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OBTAINING A SILICON ALLOY FROM A SEDIMENTARY ROCK - TRIPOLI

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ABSTRACT

The article examines the results of studies on the production of ferrosilicon from tripoli - a sedimentary rock consisting of opal-like silica. The main part of tripoli is amorphous silica. The studies included the thermodynamic modeling using the HSC-6.0 software package, based on the minimum Gibbs energy principle, combined with the second-order rotatable designs and electric melting of tripoli in an arc furnace. It was found that the interaction products in a tripoli-carbon-iron system under equilibrium conditions are $FeSi_2$, Fe_3Si, *FeSi,* Fe_5Si_3, *SiC,* $SiO_{(g)}$, *Al, Si. Formation of FeSi and* Fe_3Si *occurs at 1200°C; of* Fe_5Si_3 *and Si - at 1400°C; of* $FeSi_2$ *and SiC - at 1500°C; of aluminum - at 1700°C. An increase in the amount of iron from 25 % to 50 % by weight of tripoli allows to increase the extraction degree of silicon into the alloy at 1900°C to 82.5 %. To obtain FS45 grade ferrosilicon from tripoli, the following conditions are necessary: temperature of 1750 - 1900°C, the presence of 37.8 - 51 % of iron and 37 % of carbon. The electric smelting in an arc furnace allows producing FS50 grade ferrosilicon containing 51.1 % of silicon.*

Keywords: tripoli, silicon alloy, ferrosilicon, thermodynamic modeling, electric melting.

INTRODUCTION

When producing ferrosilicon, depending on its grade, from 370 (for FS20) to 1930 (for FS75) kg of quartzite is consumed; its main component is crystalline silica [1, 2]. The reactivity of crystalline silica during smelting ferrosilicon has its limitations. It is possible to increase the reactivity of silica in the production of ferrosilicon by using amorphous silica. The reactivity of amorphous substances is known to be higher than that of crystalline ones [3]. So, in particular, the Gibbs formation energy of crystalline silica according to [4] is 204.75 kcal, and amorphous silica is 202.8 kcal. Amorphous silica-containing rocks include at least 1 bn. t. of tripoli - a loose or weakly cemented finely porous sedimentary rock consisting of radiolarian skeletons with an admixture of clay minerals, glauconite, quartz, feldspars [5, 6]. Tripoli contains 73 - 86 % of SiO_2, the main part of which is amorphous silica [7, 8]. Tripoli is mostly (by 75 %) used in the cement industry, agriculture (as a mineral feed additive, organic fertilizers), construction (production of foam glass, heat and sound insulation materials), for purification of water, oil products, manufacture of paper and cardboard, detergents and cleaning products, insect repellents [9 - 13]. Technologies for the use of tripoli are constantly evolving. In particular, several new technologies have been patented for manufacturing an organomineral fertilizer [14], a sorbent [15 - 18], glass expanded clay and porous ceramics [19 - 21], composite materials [22 - 24], a non-firing heat-insulating material [25], a new building material [26]. However, there is no information in the periodical and patent literature about the use of tripoli in metallurgy.

This paper presents the research results on the determination of the possibility of producing ferrosilicon from tripoli.

EXPERIMENTAL

The studies included the thermodynamic modeling of the process using the software package HSC - 6.0, based on the minimum Gibbs energy, and electric melting in an arc furnace. To implement the thermodynamic modeling, the Equilibrium Compositions subprogram of the software package was used [27]. When working with the HSC-6.0 complex, the initial information is presented as a quantitative (kg) distribution of substances in the system under study. Then, according to the algorithm developed at the South Kazakhstan University, the equilibrium distribution degrees of elements (α, %) by the interaction products was determined [28].

The thermodynamic modeling was carried out using a second-order rotatable plan with obtaining regression equations and subsequent construction of volumetric and planar images of the effect of independent parameters (temperature, amount of iron) on the extraction degree of silicon into an alloy (α_{Simelt}) and its concentration in the alloy (C_{Simelt}) [29 - 31]. The initial tripoli contained 82 % of SiO_2, 3.5 % of CaO, 9 % of Al_2O_3, 4 % of Fe_2O_3, 1.5 % of MgO. The thermodynamic modeling was implemented for constant amount of carbon - 29.0 % of the tripoli weight. The error of the research results was no more than 5 %.

