

**ISSN 2518-1726 (Online),
ISSN 1991-346X (Print)**

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

ӘЛЬ-ФАРАБИ АТЫНДАҒЫ
ҚАЗАҚ ҰЛТТЫҚ УНИВЕРСИТЕТИНІҢ

Х А Б А Р Л А Р Ы

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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН

КАЗАХСКИЙ НАЦИОНАЛЬНЫЙ
УНИВЕРСИТЕТ ИМЕНИ АЛЬ-ФАРАБИ

NEWS

OF THE NATIONAL ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN

AL-FARABI KAZAKH
NATIONAL UNIVERSITY

ФИЗИКА-МАТЕМАТИКА СЕРИЯСЫ

СЕРИЯ ФИЗИКО-МАТЕМАТИЧЕСКАЯ

PHYSICO-MATHEMATICAL SERIES

3 (319)

МАМЫР – МАУСЫМ 2018 ж.

МАЙ – ИЮНЬ 2018 г.

MAY – JUNE 2018

1963 ЖЫЛДЫҢ ҚАҢТАР АЙЫНАН ШЫҒА БАСТАҒАН
ИЗДАЕТСЯ С ЯНВАРЯ 1963 ГОДА
PUBLISHED SINCE JANUARY 1963

ЖЫЛЫНА 6 РЕТ ШЫҒАДЫ
ВЫХОДИТ 6 РАЗ В ГОД
PUBLISHED 6 TIMES A YEAR

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ISSN 2518-1726 (Online), ISSN 1991-346X (Print)

Меншіктенуші: «Қазақстан Республикасының Үлттық ғылым академиясы» РКБ (Алматы қ.)
Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрағат комитетінде
01.06.2006 ж. берілген №5543-Ж мерзімдік басылым тіркеуіне қойылу туралы қуәлік

Мерзімділігі: жылдана 6 рет.

Тиражы: 300 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., 220, тел.: 272-13-19, 272-13-18,
www.nauka-nanrk.kz / physics-mathematics.kz

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Типографияның мекенжайы: «Аруна» ЖК, Алматы қ., Муратбаева көш., 75.

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«Известия НАН РК. Серия физико-математическая».

ISSN 2518-1726 (Online), ISSN 1991-346X (Print)

Собственник: РОО «Национальная академия наук Республики Казахстан» (г. Алматы)

Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан №5543-Ж, выданное 01.06.2006 г.

Периодичность: 6 раз в год.

Тираж: 300 экземпляров.

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, ком. 219, 220, тел.: 272-13-19, 272-13-18,
www.nauka-nanrk.kz / physics-mathematics.kz

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Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75.

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News of the National Academy of Sciences of the Republic of Kazakhstan. Physical-mathematical series.

ISSN 2518-1726 (Online), ISSN 1991-346X (Print)

Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty)
The certificate of registration of a periodic printed publication in the Committee of information and archives of the Ministry of culture and information of the Republic of Kazakhstan N 5543-Ж, issued 01.06.2006

Periodicity: 6 times a year

Circulation: 300 copies

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19, 272-13-18,
www.nauka-nanrk.kz / physics-mathematics.kz

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Address of printing house: ST "Aruna", 75, Muratbayev str, Almaty

NEWS

OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN

PHYSICO-MATHEMATICAL SERIES

ISSN 1991-346X

Volume 3, Number 319 (2018), 14 – 22

UDC 539.23; 539.216.1

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EFFECT OF PLASMA PARAMETERS ON THE SYNTHESIS OF CARBON NANOMATERIALS BY THE PECVD METHOD

Abstract: This work covers an experimental study of the effect of plasma parameters on the synthesis of carbon materials by the PECVD method. It was found that, depending on PECVD synthesis parameters, in particular, temperature, discharge power, gas pressure, percentage of gas mixture and etc., various carbon nanomaterials are synthesized. The obtained samples were analyzed by using analytical equipment such as Quanta 3D scanning electron microscope (SEM, FEI USA), NThegra Spectra Raman spectroscopy and Leica optical microscope. Thus, the morphology and quality of the structure of the obtained samples (carbon nanoparticles (CNP), carbon nanofibres (CNF) and nanotubes (CNTs), carbon nanowalls (CNWs) and multilayered graphene sheets) were studied by optical and electron microscopies, as well as by the method of combined light scattering. Found, that with the increase of power of the radio-frequency discharge, the production of qualitative nanostructures is complicated by the formation of their nanoclusters. For the synthesis of CNT, it is necessary to control the thickness of catalytic nanolayer, since the quality of CNT structures can be worsened by the formation of thicker nanofibers. The obtained experimental results can be used to determine the optimum PECVD synthesis condition to synthesize various carbon nanomaterials.

Keywords: carbon nanoparticles, carbon nanofibers, carbon nanotubes, carbon nanowalls, multilayer graphene, radio-frequency (RF) discharge plasma.

