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Preparation and Characterization of Carbon-Silicon Hybrid Nanostructures

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In this paper, we describe the design features of carbon-silicon (Si@C) hybrid nanostructures. Initial silicon nanoparticles (Si NPs) were prepared by acid etching from powdered (80 % Al – 20 % Si) silumin alloy. For preparing Si@C hybrid nanostructures, the use of standard thermal and chemical methods was proposed. The recommended approach involves treatment of the oxidized surface of Si NPs with polyvinylpyrrolidone as a carbon-containing precursor compound. The resulting product should be subjected to further carbonization. To prevent significant sintering of the components, Si NPs treated with polyvinylpyrrolidone were placed in a fluidized bed furnace and calcined at 400 and 500 °C in an argon atmosphere for 4 h. According to the data of atomic force microscopy, scanning electron microscopy, transmission electron microscopy (TEM), and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) imaging, we obtained the Si@C interface and the corresponding Si/C heterostructures. By using TEM and HAADF-STEM imaging, we found that 2D carbon nanostructures are formed on the structured nanosilicon surface as on the substrate. According to the visualization of the modified interface, carbon NPs are amorphous and semi-amorphous nanostructures. Carbon NPs, the Si@C interface, and the corresponding Si/C heterostructures of Si@C hybrids can be prospective as probes for the development of new sensor devices. Sensing responses of Si@C hybrid-based sensors to ammonia, methanol and ethanol vapors were measured at room temperature and compared. Notably, hybrid-based sensors are highly sensitive with a selectivity of up to 10 ppm of ammonia, contrasting to the response to 1,000 ppm of methanol and ethanol vapors. The proposed hybrids have great potential to be used in adsorption semiconductor gas sensors to determine analytes in vapors using nanocarbon bonded to silicon in hybrid nanostructures as a probe.

Keywords: Nanostructures, Silicon and Carbon, Hybrids.

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1. INTRODUCTION

Nanosilicon and silicon nanoparticles (Si NPs) [1-4] are the objects of increased scientific interest to be used as new photonic and electronic materials [1, 5], for example, in solar cells [2, 3], including the formation of semiconductor-insulator-semiconductor heterojunction solar cells [4]. Some valuable studies were performed to measure photoluminescence induced by ultraviolet irradiation and electroluminescence emission of Si NPs [5-7]. Indeed, one can use these phenomena to be integrated into photoluminescence/electro-luminescence devices [6, 7].

These and similar properties of nanosilicon open up broad prospects for its application in modern electronics [8-11]. Besides, the same nanomaterials can be integrated into light-emitting diodes, thermal detectors, and light waveguides. They can be used for creating cascade systems, and subsequent signal transmission to devices for reading and processing information is assumed and widely used [10, 14]. The latter is realized by combining emitters with devices for processing their electrical and optical signals, e.g., photocurrent, when creating integrated microcircuits. Silicon nanopowders and membranes based on them [13 16] are promising working elements for a wide range of chemical, biological, and gas sensors, electrochemical devices.

The operation of these types of sensors is based on the registration of changes in the conductive and photoluminescent properties recorded in the presence of analyte molecules in the controlled environment [15-20]. Recently, yolk-shell-structured Si@C nanocomposites and core-shell yolk-shell Si@C@void@C nanohybrids have been prepared to be used as advanced anode materials for lithium-ion batteries [9]. In general, Si NPs are promising materials in various applications in the fields of energy storage and sensors [15-17].

At present, modern materials scientists mainly deal with laser ablation and plasma-chemical synthesis. Much less attention is paid to obtaining porous silicon or silicon nanowires using anodic electrochemical etching [1, 11, 14].

The existing methods for creating effective nanomaterials based on silicon-carbon hybrids have drawbacks [18-20]. They are different for diverse methods. For example, it is necessary to use complex equipment within the framework of ablation and plasma-chemistry routines. The operation of such equipment is often characterized by a low productivity of nanopowders, which leads to a high cost of the final nanohybrids.

As a prominent alternative to hybrids prepared using known ablation and plasma-chemistry routines, we report the results of studying hybrids based on Si NPs

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obtained by etching technique and modified with carbon. Here, we present the results of microscopic studies of carbon surface layers on silicon NPs. This structural information is of interest to researchers working on the creation of silicon-based hybrids with special properties. The prepared Si@C hybrids were examined as sensing materials to measure ammonia gas in the presence of moister and alcohol vapors.