A scheme of the installation used for the electric melting of tripoli is shown in Fig. 1.

The main components of the installation are a single-electrode arc electric furnace, a transformer and a short circuit. The electric furnace is lined with chromium - magnesite bricks. The hearth of the furnace is made of a carbon-graphite block and serves as the lower current conductor. A graphite crucible with an internal diameter of 6 cm and a height of 15 cm is installed on the hearth. The space between the crucible and the lining was filled with graphite chips with a particle size of 0.1 - 0.3 cm. The upper current conductor was a 3 cm diameter graphite electrode. The furnace was equipped with a mechanical device for moving the electrode. The installation included a single-phase furnace transformer TDZhF-1002. The transformer was equipped with a terristor power regulator. The maximum power was 56 kV·A. The maximum voltage at the output of the transformer was 56 V. The short network was made of aluminum busbars. The aluminum busbar of the short network was attached to the graphite hearth by means of three copper studs. The upper electrode was connected to the aluminum bus by a flexible 2 cm diameter copper cable. A detachable refractory cover with thickness of 7 cm was installed on the upper part of lining.

The weight of the charge for melting was 0.6 - 0.7 kg. Before electric melting of the charge, the furnace was heated by an arc for 35 - 40 minutes at a voltage of 40 - 45 V and a current of 500 - 550 A. The first portion of the charge (200 - 250 g) was loaded into the heated furnace. After its melting for 7 - 12 min, the second portion was loaded, and then, after another 7 - 10 min, the last portion of the charge was loaded. The total melting time was 40 - 45 min. During the melting period, the voltage was 15 - 20 V, and the current strength was 500 - 550 A. After termination of the melting, the crucible with the products was cooled in the furnace for 3 - 3.5 hours. After that, the crucible was removed from the furnace and cooled in air for 4 - 4.5 hours. Then it was broken. The resulting products were analyzed using a scanning electron microscope.

RESULTS AND DISCUSSION

Fig. 2 shows the effect of temperature and iron on the quantitative (kg) equilibrium distribution of substances in tripoli-C-Fe systems.