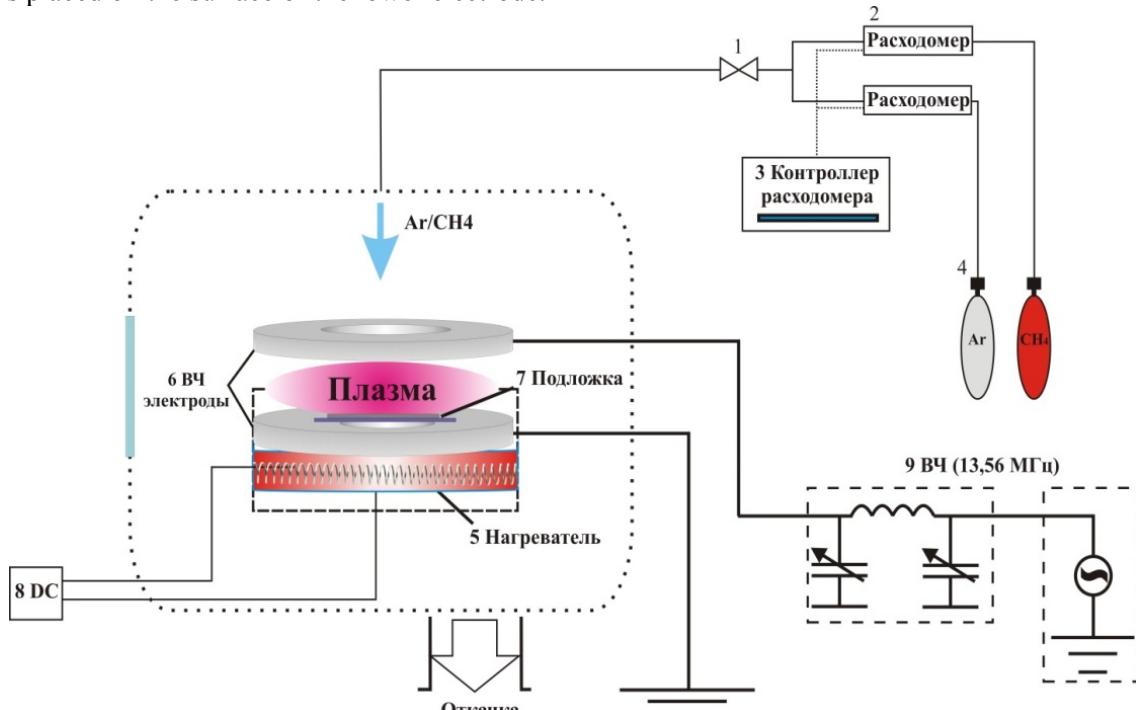
Introduction

Analysis of the state and trends in the modern development of nanoindustry objects allows us to conclude that one of the most promising areas of nanotechnology is the production of carbon nanomaterials (CNMs). As is well known, carbon exists in the solid phase in several modifications, the properties of which sharply differ from each other: carbon nanoparticles [1-2], carbon nanofibres, carbon nanotubes [3-4], graphene [5-6], carbon nanowalls [7- 8], fullerene [9] etc. The research relevance of CNMs is due to the wide range of their practical application in various areas of industry. For example, carbon nanoparticles can find their application in water purification [10,11], automobile tires as reinforcing fillers [12], in determining cancer cells at an early stage [13], etc. Scientists suggest the use of carbon nanotubes as reinforcing elements for the production of high-quality concrete [14,15] in construction. The unique properties of CNTs are also used for medicine purposes like water disinfection, production of antimicrobial coatings and drugs [16] and in targeted drug delivery [17]. CNTs and composites based on them are used in power engineering to store hydrogen [18], to create high-capacity capacitors (supercapacitors) [19]. The unique semiconductor properties of graphene allow them to be used in electronics to create highly sensitive sensors, high-speed electronic devices [20,23] etc. As for carbon

nanowalls (CNW), they are one of the allotropic modifications of carbon, which are vertically oriented graphene sheets [24,25], which find their application for energy storage as a blackbody-like material, for bolometers [26] and solar cells [27] as electrodes for supercapacitors [28,29], etc. Thus, taking into account the relevance of CNM, in this work a method of plasma-enhanced chemical vapor deposition (PECVD) was used for the synthesis of carbon nanomaterials at different plasma parameters.

Experimental part

The scheme of the experimental setup is shown in Fig. 1. The PECVD system consists of a working chamber, a system of radio-frequency electrodes (6), a heating element (5), which reduces the effect of power RF discharge on the process of dissociation of carbon-containing gas for the gas-phase deposition process of a CNT, power supply for heating element (8), a high-frequency (RF) generator (9) for igniting a plasma, a vacuum system and a gas inlet system. The substrate with the catalyst, in this case, a nickel one, is placed on the surface of the lower electrode.



