

## COLD FUSION OF SILICON CARBIDE IN SiO<sub>2</sub>-CNT COLLOIDAL NANOSYSTEM

© 2014 Dmitry Zhukalin<sup>1</sup>, Andrey Tuchin<sup>1</sup>, Dmitry Goloshchapov<sup>1</sup>, Larisa Bituytskaya<sup>1</sup>, Frank Roessner<sup>2</sup>

<sup>1</sup>Voronezh State University, Universitetskaya pl., 1, 394006 Voronezh, Russia

<sup>2</sup>Carl von Ossietzky University of Oldenburg, Ammerlander Heerstrasse 114—118, D-26129 Oldenburg, Germany  
e-mail: d.zhukalin@mail.ru, frank.roessner@uni-oldenburg.de

Received 01.10.2014

**Abstract.** Mechanisms and conditions of the formation of self-organized structures in colloid nano-system aerosol — carbon nanotubes (SiO<sub>2</sub>-CNT) were studied using numerical and natural experiments. Within the framework of cap/body CNT model we identified two mechanism of interaction of CNT with SiO<sub>2</sub>: covalent and Van-der-Waals. Covalent interactions resulted in the formation of self-organized stem structures with diameter 250—300 nm and length ~4 μm; van-der-Waals interactions resulted in the formation of spherical aggregates with the diameter ~2 μm. X-ray diffraction analysis of the obtained nanostructures revealed formation of the silicon carbide phase at room temperature.

**Keywords:** nanosystem, carbon nanotubes, silicon dioxide, electronic structure, density functional theory, charge, interface, nanocomposite, silicon carbide.

### INTRODUCTION

Electrostatic interaction plays a special role in the technology of synthesis of nanomaterials. David Walker in his review (Walker D. A., et al.) [1] demonstrated the universal character of influence of electrostatic forces on aggregation and self-assembly of charged nanoparticles with the formation of nano-objects of variable geometry. In the opinion of the authors, despite the considerable advances in research of electrostatic interactions, the area of nano-scale interactions remains poorly understood. This is especially true in the case of CNT-based nanocomposites.

Pure CNTs represent polyfunctional quantum-sized material with an array of unique physical properties [2—4]. As a rule, carbon nanotubes are considered to be electroneutral and chemically inert. The advances in technology of production of nanotubes with controlled parameters (symmetry, diameter, length) allowed to proceed to the new phase of practical applications of CNTs, such as the development of nanocomposites with defined properties. The examples of such applications include: radiation-resistant nanocomposites for use in extreme conditions (space, atomic energy) [5]; nanoelectronics [6, 7]; sensors; synthesis of hybrid materials [8, 9], etc. The unique feature of these technologies is the emphasis on localization of inter-

actions of various components with CNTs on nanoscale, based on characteristic properties: sizability; symmetry; curvature; charge states, etc. There is an ever-increasing role for quantum-chemical calculations, theoretical evaluations, and quantitative experiments [5]. This approach led to the appearance of new methods of synthesis of hybrid materials, for example size-dependent soft epitaxy of polymer chains on CNT hexagons. The synthesis of polymers (polypropylene, polyethylene, nylon, etc) takes place on the surface of single- and multi-walled CNT with diameter 0.8—1.3 nm and 5—30 nm, with the formation of hybrid structure of the new type «shish-kebab» [11, 12]. In contrast to the tubes with small diameter, the crystallization of polymer on the surface of tubes with diameter greater than 100 nm is more difficult [10].

There is great interest to the local interactions of CNTs with oxygen-containing phases. The study of the influence of atomic oxygen and hydrogen on structural characteristics of composite materials polymer-CNT (at energies up to 200 eV) using the Density Function Theory (DFT) method, there was found to be the effect of lengthening and transposition of inter-atomic bonds leading to general deformation of nanostructures, even to the point of unzipping of nanotubes [5]. In one work [13], during high-temperature annealing of composite SnO<sub>2-x</sub>/multi-walled

CNT in vacuum, there was transformation of its electronic structure along with local synthesis of tin oxycarbide.

