OBTAINING SILICON CARBIDE BASED CERAMICS BY SPARK PLASMA SINTERING

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Silicon carbide (SiC) is interesting in many spheres of human activity. Ceramic materials based on SiC are widely used due to their significant properties, such as high hardness and mechanical strength, low density, good wear resistance, excellent mechanical and chemical stability at high temperatures [1]. It is established that the properties of ceramics based on ultrafine and nanopowders are significantly different from those similar coarse-grained ceramics [2].

In this paper is considered the possibility of synthesizing silicon carbide ceramic using the spark plasma sintering (SPS) method. The main advantage of this method is a high sintering rate, which allows to obtain submicron and nanoceramics. Sintering was performed by the SPS 10-4 Thermal Technology. The basic sintering parameters: $T_{SPS} = 1750\,°C$, $p = 60$ MPa, $\Delta T/\Delta t = 100$ K/min, $\Delta t_{SPS} = 10$ min. The environment in all experiments was vacuum.

To compare the ceramics quality, several series of experiments were carried out using various powders based on the commercial hexagonal SiC F1200 powder (SiC_{com}) and cubic SiC of the plasma dynamic synthesis (SiC_{plasm}) with an average size of single-crystal particles of ~70 nm [3]. The characteristics of the ceramic products are given in Table 1. Mechanical characteristics of ceramic specimens were studied by the Galileo Isoscan HV2 OD hardness tester.

Based on the data of hardness and density of the first two experiments (Table 1), it was decided to use sintering additives, which allows to achieve significant improvement in the properties of the ceramic products [4]. The experiments on obtaining the ceramic specimens using Al-B-C mixture (4–2–2 %) were carried out for SiC_{com} and SiC_{plasm}. In both cases a significant effect is achieved in improving the mechanical characteristics (both density and hardness) of SiC ceramics.

Fig. 1 shows the results of scanning microscopy of ceramic samples. Sample 2 (Fig. 1a) has large grains and high porosity. Fig. 1b demonstrates the formation of low-porosity and dense ceramic which was obtained in experiments with SiCplasm and sintering additives. The grain size of the ceramics structure is comparable to the particle size in the original powder. It can be argued that the high values of density and hardness for sample 4 are obtained due to the saving the ultrafine structure.

The result of this work were obtaining ceramic

Table 1. Mechanical characteristics of ceramics

№	Precursors	ρ, g/cm^3	H_{av}, GPa
1	SiC_{com}	2.25	1.40±0.5
2	SiC_{plasm}	2.63	5.71±0.3
3	SiC_{com}+Al(4 %)+ +B(2 %)+C(2 %)	3.04	22.8±0.3
4	SiC_{plasm}+Al(4 %)+ +B(2 %)+C(2 %)	3.12	25.9±0.3