1 - valve, 2 - gas flowmeters, 3 - gas flowmeter controller, 4 - argon and methane gas cylinders,
5 - heating element, 6 - high frequency (RF) electrodes, 7 - substrate, 8 - power supply for heating element,
9 - HF (RF) generator with automatic matching device

Figure 1 – Structure of the PECVD system for the synthesis of nanocomposite materials based on CNTs

The experiment was carried out as follows. First, a silicon substrate with a catalytic nanolayer was annealed to form the islands of the nickel nanoclusters. To do this, the substrate is loaded into the working chamber on the surface of the lower electrode and a vacuum condition is created, after the vacuum is established, the working gas argon (Ar) flow is supplied to a pressure of the order of 1 Torr, the RF plasma is ignited in the power range 1-25 W at the temperature of 400°C and processed for 10 minutes. This process is well described in [30]. Further, for the growth of CNTs, a reaction carbon-containing gas, methane, is injected into the working chamber up to a pressure of 1.1-1.6 Torr, and then the synthesis process lasts for 15-30 minutes.

Figure 2 shows photographs of silicon substrates with a nickel catalyst before and after CNT synthesis by the PECVD method. It can be seen that after the synthesis, the surface of the substrate has a soot formation, which indicates the possible deposition of carbon nanostructures.

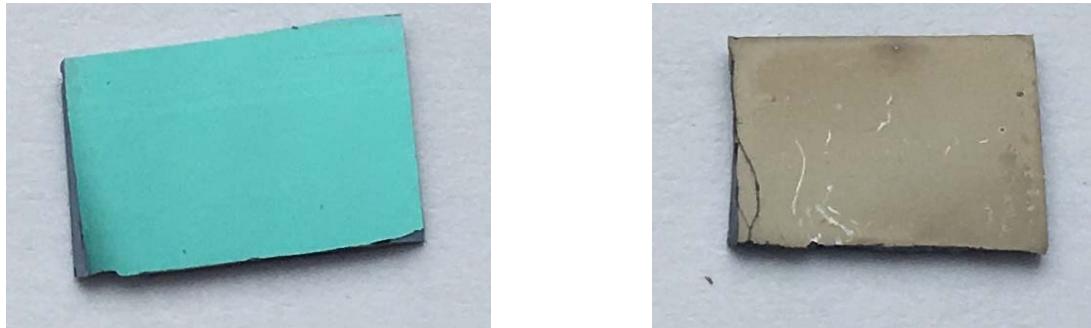


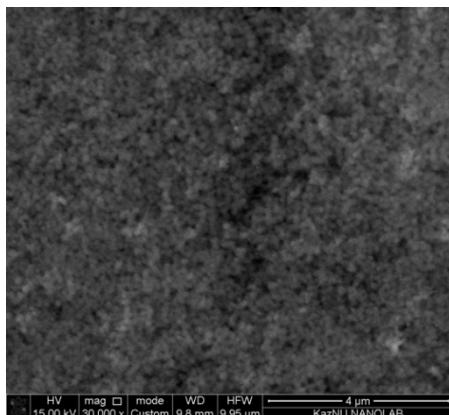
Figure 2 – Silicon substrates with nickel catalyst before (left) and after (right) CNT synthesis by the PECVD method

Results and discussion

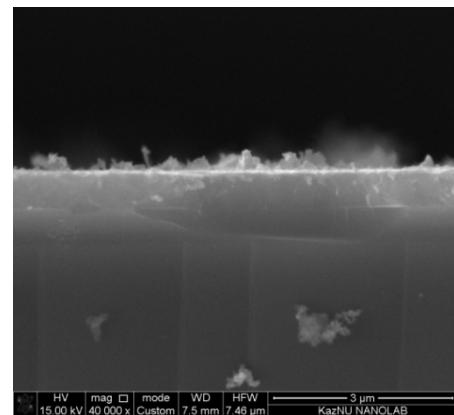
The samples synthesized by the PECVD method were investigated by scanning electron microscopy (SEM) and Raman spectroscopy (Raman spectroscopy).

SEM images and Raman spectra of the samples obtained at temperatures 400-450°C, pressure of 1.3 Torr and discharge power in the range of 1-10 W are shown in Fig. 3. As can be seen, the surface of the obtained samples has a deposition in the form of carbon nanoparticles with sizes of the order of 50 -100 nm and a carbon film. The absence of CNTs is explained by the low synthesis temperature for the formation of nanoclusters (crystalline structure).

