Guru Jambheshwar University of Science and Technology

Department of Physics

Analysis of Scanning electron microscope(SEM) images of Carbon Nanotubes(CNT)

Project of Physics of Nano-materials

Submitted to : Dr. Ravi Bhatia

Submitted By

Sourabh 220070720010 , Rimit ..12 , Sandeep ..18

February,2024

Abstract

This report is the analysis of Scanning electron microscope images of Carbon Nanotube is done by the submitter. In which, we're tells about the introduction to CNT, its synthesis and some properties of CNT. And then we are analysis the images given by the assigner.

Carbon Nano Tubes (CNT)

1.1 Introduction

CNT are the cylindrical-folded sheets of the graphene which have diameter in the nano range (10^{-9}) and length in micrometer range (10^{-6}) or greater. And due to very high length-to-diameter ratio, they have unique and interesting properties. They are first discovered by the Iijima and he get the Nobel Prize for this discovery.

1.1.1 Types of CNT

There are two type of CNT as following below:

Single Walled CNT (SWCNT)

These are just a single cylindrical tube made up of the graphene with diameter of range 0.4 nm *to* 5 nm.

Multi Walled CNT (MWCNT)

These are the concentric cylindrical tube made up of the graphene with outer diameter of range up-to 100 nm and inner diameter of 10 nm and the separation between the tube are same as the separation between the layer of the graphide (0.34 nm)

1.2 Properties

Actually the properties of CNT are unique due to its very high length-to-diameter ratio. And some of them are given bellow:

1.2.1 Hybridization

The carbon atoms in CNT are sp2 hybridized, so they have 3σ and 1π bond.

1.2.2 Mechanical Properties

CNT are very strong due to its covalent bonds.

1.2.3 Electrical Properties

The electrical Properties of the CNT is interesting, the CNT can be a semiconductor or metallic in nature and this depended upon the (n, m) and chirality of the CNT.
For **SWCNT**, the nature of tube may be metallic or may be semiconductor, it depends upon the (n, m) and the condition for metallic is $\boldsymbol{n - m = 3q}$ ***such q is integer***, and there are 1/3 of the positions are there having value of (n, m) to be a conductor in nature and all others are semiconductor. And this will happen because the unhybridized orbit of carbon lead to conduction and valance band, and the value of (n, m) lead to the different separation between the conduction and valence band. And this ratio of conducting and semiconductor CNT also get in the production.
For **MWCNT**, the tubes are conducting in nature, because they have structure similar to the graphite, and it was a conductor.[1]

1.3 Synthesis

For synthesis of CNT we need a carbon source, that can give an atomic carbon species or cluster of carbon and then we need to assemble them into the CNT with or without the catalyst . And this will happen at the temperature range $\boldsymbol{2000 - 3000^oC}$ and this can also be done with chemical action. And this leads us to some methods are given below:

1.3.1 Arc Deposition

It is method we use to make the CNT by generating the temperature range $\mathbf{2000 - 3000^oC}$ using the arc discharge method, in this we use coal or graphite electrode to generate plasma and that cause the atoms of carbon to vaporized and then the atoms are arranged them into the CNT and we can collect them on the substrate , or we use this process without
We need the inert environment for this mainly using helium. And if we need to dope element we doped them into the electrode or use its vapors.

1.3.2 Laser Ablation

It is method similar to the Arc Deposition but in this we use high power laser to vaporize the carbon source and all the other things are same.

Both of these methods are making CNT using high thermal energy and due to this, other unwanted carbon compound are also formed and that create a very difficult to extract the CNT.

1.3.3 Chemical Vapor Deposition

It is technique which mostly used for making highly pure CNT, in this method we use a feed-stoke carbon source like acetone or methane in gases state, with substrate in gases or solid state as catalysis for the process of formation CNT. In this the carbon are collected on the catalysis and form bucky-ball type chap first then CNT when concentration become the saturated.

Scanning Electron Microscope

2.1 Introduction

It is electron microscope which is used to study the surface morphology and topology of the materials with resolution of range in 10 *nanometer*. It uses the electron matter wave as the source for the microscope.

