CHARACTERIZATION OF MULTIWALLED CARBON NANOTUBES BY DC ARC DISCHARGE IN METHANE UNDER MAGNETIC FIELD INFLUENCE

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Specially dedicated to my wife, kids, parents and family.

Thank you for your sacrifice, patient and *dua'*.

May Allah grant all of us Jannah.

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ABSTRACT

Carbon nanotubes (CNTs) have gained many interest among researchers over the last two decades due to its remarkable mechanical, electrical, optical and thermal properties. High quality CNTs are in demand especially for application in nano electronics where CNTs are required to be in high crystallinity, straight and aligned orientation, having uniform diameter and less impurities to achieve the best performance. Literally, hydrogen gas is reported as the best buffer gas in producing high crystallinity and less impurities attached to CNTs by arc discharge method. However, it is not suitable for large scale CNTs synthesis due to unstable plasma formation. Recently, methane gas which contains hydrogen atoms is being studied in producing multiwalled carbon nanotubes (MWCNTs). This leads to the opportunity of investigating methane as buffer gas in producing high quality CNTs. On the other hand, the usage of magnetic field in arc discharge has been reported to have the ability to enhance the quality of CNTs in terms of narrow and uniform diameter as well as reducing impurities. Thus, this work presents a comparative study on the effect of three different arc discharge configurations to the yield of MWCNTs in methane environment. The first configuration known as Configuration A where no magnetic field assistance during CNTs arc discharge synthesis. Configuration B utilises four (4) magnets which are placed surrounding inter electrode gap while two (2) magnets are placed at anode for Configuration C. Arc discharge is generated at fixed 750 mbar of chamber pressure and fixed current about 60 A in the voltage range of 30 ~ 32 V. As a result, needle like shapes with straight orientation of individual MWCNTs for all configurations is observed under Scanning Electron Microscope. Narrow diameters are observed in configuration B with standard deviation of 2.71 mm followed by configuration C of 5.7 mm and configuration A of 8.05 mm. The results show the influence of magnetic field in producing MWCNTs with narrow and uniform diameter compared to no magnetic field assistance. The diameter distribution trend is confirmed by X-Ray powder diffraction results. High crystalline MWCNTs is confirmed by Transmission Electron Microscope images for configuration B with uniform MWCNTs inner diameter at average 2 nm. Raman spectrum shows low ratio of D band intensity over G band intensity at 0.53 for configuration B while configuration A at 0.79 which suggest fewer wall defects of MWCNTs produced in configuration B. Therefore, magnetic field assistance in methane arc discharge is proved to produce smaller and uniform diameter of MWCNTs with less wall defects. MWCNTs produced in this study can be further investigated in nanoelectronics applications such as nanowires and conductive nanofiller

