

PRODUCTION OF FERROALLOYS, CALCIUM CARBIDE, AND PHOSPHORUS FROM HIGH-SILICON PHOSPHORITE

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ABSTRACT

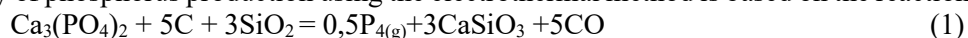
The article provides information on the interaction of Chilisai phosphorite with carbon, coke, and iron with the production of ferroalloy and calcium carbide and the extraction of phosphorus into gas. Research is conducted using computer thermodynamic modeling, mathematical planning of experiments, and electric smelting of phosphorites in an arc electric furnace. It is found that under equilibrium conditions the interaction occurs with the formation of iron silicides, calcium, silicon carbides, calcium, elemental silicon, aluminum, calcium, silicon oxide (II), gaseous phosphorus (P₄, P₂), and iron phosphides. An increase in the amount of iron at 1,500-2,000°C increases the degree of extraction of silicon in the alloy but decreases the extraction of calcium in the calcium carbide, the concentration of silicon in the alloy, and the amount of calcium carbide. In the temperature range of 1,900-2,000°C in the presence of 16.8-19.8% of iron, phosphorus completely converts to gas, and there forms an alloy with 45-47.8% of Si and 1.6-1.9% of Al and calcium carbide in the amount of 150-215 dm³/kg (with the extraction of 60-63.6% of Si into the alloy and 50-56.4% of Ca into calcium carbide). Electric smelting of phosphorite in an arc furnace produces ferrosilicon of grade FS45 (40-44.7% of Si) with the extraction of 73.8% of silicone into it, as well as calcium carbide up to the second grade in the amount of 200-252 dm³/kg. Phosphorus is almost completely (99.0-99.4%) reduced during electric smelting and converted into the gas phase.

Keywords: Phosphorite, Reduction, Thermodynamic modeling, Electrical melting, Phosphorus, Calcium carbide, Ferroalloy.

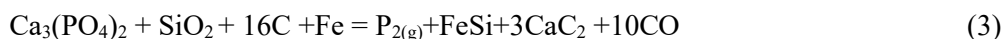
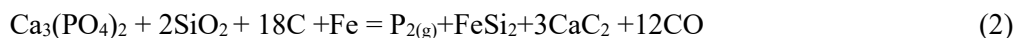
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INTRODUCTION

The modern technology of phosphorus production using the electrothermal method is based on the reaction:



which occurs at 1,400-1,550°C. During electric smelting, phosphorus is reduced (with 89-91% of it transferred into the commercial product) and ferrophosphorus (15-25% of P) and slag are formed.¹ Per each ton of phosphorus, up to 12 t of slag is transported to dumps, occupying the land and detrimentally affecting the environment. The slag, which contains up to 7% of phosphorus, has a limited demand in the construction industry since products containing phosphorus slag are sources of toxic gas emissions.² Given that the phosphate slag takes all the calcium, almost all (98-99%) silicon, and part of the iron, the degree of complex use of raw materials in electric smelting of phosphorite does not exceed 35%. Several methods of phosphorite processing with the sublimation of phosphorus (induction heating³), plasma heating,⁴ power plant,⁵ ROMELT process,⁶ recoveries with natural gas,⁷ and the use of shungite rocks (instead of quartzite and coke⁸) focus on increasing the extraction of phosphorus. Nevertheless, these technologies still produce slag. The problem can only be solved by organizing the reduction of calcium and silicon. In this connection, we propose a technology for processing phosphate rock with simultaneous production of ferroalloy and calcium carbide with the extraction of phosphorus in an ore-thermal electric furnace in accordance with the reactions:



which become possible from the thermodynamic view, respectively, at 1,904 and 1,836 K (Table- 1).

