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Structure and morphology of SiC nanostructures synthesized on Cu films

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ABSTRACT

The results of experiments on the synthesis of SiC nanostructures by chemical vapor deposition in microwave plasma are presented in this article. The single crystal silicon plates with orientation [111] which previously passed chemical purification were used as substrates. Furthermore, the substrates of porous silicon were prepared in order to activate the surface during the synthesis. The synthesis temperatures were 600 °C and 700 °C. Studies by scanning electron microscopy showed that formed nanostructures have a various diameter and a rough surface. The results of studies by Raman scattering confirmed that SiC nanostructures with structure of 3C-SiC are formed. Besides, the presence of main carbon peaks on both types of substrates which correspond to the carbon nanostructures should be noted.

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1. Introduction

Silicon carbide (SiC) is an important semiconductor material that can operate at high powers, high temperatures and frequencies. This material has excellent thermal stability and is a broadband semiconductor, which properties suggest promising applications for high-power, high-temperature (up to 600 °C) and high-frequency (up to 20 GHz) electronic devices [1,2]. It is well known that many types of sharp-pointed nanostructured materials, such as carbon nanotubes, silicon and silicon carbide nanofibers, are promising materials [3–6]. For example, one-dimensional SiC nanostructures have high compression ratios that are suitable for field emitters due to their field amplification effect [7].

Several methods are used to synthesize SiC nanostructures, including sol-gel [3], carbothermal reduction of SiO_2, decomposition of silicon organic compounds, laser ablation [8], and chemical vapor deposition [9]. To date, CVD has often been used to produce SiC nanofibers with Si sources in the solid or gas phase [10]. Considering the manifold difference of this method, the synthesis of SiC nanofibers using microwave plasma assisted chemical vapor deposition (MWCVD) has not been sufficiently investigated. Based on this, study of the influence of synthesis parameters of MWCVD on the surface morphology of SiC nanostructures is relevant.

This paper presents the results of studies of silicon carbide nanostructures obtained by the method of MWCVD on the surface of copper films previously deposited on porous silicon wafers by Raman scattering (RS), scanning electron microscopy (SEM) and X-ray analysis.

2. Experimental

2.1. Preparation and investigation of substrates

The monocrystalline silicon plates (analogue of brand KDB-20, manufacturer Siegert Wafer GmbH, Germany) 1 × 1 cm with orientation [111] was used as substrates and basis for copper films. The substrates of porous silicon (PSi) were prepared in the Educational Laboratory of Semiconductor Instrumentation, Faculty of Physics and Technology, Kazakh National University in order to activate the surface during synthesis. Crystalline silicon was pretreated in acetone and placed in a solution of H_2SO_4:H_2O_2 for 5 min, followed by washing in deionized water. The plates were then immersed in the etch HF:H_2O for 1 min, after which they were thoroughly washed. The PSi was formed by electrochemical anodizing in a modified solution with composition of HF (45%): ethoxyethanol: water in a ratio of 1:2:1. The current density and anodization time were, respectively, 15 mA and 10 min. After etching, porous silicon

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substrates were thoroughly washed in deionized water and dried in air using lighting with a red lamp for 5 min. Fig. 1 shows SEM image of PSi.

The copper films were deposited on surface of PSi plates by DC magnetron sputtering using equipment VUP-5M for 5 min. Sputtering was carried out in the flow of working gas Ar at a pressure of 10^{-2} Torr. The flow rate of Ar was 6 cm^3/min and it was controlled by the gas flow controller MCV-500SCCM. The deposition was conducted at a constant voltage on the anode target (740 V) and plasma current of 35 mA.

2.2. Synthesis of silicon carbide nanostructures

Synthesis of nanostructures was carried out in the Department of Surface and Technology of New Materials at the Institute of Materials Science, University of Siegen (Germany), on equipment of MWCVD of the ASTEX system (frequency 2.45 GHz).

