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Comprehensive Investigation Of Electron Microscopy Techniques For Structural Characterization Of Single-Walled Carbon Nanotubes

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Abstract

The present paper focuses on the growth of single-walled carbon nanotubes (SWCNTs) for device-oriented applications. Carbon nanotubes are widely regarded as one of the most promising materials of the 21st century due to their exceptional physical, chemical, and electronic properties. These materials exhibit significant potential across a broad range of applications, including highly sensitive gas sensors, field emission devices, solar cells, and super capacitors. Various techniques have been developed for the synthesis of carbon nanotubes, such as arc discharge, laser ablation, and chemical vapour deposition (CVD). In this study, plasma-enhanced chemical vapour deposition (PECVD) has been employed to achieve the controlled growth of well-aligned and high-quality SWCNTs. This technique enables the fabrication of superior nanostructures suitable for advanced device applications. Considering the remarkable properties and structural quality of SWCNTs, The as-grown SWCNTs were systematically characterized using field emission scanning electron microscopy (FESEM), high-resolution transmission electron microscopy (HRTEM), and Raman spectroscopy to evaluate their morphology, structural integrity, and quality. Furthermore, their performance in field emission and gas sensing applications was investigated. A comprehensive discussion of the experimental results and analyses is presented in this paper-wise manner, as outlined in the following sections.

Keywords *– Carbon Nanotubes (CNTs), Single-Walled Carbon Nanotubes (SWCNTs), Plasma-Enhanced Chemical Vapour Deposition (PECVD), Field Emission Scanning Electron Microscopy (FESEM), High-Resolution Transmission Electron Microscopy (HRTEM), Raman Spectroscopy, Surface Morphology, Nanomaterial's Characterization. Gas Sensors, Chemiresistive Sensors, Field Emission Properties, Nanotechnology*

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I. Introduction

Nanotechnology is the study of materials at the range of 1-100nm dimension. This technology is changing the face and structure of the world. The materials show enhanced and different properties at this Nano range. This technology includes all the subjects of the modern world like physics, chemistry, bio and other subjects. Every sphere of life is being touched by the nanotechnology. It is the understanding and control of properties of the material at the range of 1-100nm Different novel applications are being explored and enhanced at this dimension. The properties of nanomaterial differ in a fundamental way as compared to bulk materials. The experimental study shows, the colour of bulk gold particles is yellow, the same gold particles at about 90nm range show green colour and the particles at 30nm range shows red colour. The reason behind this particular phenomenon is the interaction of electron cloud of metals with photons of light falling on it. The nanoparticles at the Nano range dimension, the electron cloud begins to resonate with different wavelengths of light.

The main objectives of this research paper are

- Growth of CNTs through PECVD technique on Si substrate at low temperature.
- Deposition of Fe (Iron) catalyst for the growth of CNTs by RF Sputtering Technique.
- Characterization of as grown SWCNTs by various techniques like Field Emission Scanning Electron Microscope (FESEM),
- High Resolution Transmission Electron Microscope (HRTEM) and Raman Spectroscopy.
- Fabrication of Sensitive Chemiresistive sensors for the detection of toxic Gases like NH_3, PG, acetone methanol and NO_2 at trace level detection.

- To study various characteristics of sensors like fast response/recovery time, repeatability, selectivity, stability for long term application
- To enhance the sensor response by attaching various nanoparticles on CNTs surface

II. Complete Literature Of Carbon Nanotubes

Carbon nanotubes are built from SP2 carbon units and encompass honeycomb lattices and are an unbroken shape. They're tubular having a diameter of a few nanometers however lengths of many microns. MWCNTs are closed graphite tubules rolled like a graphite sheet. Diameters usually variety among 2 and 25 nm and unmarried-walled carbon nanotubes '(SWCNT) are made from a single seamlessly rolled graphite sheet with a regular diameter of approximately Four nm that is much like a buck ball (C60). They have a propensity to form in bundles that are parallel in contact and consist of tens to hundreds of nanotubes. Relying on how the graphene partitions of the nanotube are rolled collectively they are able to result in an armchair, Zigzag or chiral shapes (fig.1). These companies are outstanding by using their unit cells that are decided by using the chiral vector given by means of the equation: Ch= na1 + ma2 where a1 and a2 are unit vectors inside the -dimensional hexagonal lattice, and n and m are integers. Every other vital parameter is the chiral angle, which is the perspective among Ch and a1. When n = m and the chiral perspective is three hundred tiers it's miles called an armchair kind. Whilst m or n are zero and the chiral angle is same to zero the nanotube is called zigzag. Chiral nanotubes are therefore while the chiral angles are between 0 o and 30^0

Properties Of Carbon Nanotubes

Carbon nanotubes (CNTs) are considered as unique nanostructures which differ in properties as compared to other bulk materials of carbon. These extra ordinary properties are leading the CNTs are 21th century material. The elastic modulus of CNTs ranges from 1TPa and showing strength 100 times larger than the stain less steel. In case of electricity, they are conducting electricity 1000 times greater than the best conductor cooper. They are showing a high quality of strength and flexibility. On the basis of theses extraordinary properties of CNTs, they have various potential applications.

