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Synthesis of carbon nanowalls by oxy-acetylene torch method

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Abstract

This work presents a relatively new method for the synthesis of carbon nanowalls (CNWs) based on oxy-acetylene torch as a function of deposition time. The morphological and structural properties of the obtained CNW films were studied by scanning electron microscopy and Raman spectroscopy. Changes in the morphology and structural properties of the CNW films depending on the synthesis time were revealed. Shorter growth times lead to the formation of thinner CNW films with a dense labyrinth-like structure, while longer growth times lead to thicker CNW films with a petal-like structure. In addition, this study opens up the possibility of synthesizing CNWs on a production scale, since the proposed method is relatively environmentally friendly and efficient from an economical point of view.

1. Introduction

Nowadays nanomaterials and nanotechnologies play an increasingly important role in many key sectors of human activity in the new millennium [1]. The analysis of the current state and tendencies of nanotechnology development allows concluding that nanostructured carbon materials are very promising objects for practical application in different branches of industry [2-5]. Commonly known carbon nanomaterials, such as graphene [6], fullerene [7], nanodiamonds [8], carbon nanotubes (CNTs) [9], carbon nanofibers (CNFs) [10] carbon nanowalls (CNWs) [11], etc., have already been found their practical applications in medicine [12,13], supercapacitors [14-16], tissue products [17], etc.

One of the interesting and attractive carbon nanomaterials are CNWs, which are three-dimensional material with vertically oriented graphene sheets. The shape of CNWs is similar to a labyrinth-like structure with layer thicknesses ranging from a few to tens nanometers [11,18,19]. Due to their unique structure and morphology, CNWs exhibit excellent physical and chemical properties along with stability, chemical inertness, electrical conductivity, and a huge surface area to mass ratio [20-23]. Owing to attractive properties, CNWs have been used in recent years to create mass sensors [24], supercapacitors [25-27], lithium-ion batteries [28,29], electrochemical sensors [30,31], fuel cells [32], solar cells [33,34] energy storage, [35,36], memory devices [37], etc.

Given the relevance of the practical application of CNWs, the development of environmentally friendly, energy and cost-efficient methods of synthesis is one of the urgent tasks. There are two main methods of synthesis of CNWs: hot wire deposition (HWCVD) and plasma-enhanced chemical vapor deposition (PECVD). In the first method, CNWs are synthesized by heating the filament (1400°C to 2200°C), through which a flow of inert and carbon-containing gas is passed, and the substrate itself is additionally heated (500°C to 700°C) [38,39]. The second method is widely used for the synthesis of CNWs. The carbon-containing gas is partially atomized and ionized in the plasma environment, and the obtained radicals are condensed on the substrate surface. There are various methods for plasma activation of PECVD [40,41], such as microwave discharge plasma (MPECVD) [42-44], glow discharge plasma (DC-PECVD) [45,46], high frequency (RF-PECVD) capacitively coupled plasma (CCP) [47,48], ICP-PECVD inductively coupled plasma (ICP) [49,50], radical injection (RI-PECVD) [51,52], electron-cyclotron resonance (ECR-PECVD) [55] etc. The mentioned methods have their advantages and disadvantages, for example, the disadvantages include: low growth rate, the use of complex vacuum systems, high energy consumption for the heating system and plasma activation, and high cost of the equipment [54]. Considering the above, the development and optimization of the existing technology for CNWs synthesis is considered to be one of the weighty tasks.

