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Investigation of SiC and C nanostructures synthesized by the method of chemical vapor deposition on Ni films

A.K. Kenzhegulov¹, B.Z. Mansurov¹, B.S. Medyanova^{1*}, G. Partizan¹,
G.S. Suyundykova¹, B.E. Zhumadilov¹, U.P. Koztayeva², X. Jiang³

¹Center for Innovative Technologies of the Institute of Combustion Problems,
050050, 20, Polezhaeva str., Almaty, Kazakhstan

²Institute of Nuclear Physics, 050032, 1, Ibragimov str., Almaty, Kazakhstan

³Institute of Materials Engineering, University of Siegen,

Paul-Bonatz-Straße 9-11, 57076, Siegen, Germany

*e-mail: bota2192@mail.ru

The paper presents the results of experiments on synthesis of SiC and C nanostructures by the method of microwave plasma assisted chemical vapor deposition on Ni films. The plates of polished single-crystal and porous silicon were used, on the surface of which a thin layer of Ni was deposited. The dependence of the structure and morphology of the samples on the plasma power is studied. Scanning electron microscopy has shown that the formed nanostructures have a diameter of 100-170 nm and a rough surface. Analysis of the results showed that, the growth of nanostructures on the surface of porous silicon is more massive in contrast to polished Si. By means of Raman scattering the structure of the obtained samples and its dependence on the plasma power were studied. The results of studies showed the presence of silicon carbide nanostructures with 3C-SiC polytype structure. Also, the main carbon peaks in the range of ~ 1300 and ~ 1500 cm^{-1} , which correspond to carbon nanostructures were found on both types of substrates.

Key words: silicon carbide and carbon nanostructures, chemical vapor deposition, microwave plasma, porous silicon.
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1 Introduction

Nanostructuring of silicon carbide (SiC) allows changing many of its physical properties, for example, mechanical elasticity in comparison with bulk material [1]. Other interesting properties of SiC nanostructures (NS) include the presence of field emission, the threshold and properties of which are comparable to that demonstrated by carbon nanotubes [2]. These and many other properties make silicon carbide NS extremely attractive for research.

Filamentous SiC crystals, like carbon nanotubes, have a high ratio of length to diameter (100-200 and more), the diameter of crystalline SiC fibers can vary considerably from 40 to 200-500 nm [3]. The filamentary form provides improved mechanical properties of whiskers [4]. In addition to the traditional use as components of ceramics and structural materials, SiC whiskers are of interest for the development of new components of high-temperature electronic devices. Advantages of using SiC in this area are associated with its high thermal and corrosion resistance, combined with the large

value of the forbidden zone (2.4-3.3 eV, depending on the polytype of silicon carbide) [5].

The main method for SiC production is the Acheson process, which is carbothermal reduction of SiO_2 by coke at 2200-2500°C [6]. Due to high reaction temperatures and long reaction time, the synthesized SiC has often large particle size and is contaminated with impurities such as oxygen and metals [7]. SiCNTs are currently synthesized by using chemical vapor deposition (CVD) [8] and carbothermal reduction of the silica by using conventional heating [9]. However, both of these methods have the downsides in the synthesis of the SiCNTs such as CVD method can only synthesize small amounts of SiCNTs and resulted in the presence of impurities in the SiCNTs due to extensive use of chemical during the synthesis of SiCNTs. Meanwhile, long heating duration, slow heating rate and requirement for further purification step to remove impurities was the problem always associated with the use of conventional heating method in carbothermal reduction of silica. Additional production cost was also needed since large consumption of energy was required to for the

synthesis of SiCNTs. In addition, Latu-Romain et. al [10,11] in their study of synthesis of the SiCNTs from silicon nanowire (Si NW) has successfully synthesized SiCNTs by hot filament CVD at temperature of 1100°C for 30 minutes using gold as catalyst. Continuous diffusion of Si gas into the layer of SiC has resulted in the formation of SiCNTs. However, the use of gold as catalyst incurred high cost and the need to synthesize Si NW required additional processing step which are time and energy consuming [7].

Recently, many researchers have utilized microwave heating to synthesize SiC because of special characteristics of microwave heating in which microwave can volumetrically heat materials with favorable dielectric properties and can synthesize SiC with uniform grain size at higher synthesis rate and reduced reaction time [12,13]. It is more economical to synthesize materials using microwave heating as compared to conventional methods such as mechanical milling, carbothermal reduction and laser synthesis since microwave heating is well known for low energy consumption, shorter reaction time and very low impurities such as oxygen and metals reside in the end products after synthesis [14-16]. The characteristic of microwave heating is attributed to the fact that microwave is a form of electromagnetic energy with a frequency range from 300 MHz to 300 GHz and microwave can couple with certain materials which have excellent dielectric properties such as carbon and it can be absorbed into these materials volumetrically which then transformed to heat from the inside of materials [17,18]. Conventional heating method

involves the transfer of heat between objects by the mechanisms of conduction, convection and radiation [19]. Heat loss to surrounding and irregular heat transfer between the heat source and material often becomes the issue of conventional heating and causes low heating rate and high energy consumption. Li et. al [20] have reported that by using microwave heating, one can successfully synthesize nanostructured β -SiC in argon atmosphere. Compared to conventional heating, microwave heating was proven to be an efficient approach to synthesize SiC in terms of low energy consumption and time saving [7].

The paper presents the results of experiments on synthesis of SiC and C nanostructures by the method of microwave plasma assisted chemical vapor deposition on Ni films. As a result of studies, the dependence of the plasma power and type of substrate on the structure and morphology of the obtained samples was studied.

2 Experimental

2.1 Preparation of substrates

Single-crystal silicon wafers (analogue of SHB-20 brand, manufactured by Siegert Wafer GmbH, Germany) with dimensions of 1×1 cm with orientation [100] and [111] were used as substrates and basis for nickel films. In order to activate the surface during synthesis, substrates of porous silicon (PS) were prepared at the Educational Laboratory of Semiconductor Instrumentation of Faculty of Physics and Technology of KazNU (Almaty, Kazakhstan) [21]. Figure 1 shows electron microscopic images of PS surface.