Prior to the experiments, the substrates were purified with ethanol and then washed with distilled water, drying was carried out at room temperature. The synthesis was carried out at temperature of 600, 700 °C, the plasma power of 1600, 1800 W and the chamber pressure of 40, 47 Torr. The temperature was measured using infrared pyrometer of model Chino IR-AP M0011 (Japan).

The mixture of trimethylsilane ($(CH_3)_4Si$) and hydrogen was used as working gas, the flow rates of which were 10 and 400 cm^3/min, respectively. The duration of the experiments was 120 min.

2.3. Characterization

The samples of PSi were investigated by SEM in the National nanotechnological laboratory of open type (Almaty, Kazakhstan) using a microscope Quanta 3D 200i.

The obtained samples of SiC nanostructures were studied by the method of SEM, which was also carried out at the Institute of Materials Sciene of the University of Siegen. The field emission scanning electron microscope with ultra-high resolution of model Gemini Ultra 55 of the company Zeiss, with a device for X-ray microanalysis of the company «Thermo Scientific» was used to study the morphology and thickness of the samples.

The nanostructures were investigated by Raman spectroscopy using spectrometer NT-MDT NTegra Spectra (laser wavelength $\lambda = 473$ nm) at The National Nanotechnology Laboratory of open type.

The study of the structure of the obtained samples is carried out by the method of X-ray analysis using diffractometer Rigaku Mini Flex 600 XRD (X-ray analysis laboratory of Al-Farabi KazNU, Almaty, Kazakhstan). Radiographs of samples were obtained using copper radiation ($\lambda = 1.5406$ Å) in digital form. Processing of X-ray spectra to determine angular position and intensity of the reflection was performed in program OriginPro 8.1. PDXL2 software package with the base of diffractometric data PDF-2 was used for the phase analysis.

3. Results and discussion

Fig. 2 presents the results of SEM, XRD and Raman investigations of nanostructures synthesized at 600 °C. It can be seen from SEM results that nanostructures grow preferentially oriented to the substrate. The diameter of the nanostructure is ~85 nm and length is up to several micrometers. The massive growth of nanostructures over the entire surface of the substrate is observed. Fig. 2a shows that the height of the synthesized nanostructures is 1.6 μm. Fig. 2c presents X-ray spectra of the obtained nanostructures. X-ray qualitative analysis of the phase composition of the sample indicates presence of Si, SiC and Cu. The diffraction pattern of the sample demonstrates the reflection from the planes (111) and (200) ($2\theta \approx 43.1°$ and 50,4°, Fm-3 m {225}, PDF # 04-0836) which are characteristic of copper. Also, the characteristic reflection from the substrate of monocrystalline silicon Si (111) appears at angle of 28.4°. The calculation of the diffractogram shows that the reflection at angles of $2\theta \approx 35.7°$ and 59.8° corresponds to β-SiC [F-43 m {216}, PDF # 29-1129] from (111) and (220) planes. Fig. 2d shows the Raman spectra of the samples. There are peaks of silicon carbide in the region of 777.8 and 965.7 cm^{-1}, which can indicate the formation of silicon carbide film with a 3C-SiC structure [11]. In addition, one can see the main carbon peaks in the range of 1344.4 and 1573.1 cm^{-1}, which correspond to carbon nanostructures. Moreover, peaks of the second order are observed in the region of 2683.8 and 2915.7 cm^{-1}, which correspond to 2D and D + G groups [12,13]. It can be seen that the intensity of D peak is higher than that of G peak, which is inherent in defective samples. Based on this, it can be assumed that nanostructures have not high quality.

