INFLUENCE OF CHAMBER ATMOSPHERE PRESSURE ON THE PRODUCT OF SI-C PLASMODYNAMIC SYNTHESIS

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At the present time, silicon carbide is interesting in many spheres of human activity. Ceramic materials based on silicon carbide are widely used due to their high physical and chemical properties, such as melting point, thermal conductivity, oxidation resistance and mechanical strength [1].

It is established that the properties of ceramics based on nanoscale particles and powders are significantly different from those similar coarse-grained ceramics. Nanosized silicon carbide products are characterized by a lower sintering temperature. Nanoscale SiC powders are used for the production of composites with a metal matrix, ceramic foams.

As a component of high-strength ceramics, nanopowder should have the following characteristics: fine particles, about 40-100 nm, relatively uniform shape and high purity [2]. Now, there are various methods for obtaining nanopowders, including silicon carbide, which have their advantages and disadvantages [2,3].

The preparation of nanodisperse silicon carbide based on plasmodynamic synthesis of silicon and carbon. The formation of a nanosized powder is shaped in a hypervelocity jet of a carbon-silicon plasma generated by a coaxial magnetoplasma accelerator (CMPA) with graphite electrodes [4]. CMPA is powered by a capacitive energy storage (C = 6 mF, U = 3 kV).

The precursors in experiments were carbon black (soot) and silicon powder, which were mixed. Then mixture with a mass of 0.5 g, was placed (in a ratio Si : C – 3 : 1) into the plasma formation zone at the beginning of the accelerating channel. Hypervelocity plasma flowing was carried out in the hermetic reactor chamber`s atmosphere, which was filled with argon at variate pressures (p = 0.5, 1.5, 3.0, 5.0 atm.). The synthesized powder was gray. The product was collected after complete precipitation of the suspended particles on the bottom-wall and the reactor chamber.

The powder was investigated by the following methods: X-ray diffractometry (Shimadzu XRD 6000 (CuKα - radiation)); TEM-transmission electron microscopy (Philips CM 12).

Fig.1 shows powder`s X-ray diffraction which were obtained with a significant difference in pressure. The summation of reflexes of different intensity indicates the presence of several components in the products. The structure-phase analysis of XRD was complete using PowderCell 2.4 software and a PDF4+ structure database. Using them, we can conclude that in all cases the reflex with the highest intensity corresponds to cubic silicon carbide β-SiC (in the fig. 1 - ▲ 111).

