Optimal parameters for producing ferrosilicon from a mixture of diatomite and opoka

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Abstract. In view of the limited reactivity of crystalline silica used in the production of ferrosilicon, it is necessary to search for silica-containing raw materials that have this property to a higher degree. The article presents the results of studies on the production of ferrosilicon from a mixture of amorphous silica-containing rocks – opoka and diatomite in a 1:1 ratio in the presence of coke and steel shavings. The studies have included the electric smelting of the charge in a single-electrode arc furnace and using the second-order rotatable designs of experiments (Box-Hunter plans) followed by graphical optimization. The research has established that FeSi25 (24.5-30% of Si), FeSi45 (41-47% of Si), and FeSi50 (47-51% of Si) grades of ferrosilicon can be smelted from a mixture of diatomite and opoka, depending on the amount of coke and steel shavings. The optimal parameters for producing FeSi25 ferrosilicon (with the extraction of 80-82.4% of silicon into the alloy) were 35.0-37.2% of coke and 33.75-35.0% of steel shavings. FeSi45 ferrosilicon (with extraction of 80-84.6% of silicon) was formed with 38.0-45.0% of coke and 29.2-33.2% of steel shavings. At 40.4-45.0% of coke and 29.2-31.1% of steel shavings, FeSi50 ferrosilicon was formed; the extraction of silicon into the resulting alloy was more than 80-81.5%. At the balance electric smelting of the diatomite and opoka mixture in the presence of 45% of coke and 30% of steel shavings, ferrosilicon of FeSi50 grade (51.8-52.6% of Si) was produced with the 82% silicon extraction degree.

1 Introduction

Ferroalloys play a significant role in ferrous metallurgy, as they are used to impart the desired characteristics to steel and improve its quality [1-3]. Ferrosilicon, in particular, is one of the most important ferroalloys, ranking third with a share of about 19% in the global production [4,5]. The main components for its production are quartzite, coke and steel shavings [6], the cost of which is constantly growing. This predetermines the need to search for alternative natural and man-made sources of raw materials.

In the production of ferrosilicon, the amount of quartzite varies from 552 to 1930 kg per 1 tonne of the alloy [6], the basis of which is crystalline silica. However, crystalline silica has the limited reactivity when interacting with carbon [7-12]. To increase the reactivity of

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silica, its crystalline form can be replaced by rocks containing amorphous silica: diatomite, radiolarite, spongolite, tripoli, opoka, opalite, siliceous tuffs, etc. [13-16].

Staff of the M. Auezov South Kazakhstan University developed a ferrosilicon production technology with partial replacement of quartzite with opoka. This change contributed to an increase in the efficiency of silicon extraction into the alloy from 60 to 80% [17]. In continuation of this work, the article presents the results of studies on the production of ferrosilicon from a mixture of opoka and diatomite, the reserves of which in Kazakhstan amount to more than 1.8 billion tonnes [18-20].

2 Research methods

Smelting of the charge (fraction of 0.5-1 cm, weight of 0.7-0.8 kg) was carried out in a single-electrode arc furnace. A graphite crucible with an internal diameter of 8 cm and a height of 16 cm was placed on a graphite hearth of the furnace. The space between the crucible and the furnace's chromium magnesite lining was filled with graphite dust. The graphite electrode's diameter was 3 cm. The furnace was preliminary heated, and then the charge was loaded into the crucible in portions and melted. After the end of the melting process, the crucible was removed from the furnace, cooled, and broken. Then the resulting ferroalloy was separated from the slag. The degree of silicon extraction into the alloy was calculated as a ratio of the silicon mass in the ferroalloy to the silicon mass in the initial raw materials. The content of elements in the raw materials and the resulting products was determined by the atomic adsorption method using an AAS-1 device (Germany), as well as a JSM-6490LV scanning electron microscope with an INSAEnergy energy dispersive microanalysis system (Japan), and also by the pictometric method based on the density (D, g/cm^3) in accordance with equation [21]:

$$CSi(alloy)=252.405-101.848\cdot D+18.209\cdot D^2-1.243\cdot D^3 \quad (1)$$

To determine the optimal parameters of the melting process, the study was carried out using the second-order rotatable planning method (Box-Hunter designs) [22]. The degree of silicon extraction into the alloy (αSi(alloy), %) and the silicon concentration in the alloy (CSi(alloy), %) were the responses. The number of experiments (N) for a two-factor experiment is determined by the formula:

$$N=2^k+2*k+2k+1, \quad (2)$$

where k – the number of independent factors; 2k – the number of experiments in the core of the plan; 2^k – the number of experiments with star distances.