In addition to external high-energy CNT activation methods, it is important to consider the internal reserve of the nanotubes themselves related to their size qualities, surface curvature and defects. Some works [14, 15] noted the diminishing oscillations of fundamental parameters, such as ionization potential (IP), electron affinity and band gap [16—18]. Instability of the fundamental parameters by the size of chair nanotubes is related to the peculiarity of redistribution of electron density of frontier orbitals [15].

In the previous work [8] of creating the composite from the colloid solutions of clinoptilolite and CNT at normal conditions, we identified qualitative change in morphology of the original structures. Using the method of IR-spectroscopy, we found a nonlinear dependency of the intensity of oscillations of T-O bonds, based on concentration of CNTs. In order to interpret this experimental data, we suggested the model of localized charge states in the interface cap/body CNT. We define the cap/body interface of CNT as a quantum-sized transitional layer brought about by the difference in the size of carbon atom rings and the length of bonds, creating a strong local electrical field (Fig. 1) [21].

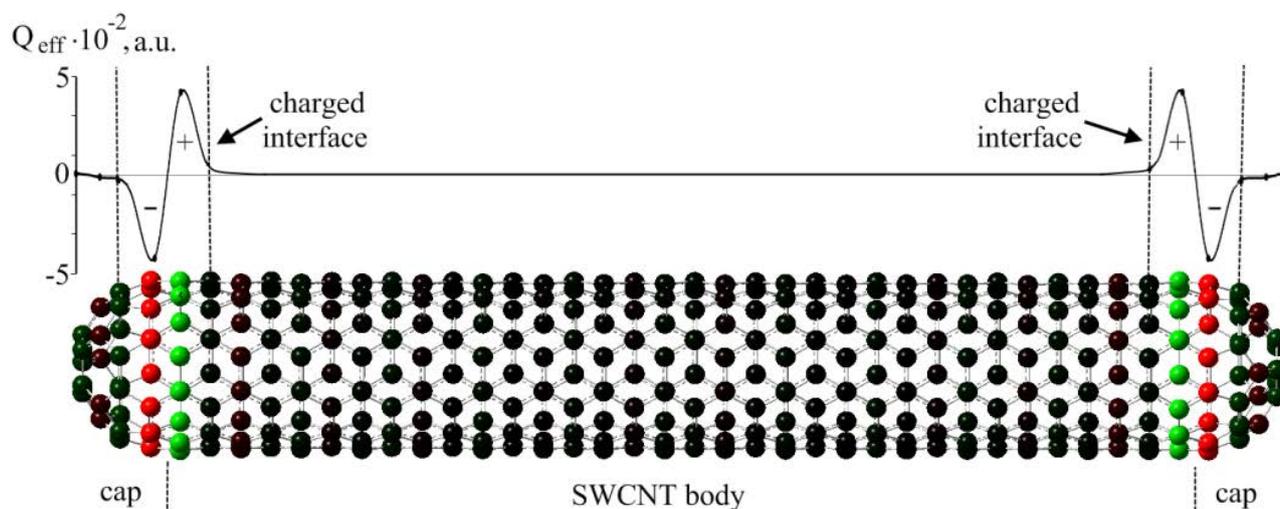


Fig. 1. The charged interface cap/body of CNT (5, 5)

Considering our finding of strong influence of short CNTs' interaction with zeolite on the intensity of T-O bond oscillations, in the present work we investigated the mechanisms and conditions of aggregation and synthesis of self-organized structures in model nanosystem SiO<sub>2</sub>-CNT using the methods of numerical and natural experiments.

### NUMERICAL CALCULATIONS

We conducted simulation of local interaction between nanofragment SiO<sub>2</sub> and carbon nanotube in the area of charged interface. Closed CNT without defects (5, 5), having length 0.6 to 6.1 nm was chosen as the model nanotube. Calculations were done using the DFT method in local spin density approximation LSDA. For calculating of charge properties we used exchange-correlation functional B3LYP. Calculations were performed using the program package Gaussian09 in the Supercomputer Center of Voronezh State University.