Table-1: The Effect of Temperature on the ΔG^0 (kJ) of the Reactions of Joint Carbothermic Reduction of Tricalcium Phosphate and Silicon Oxide in the Presence of Iron*

T, K	1,473	1,573	1,673	1,773	1,836	1,873	1,904	1,973	2,073	2,173
Reaction 2	955.3	731.6	509.0	287.8	139.5	68.3	0.0	-150	-366.6	-574.8
Reaction 3	779.7	578.2	385.0	185.6	0.0	-13.0	-74.2	-210.3	-406.4	-594.1

* – ΔG^0 calculated using HSC-6.0 software⁹

To decrease slag formation, it is necessary to reduce SiO_2 to Si and CaO to CaC_2 . This requires a temperature of at least 1,904 K (Table-1). This paper presents the results of our research on the simultaneous production of calcium carbide, ferroalloy, and phosphorus from phosphorite of the Chilisa deposit in the Aktobe basin by electrofusion. This deposit is characterized by a rather low content of P_2O_5 (10.5%).¹⁰⁻¹² For this reason, such phosphorite cannot be used for obtaining wet-process phosphoric acid (for this, the content of P_2O_5 must be $\geq 24.5\%$) or for the electrothermal production of phosphorus (for this, the content of P_2O_5 must be no less than 21%).^{13,14} Its increased content of SiO_2 (55%)¹³ allows this raw material to be used for the simultaneous production of phosphorus, siliceous ferroalloy, and (due to the presence of 20% of CaO in it) calcium carbide in an electric furnace.

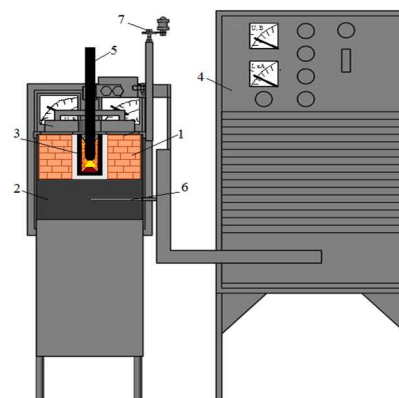
EXPERIMENTAL

The study was conducted using the method of computer thermodynamic modeling together with mathematical experiment planning and electric smelting of phosphorites in an electric arc furnace. Thermodynamic modeling was performed using the HSC-6.0 software package and Chemistry (a subprogram of Equilibrium Compositions) developed by Outokumpu Research Oy (Finland).⁹ The equilibrium distribution of elements was calculated according to the algorithm developed at the M. Auezov South Kazakhstan University.¹⁵ The unit where the electric smelting of the charge was performed is demonstrated in Fig.-1. The material was smelted for 3 to 6 minutes, after which 200-250 g of the charge was added every 4-6 minutes. In every experiment around 1,500-2,000 g of the charge was melted. The voltage was 30-35 V, and the current during smelting was 350-400 A. The ferroalloy was analyzed for metals using the AAS-1 device (Germany) and a JSM-6490LV scanning electron microscope with the INSA Energy energy-dispersive microanalysis system (Japan). In addition, the silicon content (Si(alloy), %) was estimated by its density (ρ , g/cm³), which was determined using the psychometric method.¹⁶ After that, the silicon content was established based on the formula:

$$\text{Si}(\text{alloy}) = 252.405 - 101.848 \cdot \rho + 18.209 \cdot \rho^2 - 1.243 \cdot \rho^3 \quad (4)$$



a – general appearance



b – sketch

Fig.-1: Single-Electrode Electric Arc Furnace: 1 – Chromium-Magnesite Lining, 2 – Carbon-Graphite Base, 3 – Graphite Crucible, 4 – TDJF-1002 Transformer, 5 – Graphite Electrode, 6 – Bottom Current Lead, 7 – Electrode Movement Mechanism

The quality of the obtained calcium carbide was determined by measuring the liters – the amount of C_2H_2 (dm^3/kg) emitted by the interaction of 1 kg of calcium carbide with water.¹⁷ This study used calcined phosphorite from the Chilisai deposit containing, in mass fraction: 23.8% of $Ca_3(PO_4)_2$, 57.3% of SiO_2 , 4.8% of CaO , 3.9% of $CaSO_4$, 3.5% of Fe_2O_3 , 2.7% of CaF_2 , 1.0% of MgO , 2.0% of Al_2O_3 , and 1.0% of other substances, as well as coke with 85.6% of C, 12.2% of ash, and carbon steel shavings.