The magnitude of the star distance α is calculated based on the expression ±α=2k/4. For a two-factor experiment k=2, therefore the magnitude of the star distance is ±α=22/4=1.414, and the number of experiments is:

$$N=2^2+2*2+5=13. \quad (3)$$

The confidence limit was chosen to be 95%. The amounts of coke (C, %) and steel shavings (St, % by weight of the mixture) were used as independent factors, and the degree of silicon extraction into the alloy (αSi(alloy)) and the silicon concentration in the alloy (CSi(alloy)) – as the output parameters.

Based on the obtained regression equations, 3D and planar models were built, demonstrating changing of the process' technological parameters depending on the amount of coke and steel shavings. Then, using the obtained planar images, geometric optimization of the parameters was performed.

Thermal analysis of diatomite and opoka was carried out using a Q-1500D derivatograph (Figure 1). The chemical composition of the starting materials is presented in Table 1. The weight loss for the opoka at 1000 °C was 15.6%, and for diatomite was 2.07%. The higher weight loss of the opoka is due to the fact that the opoka was not pre-heat-treated.

A B

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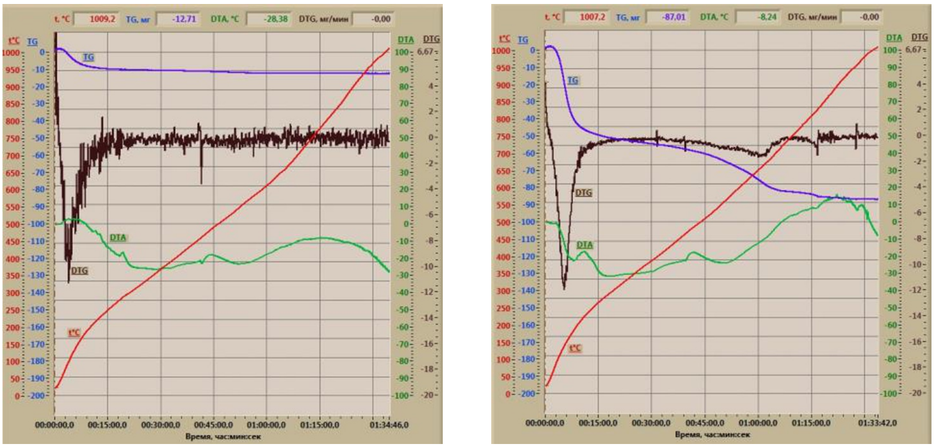


Fig. 1. Derivatograms of diatomite (A) and opoka (B)

Table 1. Chemical composition of amorphous rocks

Rock	Content, %									
	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	K_2O	Na_2O	$CaSO_4$	TiO_2	$CaCO_3$
Diatomite	78,3	6,8	2,6	1,3	3,6	4,3	1,9	0,3	0,4	0,5
Opoka	81,9	7,6	1,5	1,5	1,4	2,6	0,7	2,3	0,5	-

For carrying out the experiments we used a diatomite-opoka mixture in a 1:1 ratio (hereinafter referred to as DOM) with the following composition, wt.%: 80.0 SiO_2, 7.2 Al_2O_3, 3.5 K_2O, 2.5 Fe_2O_3, 2.1 CaO, 1.4 MgO, 1.3 Na_2O, 1.2 $CaCO_3$, 0.5 TiO_2; coke containing 85.6% of C, 4.3% of SiO_2, 2.9% of Fe_2O_3, 3.7% of CaO, 1.8% of Al_2O_3, 0.1% of MgO, 0.6% of S, 0.4% of H_2O, 0.3% of others and steel shavings containing 98% of Fe, 1.3% of C, 0.3% of Si, 0.2% of Mg, 0.2% of others.

3 Results and discussion

The first stage of the research – electric melting – included a number of exploratory experiments to determine the effect of the amount of coke (C, %) and steel shavings (St, % of the mass of the rock mixture). Figure 2 shows the effect of the amount of coke and steel shavings on the degree of silicon extraction into the alloy (αSi(alloy), %) and the concentration of silicon in it (CSi(alloy), %). The effect of steel shavings on αSi(alloy) and CSi(alloy) at 40% of coke is described by the following equations:

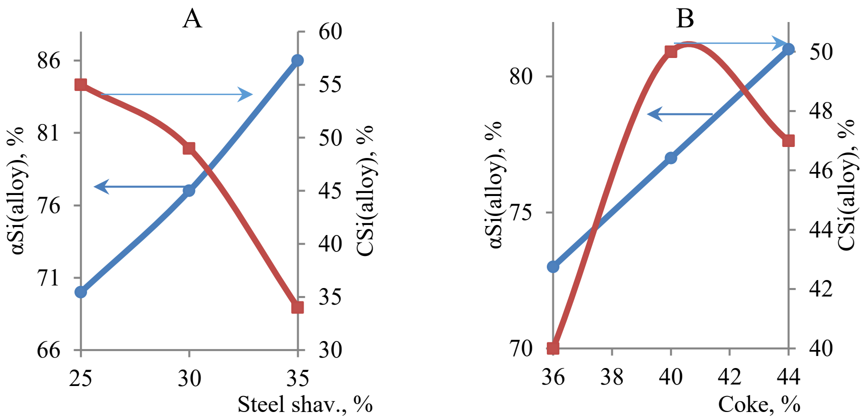
$$\alpha Si(alloy) = 65-0.8\cdot St+0.04\cdot St^2 \quad (4)$$

$$CSi(calloy)= -50+8.7\cdot St-0.18\cdot St^2 \quad (5)$$

The dependences of αSi(alloy) and CSi(alloy) on the amount of coke at 30% of steel shavings have the form:

$$\alpha Si(alloy)=37.0+C \quad (6)$$

$$CSi(alloy)=-635+ 33.375\cdot C-0.4062\cdot C^2 \quad (7)$$



A – 40% of coke, B – 30% of steel shavings

Fig. 2. Effect of coke and steel shavings on the silicon extraction degree and silicon concentration in the alloy

It can be seen that at 40% of coke, an increase in the amount of steel shavings leads to an increase in αSi(alloy). At the same time, the silicon concentration in the alloy decreases. At a constant amount of steel shavings (30%), with increasing the amount of coke, αSi(alloy) and CSi(alloy) increase to 40% of coke. However, when the amount of coke increases above 40%, CSi(alloy) decreases.

To search for conditions under which the silicon extraction degree and silicon concentration in the alloy take maximum values, further studies were carried out using the second-order rotatable designs of experiments.

The effect of the amount of coke and steel shavings on αSi(alloy) and CSi(alloy) was studied in the ranges of the variables shown in Table 2.

Table 2. Levels of factors and ranges of their variation when studying the effect of coke and steel shavings at melting the diatomite and opoka mixture

Variables	Coded kind	Natural kind	
		C, %	St, %
Main level	0	40	30
Variation interval	Δ	3.5	3.5
Top level	+1	43.5	33.5
Lower level	-1	36.5	26.5
Top star distance	+1,414	45	35
Lower star distance	-1,414	35	25

The studies were performed according to the matrix given in Table 3, which also shows the results of the melts. Based on the results of the melts, the following adequate second-order regression equations were obtained:

$$\alpha Si(alloy)=-87.712+4.493\cdot C+2.45\cdot St-3.494\cdot 10^{-2}\cdot C^2+5.796\cdot 10^{-3}\cdot St^2-3.061\cdot 10^{-2}\cdot C\cdot St \quad (8)$$

$$CSi(alloy)=-508.833+20.815\cdot C+10.527\cdot St-0.243\cdot C^2-0.196\cdot St^2-2.041\cdot 10^{-2}\cdot C\cdot St \quad (9)$$

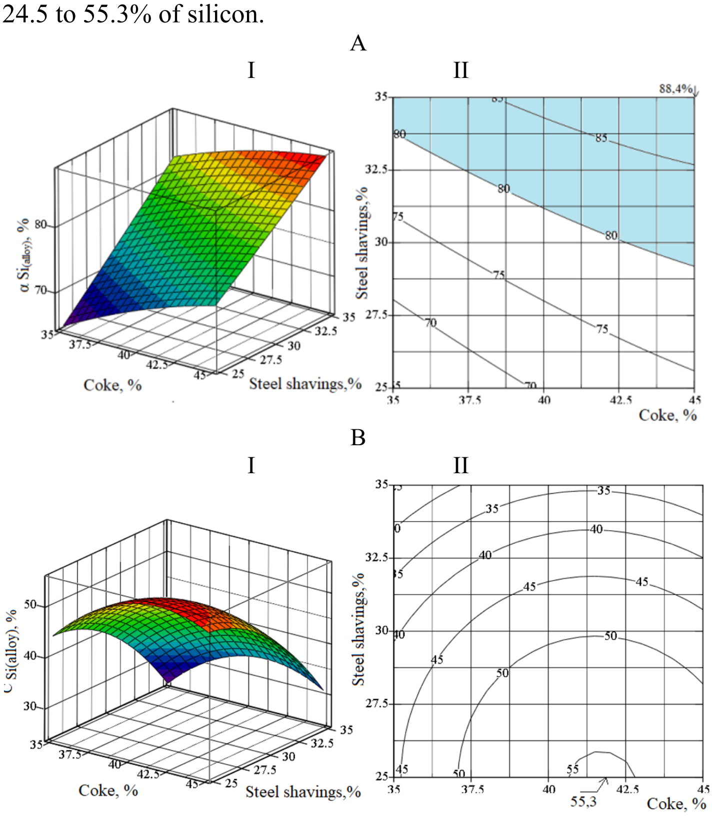
The obtained regression equations adequately describe the experimental results, since the calculated values of the Fisher criterion for equations 6 and 7 (equal to 0.38 and 2.54, respectively) are less than the tabulated value (F_{table}=6.59).

