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Research paper

# Obtaining the fine-grained silicon carbide, used in the synthesis of construction ceramics

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## Abstract

Silicon carbide is used in the production of construction and temperature-resistant goods, capable of withstanding high mechanical and thermal loads. During recent times, silicon carbide has been frequently used in the electronics industry. Since sintered silicon carbide has increasingly been used as a replacement for metal components of various devices, the process of obtaining compact goods from silicon powder has become the defining factor in the technology used for its synthesis. The selection of conditions in which the sintering is conducted depends on granulometric structure, the form and the surface condition of the initial powder. The work consists of the synthesis of silicon carbide powder using the purified form of metallurgical silicon powder and soot. The qualities of testing samples were studied, where silicon carbide was obtained using established technology, from mechanically activated elementary, fine-grained silicon and soot, by pyrolytic synthesis. It was demonstrated that synthesis produces highly pure silicon carbide powder, (α- and β-phases) with a granulometric composition that allowed subsequent sintering to produce high quality compact goods. It was established that the content of silica in synthesized silicon carbide powder does not exceed 1–2% of the total mass.


*Keywords:* Silicon carbide; The Acheson method; Form and histogram of particle size

## 1. Introduction

Modern industrial machine building, metallurgical, chemical and energy sectors impose high standards for the construction materials used to make their components. Such requirements, which cannot be met by metals and alloys, are: high chemical resistance, ability to withstand high temperatures while keeping stable and, when exposed to extensive mechanical and thermal loads, low specific density, low production cost and reliance on environmentally friendly manufacturing technologies.

Leading industrial countries all recognise that ceramic is one such material, which meets all these requirements. The determination of the level of technological development in individual countries is based on the existence of industrial production potential for ceramic material and goods.

Silicon carbide is one of the most widely used materials. Classified as oxygen-free ceramic, its field of industrial application is constantly widening. This substance has unique qualities, such as high rigidity and stability, chemical and thermal stability, high melting point and resistance to oxidation, considerable resistance to erosion etc. Thanks to these qualities, SiC can be used to produce goods designated to withstand high electric power (power electronics) [1], to build electronic components that are resistant to high temperatures, as well as equipment exposed to high friction and heat [2]. Silicon carbide is used in the industry in the production of polyethylene (ethylene) for fittings and sleeves, allowing the process temperature to be doubled [3]. Silicon carbide is increasingly being used by nano-structures [4].

The main industrial method for the production of silicon carbide is SiC synthesis, which was conceived in 1892 by

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**Nomenclature**

| | |
|---|---|
| Q | the amount of heat released during reaction, $[\frac{kJ}{mole}]$ |
| D | particle size, in nm |
| σ | standard average deviation, in nm |
| $d_{95}^{cp}$ | average particle size, for particles constituting at least 95% of total particles, in μm |

[Edward Goodrich] Acheson. The process is based on a widely used method of $SiO_2$ recovery using the carbothermic process resulting in SiC powder. The main shortcoming of this process is the multitude of stages in technology, the high energy consumption and the rather low quality of the product (low purity). Several alternative SiC production methods have been reported [5,6]. They include physical splitting of volatile silicon compounds (PVT) [4,7], chemical decomposition of compounds (CVD) [8–12], the sol-gel process [13–15], liquid phase sintering (LPS) [16] and obtaining alloys through mechanical agitation (MA) [17].

The purpose of the present paper is to describe the principles of production of high purity SiC powder, to be used in the manufacturing of compact goods, capable of withstanding high pressure, temperatures and mechanical loads, as well as in [chemically] aggressive environments. For this we will need to study the effects of the granulometric composition of the initial silicon powder on aspects of the synthesised SiC powder particles. Based on the information provided under [18], the synthesis temperature has a decisive effect on the quality of SiC, which is why its correct setting is particularly important. When producing compact goods, one of the primary requirements is to ensure uniform sintering of aggregate SiC particles. This factor will depend on the condition of the sintered particle surface. The study of the effect of synthesis temperature and air annealing of the samples on their surface is the main purpose of the exercise.

