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# VERTICALLY ALIGNED CARBON NANOTUBES FABRICATED BY MICROWAVES

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**Abstract.** The development of a highly efficient one-step technique was carried out to obtain long and aligned carbon nanotubes with or without Fe filling. The aligned carbon nanotubes (CNTs) were synthesized by microwave (MW) irradiation heating from a ferrocene $FeC_{10}H_{10}$. In this research, Scanning Electron Micriscopy (SEM) and Transmission Electron Microscopy (TEM) are used to study the growth of aligned carbon nanotubes by microwave heating. As a contribution of this method, the aligned multi-layer carbon nanotubes were obtained. CNTs have a metal particle at the tip of each tube. This carbon nanostructure promises to become important in fuel cells and in nanoscale engineering of other systems in which electrical, mechanical, and chemical interactions are integrated to produce macroscale effects. The morphology of the carbon nanotubes was studied by Scanning Electron Microscopy, Atomic Force Microscopy, and Transmission Electron Microscopy. Measurements of distinct angles, distances, and spaces between waves were made.

## 1. INTRODUCTION

Nanotechnology is now recognized to have the potential to revolutionize a host of industries. One of the important aspects of synthesis of nanomateriales relates to the formation of nanotubes and nanorods. Carbon nanotubes (CNTs) have been the focus of intensive study since their discovery in 1991 [1] because of their unique electronic and mechanical properties. A number of device applications, such as full-color displays, fieldeffect transistors, and molecular computers have been envisioned [2,3]. These applications are highly dependent on the electronic properties of the CNTs, which can be tuned by their helicity, diameter, and the introduction of defects. In recent years, novel strategies have been devoted to the altering of the physical properties of the CNTs by surface modification with organic, inorganic, and biological species [4-9].

Synthesis method for the formation of carbon nanotubes include arc-discharge [1,10], laser ablation [11], gas-phase catalytic growth from carbon monoxide [12], and chemical vapor deposition from hydrocarbons [13-15]. Silicon crystals, quartz glass, porous silicon dioxide, and aluminum oxide were used as substrates for the growth of carbon nanotubes. Carbon nanotubes collected from these substrates were used to make carbon nanotubes composites [16], for gas storage [17], for electrochemical energy storage [18], and as templates for making nanotubes of other materials [19].

Considerable efforts have been made to prepare to aligned one-dimensional nanostructures. One-dimensional (1D) nanostuctures, such as nanotubes, nanowires, and nanorods, have attracted much attention due to their excellent properties, which originate from high specific surface areas and low dimensionality.

In this communication, we report the development of a highly efficient one-step technique to produce long and aligned carbon nanotubes with or without Fe filling. The aligned CNTs were synthesized by microwave (MW) irradiation heating according to [20] at heating ferrocene $Fe(C_5H_5)_2$ .Their character-

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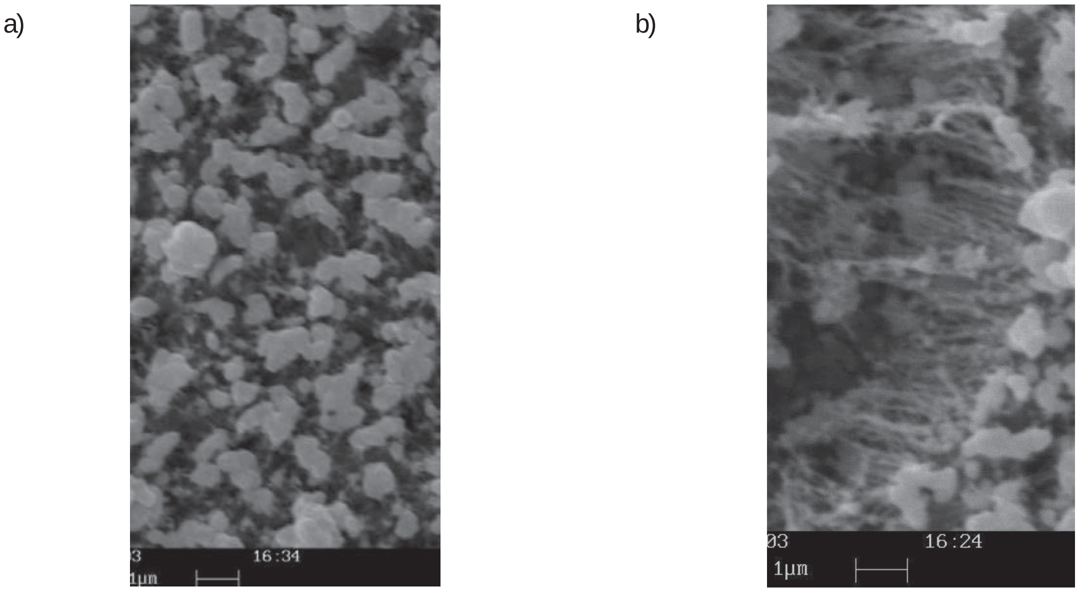