RESULTS AND DISCUSSION

Figure-2 shows the results concerning the effects of temperature on the equilibrium distribution of silicon, phosphorus, calcium, and iron from the phosphorite. The amount of carbon and iron was calculated assuming a complete reduction of phosphorus, production of ferrosilicon FS45, and calcium carbide in the amount of $230 dm^3/kg$. Figure-2 demonstrates that the main products of reduction in the considered system are FeSi, Si, SiO_g , Si, Fe_2P , FeP, Fe, P_{2g} , P_g , CaC_2 , and Ca_g . In addition, there is a small degree of transition of silicon into Fe_3Si and Fe_5Si_3 . Under temperatures higher than $1,600^\circ C$, silicone mostly transitions into FeSi and Si. The degree of silicon recovery increases with temperature: $1,600^\circ C$ – 52.5%; $1,700^\circ C$ – 66.79%; $1,800^\circ C$ – 72.94%; $1,900^\circ C$ – 83.22%; $2,000^\circ C$ – 82.27%. Phosphorus phosphides are formed at $1,000$ - $1,500^\circ C$, and gaseous phosphorus – at temperatures over $1,300^\circ C$.

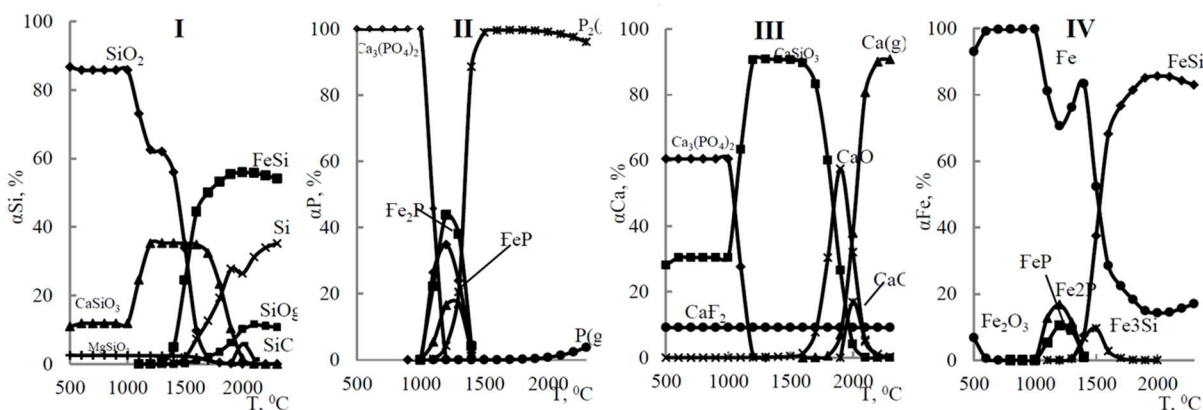


Fig.-2: Effect of Temperature on the Equilibrium Distribution of Silicon (I), Phosphorus (II), Calcium (III), and Iron (IV)

All phosphorus is converted to gas at a temperature of $\geq 1,500^\circ C$. The temperature of the onset of calcium carbide formation is $1,600^\circ C$. This process reaches its maximum at $2,000^\circ C$. Significant calcium losses due to the decomposition of CaC_2 to gaseous calcium and carbon occur at $T > 1,900^\circ C$. The presented material shows that at $1,800$ - $1,900^\circ C$ phosphorus is completely removed from Chilisai phosphorite, and 78-83% of silicon passes into the alloy. However, only 16.9% of calcium is converted into calcium carbide. Therefore, it is necessary to find conditions that ensure maximum extraction of not only silicon and phosphorus but also calcium from phosphorites. The influence of iron on the technological parameters of Chilisai phosphorite processing is presented in Fig.-3.