2. EXPERIMENTAL

2.1 Preparation

Silicon nanopowder was prepared by acid etching from powdered (80 % Al – 20 % Si) silumin alloy, which is considered as a starting material in the proposed line of chemical and thermal methods for obtaining hybrids. In a typical preparation route, the micropowder of silumin alloy was dropped into a 15 % hydrochloric acid solution for 8 h with continuous stirring until aluminum was completely leached into the solution. Then, the resulting solid residue was filtered and washed with deionized water. Finally, after being washed several times using ion-free water, the resulting silicon nanopowder was dried at 120 °C. The decoration of Si NPs with carbon nanostructures was carried out within the framework of the chemical method of surface modification. This chemical method aimed to create the Si@C interface and the corresponding Si/C heterostructures. The recommended approach, in particular, involves the reaction of the oxidized surface of Si NPs and carbon-containing precursor compounds subjected to further carbonization. This technique makes it possible to achieve a controlled (by carbonization temperature) decoration of Si NPs with carbon nanostructures.

In the framework of the considered approach, for preparing Si@C hybrids, polycrystalline Si particles weighing 100 mg were treated with polyvinylpyrrolidone solution (1 g) in absolute ethanol (100 ml) under mechanical stirring with additional sonication. The excess solvent was removed on a rotary evaporator at 35 °C; the prepared powder was dried at 75 °C for 12 h in an Abderhalden drying pistol. To obtain nanopowders without significant sintering of the components, the considered samples were placed in a fluidized bed furnace and calcined at 400 and 500 °C in an argon atmosphere for 4 h.

2.2 Characterization

The prepared Si@C hybrids were visualized by atomic force microscopy (AFM). Samples for AFM studies were prepared by drying drops of a colloidal solution on pure hydrophilic silicon substrates. The registration was performed with a Nanoscope III (Digital Instruments) in non-contact mode with Si cantilevers. Powder X-ray diffraction (PXRD) studies were performed on an automatic DRON-3M diffractometer (CuKα radiation, $\lambda = 0.15418$ nm). SEM observations were performed on a Tescan Mira 3 GMU LV-FE-SEM scanning electron microscope at an accelerating voltage of 15 kV. Element mapping was carried out with an energy dispersive X-ray (EDX) spectrometer from Oxford Instruments. To study the nanohybrids, we also used a high-resolution JEOL JEM-3000F TEM microscope with an accelerating voltage of 300 keV [21]. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) imagining was done with a Jeol ARM200F (200 keV) FEG-TEM/STEM instrument.

To characterize Si@C hybrids, their gas sensing parameters were evaluated in a homemade characterization system operating by measuring the resistance of the sensor [22]. Before testing, the corresponding nanopowder was deposited on the electrodes. Dry argon gas was bubbled through flasks containing double distilled water and ammonia solution to generate ammonia vapor and humidity. The produced moisture and ammonia vapor flowed into the gas-sensing chamber with argon as carrier gas. To get the desired concentration, we regulated the gas flow through the valves and mass flow controllers, which was introduced into the evacuated test chamber. 1,000 ppm of methanol and ethanol (CH_3OH and C_2H_5OH) vapors were used to measure response selectivity comparing with ammonia (NH_3) vapor. Using a sampling interval of 1 s, we collected data on resistance changes and accounting for relative humidity (RH). RH was measured with an RH sensor installed in the chamber.

3. RESULTS AND DISCUSSION

Because of the use of polyvinylpyrrolidone surfactant, large micelles were formed from pseudospherical Si NPs ($d \leq 50$ nm). The resulting colloids were centrifuged at 5000 rpm, and the resulting sediments were dried at 120 °C for 12 h. Further, the claimed carbonization of the precursor in an argon atmosphere was carried out at a temperature of 400 °C. This heating led to thermal decomposition of the ethoxylate shell, which allows, through heat treatment, to form a body of Si@C nanohybrids. It is clear that this technique makes it possible to obtain relatively inexpensive nanopowders of Si@C hybrids with a particle size of 5 to 100 nm.

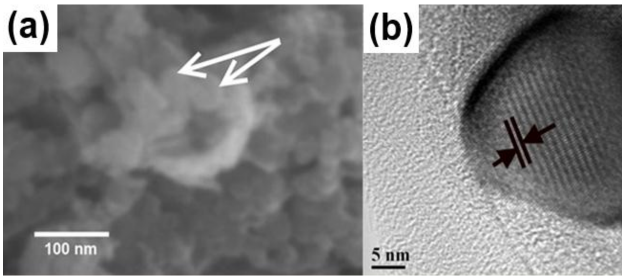

Fig. 1 – SEM (a) and TEM (b) micrographs of Si@C hybrids

Fig. 1 shows typical SEM and TEM images for such particles. From the SEM data, we observed the formation of 100 nm aggregates (Fig. 1a). Dispersion of the latter during sample preparation shows that they are composed of smaller NPs with an average size from 20 to 50 nm. The morphological analysis of Si NPs covered with a layer of unstructured carbon, the latter forms stable layers on the surface of a crystalline nanosized grain, showed that the thickness of this carbon layer ranges from 0.5 to 2 nm. It is typical for this type of NPs that the carbon layer on silicon retains the crystallinity and integrity of the original silicon nanograins, which is consistent with the TEM data (Fig. 1b). For structured Si NPs, the interplanar distances found in