Fig. 3 presents the results of studies by SEM, XRD and Raman scattering of nanostructures synthesized at 700 °C. SEM results show oriented growth of nanostructures. It was also found that nanostructures grown at 700 °C have a small diameter and a large length. In Fig. 3d, in the region of 777.8 and 972.5 cm^{-1}, characteristic peaks of SiC, corresponding to the LO and TO modes are observed. Peaks observed within 1341.2 and 1570 cm^{-1} are inherent in carbon nanotubes. It can be confirmed by the presence of second-order peaks 2D and D + G at 2675.3 and 2907.4 cm^{-1}, respectively. One can note a peak in the range of 1428.8 cm^{-1},

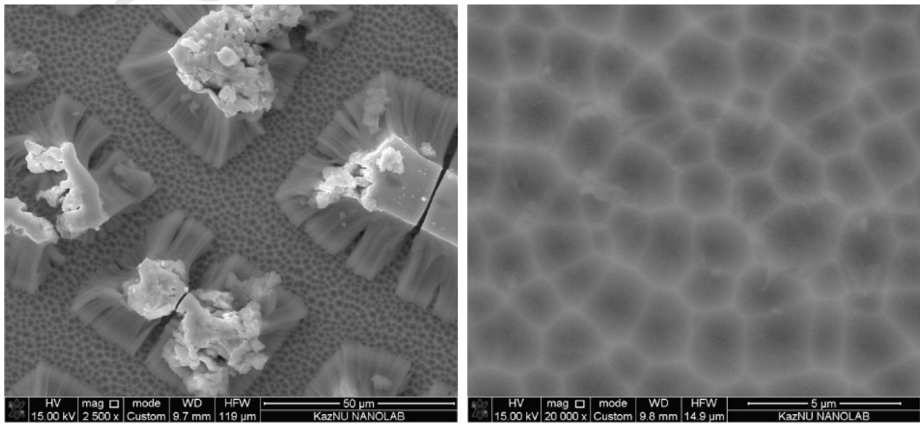

Fig. 1. SEM images of porous silicon surface.

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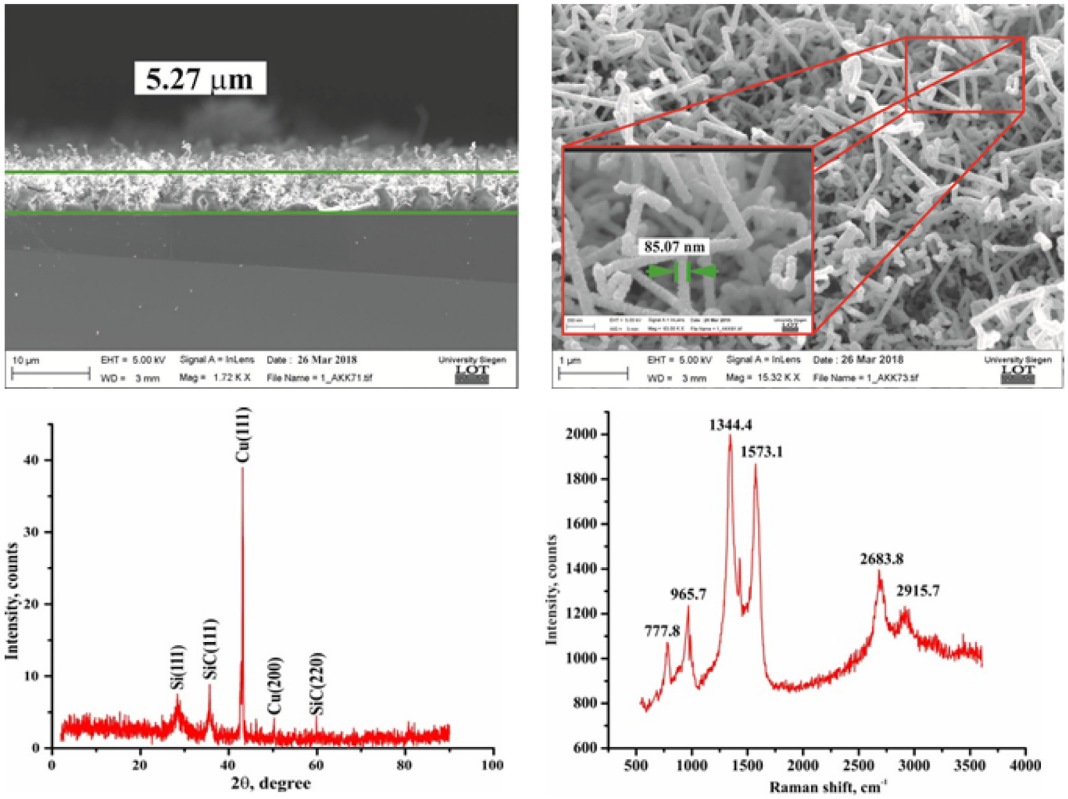

