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FORMATION OF β-SiC ON POR-Si/MONO-Si SURFACE ACCORDING TO STRANSKI - KRASTANOW MECHANISM

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We report the synthesis of β-SiC/por-Si/mono-Si heterostructure by a hybrid method, consisting of the electrochemical etching of the single-crystal silicon surface with a subsequent carbidization by a thermal annealing in a methane atmosphere. This method has a number of advantages over the known ones, because it is cheap enough and allows one to form the silicon carbide layers of high- quality. The formed structure was studied by means of SEM, EDX and XRD methods. As a result, the dense β-SiC layer, consisting of an array of the spherical islands with diameters of 2–6 µm, coated with the small pores, was formed on the por-Si/mono-Si surface. The geometric dimensions of the islands were studied by calibrating the sample image in the ImageJ software package. The maximum value of the linear size (diameter) of the island d_{max} = 5.95 µm and the minimum value d_{min} = 2.11 µm were found in the studied area. In general, the average diameter of the islands is d = 3.72 µm. The distribution has the left-sided asymmetry, that is, the smaller islets predominate. Roundness (the ratio of the area to the square of the larger axis) R = 0.86. According to the results of EDX analysis, it was found that the synthesized structure surface consists exclusively of the carbon and silicon atoms, indicating the high quality of the formed structures. It was found that the SiC film crystallizes in the cubic phase. The formation of the islands is explained by means of the layer-plus-island growth model according to Stranski-Krastanow mechanism, which is characterized by the formation of the dense wetting layer with the massive island complex on the surface. It should be also noted that the porous SiC layers of island type can, in turn, show the perspective as the buffers with the heteroepitaxy of the silicon substrate materials.

Keywords: *silicon carbide, Stranski-Krastanow mechanism, layer-plus-island growth, electrochemical etching, thermal annealing, porous layers, crystal lattice*

INTRODUCTION

Due to its unique properties, silicon carbide is the subject of many studies for a long time [1, 2]. In particular, it is characterized by thermal, chemical and radiation resistance [3, 4]. It also has the high stability of the electrical and optical characteristics [5]. This determines its suitability to be used in power electronics and optoelectronics [6].

The silicon carbide films have been widely used as substrates for forming the heteroepitaxial structures [7]. Thus, the layers of graphene [8], nitrides AlN [9], GaN [10] and AlGaN/GaN [11] are formed on the SiC surface. Such structures are applied in laser technology and for creating the integrated circuits [12].

Silicon carbide is usually grown on the single-crystal silicon substrates. The methods such as laser surfacing [13], three-dimensional printing [14], multiphase oxidation [15], *etc.* are used for this purpose.

The main problem here is the low quality of the grown films due to a large number of structural defects. This phenomenon is caused by a significant parameter mismatch of the Si and SiC crystal lattices [16]. This causes the elastic stresses, which, in turn, initiates the growth of extended defects.

The way out of this situation can be the use of buffer layers, which are able to take the relaxation loads of mechanical stresses connected with the parameter mismatch of the lattice and the difference of the thermal expansion coefficients. This will reduce the stresses and avoid the cracks of immassive defects.

The nanostructured layer of the output substrate is often used as the buffer layers. Thus, the application of the graphite layer on the por-InP/mono-InP structure of indium phosphide by annealing in a stream of fine graphite powder was demonstrated in our paper [17]. The porous layers of indium phosphide were also used as the buffer layer for growing InN [18].

The electrochemical etching in acid solutions is one of the simplest nanostructuring methods of the semiconductor surface [19, 20]. Such surface treatment leads to the formation of an array of the etching pits, which create a dense porous frame [21]. The porous layers have a high roughness

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coefficient, which provides an excellent adhesion of the deposited structures with the substrate surface [22]. This helps to eliminate the non-conformity problem of the lattice parameters.

In the proposed study, the SiC synthesis on the substrate of the porous Si is demonstrated, the morphological and structural characteristics of the formed heterostructure is analysed and the film growth mechanism is found.

EXPERIMENT

Experiment samples. Mono-Si samples, grown by the Czochralski method, were used. The ingots were cut into plates sized 1×2 cm by means of the wire cutting method with the diamond-impregnated wire. The plate thickness was 200 μm. The samples were polished on both sides to a mirror shine. The characteristics of the experiment samples are shown in Table.