The caps of the model CNT (5, 5) represent fullerene C<sub>60</sub> bisected along the perpendicular axis C<sub>5</sub>. The presence of pentagons on the tube's cap results to redistribution of electron density in such a way that the frontier ring of carbon atoms has effective negative charge Q<sub>eff</sub> (cap), and the body's ring — positive charge Q<sub>eff</sub> (body) [9, 22, 23]. The charges converging quickly to the values Q<sub>eff</sub> (cap) = -0.041 ÷ -0.037 a. u. and Q<sub>eff</sub> (body) = 0.035 ÷ 0.038 a. u. for CNT longer than 1.1 nm (a. u. — atomic units) [21]. Frontier sides of the cap's pentagons and body's hexagons are oriented perpendicular to the tube's axis, which determines the localization of the charge within the interface, the width of which is ≈ 3.7 Å. Therefore, the charges of neighboring orbitals in the cap/CNT region create localized (≈ 3.7 Å) strong (~10<sup>8</sup> V/m) short-range electrical field at interface.

Test numerical experiments were conducted on functionalization of frontier positively and negatively charged rings of carbon atoms interface with the atoms of different electronegativity: hydrogen and fluorine.

The results of potential curves calculations are presented in Fig. 2. The energies of covalent bonds of hydrogen and fluoride with the atoms of carbon from negatively charged ring of the cap were 3 and 1.5 times

higher than with positively charged ring of the tube's body. Therefore, negatively charged ring of the cap can be viewed as local activity center with increased reaction capability.

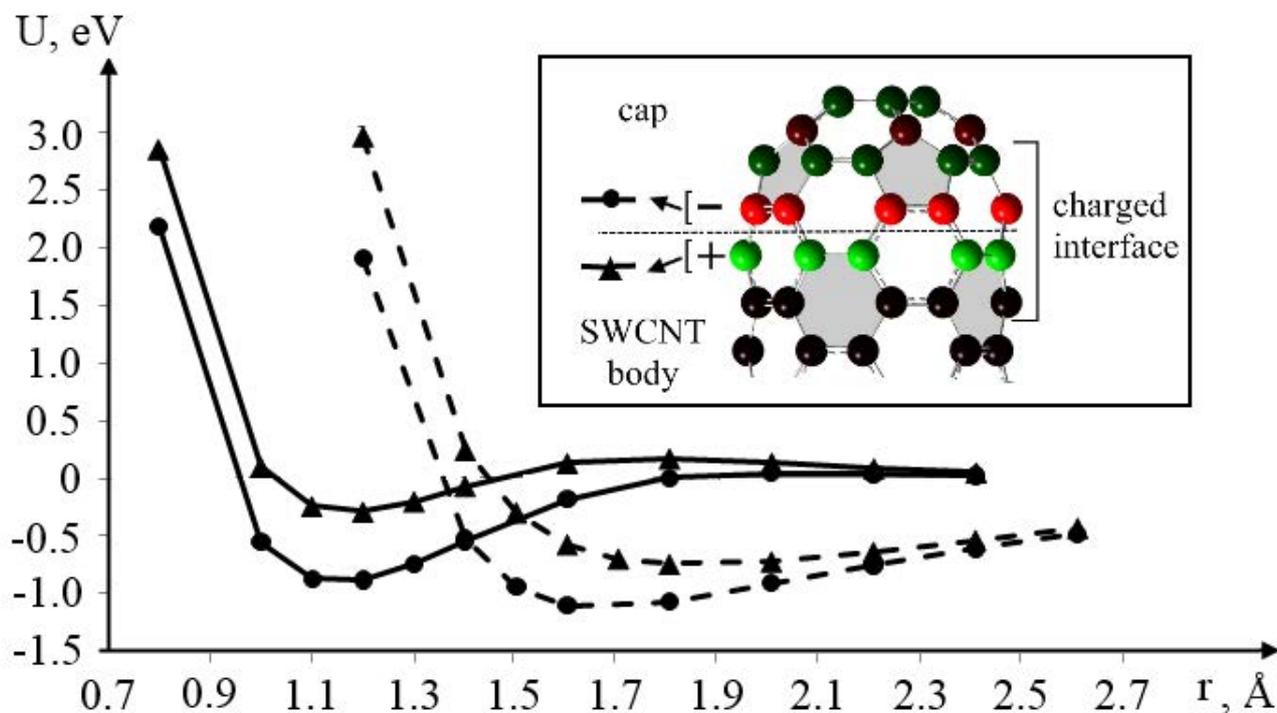


Fig. 2. Potential curves of interaction of hydrogen atoms (solid lines) and fluorine (punctate lines) with negatively-charged ring of carbon atoms (cap) and positively charged ring of SWCNT's body (5, 5). Full energy of non-interacting nanotube and hydrogen was set as zero point

