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PAPER

Low-temperature synthesis of carbon nanotubes on iron nanopowders

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Abstract

This work presents the results of experiments on synthesis of carbon nanostructures by the method of thermal chemical vapor deposition (CVD) using iron nanopowders obtained by the method of electrical explosion of wires as catalysts. The process parameters that are optimal for low-temperature growth of carbon nanotubes (CNTs) have been identified during performed experiments. Results of Raman spectroscopy and x-ray analysis showed that samples grown at temperatures below the normally used have the highest crystallinity. Studies by scanning electron microscopy using SE2 mode and results of transmission electron microscopy indicate that the synthesized structures are multiwalled CNTs with the metal clusters inside the channel of the tube. The experimental modes of synthesis of CNTs by low-temperature CVD using iron nanopowders as catalyst have been found for the first time.

1. Introduction

Historically one of the first methods of obtaining carbon nanotubes (CNTs) was electric arc discharge method [1]. However, the existence of CNTs was reported earlier by Soviet scientists LV Radushkevich and V M Lukyanovich in 1952 [2]. These works have not attracted wide attention of the scientific community related to their publication only in Russian.

At present, other techniques such as laser ablation, synthesis in flames of hydrocarbons and chemical vapor deposition (CVD) have been successfully used for the production of carbon nanostructures [3-5]. However, arc discharge and various types of CVD are the most promising methods and are used in large-scale production of CNTs and carbon nanofibers (CNFs) [6].

Currently, catalytic CVD method is considered as the only cost-effective technology for large scale production of CNTs and their integration in order to be used in different devices [7]. It is known that multiwall CNTs are synthesized under the conditions of low-temperature CVD (600 °C-900 °C), while growth of singlewalled nanotubes predominates at higher temperatures (900 °C-1200 °C) [8]. Typically, metal particles (Ni, Co, Fe and Cu) with the size of a few nanometers are used as catalysts during CVD.

Despite the fact that these methods achieved a high level of quality, they are still far from complete control of important structural features, such as the length of the nanotubes, their diameter and chirality [9].

At present, several methods of preparation of metal nanoclusters are used for the synthesis of nanostructures (CNs). One of the promising methods of obtaining nanopowders (NPs) is electric explosion of wire (EEW)—a nonequilibrium process in which a conductor is dispersed under pulsed electric current and products of the explosion are mixed with the environment. Electroexplosive NPs have several advantages compared to NPs obtained by other ways: they are resistant to oxidation and sintering at room temperature, characterized by high chemical and diffusional activity during heating [10].

The experiments are usually conducted at high temperatures (700 °C–1200 °C) and pressures below atmospheric pressure in the thermal CVD method using iron catalysts. In this case, various precursors of hydrocarbons (methane, acetylene, benzene, etc) often in combination with inert gases as well as hydrogen, nitrogen, etc are used [11, 12].

In recent years, more and more attention of researchers attracted to the low-temperature modes of obtaining carbon nanostructures by CVD. The interest caused by several reasons. Firstly, the carbon containing source gases applied in the CVD growth processes (e.g., CH_4 , C_2H_2 , C_2H_4) are normally explosive. A process at lower temperatures is much safer. The low-temperature CVD process also simplifies remarkably the equipment and reduces consequently the cost. Secondly, the CVD processes are often accompanied by the coking. The process at lower temperatures mitigates the harmful deposition from the wall of a reaction chamber as well as gas channels. Finally and the most importantly, the growth at low temperatures is of great benefit to a lot of applications when the substrate material cannot withstand a high-temperature process. For example, to apply CNFs in semiconductor industry, the CVD process has to be compatible with the CMOS technology. It means the CVD temperatures must remain below $400\,^{\circ}C-450\,^{\circ}C$ to avoid mechanical deterioration [13].