Increases in the amount of iron up to 45% and temperature to between $1,500$ and $2,000^\circ C$ positively affect the degree of silicon extraction in the alloy. The concentration of silicon in the alloy increases with temperature and naturally decreases with higher amounts of iron. As the amount of iron in the charge grows, the degree of reduction of calcium from Chilisai phosphorite to calcium carbide decreases. A similar situation is observed in the reduction of phosphorus to the gas phase. To determine the optimal conditions providing maximum extraction of Si, P, and Ca from phosphorite with the production of graded products further research involved the use of the rotatable planning method¹⁸ followed by geometrical optimization of technological parameters, described by us earlier in the processing of various natural and man-made raw materials.^{19,20} Independent factors in the study were temperature ($T, ^\circ C$) and the share of iron (Fe, %) in the mass of the phosphorite. The optimization parameters included: the degree of extraction (%) of silicone into the alloy $\alpha_{Si}(\text{alloy})$, of calcium – into calcium carbide $\alpha_{Ca}(CaC_2)$, and phosphorous – into gas $\alpha_P(\text{gas})$,

as well as the amount of calcium carbide (L), dm^3/kg . Tables-2–5 provide planning matrices for the studies and their results.

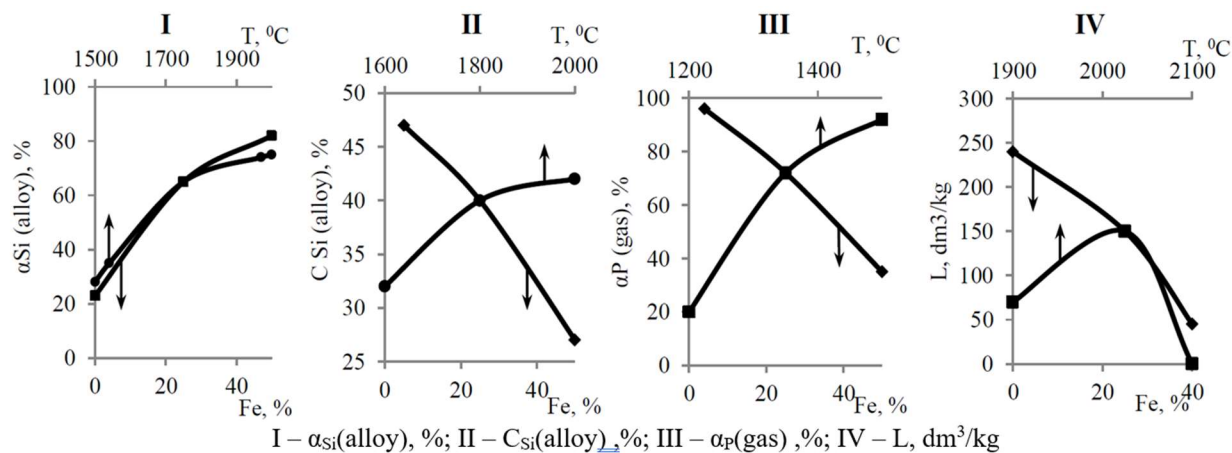


Fig.-3: Influence of Temperature and Amount of Iron on the Nature of Changes in Technological Parameters

Table-2: Planning Matrices and Results of Studies on the Effect of Temperature and Iron on $\alpha_{\text{Si}}(\text{alloy}), \%$

No.	Variables				$\alpha_{\text{Si}(\text{alloy})}$, %
	Coded		Natural		
	X ₁	X ₂	T, °C	Fe, %	
1	-1	-1	1,571	11.5	33.4
2	+1	-1	1,929	11.5	47.5
3	-1	+1	1,571	38.5	57.5
4	+1	+1	1,929	38.5	83.0
5	1.414	0	2,000	25	75.4
6	-1.414	0	1,500	25	36.6
7	0	1.414	1,750	44	80.2
8	0	-1.414	1,750	6	28.3
9	0	0	1,750	25	64.0
10	0	0	1,750	25	64.5
11	0	0	1,750	25	64.9
12	0	0	1,750	25	65.3
13	0	0	1,750	25	65.7

Table-3: Planning Matrices and Results of Studies on the Effect of Temperature and Iron on $C_{\text{Si}}(\text{alloy}), \%$