**Fig. 1.** SEM images of vertically aligned nanotubes heated by 30 min: (a) high-magnification view of the bottom-end of the array; (b) high-magnification view of the inclined vertically aligned nanotubes.

ization was performed by Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM).

## 2. EXPERIMENTAL

Preparation of nanotubes was carried out in a domestic MW-oven (power 1300 W and frequency 2.45 GHz). The MW-action allows the ferrocene heating without direct contact with energy source; the process control is achieved by varying the power and heating time from 15 to 30 min in vacuum of 10-5 Torr. The samples were prepared from powdered ferrocene (99%). The condensation of carbon vapor and accumulation of nanoparticles was used on the fused silica targets.

## 3. RESULTS AND DISCUSSION

The vertically well-aligned one dimensional nanostructures are formed (Fig. 1). From the inset of Fig.1b, it can be seen that the entire length of the arrays reaches up to 2-3 μm. The bottom sections of the arrays are nearly transparent, whereas the top sections are not. The outer diameters of the nanotubes are in the range of 50-100 nm. Same nanoballs, 200-500 nm in diameters, are clearly seen at the top-ends of the nanotubes (Fig. 2a).

The synthesis parameters, such as heating times may have an important influence on the length and thickness of the nanotubes. SEM observation of CNTs allows to conclude that the sizes of the nanotubes are increased with heating time (Fig. 2).

Two growth types for carbon nanotubes are known (Fig. 2c). In this work, we report how the vertically aligned CNTs are growing by MW heating on the one step by method number II (Fig. 2c). TEM observations allow us to gain an insight into the prepared nanotube structure. Typical TEM images of the formed nanotubes are shown in Fig.3. Each nanotube is sealed at one end with a large ball, 3-5 times larger than the characteristic nanotube dimensions. During the carbonization, the Fe aggregated into nanoparticles (CNTs). At 465 °C, the ferrocene molecules were decomposed on the surface of the Fe nanoparticles by the catalytic action of the metal. The carbon atoms were absorbed on and dissolved in the metal [21-23], transported to the interface between the iron particle and the growing end of the graphitic carbon nanotube, and incorporated into the tube. The metal particle was carried ahead as the nanotube grew longer.

To investigate the structure of nanotubes obtained by microwaves heating, CNT´s were analyzed by AFM. According to AFM studies (Fig. 4), carbon vaporization via microwave treatment produces multilayer nanotubes. The diameter of the channel of CNTs is 7.3 nm, spaces between waves ranges from 3.4 to 1.0 nm, and wave thickness of waves varies