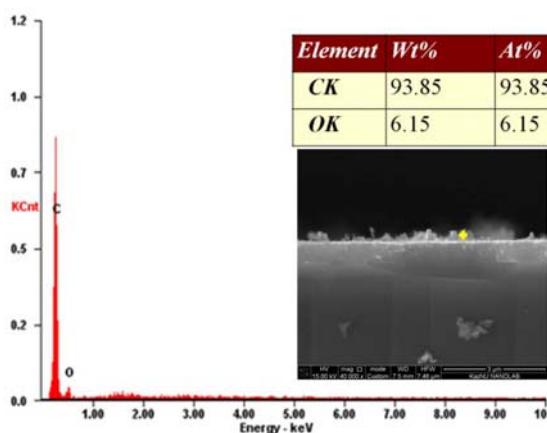
Further, the experimental work was carried out at a temperature of 500°C, a gas pressure of methane/argon of 1-1.8 Torr, and a discharge power in the range of 1-25 W.



a – SEM image, shooting angle 0⁰



b – SEM image, shooting angle 90⁰



c – chemicalcomposition

Figure 3 – SEM analysis of samples after PECVD synthesis at a temperature of 400-450°C and discharge power of 1-10W

In experiments with synthesis conditions: pressure 1-1.8 Torr, temperature 500°C and duration of 20 minutes, carbon nanowires (1-5 W) and nanotubes (5-7 W) were obtained, which was proved by the results of SEM and Raman studies, as well as optical microscopy.

Optical micrographs and SEM images of the obtained samples of carbon nanofibers and nanotubes are presented in Figures 4 and 5, respectively.

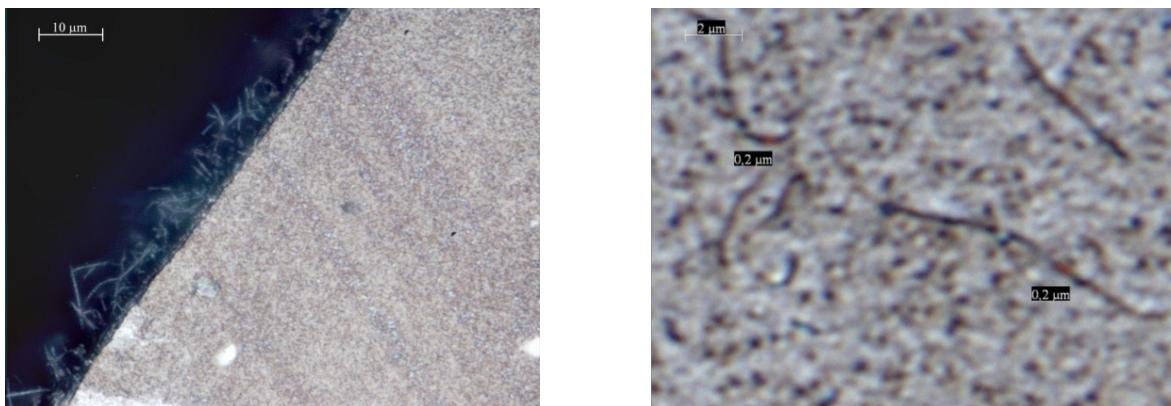


Figure 4 – Optical micrographs of carbon nanofibers synthesized at discharge power of 1-5 W and temperature of 500°C

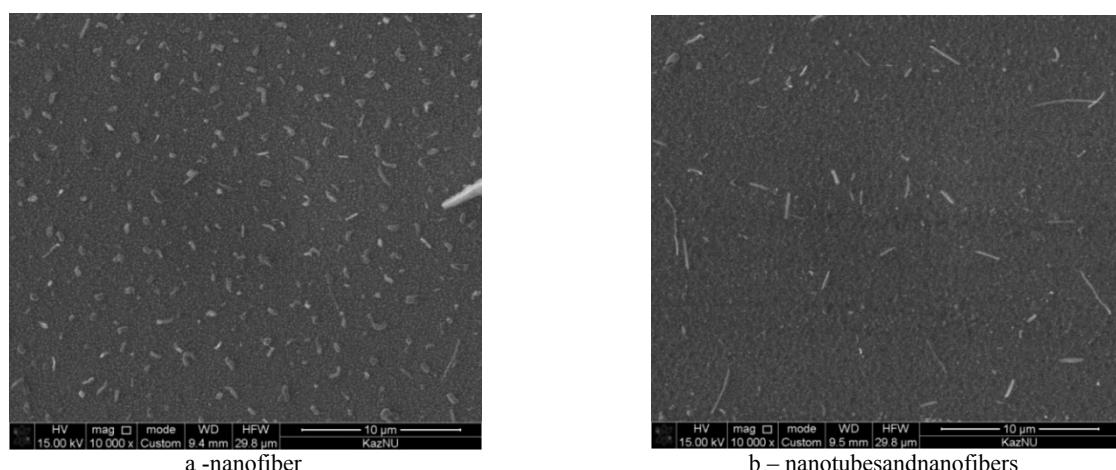


Figure 5 - SEM images of carbon nanofibers and nanotubes at a discharge power of 1-7 W and temperature of 500°C

