Original article / Оригинальная статья
DOI: http://dx.doi.org/10.21285/1814-3520-2020-5-1137-1149

Improving the quality of silicon metal by the method of x-ray radiometric separation of raw material and finished products

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Abstract: In this research, we investigate the process of X-ray radiometric separation of both raw materials (quartz, carbonaceous reducing agent) used for silicon smelting in ore-smelting furnaces and the resulting smelting products. The research objects were quartz from the Aktas field (Kazakhstan), coal from the Shubarkol field and silicon metal of various grades smelted at the Tau-Ken Temir LLP (Karaganda, Kazakhstan). X-ray diffraction analysis was performed using a Philips powder diffractometer. To determine the SiO_2 and Fe_2O_3 content, an ARL PERFORM'X X-ray fluorescence spectrometer was used. To remove impurities, a CPF1-150M single-strand radiometric separator was used. We found that the radiometric separation of original quartz samples with the Fe_2O_3 content of ~ 0.1–0.15% produces pure quartz with the Fe_2O_3 content of ≤ 0.05% and a yield of 65–70%. Provided that the Fe_2O_3 content in the original quartz sample does not exceed 0.5%, concentrates with the Fe_2O_3 content of 0.05% and a yield of 35–55% can be obtained. The yield of pure quartz with the Fe_2O_3 content of 0.01% does not exceed 15–20%. The use of radiometric separation is established to reduce the amount of phosphorus in the final product by 2–3 times. This method is effective for obtaining coal concentrates of varying ash content (2.0, 4.1 and 7.3%); the resulting concentrated product obtained with a yield of 25% contains 1.5% of ash. Separation of silicon metal (with the initial iron content of 1.2–1.5%) yields a product matching silicon grade 773 (product yield ~ 50%), 553 (~ 35%) or 441 (20%). It is concluded that radiometric separation allows the content of impurities in quartz, silicon metal and coal ash to be reduced, thus facilitating the production of higher-grade silicon.

Keywords: quartz, coal, silicon metal, X-ray radiometric separation, impurity removal

Acknowledgements: The authors express their deep gratitude to the top managers of JSC Tau-Ken Samruk National Mining Company, the parent company of Tau-Ken Temir LLP. We appreciate the help of R.A. Akberdin, the Chief Production Officer of the JSC Tau-Ken Samruk National Mining Company, who supported the funding of this research within the contract no. 472/PPC-18 as of November 19, 2018 "Investigation of the possibility of separating lump technical silicon from a slag dump of silicon production and improving the quality of coal based on the technology of X-ray radiometric separation". We thank all the operators and employees of Tau-Ken Temir LLP, employees of Irgiredmet JSC (Irkutsk, Russia), who showed patience and understanding in carrying out this work and we hope to continue mutually beneficial cooperation.

Information about the article: Received August 10, 2020; revised September 22, 2020; accepted for publication on October 30, 2020

For citation: Zobnin NN, Korobko SV, Vetkovsky DL, Moiseev AA, Aganin AO, Nurumgaliev AKh, Pikalova IA. Improving the quality of silicon metal by the method of x-ray radiometric separation of raw material and finished products. *Vestnik Irkutskogo gosudarstvennogo tehnicheskogo universiteta = Proceedings of Irkutsk State Technical University*. 2020;24(5):1137–1149. https://doi.org/10.21285/1814-3520-2020-5-1137-1149

УДК 669.782

Повышение качества металлического кремния методом рентгеновского радиометрического разделения сырьевых и готовых продуктов