Synthesis of CNs at low temperature CVD modes is carried out at different temperatures from 195 $^{\circ}$ C to 450 $^{\circ}$ C. The low-temperature CVD allows to synthesize various types of CNs, including carbon nanotubes, amorphous nanofibers, nanocoils, nanohelixs, nanosheets and branched CNs. A detailed overview of works on low-temperature synthesis of CNs is given in [13]. At present, the determined lowest temperature limit of CNFs synthesis on Fe nanoparticles is 350 $^{\circ}$ C [13, 14].

Two and three-component systems (Fe–Co, Fe–Ni and Fe–Co–Ni) are often used as a catalyst for obtaining multiwall CNTs. The lower limit of the synthesis is shifted to 400 °C in this case [13, 15–18]. However, the results of x-ray diffraction and Raman spectroscopy showed a very low crystallinity and order of the obtained samples. This is likely due to the polymeric nature of the synthesized structures that require additional high temperature thermal annealing for obtaining crystalline CNTs.

The aims of the study were determination of the lower temperature limit of the growth of carbon nanostructures on EEW Fe NPs and search for optimal conditions of low temperature (energetically favorable) synthesis using the most accessible hydrocarbons without expensive additives of inert and other gases.

2. Experimental

NPs were purchased in Tomsk Polytechnic University to be used as catalysts for the synthesis of carbon nanostructures. The procedure of obtaining NPs, experimental details and the results of studies on their morphology and structure are described in detail in works [19–21].

The joint studies on the structure and morphology of EEW NPs, their catalytic activity and the possibility of synthesis of CNs on them by thermal CVD were carried out at The Department of surface and technology of new materials at the Institute of Materials Engineering of the University of Siegen (Germany) [21, 22].

2.1. Investigation of morphology and structure of Fe NPs

2.1.1. SEM and TEM studies

Figure 1(a) shows SEM image and energy dispersive spectrum (EDX) of iron NPs. SEM images of Fe powders show that agglomeration of metal particles with a smaller size (from 10 to 50 nm) around the clusters with the size from 100 to 200 nm is present. Thus, the formation of chain-like structures from small clusters (10–30 nm) is observed. The shape of Fe particles is close to spherical. As seen from the EDX spectrum, small amounts of carbon and oxygen impurities are present in the composition of Fe powder. The presence of carbon impurities is due to the conditions of passivation procedure of powders.

Figure 1(b) presents the transmission electron microscope (TEM) image and histogram of the size distribution of Fe NPs. The results of TEM studies of iron NPs are consistent with SEM. TEM image shows that particles of iron NPs are spherical. The data of the histogram show that particles with the diameter of 40–70 nm predominate in the sample, the average diameter of which is equal to 65.5 nm. The analysis of histogram shows that distribution of iron nanoparticles according to the size is Gaussian with a standard deviation value $\sigma=30$ nm.

2.1.2. The results of x-ray analysis

Figure 2 shows the x-ray spectrum of EEW Fe nanopowders.

X-ray diffraction results show that the main phase in the composition of the studied samples is the thermodynamically stable crystal modification (space group symmetry Im3m) characteristic of massive state (Fe—PDF # 060696). However, radiographs detected asymmetry and peak splitting. Splitting of the peaks was observed in the region of the maxima—(200), (211) and (220). The decrease in the interplanar distances was

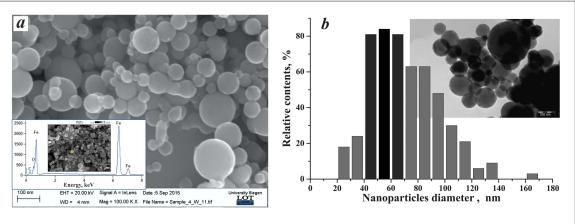
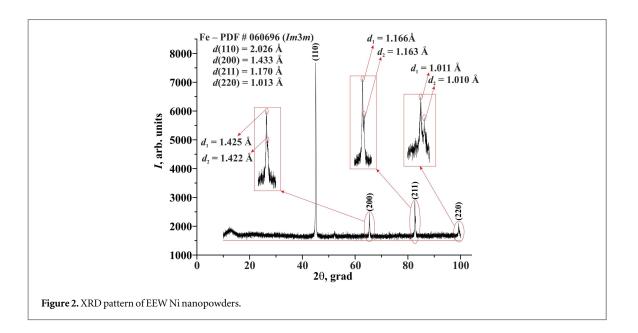