These various potential applications include solar cell, Chemi resistive sensors, field emitters, textiles, and capacitors etc. The devices are showing better efficiencies and size reduction. As they are seamless cylinders of carbon and are being used in various medical sectors also. Some main applications have been discussed in application medically they well adjustable in various branches. The needle like structures of CNTs is being widely used in medical sector. They are finding the applications as per the geometry of the CNTs. Vertically aligned CNTs are best candidates for field emitters while randomly oriented CNTs are suitable for Chemi resistive sensors. On the bases of rolling of grapheme sheet, the following structures are obtained and has been shown in fig.1.

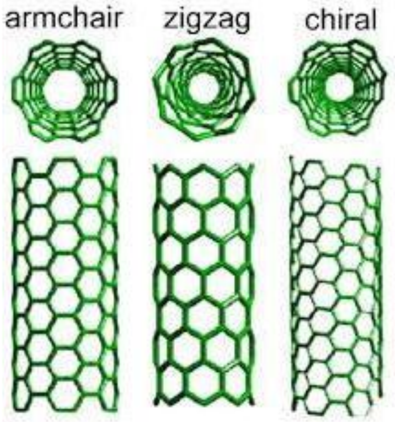

Fig.1. Rolling of grapheme to form CNTs

Classification Of Carbon Nanotubes

Carbon nanotubes are classified in following two types:
I.SWCNTs-Single-walled carbon nanotubes
II. Double wall Carbon Nanotubes
III. MWCNTs-Multiple-walled carbon nanotubes.

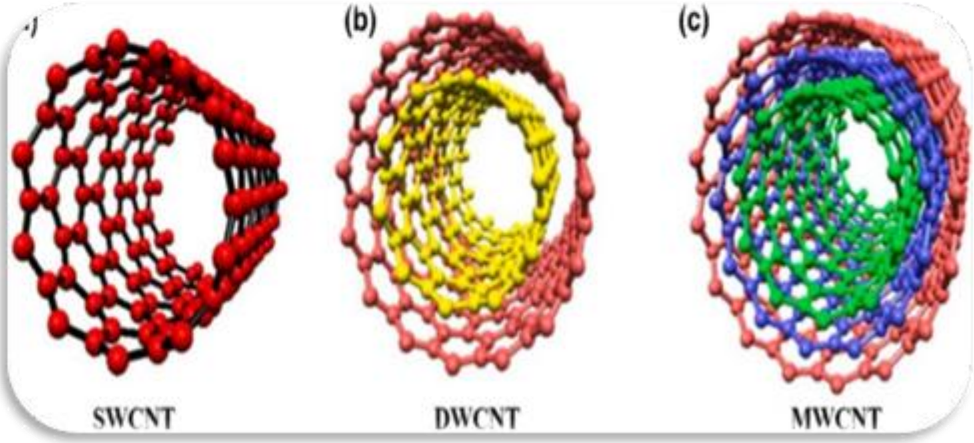

Fig.2 (a) SWCNTs, (b) DWCNTs (c) MWCNTs

III. Characterization Techniques

The Raman Spectroscopy:

Raman Spectroscopy technique is excellent for obtaining vibrational modes of the sample in a proper way. This technique was discovered by a great scientist Dr. CV. Raman. Dr. CV Raman was an Indian scientist and has got noble prize in discovering this spectroscopy technique. This technique is used by scientific community in world-wide and provides the best information regarding the sample_s vibrational modes and sample composition. It provides different peaks and every peak explain the different information regarding the sample. It works on simple mechanism of scattering of light. This mechanism involves the simple interaction of light with molecules of liquid, gas and solid. After the interaction major number of photons disperses at the same energy level as compared to incident photons. This phenomenon is known as elastic or Rayleigh scattering. From these incident photons, a very few photons will disperse with different frequency as compared to incident photon. This phenomenon is known as inelastic scattering or the Raman Effect. This name Raman Effect was given to Dr. C.V. Raman after he discovered this excellent technique. For this work, Dr. C.V. Raman was awarded the 1930 Nobel Prize in Physics. This technique has variety of applications in different fields. This field includes medical sciences and material sciences. This technique involves the collection of vibrational modes and interaction of molecules. The mechanism of this technique has been shown in fig. 3.