No.	Variables				C _{Si(ally)} , %
	Coded		Natural		
	X ₁	X ₂	T, °C	Fe, %	
1	-1	-1	1,942	11.5	41.9
2	+1	-1	1,658	11.5	48.2
3	-1	+1	1,942	38.5	30.0
4	+1	+1	1,658	38.5	34.4
5	1.414	0	2,000	25	43.6
6	-1.414	0	1,600	25	32.2
7	0	1.414	1,800	44	29.0
8	0	-1.414	1,800	6	48.3
9	0	0	1,800	25	39.7
10	0	0	1,800	25	39.1
11	0	0	1,800	25	39.3
12	0	0	1,800	25	40.4
13	0	0	1,800	25	40.9

Table-4: Planning Matrices and Results of Studies on the Effect of Temperature and Iron on $\alpha_p(\text{gas})$, %

No.	Variables				$\alpha_{P(\text{gas})}$, %
	Coded		Natural		
	X ₁	X ₂	T, °C	Fe, %	
1	-1	-1	1,244	11.5	53.6
2	+1	-1	1,456	11.5	98.5
3	-1	+1	1,244	38.5	4.6
4	+1	+1	1,456	38.5	90.0
5	1.414	0	1,500	25	96.0
6	-1.414	0	1,200	25	9.2
7	0	1.414	1,350	44	35.1
8	0	-1.414	1,350	6	92.2
9	0	0	1,350	25	74.1
10	0	0	1,350	25	74.3
11	0	0	1,350	25	75.0
12	0	0	1,350	25	73.6
13	0	0	1,350	25	73.0

Table-5: Planning Matrices and Results of Studies on the Effect of Temperature and Iron on $\alpha_{Ca}(\text{CaC}_2)$, %

No.	Variables				$\alpha_{Ca}(CaC_2)$, %
	Coded		Natural		
	X ₁	X ₂	T, °C	Fe, %	
1	-1	-1	1,929	10.6	57.0
2	+1	-1	2,071	10.6	53.4
3	-1	+1	1,929	33.4	13.0
4	+1	+1	2,071	33.4	15.0
5	1.414	0	2,100	22	41.3
6	-1.414	0	1,900	22	43.0
7	0	1.414	2,000	38	3.5
8	0	-1.414	2,000	6	65.0
9	0	0	2,000	22	44.0
10	0	0	2,000	22	44.5
11	0	0	2,000	22	46.2
12	0	0	2,000	22	46.3
13	0	0	2,000	22	37.0

Proceeding from data in Tables-2–5 and relying on the work of Grinfeld and Moiseev,¹⁸ the following regression equations were obtained:

$$\alpha_{Si(\text{alloy})} = -507.86 + 8.19 \cdot 10^{-5} \cdot T \cdot Fe - 1.4310^{-5} \cdot T^2 \cdot Fe + 0.55 \cdot Fe - 2.89 \cdot 10^{-3} \cdot z_2^2 + 1.26 \cdot Fe \quad (5)$$

$$\alpha_p(\text{gas}) = 1906.94 + 2.65 \cdot T - 3.32 \cdot 10^{-5} \cdot T \cdot Fe - 8.69 \cdot T^2 - 2.40 \cdot 10^{-3} \cdot Fe^2 + 0.37 \cdot Fe \quad (6)$$

$$\alpha_{Ca}(\text{CaC}_2) = -1892.27 + 1.72 \cdot 10^{-4} \cdot T \cdot Fe - 5.11 \cdot 10^{-5} \cdot T^2 + 1.99 \cdot Fe - 5.04 \cdot 10^{-3} \cdot Fe^2 + 3.08 \cdot Fe \quad (7)$$

$$C_{Si(\text{alloy})} = -133.75 + 1.7 \cdot 10^{-4} \cdot T - 2.48 \cdot 10^{-5} \cdot T \cdot Fe - 4.03 \cdot 10^{-6} \cdot T^2 - 2.39 \cdot 10^{-4} \cdot Fe^2 + 7.61 \cdot 10^{-3} \cdot Fe \quad (8)$$

Using the equations 5-8 by the method presented by V.F. Ochkov¹⁹ and A.M. Inkov *et al.*²⁰ we constructed volumetric and planar images of changes in $\alpha_{Si}(\text{alloy})$, $\alpha_p(\text{gas})$, $\alpha_{Ca}(\text{CaC}_2)$, and $C_{Si}(\text{alloy})$ depending on temperature and the amount of iron (Fig.-4).