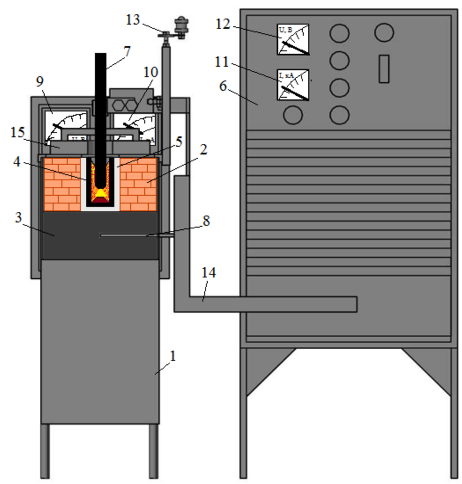
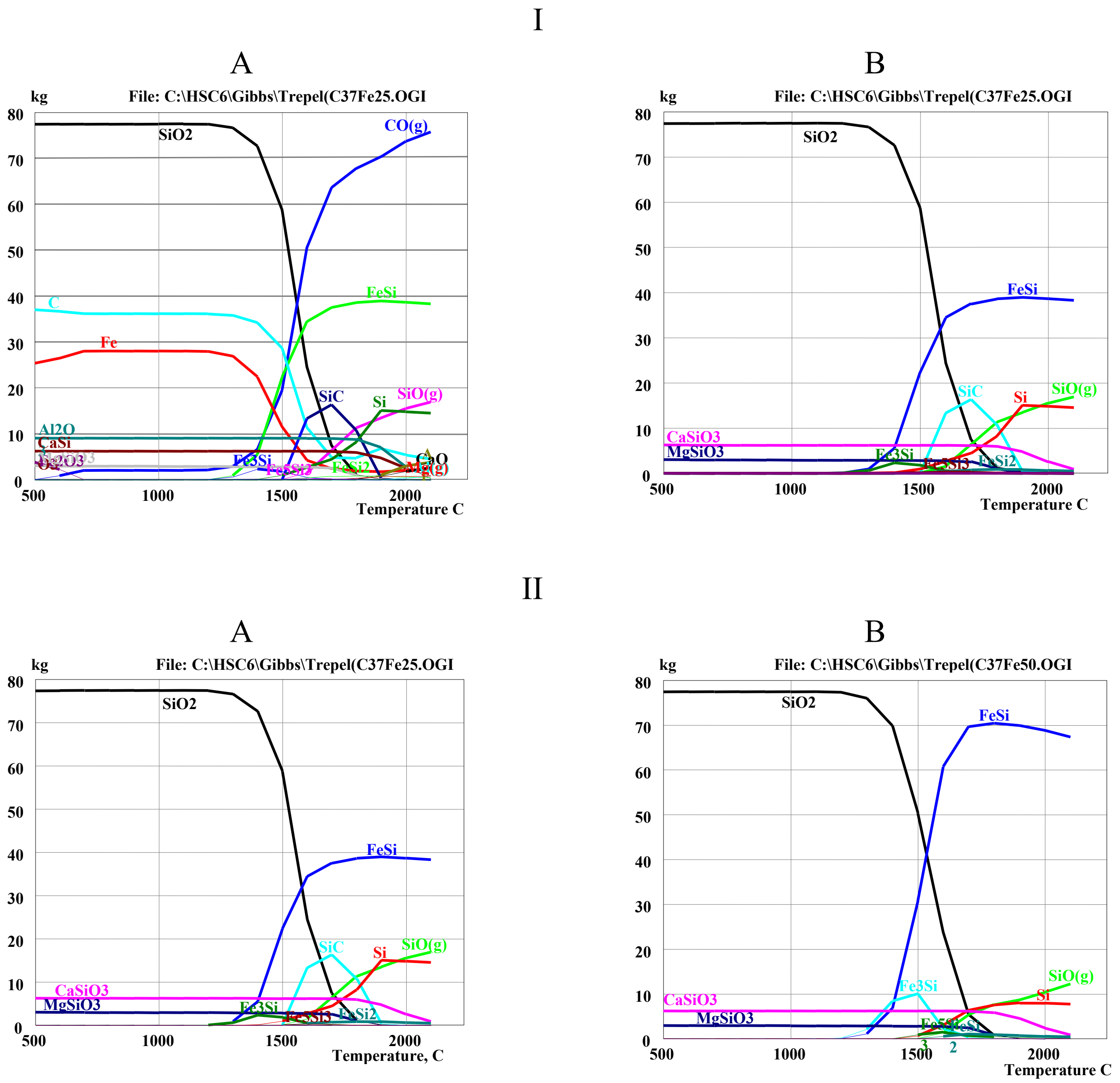


Fig. 1. Scheme of the installation for electric melting of Tripoli: 1 - furnace casing, 2 - chromium-magnesite lining, 3 - carbon graphite hearth, 4 - graphite crucible, 5 - carbon graphite layer, 6 - transformer TDZhF-1002, 7 - graphite electrode, 8 - lower current lead, 9-12 - control ammeters and voltmeters, 13 - electrode movement mechanism, 14 - flexible part of the short network, 15 - furnace cover.



I - 25 % of Fe, II - 50 % of Fe

Fig. 2. Effect of temperature and iron on the quantitative distribution of all (A) and silicon-containing (B) substances in the tripoli-carbon-iron system.

As it follows from Fig. 2, the interaction of tripoli components with carbon in the presence of iron, depending on the temperature, mainly leads to the formation of Fe_3Si, $FeSi_2$, FeSi, Fe_5Si_3, SiC, $SiO_{(g)}$, $CaSiO_3$, $MgSiO_3$, Al, Si, $CO_{(g)}$. To start the formation of FeSi and Fe_3Si (according to $\alpha Si \geq 0.1$ %), the temperature was 1200°C; for SiO(g) and Fe_5Si_3 - 1400°C; for Si, SiC, SiO(g) and $FeSi_2$ - 1500°C. Increasing the amount of iron from 25 % to 50 % leads to reducing the formation of undesirable gaseous SiO and SiC. (For instance, at 1700 - 1800°C the formation of SiO decreases by 1.25 - 1.5 times, and in the presence of 50 % of Fe, SiC is not formed at all).

