



Silicon Production Using Alternative Raw Material Source

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Abstract

During the process of the rice hulls pyrolysis at the pilot plant the silicacarbon (SC) was produced containing 51.2% of total carbon, 38.7% of silicon dioxide and 7.6% of hydrocarbons. Its secondary heat treatment with air supply resulted in generation of the two products: SC-1 (50.4% of SiO₂; 48.2% of C; 0.56% of Ca; and 0.4% of Fe), and SC-2 (76.45% of SiO₂; 22.1% of C; 0.95% of Ca; and 0.55% of Fe). After treatment with the 1% HCl solution the content of Ca and Fe in these products decreased to 0.15% and 0.16%, respectively. Silica gel (88.4% of SiO₂, 11.0% of H₂O) and carbon (95% of C, 4.5% of SiO₂) were produced from SC after its treatment with the NaOH solution.

Characteristics of the result products were studied using the infrared spectroscopy and X-ray diffraction analysis. SC is a composite alloy formed by nanoparticles of amorphous carbon (~500Å) and silicon dioxide (100–200 Å). Carbon is presented by graphite-like (Gph), polynaphthenic (Nph) and hydrocarbon (Hph) phases. Amorphous SiO₂ is presented by opal.

Three types of charging materials with SiO₂:C~2.5 proportion were produced from SC-1 and SC-2, from SC-1 and quartz, and from quartz and silica gel.

At the pressure of 20 MPascal briquettes were produced from each charging material, which upon drying at air (100°C) and furnacing (300°C) in the inert atmosphere had the strength of 25–38 kg/cm².

After smelting the briquettes in an electric arc furnace, the silicon product with the content of 98–99% Si was produced.

In industrial conditions after ladle refining the content of Ca and Fe in silicon will be reduced down to 0.3–0.4%.

Keywords: rice hulls, silicacarbon, silica, carbon, silicon, pyrolysis, briquetting

Introduction

With growth of demand for the solar-grade silicon for photovoltaic devices during the recent years there are lots of investigations for cheap sources of raw materials, as well as technologies ensuring the decrease in the production cost of the electronic-mark silicon. The special emphasis is laid on the quality of the feedstock for metallurgical-grade silicon as initially the polycrystalline silicon is produced by carbothermal reduction of silica-containing raw materials using the traditional charging materials based on quartz and coke [1].

However, currently the alternative options are considered, these are plant raw materials having in their composition silica and natural organic polymers, particularly, wastes of the rice production, i.e. the rice hulls (RH). The latter may successfully compete with the above named components of charging material in relation to both the purity of the silicon under smelting and parameters of the carbothermal process. The level of electrically active inorganic impurities in RH (Ti; Cu; Mn; Cr; Fe; Ni; P; B) is significantly lower than

that in quartz and coke, and the surface and structural characteristics of the charging material produced from rice hulls are significantly better than that of crystalline quartz and of the reducing agent of coke which is of less purity [2].

Materials and Methods

Taking into account the rice hulls composition: presence of amorphous silicon dioxide in the form of opal mineral (~15%) and organic polymers of cellulose and lignin (80% in total), the technology of complex processing of the rice hulls [3] was developed based on the RH pyrolytic decomposition with production of three marketable products during the single process cycle: organic condensate, energy gas, and solid product, that is silicacarbon (SC).

The latter is a nanocomposite which is formed by carbon nanoparticles (500Å), it is of black color and has preserved the form of wet hulls due to residual bonding forces and amorphous silicon dioxides [4] in the form of aggregates (100–200Å). Physical and chem-

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Tab. 1. Physical and chemical characteristics of silicacarbon produced from the rice hulls
Tab. 1. Wyniki analizy silikakarbonu

Product	Chemical analysis, mass %				X-ray diffraction composition in %			Physical characteristics			
	SiO ₂	C _{total}	Ca	Fe	G _{ph} [*]	N _{ph} ^{**}	H _{ph} ^{**}	Bulk weight, g/cm ³	S _{sp} , m ² /g	Σ N ₂ porosity volume, cm ³ /g	Iodine activity, %
Silicacarbon (SC)	35-37	51-52	0.2	0.15	55	35	10	0.1	150	0.15	10

* G_{ph} – graphite-like phase
** N_{ph} – polynaphthenic phase
*** H_{ph} – hydrocarbon phase

Tab. 2. The phase composition of the carbon-containing component of the carbonization product of rice hulls, cellulose and lignin
Tab. 2. Skład fazowy składników węglonośnych w produktach pirolizy łusek ryżowych, celulozy i ligniny

Carbonization product	Temperature, °C	Phase content, %		
		G _{ph}	N _{ph}	H _{ph}
RH	500	45	45	10
	650	55	35	10
	800	60	30	10
	1,000	100	abs.	abs.
Cellulose	500	45	40	15
	650	46	43	11
	800	58	30	12
	1,000	72	abs.	28
Lignin	500	44	41	15
	650	43	35	22
	800	44	38	18
	1,000	62	abs.	38

ical characteristics of silicacarbon produced from the rice hulls in stationary mode are presented in Table 1.