Let's look at the interaction between the nano-size fragment of SiO<sub>2</sub> with CNT in the area of charged interface. Numerical experiments demonstrated the presence of the initial critical distance between the atoms of carbon and silicon  $r_{\text{Si-C}}^{\text{cr}} \approx 1.94 \text{ \AA}$ , within which there is a formation of covalent bonds Si-C and O-C, as well as bridges C-Si-O-C. If the initial distance is greater than  $r_{\text{Si-C}}^{\text{cr}}$ , then the calculated value is  $r_{\text{Si-C}} \sim 2.54 \text{ \AA}$ , which is characteristic of Van-der-Waals interactions. The priority of formation of Si-C bonds depending on carbon atom's position is as follows: 1. carbon atoms of the negatively charged ring of tube's cap; 2. carbon atoms of cap's pentagons; 3. carbon atoms of tube's body. Therefore, with adequate approximation of nanosized SiO<sub>2</sub> with the charged active center of the nanotube there is local synthesis of the new oxycarbide phase, which can serve as inciting event for further structural reorganization between the tube and the surrounding nanocomponent.

In real experiments, colloid CNT solutions contain a mass assortment of nanotubes of various lengths,

symmetry, level of defects, and represent electroactive environments. The same mass quantity of long and short tubes have a different number of active centers. It is known that the aggregation of long tubes ( $>10 \text{ \mu m}$ ) with formation self-organized structures is difficult [23]. Short tubes aggregate according to fractal type without the synthesis of new phases with large ( $\sim 0.2 \text{ \mu m}$ ) particles of SiO<sub>2</sub> [9]. On macro level, the sensitivity of aggregation process to the length of CNT is proven by measuring the surface tension  $\sigma$  using the hanging drop method on OCA 15EC instrument (DataPhysics, Germany). For the short tubes ( $\sim 0.5 \text{ \mu m}$ ) used in the experiment the value was  $\sigma = 71.91 \text{ mN/m}$ , while for the long tubes ( $\sim 3 \text{ \mu m}$ ) it was  $\sigma = 72.36 \text{ mN/m}$ .

#### METHODOLOGY OF EXPERIMENT

An important condition for realization of internal reserves of short nanotubes and the synthesis of new phases is to ensure the sizability of CNTs with the interacting component. To achieve this goal, in our experiment we used amorphous SiO<sub>2</sub> of AEROSIL

brand 300 particles  $7\ \mu\text{m}$  and a «mass» of short CNTs ( $\sim 0.5\ \mu\text{m}$ ) with average diameter 20—40 nm, obtained through arc discharge method.

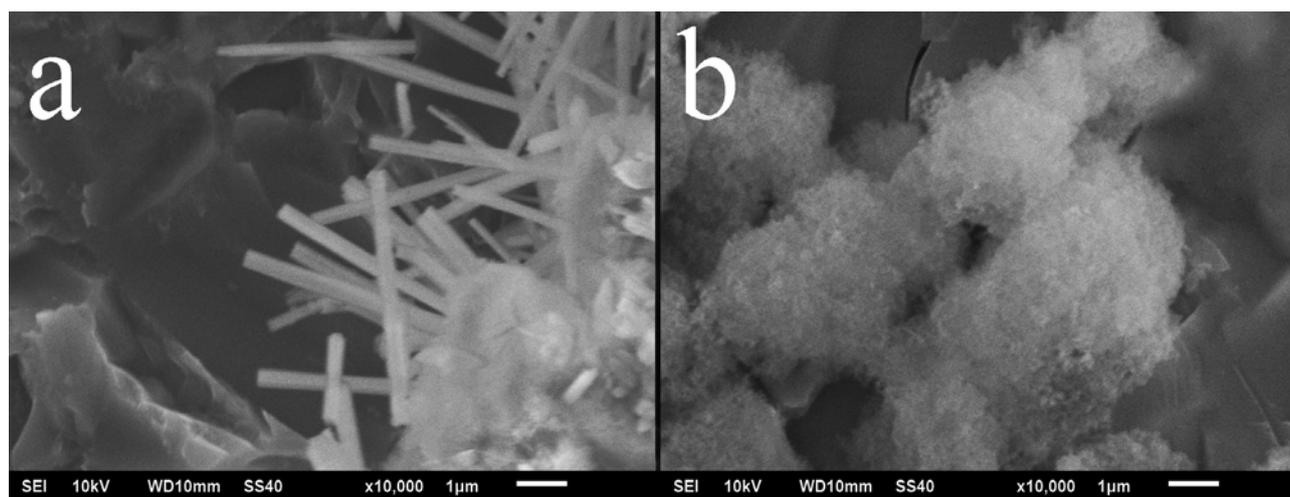
The investigation of interaction of aerosil with carbon nanotubes was conducted in homogenous environment by mixing diluted colloid solutions of  $\text{SiO}_2$  (10 g/l) and CNT (0.1 g/l) using the drop method [19, 20], with subsequent evaporation of dispersion medium — water. A drying drop is viewed as nanoreactor [20] with dynamic walls. In order to obtain a powdered composite for investigation by X-ray diffraction and IR spectroscopy, samples were evaporated from small volumes of colloidal suspensions.