The effect of temperature and iron content on the equilibrium distribution degree of the main silica-containing products (α_{Si}, %) for 25 % and 50 % of iron of the tripoli weight is presented in Fig. 3.

The incomplete transition of silicon into silicon silicide and elemental silicon is associated with the formation of calcium, magnesium, aluminum silicates, gaseous SiO, and also with the presence of SiO_2. The transition degree of Si into SiO_g decreases with an

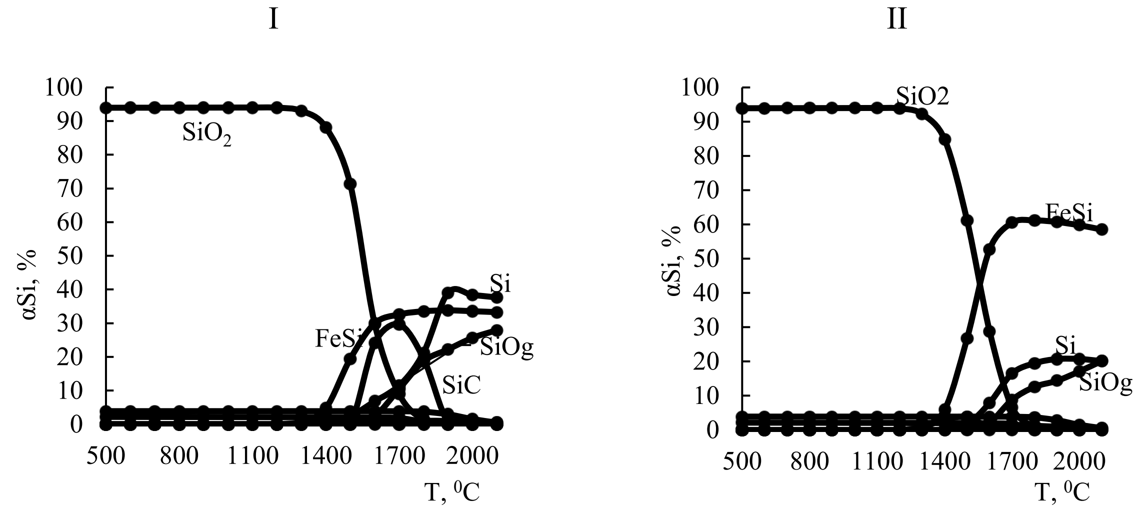
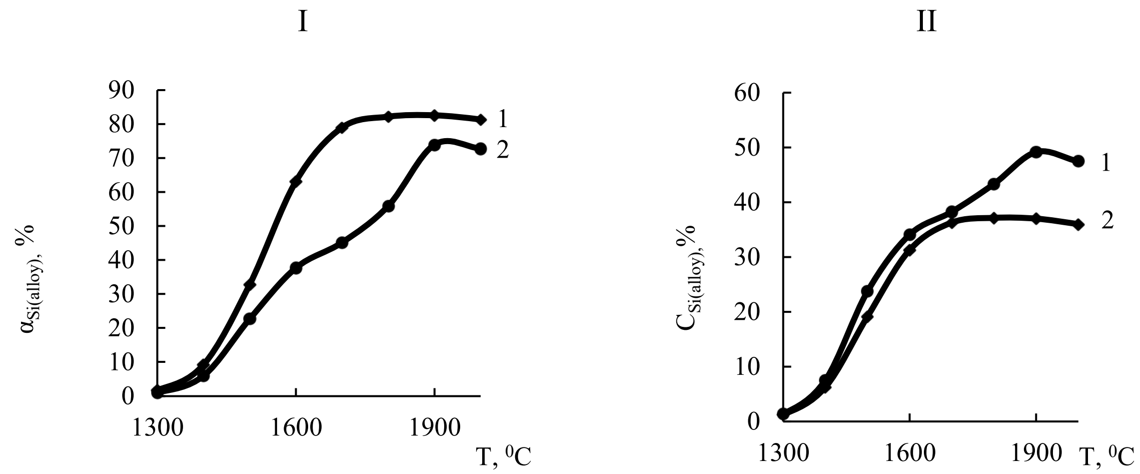


Fig. 3. Effect of temperature and amount of iron on the equilibrium distribution degree of silica-containing substances in the tripoli - carbon - iron systems, I - 25 % Fe, II - 50 %Fe.