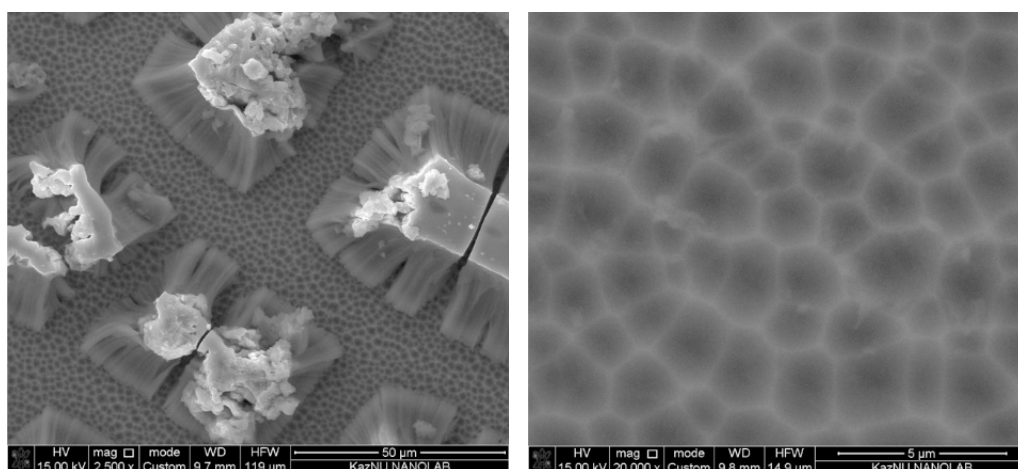


Figure 1 – Electron microscopic images of PS surface

Ni films were deposited by magnetron sputtering at a constant current in equipment VUP-5M for 5 minutes. Sputtering was carried out in a flow of working gas Ar at a pressure of 10^{-2} Torr. The Ar flow rate was $6 \text{ cm}^3/\text{min}$ and was controlled by gas flow controller MCV-500SCCM. The experiments were carried out at a voltage on the target of 600 V and plasma current of 30 mA.

2.2 Synthesis of nanostructures

Synthesis of NS was carried out in the Department of Surface and Technology of New Materials of the Institute of Materials Engineering of the University of Siegen, Germany, on equipment of microwave plasma assisted chemical vapor deposition (MWCVD) of ASTEX system (frequency 2.45 GHz).

Prior to the experiments, substrates were purified with ethanol and then washed with distilled water, drying was carried out at room temperature. The plasma power varied from 1600 to 2200 W in 200 W steps. Depending on the power, the substrate temperature was varied from 600 to 900°C and measured with infrared pyrometer of Chino IR-AP

M0011 model (Japan). The pressure in the chamber changed depending on the power to stabilize the plasma. Table 1 shows parameters of the experiments.

The experiments were carried out in atmosphere of a mixture of gases of trimethylsilane ($(\text{CH}_3)_4\text{Si}$) and hydrogen, the flow rate of which was 10 and $400 \text{ cm}^3/\text{min}$, respectively. The duration of all experiments is 120 minutes, the substrates are Ni on polished (Ni/Si) and porous (Ni /PsSi) Si of different orientations.

The obtained samples were studied by the methods of Raman scattering and scanning electron microscopy (SEM). Raman studies were carried out at the National Nanotechnology Laboratory of open type (Almaty, Kazakhstan) using spectrometer NT-MDT NTegra Spectra (laser wavelength $\lambda = 473 \text{ nm}$). Analysis of samples by SEM was carried out at the Institute of Materials Science of the University of Siegen. To study the morphology and elemental analysis of samples, high-resolution scanning electron microscope of Gemini Ultra 55 model of Zeiss company with device for X-Ray spectral microanalysis from Thermo Scientific was used.

Table 1 – Parameters of the experiments

Substrate		Si orientation	Power plasma, W	Substrate temperature, $^\circ\text{C}$	Pressure, Torr
Ni/Si	Polished	[100]	1600	600	40
			1800	700	47
			2000	800	50
			2200	900	57
	Porous	[111]	1600	600	40
			1800	700	47
			2000	800	50
			2200	900	57

3 Results and discussion

3.1 NS synthesized on substrates Ni/PSSi (100) and Ni/Si (100)

Figure 2 shows results of studies of samples synthesized at a substrate temperature of 600°C . Samples contain shifted silicon carbide peaks characteristic of 3C-SiC polytype structure, which are in the range of 778 and $898.1\text{-}928.7 \text{ cm}^{-1}$. Displacement can occur due to the presence of another crystalline structure, i.e. silicon carbide nanofibers [22]. The main carbon peaks are located in the region of 1354.6 , 1576.9 cm^{-1} and 1348.1 , 1573.7 cm^{-1} . The intensity of *D* peak and

the presence of *D + G* peak in the region of 2941.6 cm^{-1} in the samples obtained on porous substrates indicate the defectiveness of the nanostructures. The samples are crystalline, indicating the presence of *2D* peak at 2715.8 cm^{-1} and 2718.7 cm^{-1} .

The growth of NS both for polished and porous Si has a massive character over the entire surface of the substrate (*2a, d*). The formed NS have a diameter of 100-120 nm (*2c*). It is seen that the formed NS have a rough surface on SEM images (*2b, c*). Energy dispersive X-Ray spectra (Figure 3) indicate that the ratio of carbon and silicon concentrations in NS is on polished Si ~ 0.5 and on PS ~ 0.7 .

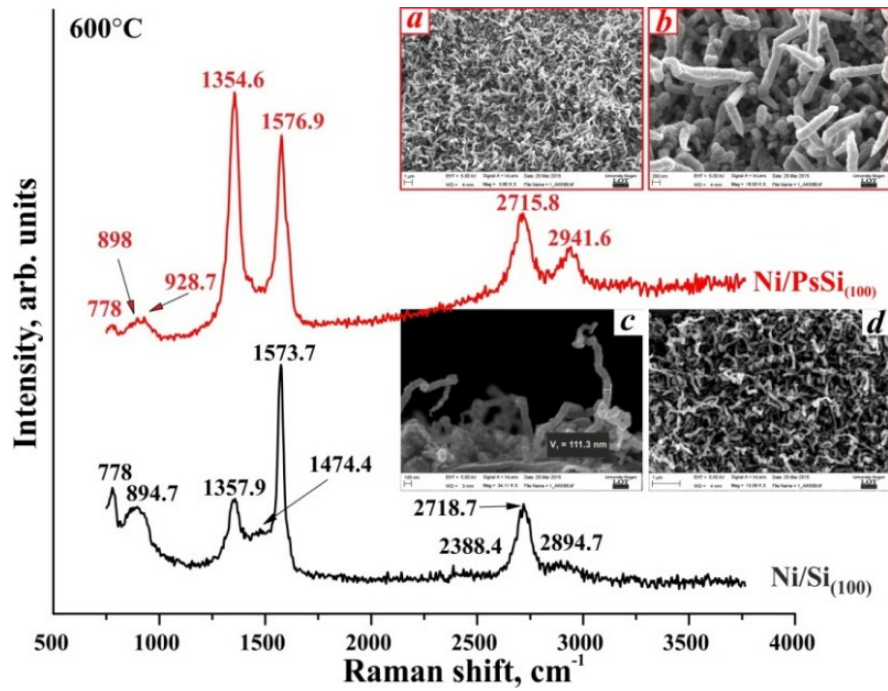


Figure 2 – Raman spectra and SEM images of samples synthesized at a substrate temperature of 600°C: *a, b* - Ni/PSSi₍₁₀₀₎; *c, d* - Ni/Si₍₁₀₀₎