ABSTRAK

Nanotiub karbon (CNTs) telah menarik minat di kalangan para penyelidik dalam tempoh dua dekad yang lalu disebabkan oleh ciri-ciri mekanikal, elektrik, optik dan termal yang luar biasa. CNTs berkualiti tinggi diperlukan terutamanya untuk aplikasi nanoelektronik di mana CNTs perlu mempunyai kehabluran yang tinggi, orientasi lurus dan sejajar, mempunyai diameter seragam dan kurang bendasing untuk memperoleh prestasi terbaik. Secara harfiah, gas hidrogen dilaporkan sebagai yang terbaik dalam menghasilkan kehabluran yang tinggi dan kurang bendasing yang melekat pada CNTs dengan kaedah discas arka. Walau bagaimanapun, ia tidak sesuai untuk sintesis CNTs berskala besar disebabkan pembentukan plasma yang tidak stabil. Baru-baru ini, gas metana yang mengandungi atom hidrogen mula dikaji dalam menghasilkan nanotiub karbon berbilang lapisan (MWCNTs). Ini membuka peluang kepada penyiasatan metana sebagai gas penampan dalam menghasilkan CNTs berkualiti tinggi. Sebaliknya, penggunaan medan magnet dalam discas arka dilaporkan mempunyai keupayaan untuk meningkatkan kualiti CNTs dari segi pengecilan diameter yang seragam serta mengurangkan bendasing. Oleh itu, kerja ini membentangkan perbandingan kesan medan magnet pada tiga konfigurasi berbeza terhadap penghasilan MWCNTs dalam persekitaran metana. Konfigurasi pertama disebut konfigurasi A di mana tiada sokongan medan magnet dalam penghasilan CNTs. Konfigurasi B menggunakan empat (4) magnet yang diletak sekeliling pembahagi antara elektrod sementara dua (2) magnet diletakkan pada anod sebagai Konfigurasi C. Discas arka dilakukan pada tekanan tetap 750 mbar dan arus tetap pada 60 A dalam julat voltan 30 ~ 32V. Hasilnya, imej *Scanning Electron Microscope* menunjukkan MWCNTs dihasilkan berbentuk jarum dengan orientasi lurus untuk semua konfigurasi. Diameter kecil diperhatikan dalam konfigurasi B dengan sisihan piawai pada 2.71 mm. Diikuti konfigurasi C pada 5.7 mm dan konfigurasi A pada 8.05 mm yang menunjukan pengaruh medan magnet dalam penghasilan MWCNTs dengan diameter kecil dan seragam berbanding tanpa medan magnet. Ianya juga dibuktikan dengan data *X-Ray powder diffraction* (XRD). Kehabluran MWCNTs yang tinggi dapat dilihat pada imej *Transmission Electron Microscope* untuk Konfigurasi B dengan diameter dalaman MWCNTs seragam pada purata 2 nm. Spektrum Raman menunjukkan nisbah intensiti jalur D terhadap intensiti jalur G yang rendah pada 0.53 bagi Konfigurasi B berbanding tanpa medan magnet pada 0.79 bagi Konfigurasi A menunjukkan kurang kerosakan pada dinding MWCNTs yang dihasilkan dalam Konfigurasi B. Oleh itu, dengan sokongan medan magnet dalam discas arka metana telah terbukti menghasilkan diameter MWCNTs yang kecil dan seragam serta kurang kerosakan pada dindingnya. MWCNTs yang dihasilkan dalam kajian ini boleh dikaji lebih lanjut untuk aplikasi nanoelektronik seperti wayar nano dan pengisi nano yang bersifat konduktif.

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LIST OF ABBREVIATION

CNTs	-	Carbon Nanotubes
MWCNTs	-	Multiwalled Carbon Nanotubes
SWCNTs	-	Single Walled Carbon Nanotubes
CH_4	-	Methane
DC	-	Direct Current
A	-	Ampere
NdFeb	-	Neodymium
PWM	-	Pulse Width Modulator
SEM	-	Scanning Electron Microscopy
TEM	-	Transmission Electron Microscopy
XRD	-	X-Ray Powder Diffraction
MHz	-	Mega Hertz
AFM	-	Atomic Force Microscopy
1D	-	One Dimensional
CVD	-	Chemical Vapour Deposition
AC	-	Alternating Current
Co	-	Cobalt
Ni	-	Nickel
Fe	-	Ferum
C	-	Carbon
He	-	Helium

Ar	-	Argon
DWCNTs	-	Double Walled Carbon Nanotubes
N_2	-	Nitrogen
H_2	-	Hidrogen
CNF	-	Carbon Nanofiber
CCD	-	Charge Coupled Device
DPDT	-	Double Pole Double Throw
FEMM	-	Finite Element Method Magnetic
RMS	-	Root Mean Square
FWHM	-	Full Width at Half Maximum
RBM	-	Radial Breath Mode

LIST OF SYMBOLS

%	-	Percentage
K	-	Kelvin
^{o}C	-	Celsius
D_v	-	Average nano-crystallite size
λ	-	Radiation wavelength
θ	-	Angle of XRD peak
ω	-	Vibrational frequency

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In modern science nowadays, extensive research on nanotechnology is rapidly growing. The application of nanotechnology is in wide areas such as agriculture, food processing, cosmetic, electronics, astronomy research, military technology for defence, etc. All electronic devices nowadays getting smaller yet their performance cannot be compromised. Thus, nanomaterial studies always become important among researchers to improve daily life routine by nanotechnology implementation especially in electronics application.

