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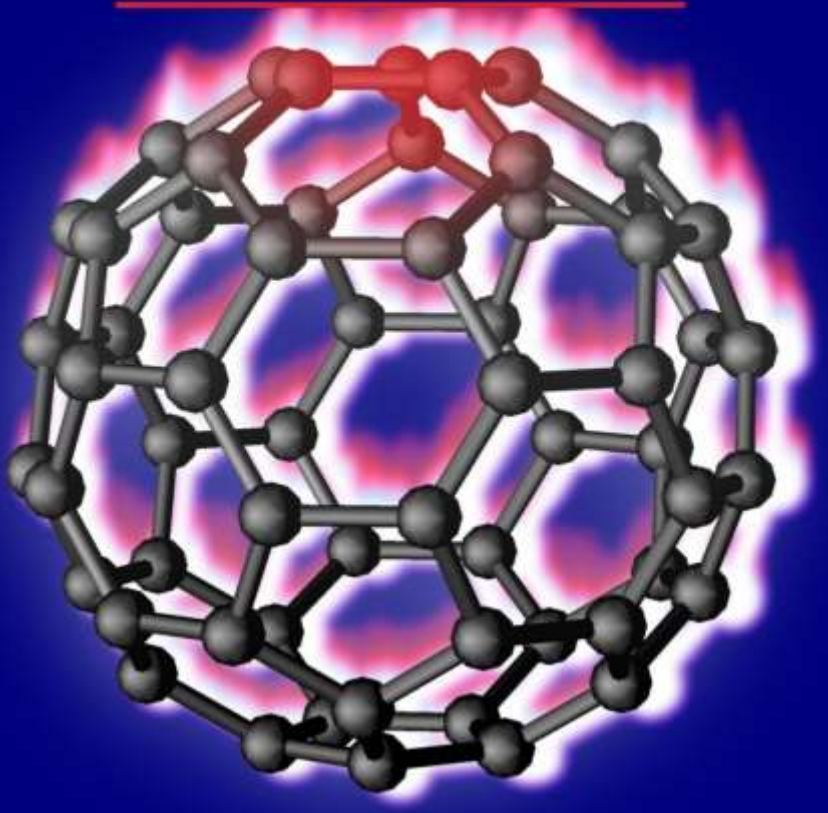
National Nanotechnology Laboratory of Open Type

X International Symposium

The Physics and Chemistry of Carbon and Nanoenergetic Materials



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National Nanotechnology Laboratory of Open Type
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Chapter 7

POSTER PRESENTATIONS

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SYNTHESIS OF SiC NANOSTRUCTURES ON THE SURFACE OF COPPER FILMS

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Abstract. The results of microwave plasma assisted synthesis of silicon carbide nanostructures by the method of CVD are shown in the paper. The porous and polished silicon plates with orientations [100] and [111] were used as substrates, the copper buffer layer was a catalyst. The experiments were carried out at temperatures of 600 and 700 ºC. The results of studies of the samples by X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) are presented. As a result of the experiments, recommendations for more efficient growth of silicon carbide nanofibers were developed.

Introduction

SiC nanostructures have become an important field of research due to their potential use as nanostructured composite materials [1]. SiC nanostructures (NS) possess high chemical resistance, thermal stability, mechanical strength and hardness, controlled by electrical conductivity and low density [2-4]. Due to these properties, SiC is considered as promising material for use in reinforcing materials, such as composites, biomaterials, high-temperature semiconductor devices used in aggressive conditions [5,6].

In recent times, various forms of SiC have been successfully synthesized, including SiC nanostructures [7], nanorods [8], nanofibers and nanotubes [9, 10]. The different methods are used to synthesize SiC fibers, such as electrospinning [11], carbothermic reduction process [12], etc. Despite this, the search for a simpler and more efficient method for the growth of SiC nanostructures will still be relevant. The process of microwave assisted chemical vapor deposition (MW-CVD) [13] is the most suitable and widely used method for obtaining fibers of various shapes with a high-purity composition and pronounced orientation.

Experimental

Silicon plates (Siegert Wafer GmbH, Germany) with orientation [100] and [111] were used as substrates. Also, in order to activate the surface during synthesis, substrates of porous silicon (PSi) were prepared at the Educational Laboratory of Semiconductor Instrumentation (KazNU). Cu films were deposited for 5 minutes by the method of magnetron sputtering based on VUP-5M. The magnitude of the potential bias between the electrodes was 600 V, the plasma current was 28-30 mA. Copper deposition was carried out in the Ar atmosphere, the working pressure of which in the chambers was 10^{-2} Torr.

NS synthesis was carried out in the Department of the Surface and Technology of New Materials of Institute of Materials Engineering of the University of Siegen (Germany) on equipment of microwave plasma assisted chemical vapor deposition (MW-CVD) of the ASTEX system (frequency 2.45 GHz). The synthesis temperature ranged from 600 to 700 °C in steps of 100 °C and was measured using infrared pyrometer of model Chino IR-AP M0011 (Japan). Plasma power was fixed at two values of 1600 and 1800 W. Depending on the power of the plasma, the pressure in the

chamber (40-47 Torr) was changed. The working gas was a mixture of trimethylsilane ($(CH_3)_4Si$) and hydrogen, the flow rates of which were 10 and 400 cm^3/min, respectively. The duration of the experiments is 120 min.