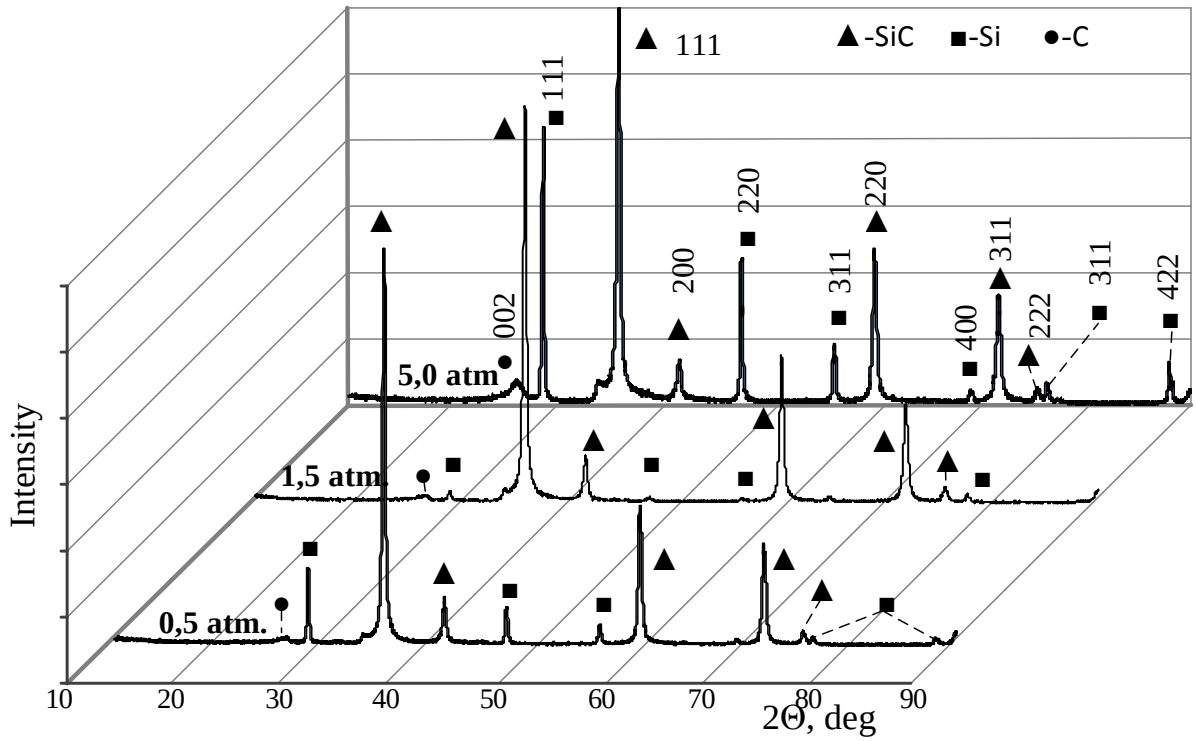

Fig. 1. X-ray diffractions of synthesized powders.

According to the summary table of the experiments (Tab. 1), the mass content of cubic silicon carbide predominates in powders of all experiments. Also, coherent-scattering region (CSR) values shows that the product of synthesis is nanodispersed. But Fig.2 shows that SiC content decreases, when atmospheric pressure in the reactor chamber increases. This dependence is determined by the concentration of the gas atmosphere which resists to movement of the plasma jet entering to the reactor chamber. Silicon carbide has no time to be formed sufficiently, so the content of additional phases (cubic silicon Si and graphite C) increase.

Tab. 1. Basic data of structural and phase analysis.

Phase	SiC		Si		C	
p, atm	Mass. %	CSR, nm	Mass. %	CSR, nm	Mass. %	CSR, nm
0.5	90.9	60	6.4	52.4	2.7	40
1.5	96.8	50	0.9	17	2.3	35
3.0	88.3	38	2.0	24	9.7	20
5.0	67.8	27	19.9	74	12.3	14

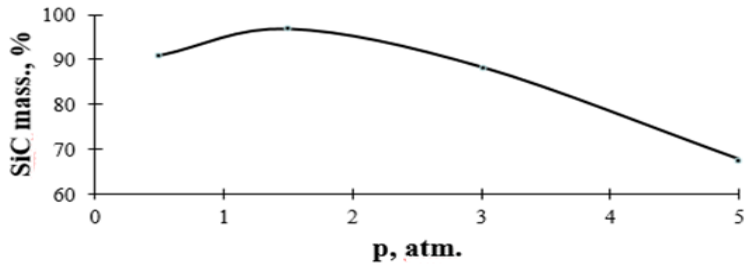

Fig. 2. Dependence of the SiC content on the atmospheric pressure of the reactor chamber.

TEM pictures (typical micrographs are shown in Fig.3-4) confirm that the increase in pressure adversely affects the purity of the product. Fig. 4 clearly shows all phase components of the synthesized product: silicon carbide, as polygons with conjugate vertices, as well as finely dispersed silicon and carbon.

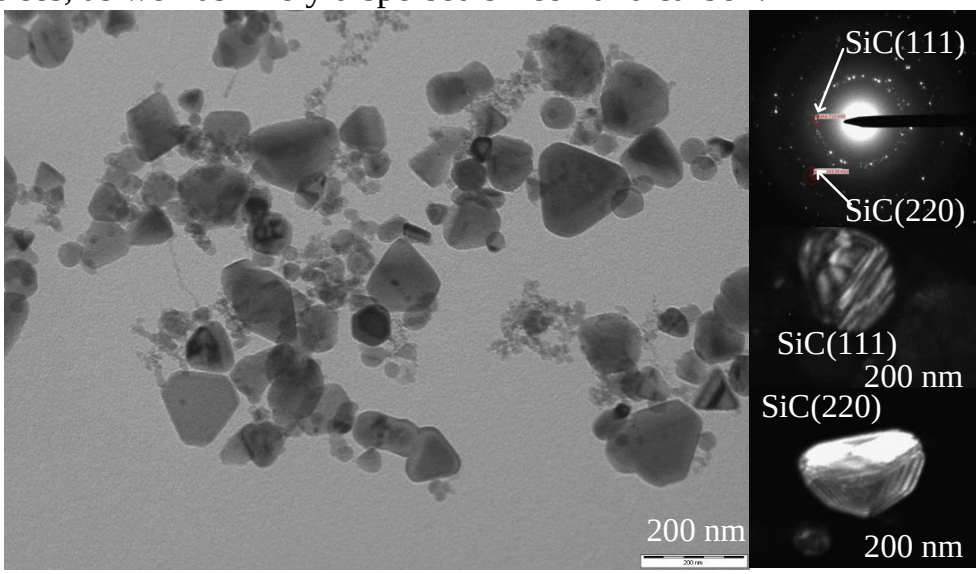

Fig. 3. A microphotograph of the product obtained at a pressure p = 1.5 atm.