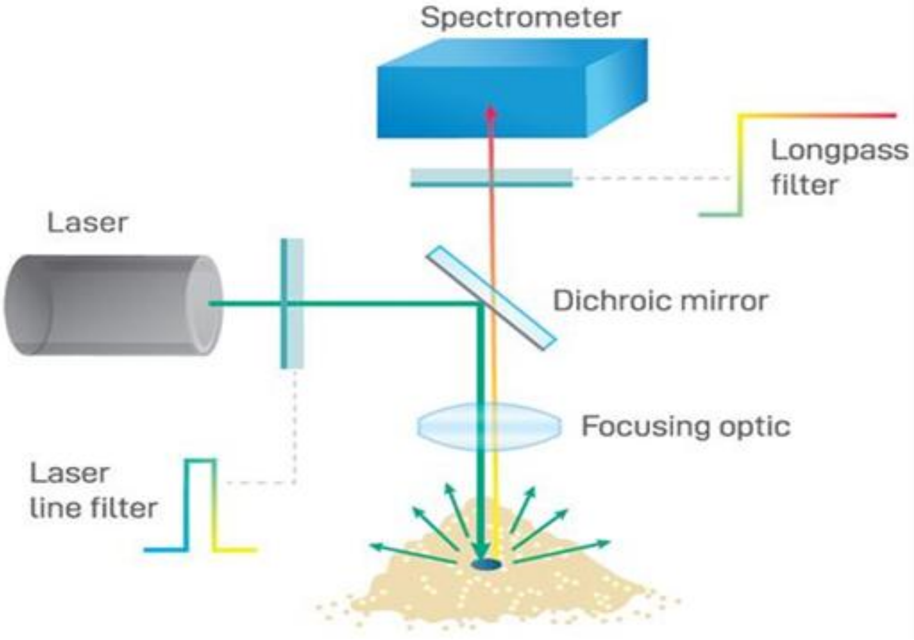

Fig.3. Raman Spectroscopy Mechanism

The different components of this technique have been shown in fig 3.1 properly. The sample is illuminated by the laser light and the interactions happen with the molecules of the sample. After the interaction the scattering and inelastic scattering are being recorded and analysed properly for the required information about the sample. The filter is being used to reflect shortwave laser light on to the sample surface and transmit the properly Raman scattered light via spectrometer. Another filter is being used to block the Raleigh scattering. Sampling optics for Raman systems come in various optical configurations: some use fiber optic coupling to allow the sampling optics to be integrated into a probe for use at a distance from the spectrometer,

While others use fully integrated sampling optics to reduce size and optical losses within the system. In simple, Raman spectrometer is able to capture and detect the all-transmitted light signals via sample surface. After that, a spectrum is obtained as a function of Raman shift verse laser frequency. This requires sufficient range, signal strength, and optical resolution. Key performance properties include high sensitivity, a good signal-to-noise ratio, and a high light collection power, expressed as the numerical aperture (NA). Spectrometer range is another important parameter to consider. This refers to the Raman shift frequency range, which is typically broken up into the fingerprint region (<1500 cm^{-1}), used for material identification, and the functional region (<3600 cm^{-1}), which provides additional chemical bond information used in research and specialized applications. The Raman mechanism has been shown in fig. 3. In this research part, the Raman spectroscopy has been used in determining the diameter of the CNTs.

Scanning Electron Microscope (SEM)

Scanning Electron Microscope working mechanism: This excellent technique to obtain the surface morphology of the samples. This technique involves the scanning process for obtaining the surface morphology of samples. The surface is being scanned property at high resolution for getting the clear and vivid surface morphology of the required sample. The sample is being scanned through a highly intense beam of electrons. The beam interacts with the surface and acquires the required information regarding the surface of the sample.

The signals are being recorded by detectors and analysed on the attached screen for obtaining excellent image. The field emission scanning electron microscopes (FESEM) are mostly used in modern days. They are able to scan the sample surface at higher level and the micrographs so obtained are highly informative and visible.

