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POSSIBILITIES OF USING PULVERIZED WASTE OF HIGH-SILICON GRADES OF FERROALLOYS IN THE PRODUCTION OF SILICON CARBIDE

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Abstract. The article discusses the process of obtaining silicon carbide from the dust of siliceous grades of ferroalloys. The results of crushing charge materials using a planetary centrifugal mill are presented, which made it possible to achieve optimal particle dispersion and improve their reactivity. The crushed charge was subjected to high-temperature processing in a resistance furnace, which contributed to the synthesis of silicon carbide with a high degree of purity. The article presents images of the charge mixture obtained using a scanning electron microscope, which made it possible to study in detail the morphology and structure of the resulting particles. Silicon carbide was studied by X-ray phase analysis, which made it possible to determine its phase composition and confirm the presence of basic phases such as β-SiC and α-SiC. The parameters affecting the synthesis efficiency, such as temperature, processing time, and the ratio of components in the charge, are also considered. The revealed patterns will make it possible to optimize the process of obtaining silicon carbide and expand its use in various industries such as electronics, metallurgy, as well as in the production of abrasive materials and refractory products.

Key words: silicon carbide; dust of siliceous grades; microsilicone; petroleum coke; phases; resistance furnace; planetary centrifugal mill.

Introduction

Currently, the Karaganda region is one of the largest in terms of industrial potential, rich in minerals and raw materials. The region is mainly known for its reserves of the coal basin, and is also famous for its reserves of quartz deposits. The largest and richest deposit is considered to be the «Aktas» deposit. The «Tau-Ken Temir» LLP plant has been operating on the basis of quartz from this deposit since 2013 on German Thyssen Krupp equipment with an annual capacity of 25,000 tons per year of metallurgical silicon. The quartz deposits of the region are also provided by the «Karaganda Ferroalloy Plant of YDD company» with an annual capacity of 240,000 tons per year of high-quality ferrosilicon. At the same time, the production of siliceous materials is accompanied by a large number of dust-like emissions, the return of which to economic circulation is an important source of secondary raw materials. According to the official websites of manufacturers, the smelting of 240,000 tons of ferrosilicon by YDD produces 18,000 tons of dust per year. The dust captured at dust and gas purification and aspiration plants is of economic, environmental and social importance for the region [1, 1353].

Silicon dust, which is subject to return, reduces the cost of ore enrichment, and the availability of raw materials increases as valuable components are extracted in the form of SiO_2. The return of aspiration dust from the silicon industry to metallurgical processing will provide a solution to environmental protection measures, protection of human activity in the region. Currently, a huge amount of stored pulverized waste packed in big bags has gathered on the territory of the enterprises. Efficient use of resources, the development of new technologies and the search for alternative sources of raw materials will help make the metallurgical industry more competitive, sustainable and environmentally friendly. For the region, it is important to solve the problems of recycling waste accumulated on the territory of production facilities.

Dust-like waste generated during the production of silicon is also called microsilicon, microsilica, or siliceous dust. The content of amorphous silicon dioxide - SiO_2 in the form of finely dispersed spherical particles in the composition of the specified metallurgical residual dust exceeds 92% [1, 67].

The dust yield varies widely and presents a difficult problem for manufacturing enterprises smelting siliceous alloys. Dust emission control is an important aspect of production and requires constant attention and monitoring. One of the most promising strategies is the development of methods for the rational use of such types of waste as ore raw materials. This requires a deeper study of the chemical composition and properties of dust waste, as well as the development of technologies and processes for their reuse. It is necessary to pay attention not only to technological aspects, but also to aspects of environmental safety and the economic feasibility of such approaches [3, 5].

The use of innovative methods and technologies can contribute not only to reducing the negative impact on the environment, but also to creating new opportunities for sustainable industrial development. It is also necessary to take into account the social aspects of introducing new practices into production, including staff training, job creation and the development of infrastructure for processing dust waste.