Table. Characteristics of experiment samples

Conductivity type	n-type
Type of crystal lattice	Cubic
Surface orientation	(111)
Impurity	P (phosphorus)
Concentration of non-basic charge carriers	1.8×10^{17} cm^{-1}
Specific resistance	2 Ohm·cm

Experiment method. The experiment was carried out in two stages. The first stage is the etching of the oxide film on the mono-Si surface and the formation of the porous layer by the electrochemical etching. The second stage is the formation of the SiC film on the silicon surface by the high-temperature deposition.

1st stage. Electrochemical etching. After cutting and polishing the single-crystal silicon surface is characterized by a large number of the broken bonds, causing the growth of its own SiO_2 oxide. The electrochemical etching in the aqueous solution of hydrochloric acid (5%) for 3 min. at a voltage of U = 2V was applied for removing the oxide from the surface. The electrochemical etching was carried out in a Teflon electrochemical cell, equipped with an airflow module and an electrolyte stirring system.

Then the samples were extracted from the electrolyte and washed in deionized water for forming a porous layer. Meanwhile, the electrolyte composition was changed to the aqueous solution of hydrofluoric acid in the following ratio of the components: HF : H_2O = 1 : 1. In this electrolyte composition the samples were etched for 7 min at a constant voltage of U = 5V. The experiment was carried out under room conditions and daylight.

During the experiment the bubbles were observed on the anode and cathode. After the experiment a small amount of the sediment was recorded at the electrolytic cell bottom.

After the experiment, the samples were washed again in deionized water and dried in dry air by means of a muffle furnace for removing the moisture residues by such successive steps:

- 3 h at 150 °C;
- 60 min at 250–300 °C;
- 60 min at 400 °C.

After that, to prevent the oxidation processes, it immediately proceeded to the deposition of silicon carbide.

2nd stage. Thermal deposition. The SiC epitaxial layers were formed by rapid thermal vacuum carbidization by means of a Jipelec JetFirst-100 device. Methane (CH_4) was used as a precursor gas. The carbidization parameters were as follows: temperature mode – gradual increase from 50 to 900 °C, pressure in the reaction chamber $P \sim 1\cdot10^{-2}$ Pa, two treatment series of 50 seconds with an interval of 1 min.

The treated samples were placed in a solution of hydrochloric acid for removing the residuals of unwanted impurities for 20 min. After that, the samples were dried in a nitrogen chamber at a temperature of 150 °C for 1 h.

Characterization. The morphology of the obtained structures was studied by means of a SEO-SEM Inspect S50-B. The component composition of the elements was studied by the energy dispersion analysis (EDX) according to the mapping technology by means of an AZtecOne spectrometer. The composition and crystallinity degree of the structures were determined by means of X-ray diffraction (XRD).

The spectra were measured by means of a Dron-3M difractor on CuK_α-emission in the angle range of 2ϑ 10–80° with a step of 0.01. 3D-modelling of the carbidization process was carried out by means of the ImageG software package.

RESULTS

SEM- analysis. Fig. 1 shows the SEM-image of the surface morphology of the synthesized β-SiC/por-Si/mono-Si heterostructure. It can be seen the presence of the convex spherical islands that tightly cover the silicon surface. The islands are separated by the grooves that do not show anisotropy, but are more random. The deeper and voluminous grooves are observed at some islet joints. Most likely, their appearance is associated with the substrate defects. The channels could be formed during the chemical treatment in HCl solution. If etching is excessive, the HCl-based electrolyte reacts strongly with weak spots in the SiC coating, forming the etching channels.

Furthermore, the ultra-fine pores, the size of which does not exceed 100 nm, can be observed on the island surface. In general, the silicon carbide is a rather inert material and usually does not show the ability to etch the pores during the chemical etching or low-temperature treatment. The appearance of the pores can be explained by etching the "weak" spots of the carbide film during the chemical etching in hydrochloric acid and thermal drying in the chamber. Such weak spots can be the localization sites of uncontrolled impurities and point defects of the crystal lattice of the silicon carbide.