The results of physical and chemical studies of silicacarbon produced from the rice hulls showed that the composition and structure of the carbon component are determined by the nature of organic polymers of cellulose and lignin presented in the rice hulls.

So, cellulose is a linear polymer molecule of which (C₆H₁₀O₅)_n presents long chains with a spatially regular structure. Cellulose fiber thermal destruction runs rapidly with formation of abundant volume of water, as well as resinous and gaseous buildups that leads to decrease in output of carbon residual down to 30%, formation of which passes through the stage of the conjugated bond system formation in the cellulose macromolecules and they are developing due to the intermolecular dehydration of cellulose [5].

Lignin is an organic polymer which in contrast to cellulose has the aromatic structure built of the structural elements C₆C₃ and belongs to the class of multifunctional organic compounds, the molecules of which contain the condensed aromatic ring with a very active carboxyl group along with other functional groups in its lateral radicals. Unlike cellulose, lignin thermal destruction runs through the polymer peripheral part breaking up with 45–47% output of the product of carbonization.

There is an opinion that the ratio between carbon enclosed in the aromatic layer and non-aromatic carbon

located in the polymer lateral chains determines the reactive capability of oil-carbon sludge polymer generated at the thermal destruction [6].

At the carbonization stage both polymers undergo reactions of dehydration, dehydrogenating, acid and spirit release with formation of atomic groups in the form of hydroxyl, carboxyl, carbonyl and other functional groups. As a result of this process the pure carbon is produced. Exactly in this period of the RH heating up to 500°C the generation of the reactive carbon microstructure is observed. That is why rice hulls silicacarbon appeared to be sufficiently prospective to use it as the charging material at smelting metallurgical silicon of high purity.

The X-ray diffraction composition of the carbon component in the carbonization products of the rice hulls and its derivatives are presented by three phases: carbon phase – graphite-like one (G_{ph} – 55%) and two hydrocarbon phases: polynaphthenic phase (N_{ph}~35%) and hydrocarbon phase (H_{ph}) of the non-identified structure.

The nanostructure of carbon phase (G_{ph}) in thermally treated samples is formed by similar graphite grids but it is less perfect if compared with the graphite structure.

One of the found out carbon phases is polynaphthenic phase (N_{ph}) which presents the clathrate structure consisting of the condensed and divided by meth-

Tab. 3. Composition of charging materials, mass %
 Tab. 3. Skład badanego materiału, % masowy

Composition	C _{total}	SiO ₂	Ca	Fe
Charging material #1 (79% of SC-2 and 21% of SC-1)	27.8	71.4	0.17	0.18
Charging material #2 (59% of SC-1 and 41% of quartz with 98.5% SiO ₂)	28.0	70.9	0.35	0.40
Charging material #3 Silica gel Carbon	- 27.0	68.0 4.0	0.002 0.11	0.013 0.12

ylene bridges of naphthenic cycles containing alkane chains. The structure of the second hydrocarbon phase (Hph) is not determined due to its non-existence in the pure form [7].

With an increase in temperature of the rice hulls processing as it is seen from Table 2, the distinct dependence of the phase content was revealed in the produced materials. The thermal treatment of plant raw materials at 500°C leads to the destruction of the intermediate phase. With the further increase in temperature up to 800°C, the amount of the graphite-like phase increases in the result of transforming the polynaphthenic component, obviously, as less heat-resistant in comparison with Hph, the share of which does not change at the temperature range up to 1,000°C. After the initial sample treatment at 1,000°C both hydrocarbon phases (Nph and Hph) are transformed to the graphite-like phase.

Cellulose thermal destruction at the temperature lower than 800°C runs similarly to that of RH; at 1,000°C the polynaphthenic phase is transformed to Gph and Hph. In the process of lignin thermal destruction the content of the graphite-like phase remains practically unchanged up to 1,000°C. In temperature interval 650÷800°C mutual transformations of Nph and Hph appear. With the temperature increase up to 1,000°C the partial Nph transition to Gph and Hph is observed similarly to that noted for cellulose. At that, lignin thermally treated at 1,000°C in comparison with the rice hulls and cellulose thermally treated under the same conditions has the least share (62%) of the graphite-like component.