To substantiate the theory that the use of microsilicon to produce silicon carbide, research was conducted to replace quartz with man-made waste. In accordance with this goal, microsilicon produced by «Tau-Ken Temir» LLP was used for the experiment. The object of the study is a by-product of the production of metallurgical silicon, which is formed during the reduction of quartz with carbon in an electric furnace. The process of melting crystalline silicon and ferrosilicon in electric furnaces, although it is a key stage in the production of ferroalloys, during the smelting of steel and semiconductor materials, is accompanied by the formation of a significant amount of dust and gas emissions. The main reason for active gas formation is the incomplete use of the initial quartzite. During smelting, up to 10-15% of the silica (SiO_2) contained in quartzite does not participate in the reaction and is carried away from the furnace in the form of dust mixed with gases. In the process of reducing silicon with carbon, an intermediate product is formed - silicon monoxide (SiO). Part of this SiO rises with the furnace gases upwards, where it is oxidized to SiO_2, forming a fine silica dust. Silica dust, like any industrial dust, poses a serious threat to the environment. In order to minimize the negative impact of dust and gas emissions, it is necessary to apply an integrated approach [4, 333].

Trapping and cleaning: special gas cleaning plants, such as bag filters, electrofilters, scrubbers, allow you to effectively capture dust from furnace gases.

Rational disposal: the captured dust, instead of ending up in landfills, can be recycled and used in other industries. For example, silica dust can be used as an additive in concrete, the manufacture of glass, ceramics and other composite materials, as well as in the production of silicon carbide. Reducing dust and gas emissions from silicon production is an important task, the solution of which requires innovative technologies and a responsible approach to environmental safety. The use of modern gas purification plants and the rational disposal of silica dust can reduce the negative impact on the environment. In this work, the basic object of the ore part of the charge is microsilicon, which is shown in Figure 1.

Materials and methods

The accumulated silica based on the enterprises of «Tau-Ken Temir» LLP and «Karaganda Ferroalloy Plant of YDD company» are labeled MKU-95, MKU85 and MK-80 according to GOST R 58894-2020. On an industrial scale, finished products are obtained by high-temperature reduction of silica from raw materials with a high content of SiO_2 carbon in electric arc furnaces. The charge for melting consists of lumpy raw materials of a certain granulometric composition. According to the results of electric arc melting, finished products and dust trapped in electric filters are obtained. The phase composition of the dust is mainly represented by the crystalline modification of silica - β-cristobalite, as well as magnetite and hematite, α-quartz and silicon carbide (α-CiC and β-SiC).The grate gases have a dust content from 10 to 100 g/m^3, the dust is a finely dispersed material with a bulk mass of 0.18-0.23 t/m^3 with a particle size of up to 5 microns. The chemical compositions of the resulting pulverized waste products of the region are shown below [5, 24].

a) general view б) type of particles

Figure 1 - Microsilicon formed during the smelting of technical silicon or ferrosilicon

To solve the problem of recycling the dust of siliceous alloys, experimental work was carried out to obtain silicon carbide using microsilicon and petroleum coke. A sample of microsilicon was selected for experimental work.

Petroleum coke is a porous, solid material from dark gray to black in color, obtained by coking petroleum raw materials. The elemental composition of crude or non-calcined petroleum coke (in %): 91-99.5 C, 0.035-4 N, 0.5-8 S, 1.3-3.8 (N+O), the rest are metals. Petroleum coke is labeled according to GOST 22898-78 [6, 173].

a) general view б) appearance after abrasion

Figure 2 - Petroleum coke

Silicon carbide is one of the most important inorganic materials, which is widely used for the production of abrasive tools, high-temperature heaters, refractory ceramics and metallurgy. Most of the silicon carbide produced by the global industry is obtained by the method proposed by Acheson at the end of the last century. The essence of the method consists in the carbon-thermal reduction of silica due to the Joule heat released when an electric current passes through the core of the furnace.