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One of the simplest, but most effective methods of obtaining carbon nanomaterials includes the method of oxy-acetylene torch, in which the deposition is conducted at atmospheric pressure, i.e., no complex vacuum and electronic apparatus are required [55]. This technology has several advantages over other methods, such as high synthesis rate, simplicity, and low cost of the equipment used [56,57]. In addition, the synthesis does not require expensive gases, including high purity ones. This method has the potential for continuous production in large scales at reduced costs compared to other methods [58-60]. To date, in the scientific literature, works on the synthesis of CNTs, CNFs, and diamond-like carbons by the oxy-acetylene torch method are presented [55,57,61]. The major difference between fabrication of CNWs and other carbon nanomaterials by the oxy-acetylene torch method is in the synthesis parameters. For example, paper [55] reports the successful deposition of CNFs on mild steel substrate (0.23% C, 0.34% Si, 0.44% Mn, 0.007% S, 0.016% P and balanced Fe) using the oxy-acetylene torch. Where the gas flow rate was adjusted so that the flame length was about 70 mm and the distance from the nozzle tip to the sample surface was about 40 mm, so that the substrate was exposed only to the middle flame zone. H. Oulanti *et al*. obtained multi-walled CNTs directly on CNFs using the oxy-acetylene method [57]. In this work, ferrocene was used as a catalyst for the growth of CNTs and was sprayed on the surface of the fibers. The synthesis parameters were an acetylene to oxygen ratio of 1.3 to 1.45, and the distance between the substrate and the flame torch was 6 mm to 10 mm. In the paper, [61] polycrystalline diamond films were grown on AISI 304 stainless steel substrate by using the oxy-acetylene torch. In this work, the gas flow rate was adjusted so that the distance from the nozzle tip to the substrate was 50 mm to 60 mm. The samples were then processed in the outer zone of the flame and then the synthesis was performed in the middle zone of the flame. The works on the synthesis of CNWs by the oxy-acetylene torch method are almost few. An example of such work is the work of L. J. Guo *et al*., [58] in which CNWs were synthesized on the surface of tungsten carbide by oxy-acetylene torch method, where CNWs grew vertically on the surface of spherical carbon particles. Due to this situation, the oxy-acetylene torch synthesis method requires additional research and optimization of the synthesis process to obtain quality CNWs. In view of the above, the synthesis of CNWs by the oxy-acetylene torch method was investigated in this work, and the effect of the synthesis duration on the structure formation was studied. It was found that the duration of synthesis has a significant effect on the morphological and structural properties of CNWs films.

The novelty of this work compared with similar oxy-acetylene torch methods is the synthesis of CNWs on the surface of silicon, where copper was pre-sprayed, as well as the use of dual-burner systems, which allows to cover more areas of the substrate.

2. Experimental

The scheme of the experimental setup is shown in Figure 1. The setup consists of two oxy-acetylene torches, a water-cooled substrate holder, and two gas mass flow meters. The mechanism of the equipment allows to change the vertical distance between the sample surface and the flame, as well as the angle of the flame front inclination in the range from 0° to 90°.

First, silicon samples of BPS-20 type (boron-doped p-type silicon, manufacturer SW GmbH, Germany) were chemically cleaned in an ultrasonic bath in a mixture of ammonia hydrate (NH_4OH), hydrogen peroxide (H_2O_2), and distilled water in a 1:1:6.5 volume ratio. After cleaning, copper films were deposited on the silicon wafer surface using a magnetron sputtering method. The deposition was carried out in a flow 6 sccm of Ar working gas at a pressure of 10^{-2} Torr. The voltage and current on the anode target were 700 V and 35 mA, respectively. The deposition time was 10 min. Figure 2 shows a SEM image of the cross-section of a thin copper film on a silicon substrate, the thickness of the copper film is about 450 nm.

The next stage consisted in a series of experiments on a silicon wafer with a thin copper film using the oxy-acetylene torch method, in which the synthesis parameters were as follows: burner nozzle diameter – 1.0 mm, flame front angle $\alpha = 90°$, distance from burner nozzle to substrate $h = 10$ mm, oxygen/acetylene concentration ratio $O_2/C_2H_2 = 0.70$, oxygen flow rate 0.70 slpm and acetylene flow rate 1 slpm, the synthesis duration varied from 5 min to 20 min with an increment of 5 min.

To characterize the surface morphology and structural quality of the obtained samples, they were examined by Quanta 3D 200i scanning electron microscopy (SEM) and NT-MDT Solver Spectrum Raman spectrometer (laser wavelength $\lambda = 473$ nm).

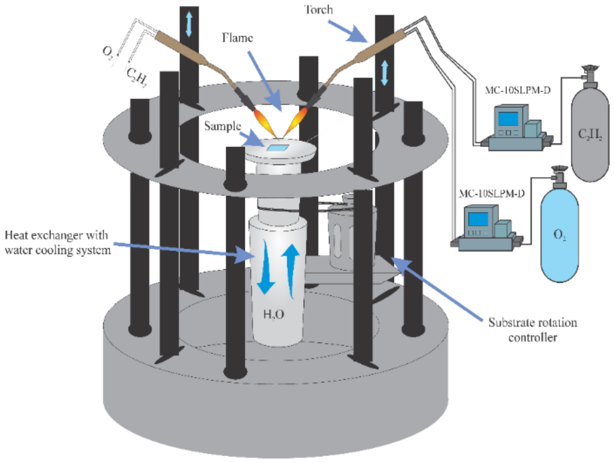

Figure 1. Scheme of oxy-acetylene torch.

