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OXIDATION OF MULTIWALLED CARBON NANOTUBES USING DIFFERENT OXIDATION AGENTS LIKE NITRIC ACID AND POTASSIUM PERMANGANATE

E.Y.MALIKOV[*], O.H.AKPEROV[*], M.B.MURADOV[*], G.M.EYVAZOVA[*], A.M.MAHARRAMOV[*], Á.KUKOVECZ[**], Z.KÓNYA[**]

[*]Baku State University
[**]Szeged University
bsuc@hotmail.com

Oxidation of multiwalled carbon nanotubes, synthesized by Chemical Vapor Deposition method using Al-Fe-Co catalyst has been carried out in this work. The main purpose of the work is the use of two famous types of oxidation agents to oxidize the multiwalled carbon nanotubes and define differences or similarities between oxidizing potentials of these agents. Two different oxidation agents like nitric acid and potassium permanganate were used for this purpose. The obtained nanostructures were characterized by FTIR, XRD, Raman spectroscopy, TEM and SEM. FTIR results show characteristic peaks of the anticipated carboxylic group. The formation of functional groups on the multiwalled carbon nanotubes was confirmed by SEM and TEM by observation of the defected sides. XRD patterns structurally characterize the obtained materials. Raman spectra give significant information about defect formation on the sidewalls of the tubes.

Key words: *carbon nanotubes, nanostructures, synthesis of carbon nanotubes, oxidation of carbon nanotubes, electron microscopy, Raman spectroscopy*

1. Introduction

Carbon nanotubes (CNTs) have received considerable attention owing to their unique electronic and mechanical properties that are expected to lead to breakthrough industrial applications. Several types of the tubes are familiar to scientists. One of them is multiwalled carbon nanotubes (MWCNTs) that can be considered as a series of singlewalled carbon nanotubes, i.e. graphene sheets rolled into seamless cylinders, arranged coaxially with regularly increasing diameter [1]. CNTs have been synthesized generally by chemical vapor deposition (CVD) [2-6], laser ablation [2,7-9] and arc discharge technique [2,8-10].

The oxidation methods of CNTs can be divided into wet chemical methods [11-18], photo-oxidation [19,20] oxygen-plasma [21], or gas phase treatment [22] and these methods have attracted a lot of attention in order to enhance the chemical reactivity of the tubes. Pristine MWCNTs can be effectively purified and oxygen-containing groups, mainly carboxyl and hydroxyl, can be introduced onto the graphitic surface using above mentioned methods [23]. This, in turn, affects the solubility and reactivity of MWCNTs and increases the possibility of further modification [24].

Chemical oxidation was carried out by Avilés et al. [25,26] using a sequential treatment based on nitric acid followed by hydrogen peroxide. For the oxidative treatment, the agglomerated MWCNTs were first mixed with HNO_3 and mechanically stirred in a stirring plate for 15 minutes. The mixture was then sonicated in an ultrasonic bath for 2h, promoting CNT disentanglement within the acid solution. Then the slurry was filtered, thoroughly washed with distilled water and the process repeated using H_2O_2.

In this paper the wet chemical oxidation method was used in order to oxidize the MWCNTs. The process was carried out using different oxidation agents like $KMnO_4$ and HNO_3. It is observed that the solubility of the MWCNTs is improved with oxidation. Therefore, the obtained material can be used as an additive for the manufacturing the advanced plastic-composite materials or an additive to construction materials for strengthening purposes.

The main purpose of this work is the use of two famous types of oxidation agents to oxidize the multiwalled carbon nanotubes and define differences or similarities between oxidizing potential of these agents.