Fig. 2. SEM images and XRD, Raman spectra of nanostructures synthesized at 600 °C.

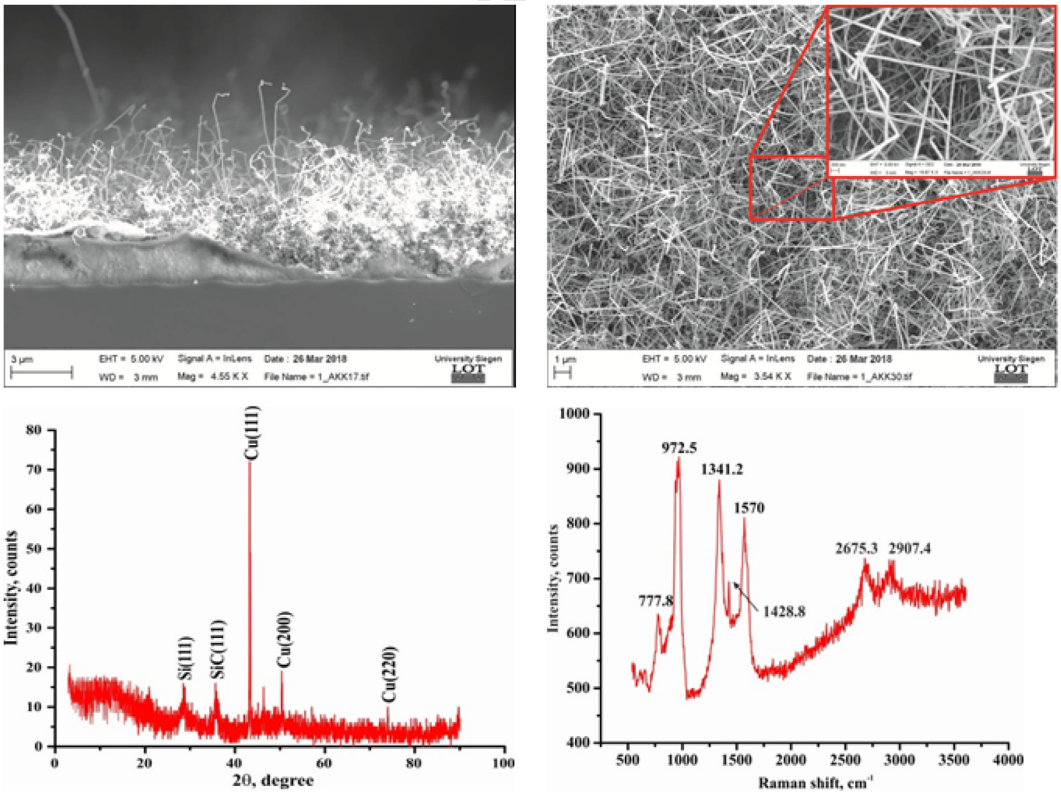

Fig. 3. SEM images and XRD, Raman spectra of nanostructures synthesized at 700 °C.

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which belongs to CH_3 groups [13]. XRD results of samples grown at 700 °C are similar to samples synthesized at 600 °C, but show higher intensity.

4. Conclusions

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 the method of scanning electron microscopy have shown that the formed nanostructures have a diameter from ten to several hundred nm and a rough surface. Analysis of the samples shows the massive growth of nanostructures on the surface of porous silicon.