The SiC production process is very laborious and requires high energy costs amounting to 7300-

7600 kWh/t. The share of electric energy in the cost structure of silicon carbide of abrasive quality is 50-60%, when loading charge materials 60-70 tons, the yield of marketable products is 10.5-11.5 tons (15-19%). Therefore, ensuring maximum product yield with rational consumption of electrical energy is an important production task. The main criterion for controlling the energy regime is the characteristic of the charge materials and the type of silicon carbide obtained [7, 380]. The dynamics of the thermal state of the furnace bath is influenced by the following factors: energy released on the core of the furnace, energy consumption due to endothermic reactions, the presence of a significant amount of exhaust gases, heat transfer to the environment. Taking into account the location of the core along the entire length of the furnace, the assumption is made about the uniform release of energy from the core surface. When assessing the dynamics of the thermal state of the furnace lining, it was assumed that the heat flows are directed only in the axial direction.

For quite a long time, the needs of metallurgy, refractory and ceramic production in silicon carbide were met by grinding abrasive materials, which unreasonably increased the cost of refractories and ceramics, and in metallurgy in some cases made the use of silicon carbide technologically and economically impractical. The situation was aggravated by the constant increase in electricity prices and the tightening of the requirements of national environmental legislation. In this regard, the main manufacturers of silicon carbide - the companies Saint - Gobian (France), Exolon - ESK (USA), Carborundum Co (USA) - and others have developed and mastered the technological processes for the production of so-called non-grinding silicon carbide, obtained, as a rule, from highly dispersed charges by furnace synthesis in the form of micro-powders with their subsequent chemical enrichment [8, 57].

According to traditional technology, silicon carbide is produced in electric furnaces in which the working resistance is a layer of coke (the so-called core), as well as directly a charge consisting of a carbonaceous reducing agent and quartz sand. Pay attention to the harmful effects of alumina in quartz sand. Therefore, quartz sands are thoroughly washed. The unit capacity of the resistance furnace is 4000-4500kVA. The furnace is a self-propelled platform, at the ends of which there are current-carrying carbon electrodes. A return, quartz sand is poured onto the bottom of the platform, and then a core is laid out from a lump of petroleum coke, which is the working resistance in the initial period of the process. A reaction charge is poured on top of the core. Quartz sand should be used pure in impurities (99.6% SiO_2; 0.3% FeO; 0.07% Al_2O_3; 0.04% CaO; 0.03% Mg0; 0.02% TiO_2). Only low-ash carbonaceous materials can be used as a reducing agent: anthracite (3% ash, 93% Ssolid); petroleum coke (0.8% ash, 94% Ssolid, 5% volatile substances, 3% moisture). 50-65 tons of charge and 3200-4500 kg of core are loaded into the furnace, then it is connected to a furnace transformer. The process of obtaining silicon carbide is controlled mainly by the consumption of electricity [9, 19].

According to the recommended technology, replacing quartz with silica will not affect the quality of silicon carbide. All hardware circuits used in the world for the production of silicon carbide are united by the fact that the process takes place in resistance furnaces of various designs, where a horizontal heater (core) lined with carbon graphite material is surrounded by a reaction and filling charge. The type, volume and capacity of the furnaces used are selected based on the volume of output, the method of obtaining silicon carbide and the technical level of the enterprise. According to the existing methods of obtaining carbide in the laboratory, high-temperature equipment with a long exposure of the charge under the influence of high temperature is used. In order to reduce the time to obtain the product, the charge materials for the production of silicon carbide were finely and superfine ground in a high-speed ball mill to increase the specific surface area of the materials. For grinding, a Retsch E_{max} mill with a drum rotation speed of up to 2,000 rpm was used for ultra-fast grinding of samples, with an integrated liquid cooling system for grinding without overheating the material and cooling stops of the grinding set, with a narrow particle size distribution due to the special design of the grinding cups, which improves sample mixing.

Efficient grinding in the high-speed E_{max} ball mill is carried out due to high-frequency impact, intense friction and controlled circular movements of the grinding cup [10, 144].

