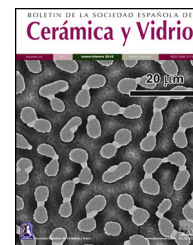




BOLETIN DE LA SOCIEDAD ESPAÑOLA DE

# Cerámica y Vidrio

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## Low-temperature synthesis of silicon carbide powder using shungite

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### ARTICLE INFO

#### Article history:

Received 26 December 2015

Accepted 12 April 2016

Available online xxx

#### Keywords:

Silicon carbide

Abrasives powder

Shungite

Low-temperature synthesis

### ABSTRACT

The paper presents the results of investigation the novel and simple method of synthesis of silicon carbide. As raw material for synthesis was used shungite, natural mineral rich in carbon and silica. The synthesis of SiC is possible in relatively low temperature in range 1500–1600 °C. It is worth emphasising that compared to the most popular method of SiC synthesis (Acheson method where the temperature of synthesis is about 2500 °C) the proposed method is much more effective. The basic properties of products obtained from different form of shungite and in wide range of synthesis temperature were investigated. The process of silicon carbide formation was proposed and discussed. In the case of synthesis SiC from powder of raw materials the product is also in powder form and not requires any additional process (crushing, milling, etc.). Obtained products are pure and after grain classification may be used as abrasive and polishing powders.

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### Síntesis de baja temperatura de polvo de carburo de silicio utilizando shungita

### RESUMEN

Este trabajo presenta un método novedoso de obtención de carburo de silicio a partir de shungita, mineral natural rico en carbono, y sílice. La síntesis se realiza a temperatura relativamente baja (1.500-1.600 °C) respecto a las temperaturas (~2500 °C) requeridas en el método Acheson. Se describen la propiedades básicas de los productos obtenidos a partir de diferentes formas de shungita y utilizando una amplia gama de temperaturas de síntesis. Se propone un proceso de formación del carburo de silicio. Cuando se obtiene el carburo de silicio a partir de materias primas en forma de polvo, el producto tiene también esta forma y no requiere ningún proceso adicional (tritución, molienda, etc.). Una vez clasificados los tamaños, el producto puede utilizarse directamente como abrasivo.

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<http://dx.doi.org/10.1016/j.bsecv.2016.04.003>

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## Introduction

Shungite is a unique coal-like mineral resource discovered in 1879 in the Onega region, which is located near the village of Shunga in Karelia, Russia. According to one of the definitions, shungite is a type of amorphous coal ranked between anthracite and graphite [1]. Therefore, carbon is the one of the main chemical component of shungite; the amount of carbon may range from 10% to 100%. Depending on carbon content in shungite, it is possible to distinguish shungite-1 with a carbon content of 98–100%; shungite-2 with a carbon content of 35–80%; shungite-3 with a carbon content of 20–35%; shungite-4 with a carbon content of 10–20% and shungite-5, in which carbon content is less than 10%. Chemical composition of shungite is determined by the location of deposit. In addition, shungite composition may include  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{K}_2\text{O}$  and S. Shungite has been known in Karelia since the middle of the 18th century. It was used for the production of paints and as a construction and decorative material in the Cathedral of the Icon of the Mother of God, the Winter Palace and the State Russian Museum in Saint Petersburg. In the 19th and 20th centuries, several attempts were made to use shungite as a substitute for hard coal. In the 1970s, an extensive use of shungite as an insulating material had its beginning. Shungite aggregates sourced from the Nigozero deposit, once subject to thermal processing at temperatures ranging from 1090 to 1130 °C, become ball-shaped particles of a low-density of 0.25–0.50 g/cm<sup>3</sup>; the particles are successfully used as light filler materials in concrete structures. Other applications of shungite include electrochemical production of P, Cu, Ni and Co ferroalloys, as a substitute for coke and fusing agents for steel melting, an acid-resistant filler and fire-resistant material, a substitute for graphite in fire-resistant materials and plaster as well as a conductive filler material [1].

There are few papers [2,3] about possibility (in favourable conditions) of carbothermic reduction of silica resulting in the formation of silicon carbide (reaction 1) or metallic silicon (reaction 2).



The natural SiC – Moissanite – has no technological importance. Because of many useful properties of SiC their synthesis is very important and various methods for obtaining silicon carbide in used [4]. The Acheson method is the most popular and well developed SiC synthesis. Also in this method, carbothermic reduction of silica is the basic reaction for SiC formation. At the initial stage, primary SiC (cubic polymorphic 3C form) is obtained. At high temperature above 2500 °C, cubic SiC undergoes decomposition and then the secondary SiC in hexagonal polymorphic 6H and 4H forms is crystallised from gaseous phase. Disadvantage of this method is necessary of crushing and milling of coarse-grained product [4].

In this paper the results of investigation the silicon carbide synthesis by carbothermic reduction of silica from shungite are presented. The syntheses were performed with various

types of reaction beds, i.e. powder, aggregates and pellets. The different temperature of reaction and temperature rate were applied. Full characteristics of synthesis products were obtained, mainly in terms of phase composition.

## Materials and methods

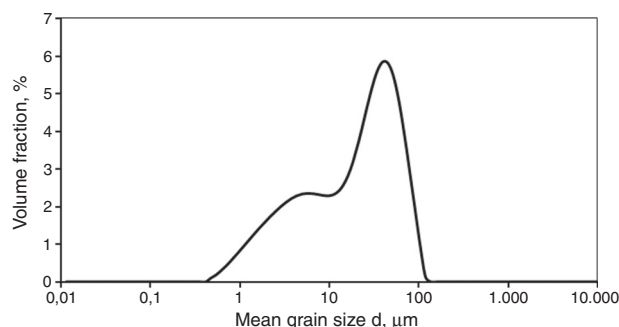
The chemical composition of used shungite is presented in Table 1. The composition of shungite was determined using the X-ray fluorescence method (XRF) and in case of carbon the thermogravimetric method was used.

The shungite used for the tests had the form of aggregates, the dimensions of which were from 5 to 30 mm. First, the aggregates were crushed to obtain approx. 1–2 mm, and then were ground in a rotating-vibration mill in ethyl alcohol for 20 h with  $\text{ZrO}_2$  as the grinding media. The particle size distribution of the obtained shungite powder is presented in Fig. 1.

Calculated – from reaction (1) – content of carbon which is required for complete silica reduction in shungite, is 33.8% by mass and is closed to the amount of carbon determined by a thermogravimetric analysis (34.3%), thus any additives were not used. Also the amount of available silicon to reaction with carbon at high temperature was lower due to high partial pressure of silicon (II) oxide and silicon. The syntheses were initially performed in a high-temperature reactor at temperatures 1600, 1800 and 2000 °C in the flow of argon. The temperature rate was 15 °C per minute. Samples were held in final temperature for one hour. The syntheses were performed on various reaction beds, i.e. shungite aggregates (approx. 10 mm in diameter), pellets about 3–4 mm in height and 13 mm in diameter, formed by uniaxial pressing under pressure of approx. 80 MPa and also powder in a SiC crucible. In second case the temperature 1500, 1550 and 1600 °C with temperature rate 2 °C per minute were applied. The products were characterised in terms of phase composition using the X-ray diffraction (XRD) method. Quantitative phase

**Table 1 – Composition of shungite.**

Phase composition	C	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	S	Rest
Contents [mass %]	34.3	56.3	3.80	0.81	1.33	1.52	1.94



**Fig. 1 – Grain size distribution of shungite powder.**

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