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Резюме: Цель – исследовать процесс рентгенорадиометрического разделения сырьевых материалов (кварца, углеродистого восстановителя), используемых для выплавки кремния в руднотермических печах, и самого продукта плавки. Объектами исследования явились кварц месторождения Актас (Казахстан), уголь Шубаркульского месторождения и металлический кремний различных марок, выплавленный в ТОО «Tau-Ken Temir» (г. Караганда, Казахстан). Рентгенофазовый анализ проводили с помощью порошкового дифрактометра фирмы «Philips». Для определения содержания SiO_2 и Fe_2O_3 использовали рентгенофлюоресцентный спектрометр ARL PERFORM'X. Для удаления примесей применяли одноручьевой рентгенорадиометрический сепаратор типа СРФ1-150М. Установлено, что при радиометрическом разделении образца исходного кварца с содержанием Fe_2O_3 ~ 0,10–0,15% обеспечивается получение чистого кварца с содержанием Fe_2O_3 менее 0,05% при выходе продукта 65–70%. При содержании в исходном кварце Fe_2O_3 до 0,5% также возможно получение концентратов с содержанием Fe_2O_3 0,05% при выходе чистого продукта 35–55%. Чистый кварц с содержанием Fe_2O_3 0,01% возможно получить с выходом лишь 15–20%. Показано, что при использовании радиометрического метода разделения достигается уменьшение фосфора в кварце в 2–3 раза. Уголь с различной зольностью (2,0, 4,1 и 7,3%) эффективно обогащается предложенным методом с получением концентрата, содержащего 1,5% золы, при выходе 25%. При обогащении металлического кремния (с исходным содержанием железа 1,2–1,5%) может быть получен продукт, соответствующий сорту кремния 773 (с выходом продукта ~ 50%), сорту 553 (с выходом ~ 35%) или сорту 441 (с выходом 20%). Радиометрический метод обогащения материалов позволяет снизить содержание основных примесей в кварце, металлическом кремнии и золе угля, что способствует получению кремния высших марок.

Ключевые слова: кварц, уголь, металлический кремний, рентгено-радиометрическое разделение, удаление примесей

Благодарности: Авторы выражают огромную благодарность руководству АО «Национальная горнорудная компания «Тау-Кен Самрук», которая является материнской компанией ТОО «Tau-Ken Temir». Особую благодарность выражаем генеральному директору по производству АО «НГК Тау-Кен Самрук» Акбердину Р.А., который поддержал финансирование исследований в рамках исполнения контракта № 472 / РРС-18 от 19 ноября 2018 г. по теме: «Исследование возможности разделения кускового технического кремния из шлакоотвала кремниевого производства и повышения качества углей на основе технологии рентгенорадиометрической сепарации». Благодарим всех операторов и сотрудников ТОО «Tau-Ken Temir», сотрудников АО «Иргиредмет» (г. Иркутск, Россия), проявившим терпение и понимание при проведении данной работы, и надеемся на продолжение взаимовыгодного сотрудничества.

Информация о статье: поступила в редакцию 10 августа 2020 г.; поступила после рецензирования и доработки 22 сентября 2020 г.; принята к публикации 30 октября 2020 г.

Для цитирования: Зобнин Н.Н., Коробко С.В., Ветковский Д.Л., Моисеев А.А., Аганин А.О., Нурумгалиев А.Х., Пикалова И.А. Повышение качества металлического кремния методом рентгеновского радиометрического разделения сырьевых и готовых продуктов. *Вестник Иркутского государственного технического университета.* 2020. Т. 24. № 5. С. 1137–1149. https://doi.org/10.21285/1814-3520-2020-5-1137-1149

INTRODUCTION

Silicon is widely used for various industrial purposes, e.g. as an agent for casting alloys, silicon bronzes, steel and cast iron or as a basis for obtaining organosilicon compounds, polycrystalline silicon and materials for 3D technologies [1–6]. The production of metal silicon in ore-smelting furnaces is steadily increasing globally, thus stimulating research into the development of novel smelting technologies based on the use alternative raw materials for improving the quality of smelted silicon and solving as-

sociated environmental problems [7–13].

Iron impurities in silicon metal present a challenging industrial problem. When the quartz components are reduced, the entire amount of iron passes into the liquid silicon phase. Oxidative refining cannot be used for removing iron, since the affinity of silicon for oxygen is higher than that of iron. Coal and its coking products are another source of iron. Tab. 1 presents the material balance calculated during the process of smelting silicon metal under the conditions of Tau-Ken Temir LLP (Karaganda, Kazakhstan) in January 2018.

It can be seen that the vast amount of iron is transferred in silicon metal from quartz and, to some extent, from coal and its thermal treatment products. In order to improve the quality of silicon metal, iron should be removed from these starting materials. In addition, the contamination of silicon metal with iron can occur while transferring the product from the furnace into the casting ladle using steel instruments. Therefore, it is also necessary to purify the finished product after its crushing and sifting into commercial fractions. A method for separating quartz from iron using magnetic and electrostatic separation was described in [14, 15]. Its disadvantage consists in the need to provide a high level of quartz grinding, which cannot be used for smelting silicon metal in a furnace with a submerged electric arc. Quartz can also be purified by flotation [16, 17], a combination of magnetic separation and acid leaching [18, 19], as well as by gravity separation [20, 21]. However, both these methods require the use of water and aqueous solutions, which complicates the process in winter and involves additional expenses for chemical reagents. The most effective method is likely to be X-ray radiometric separation, which requires neither grinding of the raw materials, nor use of aqueous media and chemical reagents [22].