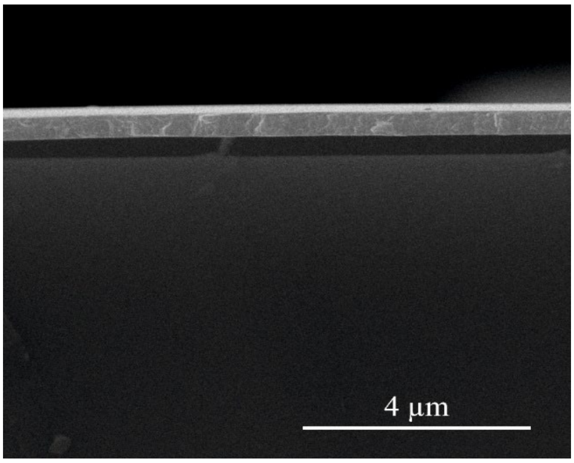

Figure 2. SEM image of cross-section of thin copper film on silicon substrate

3. Results and discussion

Figure 3 shows SEM images of CNWs synthesized at different time by the oxy-acetylene torch method. SEM images show that with increasing synthesis time, the wall thickness increases. CNWs synthesized at 5 min have a non-uniform morphology and corresponding average wall thickness is 25.9 nm. When the synthesis time is 10 min, the obtained carbon walls have a labyrinth-like shape and the average wall thickness is 29.3 nm, and at 15 min and 20 min the obtained CNWs films have a petal-like shape and the wall thickness is 39.8 nm and 44.3 nm, respectively [62] (From data of Figure 3(g)). Figure 3(e,f) shows the cross section of CNWs obtained at 5 min and 10 min. From these figures the growth rate of CNW was determined – 1.2 $\mu m \cdot min^{-1}$. The obtained samples of nanowalls at synthesis time of 15 min, 20 min are similar to the samples obtained by HWCVD method [63]. These results are consistent with the works where CNWs were synthesized at different times [21,64-66]. As can be seen from the number of walls, CNWs with a wall thickness of about 40 nm are predominantly formed in all samples. The diagram shows the thickening of the walls with increasing synthesis time (Figure 3(g)). Figure 3(h) shows the average wall spacing of the CNW samples, which were 270, 107, 308, and 263 nm for 5, 10, 15, and 20 min, respectively.