Colloidal suspensions of initial components was dispersed prior to the experiment using the ultrasound disperser for 15 minutes at 60 Vt. The size of particles in diluted colloidal suspensions was controlled using the method of dynamic light scattering (DLS) on Nicomp 380 ZLS analyzer (USA). The morphology of obtained structures was investigated on scanning electron microscope JSM-6390LV (JEOL, Japan). Elemental analysis was conducted on analytical console Bruker AXS Microanalysis GmbH (Germany). Investigation of powder-like samples of pure aerosil and aggregates  $\text{SiO}_2/\text{CNT}$  was controlled on IR-spectrometer VERTEX-70 (Germany) by method of interrupted full internal reflection in the range from 600—4000  $\text{cm}^{-1}$ . Transmission electron microscopy (TEM) performed on Carl Zeiss Libra 120.

Diffraction patterns of the samples were obtained on an automatic diffractometer PANalytical EMPYREAN with the use  $\text{Cu K}\alpha_1$  (hybrid Ge {111} monochromator on the primary beam) and a position-sensitive detector PIXcel<sup>1D</sup>. Measurements were made in reflection mode,  $\theta/2\theta$  scan with a step 0.013 ° on  $2\theta$ . Measuring range 5—60 °  $2\theta$ . The calculation of the interplanar distances and the integrated intensities used to refine the cell parameters, conducted according to the profile analysis of experimental diffraction patterns (method Pauli). Indexing of the diffraction patterns — with the use by programs ITO, TREOR. The calculations for the refinement of the diffraction patterns, the definition and refinement of cell parameters were performed using program HighScore Plus 3.0.t (3.0.5).

## RESULTS

A series of numerical calculations was confirmed experimentally. During the interaction of amorphous  $\text{SiO}_2$  with a mass of short CNTs ( $\sim 0.5\ \mu\text{m}$ ) at room temperature, we discovered 2 kinds of self-organized structures: stem (Fig. 3a) and spherical (Fig. 3b). These structures differ not only by shape, but also by the ratio of atomic concentrations of Si:O — 4:1 for stem structures, and 1:2 for spherical structures, with high carbon concentration in both cases. Based on the obtained data, it follows that there is a formation of silicene oxycarbide in the stem structures. The formation of oxycarbide phases during the interaction of  $\text{SnO}_2$  with CNT, with the formation of O-C-Sn bond was convincingly shown in work [13].



**Fig. 3.** Stem (a) and spherical (b) self-organized structures of  $\text{SiO}_2/\text{CNT}$  (X10000)

This difference in structure is explained by two types of interactions  $\text{SiO}_2/\text{CNT}$  within the framework of proposed theoretical model: the formation

of covalent bond C-Si in case of stem structures, and Van-der-Waals interaction in case of spherical structures.

X-ray diffraction analysis of the obtained nanostructures revealed formation of the silicon carbide nanophase at room temperature (Fig. 4).

The formation and synthesis of new structures in SiO<sub>2</sub>/CNT nanosystem was confirmed by the results

of DLS analysis in colloidal suspensions and IR-spectroscopy of powder-like samples of aerosil with and without CNT. Using the DLS method, we found that the addition of CNT resulted in decrease of the size of initial globules.