EXPERIMENTAL

To study the process of removing impurities, quartz of Aktas deposits (Kazakhstan) was used. At the moment, the process of mining of quartz does not exclude impurity rock. We can distinguish 5 groups of pollution (impurity rocks): “black” quartz – fig. 1, “ferruginous” quartz – fig. 2, granite – fig. 3, “ruby” quartz – fig. 5, “burgundy” quartz – fig. 6. The chemical impurity of rocks is given in tab. 2, in the same place the composition of the main mass of quartz, in fig. 4.

The X-ray radiometric properties of samples were studied using X-ray diffraction analyzers with CuKα radiation (40 kV, 30 mA) in the range 2θ) = 10–70^{o} at a goniometer rate of 2θ = 2 ^{o}/min. The content of SiO_2 and Fe_2O_3 in quartz was examined by, XRF (X-ray Fluorescence) spectrometry using an ARL PERFORM’X instrument.

Table 1. Material balance of iron in the process of smelting silicon metal at the LLP Tau-Ken Temir in January 2018
Таблица 1. Материальный баланс железа в процессе выплавки кремния металла ТОО «Tau-Ken Temir», январь 2018 года

Raw material							
Material	Expendi pendi-ture rate, t/t	Ash, %	Fe_2O_3 in ash, %	Fe_2O_3 in the material, %	Fe, %	Go Fe into	
						t	%
Quartz	3.20	–	–	0.17	0.12	3.96	63.29
Charcoal	0.64	2.04	2.90	0.05	0.04	0.26	4.23
Coal	0.93	4.10	4.41	0.18	0.12	1.17	18.79
Semicoke	0.35	7.40	4.41	0.32	0.22	0.79	12.76
Graphite electrode	0.11	0.10	19.00	0.02	0.01	0.01	0.23
Wood chips	0.84	0.25	2.90	0.00725	0.005	0.04	0.68
Total	6.07					6.26	100.00

Finished products					
Products	Expenditure rate, t/t	Fe_2O_3 in the material, %	Fe, %	Go Fe out	
				t	%
Silicon metal	1.0000	–	0.5836	5.8360	93.1644
Slag	0.0885	0.4168	0.2918	0.2582	4.1228
Microsilica	1.2138	0.0200	0.0140	0.1699	2.7126
Total	–	–	–	6.2641	100.0000

Table 2. The chemical composition of varieties of quartz
Таблица 2. Химический состав разновидностей кварца

Name	Chemical composition, %				
	Fe_2O_3	Al_2O_3	CaO	TiO_2	SiO_2
The bulk of quartz	0.02	0.14	0.003	0.003	99.83
Granite	0.62	5.46	0.108	0.026	93.79
"Ferrous" quartz	1.23	0.16	0.011	0.005	98.60
"Black" quartz	5.92	3.83	0.129	0.146	89.98
"Ruby"quartz	0.28	0.08	0.003	0.004	99.64
"Burgundy"quartz	24.01	0.19	0.028	0.017	75.75

Fig. 1. "Black" quartz
Рис. 1. «Черный» кварц

Fig. 2. "Ferrous" quartz
Рис. 2. «Железистый» кварц

Fig. 3. Granite
Рис. 3. Гранит

Fig. 4. The bulk of quartz
Рис. 4. Основная масса кварца

Fig. 5. "Ruby" quartz
Рис. 5. «Рубиновый» кварц

Fig. 6. "Burgundy" quartz
Рис. 6 «Бордовый» кварц

To remove impurity rocks, a single-strand X-ray radiometric separator of the SRF1-150M type was used. This is a new generation of Angara-type separators (developed by the Irgiredmet JSC and the Tekhnosort LLC) equipped by a highly sensitive measuring system with semiconductor detectors of reflected X-ray radiation. Fig. 7 and 8 present the signal flow and circuit operation diagrams, as well as the appearance of this equipment. The removal of impurities proceeds as follows. The feeding mechanism provides dosed continuous unloading of raw materials to the tray design, which forms the flow of raw materials with a discrete feed to the zone of measurement and sorting in the free fall mode.

The detector unit makes an assessment of the material composition of the raw material by the X-ray fluorescence method on the basis of the secondary X-ray spectrum recorded from the bulk sample, comparing the measurement result with the threshold and issuing a control signal to the pusher actuation.