It has been noted, that production of fine ceramics experienced an annual growth of 9.1% [19] and that by as early as 2000 this sector was already worth USD 8.45 billion. By 2015, the world's capacity for production of silicon carbide of all types kept increasing, churning out 1.1 million tons of goods per year, 41% of which was in China [20]. In addition to China, the growth in silicon carbide production was also recorded in India and Japan. The main consumers of silicon carbide are countries with developed metallurgical and electronic industries, where refractory ceramics are needed. In recent times, silicon carbide has also been used as a construction material, as well as being increasingly used in aviation.

In order to integrate silicon carbide further into industrial production, its quality and its physical qualities will need to be improved. With this in mind, production of construction materials from high purity silicon carbide attracts particular interest.

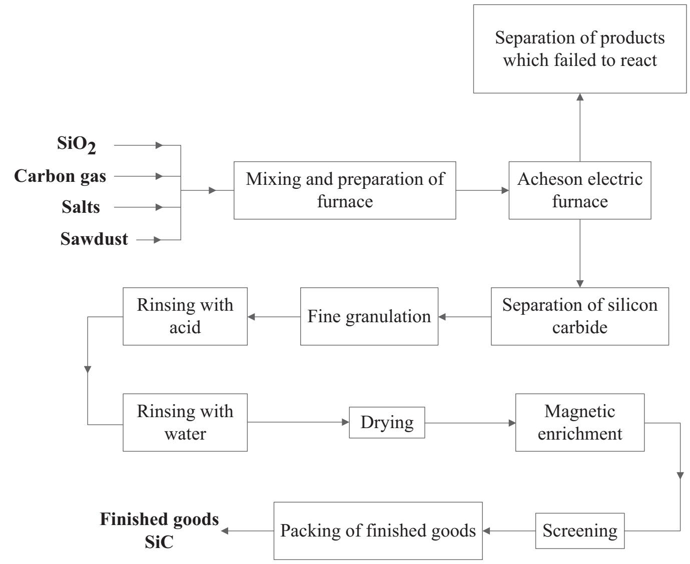


Fig. 1. A process flow diagram for the production of silicon carbide.

On an industrial scale, silicon carbide is produced [6,19] using the carbothermic method for the recovery of silica (the Acheson method) based on the following reaction:

$$SiO_2 + C \xrightarrow{>2000°C} SiC + CO + CO_2. \quad (1)$$

Fig. 1 illustrates the principle of production of general purpose silicon carbide.

## 2. Experimental research and consideration of results

In the practice of manufacturing high construction ceramics it is recommended to use pure homogeneous fine-grained powders (sub-micron) of equidimensional composition, with a homogeneous form and equal reaction to sintering. This set of excellent qualities of silicon carbide powder (rather than individually achieved characteristics) guarantees high quality exceeding of similar goods produced by leading manufacturers.

Fig. 2 is a process flow diagram illustrating the synthesis of high-quality, miniature silicon carbide powders for use in construction [21]. The raw material, which is metallurgical powder, is cleaned of iron, aluminium and calcium impurities, based on methods described in scientific literature, to a purity of 99.95–99.97% mass for silicon [22]. The material is then treated with "drowned jet", on a machine equipped with a reactor, where it is finely powdered, mechanically activated and finally classified once it reaches the purified powder stage. Synthesis of the SiC-powder from previously mechanically activated powder Si is carried out at the maximum temperature of 1600 °C. In the industry the method of SiC-synthesis from the Si-powder without preliminary mechanical activation is applied; however, such process proceeds only at 1800–1900 °C, i.e. at a temperature at 300–400 °C above, than the offered process.

Only finely graded silicon powders from two groups are used in the synthesis, each with an average particle diameter of up to $d_{av} \leq 1.0$ μm and $d_{av} \leq 5.0$ μm, respectively, in a mass of up to