Carbon nanotubes, (CNTs) often said as one of promising nanoscale material in electronics application to be used as nanowires, sensing element, optoelectronic devices, field emitter for display, and transistor channel [1-3]. CNTs being investigated in the past 20 years since founded by Japanese scientist, Sumio Iijima in 1991 [4]. During that time, arc discharge was the first method known to produce CNTs. A few years later, CNTs was successfully produced by chemical vapour deposition and laser ablation method [5, 6]. Among them, arc discharge is considered the best method in producing high crystalline CNTs with fewer defects whilst not producing hazardous toxic gas and considered as an environmental friendly method in producing CNTs [7-9].

Basically, arc discharge is electrical breakdown phenomena which resulting the creation of hot plasma due to high current. In CNTs synthesis application, arc discharge system consists of two electrodes acting as anode and cathode, power supply and carbon precursor such as pure graphite which actually acting as anode for anodic arc discharge. It can be happening in tight chamber filled with certain gas at certain pressure as well as in liquid environment by submerged both anode and cathode. However, the large number of reports found to produced CNTs in tight chamber filled with certain gas [10].

In arc discharge method, both electrodes (anode and cathode) was brought to contact at initial stage allow for current flowing, causing resistive heating that increases electrode temperature [10]. Sudden change of interelectrode gap led to electrical breakdown in gaseous environment hence resulting the formation of plasma between electrodes. The stability of plasma corresponds to the stablity of anode and cathode voltage which related to interelectrode gap. This continuous hot plasma formation converts the carbon precursor into carbon vapour state. The basic fundamental of CNTs growth in arc discharge is the phase change of carbon precursor from solid to carbon vapour and then turn to liquid state before solidify again and stick at cathode tip which much cooler compared to anode. The carbon deposited at cathode tip usually contain CNTs.

Several arc discharge parameters were taken into account in producing CNTs in better yield such as arc current, arc voltage, type of gas, chamber pressure, type of power supply, catalyst used, shape and type of electrodes as well as carbon precursor itself. The variation of these parameters has gone through along the past 20 years to understand the optimal condition in producing CNTs in high quality and high quantity. Until this report is written, both studies on the synthesis and application of CNTs still running in parallel however, there is still much more to be understood in the production of quality CNTs by arc discharge method. A lot of improvement has been achieved yet there is still a gap in academic research field to produce much better CNTs as required to be used efficiently in electronics application that need specific structure, diameter and length of CNTs.

In the beginning, CNTs was grown in Argon environment at 100 Torr of chamber pressure [4]. Since then, other types of gases being investigated to evaluate the yield of CNTs produce. Different types of gases have different ionization potential and have unique thermal characteristic which plays important role in achieving breakdown condition hence led to formation of plasma. Due to its highest thermal conductivity, Hydrogen is said as the best gas for promoting the growth of CNTs with less carbonaceous materials [7]. However, in large scale CNTs production, it was found that Hydrogen is difficult to control due to unstable plasma formation [11]. Thus, most of works later reported using Helium tested at various parameters. Other gases have been investigated including Nitrogen, Air, and hydrocarbon such as Methane, CH_4 [12-14]. However, only a few works reported conducting arc discharge CNTs synthesis with hydrocarbon as buffer gas. Limited reports on the use of hydrocarbons in the production of CNTs by arc discharge method have opened up opportunity for researchers to explore more on the capabilities of hydrocarbon in producing good quality CNTs.

The external magnetic field introduced in arc discharge CNTs synthesis reported increases the yield of CNTs [15-17]. It has been confirmed that plasma confinement and other plasma parameters in arc discharge can be controlled by magnetic field [18]. Thus, the exploitation of magnetic field in assisting CNTs synthesis is worth to be expanded and understandable in combination with other arc discharge CNTs parameters such as type of gas and pressure, arc voltage and arc current as well as carbon precursor. Types of magnet, configuration and magnetic strength also play important role to the formation of stable confined plasma and hence producing better quality of CNTs.

1.2 Problem Statement

In electronics application, Single Walled Carbon Nanotubes (SWCNTs) often gets more attention due to its remarkable electronics properties where it can demonstrate both metallic and semiconducting behaviour depend on its chirality.

Multi Walled Carbon Nanotubes (MWCNTs) is said to always exhibit metallic behaviour. However, it also demanded for many applications such as flat panel display, electron source in cathode ray tube, individual nanoprobe, high brightness beam in electron microscope, nanoscale X-Ray source and nanoscale linear bearings [19-24].