When quality requirements are not met, an electromagnetic or electropneumatic slide type pusher changes the falling trajectory of the sample. Samples of quality raw materials fall without deviating their trajectory. The control signal for duration and impact force is proportional to the linear size of the sorted sample. To create a control signal, spectral ratios for iron, calcium and strontium were used:

$$P_{Ca} = N_{Ca}/ N_S; P_{Fe} = N_{Fe}/ N_S; P_{Sr} = N_{Sr}/ N_S; \quad (1)$$

where: N_{Ca}, N_{Fe}, N_{Sr} are the number of pulses of characteristic X-ray radiation of the elements Ca (3.7 keV), Fe (6.4 keV) and Sr (14.2 keV) reflected from a freely falling sample of raw material; N_S is the secondary scattered X-ray radiation recorded from a sample of raw material together with the characteristic radiation of the elements contained there in.

Initially, the samples were estimated according to the statistical distribution of the intensity of reflected characteristic X-radiation. This assessment was carried out on such a generalized mass of samples that the statistical distribution did not change its character within a statistical error of 5%. The obtained mass was established as the limit of its representativeness from the volume of the studied sample. The validity of checking this limit was verified in static and dynamic conditions. Next, an analysis was made of the statistical distribution of the magnitude of the spectral ratio within the variation range in order to identify the threshold separation value. As a criterion that determined the values of the threshold spectral ratio, the yield of concentrates and tailings was used. The yield was estimated based on a histogram of statistical distribution. In addition, the value of X-ray reflected radiation most pronounced the obtained statistical distribution was determined. On the basis of this analysis, the element contained in the material under study by which the division should be made was determined, as well as the separation principle. After that, control enrichment of a representative sample was performed. The resulting separation products were averaged, reduced and subjected to chemical analysis. In cases where the result was unsatisfactory, the yield of the separation products was changed by varying the threshold spectral ratio, the element used for the separation and the separation principle. When splitting into two products, a one-step scheme was used with a single value of the separation criterion. When splitting into three products, a two-stage scheme was used, etc. These studies were conducted on different size classes, because the size of raw materials also affects the separation rate.

RESULTS AND DISCUSSION

Fig. 9 shows the ratio between the number of pulses of reflected X-ray radiation from the sample under examination over the measurement period and the frequency, keV. As can be seen from the graph, there is a good agreement between the magnitude of the peak in the region of the characteristic X-ray frequency of iron and its content in quartz. The maximum peak of 1350 pulses was observed from “black” quartz with an iron content of 5.92%. The minimum peak of 150 pulses corresponds to “ruby” quartz with an iron content of 0.28%. Based on this relationship, the spectral ratio P_{Fe} was chosen as the sorting criterion during separation of the investigated material. Tab. 3 presents the results of separation obtained by the method described above.