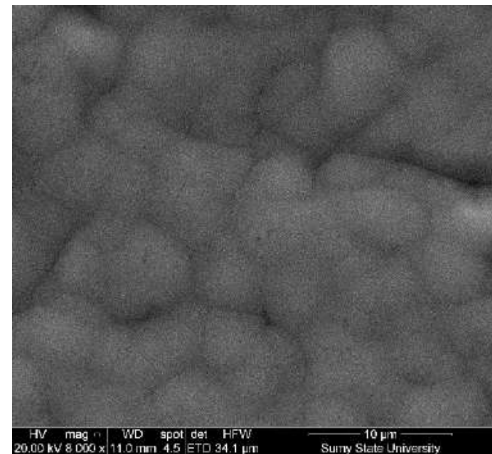

Fig. 1. Morphology of β-SiC/por-Si/mono-Si surface, obtained by means of SEM

The geometric dimensions of the islands were studied by calibrating the sample image in the ImageJ software package (Fig. 2). The maximum value of the linear size (diameter) of the island d_{max} = 5.95 μm and the minimum value d_{min} = 2.11 μm were found in the studied area. In general, the average diameter of the islands is d = 3.72 μm. The distribution has the left-sided asymmetry, that is, the smaller islets predominate. Roundness (the ratio of the area to the square of the larger axis) R = 0.86.

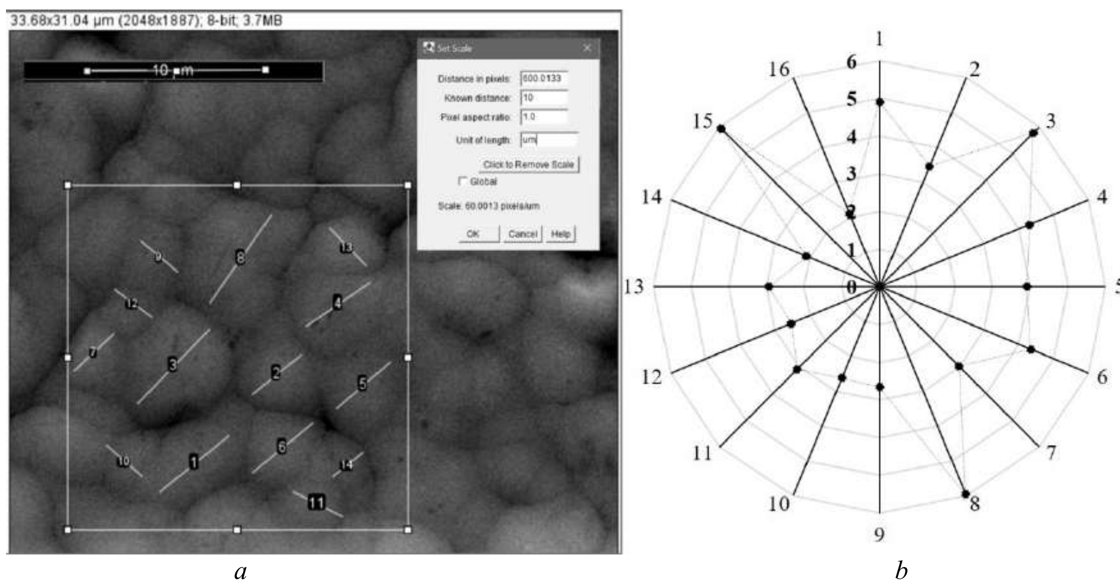

Fig. 2. Calibration of SEM-image of β-SiC/por-Si/mono-Si surface by means of ImageJ software package (*a*) and distribution diagram of linear sizes of islands (*b*)

EDX-analysis. Fig. 3 shows the EDX-mapping of formed structure surface. It can be seen that the β-SiC/por-Si/mono-Si surface contains only the Si and C atoms. No other atoms were detected, *i.e.*, the heterostructure was not oxidized, as well as the uncontrolled impurities were not observed. It can be also seen that the carbon is in much lower concentrations than the silicon. This may indicate a small thickness of the β-SiC film, which causes the reflexes to be appeared from the silicon substrate.

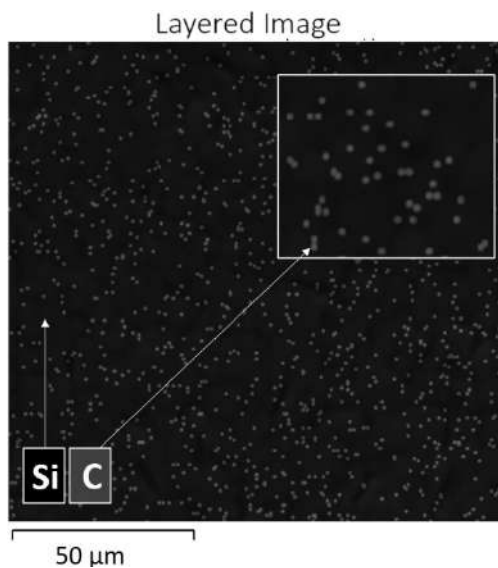

Fig. 3. Energy dispersive X-ray (EDX) mapping analysis of sample

It should be noted that the carbon is distributed evenly over the entire sample surface. This indicates the uniform carbidization of the silicon surface as well as the good quality of the formed structure.