1- 25 % of Fe, 2 - 50 % of Fe

Fig. 4. Effect of temperature and iron on the extraction degree of silicon in the alloy (I) and the concentration of silicon in the alloy (II).

increase in the amount of iron. So, for example, at 1800°C this indicator decreases from 18.72 % to 12.60 %. The degree of formation of elemental silicon is practically independent of temperature in the interval of 1600 - 1800°C, but at 1900°C it increases from 9.0 % to 14.7 %. The degree of silicon transition into its elemental state decreases from 39.05 % to 20.68 %. The transition degree of silicon into FeSi significantly increases. So, at 1800°C and 25 % of iron, 33.5 % of silicon passes into FeSi, and in a case of 50 % of Fe - 61.2 %.

Fig. 4 shows the effect of temperature and iron on the extraction degree of silicon into the alloy as Si, Fe_3Si, Fe_5Si_3, FeSi, $FeSi_2$ and the silicon concentration in these compounds.

Judging by Fig. 4, an increase in the amount of iron will increase $\alpha_{Si(alloy)}$ in the temperature range of 1600 - 1900°C. The maximum $\alpha_{Si(alloy)}$ is at 1700 - 1900°C and 50 % of iron (78.96 % - 82.55 %). However, in this case, the concentration of silicon in the alloy decreases. The noticeable reduction of aluminum begins at 1800°C. Moreover, with an increase in the amount of iron from 25 % to 50 %, the transition degree of aluminum into

Table 1. Matrix for planning the experiments on the producing ferroalloy from tripoli and their results.

Experiment	Variables in a code kind		Extraction of Si into alloy, %				Concentration of Si in alloy, %			
			Variables in a natural kind		Optimization parameter - $\alpha_{Si(alloy)}$, %		Variables in a natural kind		Optimization parameter - $C_{Si(alloy)}$, %	
	X_1	X_2	T, °C	Fe, %	Exp.	Calc.	T, °C	Fe, %	Exp.	Calc.
1	+1	+1	1956	47.0	82.1	82.0	1870	47.0	45.1	45.2
2	+1	-1	1956	29.0	78.1	78.2	1730	29.0	40.0	39.3
3	-1	+1	1744	47.0	78.2	74.8	1870	47.0	39.4	40.1
4	-1	-1	1744	29.0	60.3	59.6	1730	29.0	38.1	38.3
5	+1.414	0	2000	38.0	79.9	79.5	1900	38.0	42.7	43.2
6	-1.414	0	1700	38.0	65.0	66.0	1700	38.0	39.4	38.8
7	0	+1.414	1850	50.7	83.2	83.7	1800	50.7	43.5	43.1
8	0	-1.414	1850	25.3	65.5	65.4	1800	25.3	37.7	37.6
9	0	0	1850	38.0	77.9	78.06	1800	38.0	41.7	41.8
10	0	0	1850	38.0	77.5	78.06	1800	38.0	41.8	41.8
11	0	0	1850	38.0	78.3	78.06	1800	38.0	41.3	41.8
12	0	0	1850	38.0	78.0	78.06	1800	38.0	42.4	41.8
13	0	0	1850	38.0	78.6	78.06	1800	38.0	42.0	41.8

its elemental state decreases. So, at 1900°C and 25 % of Fe, aluminum is reduced by 21.36 %, and at 50 % of iron - by 15.47 %. The aluminium concentration in the alloy also decreases from 1.8 % to 0.9 %.

