# CHARACTERIZATION OF CARBON NANOTUBES SYNTHESIZED USING MAGNETIC FIELD ASSISTED ARC DISCHARGE TECHNIQUE

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# CHARACTERIZATION OF CARBON NANOTUBES SYNTHESIZED USING MAGNETIC FIELD ASSISTED ARC DISCHARGE TECHNIQUE

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Dedicated to my beloved mother, father, wife,
lecturers, and all my friends.……

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# ABSTRACT

The unique properties of multi-walled carbon nanotubes (MWCNTs) attract enormous attentions as promising nanomaterials for numerous applications. In this research, the growth of MWCNTs using direct current (DC) arc discharge plasma under magnetic field effect in different ambient environments and pressures were investigated. Arc plasma was generated inside a vacuum chamber by contact ignition between two graphite rods. A DC power supply was used to supply an arc current of 70 A and a voltage of 12 V across the graphite rods. Magnetic field was applied in transverse and axial configurations with field strength of 30 mT. Arc discharge experiments were conducted in air, hydrogen and argon environment for different ambient pressures of $10^{-2}$, $10^{-1}$, 1, $10^{1}$ and $10^{2}$ mbar. The structural and physical properties of MWCNTs were characterized using Transmission Electron Microscopy, Field Emission Scanning Electron Microscopy, Raman spectroscopy, Fourier Transform Infra-red spectroscopy and X-Ray diffractometry. Results showed that MWCNTs growth in arc discharge plasma was significantly influenced by the ambient environment, gas pressure, axial and transverse magnetic field. The applied transverse magnetic field on arc discharge plasma enhanced the growth of MWCNTs with smaller tube dimeter. The axial magnetic field on the other hand allowed the growth of long tubular structure with lesser impurities as observed under electron microscope. Results from Raman spectroscopy showed that high rise of G band as compared with D band indicates high rise of graphitic structure of MWCNTs. The MWCNTs quality was measured in terms of intensity ratio of the peak D band to the peak G band. High quality MWCNTs with less defective sites were obtained for sample prepared in hydrogen environment. The increase in the length of the MWCNTs was observed for sample prepared in the presence of inert gas argon. In conclusion, high quality MWCNTs were successfully synthesized using magnetic field assisted arc discharge technique.

# ABSTRAK

Keunikan sifat pelbagai lapisan karbon tiub nano (MWCNTs) menarik banyak perhatian sebagai bahan nano yang unggul untuk pelbagai kegunaan. Dalam penyelidikan ini, pertumbuhan MWCNTs menggunakan arus terus (DC) nyahcas plasma arka dibawah kesan medan magnet dalam persekitaran dan tekanan berbeza telah diselidiki. Plasma arka yang terjana didalam ruangan vakum melalui cucuhan sentuhan antara dua rod grafit. Sumber kuasa DC digunakan untuk menyalurkan arus arka 70 A dan voltan 12 V merentasi rod grafit. Medan magnet dikenakan secara konfigurasi melintang dan menegak dengan kekuatan medan sebanyak 30 mT. Eksperimen nyahcas arka dijalankan dalam persekitaran udara, hidrogen, dan argon pada tekanan berbeza iaitu $10^{-2}$, $10^{-1}$, 1, $10^{1}$ dan $10^{2}$ mbar. Struktur dan sifat fizikal MWCNT dicirikan menggunakan Mikroskop Transmisi Elektron, Mikroskop Pengimbas Medan Pelepasan Elektron, Spektroskopi Raman, Spektroskopi Transformasi Fourier Infra-Merah dan pembelauan Sinar-X. Keputusan menunjukkan MWCNTs tumbuh melalui nyahcas plasma arka amat dipengaruhi oleh faktor persekitaran, tekanan gas, medan magnet melintang dan menegak. Medan magnet secara melintang dikenakan pada nyahcas plasma arka meningkatkan pertumbuhan MWCNTs dengan diameter tiub bersaiz kecil. Selain itu, kesan medan magnet secara menegak menghasilkan struktur tiub yang panjang dengan bendasing yang rendah seperti yang diperhatikan dibawah mikroskop elektron. Keputusan spektroskopi Raman menunjukkan kenaikan tinggi puncak G berbanding puncak D menunjukkan kadar struktur grafitik yang tinggi pada MWCNTs. Kualiti MWCNTs diukur dengan menentukan nisbah keamatan puncak jalur D kepada puncak jalur G. MWCNTs berkualiti tinggi dengan kecacatan yang rendah diperoleh bagi sampel yang dihasilkan didalam persekitaran hidrogen. Peningkatan panjang MWCNTs dapat dilihat pada sampel yang dihasilkan dalam gas lengai argon. Kesimpulannya, MWCNTs berkualiti tinggi telah berjaya disintesiskan menggunakan kesan medan magnet berbantukan teknik nyahcas plasma arka.