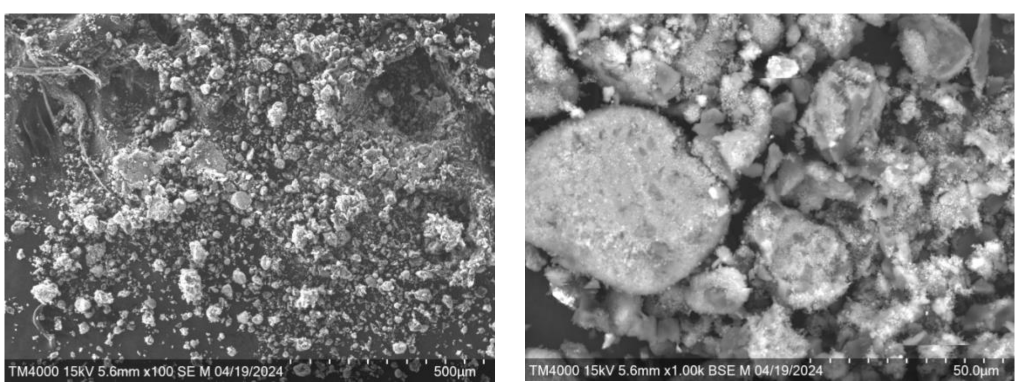
Grinding cups with a volume of 50, 80 and 125 ml are offered made of materials such as stainless steel, tungsten carbide and zirconium oxide, which prevent cross-contamination of the sample. Grinding balls, depending on the material, have sizes from 0.1 mm to 25 mm.

Two oval-shaped grinding cups are mounted on two discs, giving them circular movements in one direction. The combination of the shape of the grinding cup and vibration creates strong friction between the grinding balls, the material and the walls of the glass, as well as the acceleration with which the balls hit the walls of the glass, crushing the material. All this makes it possible to achieve high-quality mixing and a high degree of fineness, as well as a narrower particle size distribution compared to traditional ball mills.

Figure 3 - Retsch E_{max} high-speed ball mill, grinding cup and grinding balls

Results and discussions

The grinding of the charge mixture was carried out carefully and accurately. After preparing the materials for grinding, the necessary operating modes were set (rotation speed, grinding time, etc.). The rotation speed was selected at 1500 rpm, the grinding time was 3 minutes, the cooling pause was 1.5 minutes, the grinding duration was 30 minutes. Based on the literature, the estimated grinding time was established, which was 10 hours. Below is an image of the SEM morphology of the charge particles.



а б

Figure 4 - SEM image of the morphology of the charge mixture particles at magnification a)*100, b)*1000

The resulting charge mixture, due to the peculiarities of the technology of further processing, was subjected to pelletizing by briquetting. The need for fumigation is due to the fact that pyrometallurgical processing of small materials is impossible under the conditions of technology and is accompanied by high dust removal. The charge mixture consisting of silica and petroleum coke was connected by a binder in the form of liquid glass and formed under a press. According to the ratio of microsilica and petroleum coke, the following briquettes were obtained.

After that, the resulting briquettes were placed in a drying cabinet to remove excess moisture and harden the material. The temperature for drying briquettes is 125°C and the holding time is 120 minutes.

To carry out the work, a basic laboratory furnace for the production of silicon carbide was designed. The body of a high-temperature Tamman furnace with a coal tube was used for the construction of the furnace. The Tamman furnace was converted to an electric arc furnace with a graphite electrode. The current was supplied through a current guide to the graphite electrode through it to the charge layer.

The silicon carbide furnace is a research facility used to simulate carbidization processes. This high temperature unit is equipped with a carbon piping workspace and a graphite electrode. The temperature is measured using a tungsten-rhenium thermocouple BP-5/20, the place of hot welding of which on a reinforced corundum lid is brought to the bottom of the charge layer.

The resulting briquettes were cut into pieces and placed in a laboratory oven. Heating was performed linearly at a rate of 15°C per minute. The holding temperature was set to 50-100°C above the theoretical one, which is explained by the fact that the heating was carried out indirectly. The temperature of silicon carbide production in a high-temperature laboratory furnace was 1800°C.

When the set temperature was reached in the furnace, an isothermal exposure was carried out, which amounted to 80 minutes. High mass loss rates at temperatures above 1400°C is associated with the transition of part of SiO2 to the gas phase in the form of silicon monoxide (SiO). The formation of silicon carbide occurs in the temperature range above 1700 °C.