Figure 1. SEM image and EDX spectrum (a), TEM image and histogram of the size distribution (b) of Fe NPs.



revealed for all three planes ($\Delta'_{(200)} = 0.008$ Å, $\Delta''_{(200)} = 0.011$ Å, $\Delta'_{(211)} = 0.004$ Å, $\Delta''_{(211)} = 0.007$ Å, $\Delta'_{(220)} = 0.002$ Å, $\Delta''_{(220)} = 0.003$ Å). The obtained results of splitting of the peaks could show itself on account of size effects. In particular, in [23] it is related to the influence of the oxide layer.

Distortion of the lattice is possible in the course of formation at the metal/oxide interface due to the orienting influence of metal oxide lattice (the energies of crystal lattices differ by several times). However, in [24], splitting of peaks is attributed to the change in the lattice parameter of metal nanoclusters.

2.2. Synthesis of CNs

The growth of CNs was conducted in the volume of quartz reactor with an internal diameter of 90 mm placed inside 1150 mm long horizontal three-zone tube furnace (Carbolite Limited). The catalyst was loaded into a ceramic boat and mechanical pumping is carried out by backing pump for 30 min (up to pressure 10 mbar) after mounting of the sample to the volume of reactor. The reactor was heated to the desired temperature at a controlled rate (rate of heating varied between 5 $^{\circ}$ C and 10 $^{\circ}$ C min $^{-1}$). The working gas—acetylene was fed in the reactor to the required value of pressure after reaching the desired temperature. Pumping of residual gases and cooling the reactor to room temperature were carried out at a predetermined speed at the end of the synthesis (the time of experiments varied from 1 to 3 h).

The scheme of technological equipment for synthesis of carbon materials by method of thermal CVD and the procedure of conduction of experiments were described in detail in works [21, 22].

2.3. Methods and apparatus for the study of the morphology and structure of CNs

The field emission scanning electron microscope with ultra-high resolution of model Gemini Ultra 55 of the company Zeiss, with a device for x-ray microanalysis of the company ≪Thermo Scientific≫ was used to study

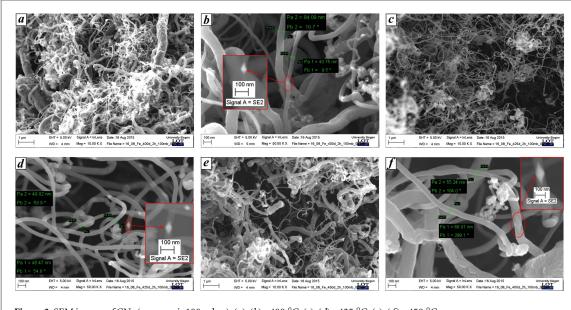


Figure 3. SEM images of CNs (pressure is 100 mbar): (*a*), (*b*)-400 °C, (*c*), (*d*)-425 °C, (*e*), (*f*)-450 °C.

the morphology of the samples. Two basic modes of shooting InLens and SE2 were selected for the study. InLens method provides the best resolution of SEM images of the surface morphology or cross section of the scanned sample. Shooting mode SE2 is the most preferred for obtaining accurate characterisctics of the surface topography of the scanned sample. Investigations were carried out at the Institute of Materials Engineering of the University of Siegen (Germany).