# TABLE OF CONTENTS


| CHAPTER | TITLE | PAGE |
|---|---|---|
| | **DECLARATION** | ii |
| | **DEDICATION** | iii |
| | **ACKNOWLEDGEMENT** | iv |
| | **ABSTRACT** | v |
| | **ABSTRAK** | vi |
| | **TABLE OF CONTENTS** | vii |
| | **LIST OF TABLES** | x |
| | **LIST OF FIGURES** | xi |
| | **LIST OF SYMBOLS** | xvii |
| | **LIST OF ABBREVIATIONS** | xix |
| | **LIST OF APPENDICES** | xx |
| **1** | **INTRODUCTION** | |
| | 1.0. Background of Study | 1 |
| | 1.1. Problem Statement | 2 |
| | 1.2. Objectives | 4 |
| | 1.3. Scope of Study | 4 |
| | 1.4. Significance of study | 4 |
| | 1.5. Thesis Outline | 5 |

**2 LITERATURE REVIEW**

2.0. Introduction 6
2.1. Carbon Allotropes 7
2.2. Carbon Nanotube and Related Carbon-Based Product 9
2.3. Carbon Nanotube Physical Properties 10
2.4. Carbon Nanotube Growth Method 14
2.4.1. Catalyst Assisted Carbon Nanotube Growth 16
2.4.2. Carbon Nanotube Growth on Substrate 18
2.5. Carbon Nanotube Electrical And Mechanical Properties 20
2.6. Filling and Functionalize of Carbon Nanotube 22
2.7. Application of Carbon Nanotube 23
2.8. Experimental Work Development 25
2.8.1. Arc Discharge Plasma 26
2.8.2. Inter-electrode Phenomena 27
2.8.3. Parametric Studies of Carbon Nanotube Growth 30
2.8.4. Effect of Direct Current Electric Field 30
2.8.5. Effect of Applied External Magnetic Field 31
2.9. Sample Analysis of Carbon Nanotube Structure 32
2.10. Modeling and Simulation Development 33

**3 RESEARCH METHODOLOGY**

3.1. Introduction 37
3.2. Experimental Equipment 37
3.2.1. Reaction Chamber 38
3.2.2. Vacuum system 39
3.2.3. Gas Regulatory System 42
3.2.4. Power Supply 44
3.2.5. Graphite Rod 45
3.2.6. Permanent Magnet 45
3.2.7. Motorized Stage 47
3.3. Experimental Procedure 48