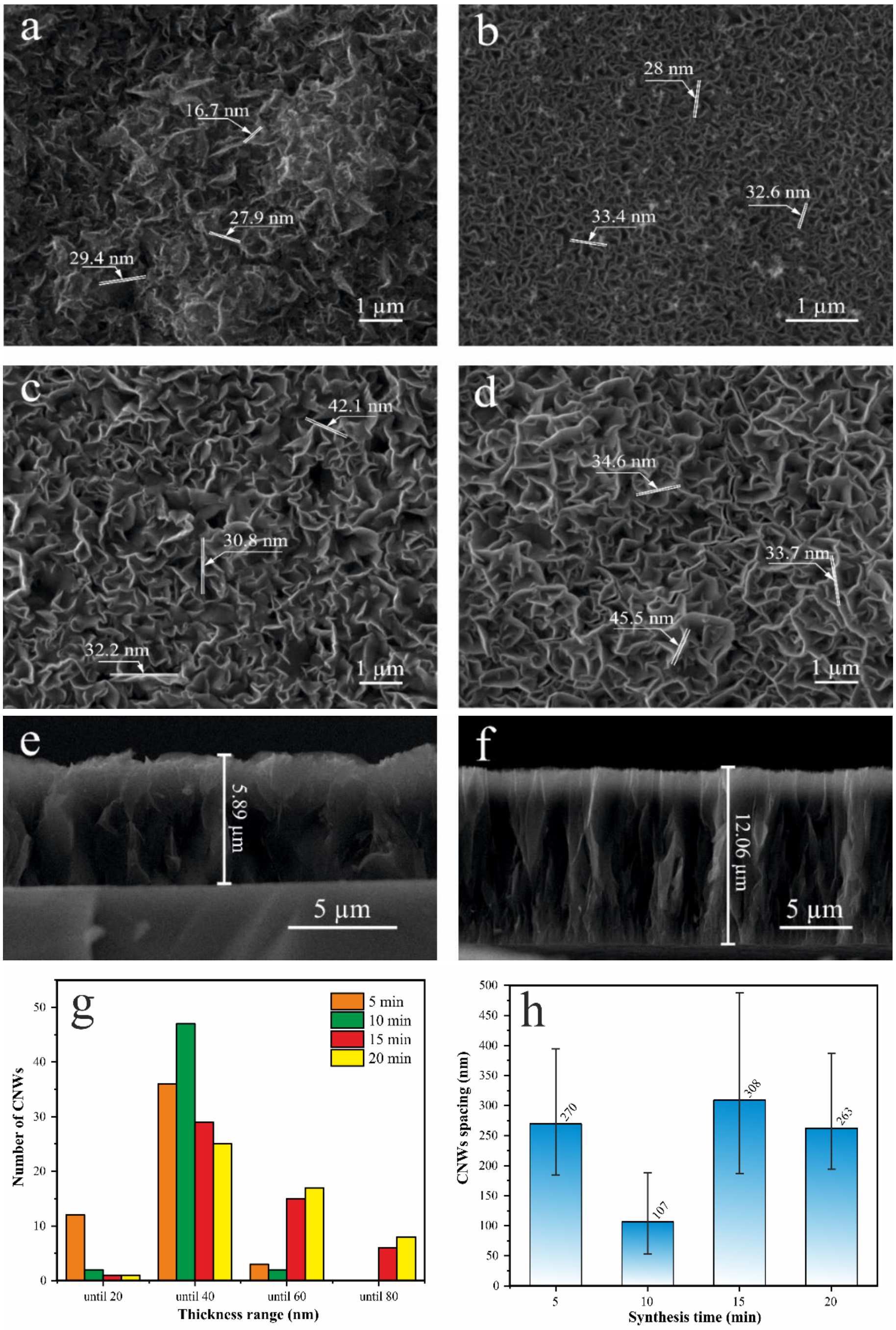

Figure 3. SEM images of CNWs synthesized at different time: (a) 5 min, (b) 10 min, (c) 15 min, (d) 20 min, cross-section- view SEM images of CNWs synthesized at (e) 5 min, (f) 10 min, and (g) thickness of CNWs, and (h) CNWs spacing.

Figure 4 shows an analysis of Raman spectra of CNWs synthesized by the oxy-acetylene torch method. From Figure 4(a) it is seen that the samples demonstrate pronounced Raman bands G, D, 2D, and D + G. The D band is induced by lattice defects [67], and the G band is a tangential mode of vibration in crystalline graphite [68]. There is also a small shoulder peak, called D', with much lower intensity than the G peak. The D' band corresponds to the symmetry breaking of the finite size of the sp^2 crystal units and is typical for graphene edges [66]. A second-order 2D band and a combination of the D + G peaks are also observed. The 2D peak is an indication of the graphitic structure content in carbon materials [69]. As the synthesis time increases, the relative intensity of the G peak decreases. The increase of intensity ratio I_D/I_G as a function of the synthesis time indicates the growth of structural defects which is very likely in the case of intersecting graphene sheets (see Figure 4(b)) [70]. The growth trend in the I_D/I_G peak intensity ratio depending on the synthesis time can also be related to the change in the morphology of CNWs from a labyrinth-like to petal-like. In the paper of Kurita *et al.*, [71] it is reported a linear increase in the I_D/I_G intensity ratio with decreasing CNWs size. Using the intensity ratio of the I_D/I_G peaks, the crystallite size L_a was estimated according to the Tuinstra-Koenig Relation formula: [72].

$$L_a = \frac{2,4 \times 10^{-10} \times \lambda^4}{I_D/I_G} \qquad (1)$$

where L_a is the crystallite size, λ is the laser wavelength, I_D/I_G is the ratio of the D and G peak intensities. Figure 4(b) shows the decrease of L_a as a function of the synthesis time. Thus, the increase in the D peak and decrease in the G peak can also be attributed to the decrease in crystallite size. An important parameter to characterize the structure of graphite-like carbon materials is the full width at half maximum (FWHM) of the G peak. It is observed that increasing synthesis time of the CNW film leads to a decrease in the FWHM G as shown in Figure 4(b), indicating a lower defect density. This is an indication of the increasing degree of crystallinity of the graphite-like sheets of the obtained CNW films. The data of the analysis of the Raman spectra of the CNW films are shown in Table 1.