To perform the reaction of the carbothermal reduction of silicon dioxide $\text{SiO}_2 + 2\text{C} = \text{Si} + 2\text{CO}$, the stoichiometrical ratio of SiO₂ to C shall be equal to 2.5. Such ratio provides the required concentration of monoxide (SiO) in the gas phase, otherwise it is impossible to achieve the complete extraction of silicon due to the non-productive SiO and CO carry-over from the reaction zone. However, according to the chemical analysis data, there is the superabundance of carbon in silicacarbon with the C/SiO₂ ratio equal to 1.5.

In experimental complex of the Republic State-Owned Enterprise “The National Center on Complex

Processing of Mineral Raw Materials of the Republic of Kazakhstan “, on the pilot pyrolysis plant operating in the continuous mode a 200 kg pilot batch of pyrolysis silicacarbon (mass %: SiO₂ – 28.92; C_{total} – 55.8; volatile - resinous – 27.3) was produced to use as a charging material for smelting of the metallurgical silicon.

In order to remove the volatile matters, the pyrolysis silicacarbon underwent the oxidizing burning at 550–600°C that was necessary as the resinous components pull the briquettes apart during the briquetting process.

As the result two products SC-1 and SC-2 were produced with the content in mass %: SC-1 (SiO₂ – 50.4; C – 48.2; Ca – 0.56; Fe – 0.4) and SC-2 (SiO₂ – 76.45; C – 22.1; Ca – 0.95; Fe – 0.55).

According to the chemical analysis, after the silicacarbon oxidizing burning the content of Ca and Fe impurities in it increased significantly. To remove these impurities both batches of silicacarbon were treated by the hot (70–80°C) 1% HCl solution. The residuals were separated from the solution, washed by hot water till the rinse water neutral reaction, and dried at 105°C.

The dried residuals of SC-1 and SC-2 have the following composition, respectively, in mass %: (SiO₂ – 49.4; C – 47.4; Ca – 0.13; Fe – 0.15) and (SiO₂ – 74.28; C – 22.5; Ca – 0.17; Fe – 0.18).

In the silicacarbon products SC-1 and SC-2 washed with acid the content of Ca and Fe impurities decreased by 4.5 and 2.8 times, respectively.

The required SiO₂:C=2.5 ratio in the charging materials 1 and 2 was achieved by the correction in the compositions. Carbon insufficiency in SC-2 to achieve the stoichiometry was compensated by adding SC-1, and silica insufficiency in SC-1 by adding the required amount of quartz.

The stoichiometrical compositions of charging material #1 and charging material #2 are presented below in Table 3.

In the technologies using the RH oxidizing burning with producing carbon black (SiO₂~43-45%) and silica white (SiO₂~98.5%) a large amount of the pure valuable reducing agent is lost [8]. That is why charging

Tab. 4. The results of the chemical analysis of the carbon-containing products
 Tab. 4. Wyniki analizy chemicznej produktów węglonośnych

Product name	Mass fraction of the elements under determination, %						
	SiO ₂	Fe	Ca	Mg	Na	K	P
Silicacarbon prior to alkali treatment	41	0.57	0.32	0.12	0.15	0.33	0.003
Carbon product after alkali treatment	4.2	0.65	0.41	0.20	0.56	0.42	-
Carbon product after acid treatment	4.2	0.12	0.11	0.04	0.02	0.014	0.003

Tab. 5. Chemical composition of components of charging material #3
 Tab. 5. Wyniki analizy chemicznej produktów węglonośnych #3

Product name	Composition in mass %								
	SiO ₂	Ca	Fe	Mg	Na	K	P	B	H ₂ O cryst
Silicon dioxide	88.4	0.002	0.013	0.012	0.12	0.003	0.02	0.0001	11.0
Carbon product	4.5	0.11	0.12	0.04	0.002	0.014	0.003	0.0003	-

material #3 based on SC was prepared with preservation of its two components: silica and carbon.

This charging material production technology assumes the SC treatment with NaOH solution at 70–80°C during one hour. In such conditions the silica passes into solution with generation of the alkali-silicate solution. The residuary solid carbon is nutsche-filtered, washed with de-ionized water till achieving the rinse water neutral reaction, dried at 105–110°C and analyzed for the following components: SiO₂, Ca, Fe, Na, Mg, K, P.

As a result of the SC alkali treatment it was stated that the mineral impurities do not pass into solution but all are concentrated on the carbon product so the produced carbon was washed with 1% HCl solution with simultaneous heating during one hour. Then the solid residual was nutsche-filtered and rewashed with the 0.5% HCl solution based on deionized water.