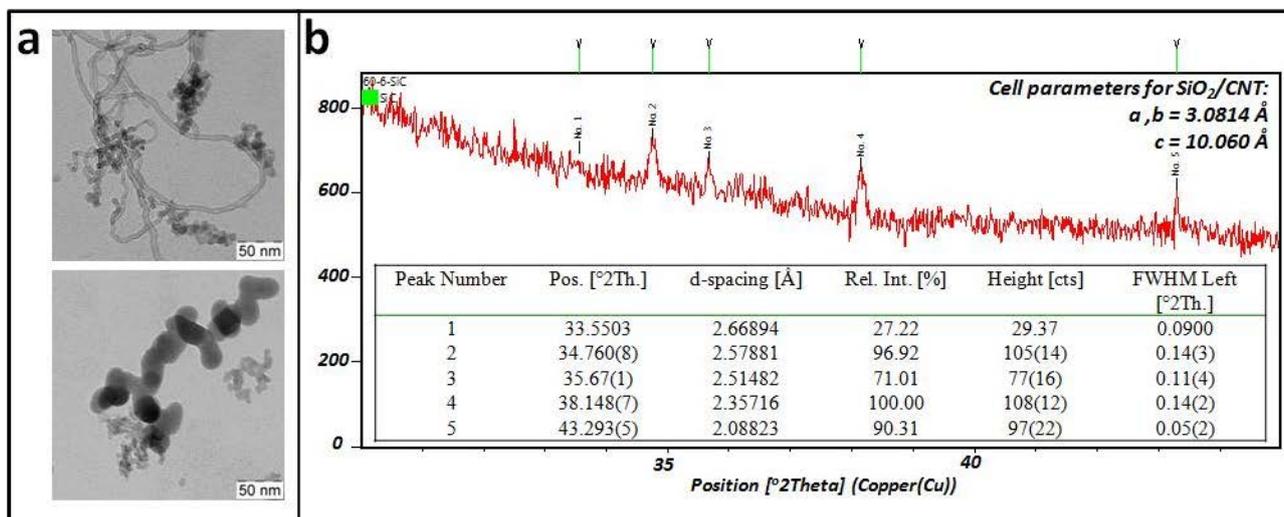


Fig. 4. TEM image (a) and diffraction pattern of the nanostructure of SiO<sub>2</sub>/CNT (b)

Investigation of the nanotubes length dependence on interaction between CNT and SiO<sub>2</sub> was done by IR spectroscopy. Aerosil compared with aerosil doped CNT with length < 0.5 μm and aerosil doped CNT with length < 3.0 μm (Fig. 5).

Oscillation at 1639 cm<sup>-1</sup> corresponding to the bending vibrations of adsorbed molecules of water.

Oscillation mode at 1211 cm<sup>-1</sup> represents the antisymmetric stretching vibrations of the Si-O-Si siloxane groups flattened chains of tetrahedra SiO<sub>4</sub>, the corners between which are equal to 180° [24]. Adding short CNTs leads to a shift mode in the long wavelength region of the spectrum.