The study on the structure of CNs is carried out by the method of x-ray analysis using diffractometers Philips X'Pert PRO MRD (Institute of Materials Engineering of the University of Siegen, Germany) and Rigaku D/max/2400 XRD (Shenyang National Laboratory of Materials Science, Chinese Academy of Sciences). Radiographs of samples were obtained using copper radiation ($\lambda=1.5406~\text{Å}$) in digital form. Processing of x-ray spectra to determine the angular position and intensity of the reflection was performed in program OriginPro 8.1. PCPDFWIN program with the base of diffractometric data PDF-2 was used for the phase analysis.

The samples were investigated by Raman spectroscopy using spectrometer NT-MDT NTegra Spectra (laser wavelength $\lambda = 473$ nm) at The National Nanotechnology Laboratory of open type (Almaty, Kazakhstan).

The analysis of samples by TEM was performed at the Institute of Nuclear Physics (Almaty, Kazakhstan) with a TEM JEOL JEM-2100F.

3. Results and discussion

3.1. SEM studies

The experiments were performed at different temperatures ($200 \,^{\circ}\text{C}$ – $700 \,^{\circ}\text{C}$) and pressures (100– $400 \,^{\circ}\text{mbar}$) to determine the optimal conditions for CNs synthesis and lower temperature limit. Studies have shown that the lower temperature limit is $400 \,^{\circ}\text{C}$. It is evident that catalytic decomposition of acetylene does not occur at lower temperatures. Thus, the results of experiments made it possible to allocate the optimum range for the low-temperature synthesis: temperature is $400 \,^{\circ}\text{C}$ – $450 \,^{\circ}\text{C}$, pressure is 100– $300 \,^{\circ}\text{mbar}$ [25–27].

Further, more detailed studies were carried out in these experimental ranges. Figures 3-5 show SEM images (with different shooting modes—InLens and SE2) of CNs obtained at temperatures of $400\,^{\circ}\text{C}-450\,^{\circ}\text{C}$ and pressures of 100-300 mbar.

As seen from SEM images, the massive growth of CNs starts at 400 °C during the entire investigated range of pressures. The steady growth of CNs with a fairly large dispersion of diameter from 40 to 100 nm and a different morphology (from spiral to straight) is observed in certain experiments in the temperature range of 400 °C–450 °C. SE2 shooting mode allowed us to determine that iron clusters are inside of CNs.

3.2. X-ray analysis of CNs

Figure 6 shows the x-ray spectra of CNs obtained at different temperatures ($400 \,^{\circ}\text{C}$ – $450 \,^{\circ}\text{C}$) and pressures (100– $300 \,\text{mbar}$).

The diffraction patterns of all samples present reflection from plane (002) ($2\theta \approx 26.38^{\circ}$, PDF #41-1487) which is the most characteristic of graphite. It should be emphasized that the intensity of the graphite peak

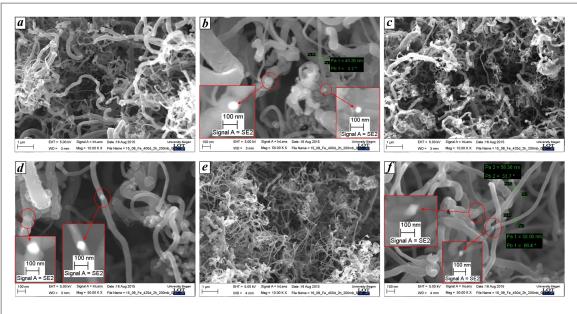
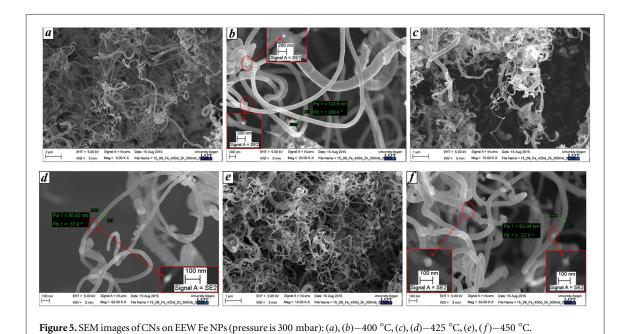
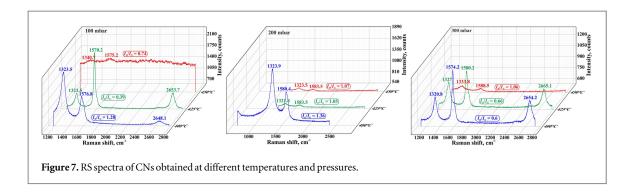