XRD- analysis. The radiograph of the obtained heterostructure is shown in Fig. 4. The peak at 2ϑ = 28.5° corresponds to the reflection from the plane (111) of the elemental silicon [23]. A slight shift to the low-frequency spectrum part is due to the quantum-dimensional effects because of the presence of the porous layer. It can be also due to the curvature of the silicon lattice as a result of the elastic stresses to be appeared between the Si and SiC layers because of the parameter mismatch of the crystal lattices. The low intensity of the spectrum is due to the overgrowth of the porous silicon surface with the SiC film.

The peaks at the values of 2 ϑ 35.6° and 59.9° correspond to the reflection from (111) and (220) and the cubic β-SiC planes, respectively [24]. The absence of other intense peaks indicates the good crystallization of the obtained layers.

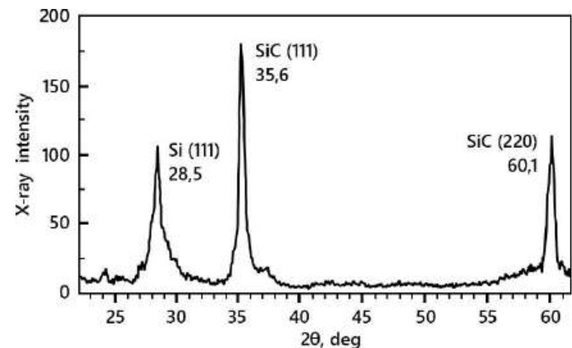

Fig. 4. XRD pattern of β-SiC/por-Si/mono-Si

DISCUSSION

As mentioned above, the parameters of the Si and SiC crystal lattices are significantly differed (Fig. 5). This usually significantly complicates the carbidization of the silicon surface. As a result, the uneven SiC layers, which are prone to amorphization and the appearance of a significant number of the structural defects, are formed [24]. We have demonstrated that the structurally and morphologically uniform SiC layer can be formed on the silicon surface due to the use of the buffer porous layer.

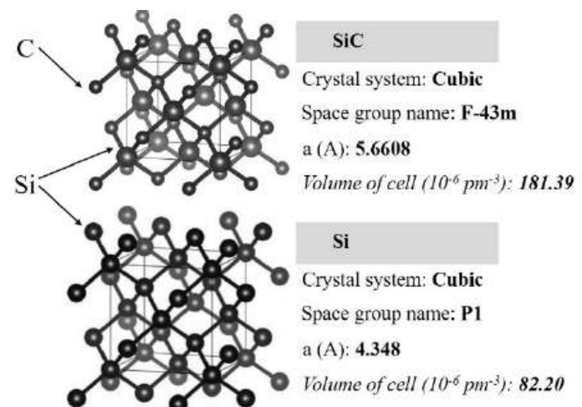

Fig. 5. Crystal lattices and structural parameters of SiC and Si

One may assume such growth mechanism of the SiC film on the por-Si/mono-Si surface. The carbon atoms, coming to the silicon surface, react with the substrate atoms. The reaction result is the formation of the Si-C compounds. The silicon atoms come into the boundary due to the diffusion. This creates openings in the near-surface layers of the substrate. On the other side, the silicon surface contains the porous layer. The

gaps between the pores are the protrusions, they are also silicon donors for forming the SiC layer. The pores (nanocavities) are, in turn, the places of origin of the silicon and carbon compounds. They reduce the deformation of the substrate and the grown SiC film. That is, the buffer pore layer of the silicon directly participates in the chemical carbidization reaction. At that the point defects – openings and the internodal carbon atoms are also formed in the growing SiC layer, which become effective channels for relaxing the elastic stresses in the Si-SiC system. This, in turn, explains the subsequent formation of the etching pits and pores on the SiC island surface.

Thus, the diffusion of the silicon atoms to the SiC-Si boundary and the evolution of the openings, caused by this diffusion, depend on the elastic deformation fields, the presence of nanovoids and the structure of the por-Si buffer layer. This opens up prospects for optimizing and improving the quality level of the SiC layers in the presence of buffer "soft" por-Si layers.