3.3.1. Assembly Fitting 48

3.3.2. Controlled Environment 52

3.3.3. Arcing Process 52

3.3.4. Sample Collection 53

3.4. Research Flow Chart 56

**4 RESULTS AND DISCUSSION**

4.1. Introduction 57

4.2. Electron Microscopy 57

4.2.1. Transmission Electron Microscopy 57

4.2.2. Deviation of Nanoparticle Size 71

4.2.3. Field Emission Electron Microscopic Analysis 75

4.3. Spectroscopic Characterization of Synthesized CNTs 90

4.3.1. Raman Spectroscopy Analysis 90

4.3.2. Fourier Transform Infrared Spectroscopy 99

4.4. X-Ray Diffraction Analysis 104

4.4.1.D-Spacing MWCNT 109

4.4.2.Crystallite Size 112

**5 CONCLUSION AND RECOMMENDATION**

5.1. Conclusion 116

5.2. Recommendation for Future Study 117

**REFERENCES** 119

Appendix A-D 130-140

# LIST OF TABLES


| TABLE NO. | TITLE | PAGE |
|---|---|---|
| 3.1 | Parameters and ambient environment for arc discharge process to synthesize carbon nanotube. | 55 |
| 4.1 | Statistic data for carbon nanotube diameter and length grow under different ambient condition measured by TEM. | 72 |
| 4.2 | Statistic data for carbon nanotube diameter and length grow under different ambient condition measured by FESEM. | 88 |
| 4.3 | Raman features of CNT in range between 1200 and 1800 $cm^{-1}$. | 95 |
| 4.4 | Distribution of XRD diffraction plane (002) and plane spacing $d_{(002)}$ in different environment of arc discharge plasma. | 110 |
| 4.5 | Crystallite size calculated using Debye-Scherrer equation in different applied ambient environment. | 113 |

# LIST OF FIGURES


| FIGURE | CAPTION | PAGE |
|---|---|---|
| 2.1 | $Sp^2$ configuration of carbon in honeycomb lattice layer. | 8 |
| 2.2 | Single-Walled Carbon Nanotube | 9 |
| 2.3 | Geometric structure of an (n,m) single walled carbon nanotube. | 11 |
| 2.4 | Graphene layer specifies triangular coordination of carbon atom identifying types of carbon nanotube upon rolled across dotted line. | 12 |
| 2.5 | Three main type of carbon nanotube; a) armchair, b) zig-zag, and c) chiral formed with hemispherical capped at both end. | 13 |
| 2.6 | Electronic band structure of and density of state for (a) (10, 10) armchair carbon nanotube showing metal characteristic and (b) (20, 0) zig-zag carbon nanotube with the gap between conduction and valence band showing semiconductor characteristics [42]. | 14 |
| 2.7 | Nano-particle catalyst deposition. | 19 |
| 2.8 | Image of brain scan using SWCNT | 25 |
| 2.9 | Inter-electrode gap between two graphite electrodes. | 27 |
| 3.1 | (a) Stainless steel reaction chamber and (b) schematic diagram of reaction chamber. | 38 |
| 3.2 | Vacuum system | 39 |
| 3.3 | Diffstak 100 Diffusion pump. | 40 |

| | | |
|---|---|---|
| 3.4 | RV5 Rotary vane pump. | 40 |
| 3.5 | Wide Range Gauge pressure gauge. | 41 |
| 3.6 | Pirani gauge. | 41 |
| 3.7 | TIC instrument controller pressure display meter. | 42 |
| 3.8 | Mass flow controller for (a) Hydrogen and (b). Argon | 43 |
| 3.9 | Flowmeter channel readout. | 43 |
| 3.10 | Double stage gas regulators. | 44 |
| 3.11 | DC power supply. | 44 |
| 3.12 | Anode and Cathode graphite electrodes. | 45 |
| 3.13 | Permanent magnet; (a) disc and (b) ring magnet. | 46 |
| 3.14 | Magnet configuration (a) transverse and (b) axial. | 47 |
| 3.15 | One dimensional motorized translational stage with minimum step 5nm. Inset, motor controller model TDC001. | 47 |
| 3.16 | Full system of arc discharge plasma. | 49 |
| 3.17 | Block diagram of experimental setup | 50 |
| 3.18 | Fitting assembly inside vacuum chamber. | 51 |
| 3.19 | (a)Transverse and (b) Axial magnet configuration. | 52 |
| 3.20 | Arc discharge plasma. | 53 |
| 3.21 | Cathode and anode after discharge process. | 53 |
| 3.22 | CNT powder grained from cathode deposit. | 54 |
| 3.23 | Flow chart of research methodology for synthesis carbon nanotube by arc discharge technique in different applied conditions. | 56 |
| 4.1 | TEM micrograph with different nano-structures. | 58 |
| 4.2 | TEM micrographs of Carbon nanotubes samples prepared in Air for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field. | 59 |
| 4.3 | TEM micrographs of Carbon nanotubes samples prepared in $H_2$ for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field. | 60 |

