of Zn and 97.8% of Pb pass, the zinc content was 25.2% and the lead content was 11.7%. That is, the sublimates are a polymetallic concentrate, in which the zinc content is 8.1 times higher than in the base mixture, and the ∑ Zn and Pb is 9.4 times higher.

Conclusions

Based on the results obtained during the electric smelting of the mixture of ore and beneficiation tailings with coke, magnetite concentrate, and steel cuttings, the following conclusions can be drawn:

A high (75-82.8%) degree of silicon extraction in the alloy occurs when the mixture is smelted in the presence of 15-33% of coke and with 14.6-91.9% of iron replacement in the magnetite concentrate with iron in the steel cuttings.

The silicon concentration in the alloy varies from 30 to 44.2%. Moreover, the formation of FeSi45 grade ferrosilicon (41-44.7% of Si) occurs in the presence of 26.8-33% of coke and 19.4-97.2% of γ.

The main part of zinc (98.8%) and lead (97.81%) is extracted from the mixture into the sublimates, which contain 25.2% of Zn and 11.7% of Pb. The zinc concentration in the sublimates is 8.1 times higher than that in the base mixture, the zinc and lead sum concentration is 9.4 times higher.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **V. Shevko:** Conceptualization, Formal Analysis, Software, Methodology, Investigation, Writing – original draft; **B. Makhambetova:** Project administration, Investigation, Writing – original draft; **D. Aitkulov:** Validation, Resources, Funding Acquisition, Writing – review & editing; **A. Badikova:** Investigation, Visualization, Writing – review & editing.

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Орташа кремнийлі ферросилиций алу үшін Шалқия сульфидті кені мен оның байыту қалдықтарын біріктіріп электрмен балқытуды оңтайландыру

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[2] Қазақстан Республикасының минералдық шикізатты кешенді қайта өңдеу жөніндегі ұлттық орталығы, Алматы, Қазақстан

Мақала келді: *22 мамыр 2024*
Сараптамадан өтті: *3 шілде 2024*
Қабылданды: *17 шілде 2024*

ТҮЙІНДЕМЕ

Құрамында 4-6% ∑ Pb және Zn бар Шалқия кен орнының сульфидті жоғары кремнийлі кенін флотациялық байыту барысында <80% мырыш пен <60% қорғасынды концентратын алу тиімсіз және 1 тонна кенге шаққанда 0,93 т-ға дейін қалдық түзіледі. Мақалада Шалқия кенінің электрлік балқыту қоспасын және оны байыту қалдықтарын кокс, болат үгінділері және сульфидтерді тотықтырғыш және темір жеткізуші қызметін атқаратын магнетит концентратының қатысуымен өңдеудің тәжірибелік зерттеулерінің нәтижелері берілген. Тәжірибелерді жоспарлау және оларды оңтайландыру әдісін қолдана отырып, кремнийдің қорытпаға алынуына кокстың әсері және магнетит концентратындағы темірді болат үгінділеріндегі темірмен алмастыру дәрежесі және ондағы осы элементтің құрамы зерттелді. Қоспадан кремнийдің 75-тен 82,8%-ға дейін кремнийі бар қорытпаға алынатыны анықталды. Қорытпадағы кремний мөлшері 30-дан 44,2%-ға дейін өзгерді. Құрамында 41-47,7% кремнийі бар FeSi45 маркалы ферросилицийдің түзілуі 26,8-33,0% кокс қатысында және 1:1 кен мен оның байытылған қалдық қоспасын балқытқанда және магнетит концентратының темірін болат үгінділерімен 19,4-тен 97%-ға дейін алмастырғанда пайда болады. Электрлік балқыту кезінде түзілетін возгондар құрамында 25,2% Zn және 11,7% Pb болады. Олар бастапқы қоспамен салыстырғанда ∑Zn және Pb-ға 9,4 есе бай.

Түйін сөздер: сульфидті кен, байыту қалдықтары, қорғасын-мырыш кені, электрлік балқыту, мырыш возгоны, ферросилиций.

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Оптимизация совместной электроплавки сульфидной руды Шалкия и хвостов ее обогащения с получением среднекремнистого ферросилиция

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АННОТАЦИЯ

Флотационное обогащение сульфидной высококремнистой руды месторождения Шалкия, содержащей 4-6% ∑ Pb и Zn происходит не эффективно с извлечением в концентрат <80% цинка и <60% свинца и образованием на 1 т руды до 0,93 т хвостов. В статье приводятся результаты экспериментальных исследований переработки электроплавкой смеси руды Шалкия и хвостов ее обогащения в присутствии кокса, стальной стружки и магнетитового концентрата, выполняющего роль окислителя сульфидов и поставщика железа. Методом планирования экспериментов и их оптимизацией исследовано влияние кокса и степени замены железа магнетитового концентрата на железо стальной стружки на извлечение кремния в сплав и содержание в нем этого элемента. Установлено, что из смеси в кремнийсодержащий сплав извлекается от 75 до 82,8% кремния. Содержание кремния в сплав изменяется от 30 до 44,2 %. Образование ферросилиция марки FeSi45, содержащего 41-47,7% кремния происходит при плавке смеси руды и хвостов с отношением 1:1 в присутствии 26,8-33,0% кокса и заменой железа магнетитового концентрата на железо стальной стружки от 19,4 до 97%. Возгоны, образованные при электроплавке содержат 25,2% Zn и 11,7% Pb. Они богаче по ∑Zn и Pb в сравнении с исходной смесью в 9,4 раза.

	Ключевые слова: сульфидная руда, хвосты обогащения, свинцово-цинковая руда, электроплавка, цинковые возгоны, ферросилиций.

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