Fabrication of carbon nanopowder by arc discharge technique

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Abstract

Carbon Nanopowder was fabricated by arc discharge technique at deposition pressure of 10^{-5} mbar Argon gas on glass substrates. The prepared carbon nano- powder was collected from chamber and purified with nitric acid at 323K .The morphology and crystalline structure of the prepared powder was examined by X-Ray Diffraction (XRD), Atomic Force Microscope (AFM), and Scanning Electron Microscope (SEM). XRD spectrums showed that the powder exhibits amorphous structure and after purification, the powder showed hexagonal structure with a preferential orientation along(002) direction ,where AFM and SEM gave very compatible estimation on the grain size and shape of the nanopowder.

Key words

carbon nanopowder, arc discharge, surface morphology

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تصنيع مسحوق الكاربون النانوي باستخدام تقنية التفريغ الكهربائي القوسي

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الملخص

تم تصنيع مسحوق الكربون النانوي باستخدام تقنية التفريغ ألقوسي(تحت ضغط بحدود10^{-5} ملي بار غاز الاركون)على شرائح زجاجية ،جمع المسحوق من الحاضنة وتنقيته باستخدام حامض النتريك عند درجة حرارة 323 كلفن .تم فحص التركيب البلوري وسطح العينات خلال حيود الأشعة السينية (XRD) ومجهر القوة الذرية (AFM) ومجهر الماسح الالكتروني (SEM) .أظهرت نتائج الأشعة السينية تركيبا عشوائيا للمسحوق الغير معامل ومتعدد التبلور للمسحوق المعامل مع تركيب سداسي مفضل باتجاه (002) .كما ان فحوصات (AFM)، (SEM) اظهرت توافقا مع الحجم الحبيبي المحسوب وشكل المسحوق النانوي المصنع.

Introduction

During the last few years, advances in the solid state physics have been characterized by a change from bulk crystal to a very small in least one of their three dimensions. Semiconductor nanocrystals are the subject of a rapidly developing field. It can be defined as crystals with dimensions ranging from 10- 100 nm; above this size, they are termed microcrystals. When the dimensions of a solid are reduced from a large size to the size of the characteristic lengths of electrons i.e. de Broglie wavelength λ_B, coherence length and localization length then the particles behave wave-like and the crystal size becomes smaller leading to the semiconductor energy levels to be more separated

from each other and the effective band gap to increase, therefore new physical properties due to quantum effect is observed, such as: quantum conductance oscillations, quantum Hall effects, resonant tunneling single electron transport, etc. These properties are necessary to built nanostructure semiconductor heterojunction, superlattice, etc. [1]. The low dimensions materials are classified according to the number of dimensions in nanometer size into three types[2] Quantum wells, Quantum wires, Quantum dots.

The synthesis of nanoparticles of different materials and the study of their properties and possible applications are at present among the most active research areas. Nanostructured films are expected to offer efficient and flexible tools for applications in different fields such as catalysis, information storage, nonlinear optics, optical and chemical sensors and many others. The functional properties of the Nanostructured films are largely determined by composition, size, morphology and surface properties of the nanoparticles forming the film. Nanoparticles can have very different properties with respect to the relative bulk material due to quantum confinement effects [3].

Three main techniques to prepare carbon nanotubes CNTs are as follows: (i) arc-discharge technique; (ii) laser ablation technique; (iii) chemical vapor deposition (CVD) technique [4]. Nanotubes were observed in 1991 in the carbon soot of graphite electrodes during an arc discharge, by using a current of 100 Amps, that was intended to produce fullerenes[5]. The carbon arc-discharge method is the first technique that was used to grow CNTs; However the first macroscopic production of CNTs was made in 1992 by two researchers at NEC's[6]. During this process, the carbon contained in the negative electrode sublimates because of the high-discharge temperatures, because nanotube was initially discovered using this technique; it has been the most widely-used method of Nanotube synthesis.

The yield for this method is up to 30% by weight and it produces both single- and multi-walled Nanotube with lengths of up to 50 micrometers with few structural defects[7]. The process is carried out in a vacuum chamber with two carbon electrodes as carbon source. Inert gas (typically helium) is supplied to increase the speed of carbon deposition. When high dc voltage is applied between the carbon anode and cathode, plasma of the inert gas is generated to evaporate the carbon atoms .The ejected carbon atoms are then deposited on the negative electrode to form CNTs. Both SWNTs and MWNTs can be grown by this method, while the growth of SWNTs requires catalysts. It is the principal method to produce high-quality CNTs with nearly perfect structures. In the laser ablation technique, a carbon target is ablated by intense laser pulses in a furnace in the presence of an inert gas and a catalyst. CNTs are formed and collected on a cold substrate. Both the arc-discharge and laser-ablation methods require high growth temperature, which is about 3000–4000 $^{\circ}$C for the evaporation of carbon atoms from solid carbon source. The laser ablation method yields around 70% and produces primarily single-walled carbon Nanotube with a controllable diameter determined by the reaction temperature. However, it is more expensive than either arc discharge or chemical vapor deposition[1].