Also, in our opinion, an interesting explanation is the formation of the SiC layer, consisting of the densely packed islands. As you may know, there are three film growth mechanisms on the semiconductor substrates, namely [25] (Fig. 6):

- Frank – van der Merwe growth mechanism;
- Volmer – Weber island growth mechanism;
- Stranski – Krastanow layer-plus-island growth mechanism.

Frank – van der Merwe growth mechanism is observed in the systems with a very low mismatch of the crystal lattices between the deposited film and the substrate (Fig. 6 *a*). Under such conditions, the film (the so-called "wetting layer") increases due to the formation of single-atom degrees [26]. Volmer – Weber mechanism is typical for the systems with a high mismatch of the constant lattices (Fig. 6 *b*). It is characterized by the island growth without forming the wetting layer [27].

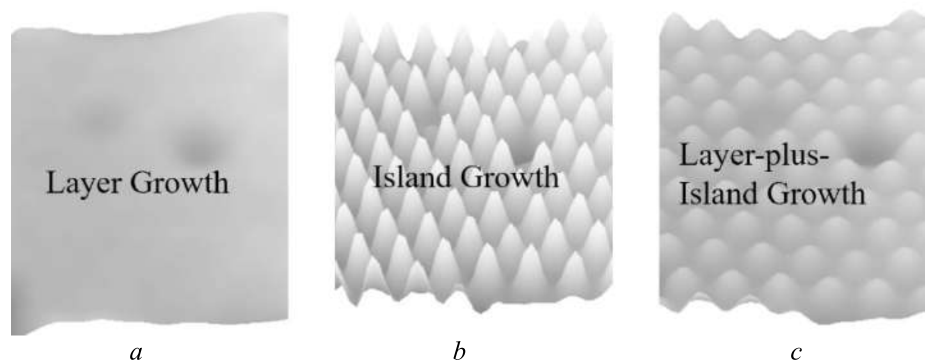

Fig. 6. Film growth mechanisms on semiconductor surface: *a* – Frank – van der Merwe, *b* – Volmer – Weber, *c* – Stranski – Krastanow growth mechanisms

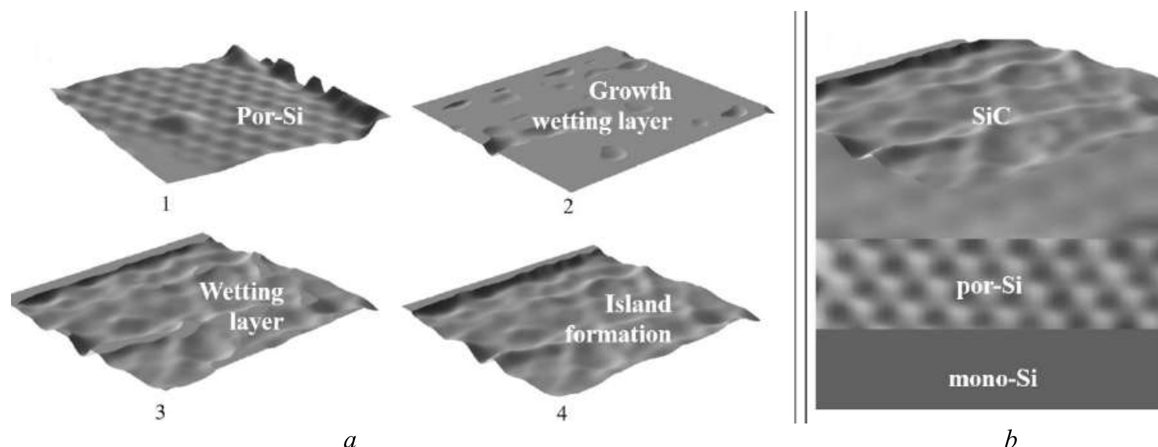

Fig. 7. SiC growth mechanism on por-Si surface (*a*) and model of β-SiC/por-Si/mono-Si heterostructure (*b*)

Stranski-Krastanow mechanism is the intermediate one (Fig. 6 *c*). According to this mechanism, the layer-by-layer growth is occurred at the initial deposition stages due to the gradual increase of the wetting layer [28]. The formation of the elastic deformations, which induces the film to compress, is observed due to the slight parameter mismatch of the crystal lattices. Due to this, the spherical islands are formed. Thus, a three-dimensional crystallite system is formed on the substrate coated with the wetting layer.

In our case, the SiC growth on the Si surface is characterized by the fact that the lattice mismatch is significantly minimized by the presence of the porous layer. Therefore, it becomes possible to form the heterostructure according to Stranski-Krastanow mechanism (Fig. 7 *a*).