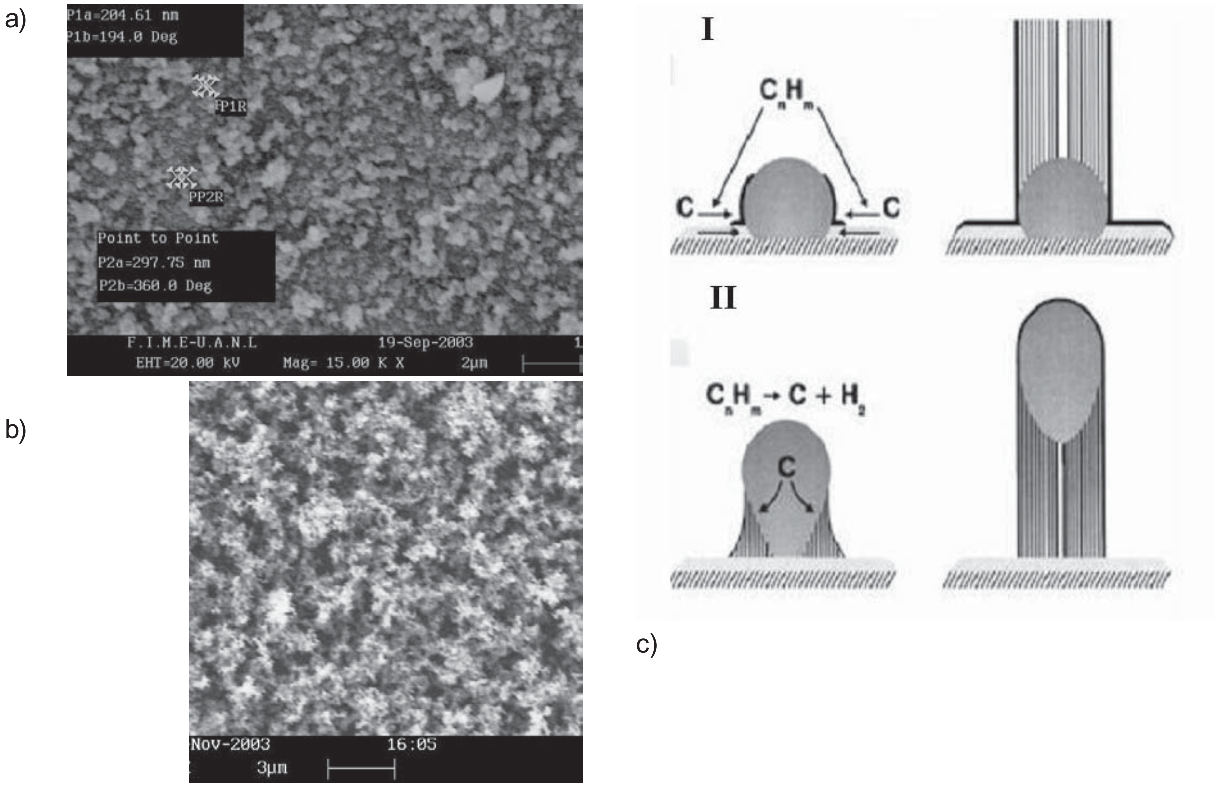


**Fig. 2.** High-magnification view the SEM image of the nanotubes: (a) the bottom-end with the dimensiones (heating 30 min); (b) CNTs by MW- heating ferrocene at 15 min; (c) Visualization of possible growth mechanisms of a carbon nanotube with metallic catalizator.

from 9.27 Å to 10.4 Å, (Fig. 5). The tube contains approximately 20-50 graphite wall layers. This result agree well with data reported in [21].

## 4.CONCLUSIONS

In summary, a novel method for synthesis of vertically aligned carbon nanotubes at short time was studied. The optimal process time was 30 min. As a contribution of this method, the aligned multi-layer carbon nanotubes were obtained. CNTs have a metal particle at the tip of each tube. The diameter of the channel of CNTs is 7.3 nm, spaces between layers ranges from 3.4 to 1.0 nm, and thickness of layers varies from 9.27 to 10.4 Å. The tube contains ap-