Table 1 shows that all samples show major carbon peaks D and G. The values of D and G peaks are almost the same. In addition to them, the second order groups D′, 2D and D + G are also presented. The presence of these peaks indicates the presence of graphene. Also from Table 1 we can see that with increasing synthesis time the crystallite size of L_a and FWHM G decreases and the ratio of I_D/I_G intensities increases. According to these data, it can be assumed that the obtained structures are CNWs.

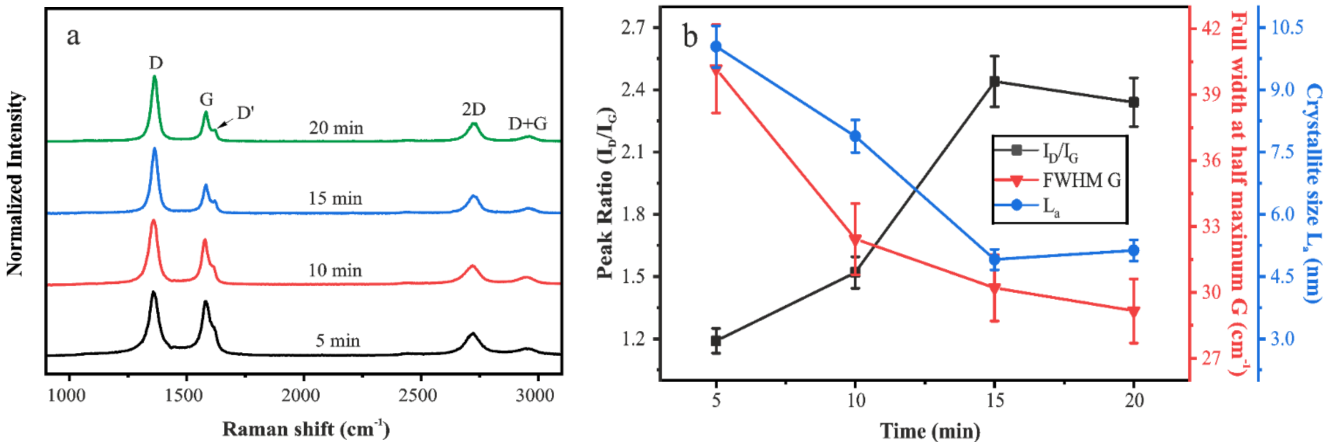

Figure 4. Raman spectroscopy of CNWs: (a) Raman spectra of CNWs synthesized at different time durations, (b) dependences of I_D/I_G, FWHM of G peak, and crystallite sized L_a on synthesis time.

Table 1. Data of Raman spectroscopy analysis

	5 min	**10 min**	**15 min**	**20 min**
G peak position (cm^{-1})	1582.15	1577.60	1581.98	1581.75
D peak position (cm^{-1})	1360.24	1357.92	1363.13	1363.08
D' peak position (cm^{-1})	1617.17	1612.53	1619.18	1619.07
2D peak position (cm^{-1})	2719.48	2716.79	2723.71	2723.76
D + G peak position (cm^{-1})	2947.71	2947.71	2955.95	2961.44
FWHM G (cm^{-1})	40.18	32.43	30.21	29.16
FWHM D (cm^{-1})	47.20	42.14	32.73	32.23
FWHM D’ (cm^{-1})	24.70	21.46	18.75	18.50
I_D/I_G	1.19	1.52	2.44	2.34
I_{2D}/I_G	0.40	0.41	0.65	0.64
Crystallite Size L_a (nm)	10.04	7.88	4.91	5.13

4. Conclusions

In this work, the synthesis of carbon nanowalls by oxy-acetylene torch method is presented and the dependence of their structural properties on synthesis time is studied. The results of SEM analysis indicate changes in the morphology of CNWs from labyrinth-like to petal-like depending on the duration of synthesis. Analysis of Raman spectra of the samples showed that the obtained materials are CNWs with typical Raman band positions. The FWHM of the G peak decreases with increasing synthesis time despite the fact that I_D/I_G ratio increases sufficiently. These dependencies indicate that the synthesized CNWs consist of small crystallites with a high degree of graphitization. The obtained results open a promising path for the synthesis of CNWs on an industrial scale since the proposed method can be considered relatively ecologically friendly and economically efficient.

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