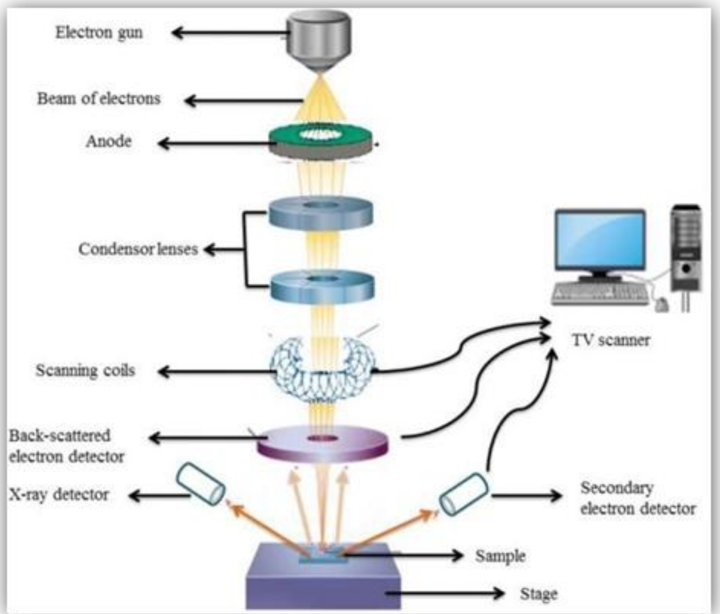

Fig.4. Scanning Electron Microscope (SEM)

Electron Microscope is in the form of a tall vacuum column that is vertically mounted. It involves the following components:

Electron gun

The electron gun consists of a heated tungsten filament, which generates electrons. The electrons are being generated at high intensity and are being projected on the surface of the sample.

Electromagnetic lenses

The condenser lens usually used to focus the electron beam on the specimen. A second condenser lens forms the electrons into a thin tight and intense beam.

- The electron beam are coming out of the specimen and passes down the second of magnetic coils called the objective lens, which has high power and forms the intermediate magnified image.
- The third set of magnetic lenses called **projector (ocular) lenses** produce the final further magnified image.
- Each of these lenses acts as an image magnifier all the while maintaining an incredible level of detail and resolution.

Specimen Holder

- The specimen holder is an extremely thin film of carbon or collodion held by a metal grid.

Image viewing and Recording System

- The final image is projected on a fluorescent screen.
- Below the fluorescent screen is a camera for recording the image.

Transmission Electron Microscope

Transmission electron microscope is an excellent technique for obtaining the internal structures of the samples. It is commonly known as high resolution transmission electron microscope (HRTEM). This technique involves the transmission of electron beam through the samples for obtaining the internal structures with magnification. The working principle of HRTEM has been shown in fig.. The different parts of this technique has been shown and explained one by one. Every part is important and specific in this technique. The intense electron beam transmits through the sample and is projected on to the photo sensitive screen. The micrograph is being analysed properly on the screen and is being saved with good magnification for future study. It is a costly instrument for analyzing the different samples. This techniques provides us the high quality micrographs of internal structures and has high resolution.

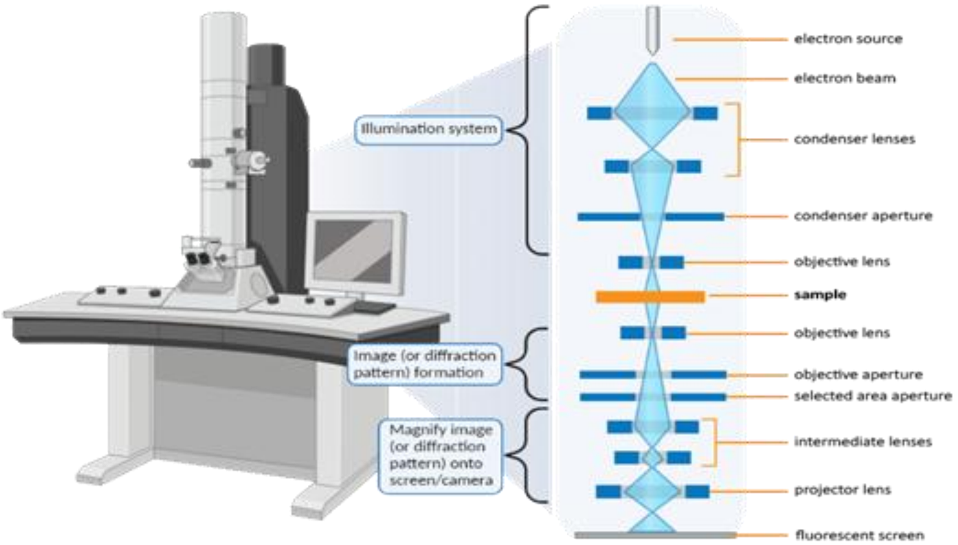

Fig. 5 Transmission Electron Microscope (TEM)

The technique is widely used for different nanomaterial. The different parts with specific work have been shown in fig. 5. The main working parts includes

Electron gun

This part is being used to generate high intense beam of electrons which is responsible for obtaining the information of the sample.