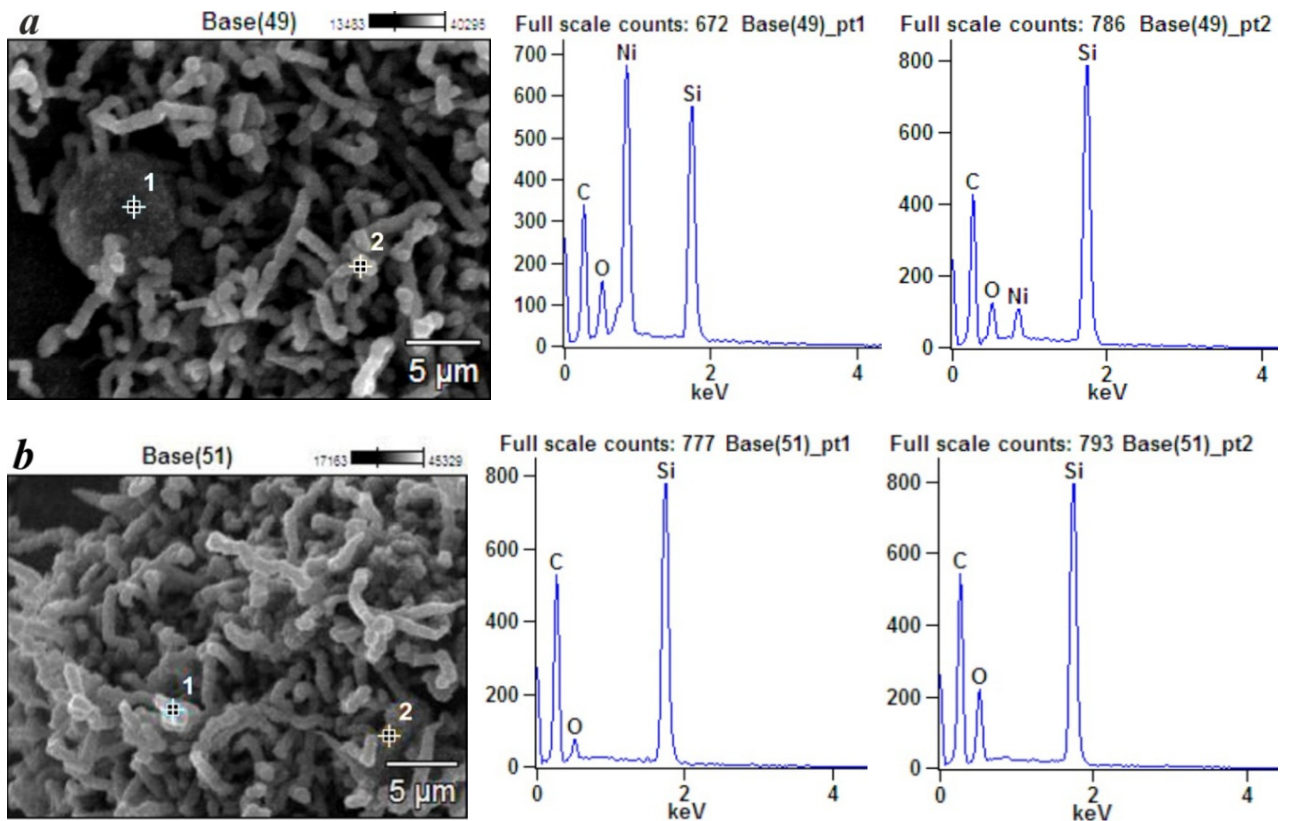


Figure 3 – Energy dispersive X-Ray spectra of samples synthesized at a substrate temperature of 600°C: *a* - Ni/Si₍₁₀₀₎; *b* - Ni/PSSi₍₁₀₀₎

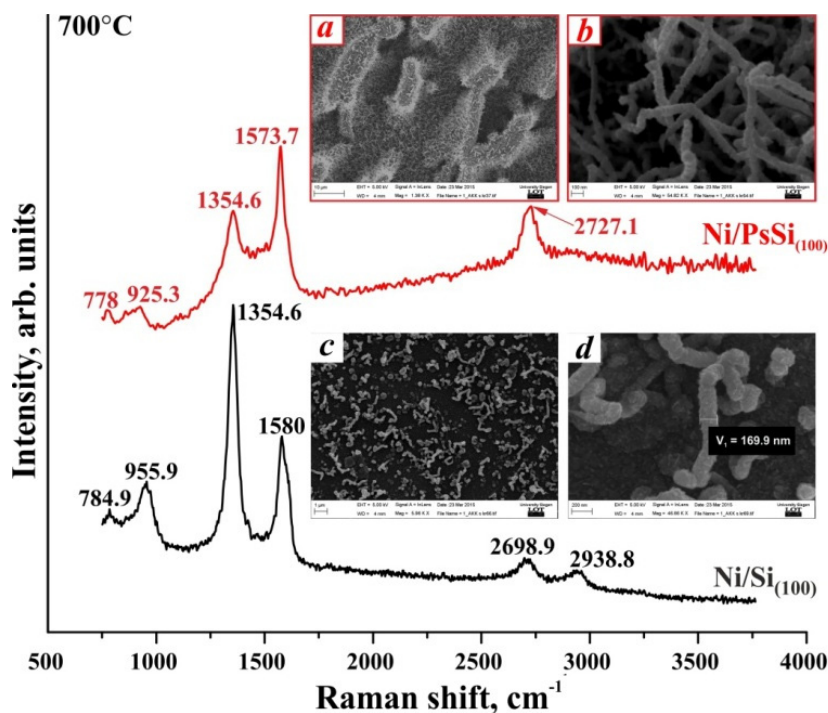


Figure 4 – Raman spectra and SEM images of samples synthesized at a substrate temperature of 700°C: *a, b* - Ni/PsSi₍₁₀₀₎; *c, d* - Ni/Si₍₁₀₀₎

Figure 4 shows Raman spectra and SEM images of samples obtained at a substrate temperature of 700°C. One can see that, silicon carbide groups are also shifted to the low-frequency region of 771.2, 785 and 925.3, 955.9 cm⁻¹. The main carbon peaks are located within the limits of 1355-1358 and 1554.6-1580 cm⁻¹. Peaks of the second order in the region of 2699-2704.6 and 2960.9 cm⁻¹, which correspond to the groups 2D and D + G are present in the high-frequency region of the spectrum. It can be assumed from intensity of D peak that nanostructures obtained on porous silicon substrate are less defective than nanostructures on polished substrates.