TEM micrographs (Fig. 1b) are 0.31 nm; they can be attributed to the Si (111) plane. In Fig. 1b, nanocarbon layers are shown. However, they are characterized by a low degree of crystallinity. Therefore, 3D structuring of thin nanocarbon layers at the Si/m-C interface is probably impossible. According to the TEM data, the formation of a 2D silicon-carbon interface occurs for the obtained pseudo-spherical nanoparticles (20 nm in diameter). EDX data show the formation of a particle of the core-shell type. In them, the silicon core is covered by a multilayer carbon shell. According to microscopic data, it is impossible to assert that the formation of multilayers based on a 2D carbon nanostructure is realized. These NP are likely to have a more complex structure.

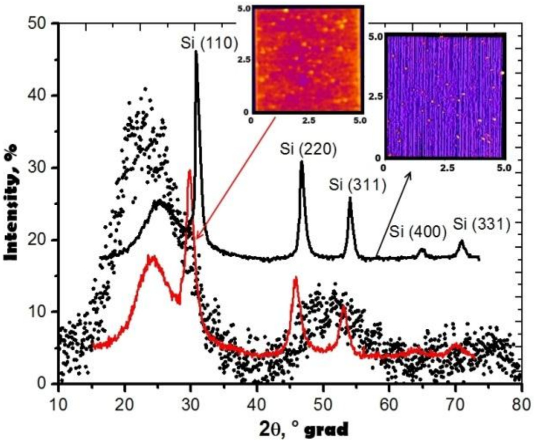

Fig. 2 – PXRD patterns: Si NPs used as precursor (black line) and Si@C hybrids (red line); points – before carbonization at 400 and 500 °C. Inserts are AFM images of Si NPs and Si@C hybrids. Scale in micrometers

Fig. 2 shows PXRD patterns of agglomerated nanohybrids. They are characterized by a halo in the range of 2θ angles of 15-30°. Here diffuse reflections from the amorphous component are observed, with an assumed maximum at 25°. Such a wide peak in PXRD patterns can be attributed to amorphous carbon in the annealed agglomerates. For all patterns, the same features are observed. They indicate the presence of both structured and unstructured components in nanohybrids. After carbonization, we observed narrow reflections of silicon crystallites. PXRD patterns are characterized by peaks at 28-30°, 46-48° and 53-55°, which correspond to crystallographic reflections from planes (110), (220), (311), (400), and (311) for the elementary silicon lattice.

Detailed information on the size distribution of nanoparticles in nanohybrids was obtained by analyzing TEM and AFM images (typical images are shown in Fig. 2, Fig. 3). The particle size distribution (see AFM in Fig. 2, obtained with about 100 particles for ensemble images from different parts of the coordinate grid) can be quite well represented as such, which is formed by NPs with an average diameter of 20 to 40 nm, the standard deviation of this estimate is about 1 nm.

Broadening of diffraction maxima (according to the PXRD data) indicates the ultradispersed state of hybrids. The TEM micrograph of agglomerated hybrids shows the formation of 2D carbon nanostructures (Fig. 4). The formation of surface structures requires the observance of some conditions that make it possible either to select the experimental parameters, under which 2D nanostructures become thermodynamically advantageous, or, as implemented in the present study, to limit the spatial growth of NPs, while ensuring a high locality of the structure-forming precursor.