| | | |
|---|---|---|
| 4.4 | TEM micrographs of Carbon nanotubes samples prepared in Ar for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field.. | 62 |
| 4.5 | TEM micrographs of Carbon nanotubes samples prepared in Air for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of transverse magnetic field. | 63 |
| 4.6 | TEM micrographs of Carbon nanotubes samples prepared in $H_2$ for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of transverse magnetic field. | 65 |
| 4.7 | TEM micrographs of Carbon nanotubes samples prepared in Ar for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of transverse magnetic field. | 66 |
| 4.8 | TEM micrographs of Carbon nanotubes samples prepared in Air for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 67 |
| 4.9 | TEM micrographs of Carbon nanotubes samples prepared in $H_2$ for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 69 |
| 4.10 | TEM micrographs of Carbon nanotubes samples prepared in Ar for ambient pressures (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 70 |
| 4.11 | Average carbon nanotube diameter and length growth in different condition applied measured from TEM. | 73 |
| 4.12 | FESEM micro graphs of **c**arbon nanotube in Air ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field. | 76 |
| 4.13 | FESEM micro graphs of **c**arbon nanotube in $H_2$ ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field. | 77 |

| | | |
|---|---|---|
| 4.14 | FESEM micro graphs of **c**arbon nanotube in Ar ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in absence of magnetic field. | 79 |
| 4.15 | FESEM micro graphs of **c**arbon nanotube in Air ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of external transverse magnetic field. | 80 |
| 4.16 | FESEM micro graphs of **c**arbon nanotube in $H_2$ ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of external transverse magnetic field. | 81 |
| 4.17 | FESEM micro graphs of **c**arbon nanotube in Ar ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of external transverse magnetic field. | 82 |
| 4.18 | FESEM micro graphs of **c**arbon nanotube in Air ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 84 |
| 4.19 | FESEM micro graphs of **c**arbon nanotube in $H_2$ ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 85 |
| 4.20 | FESEM micro graphs of **c**arbon nanotube in Ar ambient environment at pressure (a) $10^{-2}$ mbar, (b) 1 mbar and (c) $10^{2}$ mbar in presence of axial magnetic field. | 86 |
| 4.21 | FESEM micrograph and EDX spectrum of CNTs samples | 87 |
| 4.22 | Raman spectra for CNTs grown in different ambient pressures $10^{-2}$, $10^{-1}$, 1, $10^{1}$ and $10^{2}$ mbar and absence of magnetic field under ambient environments (a) Air (b) $H_2$ and (c) Ar | 91 |
| 4.23 | Raman spectra for CNTs grown in different ambient pressures $10^{-2}$, $10^{-1}$, 1, $10^{1}$ and $10^{2}$ mbar and presence of | 93 |