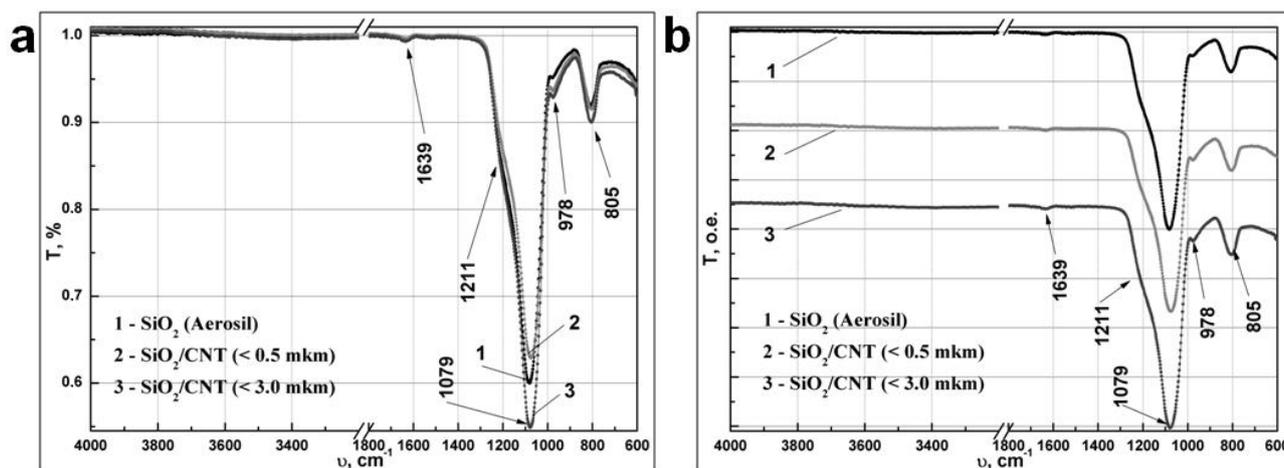


Fig. 5. Comparison of the IR spectrum mode aerosil (1) with tabulated values (a) and the spectrum changes when adding CNT length of 0.5 μm (2) and 3 μm (3) (b)

The intensity of the oscillation mode at 1079 cm<sup>-1</sup> in the sample aerosil/short CNT decreases, as compared with initial aerosil, and shifts toward longer

wavelengths. It is known that the absorption band in the IR spectra in the region 1100 (SiO<sub>2</sub>) — 980 — (SiO) — 943 cm<sup>-1</sup> (Si) linked to the stretching vibra-

tions of the bridging oxygen Si-O-Si (stretching mode Si-O-Si) and used for determining the composition of the layers SiO<sub>x</sub> [25]. Displacement of oscillation mode to lower frequencies indicates the increasing concentration of silicon in the sample aerosil/short CNT [26].

Fluctuations at 978 cm<sup>-1</sup> correspond to the stretching vibrations Si-(OH). Interval 900—500 cm<sup>-1</sup> belongs to the symmetric vibrations of Si-O [24].

In the case of adding of long CNT (~ 3.0 μm), at the spectrum of the sample is observed increase in the intensity of the vibrational modes, without asymmetries of oscillation at 1078 cm<sup>-1</sup> (Fig. 5a). These facts confirm the weak interaction of long CNT with aerosil.

### CONCLUSIONS

On the basis of IR spectroscopic studies and elemental analysis, we can conclude that composite on aerosil and short CNTs is forming with a high content of silicon. Doping by long nanotubes leads to aggregation of the globules of the aerosil without changing the composition.

Therefore, during the study of interactions between nanomaterials in CNT-aerosil system at room temperature in drying drop, we found that with nanotubes of lengths less than 0.5 μm there were two types of structures forming: spherical aggregates with diameter ~2 μm, and self-organized stem structures with diameter 250—300 nm and length ~4 μm. X-ray diffraction analysis of the obtained nanostructures revealed formation of the silicon carbide phase at room temperature. Theoretical analysis confirmed that the mechanism of formation of such structures is due to the presence of charged interface within the closed short nanotubes, near the border of cap/body, which increases their reaction capability. The formation of stem structures is due to covalent interaction, while the formation of spherical aggregates is due to van-der-Waals interaction.

*The authors would like to thank Marina Langer and DataPhysics Instruments GmbH company for their help in experiments to determine surface tension, as well as the center of collective use of scientific equipment of Voronezh State University for conducting SEM, TEM and X-ray diffraction.*

*This work was supported by the project FP7-IRSES-295260 «ECONANOSORB» under Marie Curie Actions of the 7th Framework Program of European Union and by RFBR, research projects N 14—02—31315.*

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УДК 538.975

## ХОЛОДНЫЙ СИНТЕЗ КАРБИДА КРЕМНИЯ В КОЛЛОИДНОЙ НАНОСИСТЕМЕ SiO<sub>2</sub>-УНТ

© 2014 Д. А. Жукалин<sup>1</sup>, А. В. Тучин<sup>1</sup>, Д. Л. Голощапов<sup>1</sup>, Л. А. Битюцкая<sup>1</sup>, Ф. Ресснер<sup>2</sup><sup>1</sup>Воронежский государственный университет, Университетская пл., 1, 394006 Воронеж, Россия<sup>2</sup>Университет имени Карла фон Осецкого, Ольденбург, Германия  
e-mail: d.zhukalin@mail.ru, frank.roessner@uni-oldenburg.de