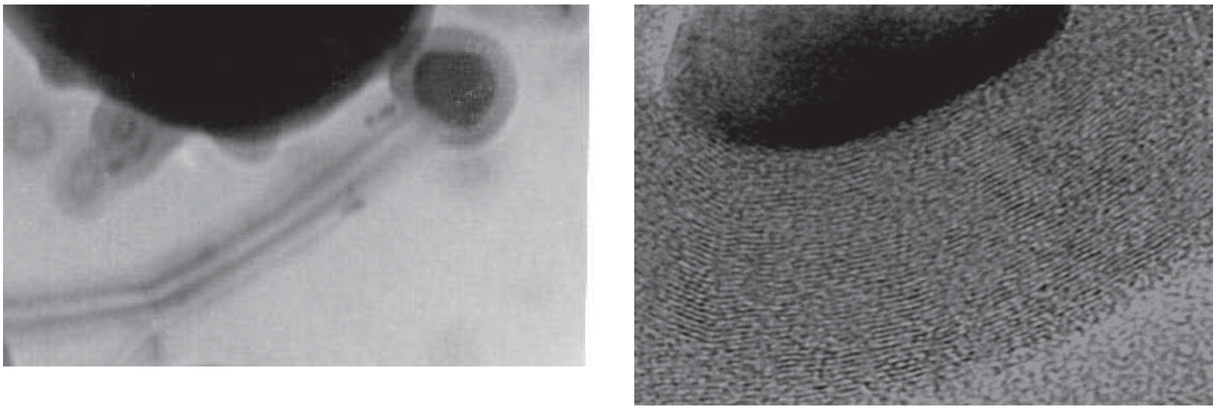
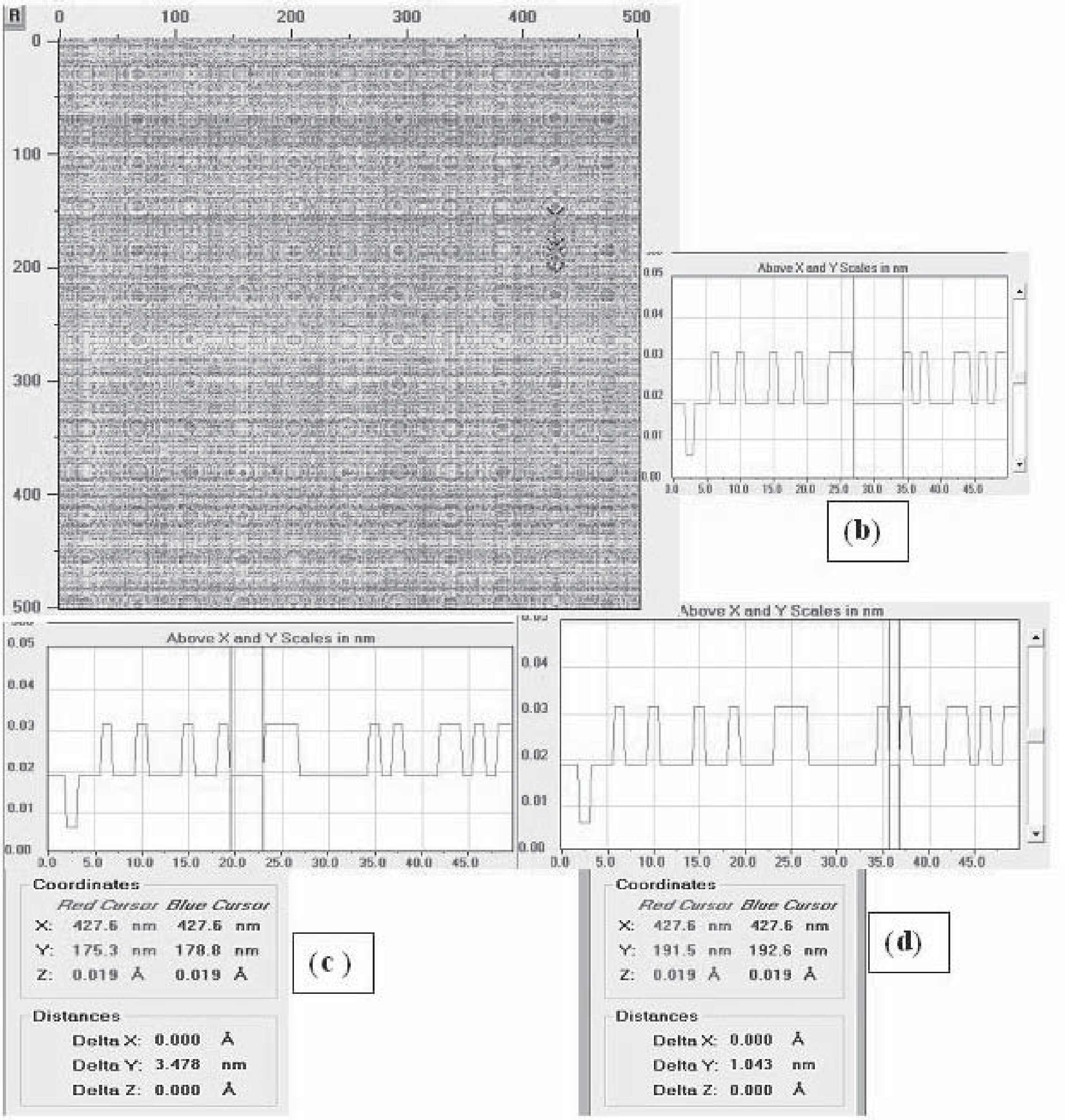