2. Experimental

2.1 Materials and methods

All the chemicals were of analytical reagent grades. Transmission Electron Microscopy images were taken with a FEI TECNAİ G2 20 X–TWIN Transmission Electron Microscope using 200 kV voltage. The TEM specimens were prepared by dripping the droplets of sonicated solutions of the samples onto the carbon coated copper grid. Scanning Electron Microscopy images have been taken with Hitachi S-4700 SEM device in 10 kV accelerating voltage. Powder XRD patterns were recorded on a Rigaku MiniFlex Desktop X–ray Diffractometer. X-ray diffraction measurements were performed using CuK_α radiation (1.5418 Å). FTIR spectra were recorded on a BRUKER Vertex 70 IR spectrometer. Raman investigations were done using Thermo Scientific DXR Raman Microscope at 532 nm and 10 mW laser parameter.

2.2. Synthesis of MWCNT

MWCNTs were synthesized by the CVD method. The experimental set-up used for this process consists of a Lenton LTF 14/75/610 type horizontal furnace, quartz tube (Tube diameter: 6 cm, Tube length: 95cm) and gas flow meters. An Al-Fe-Co catalyst was used to direct the process. It was prepared using $Al(OH)_3$, $Fe(NO_3)_3$ x 9 H_2O and $Co(NO_3)_2$ x 6 H_2O and alcohol solvents under ultrasonic agitation. Then it was evaporated in vacuum and dried completely.

MWCNT growth on catalyst was performed using acetylene (acetylene purity: 3.0 [>=99.9%]) (acetylene flow rate=30ml/min) as the carbon source and nitrogen (nitrogen flow rate=300ml/min) as the gas for flow and inert condition. The process lasted for 2 hours at 650°C.

2.3. Cleaning of the MWCNT

There are some trapped catalyst particles in and out of MWCNTs. They can affect the next processes. For that reason they should be cleaned. Following cleaning procedure was used for this purpose. The MWCNTs with catalyst impurity were mixed with concentrated HCl (36%) and the mixture has been undergone to the boiling process on a magnetic stirrer. The process was continued for 16 hours. Then it was filtered and washed. Obtained MWCNTs were mixed in NaOH (10M) and boiled for 16 hours again. Then it was filtered and washed again. Final powders are the clean MWCNTs.

2.4. Oxidation of MWCNT

MWCNTs are not reactive. They should be activated for the future use. Activation can be done with formation of functional groups on the sidewalls and end caps of the tubes, which makes them more reactive. The MWCNTs were undergone to the oxidation process using different oxidation agents like $KMnO_4$ and HNO_3. The clean MWCNTs were mixed with 0.1M $KMnO_4$ solution and the temperature was raised to 80°C and it was performed for 3 hours with stirring on a magnetic stirrer. Bubbles can be seen during the process which is the proof of the oxygen formation. This oxygen oxidizes the MWCNT.

The oxidation of MWCNTs using nitric acid was performed in two steps. Firstly 1 g of MWCNTs were mixed with 200 ml of concentrated (65 %) nitric acid in a beaker. Then the mixture was sonicated in 28 kHs for 30 minutes. In the second step it was boiled with stirring on a magnetic stirrer for 12 hours. The process was continued with washing, filtering and drying.

As the result of the oxidation carboxyl, hydroxyl, aldehyde, anhydride and other groups can be formed on the defected sides of the tube.

3. Results and discussion

3.1. FTIR investigation

Figure 1 shows the FTIR spectra for pristine MWCNT (1), MWCNT oxidized with $KMnO_4$ (2) and MWCNT oxidized with HNO_3 (3). The aromatic C=C stretching peaks at 1539 cm^{-1}, 1541 cm^{-1} and 1558 cm^{-1} as well as C-C vibrations at 1456 cm^{-1} and 1506 cm^{-1} characterize the pristine MWCNTs [27-29]. The same peaks can be observed in the spectrum of the MWCNTs oxidized with $KMnO_4$ (figure 1(2)) and the MWCNTs oxidized with HNO_3 (figure1(3)). Oxidation of MWCNTs with $KMnO_4$ is confirmed by the appearance of the C=O (at 1635 cm^{-1}, 1697 cm^{-1}, 1715 cm^{-1} and 1732 cm^{-1}) and C-O (at 1163 cm^{-1} and 1295 cm^{-1}) bands. The broad band with a maximum at 3381 cm^{-1}