Figure 4 also shows SEM images of samples obtained at a substrate temperature of 700°C. As can be seen in Figure 4d, the synthesized NS have an average diameter of 100-170 nm. Figure 4b confirms that the growth of NS on PS surface is more massive than on polished Si.

Energy dispersive X-Ray spectra (Figure 5) show that the ratio of carbon and silicon concentrations in NS is on polished Si ~ 0.5-0.7 and on PS ~ 0.3-0.5.

The following samples are obtained at a substrate temperature of 800°C. The spectra also contain carbon nanostructures of silicon carbide,

TO and LO groups have shifted to 771.2-788.4 and 928.7-970.1 cm⁻¹. The main carbon peaks D and G are observed in the region of 1354.7-1364.4 and 1564.2-1586.4 cm⁻¹. The second-order 2D group is located in the region of 2704.6 and 2721.5 cm⁻¹, while D + G peaks are observed a 2941.6 and at 900.2 cm⁻¹. It can be judged by the intensity of 2D and D peaks, that samples are rather crystalline.

SEM image (Figure 6a) shows that islets of silicon carbide nanostructures are formed in the process of growth. It can be noted that the massive growth of nanostructures occurs on both substrates. Figure 6b and 6d show that structures formed on polished substrates have a smaller diameter. The energy-dispersive X-Ray spectrum (Figure 7) shows that carbon content in NS for both substrates is ~ 0.6-0.7.

As in all samples, shifted silicon carbide peaks are present and the position of the peak of transverse mode has not changed. Peaks within 1426 cm⁻¹, which belong to CH₃ groups [23] can be noted. Peaks D and G are located in the area of 1348.1-1364.4 and 1580 cm⁻¹. There are groups of the second order 2D and D + G within the limits of 2701.7-2729.9 and 2897.4-2941.6 cm⁻¹ on both samples. The second sample contains additional

peaks in the region of 2438 and 3252.6 cm^{-1} . The first peak corresponds to the group D' , which is the characteristic group of graphene and allows us to

speak about its presence [24]. Whereas, the second one speaks about the presence of C-H bonds in the samples [25].

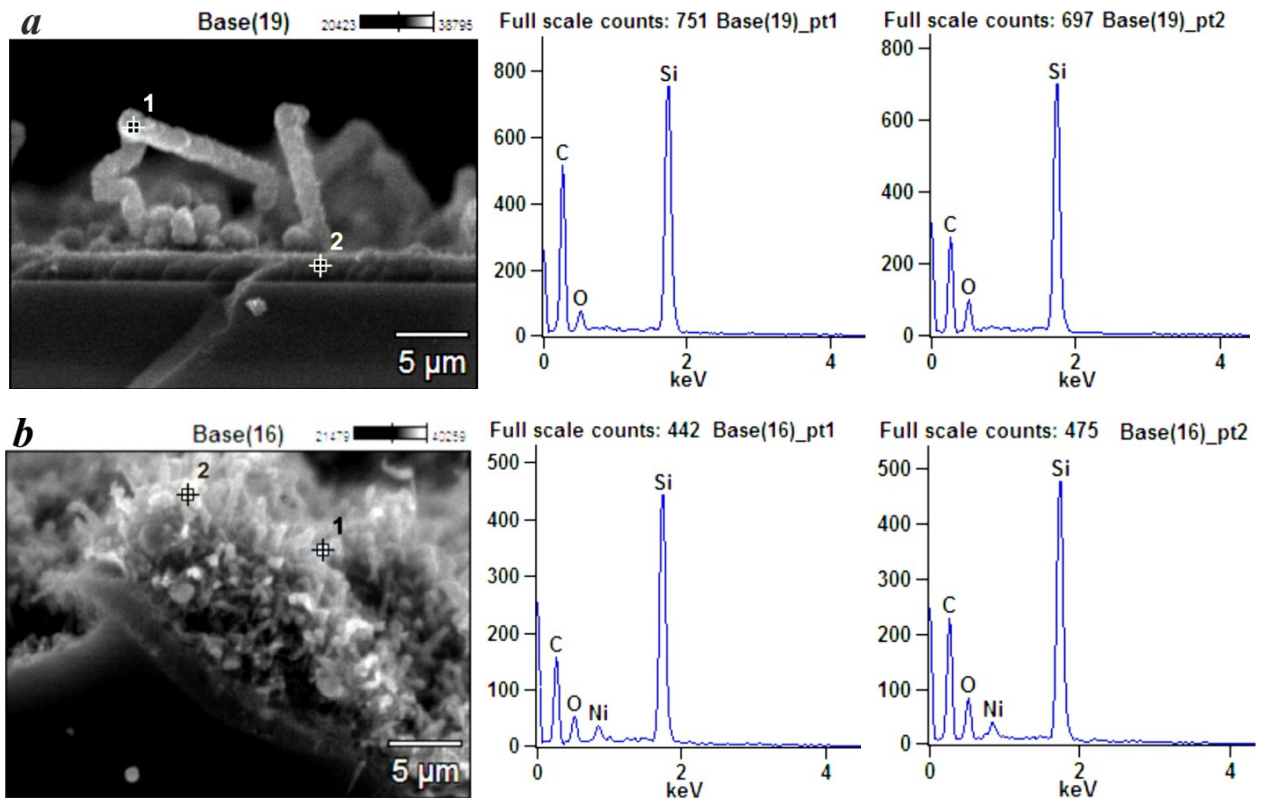


Figure 5 – Energy dispersive X-Ray spectra of samples synthesized at a substrate temperature of 700°C: *a* - Ni/Si₍₁₀₀₎; *b* - Ni/PSSi₍₁₀₀₎

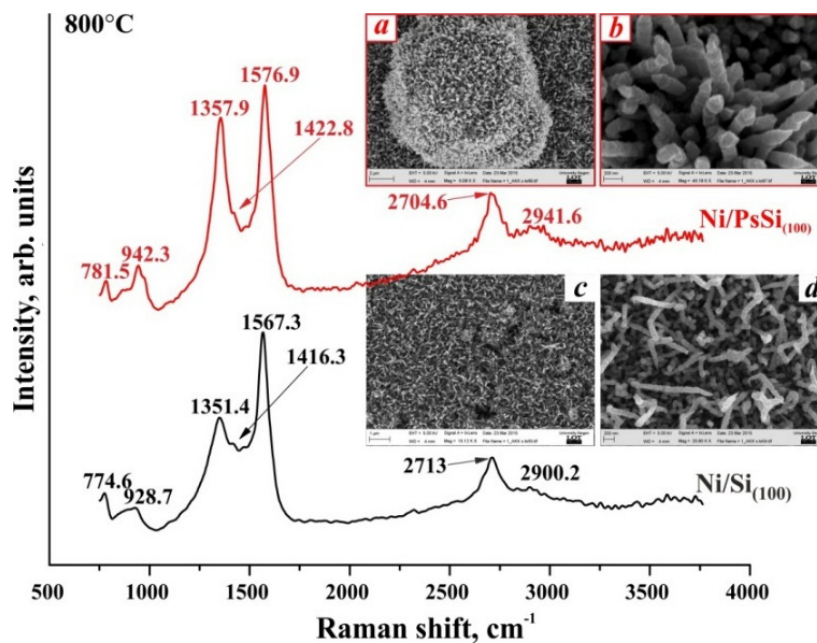


Figure 6 – Raman spectra and SEM images of samples synthesized at a substrate temperature of 800°C: *a*, *b* - Ni/PSSi₍₁₀₀₎; *c*, *d* - Ni/Si₍₁₀₀₎