**Fig.3.** TEM images of Fe-filled CNTs at differnt scale: a) 255,000 X; b) 400,000X.



(c)

Coordinates

| | Red Cursor | Blue Cursor |
|---|---|---|
| X: | 427.6 nm | 427.6 nm |
| Y: | 175.3 nm | 178.8 nm |
| Z: | 0.019 Å | 0.019 Å |

Distances

| | | |
|---|---|---|
| Delta X: | 0.000 | Å |
| Delta Y: | 3.478 | nm |
| Delta Z: | 0.000 | Å |

(d)

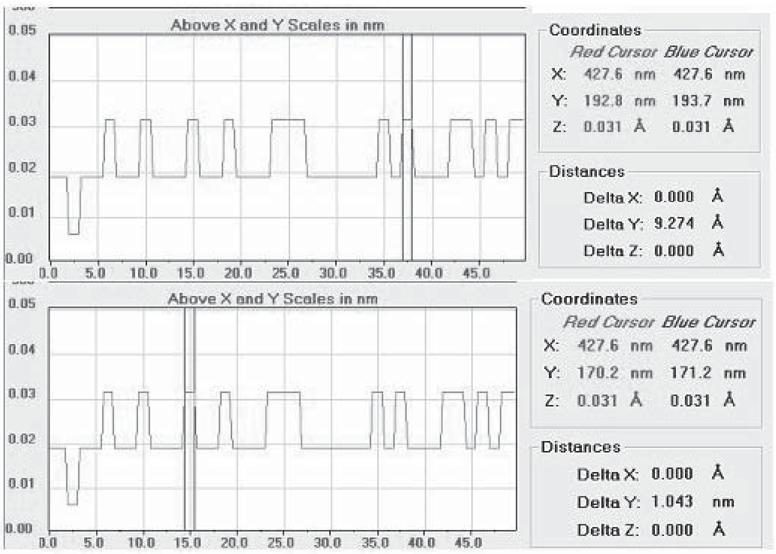
Coordinates

| | Red Cursor | Blue Cursor |
|---|---|---|
| X: | 427.6 nm | 427.6 nm |
| Y: | 191.5 nm | 192.6 nm |
| Z: | 0.019 Å | 0.019 Å |

Distances

| | | |
|---|---|---|
| Delta X: | 0.000 | Å |
| Delta Y: | 1.043 | nm |
| Delta Z: | 0.000 | Å |

**Fig. 4.** (a) AFM image of the CNT´s ; (b) measurement of internal diameter of the CNT;(c) and (d) different distances between waves .



Coordinates

| | Red Cursor | Blue Cursor |
|---|---|---|
| X: | 427.6 nm | 427.6 nm |
| Y: | 192.8 nm | 193.7 nm |
| Z: | 0.031 Å | 0.031 Å |

Distances

| | | |
|---|---|---|
| Delta X: | 0.000 | Å |
| Delta Y: | 9.274 | Å |
| Delta Z: | 0.000 | Å |

Coordinates

| | Red Cursor | Blue Cursor |
|---|---|---|
| X: | 427.6 nm | 427.6 nm |
| Y: | 170.2 nm | 171.2 nm |
| Z: | 0.031 Å | 0.031 Å |

Distances

| | | |
|---|---|---|
| Delta X: | 0.000 | Å |
| Delta Y: | 1.043 | nm |
| Delta Z: | 0.000 | Å |

**Fig.5.** Measurement of the layer widths.

proximately 20-50 graphite wall layers. This carbon nanostructure, promises to become important in fuel cells and in nanoscale engineering of other systems in which electrical, mechanical, and chemical interactions are integrated to produce macroscale effects.

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