can be assigned as the O-H stretching peak of the carboxylic group formed by the oxidative treatment. The same bands of the C=O (at 1632 cm^{-1} and 1717 cm^{-1}) and C-O (at 1145 cm^{-1} and 1385 cm^{-1}) can also be observed in the spectrum of the MWCNTs oxidized with HNO_3. Moreover the broad band between 3024 cm^{-1} and 3663 cm^{-1} is assigned as the stretching peak of O-H which is the main part of the carboxylic group obtained by oxidation with HNO_3. Another fact of the defect formation in MWCNTs due to the treatment with HNO_3 is the sp^3 C-H stretching of benzene ring in the MWCNT observed at 2849 cm^{-1} and 2916 cm^{-1} [30].

The above mentioned FTIR results reveal that both of the oxidation agents used in this work are very useful for oxidation of MWCNTs.

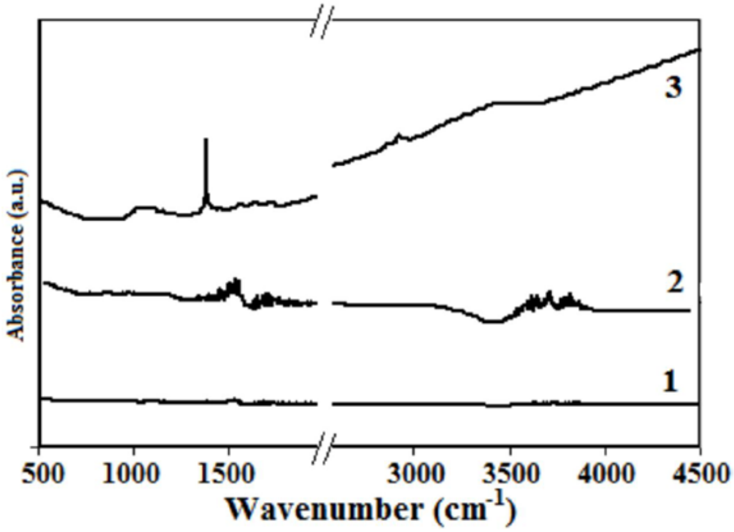

Fig. 1. FTIR spectra for pristine MWCNT (1), MWCNT oxidized with $KMnO_4$ (2) and MWCNT oxidized with HNO_3 (3)

3.2. XRD spectroscopy

Figure 2 shows the X-ray patterns for pristine MWCNT, MWCNT oxidized with HNO_3 and MWCNT oxidized with $KMnO_4$. Characteristic (002), (100), (101) and (004) peaks were observed in the pattern for MWCNTs. The average coherence length (L_c) or mean crystalline size along the c-axis (crystallographic c-axis is the perpendicular axis to the long axis of the MWCNTs) was estimated by the Scherrer`s equation [31]:

$$L_{hkl}=(0.9 \text{ x } \lambda)/(\beta \text{ x } \cos\theta)$$

where λ corresponds to the X-ray wavelength, θ is the Bragg angle and β is the half-peak width in radians. The (002) Bragg peak was used to calculate L_c. The results were about 11 nm (pristine MWCNT), 10 nm (MWCNT oxidized with HNO_3) and 6 nm (MWCNT oxidized with $KMnO_4$). These values represent an average stacking height of graphitic planes in the MWCNT walls. It can be seen from the results that the value of L_c decreases in the line from pristine MWCNT to oxidized MWCNT. With the oxidation using HNO_3 and $KMnO_4$, outer layers of the MWCNTs lose their symmetry and become defected. But the nature and the quantity of the defect formation depend on the type of the oxidation agent. As it can be seen from the results, the L_c value for the MWCNT oxidized with $KMnO_4$ is quite less than the value for another one. It means that $KMnO_4$ is stronger oxidative agent in comparison with HNO_3.