Based on the obtained regression equations using the MathCAD program, 3D models and horizontal response surfaces of the effect of coke and steel shavings on the process parameters were constructed.

Table 3. Matrix of planning the experiments and their results

#	Variables				Technological parameters			
	Coded		Natural		αSi(alloy) exp., %	αSi(alloy) calc., %	CSi(alloy), exp., %	CSi(alloy), calc., %
	X1	X2	C, %	St, %				
1	-1	-1	36.5	26.5	69.4	69.14	47.8	48.12
2	+1	-1	43.5	26.5	75.5	75.36	53.6	53.82
3	-1	+1	36.5	33.5	80.8	80.91	34.6	34.10
4	+1	+1	43.5	33.5	85.4	85.63	39.4	38.79
5	1,414	0	45	30	81.2	81.13	46.6	46.81
6	-1,414	0	35	30	73.3	73.39	39.4	39.46
7	0	1,414	40	35	86.3	86.05	33.3	34.02
8	0	-1414	40	25	70.2	70.47	55	54.56
9	0	0	40	30	77.4	78.12	48.6	49.1
10	0	0	40	30	78.1	78.12	48.9	49.1
11	0	0	40	30	78	78.12	49	49.1
12	0	0	40	30	78.3	78.12	49.2	49.1
13	0	0	40	30	78.8	78.12	49.8	49.1

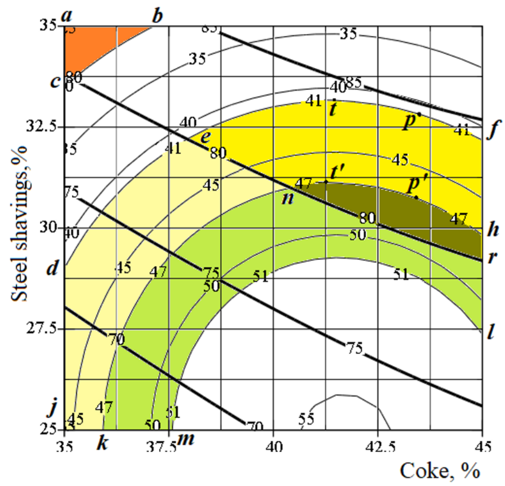
From Figure 3 it can be seen that a high (80-88.4%) degree of silicon extraction into the alloy takes place at 35-45% of coke and 29.2-35% of steel shavings. The resulting alloy contains from 24.5 to 55.3% of silicon.



I – 3D model, II – horizontal section

Fig. 3. Effect of the amount of coke and steel shavings on the silicon extraction degree (A) and silicon concentration in the alloy (B)

To determine the optimal parameters for electric melting of the diatomite and opoka mixture, Figure 4 shows a combined image of changing the technological parameters depending on coke and steel shavings. Values of the technological parameters in the boundary points in various areas are given in Table 4. It can be seen that the result of the electric melting of the diatomite and opoka mixture is ferrosilicon of FeSi25, FeSi45, and FeSi50 grades (Table 5).