**Аннотация.** Цель работы заключалась в исследовании взаимодействия наноразмерных компонентов: аморфного диоксида кремния SiO<sub>2</sub> со средним размером частиц 7 нм и массива коротких УНТ со средним диаметром 20—40 нм, полученных электродуговым методом. Изучая взаимодействие наноматериалов в коллоидной наносистеме SiO<sub>2</sub>-УНТ при комнатной температуре, в высыхающей капле (нанореакторе) обнаружено, что при длине трубок менее 0.5 мкм образуются 2 типа структур: сферические агрегаты с диаметром ~2 мкм и самоорганизованные стержневые гибридные структуры диаметром 250—300 нм и длиной ~4 мкм. Теоретически доказано, что механизм формирования подобного рода структур обусловлен наличием в закрытых коротких трубках заряженного интерфейса вблизи границы шапка/остов УНТ, повышающего их реакционную способность. Формирование стержневых структур обусловлено ковалентным взаимодействием, сферических агрегатов — взаимодействием Ван-дер-ваальса. Формирование и синтез новых структур в наносистеме SiO<sub>2</sub>-УНТ подтвердились результатами DLS анализа в коллоидных взвешях и ИК-спектроскопией порошкообразных образцов аэросила без и с УНТ. Методом DLS было обнаружено, что добавление УНТ привело к уменьшению размера исходных глобул, а анализ ИК-спектров выявил перераспределение интенсивностей и асимметрию мод колебаний. Проведенный дифракционный анализ полученных наноструктур выявил формирование нанофазы карбида кремния. Таким образом, в коллоидной наносистеме SiO<sub>2</sub>-УНТ проведен холодный синтез карбида кремния.

**Ключевые слова:** наносистема, углеродные нанотрубки, оксикарбид кремния, электронная структура, функционал плотности, заряд, нанокомпозит, карбид кремния.

Жукалин Дмитрий Алексеевич — аспирант кафедры физики полупроводников и микроэлектроники, Воронежский государственный университет; тел.: +7 (951) 568525, e-mail: d.zhukalin@mail.ru

Zhukalin Dmitry A. — post graduate student, Department of Physics of Semiconductors and Microelectronics, Voronezh State University; ph.: +7 (951) 5685250, e-mail: d.zhukalin@mail.ru

Тучин Андрей Витальевич — аспирант кафедры физики полупроводников и микроэлектроники, Воронежский государственный университет; тел.: +7 (908) 1485775, e-mail: a.tuchin@bk.ru

Tuchin Andrey V. — post graduate student, Department of Physics of Semiconductors and Microelectronics, Voronezh State University; ph.: +7 (908) 1485775, e-mail: a.tuchin@bk.ru

*Голощанов Дмитрий Леонидович* — к. ф.-м. н., ведущий инженер кафедры физики твердого тела и наноструктур, Воронежский государственный университет; тел.: +7 (905) 0531234, e-mail: goloshchapovdl@gmail.com

*Битюцкая Лариса Александровна* — к. х. н., доцент кафедры физики полупроводников и микроэлектроники, Воронежский государственный университет; тел.: +7 (473) 2208481, e-mail: me144@phys.vsu.ru

*Ресснер Франк* — д. х. н., профессор кафедры технической химии, Ольденбургский университет имени Карла фон Осецкого; тел.: +49 (441) 7983355, e-mail: frank.roessner@uni-oldenburg.de

*Goloshchapov Dmitry L.* — Cand. Sci. (Phys.-Math.), Chief Engineer, Senior Researcher, Department of Solid State Physic and Nanostructures, Voronezh State University; ph.: +7 (905) 0531234, e-mail: goloshchapovdl@gmail.com

*Bityutskaya Larisa A.* — Cand. Sci. (Chem.), Associate Professor, Department of Physics of Semiconductors and Microelectronics, Voronezh State University; ph.: +7 (473) 2208481, e-mail: me144@phys.vsu.ru

*Roessner Frank* — Dr. Sci. (Chem.), Professor of Industrial Chemistry Department, Carl von Ossietzky University of Oldenburg; ph. +49 (441) 7983355, e-mail: frank.roessner@uni-oldenburg.de