As can be seen from Fig.-4, an increase in temperature and iron content positively affects $\alpha_{Si}(\text{alloy})$ in the temperature range of 1,665-2,000°C and with 24-44% of iron, in which case $\alpha_{Si}(\text{alloy})$ amounts to 70-90%. The concentration of silicon in the alloy varies from 24.8 to 51.6%. According to the silicon content in the temperature range of 1,600-1,770°C and with 34.5-44% of iron, an alloy containing 41-47% (FS 45) is formed in a fairly wide temperature range (1,600-2,000°C) at 6-29.3% of iron. Ferrosilicon of FS50 grade is formed in the temperature range of 1,775-2,000°C with a relatively small amount of iron (6-18%). The

complete transition of phosphorus from phosphorite to the gas phase occurs along the fn line, i.e., with temperature increase from 1,380°C (at 6% of iron) to 1,500°C (at 23.6% of iron). Calcium is maximally transferred from phosphorite to CaC_2 at 1,975°C with 6% of iron.

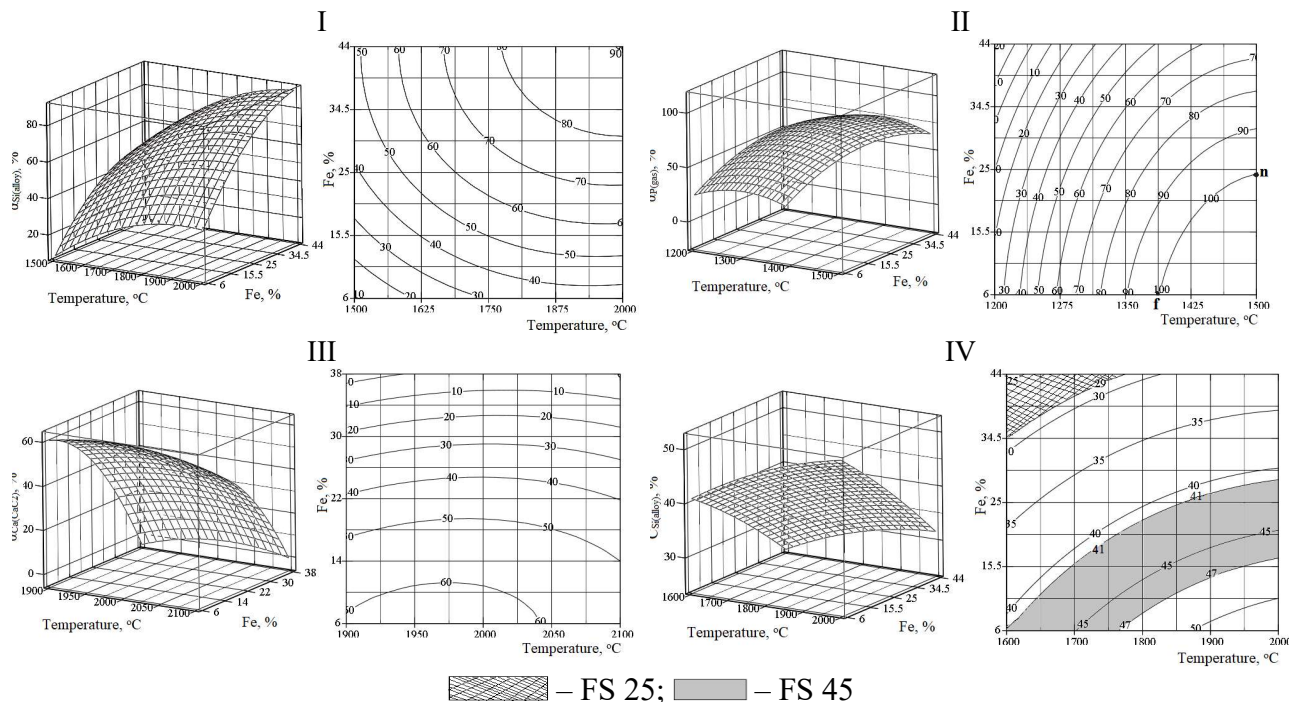


Fig-4: Volumetric and Planar Illustrations of the Effect of Temperature and Iron on $\alpha_{\text{Si(alloy)}}$, (I), $\alpha_{\text{P(gas)}}$, % (II), $\alpha_{\text{Ca(CaC}_2\text{)}}$, % (III), and $\text{C}_{\text{Si(alloy)}}$, % (IV)