The results of the chemical analysis of the products shown in Table 4 indicate that the content of inorganic elements in modified silicacarbon grows after the alkali treatment and decreases sufficiently after the acid treatment.

On the diffraction pattern of the carbon product produced after the silicacarbon alkali treatment there is only one broad halo peculiar to the graphite-like carbon phase with $d=3.345\text{--}3.857\text{Å}$, that makes it possible to consider the amorphous carbon component as the favorable reducing agent for the carbothermal process of the metallurgical silicon smelting [9].

Silica component of the charging material is produced by the known method of neutralizing the alkali-silicate solution with the HCl mineral acid till reaching pH=9 [10]. After the complete precipitation of silica gel it is washed with deionized water by decantation, squeezed and dried at 125°C.

It is established that silica gel produced in such way coagulates rapidly and transforms into transparent hydrogel presenting large aggregates from 200 to 1,000 Å.

According to the chemical analysis data, the silica gel (SiO₂·nH₂O) contains SiO₂~88%; H₂Oconstr~11% and its purity is adequately high in relation to inorganic impurities <0.2% (Table 5).

Therefore, after the alkali treatment of silicacarbon two amorphous products were produced, these are carbon and silica gel with a low content of impurities and high surface characteristics. These components were used to prepare the stoichiometrical charging material (#3) for the metallurgical silicon smelting.

In total three charging materials were prepared on the base of SC-1, SC-2, quartz, silica gel and carbon from which briquettes were produced for metallurgical silicon smelting in an electric arc furnace.

Operations for the briquette preparation include: the initial material grinding to 0.1 mm fraction, intensive mixing of the components in the required proportion, briquetting using a bonding agent, drying and furnacing of the briquettes.

It is known that in metallurgy the rigid requirements are imposed to ore briquettes not only in relation to their chemical composition but also to their physical and chemical features. In particular, briquettes shall have an adequate mechanical strength to withstand loads at transportation and filling into a metallurgical furnace, and shall also have a high water resistance, atmospheric stability, and adequate porosity increasing their reactive capabilities, etc.

These indicators are achieved due to adding a bonding agent into the charging material.

The two products were tested as the bonding agent: the traditionally used sodium silicate with the 2.6% base-to-silica (15 mass %) and resinous product (mass 10%) obtained at the rice hulls pyrolysis.

Charge material briquetting was performed in the press form with $d=52$ and $h=50\text{mm}$ using a mechanical press at the pressure of 20 MPascal.

Briquettes were air-dried at 100°C and furnaced at 300°C in absence of air.

After drying all the briquettes were tested to determine the mechanical strength for dropping and crushing.

Briquettes based on sodium silicate after drying at 100°C withstood the 12 kg/cm² load under compression. After the 300°C heating during 15 minutes the briquettes were hardened and withstood the 25 kg/cm² load under compression.

A different situation is observed with the briquettes produced using resinous products. Their mechanical strength after pressing and drying at air is sufficiently high; they withstood the load up to 38 kg/cm² under compression. But the briquettes failed to withstand a heat load; when the briquettes were heated up to 300°C they broke down as the resin melted and fled.

Taking into consideration the test results it is recommended to use the combined bonding element composed of sodium silicate and settling-vat tar (15% and 10% mass percent, respectively) as the optimum option for the briquettes production using silicacarbon products of the rice hulls processing. This proportion is determined experimentally.

The briquettes based on the combined bonding additive are sufficiently strong both when they are dried at 100°C and heated up to 300°C. Obviously, the resin works at low temperatures, while sodium silicate hardens the briquettes at the temperatures of 300°C and higher. Positive effect from using the combined bonding element is also in that resin evaporation at calcinating will facilitate the increase in gas permeability of the briquette.

It should be noted that the application of the combined bonding element has another positive effect, i.e. the resin evaporation during the furnacing facilitates the increase in gas permeability of the briquettes.

Due to using the charging material having an increased porosity in the silicon smelting, the silicon extraction increases and electric energy consumption is reduced because of the high sorption activity of the charging material in relation to gaseous silicon oxide and decrease in losses of silicon with the exhaust gases.

For authenticity of the researches ten briquettes were prepared using each charging material and data on their mechanical strength were taken as an arithmetical average.

Tests on the silicon smelting were conducted in "Steklo M" LLP (Shymkent).

Briquettes prepared using charging materials #1, #2 and #3 at the amount of 16, 30, 28 kg, respectively, were smelted in a 60 kW one-electrode furnace at the 80 V arc voltage, 300-400 A current strength and 13 A/cm² current density.