| | | |
|---|---|---|
| | magnetic field with transverse configuration under ambient environments (a) Air (b) $H_2$ and (c) Ar. | |
| 4.24 | Raman spectra for CNTs grown in different ambient pressures $10^{-2}$, $10^{-1}$, 1, $10^{1}$ and $10^{2}$ mbar and presence of magnetic field with axial configuration under ambient environments (a) Air (b) $H_2$ and (c) Ar. | 94 |
| 4.25 | $I_D/I_G$ of Raman spectra for CNTs grown in different ambient pressures $10^{-2}$, $10^{-1}$, 1, $10^{1}$ and $10^{2}$ mbar for (a) in absence of magnetic field, (b) in presence of magnetic field and (c) axial magnetic field configuration under different ambient environments; Air, $H_2$ and Ar. | 97 |
| 4.26 | FTIR spectra of sample synthesized in absence of magnetic field under (a) air (b) hydrogen and (c) argon ambient environment | 100 |
| 4.27 | FTIR spectra of sample synthesized in presence transverse magnetic field under (a) air and (b) hydrogen and (c) argon ambient environment. | 101 |
| 4.28 | FTIR spectra of sample synthesized in presence axial magnetic field under (a) air (b) hydrogen and (c) argon ambient environment. | 102 |
| 4.29 | XRD pattern for carbon arc discharge sample in Air in absence of magnetic field under different pressure | 103 |
| 4.30 | XRD spectrum of CNT samples in absence of magnetic field under different ambient environment. | 105 |
| 4.31 | XRD spectrum of CNT samples in presence of transverse magnetic field under different ambient environment | 106 |
| 4.32 | XRD spectrum of CNT samples in presence of axial magnetic field under different ambient environment. | 107 |
| 4.33 | $d_{(002)}$ spacing distribution in absence of magnetic field, $B_0$, in presence of transverse magnetic field, $B_T$, and axial | 111 |

magnetic field, $B_A$ in three different ambient environment; (a) air, (b) hydrogen, and (c) argon

4.34 Carbon nanotube wall in synthesized in different ambient environment without magnetic field, $B_0$, with applied transverse magnetic field, $B_T$ and with applied axial magnetic field, $B_A$. 114

## LIST OF SYMBOLS

| | | |
|---|---|---|
| *A* | - | Ampere |
| *V* | - | Voltage |
| *DC* | - | Direct Current |
| $B_0$ | - | In absence of magnetic field |
| $B_T$ | - | In presence of transverse magnetic field |
| $B_A$ | - | In presence of axial magnetic field. |
| *mbar* | - | milibar |
| $S\ m^{-1}$ | - | Siemen per meter |
| *GPa* | - | GigaPascal |
| *nm* | - | nanometer |
| $C_h$ | - | Chiral vector |
| *J* | - | Current density |
| $\mathbf{B}$ | - | Magnetic field |
| $\lambda_{eff}$ | - | Effective mean free path electron |
| $\pi^*$ | - | Delocalized electron orbital |
| $E_F$ | - | Fermi Energy |
| $\Gamma$ | - | Mass flux rate |
| $p_{sat}$ | - | Saturation pressure |
| $I_D$ | - | Raman D-band Intensity |
| $I_G$ | - | Raman G-band Intensity |
| $d_{002}$ | - | 002 plane spacing |
| $H_2$ | - | Hydrogen |
| *Ar* | - | Argon |
| $P_{kin}$ | - | Kinetic Pressure |

| | | |
|---|---|---|
| $n$ | - | density |
| $k$ | - | Boltzmann constant |
| $T$ | - | Temperature |

## LIST OF ABBREVIATION

| | | |
|---|---|---|
| *CNT* | - | Carbon Nanotube |
| *SWCNT* | - | Single-Walled Carbon Nanotube |
| *DWCNT* | - | Double-Walled Carbon Nanotube |
| *CVD* | - | Chemical Vapor Deposition |
| *PECVD* | - | Plasma Enhanced Chemical Vapor Deposition |
| *TEM* | - | Transmission Electron Microscopy |
| *FESEM* | - | Field Emission Scanning Electron Microscopy |
| *FTIR* | - | Fourier Trasnform Infra-Red |
| *XRD* | - | X-Ray Diffraction |
| *TGA* | - | Thermogravimetric Analysis |
| *DTA* | - | Derivative Thermogravimetric Analysis |
| *0D* | - | Zero Dimensional |
| *1D* | - | One Dimension |
| *3D* | - | Three Dimension |

# LIST OF APPENDICES


| APPENDIX | TITLE | PAGE |
|---|---|---|
| A | Carbon nanotube diameter and length measured by TEM | 126 |
| B | Metal polish used to clean the vacuum chamber. | 134 |
| C | Computer interface to measure distance travel by electrode and time elapse | 135 |
| D | Tesla meter used to measure magnetic field strength. | 136 |