XRD results show that the obtained nanostructures are 3C-SiC polytypic silicon carbide nanostructures. It is also confirmed by results of studies by Raman scattering. Besides, the presence of main carbon peaks in the range of ~1300 and 1500 cm^{-1}, which corresponds to the carbon nanostructures should be noted.

It is necessary to conduct additional studies using transmission electron microscopy and diffraction of electrons for a more detailed analysis of the structure of the obtained nanostructures. Furthermore, the possibility of using polycrystalline silicon as a substrate for the synthesis of silicon carbide nanostructures will be studied.

CRediT authorship contribution statement

Bauyrzhan Zhumadilov: Software, Validation. **Gulnur Suyundykova:** Investigation, Software, Visualization. **Gulmira Partizan:** Data curation, Writing - original draft, Validation. **Aidar Kenzhegulov:** Conceptualization, Methodology. **Botagoz Medyanova:** Formal analysis, Software. **Bakhodir Aliyev:** Supervision, Formal analysis.

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References

[1] G. Pensl, H. Morkoc, B. Monemar, E. Janzen, Silicon Carbide, III-Nitrides and Related Materials, Materials Science Forum, Trans Tech Publications, Switzerland, 1998.
[2] G.L. Harris, Properties of SiC, EMIS Data reviews Series, 13, The Institute of Electrical Engineers, London, 1995.
[3] Y.J. Hao, G.Q. Jin, X.D. Han, X.Y. Guo, Synthesis and characterization of bamboo-like SiC nanofibers, J. Mater. Lett. 60 (2006) 1334–1337.
[4] S. Fan, M.G. Chapline, N.R. Franklin, T.W. Tombler, A.M. Cassel, H. Dai, Self-oriented regular arrays of carbon nanotubes and their field emission properties, J. Sci. 283 (1999) 512–514.
[5] J. Li, W. Lei, X. Zhang, X. Zhou, Q. Wang, Field emission characteristic of screen-printed carbon nanotube cathode, J. Appl. Surf. Sci. 220 (2003) 96–104.
[6] Y.B. Li, Y. Bando, D. Golberg, ZnO nanoneedles with tip surface perturbations: excellent field emitters, J. Appl. Phys. Lett. 84 (2004) 3603–3605.
[7] M.B. Rizk, C. Assouar, M. Gatel, J. Belmahi, J. Lambert, Bougdira, Synthesis of carbon coated β-SiC nanofibers by microwave plasma assisted chemical vapour deposition in CH4/H2 gas mixture, J. Diamond Related Mater. 17 (2008) 1660–1665.
[8] W. Shi, Y. Zheng, H. Peng, N. Wang, C. Sing Lee, S.-T. Lee, Laser ablation synthesis and optical characterization of silicon carbide nanowires, J. Am. Ceram. Soc. 83 (12) (2000) 3228–3230.
[9] S.I. Honda, Y.G. Baek, T. Ikuno, H. Kohara, M. Katayama, K. Oura, T. Hirao, SiC nanofibers grown by high power microwave plasma chemical vapor deposition, J. Appl. Surf. Sci. 212–213 (2003) 378–382.
[10] K. Dul, S. Jonas, B. Handke, The nanostructure and microstructure of SiC surface layers deposited by MWCVD and ECRCVD, J. Appl. Surf. Sci. 425 (2017) 965–971.
[11] S.P. Frank, Application of Infrared, Raman and Resonance Raman Spectroscopy in Biochemsitry (1983) 550 p..
[12] A.C. Ferrari, J. Robertson, Interpretation of Raman spectra of disordered and amorphous carbon, J. Phys. Rev. B. 61 (2000) 14095–14107.
[13] A.C. Ferrari, Raman spectroscopy of graphene and graphite: Disorder, electron-phonon coupling, doping and nonadiabatic effects, J. Solid State Commun. 143 (2007) 47–57.

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