2.2 Principle

- **de Broglie's Hypothesis:** Matter have a matter wave associated with it when they are in motion.

$$\boldsymbol{\lambda = \frac{h}{mv}} \tag{2.1}$$

  where h is plank constant, m is the rest mass of the particle moving with velocity v.
  And when the electron is used which is accelerated by the potential V then it's de Broglie wavelength λ become

$$\boldsymbol{\lambda = \frac{h}{\sqrt{2meV}}} \tag{2.2}$$

  And wavelength of electron is very small compare to the light.

- **Rayleigh Criterion:** The size of wave use to observe the particle have the same order of wavelength as the size of the particle.

2.3 Phenomenons

There are many phenomena is happening when the electron beam strikes the sample mainly bulk samples. These phenomena happen on surface or into very slight inside the sample. And these phenomenons are listed bellow:

- **Secondary electron generation:** When the electron beam strikes the sample then the electron have a very high kinetic energy and when they collide with atoms and knockout the atom's outer shell electron or ionized the atom.
- **Back-scattered electron:** The electrons are those who scattered back to the direction of the beam and they can be back scattered with or without any loose of energy.
- **Auger Electron:** When the electron strike the atom and remove the electron from the inner shell of electron let K then from its L shell electron get dexcited into L with transfer of the energy to outer shell electron then the emitted electron have specific energy related to the atom's atomic number. And this is the auger effect.
- **Characteristic X-ray:** It have same process as auger electron emission but this time the L shell electron loose there energy by emission of photon and this photon have energy equal to the energy gap between the shells. They have unique energy value for every atom , and it's related to its atomic number.
  And this detect with extension attachment of EDX (Energy dispersive x-ray analysis)

2.4 Construction

Electron microscope consists of these parts:

- **Electron Gun:** The electrons are generated by the heating of a tungsten filament about 1200^oC and then accelerate by applying a voltage and refined into the beam and voltage have order of few Kilo volts.
- **Condenser Lens:** These are the electromagnetic lens which can condense the electron beam and focus into a very small section and this

help to zoom into the sample and focus of microscope can be controlled by the voltage we use to condense the electron. This can be calculated by Lorentz's force equation and electron follow a helical path after pass through the lens.

- **Sample holder:** It is just a holder to hold the sample it different for different type of samples and it can also cryo cooled for biological sample.
- **Detector:** There are many types of detectors is used to detect the different phenomenon like:
  - **Everhart-Thornley detector:** It is use for detection of secondary electron in SEM.
  - **Semiconductor Detector:** It is made up of highly pure semiconductor and it is use for detection of back scattered electrons.
  - **Energy Dispersive x-ray analysis**
- **Vacuum Pump:** It is necessary to there is no air when the SEM is working because that cause the defection in electron beam. So we use high vacuum ($10^{-7} pascal$) when SEM is used. And this can be achieved by using first using rotatory pump and then diffusion pump for ultra vacuum.

2.5 Working

The heated filament produces the electrons then we apply voltage and accelerate the electron and make a electron beam. Then with the help of condenser electromagnetic lens the electron beam is confined a make a very small spot. And when the electron beam fall on the sample then the back scattered electron, secondary electron, auger electron and characteristic x-ray is produced and the detector detect them and analysis the result and we get the image. And we can move the sample holder and scan the whole sample.

2.6 Advantages of SEM

- It is very less expensive compare to the Transmission electron microscope.

- It have very high resolution compare to the simple microscope.
- With EDX extension we can also get the composition of the sample.
- It's sample preparing is not so difficult, we need to coat gold or platinum on the insulator sample.