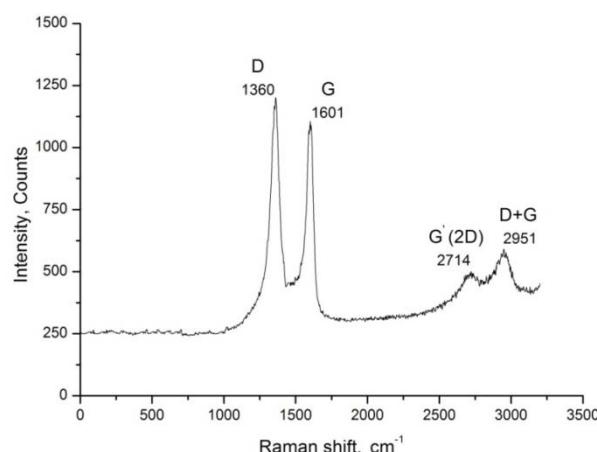
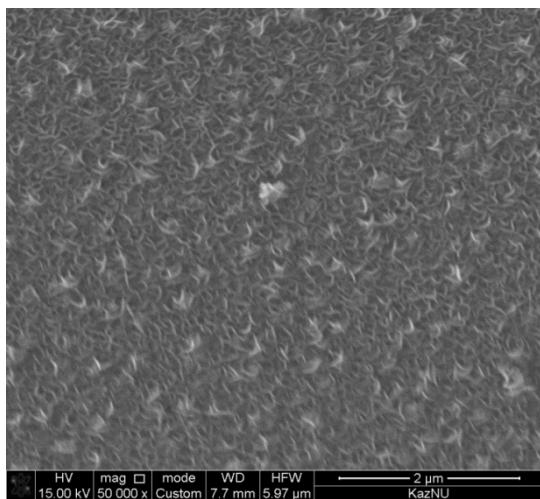


Figure 6 – Raman spectrum of the obtained nanofibers (nanotubes) at a discharge power of 1-7 W and a temperature of 500°C

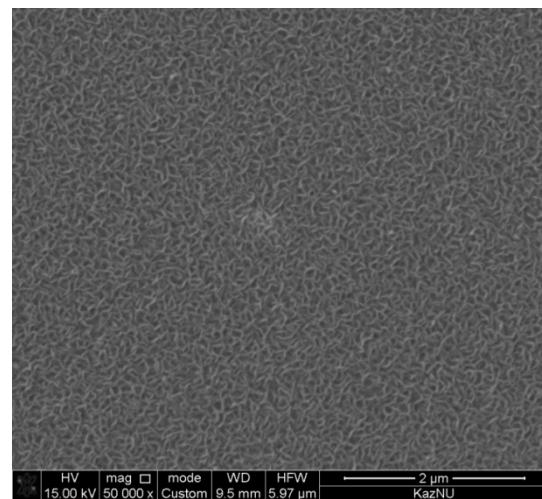
The result of Raman study of the obtained samples is shown in Fig. 6. The obtained spectrum is typical for the multiwalled carbon nanotubes (MWCNT) and demonstrates G mode, which is usually observed in graphite-like materials, D mode, which is associated with defects in the structure, the second harmonic of the D mode - G'(2D) mode and G+D mode, the origin of which is not fully understood yet.

It can be seen from the spectrum that the obtained MWCNTs are not of the high quality. The intensity ratio of D and G peaks, responsible for the perfection of the graphene structure, is equal to 1.1, where for CNTs it is about 1.5 and higher. In addition, the position and partial overlapping of D and G peaks indicate amorphous structure. The low quality of MWCNTs is probably due to the presence of additional constituents like nanofibers, which have higher diameter and more disordered structure.

With the increase in discharge power in the range of 10-15 W, the formation of carbon nanowalls (CNW) was detected, which was confirmed by the results of SEM, Raman analysis (Figures 7-9).



a – 15ВТ



b – 10ВТ

Figure 7 – SEM images of CNW synthesized on Ni/Si substrates at a discharge power of 15 W (a) and 10 W (b) at a temperature of 500°C

As can be seen from the SEM image, after the synthesis process, the surface of the silicon substrate becomes covered with the vertically aligned carbon sheets, also known as carbon nanowalls (CNW). As the discharge power is increased, it is possible to observe the agglomeration of nanolayers - the formation of nanoclusters of the walls (Figures 7a and 8).