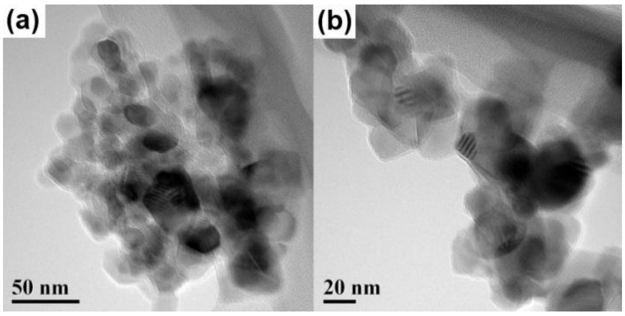

Fig. 3 – TEM micrographs of agglomerated Si@C hybrids

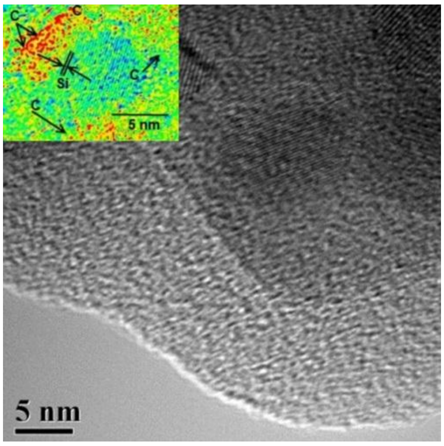

Fig. 4 – TEM and HAADF-STEM (insert) micrographs of Si@C hybrid prepared at 400 °C

To avoid uncontrolled phase formation at 400 °C, the implementation of fast pyrolysis by rapid heating to 500 °C allows one to regulate the formation of surface structures, during which small clusters grow in size with subsequent growth of large 2D nanostructures, mainly as ellipsoids (Fig. 5). The insert in Fig. 5 shows interplanar silicon distances in the structure-forming nanostructures in the region of formation of a carbon spacer. The HAADF-STEM micrograph shows the adhesion of two Si NPs by the formation of a silicon-carbon-silicon interface. As seen from Fig. 5, the surface of silicon nanostructures is decorated with carbon NPs, which are indicated by arrows in the TEM micrograph. According to the results, these carbon particles are nanostructures with a size of about 5 to 7 nm.

Microscopic data (Fig. 5) show that these carbon NPs are semi-amorphous; the thickness of their layer does not exceed 1 nm and, probably, performs an auxiliary function, regulating the size of NPs and preventing their aggregation. A surface interface of this type performs a passivation function, preventing deep amorphization during oxidation of carbon NPs [23], as a result of interaction with the environment during the operation of devices, and plays the role of a spacer between carbon (amorphous) NPs and structured Si NPs, forming the body and structure of nanohybrids.

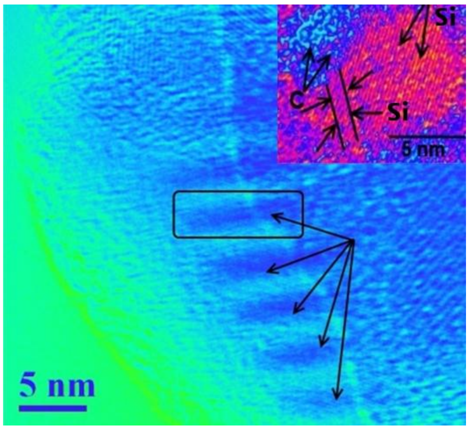

Fig. 5 – TEM and HAADF-STEM (insert) micrographs of Si@C hybrid prepared at 500 °C

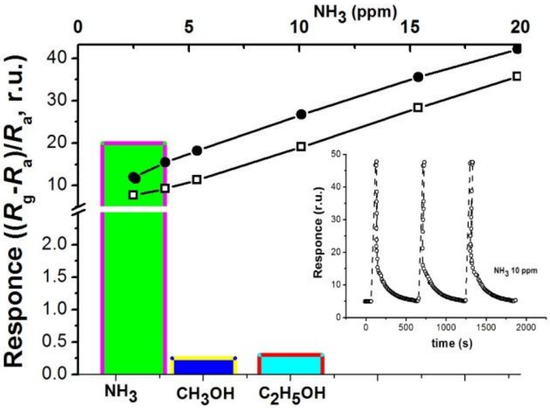

Fig. 6 – Response to NH_3 (insert), comparison of responses at 2.5 to 20 ppm of NH_3 vapor, and response of a sensor based on Si@C hybrid prepared at 500 °C to alcohols versus ammonia vapors (bar chart)

The large curvature of the surface of Si@C hybrids could change the topology of atomic bonds on the surface. This rearrangement of bonds will lead to a change in their chemical potentials, as a result of which the reactivity and catalytic ability of hybrids can increase significantly. Upon exposure to NH_3, the direct interaction between 2.5 to 20 ppm of NH_3 vapor and the surface of Si@C hybrids prepared at 400 and 500 °C leads to a change in resistance of both studied sensors, see white and black data points, respectively, in Fig. 6.

