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Production of Crystalline Silicon by Carbothermal Method

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Abstract
In the auricle is described the process of obtaining crystalline, i.e., technical silicon by the carbothermal method in the Zestafoni Ferroalloys plant of "Georgian Manganese", in a single-phase electric arc furnace of circular section, specially built for this purpose which is powered by a 5.5 MVA transformer. Quartz of 98.9% purity is used as the starting material, petroleum coke with 95.94% solid carbon content and charcoal with 71% carbon content is used as the regenerator.
During 60 hours, 12 t of burden is processed in the form of 24 servings of 500 kg. 3055 kg of quartz (silicon extraction ratio 65.4%), 1341.5 kg of charcoal, 332.9 kg of petroleum coke, 166.5 kg of wood chips, 61 kg of graphite electrode and 14673 kWh of technological electricity were used for 1 ton of products.

Key words: furnace, crystalline silicon, "solar silicon", quartz, coke.

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I. Introduction

(the essence of the problem). The inexorable increase in energy consumption on earth, the reduction in reservesof mineral energy resources (oil, coal, gas, peat) and the permanent increase in their prices have forced the world community to search for new energy sources. Scientists had high hopes on regulated thermonuclear processes and nuclear power plants known as the "Tokomaki" project. But, unfortunately, the implementation of the "Tokomaki" project has moved into the distant perspective, and therefore, many states have stopped funding it. On the other hand, the disasters at the Chernobyl and Fukushima nuclear power plants forced the leaders of leading Western countries to think and decide to sharply reduce nuclear energy.Therefore, the search for alternative, renewable energy sources such as wind energy, biofuels, solar energy and others is becoming increasingly important. Solar energy has already acquired a global character. The European Union is discussing a project to build a complex of solar power plants in the Sahara Desert, which will unite about a hundred giant power plants equipped with solar panels. If the project is implemented, Europe will no longer be dependent on energy resources from Russia and the Middle East[1].

According to forecasts, the share of solar energy in world energy consumption will increase annually and will reach 50% by 2050, and in some countries - 60%.

The actualization of the use of solar energy has sharply increased the demand for the production of photoelectric converters (PEC) modules, where high-purity (99.99%) silicon plates called "solar silicon"[1]are used as the working element.

The modern world produces and consumes several million tons of silicon each year, mainly in the form of metallurgical (technical) MG-Si silicon, often referred to as crystalline silicon. A wide variety of silicon-

[1]The name "solar silicon" (Terrestrial Solar Grade-TSG) defines the purity of silicon used for the production of photovoltaic converters, in which the content of electrically active impurities (Al, Fe, Ti, V, P, B) must be within
10^{-5}-10^{-3} atomic percent

containing quartz (SiO2) minerals, widely distributed in nature, are used as initial raw materials. From here, elemental silicon is restored by the carbothermal, less often magnesium thermal method. In the first case, a high-quality low-ash carbonaceous reducer is used (charcoal, graphite, petroleum coke, etc.), in the second case - metallic magnesium [2].

Most of the silicon mined in the world, relatively less pure silicon (95% Si), is widely used in modern industry, while up to 2% of the total product, with a higher silicon content (98-99% Si), is deeply purified by various methods and used for electronics and solar energy purposes.

Thus, the production of silicon for photovoltaic modules by the direct carbothermic method is considered one of the promising directions for solving the problem, which implies the production of technical silicon with a purity of 98% by weight in ore-thermal furnaces from clay-containing raw materials.

The present work carried out in the Zestafoni Ferroalloys Factory (ZFF) also concerns the smelting of technical silicon useful for the production of "solar silicon" by carbothermal process.

Experimental furnace. In 2022, in order to promptly solve the scientific and technical problems posed in the ZFF, and among them for the extraction of high-purity technical silicon, a single-phase experimental furnace of circular cross-section (Fig. 1) was installed, with a 600 mm diameter graphite electrode placed in its center[3]. The electric arc is connected to a steel plate located at the bottom of the furnace, which, like the walls of the furnace, is built with coal blocks and fireclay bricks.

Furnace dimensions:
- furnace shell diameter - 3000 mm;
- shell height- 3130 mm;
- internal diameter of the lining - 1950 mm;
- height of lining to coal blocks- 750 mm;
- total height of the lining - 1355 mm.