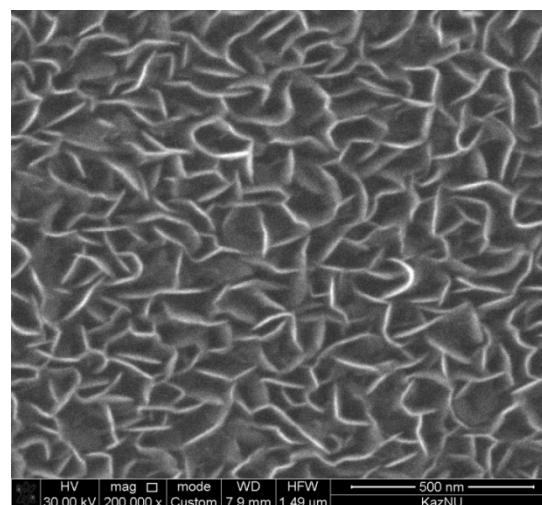
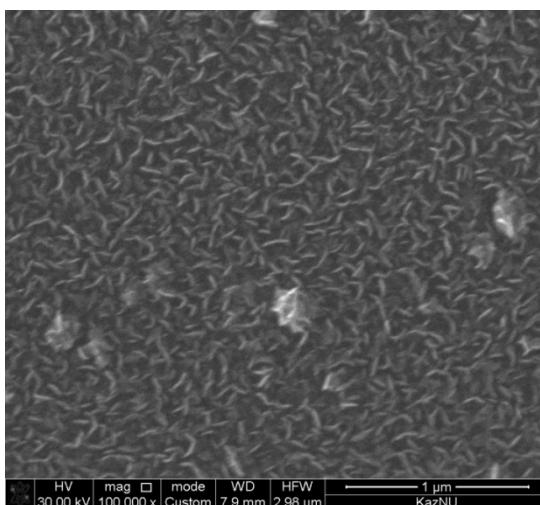


Figure 8 – SEM images of CNW synthesized on Ni/Si substrates at discharge power of 15 W and the temperature of 500°C

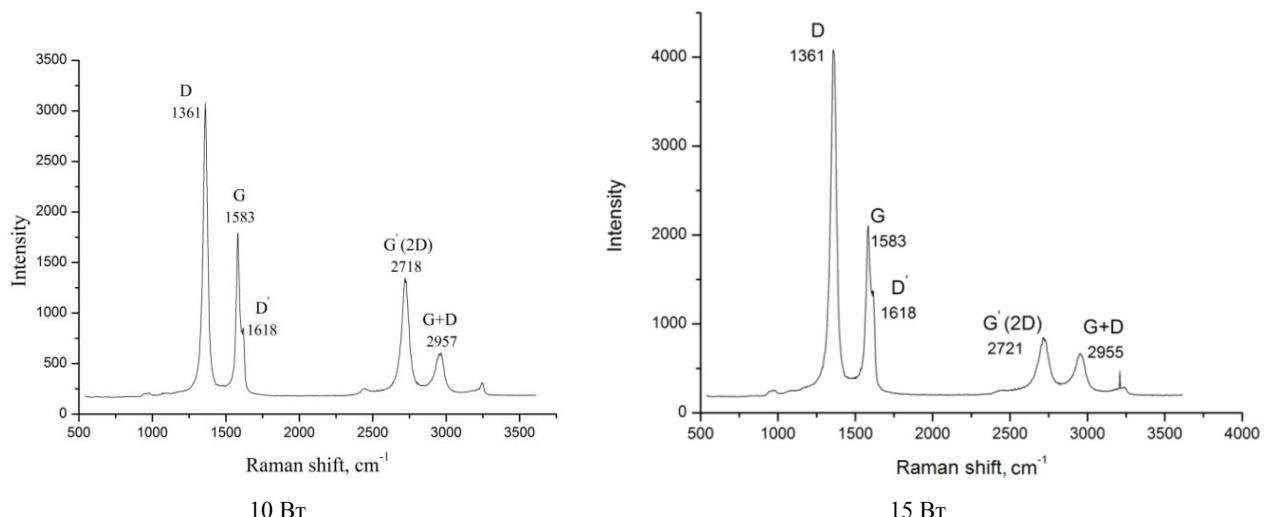
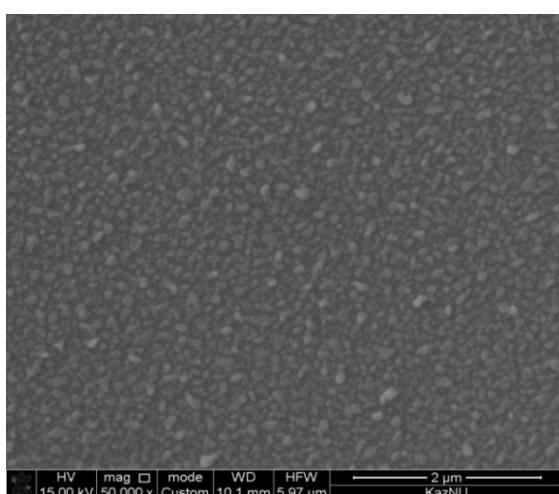