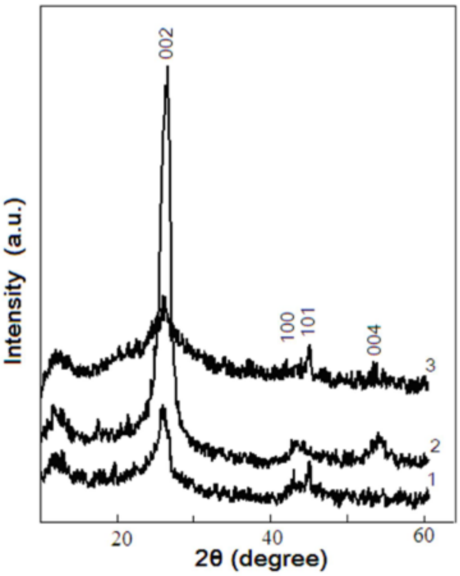

Fig. 2. XRD patterns for pristine MWCNT (1), MWCNT oxidized with HNO_3 (2) and MWCNT oxidized with $KMnO_4$ (3)

3.3. Raman spectroscopy

Raman spectroscopy is the most significant technique for CNTs and gives useful information like doping, defects, chirality, curvature and diameter. In this work Raman investigations were performed using a 532 nm green excitation laser. The spectra for pristine MWCNT, MWCNT oxidized with $KMnO_4$ and MWCNT oxidized with HNO_3 were summarized in Figure 3. In general Raman spectrum of CNT consists of 3 peaks like D band, G band and G` band [32]. The D band, which is observed at ~1340 cm^{-1} represents the scattering from defects and amorphous carbon impurities present in the MWCNTs samples. It is a disorder induced peak. The G band peak (~1570 cm^{-1}) is a result of ordered high-frequency in-plane stretching of the C-C bonds, which is characteristic for defect free carbon nanotubes. It is called Graphite band. The G` band (~2670 cm^{-1}) is the result of double resonance process. This band does not require an elastic defect-related scattering process and is observable for defect-free sp^2 carbons. The ratio between the intensities of D and G bands (I_D/I_G) is proportional with the extent of defect formation, thus it can be used to quantify the oxidation. As it can be seen from figure 3, the ratios for pristine

MWCNT, MWCNT oxidized with HNO_3 and MWCNT oxidized with $KMnO_4$ are 0.87, 0.93 and 0.95 respectively. An increase in the I_D/I_G ratio indicates an increase in the number of defects on the sidewalls of MWCNTs. Obtained results show that the MWCNTs oxidized with $KMnO_4$ contain more defects than the MWCNTs oxidized with HNO_3.

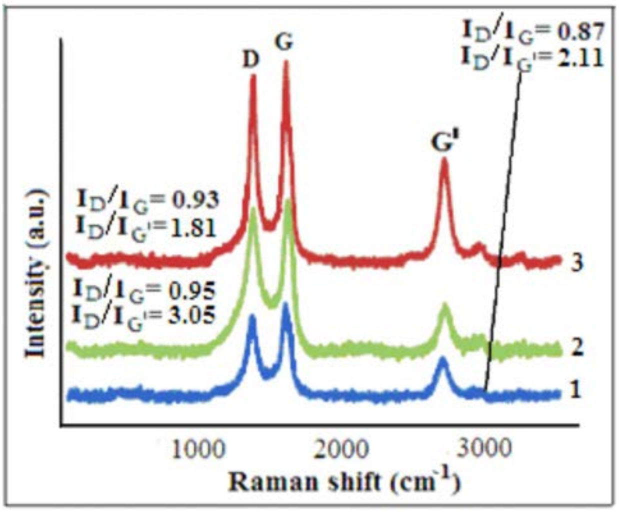

Fig. 3. Raman spectra of pristine MWCNT (1), MWCNT oxidized with $KMnO_4$ (2) and MWCNT oxidized with HNO_3 (3)