To determine the optimal parameters for the extraction of silicon from tripoli in grade alloy, further studies were carried out by the planning method. The planning matrix and research results are shown in Table 1.

Based on Table 1, the following regression equations were obtained:

$$\alpha_{Si(alloy)} = -1079.73+1.03\cdot T+7.775\cdot Fe-2.35\cdot 10^{-4}\cdot T^2-2.143\cdot 10^{-2}\cdot Fe^2-2.94\cdot 10^{-3}\cdot T\cdot Fe \quad (1)$$

$$C_{Si(alloy)} = -175.5+0.255\cdot T-1.895\cdot Fe-8.1\cdot 10^{-5}\cdot T^2-8.93\cdot 10^{-3}\cdot Fe^2+1.55\cdot 10^{-3}\cdot T\cdot Fe \quad (2)$$

The resulting regression equations are adequate, since the calculated F-criterion for the changing $\alpha_{Si(alloy)}$ is 0.279, and its abular value is 6.59, and for $C_{Si(alloy)}$ this criterion is characterized by the following inequality: 1.248 < 6.59. Using equations 1 and 2, volumetric and planar images of $\alpha_{Si(alloy)}$ = f(T, Fe) and $C_{Si(alloy)}$ = f(T, Fe) were constructed. These curves are shown in Figs. 5 and 6.

It can be seen from Fig. 5 that to achieve $\alpha_{Si(alloy)}$ from 80 % to 89.1 %, the melting of the tripoli should be carried out in the temperature range of 1722 - 2000°C in the presence of 38 % - 50.7 % of iron (shaded area of Fig. 5). As it follows from Fig. 6, the resulting ferroalloy contains 37.1 % - 46.2 % of silicon and refers to FS 45 grade ferrosilicon (shaded area of Fig. 6).

Fig. 7 contains the combined information about $\alpha_{Si(alloy)}$ and $C_{Si(alloy)}$. The boundary technological parameters to produce FS45 and FS50 ferrosilicon (in accordance with State Standard [32]) are shown in Table 2.

Based on Fig. 7 and Table 2, we can conclude that the formation of FS45 ferrosilicon occurs in the *abcd* area at 1754 - 1900°C, 37.0 % of carbon, 38.6 - 50.7 % of iron. The extraction degree of silicon in the ferroalloy is 80 % - 83.5 %.

Producing the ferroalloy from the tripoli was carried out in an arc furnace using a charge containing 59 % of tripoli, 22 % of coke and 19 % of steel shavings.

The chemical composition of initial tripoli was 60.7 % of SiO_2, 8.0 % of Al_2O_3, 12.3 % of $CaCO_3$, 3.0 % of

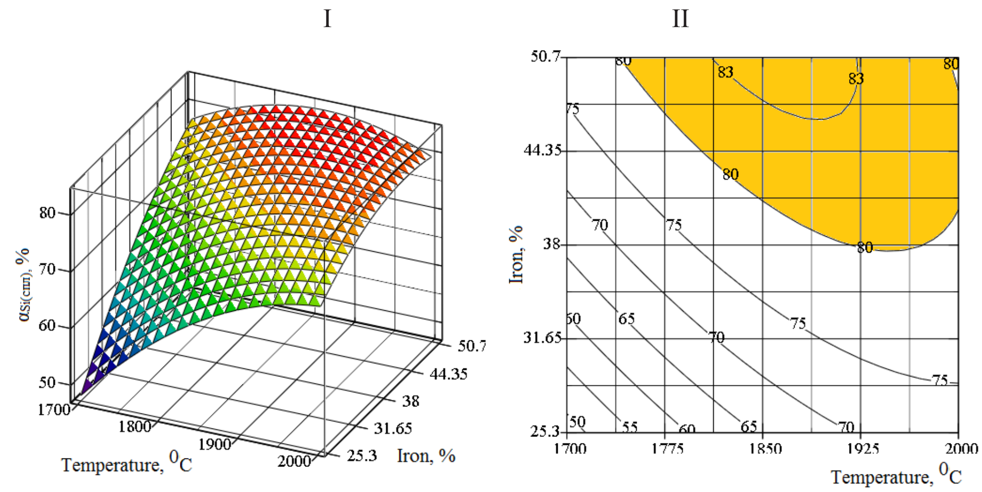


Fig. 5. Effect of temperature and iron on the extraction degree of silicon into ferroalloy, I - volumetric image, II - planar image.