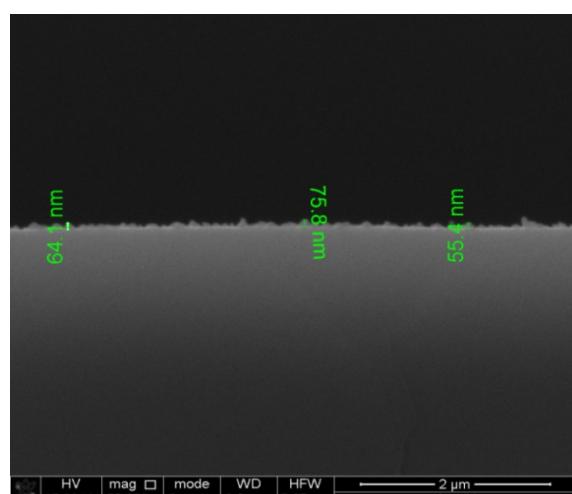
Figure 9– Raman spectrum of CNW synthesized on Ni/Si substrate at discharge power of 10 and 15 W at the temperature of 500°C

The Raman spectrum of the obtained samples corresponds to the typical spectrum of CNW [31-33], while the G-mode, which is usually observed in graphite materials, has a redshifted shoulder - D' peak. The spectrum also contains characteristic D-peak, associated with defects in the sp² structures, 2D (G') - peak, which corresponds to the second order of the D-mode and the G+D mode. The intensity ratio of the D and G modes, which indicates the degree of disorder (defectiveness) in the crystal lattice of the graphene sheet, ranges from 1.7 to 1.9. The calculated in-plane correlation length L_a varies from 2.5 to 2.3 nm, the region in which the CNW can be considered as defect-free. As mentioned above, with the increase of discharge power, the agglomeration of nanowalls occurs, and the ratio I(D)/I(G) increases as well, which indicates the formation of defects in the structure.

With a further increase in the discharge power in the range of 20-25 W and under the same parameters of temperature and pressure, multilayer graphene sheets were obtained (Fig. 10,11). As can be seen from the SEM images, the islands of multilayer graphene with a thickness of the order of 50-75 nm were formed on the surface of the silicon substrate. Raman studies indicate that the obtained structures correspond to the graphite-like material. The in-plane correlation length L_a is 2 nm, and the ratio I(D)/I(G) is 1.4.



a – SEM image, shooting angle 0⁰



6 – SEM image, shooting angle 90⁰

Figure 10 – SEM analysis of the samples after PECVD synthesis at 500⁰C and discharge power of 15-25 W

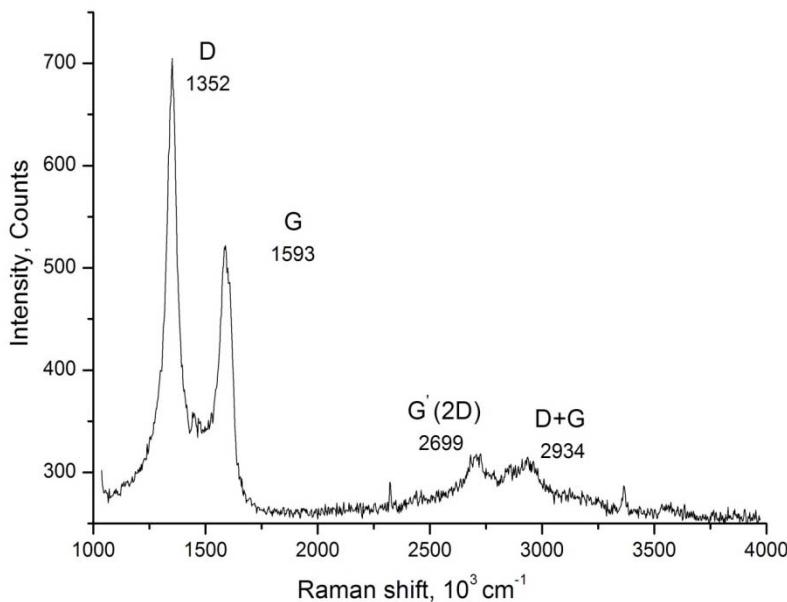


Figure 11 – Raman spectrum of the samples after PECVD synthesis at 500°C and discharge power of 15-25 W

Table 1 represents parameters for the synthesis of carbon nanomaterials by PECVD method

Table 1 – Parameters for the synthesis of carbon nanomaterials by PECVD method

Resulting product	Synthesis parameters			
	Pressure, torr	Temperature, $^\circ\text{C}$	Discharge power, W	Gas mixture
Carbon nanoparticles	1.1-1.6	400-450	1-15	Ar/CH ₄
Carbon nanofibers and nanotubes	1.1-1.6	500	1-7	Ar/CH ₄
Carbon nanowalls	1.1-1.6	500	8-15	Ar/CH ₄
Multilayered graphene	1.1-1.6	500	20-25	Ar/CH ₄

Conclusion

Thus, various carbon nanomaterials were obtained and characterized. It was experimentally revealed that the type of the synthesized nanomaterial depends on the value of discharge power. At temperatures of 400 - 450°C and discharge power of 1-15 W carbon nanoparticles are synthesized, with increasing temperature up to 500°C carbon nanostructures are formed, in particular, at discharge power of 1-7 W - carbon nanofibres and nanotubes, 8-15 W - carbon nanowalls, 20-25 W multi-layered graphene. The obtained experimental results can be used to determine the optimum PECVD synthesis condition to synthesize various carbon nanomaterials.