On the one side, the formation of the islands reduces the system energy by reducing the bulk chemical component. However, along with this, the system energy can be increased by increasing the effective surface area. This induces the origin of the islands on the stressed layer, which leads to the relaxation of the energy system. Thus, the presence of the wetting layer with the island complex on the surface is observed. Fig. 7 *b* shows the model of the formed β-SiC/por-Si/mono-Si heterostructure.

Thus, the porous layer of the silicon serves as a reliable "soft" substrate for forming the β-SiC on the Si surface. It helps to relieve the elastic stresses that usually occur at the SiC/Si boundary as well as "smoothes" the non-conformities of the crystal lattices of these materials. It should be also noted that the porous SiC layers of island type can, in turn, show the perspective as the buffers with the heteroepitaxy of the silicon substrate materials.

CONCLUSIONS

In our paper the formation technology and mechanism of the β-SiC/por-Si/mono-Si heterostructure are demonstrated. According to the proposed method, the porous layer of the silicon was formed by the electrochemical etching on the single-crystal Si surface in the aqueous solution of hydrofluoric acid. The β-SiC layers were formed by the rapid thermal vacuum carbidization in the methane atmosphere.

As a result, the dense β-SiC layer, consisting of an array of the spherical islands with the diameters of 2–6 μm, coated with the small pores, was formed on the por-Si/mono-Si surface. According to the results of EDX analysis, it has been found that the synthesized structure surface consists exclusively of the carbon and silicon atoms, indicating the high quality of the formed structures.

The XRD-spectrum analysis allowed to reveal the presence of the Si (111) and the cubic phase of the β-SiC with the reflection from the planes (111) and (220). These results indicate the good crystallization of the heterostructure.

It has been found that the β-SiC islands are growing according to Stranski – Krastanow layer-plus-island growth mechanism, which is characterized by the formation of the dense wetting layer with the massive island complex on the surface.

It has been shown that the por-Si is a reliable buffer layer, which reduces the elastic stresses occurring at the SiC – Si boundary and allows to minimize the effect of the parameter mismatch of the crystal lattices on the silicon carbidization processes.

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Формування β-SiC на поверхні por-Si/mono-Si за механізмом Странського – Крастанова

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Ми повідомляємо про синтез гетероструктури β-SiC/por-Si/mono-Si гібридним методом, який включає електрохімічне травлення поверхні монокристалічного кремнію з наступною карбідізацією термічним відпалом у атмосфері метану. Такий метод має низку переваг над відомими, оскільки є достатньо дешевим та дозволяє формувати шари карбіду кремнію високої якості. Сформовану структуру було досліджено за допомогою SEM, EDX та XRD методів. В результаті на поверхні por-Si/mono-Si утворився щільний шар β-SiC, який складається з масиву сфероподібних острівців діаметрами 2–6 μm, вкритих дрібними порами. Геометричні розміри острівців було досліджено за допомогою калібрування зображення зразка в ImageJ. Встановлено, що у досліджуваній області максимальне значення лінійного розміру (діаметр) острівка d_{max} = 5.95 μm, мінімальне значення d_{min} = 2.11 μm. Загалом, острівки мають середній діаметр d = 3.72 μm. Розподіл має лівосторонню асиметрію, тобто переважають більш дрібні острівки. Округлість (відношення площі до квадрату більшої вісі) R = 0.86. За результатами EDX аналізу встановлено, що поверхня синтезованої структури складається виняткова з атомів вуглецю та кремнію, що свідчить про високу якість сформованих структур. Встановлено, що плівка SiC кристалізується в кубічній сингапії. Утворення острівків пояснено за допомогою layer-plus-island моделі росту за механізмом Странського – Крастанова, який характеризується утворенням щільного змочувального шару з ансамблем масивних острівців на поверхні. Показано, що por-Si є надійним буферним шаром, який зменшує пружні напруження, що виникають на межі розділу SiC–Si, і дозволяє мінімізувати вплив невідповідності параметрів кристалічних ґраток на процеси карбідізації кремнію. Необхідно також зазначити, що поруваті шари SiC острівкового типу можуть, в свою чергу, показати перспективність як буфери при гетероепітаксії матеріалів на кремнієві підкладки.

***Ключові слова**: карбід кремнію, механізм Странського – Крастанова, layer-plus-island модель росту, електрохімічне травлення, термічний відпал, поруваті шари, кристалічна ґратка*

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