Thus, the grinding of the charge mixture in a high-speed ball mill and the pelletizing of the charge mixture made it possible to carry out a high-temperature experiment and clarify the formation temperatures of silicon carbide.

The samples obtained were transferred for chemical analysis. The following is an X-ray diffractometric analysis of the resulting product after a high-temperature experiment. The phase composition of the materials was studied by X-ray phase analysis on an Empyrean Malvern Panalytical X-ray diffractometer located at the Abylkas Saginov Karaganda Technical University. The diffractometer is equipped with an X-ray tube, the anode material is Cu (Ka1= 1.541874 Å). The measurements were carried out at room temperature in the range of angles 2°, in the range from 0° to 90° in step-by-step scanning mode with a step of 0.013 degrees.

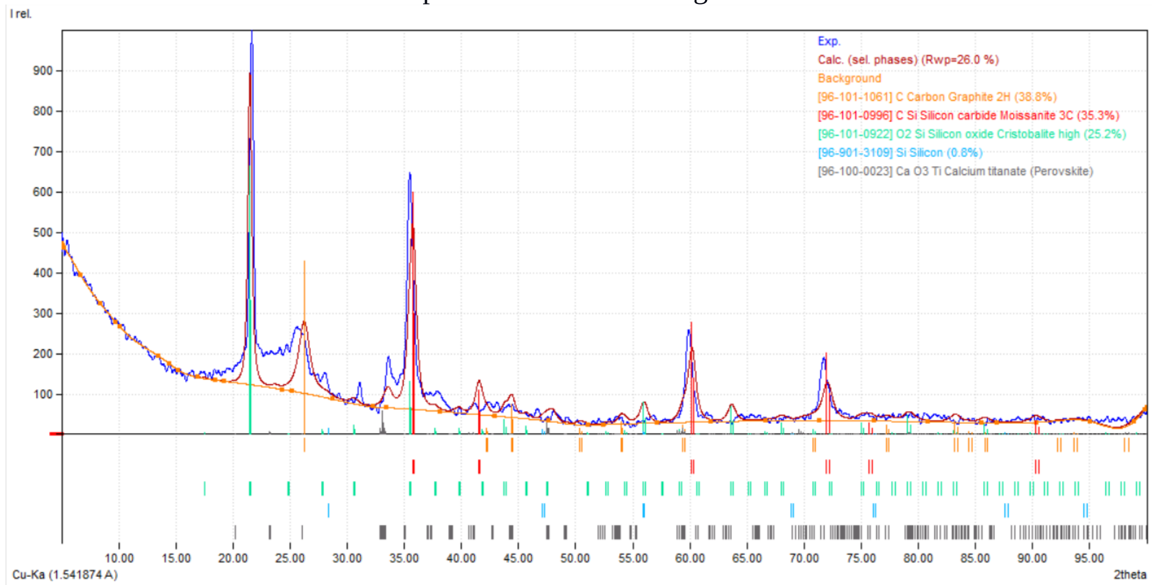


Figure 8 - X-ray of the obtained silicon carbide

According to the X-ray data, when the charge mixture is heated at high temperatures with isothermal exposure, peaks are observed related to the main characteristic diffraction maxima of silicon dioxide in the form of cristobolite. The peaks forming on the X-ray have characteristic reflexes of the α SiC phase in the form of moissanite.Also, the X-ray image shows the formation of a new peak, which indicates the beginning of the formation of the crystalline phase of carbon-graphite. The X-ray image shows the formation of a new peak, which indicates the beginning of the formation of the crystalline phase of carbon-graphite. The peaks forming on the X-ray have characteristic reflexes of the Si phase. The percentage of peaks formed on the RFA is shown in Table 5.

Table 5 – X-ray of the obtained silicon crab

Formula	Name of phases	Content, %
SiC	Moissanite	35,3
SiO_2	Cristobalite	25,2
Si	Free silicon	0,8
C	Graphite	38,8

Conclusion

According to the results of the study, the optimal technological parameters of the process of obtaining silicon carbide from silica were determined. High temperature heat treatment at 1800°C with an isothermal exposure of 80 minutes, it was shown that the resulting final material is a valuable product of silicon carbide. Based on the conducted research, the possibility of obtaining silicon carbide from man-made waste has been established.