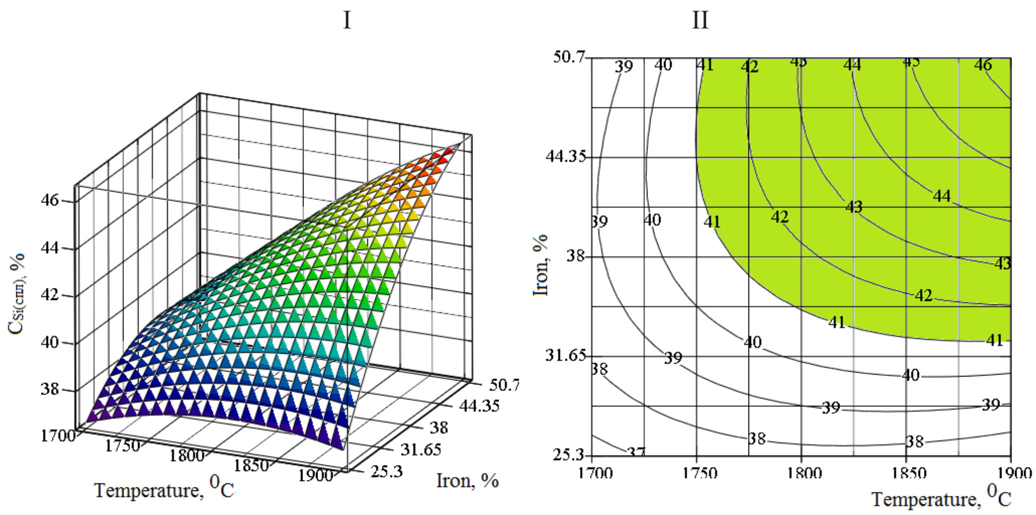
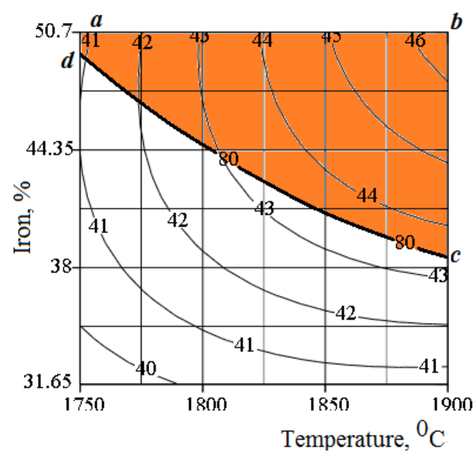


Fig. 6. Effect of temperature and iron on the concentration of silicon in the ferroalloy, I - volumetric image, II - planar image.



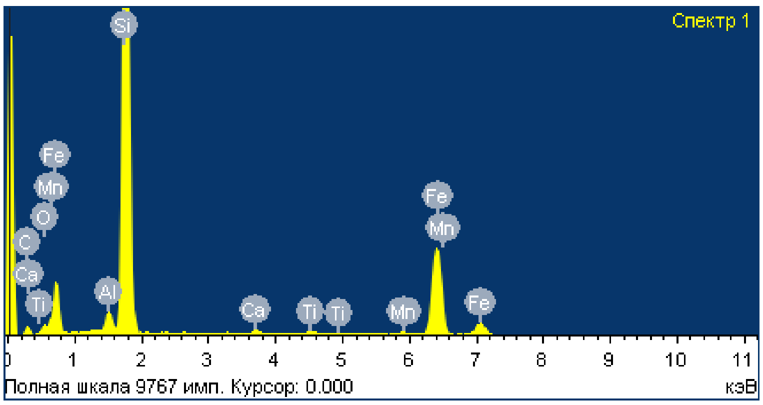
(–) -$\alpha_{Si(alloy)}$, (—)$C_{Si(alloy)}$

Fig. 7. Combined information on the effect of temperature and iron on the extraction degree of silicon into the alloy and the silicon concentration in the alloy.