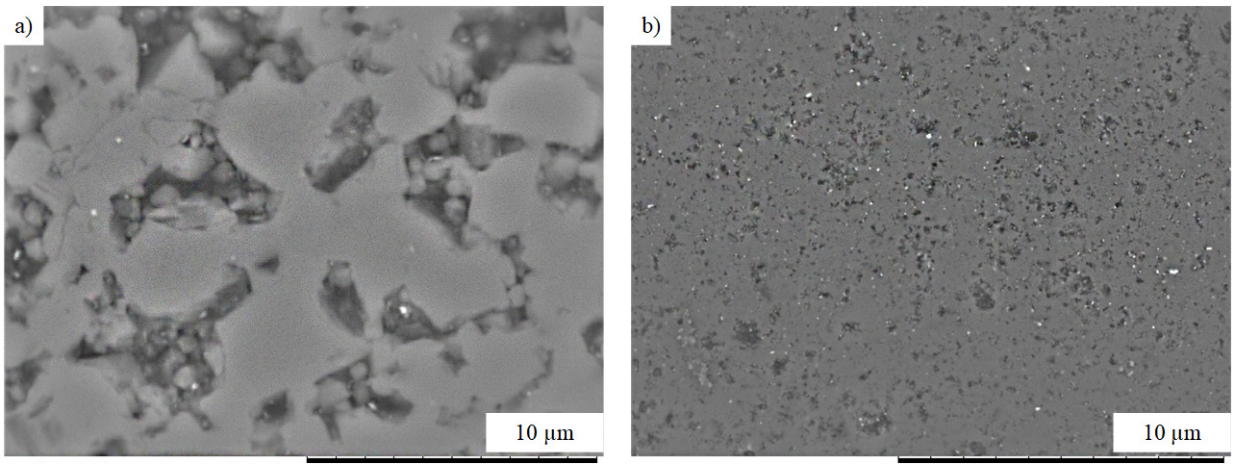

Fig. 1. *SEM-images of ceramic products*

samples from silicon carbide by spark plasma sintering. The ceramics products were investigated by scanning electron microscopy (SEM) by Hitachi TM 3000. It has been established that using sintering additives and ultrafine powders it is possible to receive high-strength silicon carbide ceramics. The sample with the highest density and hardness values was obtained in a series of experiments using silicon carbide powder from plasmadynamic synthesis and using Al–B–C sintering additives: $\rho = 3.12$ g/cm^3; $H_{av} = 26$ GPa.

References

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ARYLATION OF ALKANES USING ARENDIAZONIUM TOZYLATES

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Aromatic diazonium salts have been known as one of the most valuable building blocks in organic synthesis. Considerable attention has been paid to the evaluation of their reactivity in the various transformations with the formation of new carbon-carbon bonds [1–3]. One of the most well-known reactions is the arylation of alkenes by the Matsuda-Heck mechanism.

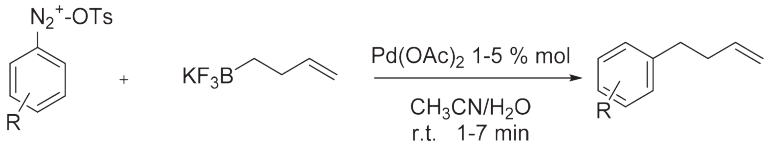
Previously, we showed that arenediazonium tosylates is able to arylate potassium vinyltrifluoroborate in the presence of 1 mol% $Pd(OAc)_2$ at room temperature [4]. In this contribution, we investigated the possibility of the formation of C_{sp2}–C_{sp3} bonds in Pd-catalyzed cross-coupling with potassium alkytrifluoroborates.

As a model substrate, we selected potassium methyltrifluoroborate and potassium (3-butynyl) trifluoroborate. The reaction was carried out under similar conditions, as shown earlier [4].

It was found that the arylation of potassium (3-butynyl)trifluoroborate proceeded smoothly with high reaction rate forming the desired product with high yields (from 57 to 92%).

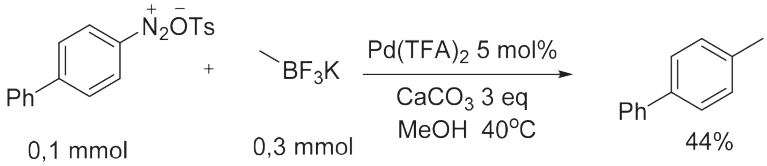
On the other hand, the arylation of potassium methyltrifluoroborate required the increased temperature. The optimization of the reaction conditions allowed to achieve the 44% yield of desired methylbiphenyl in the presence of $Pd(TFA)_2$ and $CaCO_3$ as a base.

At present, the optimization of the reaction conditions is finished. When varying reaction conditions, we obtained better results using $Pd(OAc)_2$ in methanol. It was found that arylation of potassium methyltrifluoroborate with biphenyldiazonium



R= 4-NO_2 83 %; 2-NO_2 92 %; 4-Br 54 %; 3-CF_3 57 %; 4-COOEt 76 %

Scheme 1.



Scheme 2.