Experimental part

1. Evaporation system

The evaporation system is consisted of two graphite rods (American Elements C-GR-04-R purity of 99. 99%) of different diameters 3mm and 7mm used as an anode and cathode electrodes, respectively. The design and shape of the rods are illustrated in Fig.1. These two electrodes were installed in the center of the vacuum chamber of coated unit type Edward 306A, end to end separated by approximately 1mm and all other arrangements necessary for evaporation process such as the evaporation source, substrate holder and radiation heater are fixed inside the chamber. As for evaporation deposited films of carbon nanopowder, the substrate consists of glass slide which are putted at a normal distance (h) equal to 5 cm to the graphite rods.

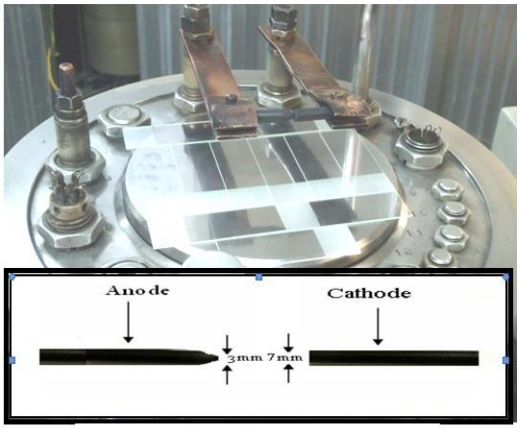

Fig.1: Shapes of graphite electrodes

2. Synthesis of carbon nanopowder

Arc discharge evaporation method in a high vacuum and low pressure was used to evaporate graphite. This method is a direct deposition technique in which the material is deposited by vaporization after plasma is produced. After the arc was carried out, much soot was deposited on the chamber wall .The soot was collected from the chamber wall and taken out to the purification treatment.

3. Purification of carbon anopowder

When the powder was collected from the chamber, it contained amorphous carbon and this is purified by the flowing:

Firstly the soot was placed in a beaker containing of 5ml conc. $HNO_3$and 10ml distilled water stirred in at 323 K for 90 minutes. The sample was washed with distilled water, dried and dispersed in ethanol under sonication and filtered using filter paper. To insulate the amorphous carbon from carbon Nanopowder, the samples were collected on the filter paper. The powder examined by Scanning Probe Microscope System (AA3000 SPM) and Scan Electron Microscope (SEM)Hitachi FE-SEM model S-4160 in University of Tehran - College of Engineering (Electricity engineering and Computer Dept).

Results and discussion

This method must be carried out with care and precision as flow: When the system is pumped down to a vacuum of $2X10^{-6}$ mbar, then it is broken out by entering the Ar gas to the chamber using the valve control, the passage of direct current creates a high temperature discharge between the two graphite electrodes because in DC arc discharge technique the cathode was used always larger than anode in the diameter about 3mm and 7mm as an anode and cathode electrodes, respectively. DC current of approximately 50 A with voltage of 25V was applied between two electrodes. The current density of cathode was significantly smaller than that of the anode; hence there was a notable temperature gradient between the anode and the cathode. This temperature gradient to the heat of the graphite rod and the plasma was produced; result in the evaporation of the one of graphite electrodes (anode)

to form rod shape on the cathode and chamber wall. After the arc was carried out, carbon anions arrived at the cathode, there were deposited and formed several forms of carbon which include Nanotube, then the current supply was switched off and the samples were left in the high vacuum for one day, then the air was admitted to the chamber, and the films were taken out from coating unit and kept in the desiccators until the measurements were made. The carbon nanopowder characterized by x-ray diffraction (XRD), Atomic Force Microscopy (AFM), and Scan Electron Microscopy (SEM).

3.1 X-Ray Diffraction

The structure of carbon nanopowder at disposition pressure (10^{-5} mbar argon gas) under vacuum by arc discharge technique has been examined by x-ray diffraction measurements at low angle of diffraction. X-ray system Shimadzu XRD 7000 (Voltage 4kV 30mA) maxima Ni filter scan speed 10 deg/min resolution with capper K_α radiation of wavelength λ =1.54 Å was used for measurements, the scanning angle (2θ) varied in the range (10-90)^o with speed 2cm/min. Fig.2 showes x-ray diffraction of CNpowder as received from chamber and that purified by acid-treated prepared at pressure (10^{-5} mbar Argon gas) the figure reveal find highly oriented along the c-axis giving a peaks at 2θ= 26.58^o and 54.69^o which belongs to (002) and (004) respectively for purification CNpowder by acid treated which agreement with reference to JCPDS No.56-159[8].