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ПЛАЗМА ПАРАМЕТРЛЕРИНІҢ ҚӨМІРТЕКТІ НАНОМАТЕРИАЛДАРДЫҢ PECVD ӘДІСІМЕН СИНТЕЗІНЕ ӘСЕРІ

Аннотация. Аталған жұмыс плазма параметрлерінің қөміртекті наноматериалдардың PECVD әдісімен синтезіне әсірін эксперименталды зерттеуге арналған. PECVD параметрлеріне, температура, разряд қуаты, газ қысымы, газдардың пайыздық үлесіне байланысты әртүрлі қөміртекті наноматериалдар синтезделетіні анықталды. Алынған үлгілер сканерлеуші электрондық микроскоп Quanta 3D (CЭМ, FEI USA), Рамандық спектрометр NThegra Spectra, оптикалық микроскоп Leica сияқты аналитикалық қондырығылар қөмегімен зерттелді. Осылайша, оптикалық және электрондық микроскоп, және де комбинациялық жарық шашырау қөмегімен құрылымдардың морфологиясы мен сапасы зерттелді: қөміртекті нанобөлшектер(KНБ), қөміртекті наноталшық (КНТ) пен нанотүтікше (КНТ), қөміртекті наноқабырға (КНҚ) және көпқабатты графен паракшалары. ЖЖ разряд қуатын өсірген кезде наноқабырғалардың нанокластерге құрылуы қыындаудытыны анықталды. КНТ синтезі үшін каталитикалық наноқыбықтықшаның қалындығын басқару қажет, себебі КНТ құрылымы нашарлап наноталшықтың өсуіне алып келеді. Алынған нәтижелер, PECVD әдісімен әртүрлі қөміртекті наноматериалдарды синтездеуде қолданылуы мүмкін.

Тірек сөздер: қөміртекті нанобөлшектер, қөміртекті наноталшықтар, қөміртекті нанотүтікшелер, қөміртекті наноқабырғалар, көпқабаттыграфен, жогары жиілікті разряд плазмасы.

УДК 539.23; 539.216.1

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ВЛИЯНИЕ ПАРАМЕТРОВ ПЛАЗМЫ НА СИНТЕЗ УГЛЕРОДНЫХ НАНОМАТЕРИАЛОВ МЕТОДОМ PECVD

Аннотация. Данная работа посвящена экспериментальному исследованию влияния параметров плазмы на синтез углеродных материалов методом PECVD. Установлено, что в зависимости от параметров PECVD синтеза в частности, температуры, мощности разряда, давление газа, процентное соотношение смеси газов и т.д., синтезируются различные углеродные наноматериалы. Полученные образцы были исследованы с помощью аналитических оборудований, таких как сканирующий электронный микроскоп Quanta 3D (CЭМ, FEI USA), Рамановский спектроскоп NThegra Spectra, оптический микроскоп Leica. Таким образом, с помощью оптической и электронной микроскопии, а также методом комбинированного рассеяния света были исследованы морфология и качество структуры полученных образцов: углеродные наночастицы (УНЧ), углеродные нановолокна (УНВ) и нанотрубки (УНТ), углеродные наностены (УНТ) и многослойные графеновые листы. Установлено, что с увеличением мощности ВЧ разряда получение качественных наностен усложняется формированием их нанокластеров. Для синтеза УНТ необходимо контроль толщины каталитического нанослоя, так как качество структур УНТ может ухудшиться формированием более толстых нановолокон. Полученные результаты могут быть использованы для определения оптимальных условий PECVD метода для синтеза различных углеродных наноматериалов.

Ключевые слова: углеродные наночастицы, углеродные нановолокна, углеродные нанотрубки, углеродные наностены, многослойныйграфен, плазма высокочастотного разряда.

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ISSN 2518-1726 (Online), ISSN 1991-346X (Print)

Редакторы М. С. Ахметова, Т.А. Апендиев, Д.С. Алеков
Верстка на компьютере А.М. Кульгинбаевой

Подписано в печать 05.06.2018.
Формат 60x881/8. Бумага офсетная. Печать – ризограф.
10 п.л. Тираж 300. Заказ 3.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-18, 272-13-19