Table 2. Values of technological parameters at the boundary points (*abcd* area in Fig. 7).

Point in Fig. 7	Technological parameters				
	C, %	Fe, %	T, °C	$\alpha_{Si(alloy)}$, %	$C_{Si(alloy)}$, %
a	37.0	50.7	1759	81.0	41.0
b	37.0	50.7	1900	83.5	46.4
c	37.0	38.6	1900	80.0	43.4
d	37.0	49.4	1754	80.0	41.0

Fig. 8. A photograph of the resulting ferroalloy.



Element	Weight %
C	13.69
O	3.97
Al	2.02
Si	43.84
Ca	0.50
Ti	0.60
Mn	0.63
Fe	34.75
Итоги	100.00

Fig. 9. SEM analysis of produced ferroalloy.

$MgCO_3$, 3.6 % of Fe_2O_3, 2 % of K_2O, 0.5 % of TiO_2, 2 % - others. After the calcination, the contents of SiO_2 and Al_2O_3 in the tripoli were 67.4 % and 9.3 %, respectively.

Fig. 8 presents a photograph of the resulting ferroalloy, and Fig. 9 shows its SEM analysis.

Fig. 9 shows that the resulting ferroalloy, in addition to the metals, contains carbon and oxygen. This is due the fact that in a case of crucible melting the alloy and the slag formed during the process are unsufficiently completely separated from each other (in particular, dark coke particles visible in the photographs). Therefore, the true composition of the ferroalloy differs from the composition shown in Fig. 9. To determine the real silicon content in the alloy, it is necessary to ignore the carbon and the oxygen containing in SiO_2 of the charge. The content of silicon in the alloy, excluding carbon and oxygen in it, was calculated using the expression:

$$C_{Si(alloy)} = \left[\frac{C_{Si(p)} - C_{O(p)} \frac{A\,Si}{M\,O_2}}{100 - C_{C(p)} - C_{O(p)} - C_{O(p)} \frac{A\,Si}{M\,O_2}} \right] \cdot 100 \qquad (3)$$

where $C_{Si(p)}$, $C_{O(p)}$, $C_{C(p)}$ - content of silicon, oxygen and carbon in the ferroalloy according to the SEM analysis, %; $C_{O(p)} \frac{A\,Si}{M\,O_2}$ - silicon bonded with oxygen in the ferroalloy as SiO_2, %.

In our case, the true silicon content in the alloy is:

$$C_{Si(alloy)} = \left[\frac{43{,}84-3{,}97\frac{28}{32}}{100-13{,}69-3{,}97-3{,}97\frac{28}{32}}\right]\cdot 100 = 51{,}1\ \% \quad (4)$$

Such a ferroalloy, in accordance with [32], refers to FS50 grade ferrosilicon; the extraction degree of silicon into the ferroalloy in this case was 86.8 %.

CONCLUSIONS

On the basis of the results, obtained at the interaction of tripoli with carbon in the presence of iron, the following conclusions can be drawn:

- At equilibrium conditions
- the interaction products are $FeSi_2$, Fe_3Si, FeSi, Fe_5Si_3, SiC, $SiO_{(g)}$, Al, Si; the formation of FeSi and Fe_3Si takes place at 1200°C, Fe_5Si_3 and Si - at 1400°C, $FeSi_2$ and SiC - at 1500°C, aluminum - at 1700°C with a constant amount of iron in the charge;
- an increase in the amount of iron from 25 % to 50 % allows to increase the extraction degree of silicon into the alloy: from 72.3 % to 82.55 % at 1900°C;
- to obtain FS45 ferrosilicon from the tripoli, the following conditions should be met: temperature of 1754 - 1900 °C, 37.0 % of carbon, 38.6 - 50.7 % of iron.
- As a result of the crucible electric melting of the tripoli mixed with coke and steel shavings, at the extraction of 86.8 % of Si into alloy, the resulting ferroalloy in terms of silicon content (51.1 %) corresponds to FS50 grade ferrosilicon.

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