The peak at 2θ=26.58^0 which was strong and sharp, was corresponding to (002) reflections of CN powder.

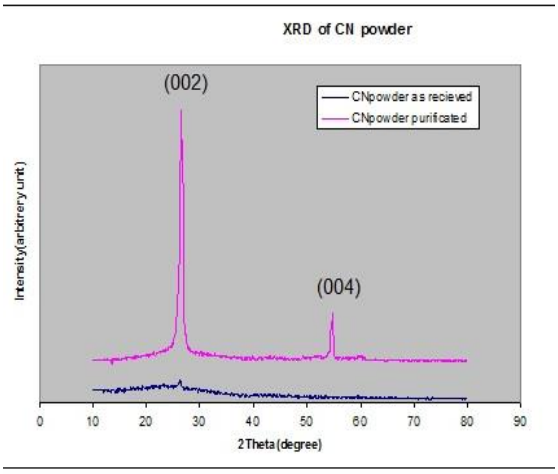

Fig.2: X-Ray Diffraction of CNpowder for as fabricated and purified by acid-treated prepared at pressure (10^{-5} mbar Argon gas).

The interplanar spacing was calculated to be $d_{(002)}$=3.351 Å the other beak which was corresponding to (004) at 2θ= 54.692^0.The interplanar spacing was calculated to be $d_{(004)}$=1.677 Å**.** The average grain size of the nanoparticles of CN powder purified by HNO_3 treated was estimated by using the standard Scherer formula[9], Table1 indicated for the grain size estimation

Table1: The value of (2θ) of the strong two peaks and grain size of purified CNpowder calculated by Scherer formula

Peak No.	2 θ	FWHM (deg)	hkl	d_{hkl} (Å)	G.S (nm)
1	26.580	0.538	002	3.351	14
2	54.692	0.197	004	1.677	43

2. Morphological characteristic of CNpowder

Atomic Force Microscope (AFM) & Scanning Electron microscope (SEM) were examined the powder fabricated of two types of powder.

A. CNpowder as received from chamber

AFM images of CNpowder as received from chamber was shown in Fig.3. The Granularity normal distribution of the particles collected as a result of the preparation of CNpowder, where the size of the grain rate ranged up to 60nm.

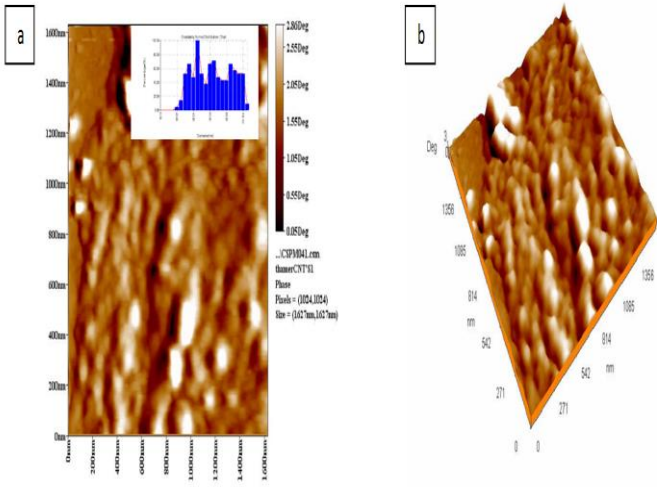

Fig. 3: (AFM)images of the untreated powder CNpowder(a)Two dimension and (b)Three dimension

SEM images of untreated CNpowder were shown in Fig.4. The diameters were measured to be about 60 nm and the side walls were non uniform reflected because more amorphous carbon in the structure.

B. Purified CNpowder by nitric acid

In order to dissolve the amorphous carbon, we purified CNpowder treated by nitric acid. Fig.5 shows (AFM) images of the treated powder (a) two imension and (b) three dimensions.

The Granularity normal distribution of the particles collected as a result of the preparation and purified of CNpowder, where the size of the grain rate ranged up to 50nm. SEM images Fig.6 shows morphological of purified CNpowder treated by nitric acid magnified 60000 times and shown the grain size about 50 nm.