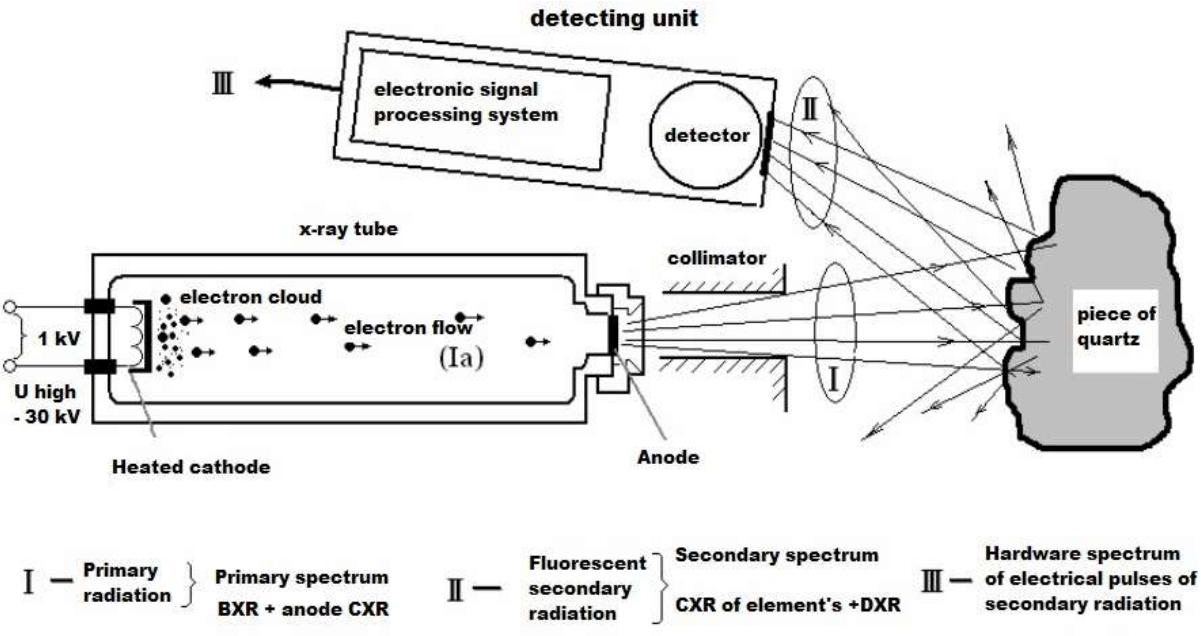

Fig. 7. Schematic diagram of signal movement in an SRF1-150M X-ray radiometric separator:
BXR – Brake X-ray; CXR – Characteristic X-ray; DXR – Diffuse X-ray
Рис. 7. Принципиальная схема движения сигналов в рентгенорадиометрическом сепараторе СРФ1-150М:
BXR – тормозной рентгеновский снимок; CXR – характеристический рентгеновский снимок;
DXR – диффузный рентгеновский снимок

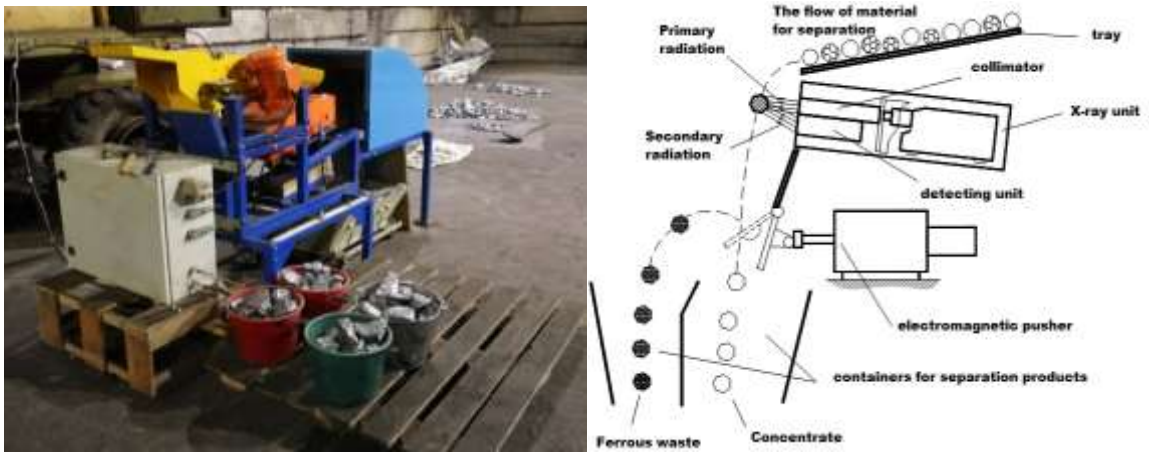

Fig. 8. Schematic diagram of the operation and appearance of an SRF1-150M X-ray radiometric separator
Рис. 8. Принципиальная схема работы и внешний вид рентгенорадиометрического сепаратора СРФ1-150М

As can be seen from the results presented in tab. 3, quartz with a Fe_2O_3 content of less than 0.05% with a yield of 65–70% was obtained from quartz with the initial Fe_2O_3 content of 0.10–0.15%. Provided that the initial quartz contains up to 0/5% of Fe_2O_3 the yield of pure product reaches 35–55%. Pure quartz with a Fe_2O_3 content of 0.01% can be obtained with a yield of 15–20%. The process enables removal of such impurities –aluminium and calcium oxides, phosphorus. In comparison with the initial concentration, a two- or three-fold decrease in the amount of phosphorus can be achieved. It should be noted that the best enrichment conditions are achieved when separating the feedstock into fractions of 20–40 and 40–80 mm. With the enrichment of the quartz fraction of 20–80 mm, the yield of a suitable product decreases from 70 to 50%.

When separating coal from the Shubarkul deposit (Kazakhstan), similar results were obtained (tab. 4). This coal is also used in the production of silicon metal at the Tau-Ken Temir LLP.