We produced 9; 5; 6.5 kg of silicon, respectively, with an average content of Si~98.8%. The high content of impurities (~1.2 %) in the produced silicon samples is because of the small area of the melting chamber

of the electric arc furnace where the high ratio of the melting pot and electrode surface to the mass of smelted silicon caused its contamination with harm impurities contained in the refractory and electrode and the non-productive SiO carry-over from the reaction zone.

When smelting the proposed charging materials in the furnaces of larger sizes with a high furnace throat, these deficiencies will be neutralized and an essential decrease in impurities in silicon down to 0.3–0.4 % is expected subject to the additional ladle refining of silicon.

Conclusion

Rice hulls are the alternative raw material for metallurgical silicon smelting due to the unique composition: the amorphous silica and two organic polymers namely cellulose and lignin.

Silicacarbon produced by the pyrolytic decomposition of rice hulls is the nanocomposite formed by nanoparticles of carbon (500Å), and silicon dioxides (100-200Å).

The charging material based on silicacarbon compares favorably with the charging material based on quartz and coke due to the low content of inorganic impurities including electrically active ones (<0.4%), as well as a sufficiently high specific surface of amorphous components of silicon dioxide and graphite-like carbon.

A pilot batch of pyrolysis silicacarbon (SC) was produced and tested on the pilot plant and after its oxidizing burning the two products were produced: SC-1 and SC-2 of the following composition in mass %: SiO₂~50.4; C~48.4; Ca~0.56; Fe~0.4 and SiO₂~74.4; C~22.1; Ca~0.95; Fe~0.55, respectively. After they were washed with the 1 % HCl solution, the content of Ca and Fe impurities decreased in 4.5 and 2.5 times, respectively. Two stoichiometrical charging materials #1 and #2 were prepared using SC-1 and SC-2 with adding quartzite by means of correction, and charging material #3 was produced by the mixing individual components of carbon and silica gel which had been separated during the alkali treatment of the pyrolysis silicacarbon.

Briquettes prepared using charging materials #1, #2 и #3 were smelted in in a 60 kW one-electrode furnace at the 80 V arc voltage, 300-400 A current strength and 13 A/cm² current density. 9; 5; 6.5 kg of silicon were produced, respectively, with an average content of Si~98–99%.

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Produkcja krzemu ze źródeł alternatywnych

W procesie pirolizy łuski ryżowej w instalacji pilotowej wyprodukowano silikakarbon (SC) zawierający 51,2% całkowitego węgla, 38,7% dwutlenku krzemu i 7,6% węglowodorów. Jego wtórna obróbka cieplna za pomocą powietrza doprowadziła do wytworzenia dwóch produktów: SC-1 (50,4% SiO₂, 48,2% C, 0,56% Ca i 0,4% Fe) i SC-2 (76,45% SiO₂, 22,1% C, 0,95% Ca i 0,55% Fe). Po obróbce 1% roztworem HCl zawartość Ca i Fe w tych produktach obniżyła się odpowiednio do 0,15% i 0,16%. Żel krzemionkowy (88,4% SiO₂, 11,0% H₂O) i węgiel (95% S, 4,5% SiO₂) wytworzono z SC po traktowaniu roztworem NaOH. Charakterystykę produktów badano przy użyciu spektroskopii w podczerwieni i analizy dyfrakcji rentgenowskiej. SC jest stopem kompozytowym utworzonym z nanocząstek bezpostaciowego węgla (~ 500Å) i dwutlenku krzemu (100–200 Å). Węgiel jest reprezentowany przez fazy grafitopodobne (Gph), polinefeninowe (Nph) i węglowodorowe (Hph). Amorficzny SiO₂ jest prezentowany przez opal. Trzy rodzaje materiałów z udziałem SiO₂: C 2,5 zostały wytworzone z SC-1 i SC-2, z SC-1 i kwarcu oraz z kwarcu i żelu krzemionkowego.

Brykiety wytworzono pod ciśnieniem 20 MPa po wysuszeniu na powietrzu (100°C) i w piecu (300°C) w atmosferze obojętnej. Brykiety miały wytrzymałość 25-38 kg/cm². Po wytopieniu brykiety w elektrycznym piecu łukowym wytworzono produkt krzemowy o zawartości 98–99% Si.

W warunkach przemysłowych po rafinacji zawartość Ca i Fe w krzemie zostanie zmniejszona do 0,3–0,4%.

Słowa kluczowe: łuski ryżowe, węgiel krzemu, krzemionka, węgiel, krzem, piroliza, brykietowanie