The furnace is served by a single-phase 5.5 MVA power transformer. To represent the scale, we remind you that the furnace capacity of the Didube pilot plant, where ferromanganese production technology was developed for the first time in 1930, was 0.9 MVA.

Figure1. Experimental furnace

Materials and experiment. In order to obtain crystalline silicon, the factory purchased three kinds of charging material[2]:

- quartz (98.9%-SiO_2, 0.3%-Fe_2O_3, 0.1%-Al_2O_3, 0.1%-CaO, 0.1%-MgO);
- petroleum coke (95.94%-solid carbon, 2.63%-volatile, 0.,73%- ash, 0.7%- sulfur);
- charcoal (71%-solid carbon, 26%- volatile, 1.6%- ash, 0,03%- sulfur).

According to theoretical calculation of the burden 24 servings of 500 kg with the following composition were made:
quartz- 312 kg;
charcoal - 137 kg;
petroleum coke - 34 kg;
wood chips - 17 kg;
totally 500kg,

[2]Composition in mass percentages

which was spent in 60 hours.
The material balance of the burden looks like this:
quartz– 7.488 t;
charcoal – 3.288 t;
petroleum coke– 0.816 t;
wood chips– 0.408 t;
totally12 t.

In December 2022, the operation of the experimental furnace began. This was preceded by a 47-hour wood-burning drying of the furnace and then a 31-hour electric and coke heating process until reddening the coal blocks of the furnace bath wall. After that, the loading of the burden into the furnace and the melting of crystalline silicon began, which was going on for 60 hours.
Thus, in 2.5 days, 12 tons of burden with a total weight of 500 kg were consumed in the form of 24 servings.
In order to avoid the expected segregation due to the different bulk weights of the burden materials, the burden was manually supplied to the furnace mouth.

Research results and future perspective. It should be noted that when receiving crystalline silicon, the experimental furnace worked in a hot state, in some cases we had to hold the electrode in a higher than average position in order to maintain the electric mode. The rise of gases was clearly visible both on the furnace mouth and on its arch. Twice there was a case when we had to open the furnace door due to the high temperature (145-150^{0}C) in the air cleaner (in front of the filters). There was no gas or burden outbursts from the furnace top.
Melting of crystalline silicon in an experimental furnace is characterized by the following parameters:
1. Average power 1.25-1.35MW;
2. Electrode voltage70-74 V;
3. Electrodecurrent22-24 ka;
4. current in high area 280-320 amp;
5. Voltage in the high area 6.0 kV
6. Electrode position 100-230 mm;
7. Temperatures of half rings and jaws 20-40^{0}C;
8. Temperature of gases at the furnace 240-310^{0}C;
9. Temperature of gases in the filters115-145^{0}C;

In the course of this whole process 75.964 kWh of electricity was consumed, including about 40 000kWh of electricity for drying and heating the furnace and 35.964 kWh for receiving products.

Figure2. Crystalline silicon

Crystalline silicon with its qualitative indicators must comply with the technical conditions of the state standard 2169-69. It is dark gray and shines like steel. Its crystalline lattice is of the diamond type and because of that it is very strong, it even cuts glass.

98.7% pure crystalline silicon was obtained in the very first trial with the carbon reduction process, the main impurities of which are: iron - 0.5%, Aluminum-0.4% and calcium-0.4%. Naturally, this result can be improved in the future by searching for purer burden materials.

For 1 ton of the obtained product there were spent 3055 kg of quartz (silicon extraction ratio 65.4%), 1341.5 kg of charcoal, 332.9 kg of petroleum coke, 166.5 kg of wood chips, 61 kg of graphite electrode and 14673 kWh of technological electricity. Such a high energy consumption is caused by the alloy and the burden remaining in the furnace bath after 2.5 days of operation.

Thus, 3173.2 US dollars were spent to obtain 1t of crystalline silicon.

This result is of great importance and may have a decisive influence on the factory's activities in the future. A certain part of the products produced as a result of further processing of technical silicon is called Silicon of semiconductor purity, "electronic silicon" (99.999% by weight) will be used in various fields of radio electronics for the manufacture of radio components and microcircuits, and the largest part - for the manufacture of "solar" elements.

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