3.4. Microscopic investigations

3.4.1. TEM investigation

Figure 4 shows the TEM view of pristine MWCNT, MWCNT oxidized with HNO_3 and MWCNT oxidized with $KMnO_4$. It includes the images in the scales of 20 nm, 50 nm and 100 nm. It clearly shows the difference between pristine, and two types of oxidized MWCNTs. It can be deduced from the comparison of the figures that the differences are the defects. The pristine MWCNTs (figure 4a) look smooth, because they do not contain considerable defects on the sidewalls. On the other hand we can see the defect regions on the sidewalls of the oxidized MWCNTs (figure 4b,c,d). The sidewalls of the MWCNTs oxidized with nitric acid (figure 4b) are visibly defected. An articulated structure of the tubes and dot-like fragments on the background are the evidences of oxidation. Figure 4c,d shows that, $KMnO_4$ is much more aggressive oxidation agent than HNO_3. Because, with this oxidation the outer layers of the sidewalls of the MWCNTs are exfoliated with strong oxidation effect of this agent and changed their shape partly to the graphene-like structure. That means the inner walls of the tubes become uncovered and can easily be undergone to the oxidation. That is the priority feature of this process, because it improves the activity of the MWCNTs. But negative side of this oxidation agent is that, it is more destructive than HNO_3. The MWCNTs can acquire chemical activity but loose their mechanical excellence with this process. But it has a lot of advantages like obtaining much more oxidation effects even at mild conditions.

3.4.2. SEM investigation

SEM images show that the most of the structure consists of carbon nanotubes than amorphous carbon. Bindings and defect regions can be seen in the SEM images (Figure 5). In the Figure 5a,b it can be seen that, the sidewalls of the MWCNTs are not defected. Since they are not oxidized, they show smooth surface. In contrast, the sidewalls of MWCNTs described in Figure 5c are considerably defected. That is the result of the destructive effect of oxidation agent to the outer layers of the walls of MWCNTs which accompanied with functional group formation. Figure 5d is the SEM image of the MWCNT oxidized with $KMnO_4$. It is quite visible from the image that the defect regions are highly spread for the tubes oxidized with $KMnO_4$ which proves that this type of oxidation agent is stronger than the HNO_3.