# CHAPTER 1

# INTRODUCTION

## 1.0 Background of study

Nanotechnology has brought up a new era of fascinating atomic scale study. While pursuing technological advances, carbon element come out with spectacular nanostructure designated in 0D, 1D, and 3D honeycomb lattice structure. Carbon nanotube is one of the carbon allotropes build from 1D $sp^2$ hybridized bonding of carbon atom. Carbon nanotube was accidentally founded in 1991 during an arc discharge process of synthesize buckyball structure of carbon [1]. The unique properties of carbon has tremendously pledge researcher to exploit advantage of carbon nanotube for vast application from small scale nano size device [2] to macro size golf club [3] .

Carbon nanotube is a form of straw-like structure made of single layer of carbon atom recognized as Single Walled Carbon Nanotube (SWCNT). In different ways the carbon atom bound together and defines whether SWCNT has metallic or semiconductor properties. The tube can even exist in Multi-layer form by spacing 0.34 nm. The band gap of semiconductor nanotube depends on the configuration and diameter of the tube [4]. This remarkable structure builds up from hexagonal lattice structure of carbon atom layer bind by strong covalent bond. The delocalized $\pi$ orbital of $sp^2$ hybridized orbital enables high electron transport in carbon nanotube structure.

The unique carbon nanotube properties are its high tensile strength maximum 100 GPa [5], high thermal conductivity 2000 $Wm^{-1}$ K [6], and high electrical conductivity 10,000 S $m^{-1}$ [7]. Carbon nanotube is strong but lightweight and highly flexible. The high elasticity also makes carbon nanotube absorb high kinetic energy. Likewise this makes carbon nanotube a good material to use as functional structure which can withstand high temperature and high conductivity for electrical and electronics application and also for high durable mechanical application technology. In particular carbon nanotube structure can be modified for desirable application. Hemispherical cap can be open and the tube can be filled with nanoparticle for drugs delivery applications [8]. Chemical functional groups are able to attach with carbon nanotubes sidewall or body [9] for variety of application. Carbon nanotube composite certainly used for many applications including photovoltaic solar cells [10] and sporting goods [11], nanoprobe [12], nano-electronic integrated circuit [13], stretchable sensors [14], and nanowire for supercapacitor and battery application [15]. Carbon nanotube can unzipped into form of graphene nano ribbon for memory device and processing device application [16, 17].

Typically, there are three main techniques used to synthesize carbon nanotube; arc discharge, laser ablation, and chemical vapour deposition (CVD). In vacuum arc discharge, plasma is generated between the different potential of electrodes. This generated plasma then releases highly energetic carbon particle from anode toward cathode contact area. The high erosion of anode from arc discharge induces carbon nanotube growth. The arc discharge technique has an advantage to grow carbon nanotube efficiently as it does not require high cost such as laser and delivers in shorter time as compared to CVD process. In addition, the arc discharge technique is capable to grow straight, highly crystalline and highly graphitized carbon nanotube structure [18].

## 1.1 Problem Statement

Carbon nanotube grows in high temperature process. Many different techniques have been employed enabling growth for carbon nanotube including arc discharge [1], laser assistance grow [19], thermal growth [20] and chemical vapor deposition [21]. There are many technique that have been used to synthesize carbon nanotube. The arc discharge technique is one of the efficient technique to grow fast fine structure carbon nanotube. Arc plasma recognized as one of efficient method to grow carbon nanotube among others because it is fast and able to grow highly crystalline and graphitized tube structure. The growth phenomena of this fascinating nanostructure is still on-going debate by many researcher. However, the condition applied will give significant impact on the growth of carbon nanotube structures.

Close contact between electrodes can lead to ignition spark to generate arc plasma. The ionization of carbon releases electron creating current within electron through a plasma bridge. Highly erosion anode feed ionic carbon to grow fine nanostructure at cathode deposit. In arc discharge method, arc plasma play important role towards the growth of carbon nanostructure. The physical parameter such as applied voltage and current, external magnetic field, electrode geometry and dimension, ambient environment and pressure, have very strong influence on the dynamics of plasma thus certainly affect the growth of carbon nanotube structure.