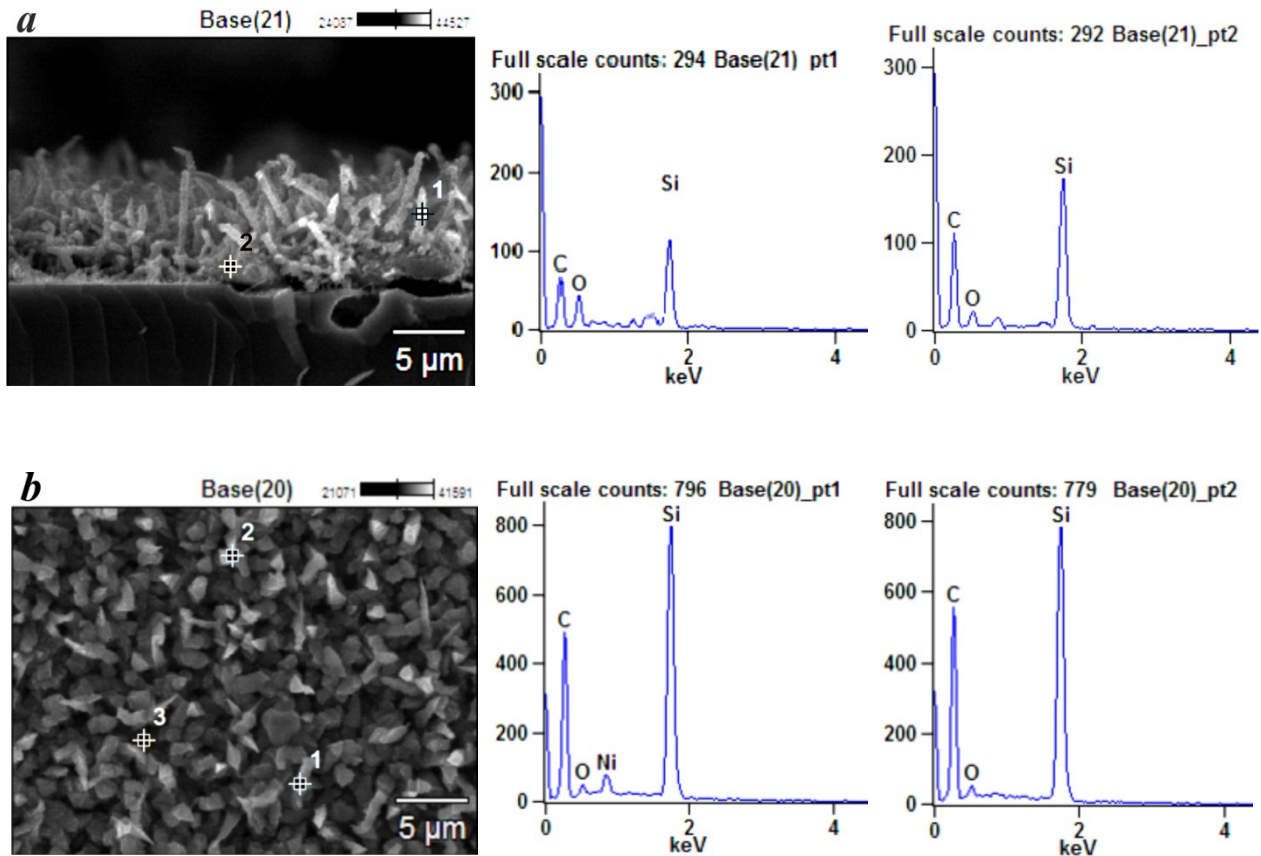


Figure 7 – Energy dispersive X-Ray spectra of samples synthesized at a substrate temperature of 800°C: *a* - Ni/Si₍₁₀₀₎; *b* - Ni/PSSi₍₁₀₀₎

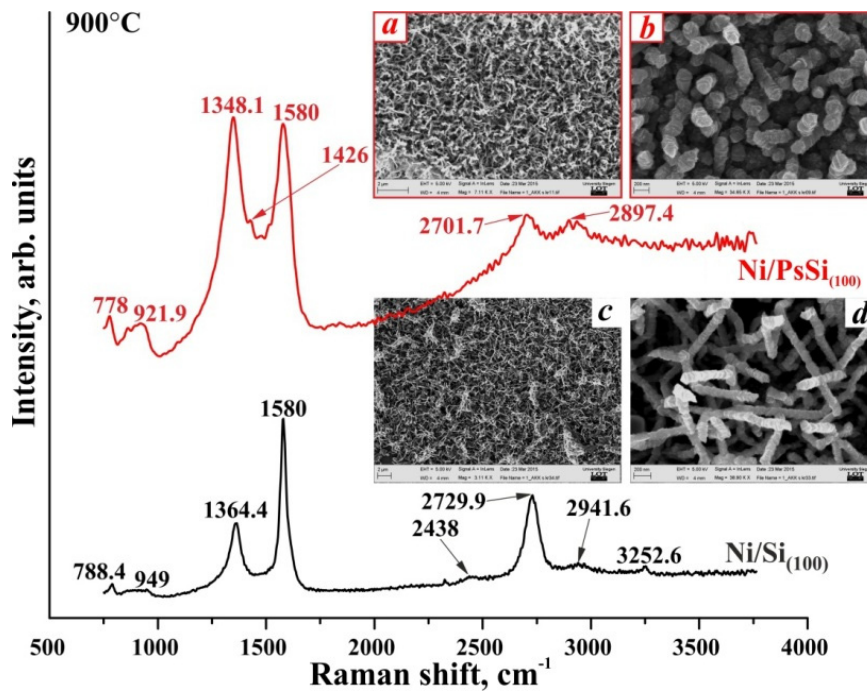


Figure 8 – Raman spectra and SEM images of samples synthesized at a substrate temperature of 900°C: *a*, *b* - Ni/PSSi₍₁₀₀₎; *c*, *d* - Ni/Si₍₁₀₀₎

As can be seen in Figure 8b and 8d, the formed NS are quite straight and grows oriented, which, presumably, can be influenced by the orientation of the substrate. In addition, it can be seen that NS formed on PS have an average diameter of 200 nm and, as in the previous experiments, a rough surface.