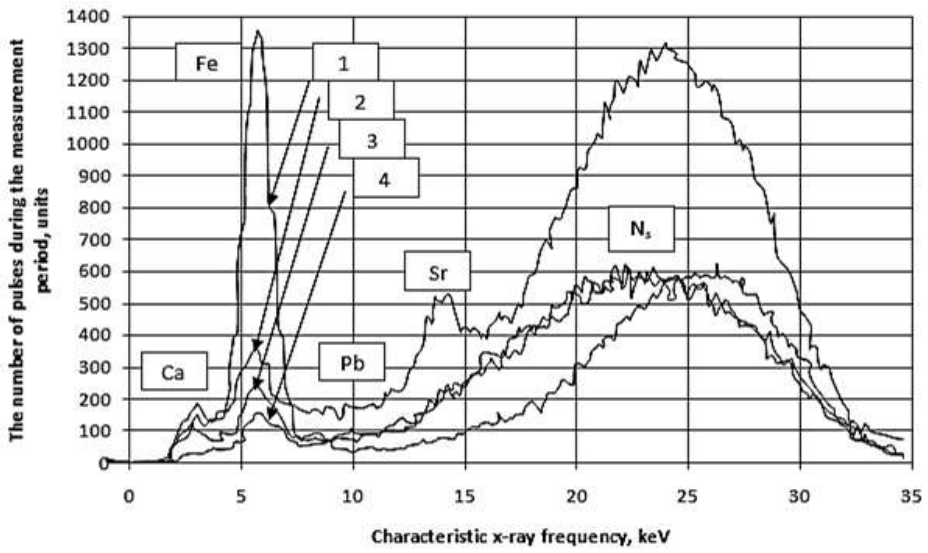

Fig. 9. Dependence of the number of pulses of reflected X-ray radiation over the measurement period on frequency, keV: 1 – "Black" quartz; 2 – "Ferrous" quartz; 3 – Granite; 4 – "Ruby" quartz
Рис. 9. Зависимость количества импульсов отраженного рентгеновского излучения за период измерения от частоты: 1 – «черный» кварц; 2 – «железистый» кварц; 3 – гранит; 4 – «рубиновый» кварц

The studies were carried out using the separation of spectral ratios by iron and strontium as a criterion. High-quality ash removal was achieved when using the spectral ratio for iron as a criterion for the separation. As can be seen from tab. 4, coal can be effectively enriched by the X-ray radiometric separation. Regardless of the initial concentration (2.0, 4.1, 7.3%), coal concentrate can be obtained from the content of 1.5% and yield of 25%.

Using a similar technique, separation of silicon metal was carried out. The respective results and separation curves (lines of Henri Poincaré) are presented in fig. 10–12. As can be seen from fig. 10–12, silicon metal with the initial iron content of 1.2–1.5% can be obtained as separation products corresponding to 773 metal silicon grade and a yield of 50%, 553 metal silicon grade with a yield of 35% or 441 metal silicon grade with a 20% yield. This depends on the demand for and cost of a particular metal silicon grade. Silicon metal of the grade 553 with a yield about 20% can be produced from silicon metal of the grade 773 (initial iron content of 0.6%). Silicon metal of the grade 4403 can be obtained from silicon metal of the grade 3301 with a yield of about 60%. Moreover, the lower the initial content of harmful impurities in silicon metal, the lower the purifying effect that can be achieved as a result of X-ray radiometric separation. This method is most effective when purifying ferrous silicon with ferrous inclusions obtained in the process of release from the furnace using steel tools.

In addition, experiments were carried out on the X-ray radiometric separation of slag produced in the smelting of silicon metal. The separation of the enrichment products by X-ray spectra was qualitative. In addition to the elemental silicon, the oxide component is present in the concentrates. The enrichment wastes in the oxide base contain small (0–10 mm) particles of elemental silicon. Visually, the oxide part in the concentrate is represented by inclusions of unreacted quartz rather than by slag. The spectrum of quartz is close to that silicon; therefore, it was not separated during purification. The presence of the initial quartz in the slag points to massive violations of the technological regime during smelting of silicon metal. Such violations are described in [23]. The methods described in this paper [23] can be used to avoid the possibility of contamination of slags with quartz and to obtain concentrates without oxide inclusions during X-ray radiometric separation. This product can already be defined as a silicon metal of a certain brand. In this regard, the authors plan to return to this work after the implementation of the above recommendations.