2.7 Disadvantages of SEM

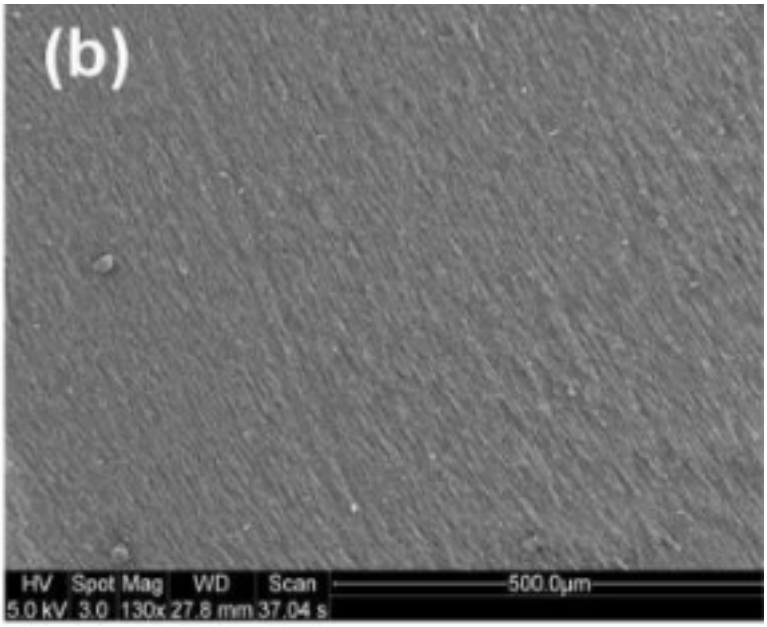
- It's resolution is still lower than the TEM or STEM.
- It only tell about the topology and surface morphology it can't tell about the inside the bulk.
- It is not use for very thin films.
- It need a trained personal to operate.
- It's needs the vacuum to work.

Analysis of Given SEM images

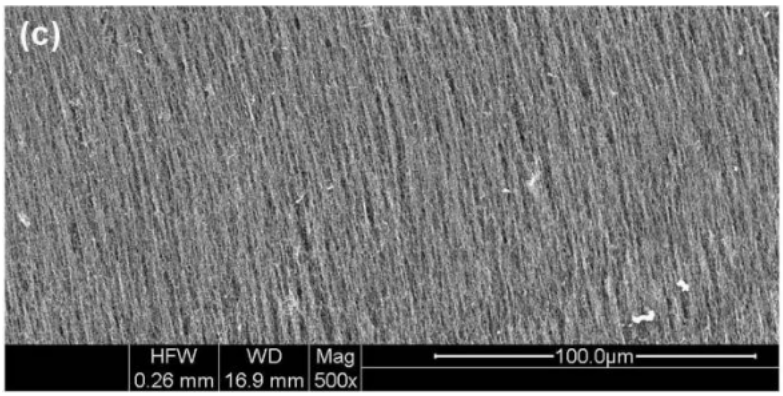
3.1 Abbreviations

- **HFW:**Horizontal Field of View, It is length of side of square field of view.
- **HV:** High Voltage, It is the accelerating voltage apply to the electron.
- **Spot:** It is the spot size.
- **Mag:** Magnification of the microscope.
- **WD:** Working Distance, It is the distance between the electron gun and the sample holder.
- **Scan:**Scanning time, It is the time for which scan is done.