The diameter of nanostructures on polished substrates is half as much, as can be seen from Figure 8d. It can be noted that growth of NS is massive on both polished Si and PS surfaces. The energy-dispersive X-Ray spectra in Figure 9 indicate that carbon content in NS on both substrates is up to ~ 0.5 .

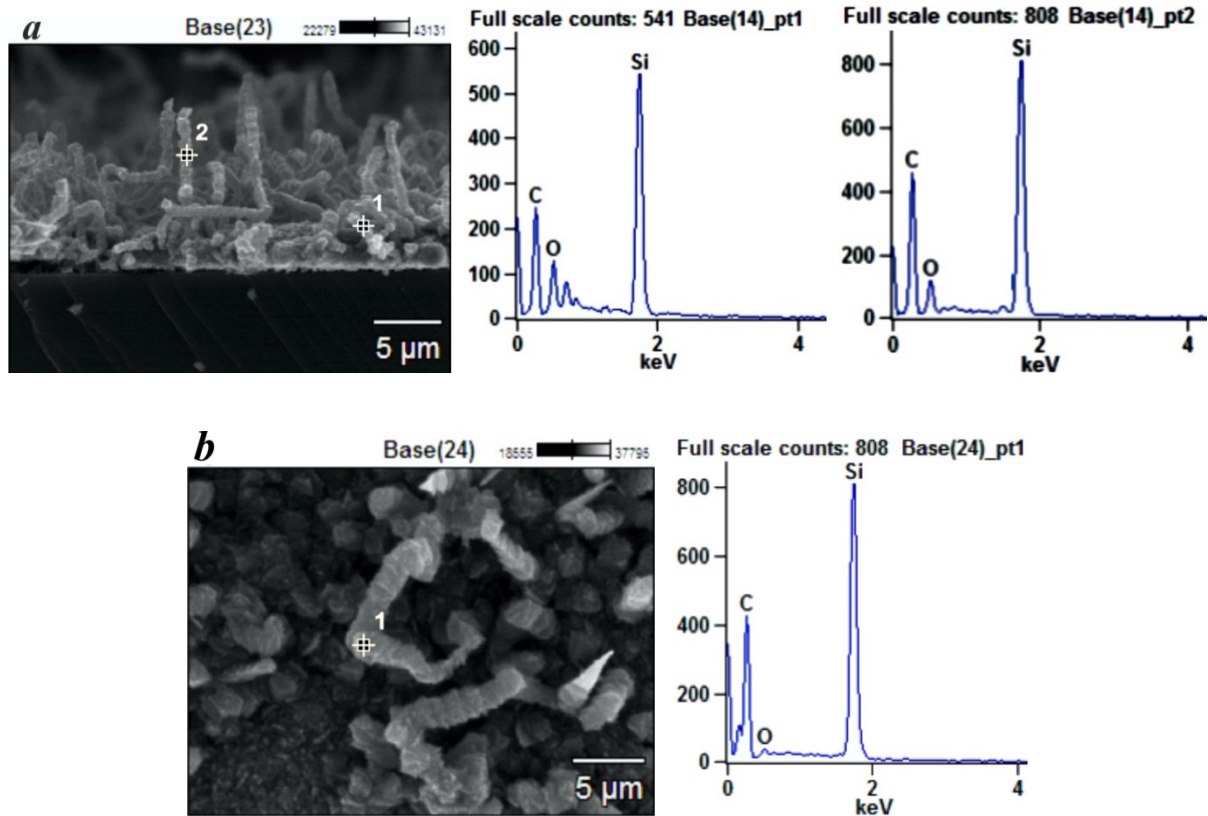


Figure 9 – Energy dispersive X-Ray spectra of samples synthesized at a substrate temperature of 900°C: *a* - Ni/Si₍₁₀₀₎; *b* - Ni/PSSi₍₁₀₀₎

3.2 NS synthesized on substrates Ni/PSSi₍₁₁₁₎ and Ni/Si₍₁₁₁₎

Figure 10 shows Raman spectra of some samples obtained on nickel films deposited on the surface of polished and porous silicon (111) (Ni/Si₍₁₁₁₎, Ni/PSSi₍₁₁₁₎).

Further, spectra of carbon nanostructures obtained at different temperatures on the surface of polished silicon substrates with the orientation [111] were considered. Figure 10a shows spectra of nanostructures on polished substrates. The first sample contains the main carbon peaks in the region of 1348.1 and 1596 cm⁻¹. One can note the shift of G

peak to the high-frequency region ~ 1600 cm⁻¹. In addition to these, displaced silicon carbide groups at 784.9 and 945.7 cm⁻¹ can be observed. The peak located within 1416.3 cm⁻¹ corresponds to the CH₃ groups [23]. The peak in the region of 2900 cm⁻¹ corresponds to 2D zone and associated with the boundary point K in the Brillouin zone of graphite, which depends on the packing in three-dimensional space. These lines are associated with resonance scattering of light and the participation of two phonons with the same energy but opposite direction of the pulse and give information on the ordering of graphite (graphene) layers [26].