The ionization of carbon release caused current flows within the electrons through the plasma. There are several issues regarding carbon nanotube growth in arc discharge process including entanglement, by product growth, low density grows, and lack of graphitic structure. These are caused by non-uniform plasma expansion. In the arc discharge process, the two contacting graphite electrodes by small gaps will create arc plasma which will then evaporate carbon material and deposit carbon nanotube at the cathode surface area. The effect of physical parameters including current and voltage applied, electrode dimensions, also ambient environment and pressure onto the arc plasma will affect the growth of carbon nanotube structure. The optimization of physical parameter will enhance plasma stability and contribute toward optimized growth of carbon nanotube [22].

The novelty of this study is by applying magnetic field in the axial and transverse configuration assisted onto arc plasma to grow carbon nanotube under different ambient environment and pressure. This research will focus on the grow phenomena and the physical properties of carbon nanotubes based on different physical parameters. The discharge process is carried out in different pressures ranging from $10^{-2}$ to $10^{2}$ mbar in three different environments; hydrogen, argon, and air. The growth of carbon nanotube structure in presence of magnetic field in different environment and pressure has been investigated comprehensively The nanotube samples collected will be analyzed comprehensively under different microscopic and spectroscopic techniques.

## 1.2 Objectives

The main objective of the study is to investigate in details the effect of magnetic field toward the growth of carbon nanotube by arc discharge technique in different ambient conditions. The specific objectives are to:

i. Investigate growth of carbon nanotube by magnetic assisted arc discharge process by transverse and axial magnetic field configuration with field strength 30mT.
ii. Determine the effect of ambient environment including Air, Hydrogen, and Argon and different pressures applied at $10^{-2}$, $10^{-1}$, 1, $10^{1}$, and $10^{2}$ mbar on the growth of carbon nanotube.
iii. Characterize the growth of carbon nanotube using microscopic and spectroscopic techniques.

## 1.3 Scope of Study

Experiment has been conducted with electrode made of high purity 99.999% graphite with anode dimension outer and inner diameter used were 9 mm and 5.5 mm while cathode diameter 12 mm. The hole for inner diameter has depth 5 cm. Constant Magnetic field with strength 30 mT has been applied across arc plasma in transverse and axial configuration. Three different ambient environments have been applied in this study, air, hydrogen, and argon. Five different pressure has been applied in this study, $10^{-2}$, $10^{-1}$, 1, $10^{1}$, $10^{2}$ mbar. Sample of carbon nanotube is collected on cathode deposit. The samples are analysed under electron microscopy, Raman Spectroscopy, X-Ray diffraction, and Fourier transform Spectrophotometer.

## 1.4 Significance of Study

The study significantly contributes to the understanding towards optimize growth of carbon nanotube and role of external magnetic field. The carbon nanotube structural properties and characteristic grow in different magnetic configuration applied across arc plasma in different ambient environment and pressure are outlined in this study. The combination of all carbon nanotube features and characteristic will construct a database for optimization of the technique used for further practical application.

## 1.5 Thesis Organization

The first chapter presents an overview of carbon nanotube structure, research background, problem statement, objectives, and scope of study. The second chapter provides a review of related literature on nanotube nanostructure and its physical properties, different technique to synthesize carbon nanotube, factor influence carbon nanotube growth, and the application of carbon nanotube. In the third chapter, the methodology and research framework are discussed in details. This chapter described the equipment used, experimental setup, details of reaction chamber, parameter manipulation, and experimental procedure. The fourth chapter presents the results of characterization of carbon nanotube sample by direct and indirect method using TEM, FESEM, Raman, XRD, FTIR, and TGA. Chapter five, the final chapter presents the summary of the entire study, discusses conclusion and some recommendation for future study of this unique nanomaterial.

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