Basically, there are three usually employed methods in synthesis MWCNTs. The three methods are chemical vapour deposition, laser ablation and arc discharge methods. Among three of them, arc discharge is said capable of producing well graphitized MWCNTs [25, 26] . Moreover, arc discharge does not require metal catalyst in synthesis MWCNTs. Previously, hydrogen was claimed by Zhao et al. as the best gas in producing high quality MWCNTs by arc discharge method [27]. However, it was not suitable for large scale production due to unstable plasma formation [28]. As option, Ando et al. suggested the use of CH_4 in synthesising MWCNTs since the decomposition of hydrogen atom contained in CH_4 molecules produces high crystallinity MWCNTs [29]. More recently, there is a work also uses CH_4 as buffer gas at various chamber pressures with different arc currents [14].

In order to fulfil demand for high quality MWCNTs to well suit to the application mentioned above, high crystallinity MWCNTs with uniform diameter formed in well-arranged and straight structure are required. Many reports have mentioned the use of magnetic field in arc discharge has improved the yield of SWCNTs in term of high crystallinity, high quantity, increasing the length and narrows down the diameter of SWCNTs [16, 17, 30-32]. A few works reported of producing less impurities in MWCNTs with magnetic field assistance [15, 33]. To the best of the author’s knowledge, there is no report on the production of high crystallinity MWCNTs with uniform diameter formed in well-arranged structure by arc discharge method. Thus, this work presents the effect of combination magnetic field and CH_4 to the diameter distribution, quantity, high crystallinity, structural defect, shape and orientation as well as impurity level of MWCNTs produced.

1.3 Research Objectives

Based on problem statement and current situation, three research objectives have been defined as in the following:

i. To investigate the effect of magnetic field in different configurations to diameter distribution of MWCNTs grown by DC arc discharge in CH_4.
ii. To investigate the influence of combination of magnetic field and CH_4 in producing high crystallinity and less defect of MWCNTs structure.
iii. To learn the ability of magnetic field in controlling the shape and orientation of MWCNTs produced in CH_4 environment.

1.4 Scope of Project

In order to fulfil project timeline while successfully achieving all three objectives, the research parameters are confined as mention in the following:

i. MWCNTs grown in CH_4 environment at fixed 750 mbar of chamber pressure.
ii. Arc discharge controlled at fixed 60A of DC current for all configurations (with and without magnetic field assistance).
iii. The magnets used are Neodymium, (NdFeB) permanent magnet in grade N52 with axially magnetized direction.
iv. All CNTs synthesis experiment last at approximately 60 seconds.
v. Interelectrode gap distance maintained manually by Pulse Width Modulator, PWM linear motor controller.
vi. High purity graphite (99.99%) were used as anode and cathode.
vii. CNTs were characterized by Scanning Electron Microscopy, Transmission Electron Microscopy, X-Ray powder diffraction, Raman Spectroscopy.

1.5 Thesis Layout

This thesis consists of five chapters which explained as follows:

Chapter 1 starts with the background study to introduce briefly about the subject under study. The problem statement described in this chapter to highlight the gap in recent works related to synthesis of CNTs by arc discharge method, the effect of magnetic field in CNTs formation as well as the gas has been used as buffer gas. This chapter also highlights the objectives of study as well as research scopes.

Chapter 2 explaines the introduction of CNTs to give understanding about the core material which being under study. Three main methods in producing CNTs also described. In this chapter, previous works from other researcher related to synthesis of CNTs by arc discharge method with magnetic field assisted, growth of CNTs in various buffer gas as well as the effect of buffer gas in CNTs production by arc discharge method are presented. As additional, common characterization methods of CNTs in also included.

In Chapter 3, the research methodology is presented. The detail on experimental setup, magnetic field configuration study, equipment used as well as the experimental procedure are described. The overall research works are graphically illustrated in a process flow chart for ease of understanding. Preparation and steps taken for CNTs characterization and analysis are also discussed.

Experimental results are presented in Chapter 4. In this chapter, characterization of CNTs produced are explained and discussed. The results were including morphology of CNTs analysed by Scanning Electron Microscopy, and TEM, spectroscopic analysis by Raman Spectrometer and crystalline structure study analysed by XRD method.

Finally, Chapter 5 gives the conclusion of the findings in Chapter 4. It also highlights the contribution of the research work based on results achieved as well as recommendation related to this topic for future research.

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