(———) – αSi(alloy), %, (———) – CSi(alloy), %

Fig. 4. Combined graphic information on the effect of coke and steel shavings on the melting process' technological parameters

Table 4. Technological parameters in the areas of Figure 4

Points in Figure 4	Coke, %	Steel shavings, %	αSi(alloy), %	CSi(alloy), %
a	35.0	35.0	82.1	24.5
b	37.2	35.0	84.1	30.0
c	35.0	33.7	80.0	30.0
d	35.0	29.0	71.6	41.0
e	38.0	32.3	80.0	41.0
f	45.0	32.2	84.3	41.0
h	45.0	29.9	81.0	47.0
j	35.0	25.0	64.9	44.3
k	35.9	25.0	66.0	47.0
l	45.0	27.5	77.6	51.0
m	37.5	25.0	67.9	51.0
n	40.4	31.0	80.0	47.0
r	45.0	29.2	80.0	48.3
t	41.3	33.2	84.0	41.0
p	43.5	32.8	84.6	41.0

Table 5. Technological parameters for the areas of Figure 4 with grades of the resulting alloys

Area	Coke, %	Steel shavings, %	αSi(alloy), %	CSi(alloy), %	Grade of the alloy
abc	35.0-37.2	33.75-35.0	80.0	24.5-30	FeSi25
denkj	35.0-40.4	25.0-32.3	64.9-80.0	41.0-47.0	FeSi45
eftphn	38.0-45.0	29.2-33.2	80.0-84.6		
knrlm	35.9-45.0	25.0-31.0	66.0-80.0	47.0-51.0	FeSi50
nhr	40.4-45.0	29.2-31.1	80.0-81.5		

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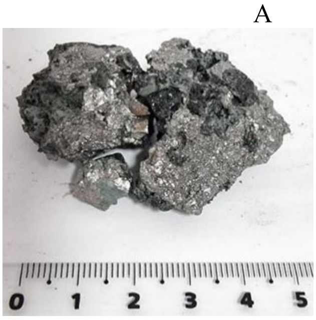
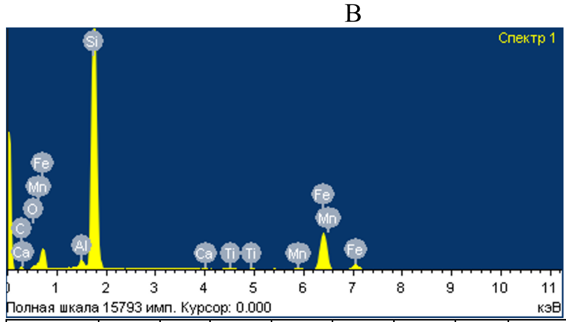
Based on the table presented, it follows that ferrosilicon of FeSi25 grade with the extraction of 80% of silicon into the alloy is formed in a case of 35.0-37.2% of coke and 33.75-35.0% of steel shavings; ferrosilicon of FeSi45 grade with the extraction of > 80% of silicon into the alloy – at 38.0-45.0% of coke and 29.2-33.2% of steel shavings; FeSi50 grade ferrosilicon with the extraction of > 80% of silicon into the alloy – at 40.4-45.0% of coke and 29.2-31.1% of steel shavings.

A photograph and SEM analysis of the ferroalloy produced by the balance electric smelting from a mixture of the rocks, 45% of coke and 30% of steel shavings are shown in Figure 5. The density of the resulting ferrosilicon was 4.54 g/cm3. The silicon content in the alloy, based on the dependence of CSi(alloy)=f(D), was 51.8%. According to the SEM analysis, the silicon content in the alloy was 52.6%. Based on this, the ferroalloy, smelted from the diatomite and opoka mixture, coke and steel shavings, in accordance with State Standard1415-93 refers to ferrosilicon of FeSi50 grade. The degree of silicon extraction into the alloy was 82%.

A



B



Element	C	O	Al	Si	Ca	Ti	Mn	Fe
Wt %	9.95	3.2	1.55	52.6	0.29	0.44	0.6	31.37

Fig. 5. Photograph (A) and SEM analysis (B) of the resulting ferroalloy

4 Conclusion

The electric melting the mixture of diatomite and opoka in the arc furnace allowed us to establish that:

- FeSi25, FeSi45, and FeSi50 grades of ferrosilicon can be smelted from the 1:1 diatomite and opoka mixture (depending on the amount of coke and steel shavings);

- the optimal parameters for producing FeSi25 ferrosilicon (with the silicon extraction degree of 80-82.4%) are 35.0-37.2% of coke and 33.75-35.0% of steel shavings. FeSi45 ferrosilicon (αSi(alloy)=80-84.6%) is formed with 38.0-45.0% of coke and 29.2-33.2% of steel shavings. FeSi50 ferrosilicon (αSi(alloy) > 80-81.5%) can be produced when the amounts of coke and steel shavings are 40.4-45.0 and 29.2-31.1%, respectively;

- the balance electric melting the diatomite and opoka mixture with 45% of coke and 30% of steel shavings allowed to produce FeSi50 ferrosilicon (51.8-52.6% of Si); αSi(alloy) in this case was 82%.

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