Figure 4. SEM images of CNs (pressure is 200 mbar): (*a*), (*b*) $-400\,^{\circ}$ C, (*c*), (*d*) $-425\,^{\circ}$ C, (*e*), (*f*) $-450\,^{\circ}$ C.



100 mbar 2240 g 2500 mbar 2250 g 2500 mbar 2250 g 2500 mbar 2550 g 2500 mbar 2550 g 25

 $\textbf{Figure 6.} \ \text{X-ray spectra of CNs obtained at different temperatures and pressures.}$



increases with the decrease in temperature. Also, x-ray qualitative analysis of the phase composition of the sample indicates that α -Fe, Fe₃C and C are contained in the composition of the sample. The presence of a considerable number of reflections Fe₃C suggests that growth of CNs occurs by vapor–liquid–solid mechanism with catalytic decomposition of acetylene on carbide cycle [28, 29].

3.3. Investigation of CNs by Raman scattering

Figure 7 presents the Raman spectra of CNs synthesized in the investigated range of temperature and pressure. All Raman spectra of CNs grown at 100 mbar predominantly show two groups of first order D and G. These groups are located in the region of 1323.5 cm⁻¹ and 1576.8 cm⁻¹ for the samples obtained at 400 °C, their full width at half maximum (FWHM) are $66 \, \mathrm{cm}^{-1}$ and $77 \, \mathrm{cm}^{-1}$, respectively. But intensity of D peak is higher compared to the G peak, which is a sign of high degree of crystalline disorder of the sample. Group G appears due to interplanar fluctuations of C–C bonds. Also, there is a second order group 2D at $2648.1 \, \mathrm{cm}^{-1}$, which is an overtone of group D [30]. This group is an indicator of the presence of long-range order in the sample and appears due to two-phonon secondary scattering, leading to the formation of inelastic phonon [31]. It may be associated with a level of crystallinity of the carbon nanotubes [32]. The intensity of these peaks depends on the metal properties of nanotubes [33].

The nanotubes grown at a temperature of 425 °C showed the best ratio of the intensities $I_D/I_G(0.39)$. D and G peaks are observed at 1323.5 cm⁻¹ and 1570.2 cm⁻¹, respectively. One can speak about high crystallinity of the sample judging from the width and intensity of the peaks and the presence of second order in the area of 2653.7 cm⁻¹. In addition, it can be seen that the intensity of D peak is quite small, which is also indicative of good quality of nanotubes. FWHM of samples are 77 and 34.6 cm⁻¹, respectively. The sample synthesized at 450 °C shows a rather blurred spectrum, indicating a defect structure. The shift of group D is also observed in the range of 1340.7 cm⁻¹, and group G is located in the range of 1575.2 cm⁻¹. FWHM of group D is much greater than that of the first two samples, indicating the presence of large number of defects and low crystallinity [34].

It can be said that multiwall nanotubes of good quality were obtained at a pressure of 100 mbar and temperature of 425 °C based on the RS results.

The spectra of CNs obtained at 200 mbar show that the intensity of D peak is higher than that of G peak. This suggests that the nanotubes have a defective structure. Group G is in the range of 1580.4 in the first sample and 1583.5 cm⁻¹ in the last two samples (425 °C and 450 °C), indicating a significant graphitization of sample [31]. Also, this may be indicated by the absence of 2D peak.