The electrons are being produced by a cathode which is known as tungsten filament and is usually in V shaped and is heated when it is in operational form. The cathode is always negatively charged.

The electron beam is subjected to transmit through the sample with high voltage with a definite constant energy. The beam is being focussed through the specimen; this beam produces the high quality images of the sample.

The beam passes through different condenser lenses which focus the beam with intensity to create or produce the excellent micrographs of the sample.

Image- Producing system

- These parts are always made up of objective lenses with movable stage and are attached with intermediate and projector lenses. They focus the electron beam via specie and form highly magnified images. The objective lens which has shorter focal length of about 1-5nm which produces an intermediate image and then these images are transmitted to projector lenses which lead the magnification of images.
- In HRTEM, The projector lenses are classified into two types (i) intermediate and (ii) Projector lens, the intermediate lens produce good images but projector lenses produces the images at high magnification.
- To obtained excellent images both the lenses require high power supplies with good stability for better resolution of the images.

Image-Recording System

- This part of the HRTEM is highly sensitive and important; it records the images and the information regarding the images.
- This part is made up of a fluorescent screen which is used to view and focus the images. This part is also having the digital cameras which records the images and save it permanently.
- This part has a vacuum system so that they will capture the images without disturbing and pure qualities of images are being obtained. With the help of vacuum pure and excellent type of images are recorded.
- The high quality of vacuum system is obtained with the help of pumps, gauge and power supply
- The image obtained is so called monochromatic images which are usually black or white
- To make these images visible to human eyes, the electrons are allowed to pass via fluorescent screen fixed at the base of the microscope.
- The micrographs are captured digitally on screen/computer in the form of JPEG or TIFF format During the time of storage, the micrographs are manipulated from its monochromatic

IV. Conclusion

This study presents a systematic investigation into the growth and characterization of single-walled carbon nanotubes (SWCNTs) synthesized using the plasma-enhanced chemical vapour deposition (PECVD) technique. The controlled deposition of iron (Fe) catalyst using RF sputtering enabled the formation of well-aligned and high-quality CNT structures on silicon substrates at relatively low temperatures. Comprehensive characterization using advanced electron microscopy techniques such as FESEM and HRTEM provided detailed insights into the surface morphology, alignment, and internal structure of the synthesized nanotubes. FESEM analysis confirmed the uniform distribution and vertical alignment of CNTs, while HRTEM revealed their crystalline structure and nanoscale diameter. Raman spectroscopy further validated the structural integrity and quality of SWCNTs through characteristic peaks, indicating minimal defects and high graphitization. The fabricated CNT-based chemiresistive sensors demonstrated promising performance in detecting toxic gases such as NH_3, NO_2, acetone, methanol, and PG at trace levels. The sensors exhibited fast response and recovery times, good selectivity, repeatability, and long-term stability. Additionally, surface functionalization with nanoparticles significantly enhanced sensing performance. Overall, the integration of precise growth techniques with advanced characterization methods confirms that SWCNTs are highly suitable for next-generation Nano electronic and sensing applications.

V. Future Scope

The present work opens several avenues for further research and technological advancements:

Advanced Functionalization: Future studies can focus on functionalizing CNTs with metal and metal oxide nanoparticles (such as Au, Ag, ZnO) to further enhance sensitivity and selectivity for specific gases.

Integration with AI/ML: Incorporating machine learning algorithms for pattern recognition in gas sensing data can improve detection accuracy and enable smart sensing systems.

Flexible and Wearable Sensors: Development of flexible, lightweight, and wearable CNT-based sensors for real-time environmental and biomedical monitoring.

Hybrid Nanostructures: Exploration of CNT-based hybrid materials (e.g., graphene-CNT composites) for improved electrical and mechanical properties.

Miniaturization and Device Integration: Integration of CNT sensors into microelectronic and Nano electronic systems for compact and portable sensing devices.

- Energy Applications: Investigation of CNTs in energy storage devices such as supercapacitors and batteries for enhanced efficiency.
- High-Resolution Characterization: Use of more advanced techniques such as Atomic Force Microscopy (AFM) and X-ray Photoelectron Spectroscopy (XPS) for deeper surface and chemical analysis.
- Scalability and Industrial Application: Optimization of PECVD processes for large-scale, cost-effective production of CNTs for industrial applications.

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