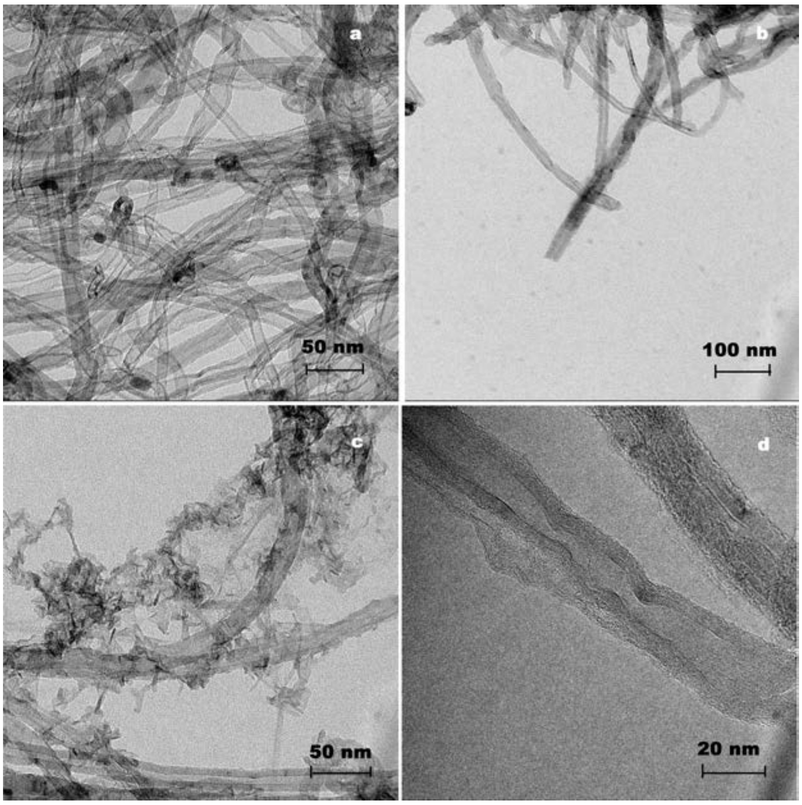

Fig. 4. TEM images of pristine MWCNT (a), MWCNT oxidized with HNO_3 (b) and MWCNT oxidized with $KMnO_4$ (c,d)

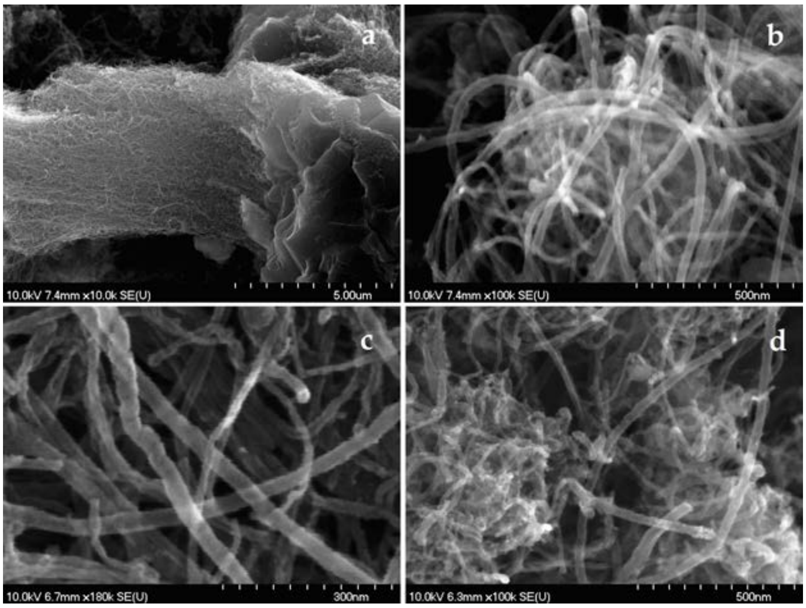

Fig. 5. SEM images for the pristine MWCNT (a,b), MWCNT oxidized with HNO_3 (c) and MWCNT oxidized with $KMnO_4$ (d)

4. Conclusion

The MWCNTs synthesized via the chemical vapor deposition method using the Al-Fe-Co catalyst, acetylene as the carbon source, nitrogen as the inert and flow gas, and horizontal furnace as the set up of the process were undergone to the cleaning process for removing the trapped catalyst particles. Then clean MWCNTs were oxidized with using two different oxidation agents like $KMnO_4$ and HNO_3 to obtain functional groups on the defect walls and end caps. The proof of formation of the functionalities was obtained using FTIR technique. The XRD, Raman, TEM and SEM results show defects which are the evidences of formation of the functional groups through the oxidation processes. Visually, defects can easily be seen on the surface of the MWCNTs in the microscopic images. All these results express that, $KMnO_4$ and HNO_3 are useful compounds for oxidation of MWCNTs. But it can be concluded that, $KMnO_4$ is better for the oxidation potential and much more fertile than HNO_3. Because, quite miserable amount of $KMnO_4$ is enough to get rather considerable amount of oxidized MWCNTs. Moreover, this oxidation proceeds under mild conditions which makes it cheaper in comparison with oxidation using HNO_3. The oxidation process is necessary to make the MWCNTs reactive and mixable in solvents. It is possible to use it as the first stage of the manufacturing process in the future to produce composites on this basis.