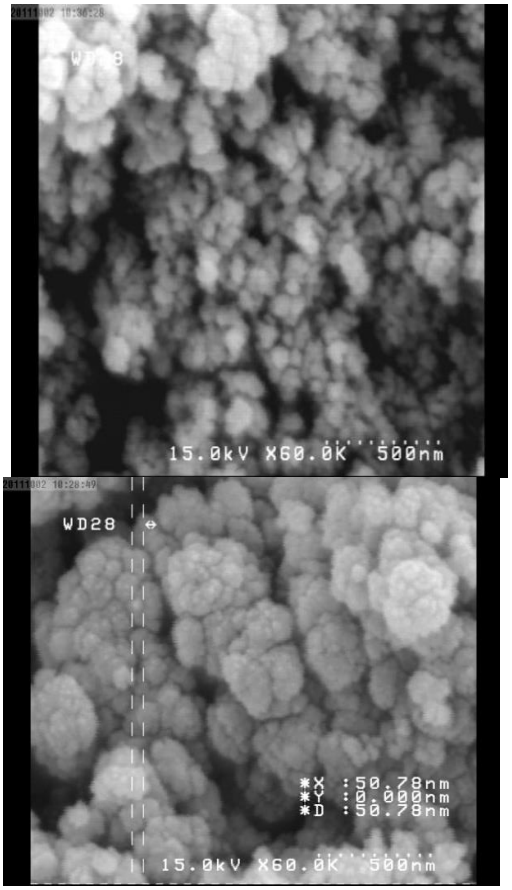

Fig.4: SEM images of untreated CNpowder magnified 60000 times showed the grain size

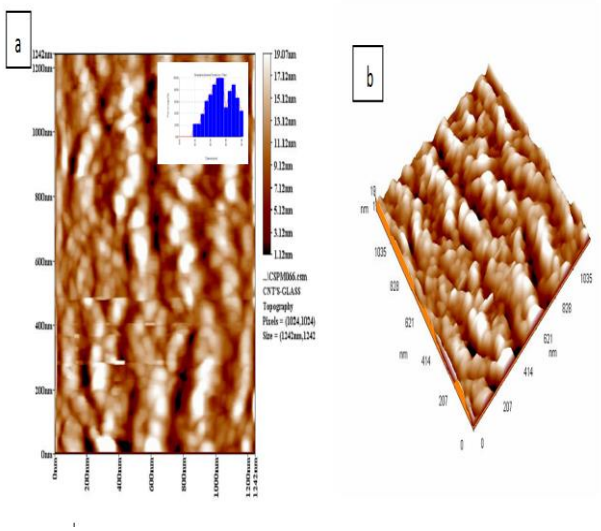

Fig.5: (AFM) images of the purified CNpowder(a)Two dimension and (b)Three dimension

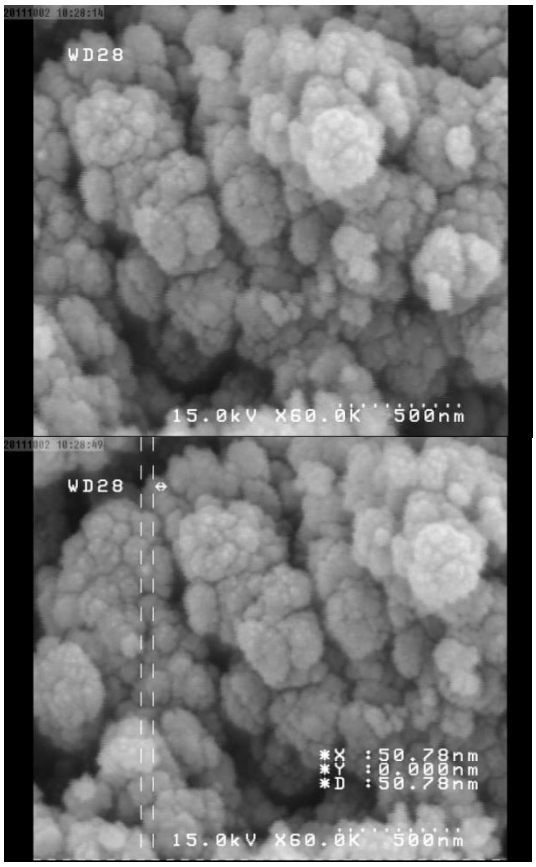

Fig.6: SEM images of treated CNpowder by nitric acid magnified 60000 times show the grain size

Conclusions

1. CNpowder of diameter 40-60 nm prepared by arc discharge method
2. The purification of the product using acid leaching led to more crystalline structure
3. XRD spectrum revealed two main planes 002 and 004 Scherrer formula showed crystal grain size of 14nm, 43nm
4. AFM and SEM showed similar surface morphology and Particle size distribution around 50 nm

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