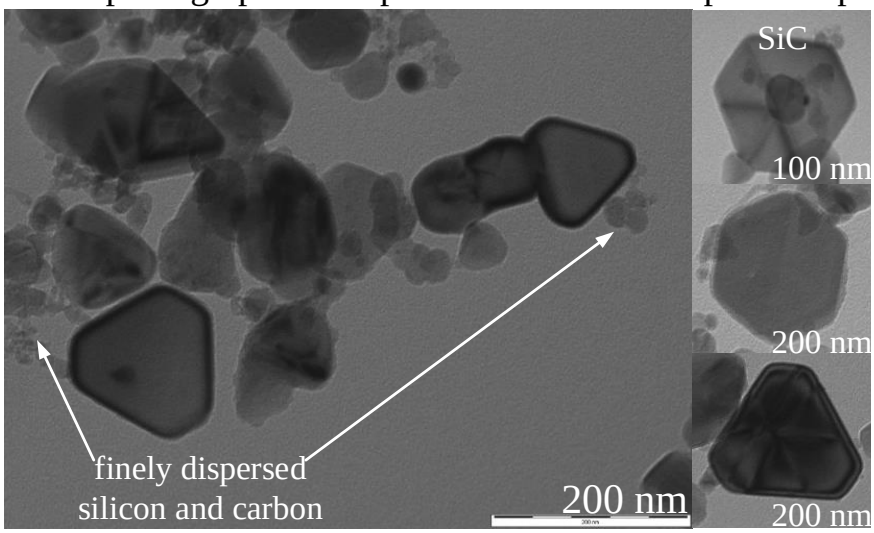

Fig. 4. TEM images of cubic silicon carbide.

The experimental data proves that nanodispersed cubic silicon carbide β-SiC was obtained in all experiments. It is empirically established that the content of silicon carbide decreases when reactor chamber`s atmospheric pressure rises. The characteristics required for high-strength ceramics: a high content of cubic silicon carbide (96.8%) and a nanodispersed composition are reached at p = 1.5 atm.

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СИНТЕЗ КАРБИДА КРЕМНИЯ КОМБИНИРОВАННЫМ МЕТОДОМ С ИСПОЛЬЗОВАНИЕМ НАНОВОЛОКНИСТОГО УГЛЕРОДА

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Неметаллические тугоплавкие соединения обладают такими свойствами, как высокая температура плавления, твердость, химическая инертность, благодаря чему они широко применяются в качестве основы конструкционных, полупроводниковых, жаропрочных и абразивных материалов. Одним из наиболее важных представителей соединений данного класса является карбид кремния.

Основными способами получения карбида кремния считаются карботермическое восстановление, проходящее с поглощением теплоты при температурах свыше 2000 ºС, и синтез из элементов, который можно охарактеризовать как самораспространяющийся высокотемпературный синтез (СВС). В данной работе исследуется возможность объединения двух этих технологий с целью получения высокотемпературной модификации карбида кремния при более низких затратах энергии.

Шихта готовилась в соответствии со стехиометрией обеих реакций и далее смешивалась в соотношениях карботермическое восстановление : синтез из элементов 1:1. Суммарное уравнение химической реакции комбинированного метода синтеза карбида кремния в общем виде:

$$SiO2 + Si + 4 \cdot C = 2 \cdot SiC + 2 \cdot CO$$

В качестве углеродного материала для синтеза карбида кремния был выбран нановолокнистый углерод, характеризующийся высоким значением удельной поверхности (~150 $м^2/г$) [1]. Ранее нановолокнистый углерод для синтеза карбида кремния не использовался.

Процесс синтеза проводился в индукционной печи тигельного типа в среде аргона. Было решено провести эксперименты с разными температурами синтеза: 1600 °С (образец SiC-16) и 1800 °С (образец SiC-18). Продолжительность каждого синтеза составила около 20 минут. Полнота прохождения процесса, протекающего с участием газовых компонентов, определялась по убыли массы.