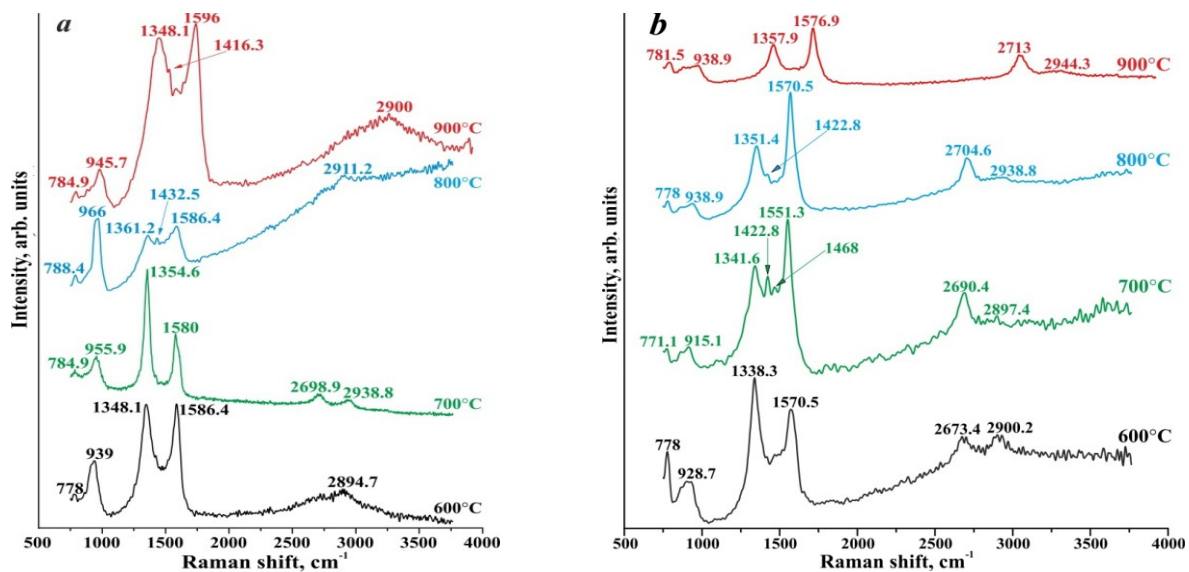


Figure 10 – Raman spectra of samples synthesized on substrates: a – Ni/Si(111), b – Ni/PSSi(111)

In the next sample, silicon carbide peaks are not strongly shifted and the most intense than in the remaining samples. One can notice the broadening of the peaks, which may be due to the amorphous structure. The proof of this is the displacement of *D* and *G* peaks to the high-frequency region: 1361.2 and 1586.4 cm⁻¹ and the presence of *D* + *G* peak in the region of 2911.2 cm⁻¹. There is also a peak corresponding to CH₃ groups.

There are shifted silicon carbide peaks in the sample obtained at 700°C. But you can see that position of peak of the transverse mode has not changed. *D* and *G* peaks are located in the region of 1348.1 and 1586.4 cm⁻¹. The sample is defective, since the intensity of *D* peak is very high and there is *D* + *G* peak at 2894.7 cm⁻¹.

The main carbon groups "merged" into one peak in the last sample. This can happen because of close location of peaks. There are 3 peaks located at 1367.7, 1442.2 and 1551.3 cm⁻¹, which correspond to the groups *D*, CH₃ and *G*. As in the remaining samples, there are peaks of carbon nanostructures of silicon carbide. *D* + *G* peak is located in the region of 2897.4 cm⁻¹.

In the spectra of Figure 10b, peaks of silicon carbide nanostructures are present in the range from 771.1 to 781.5 cm⁻¹. The position of the peak of the transverse mode does not change and lies in the region of 778 cm⁻¹ in the samples obtained at 600 and 800°C. The peaks corresponding to the longitudinal mode are observed in the range from 915.1 to 938.9

cm⁻¹. The position of the peak of the longitudinal mode does not change in the first two samples. *D* peak shifts with a decrease in temperature to the low-frequency region, and its intensity also increases. This indicates an increase in the defectiveness of structures. *G* peak is located at 1570.5 cm⁻¹ in the samples obtained at 600 and 800°C. It is shifted to the low-frequency region 1551.3 cm⁻¹ in a sample synthesized at 700°C, and shifted to the high-frequency region of 1576.9 cm⁻¹ in a sample obtained at 900°C. 2*D* peak is observed at 2673.4 - 2713 cm⁻¹. Also there is *D* + *G* peak, which is within the range of 2897.4 - 2944.3 cm⁻¹ in all spectra. All obtained carbon nanostructures are carbon nanotubes. Also there are additional peaks at 700 and 800°C in the region of 1422.8 and 1468 cm⁻¹, which appear due to the presence of CH₃ groups [23].

Figure 11 shows results of studies of samples synthesized on nickel films deposited on the surface of polished silicon (111) by SEM. SEM images of the formed NS show that they have a rough surface. It can be seen that the growth of NS becomes more massive and oriented with increasing temperature. In addition, as can be seen from the figures, the temperature increase also helps to straighten the fibers. It can be seen from Figure 11g, h that fibers are curved at a certain angle, which may indicate the influence of the orientation of the substrate on their growth. Figure 12 shows SEM images of samples synthesized on nickel films deposited on the surface of porous silicon (111).