3.2 Images on First page:

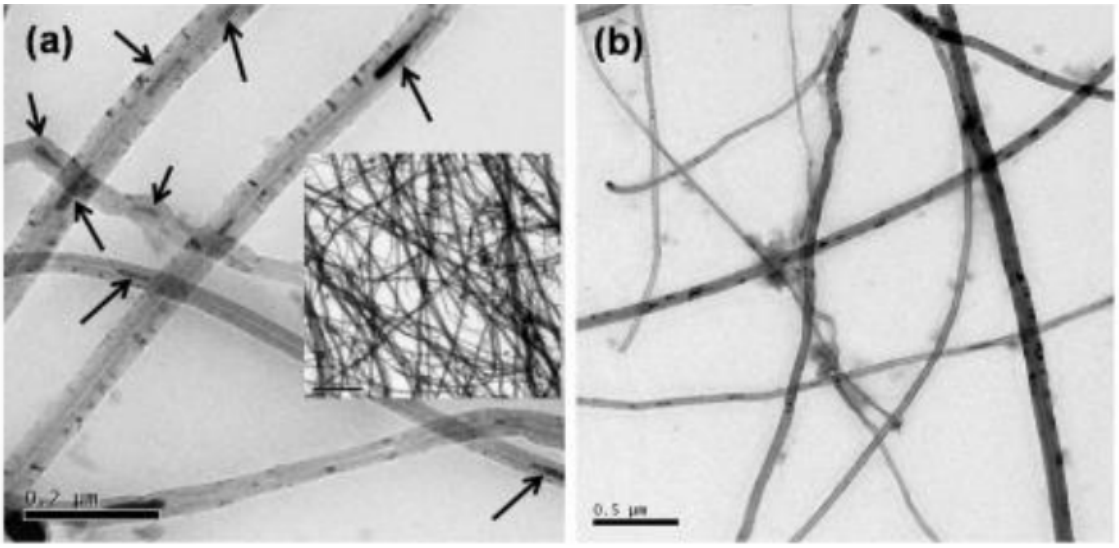


This is the image of CNT with 130x magnification with accelerating voltage of 50kV and having WD of 27.8 mm, and Scan for 37.04 seconds.



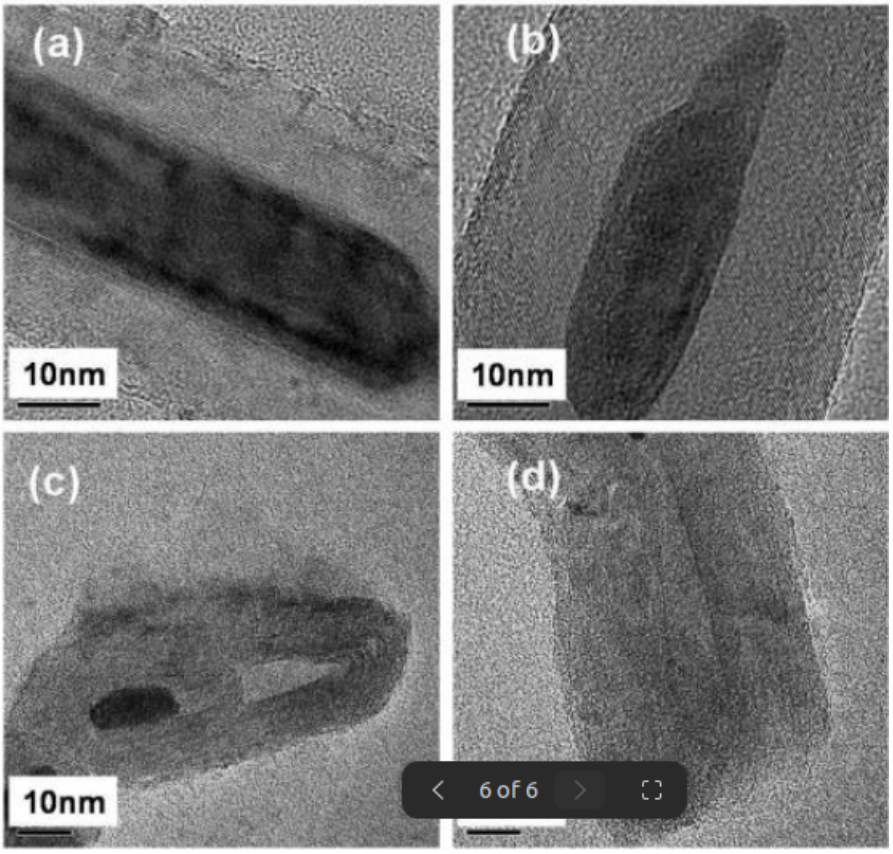
This images is taken with 500x magnification with HFW of 0.28 mm,and working distance of 16.9 mm.

3.3 Images of Second page:



The images are taking with same Magnification, Accelerating voltage and same WD. But with different HFW. In this images we can see that there are CNT with single walls and having a clump of atoms of some element (As Prof. direction it is Iron(Fe)) and there are made clump of nano size.

3.4 Images of Third page:



In this images we iron atoms clump are of diameter of about 10 nm and length of about 50 60 nm. And CNT have diameter of about 30 nm.