As a final point, we evaluate the selectivity of Si@C hybrid-based sensors. The sensing responses of Si@C hybrids prepared at 400 and 500 °C to exposure to methanol and ethanol vapors at 1,000 ppm were measured at room temperatures and compared with those at 1 to 10 ppm NH_3. Both Si@C hybrid based sensors are more sensitive to exposure to NH_3 at 10 ppm than to alcohol vapors at 1,000 ppm. The ratio of the selectivity coefficients reaches the values of 20/0.25 and 20/0.3, as the ratio of the response signal upon exposure to NH_3 versus that to methanol and ethanol vapors, respectively, for a sensor based on the Si@C hybrid prepared at 500 °C (see bar chart in Fig. 6). We found an increase in the baseline resistance of both sensors with RH, and as found, the 45 % RH degrades the sensing response. Moreover, Si@C hybrid based sensors show a linear response between 1 and 20 ppm NH_3 ranged from 5 to 43 r.u. and have excellent selectivity towards NH_3.

4. CONCLUSIONS

Thus, within the framework of this work, we showed the possibility of preparing Si@C hybrids based on structured silicon with a particle size of 7-20 nm by chemical and thermal methods using silicon NPs prepared by the etching method. These hybrids were prepared from polyvinylpyrrolidone precursor and formed by crystalline pseudo-spherical Si NPs decorated with carbon NPs. The latter have a two-dimensional structure and are roentgen amorphous or semi-amorphous. Using the proposed method, we achieved the desired result by decorating Si NPs with small carbon NPs. This result attracts attention, and the developed approach is of direct interest for further optimization and approbation in the functionalization of silicon nanowires as probes. The structural features of Si@C hybrids and the represented unique character of the formation of carbon NPs at the interface and edge dislocations of nanosilicon are unique. The proposed method has great potential for creating sensitive Si@C hybrids for adsorption semiconductor gas sensors. In particular, one can adapt these hybrids in devices, the principle of operation of which involves the use of various detection methods of adsorption response to analytes in vapors through the use of nanocarbon bonded with silicon in hybrid nanostructures as a probe. Our data revealed that both prepared Si@C hybrids, as sensing materials, show high selectivity towards NH_3 and can be used in alcohol/ammonia mixed vapors.

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Приготування та характеризація вуглецево-кремнієвих гібридних наноструктур

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У статті ми описуємо особливості будови гібридних наноструктур вуглець-кремній (Si@C). Вихідні кремнієві наночастинки (Si НЧ) були отримані кислотним травленням із порошкоподібного силумінового (80 % Al – 20 % Si) сплаву. Для приготування гібридних наноструктур Si@C було запропоновано використання стандартних термічних та хімічних методів. Рекомендований підхід передбачає обробку окисленої поверхні Si НЧ полівінілпіролідоном, як сполукою-попередником, що містить вуглець; отриманий продукт слід піддавати подальшій карбонізації. Для запобігання значного спікання компонентів, Si НЧ, оброблені полівінілпіролідоном, поміщали в піч з киплячим шаром і прожарювали при 400 та 500 °C в атмосфері аргону протягом 4 годин. За даними атомно-силової мікроскопії, скануючої електронної мікроскопії, трансмісійної електронної мікроскопії (ТЕМ) та скануючої трансмісійної електронної мікроскопії з висококутовою кільцевою діаграмою темного поля (СТЕМ-ВКДТП), ми отримали інтерфейс Si@C та відповідні Si/C гетероструктури. Використовуючи зображення ТЕМ та СТЕМ-ВКДТП, ми виявили, що 2D вуглецеві наноструктури утворюються на структурованій поверхні нанокремнію, як на підкладці. Згідно візуалізації модифікованого інтерфейсу, вуглецеві НЧ є аморфними та напіваморфними наноструктурами. Вуглецеві НЧ, інтерфейс Si@C та відповідні Si/C гетероструктури гібридів Si@C можуть бути перспективними як зонди для розробки нових сенсорних пристроїв. Сенсорні відгуки датчиків на гібридній основі Si@C на пари аміаку, метанолу та етанолу вимірювали при кімнатній температурі та порівнювали. Слід зазначити, що датчики на гібридній основі мають високу чутливість та вибірковість до 10 мд. аміаку, на відміну від реакції на 1000 мд. парів метанолу та етанолу. Запропоновані гібриди мають великий потенціал щодо використання в адсорбційних напівпровідникових газових датчиках для визначення аналітів у парах з використанням нановуглецю, зв'язаного з кремнієм, у гібридних наноструктурах в якості зонду.

Ключові слова: Наноструктури, Кремній та вуглець, Гібриди.