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NİTRAT TURŞUSU VƏ KALİUM PERMANQANAT KİMİ MÜXTƏLİF OKSİDLƏŞDİRİCİ AGENTLƏRİN İSTİFADƏ EDİLMƏSİ İLƏ ÇOXDİVARLI KARBON NANOBORULARIN OKSİDLƏŞMƏSİ

E.Y.MƏLİKOV, O.H.ƏKBƏROV, M.B.MURADOV, Q.M.EYVAZOVA,
A.M.MƏHƏRRƏMOV, A.KUKOVEÇ, Z.KONYA

XÜLASƏ

Bu işdə Al-Fe-Co katalizatoru istifadə edilməklə Kimyəvi Buxar Çökdürmə metodu ilə sintez edilmiş çoxdivarlı karbon nanoboruların oksidləşdirilməsi həyata keçirilmişdir. İşin əsas məqsədi çoxdivarlı karbon nanoboruların oksidləşməsi üçün iki oksidləşdirici agentin istifadəsi və bu agentlərin oksidləşdiricilik qabiliyyətləri arasındakı müxtəlifliklər və ya eyniliklərin müəyyən edilməsidir. Bu məqsədlə nitrat turşusu və kalium permanqanat kimi iki müxtəlif oksidləşdirici agent istifadə edilmişdir. Alınmış nanostrukturlar FTİQ, Rentgen difraksiya, Raman spektroskopiya, KEM və SEM vasitəsilə xarakterizə olunmuşdur. FTİQ nəticələri gözlənilən karboksil qrupunun xarakterik piklərini göstərir. Çoxdivarlı karbon nanoborularda funksional qrupların formalaşması SEM və KEM vasitəsilə defektli tərəflərin müşahidəsi ilə təsdiqlənmişdir. Rentgen difraksiya diaqramları alınmış materialları struktur baxımından xarakterizə edir. Raman spektrləri boruların yan divarlarında defekt formalaşması haqqında əhəmiyyətli məlumat verir.

Açar sözlər: karbon nanoborular, nanoquruluşlar, karbon nanoboruların sintezi, karbon nanoboruların oksidləşməsi, elektron mikroskopiya, raman spektroskopiya

ОКИСЛЕНИЕ МНОГОСЛОЙНЫХ УГЛЕРОДНЫХ НАНОТРУБОК С ПОМОЩЬЮ РАЗЛИЧНЫХ ОКИСЛИТЕЛЬНЫХ АГЕНТОВ, ТАКИХ КАК АЗОТНАЯ КИСЛОТА И ПЕРМАНГАНАТ КАЛИЯ

Э.Я.МЕЛИКОВ, О.Г.АКПЕРОВ, М.Б.МУРАДОВ, Г.М.ЭЙВАЗОВА,
А.М.МАГЕРРАМОВ, А.КУКОВЕЧ, З.КОНЯ

РЕЗЮМЕ

В работе осуществлено окисление многослойных углеродных нанотрубок, полученных с помощью химического осаждения из пара используя катализатор Al-Fe-Co. Целью исследования является использование двух известных агентов для окисления многослойных углеродных нанотрубок и определение различия и сходства между окислительными способностями этих агентов. Для этой цели были использованы два различных окислителя, такие как азотная кислота и перманганат калия. Полученные наноструктуры были охарактеризованы с помощью ИК-Фурье, рентгеновской дифракции, рамановской спектроскопии, ПЭМ и СЭМ. Результаты ИК-Фурье показывают характерные пики ожидаемых карбоксильных групп. Формирование функциональных групп на многослойных углеродных нанотрубках было подтверждено на СЭМ и ПЭМ путем наблюдения дефектов на стенках. Рентгенограммы характеризуют структуру полученных материалов. Спектры комбинационного рассеяния дают важную информацию о дефектообразованиях на боковых стенках труб.

Ключевые слова: углеродные нанотрубки, наноструктуры, синтез углеродных нанотрубок, окисление углеродных нанотрубок, электронная микроскопия, рамановская спектроскопия

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