Zeiss scanning electron microscope (SEM) of the model Gemini Ultra 55 with a device for X-ray spectral microanalysis (Thermo Scientific) was used to study the morphology and elemental analysis of samples. X-ray diffraction was carried out using Rigaku MiniFlex 600 XRD diffractometer (Laboratory of X-ray diffraction analysis of Al-Farabi KazNU).

Results and discussion

Analysis of SEM images of the samples shows that the growth of NS on PSi surface is more massive unlike polished Si. According to X-ray spectral analysis, the carbon content in the samples of NS varies from 30 to 70%. Analysis of the diffractograms shows that the samples consist of several phases that correspond to the following structural models: cubic silicon carbide β-SiC, cubic copper Cu crystal, graphite gC.

Samples obtained at a substrate temperature of 600 °C are amorphous carbon films with fibrous formations on the surface. It can be noted that longer NS are formed on porous substrates in comparison with NS synthesized on polished substrates. The main difference on diffractograms is that the intensity of X-ray reflection from SiC NS grown on polished silicon is higher. The peaks corresponding to β-SiC [PDF #29-1129] from the (111) and (220) planes were identified on diffractogram. The reflection inherent in copper Cu [PDF #04-0836] appears from the planes (111) and (200)at the angles of 43.1° and 50.4°.

Figure 1 shows the diffraction patterns and SEM images of SiC NS synthesized on polished Si and PSi with orientation [111] at 700 °C.

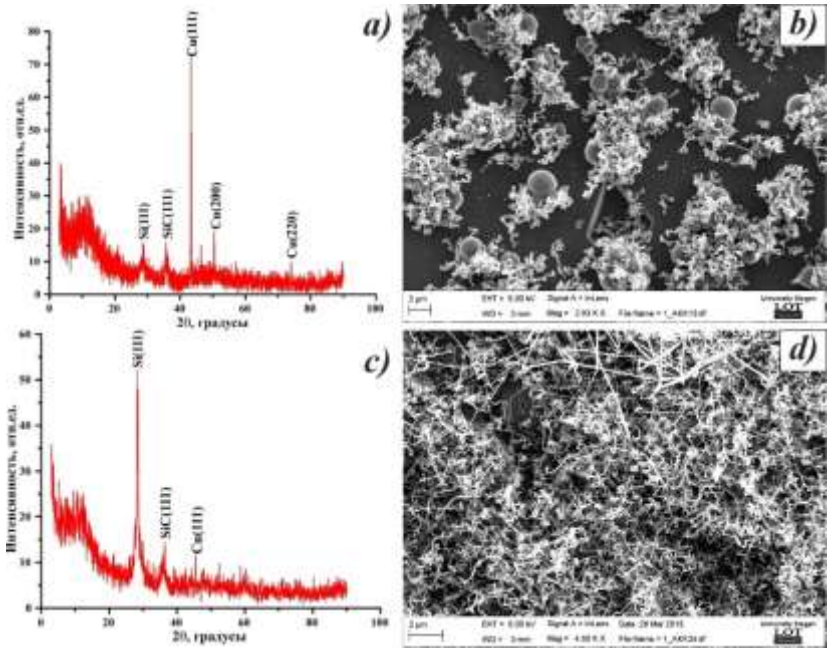

Figure 1 - Diffractograms and SEM micrographs of samples synthesized at a substrate temperature of 700 °C on polished (*a*, *b*) and porous (c, *d*) Si (111)

The analysis of the diffractograms (Fig. 1) shows that the reflection at angle of $2\theta \approx 35.7°$ corresponds to β-SiC [F-43m {216}, PDF #29-1129] from the (111) plane. The reflection from the

angles of 43.1°, 50.4° and 74.2° is due to the presence of copper Cu [Fm-3m {225}, PDF # 04-0836] from planes (111), (200) and (220). Comparative XRD shows that the reflection from the buffer layer of copper is more intense on the PSi. This indicates that the buffer layer of copper on these samples is thicker, but this has slight effect on the growth of silicon carbide. An interesting trend is observed in all diffractograms, when reflection from copper oxide appears on the substrate with orientation [100], whereas it is absent for orientation [111]. This legitimacy is valid for both porous and polished silicon. This is explained by the difference in the reticular density of the buffer layer.

Conclusion

In the course of the research, experiments on the synthesis of SiC nanostructures by the method of microwave plasma assisted chemical vapor deposition were carried out. Studies by SEM have shown that the obtained NS have a diameter of 80-100 nm and a rough surface. The growth of NS is more massive on the surface of PSi, in contrast to the polished Si. According to the results of X-ray diffraction study, it can be said that the samples consist of phases of cubic silicon carbide β-SiC, a cubic copper crystal Cu and gC graphite. Thus, amorphous carbon films are formed at a temperature of 600 °C, whereas β-SiC nanostructures are formed at 700 °C.

For a more detailed analysis of the NS structure, it is necessary to carry out additional studies using transmission electron microscopy and diffraction of electrons.

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