The extremum of $\alpha_{\text{Ca(CaC}_2\text{)}}$ at 1,975°C is associated with the decomposition of calcium carbide into gaseous calcium and carbon.¹⁷ The equation for the effect of temperature and iron on the amount of calcium carbide (L , dm^3/kg) is as follows:

$$L = -22323.11 + 1.3 \cdot 10^{-3} \cdot T \cdot \text{Fe} - 5.64 \cdot 10^{-4} \cdot T^2 + 22.54 \cdot \text{Fe} - 0.28 \cdot \text{Fe}^2 - 21.53 \cdot \text{Fe} \quad (9)$$

Figure-5 suggests that calcium carbide at high relative volume ($>200 \text{ dm}^3/\text{kg}$) is formed with relatively low iron content (6-22%) in the temperature range of 1,930-2,080°C. The maximum volume of calcium carbide ($230 \text{ dm}^3/\text{kg}$) is noted at 2,000°C in the presence of 6% of iron.

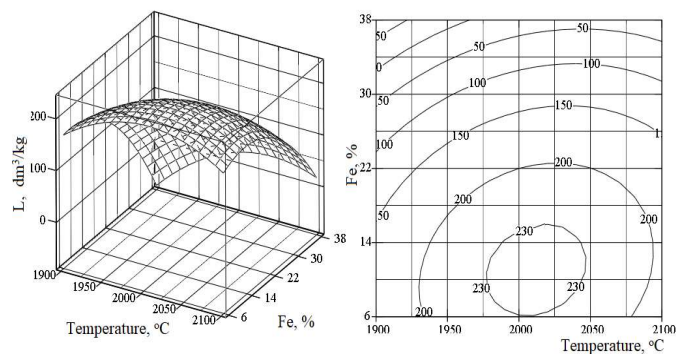


Fig-5: Volumetric and Planar Illustrations of the Effect of Temperature and Iron on the Amount of Calcium Carbide in Liters

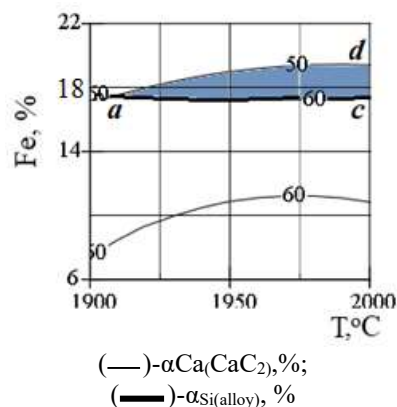


Fig-6: The Combined Information on the Effect of Temperature and Amount of Iron on Technological Parameters

To determine the optimal conditions for processing Chilisai phosphate rock, the effects of temperature and iron on the technological parameters of extraction and concentration must be considered together. The

difficulty in finding this mode lies in the opposite nature of changes in the extraction of Si in the alloy and Ca in the calcium carbide and its volume.

Figure-6 demonstrates the combined effect of temperature and the amount of iron on the process parameters under the condition of $\alpha_{\text{Si}}(\text{alloy}) \geq 60\%$ and $C_{\text{Ca}}(\text{CaC}_2) \geq 50\%$. The values of the parameters in the area *acd* in which these conditions are satisfied are given in Table-6.