Table 3. The results of X-ray radiometric separation of quartz from the "Aktas" deposit
Таблица 3. Результаты рентгенорадиометрической сепарации кварца месторождения «Актас»

Fraction of quartz, mm	Experiment no.	P_{Fe}	Product name	Weight, kg	Product yield,%	Chemical composition, %					
						Fe_2O_3	Al_2O_3	CaO	TiO_2	P_2O_5	SiO_2
-80+40	1	0.030/0.038	Concentrate	16.4	25.1	0.03	0.09	0.02	0.0032	0.0027	99.76
			Intermediate product	27.2	41.6	0.06	0.31	0.06	0.0105	0.0043	99.45
			Waste	21.8	33.3	0.33	0.74	0.51	0.030	0.0112	98.27
			Source quartz	65.4	100.0	**0.142**	**0.398**	**0.200**	**0.015**	**0.006**	**99.135**
	2	0.030/0.042	Concentrate	31.5	63.0	0.02	0.12	0.02	0.0044	0.0027	99.74
			Intermediate product	6.0	12.0	0.11	0.52	0.07	0.0210	0.0066	99.17
			Waste	12.5	25.0	0.47	1.17	0.51	0.061	0.0231	97.67
			Source quartz	50.0	100.0	**0.143**	**0.431**	**0.149**	**0.021**	**0.008**	**99.154**
	3	0.040	Concentrate	22.6	58.9	0.04	0.19	0.03	0.0051	0.0034	99.63
			Waste	15.8	41.1	0.18	0.69	0.05	0.025	0.0066	98.94
			Source quartz	38.4	100.0	**0.098**	**0.396**	**0.038**	**0.013**	**0.005**	**99.346**
-40+20	4	0.040	Concentrate	18.9	52.1	0.05	0.21	0.03	0.0061	0.0046	99.60
			Waste	17.4	47.9	0.17	0.72	0.13	0.0297	0.0119	98.82
			Source quartz	36.3	100.0	**0.107**	**0.454**	**0.078**	**0.017**	**0.008**	**99.226**
	5	0.045	Concentrate	14.1	42.9	0.04	0.28	0.02	0.0082	0.0021	99.55
			Waste	18.8	57.1	0.46	0.91	0.52	0.0412	0.0174	97.96
			Source quartz	32.9	100.0	**0.280**	**0.640**	**0.306**	**0.027**	**0.011**	**98.642**
	6	0.028/0.038	Concentrate	16.2	39.2	0.02	0.17	0.05	0.0059	0.0040	99.65
			Intermediate product	12.8	31.0	0,07	0.31	0.05	0.0127	0.0048	99.46
			Waste	12.3	29.8	0.35	0.93	0.46	0.043	0.0184	98.09
			Source quartz	41.3	100.0	**0.134**	**0.440**	**0.172**	**0.019**	**0.009**	**99.126**
-80+20	7	0.042	Concentrate	9.8	37.7	0.05	0.20	0.033	0.009	0.0001	99.62
			Waste	16.2	62.3	0.61	0.53	0.172	0.028	0.0200	98.54
			Source quartz	26.0	100.0	**0.399**	**0.406**	**0.120**	**0.021**	**0.012**	**98.947**
	8	0.042	Concentrate	16.0	40.0	0.03	0.16	0.03	0.0058	0.0035	99.67
			Waste	23.8	60.0	0.44	1.08	0.55	0.0490	0.0203	97.79
			Source quartz	39.8	100.0	**0.276**	**0.712**	**0.342**	**0.032**	**0.014**	**98.542**

Table 4. The results of X-ray radiometric separation of coal from the “Shubarkol” deposit
Таблица 4. Результаты рентгенорадиометрической сепарации угля месторождения «Шубарколь»

Initial coal quality	Fraction of coal, mm	Product name	Sort indicators					
			P_{Fe} – 0,022/0,028			P_{Sr} – 0,026/0,030		
			Weight, kg	Product yield, %	Ash content, %	Weight, kg	Product yield, %	Ash content, %
High	-40+20	Concentrate	11.1	28.2	1.6	11.5	52.0	1.6
		Intermediate product	25.0	63.6	2.0	5.1	23.1	1.8
		Waste	3.2	8.1	2.9	5.5	24.9	1.9
		Source coal	39.3	100.0	2.0	22.1	100.0	1.72
Average	-40+20	Concentrate	10.2	56.7	2.8	5.0	28.2	2.2
		Intermediate product	6.4	35.6	3.5	5.9	33.3	4.5
		Waste	1.4	7.8	16.5	6.8	38.4	4.3
		Source coal	18	100.0	4.1	17.7	100.0	3.77
Low	-40+20	Concentrate	13.1	62.7	3.8	7.5	42.1	5.3
		Intermediate product	5.9	28.2	7.1	5.6	31.5	4.6
		Waste	1.9	9.0	32.1	4.7	26.4	4.5
		Source coal	20.9	100.0	7.42	17.8	100.0	4.87