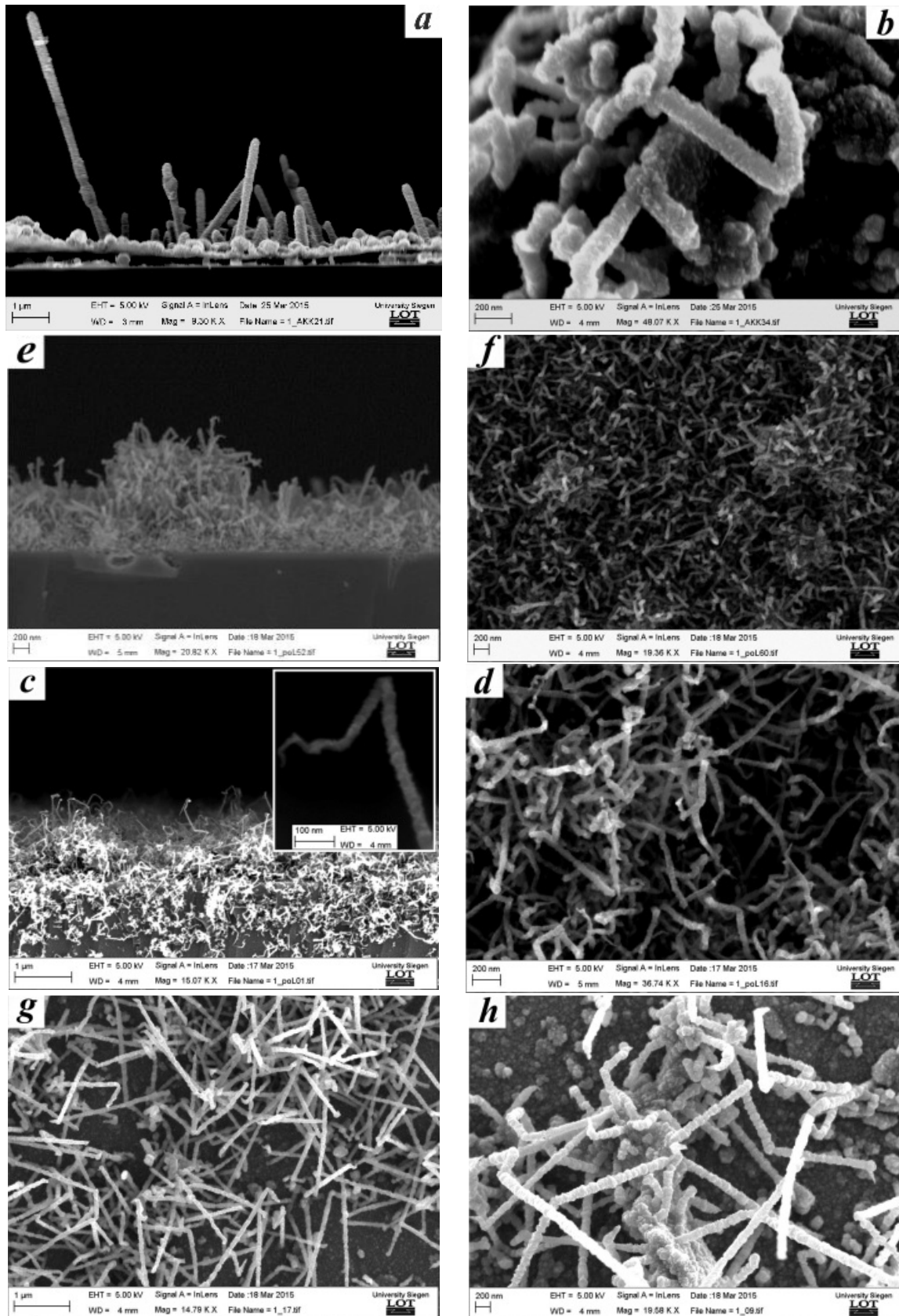


Figure 11 – SEM images of samples synthesized on Ni/Si₍₁₁₁₎ at different substrate temperatures: *a, b* - 600°C; *c, d* - 700°C; *e, f* - 800°C; *g, h* - 900°C

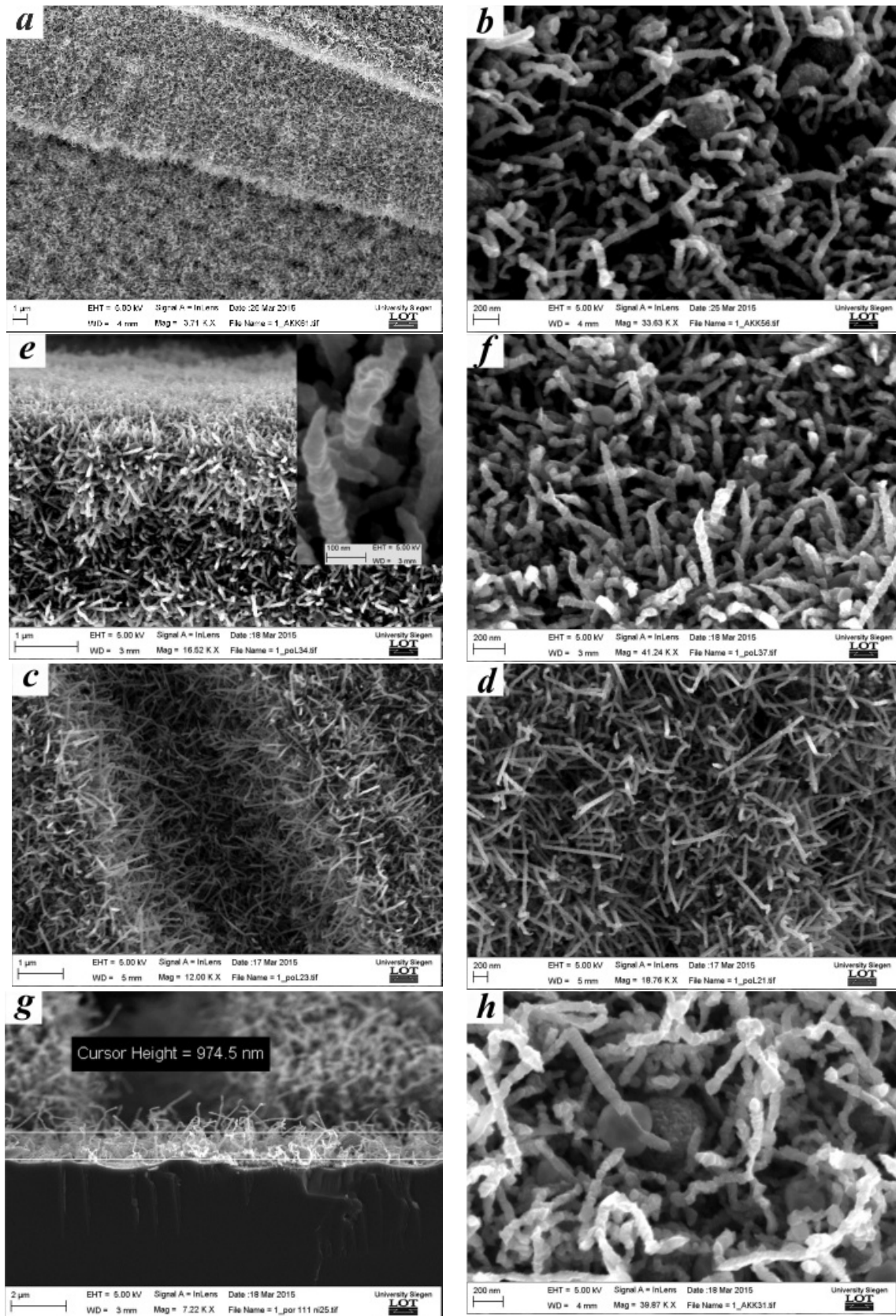


Figure 12 – SEM images of samples synthesized on Ni/PSSi₍₁₁₁₎ at different substrate temperatures: *a, b* - 600°C; *c, d* - 700°C; *e, f* - 800°C; *g, h* - 900°C

It can be noted that growth of NS on porous silicon substrates is more massive in comparison with polished substrates. The samples obtained at 700°C grow in oriented manner and are quite straight. In comparison with Figure 11, the dependence of NS growth on temperature is not traced in this series of experiments. Figure 12g show that the height of synthesized NS is 974.5 nm. According to figures 11c and 12e, the obtained structures have a diameter of ~ 100 nm.

4 Conclusions

Experiments on synthesis of SiC and C nanostructures on Ni buffer layer by the method of microwave plasma assisted chemical vapor deposition were carried out.

Studies by scanning electron microscopy have shown that the formed nanostructures have a diameter of 100-170 nm and a rough surface. Analysis of results of scanning electron microscopy of samples shows that the growth of nanostructures on the surface of porous silicon is more massive than on polished Si. Energy dispersive X-Ray spectra indicate that carbon content in NS varies from ~ 40 to ~ 80%.

The results of studies by Raman scattering confirmed that there are carbon nanostructures of silicon carbide with 3C-SiC polytype structure. It was determined that the most optimal temperature for obtaining nanostructures is 800°C. It is also possible to note the presence of the main carbon peaks in the range of ~1300 and ~1500 cm^{-1} , which correspond to carbon nanostructures on both types of substrates.

For a more detailed analysis of the structure of the produced NS, it is necessary to conduct additional studies using transmission electron microscopy and diffraction of electrons, as well as X-Ray diffraction analysis.

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