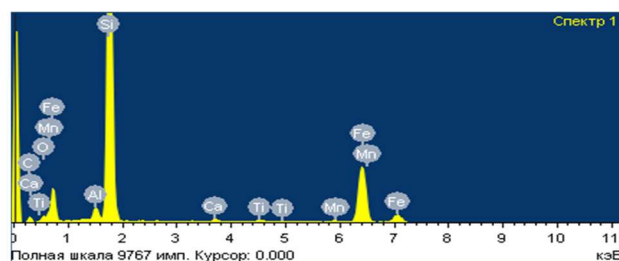
Table-6: Technological Parameters at the *acd* Area Boundary Points

Point in Fig.-6	T, °C	Fe, %	$\alpha_{\text{Si}}(\text{alloy})$, %	$\alpha_{\text{Ca}}(\text{CaC}_2)$, %	$C_{\text{Si}}(\text{alloy})$, %	(L), dm ³ /kg
a	1,900	17.0	60.0	50.9	45.9+	150
c	2,000	16.8	60.0	56.4	45.0+	215
d	2,000	19.8	60.0	50.0	47.8+	208

Table-6 and Figure-6 evidence that with the satisfaction of the conditions $\alpha_{\text{Si}}(\text{alloy}) \geq 60\%$ and $C_{\text{Ca}}(\text{CaC}_2) \geq 50\%$ (50.0-56.4%), the resulting alloy contains 45.0-47.8% of Si and the carbide reaches the volume of 150-215 dm³/kg. Notably, in this case, phosphorus completely turns into gas in the form of P₂ and the degree of aluminum reduction does not exceed 60-70%. The content of aluminum in the alloy amounts to 1.6-1.9%. During the electric smelting of Chilisai phosphorite, the degree of silicon extraction into the ferroalloy is 73.8%. This indicator is 73.8-60=13.8% higher than that under-equilibrium conditions. This is due to the fact that thermodynamic analysis does not account for the presence of blast furnace gas, which traps gaseous SiO, SiO₂, and Si. Therefore, the degree of silicon extraction into the alloy during smelting is higher than that obtained through thermodynamic modeling. The obtained ferroalloy and calcium carbide are shown in Fig.-7. By the psychometric method, based on the density of the obtained ferroalloys (5.5-5.1 g/cm³) and using equation 8, the content of silicon in the alloy is found to be 40.1-44.7%. Figure-8 showing SEM analysis of the ferroalloy reveals that the alloy contains 43.84% of silicon. Based on this indicator, the alloy corresponds to the FS 45 grade of ferrosilicon.²¹



I – ferroalloy, II – calcium carbide
Fig.-7: Products of Chilisai phosphorite smelting



Element	C	O	Al	Si
Weight, %	13.69	3.97	2.02	43.84
Element	Ca	Ti	Mn	Fe
Weight, %	0.5	0.6	0.63	34.75

Fig.-8: SEM Analysis of the Ferroalloy

The obtained calcium carbide at the volume of 200-252 dm³/kg, in accordance with GOST 1460-2013,²² belongs to the third and second grades. The extraction of calcium in carbide was 62-68% and of phosphorus in gas – 99.0-99.4%. The obtained calcium carbide without a grade ($L < 230$ dm³/kg) can be recommended for use in vegetable production. For example, the application of calcium carbide to 1 ha of podzolic loamy soil increased yields of cucumbers by 30-50%.²³⁻²⁵ Based on the results of research on the processing of phosphate rock, a patent was received.²⁶

CONCLUSION

The study of the interaction of Chilisaï phosphorite with carbon, coke, and iron lead showed:

1. In equilibrium conditions:

- increase in the amount of iron at 1,500-2,000°C enhances the extraction of silicon in the alloy, but also reduces the extraction of calcium in the calcium carbide, the concentration of silicon in the alloy, and the volume of calcium carbide;
- in the temperature range of 1,900-2,000°C in the presence of 16.8-19.8% of iron, phosphorus is completely converted into gas, the resulting alloy contains 45.0-47.8% of Si, and carbide reaches the volume of 150-215 dm³/kg (with the extraction of 60-63.6% of Si in the alloy and of 50-56.4% of Ca in calcium carbide).

2. Electric smelting of phosphorite in an arc furnace produces ferrosilicon of grade FS45 (40-44.7%) with the extraction of 73.8% silicon in it, as well as calcium carbide of up to the second grade in the amount of 200-252 dm³/kg. Phosphorus is almost completely (99.0-99.4% of Si) reduced during electric smelting and transformed into the gas phase

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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