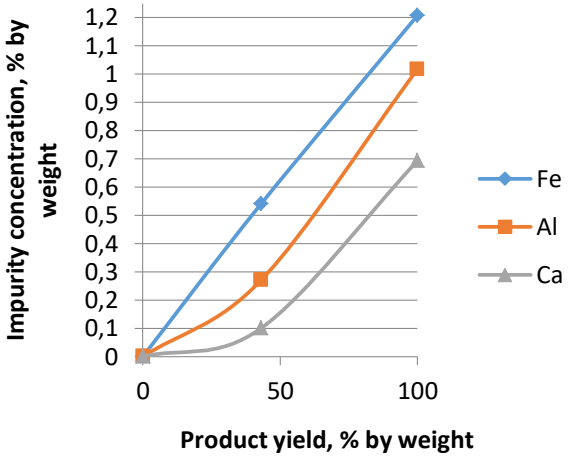

Fig. 10. Ferric silicon enrichment line
Рис. 10. Линия обогащения железистого кремния

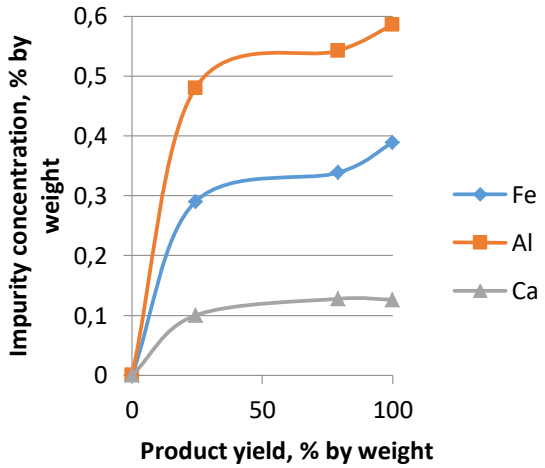

Fig. 11. Silicon Grade 773 Enrichment Line
Рис. 11. Линия обогащения кремния 773

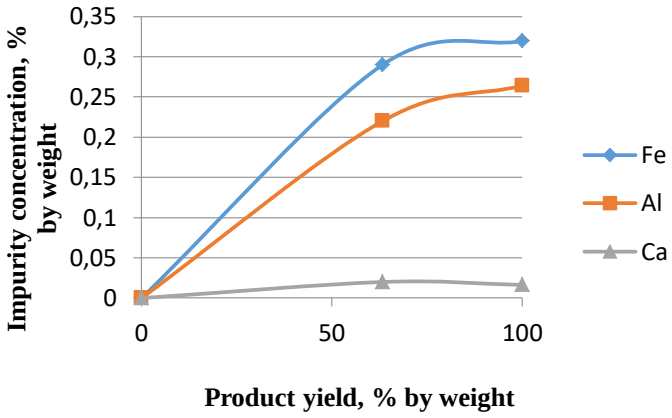

Fig. 12. Silicon Grade 4403 Enrichment Line
Рис. 12. Линия обогащения кремния 4403

CONCLUSIONS

The relationship between the number of reflected X-ray pulses over the measurement period and the frequency of quartz of different chemical composition was established. On the basis of this dependence, an X-ray radiometric separation technique for quartz, coal, metal silicon and silicon slag was developed and tested. This technique solves the practical problem of improving the quality of silicon metal obtained by smelting. The possibility of reducing the concentration of such harmful impurities as iron, titanium and phosphorus in the resulting silicon metal was demonstrated. This can significantly reduce the cost of solar-grade silicon production.

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Зобнин Н.Н., Коробко С.В., Ветковский Д.Л., Моисеев А.А., Аганин А.О., Нурумгалиев А.Х., Пикалова И.А. заявляют о равном участии в получении и оформлении научных результатов и в равной мере несут ответственность за плагиат.

Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this article.

Конфликт интересов

Авторы заявляют об отсутствии конфликта интересов.

The final manuscript has been read and approved by all the authors.

Все авторы прочитали и одобрили окончательный вариант рукописи.

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