The samples obtained at a pressure of 300 mbar and temperatures of 400 °C and 425 °C show high crystallinity judging from the intensity of 2D peak. It can be assumed that they are quite ordered according to the intensity of D peak. It is located at 1320.8 and 1327 cm⁻¹, respectively. Group G is observed at 1574.2 and 1580.2 cm⁻¹. The third sample shows not smooth spectrum as previous. D peak intensity is higher than that of G peak, the latter is shifted to 1588.5 cm⁻¹. There is no long-range order in this sample due to the absence of 2D peak.

The lowest value of the ratio of the intensities of D and G peaks corresponds to nanotubes obtained under the conditions of 100 mbar and 425 °C. These are the most crystalline nanotubes. Also good results were obtained for nanotube grown at 300 mbar and 400 °C, 425 °C. It can be seen that more ordered nanotubes are synthesized with increase of temperature for 200 mbar, whereas their quality goes down at 300 mbar. The samples obtained under the conditions of 100 mbar and 400 °C, 200 mbar and 400 °C show the lowest quality. It can be concluded that the most optimal conditions for obtaining nanotubes are 100 mbar and 425 °C after analyzing the Raman spectra.

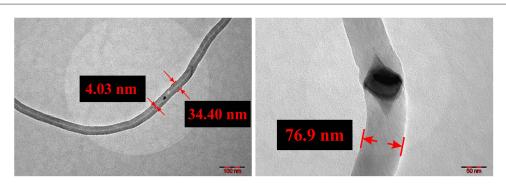


Figure 8. TEM images of CNTs synthesized at a pressure of 100 mbar and a temperature of 425 °C.

3.4. TEM studies of CNs

The samples synthesized at a pressure of 100 mbar and a temperature of 425 °C were further studied by TEM based on analysis of SEM images, Raman spectra and diffraction patterns. Figure 8 shows TEM images of these samples.

As seen, the obtained CNs are multi-walled CNTs. This agrees well with the results of x-ray diffraction and Raman spectroscopy. Metal clusters are arranged inside the channel of tube, which correlates with the SEM images taken in mode SE2.

4. Conclusion

The conducted experiments demonstrated the possibility of using Fe NPs obtained by the EEW as catalysts for growth of CNs. Stable growth of CNs is carried out at temperatures significantly lower than commonly used by thermal CVD on iron catalysts. The studies have shown that the lower temperature limit is 400 °C. The massive growth of CNs is observed in the entire investigated range of pressures at the same temperature. SEM studies in SE2 mode and TEM results allowed to determine that the iron clusters are located inside the CNT.

Raman spectroscopy showed that nanostructures grown on NPs at 100 mbar and 425 $^{\circ}$ C have the highest crystallinity according to the intensities of the D and G peaks and presence of 2D group. These findings are consistent with the results of x-ray analysis. TEM results confirmed that the obtained CNs are multiwall nanotubes with metal clusters inside the channel. Thus, experimental modes of synthesis of carbon nanotubes by low-temperature CVD using iron nanopowders as catalyst have been found for the first time. In the course of the studies carried out by us it have shown that Fe nanoscale particles obtained under nonequilibrium conditions of electric explosion of conductors have a crystalline structure with a lattice parameter different from the standard. We suppose that the reduction of the lower temperature boundary is associated with increased catalytic activity of EEW nanopowders, which in turn depends on the changes in the crystal structure parameters of Fe nanoclusters.

The results obtained in the course of research have a high potential for the development of efficient, low-energy, low-cost technology for obtaining CNTs and CNFs, without the use of expensive gas and the ability to control the structure and properties of macroscopic parameters of CNs. The synthesized CNTs do not have high crystallographic properties (degree of crystallinity, the geometric orientation, etc). However, the use of such CNs is promising in the fields of industry, which do not require structural perfection (such as reinforcing component in the concrete, polymers, plastics, ceramics and other materials; the basis for active sorbents; composite materials in the manufacture of medical prostheses).

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