It is important to note that effective dust waste management in silicon production not only contributes to improving the environmental situation in the region, but can also become an example of innovative approaches to sustainable production in other industries. Thus, the integration of new waste disposal methods can lead to a reduction in the negative impact on the environment and contribute to the development of environmentally friendly technologies in industry.

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ВОЗМОЖНОСТИ ИСПОЛЬЗОВАНИЯ ПЫЛЕВИДНЫХ ОТХОДОВ ФЕРРОСПЛАВОВ С ВЫСОКИМ СОДЕРЖАНИЕМ КРЕМНИЯ В ПРОИЗВОДСТВЕ КАРБИДА КРЕМНИЯ

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Аннотация. В статье рассматривается процесс получения карбида кремния из пыли кремнистых марок ферросплавов. Приведены результаты измельчения шихтовых материалов с использованием планетарно-центробежной мельницы, что позволило достичь оптимальной дисперсности частиц и улучшить их реакционную способность. Измельченная шихта подвергалась высокотемпературной переработке в печи сопротивления, что способствовало синтезу карбида кремния с высокой степенью чистоты. В статье представлены снимки шихтовой смеси, полученные с помощью сканирующего электронного микроскопа, которые позволили детально изучить морфологию и структуру получаемых частиц. Карбид кремния был исследован методом рентгенофазового анализа, что позволило определить его фазовый состав и подтвердить наличие основных фаз, таких как β-SiC и α-SiC. Также рассмотрены параметры, влияющие на эффективность синтеза, такие как температура, время обработки и соотношение компонентов в шихте. Выявленные закономерности позволят оптимизировать процесс получения карбида кремния и расширить его использование в различных отраслях, таких как электроника, металлургия, а также в производстве абразивных материалов и огнеупорных изделий.

Ключевые слова: карбид кремния, пыль кремнистых марок, микрокремнезем, нефтяной кокс, фазы, печь сопротивления, планетарная центробежная мельница.

КРЕМНИЙ КАРБИДІ ӨНДІРІСІНДЕ ЖОҒАРЫ КРЕМНИЙЛІ ФЕРРОҚОРЫТПАЛАРДЫҢ ШАҢ ТӘРІЗДІ ҚАЛДЫҚТАРЫН ПАЙДАЛАНУ МҮМКІНДІКТЕРІ

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Аңдатпа. Мақалада кремний карбидін ферроқорытпалардың кремний маркаларының шаңынан алу үрдісі қарастырылады. Бөлшектердің оңтайлы дисперсиясына қол жеткізуге және олардың реактивтілігін жақсартуға мүмкіндік беретін планеталық-центрифугалық диірменді қолдана отырып, шикіқұрам материалдарын ұнтақтау нәтижелері келтірілген. Ұнтақталған шихта қарсылық пешінде жоғары температурада өңделді, бұл жоғары тазалықтағы кремний карбидінің синтезіне ықпал етеді. Мақалада алынған бөлшектердің морфологиясы мен құрылымын егжей-тегжейлі зерттеуге мүмкіндік беретін сканерлеуші электронды микроскоптың көмегімен алынған шихта қоспасының суреттері келтірілген. Кремний карбиді рентгендік фазалық талдау әдісімен зерттелді, бұл оның фазалық құрамын анықтауға және β-SiC және α-SiC сияқты негізгі фазалардың болуын растауға мүмкіндік берді. Температура, өңдеу уақыты және шикіқұрамдағы компоненттердің қатынасы сияқты синтез тиімділігіне әсер ететін параметрлер де қарастырылады. Анықталған заңдылықтар кремний карбидін өндіру үрдісін оңтайландыруға және оны электроника, металлургия сияқты әртүрлі салаларда, сондай-ақ абразивті материалдар мен отқа төзімді бұйымдар өндірісінде пайдалануды кеңейтуге мүмкіндік береді.

Түйін сөздер: кремний карбиді, кремнийлі маркалы шаң, микрокремнезем, мұнай коксы, фазалар, қарсылық пеші, планетарлық центрифугалық диірмен.