UNIVERSITI PUTRA MALAYSIA

PLASMA TECHNIQUE OF CARBON NANOTUBE SYNTHESIS USING COMMERCIAL MICROWAVE OVEN

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By

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PLASMA TECHNIQUE OF CARBON NANOTUBE SYNTHESIS USING COMMERCIAL MICROWAVE OVEN

By

KURE NICODEMUS

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Carbon nanotubes (CNTs) research have been the focus of revolutionizing nanotechnology, which stimulate attention from scientific communities to understand its exceptional properties, syntheses and potential applications such as sensor, field emission, hydrogen storage and biomedicine. Advances have been made by researchers, working extensively to develop diverse techniques such as arc discharge, laser ablation, thermal chemical vapor deposition and plasma enhanced chemical vapor deposition to synthesize CNTs, but these techniques are expensive and time consuming. The aim in this study is to develop a plasma technique using commercial microwave oven, to synthesize CNTs which can be more economical and less time consuming.

The technique employed comprises of two parts; first part id to monitor the pressure level and plasma region while the second part deals with synthesis where require carbon source and catalyst are investigated. Commercial microwave oven with operating power of 600 W was used to irradiate the carbon source and coated substrate at atmospheric pressure with 2.45 GHz frequency, which leads to the formation of plasma in the tubular reactor.

From the study shows that for CNTs growth, certain parameters are necessary to be controlled such as plasma, pressure at 0.81 mbar, temperature at 750 °C, catalyst (iron(III) nitrate nonahydrous), and carbon source (Polyethylene). The obtained CNTs were characterized via Raman spectroscopy which shows CNTs quality of 1.01, average tubes diameter at (6.0 to 10.0) ± 0.5 nm, twisted and oriented structures with interlayer spacing of about 0.35 nm and carbon purity of about 99.86%. The plasma technique results obtained shows that the technique is economical and fast process of synthesis.

Abstrak tesis yang dibentangkan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains.

SINTESIS DAN PENCIRIAN TIUB NANO KARBON (CNTs) DENGAN TEKNIK PLASMA MENGGUNAKAN KETUHAR GELOMBANG MIKRO KOMERSIAL

Oleh

KURE NICODEMUS

November 2015

Pengerusi : Profesor Madya Mohd Nizar Hamidon
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Penyelidikan tiub nano karbon (CNTs) telah menjadi fokus dalam merevolusikan teknologi nano, yang merangsang perhatian daripada masyarakat saintifik untuk memahami ciri-ciri yang luar biasa, sintesis dan aplikasi yang berpotensi seperti sensor, pancaran medan, penyimpanan hidrogen dan bioperubatan. Kemajuan telah dibuat oleh penyelidik, yang bekerja secara meluas untuk membangunkan pelbagai teknik seperti nyahcas arka, ablasi laser, pemendapan wap kimia haba dan pemendapan wap kimia haba plasma dipertingkat ntuk mensintesis CNTs, tetapi teknik ini mahal dan mengambil masa. Tujuan kajian ini adalah untuk membangunkan satu teknik plasma menggunakan ketuhar gelombang mikro komersial, untuk mensintesis CNTs.

Teknik yang digunakan terdiri daripada dua bahagian; bahagian pertama untuk memantau tahap tekanan dan kawasan plasma manakala bahagian kedua membincangkan sintesis di mana sumber karbon dan pemangkin yang diperlukan disiasat. Ketuhar gelombang mikro komersial dengan kuasa operasi sebanyak 600 W digunakan untuk menyinari sumber karbon dan substrat bersalut pada tekanan atmosfera dengan frekuensi 2.45 GHz, yang membawa kepada pembentukan plasma dalam reaktor tiub.

Daripada kajian menunjukkan bahawa bagi pertumbuhan CNTs, parameter tertentu yang perlu dikawal adalah seperti plasma, tekanan pada 0.81 mbar, suhu pada 750 °C, pemangkin (ferum (III) nitrat nonahydrous), dan sumber karbon (Polyethylene). CNTs yang diperolehi dicirikan melalui spektroskopi Raman yang menunjukkan kualiti CNTs daripada 1.01, purata diameter tiub pada (6,0-10,0) ± 0.5 nm, struktur dipintal dan berorientasikan dengan jarak antara lapisan kira-kira 0.35 nm dan ketulenan karbon kira-kira 99.86%. Keputusan teknik plasma yang diperoleh menunjukkan bahawa teknik ini adalah lebih menjimatkan dan mempercepatkan proses sintesis.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CNTs	Carbon nanotubes
CNS	Carbon nanostructures
0D	Zero dimensional
1D	One dimensional
2D	Two dimensional
3D	Three dimensional
FESEM	Field emission scanning electron microscopy
HRTEM	High resolution transmission electron microscopy
EDX	Energy dispersive X-ray
XRD	X-ray diffraction spectroscopy
MWCNTs	Multiwalled carbon nanotubes
SWCNTs	Singlewalled carbon nanotubes
Rf	Radio frequency
Dc	Direct current
Mw	Microwave
Ar	Argon
He	Helium
Y-Ni	Yttrium-Nickel
Fe	Iron
Ni	Nickel
Co	Cobalt
Mo	Molybdenum
CVD	Chemical vapor deposition
TCVD	Thermal chemical vapor deposition
PECVD	Plasma enhanced chemical vapor deposition
ACCVD	Alcohol catalytic chemical vapor deposition
OH	Hydroxyl
FCCVD	Floating catalyst chemical vapor deposition
NPs	Nanoparticles
RBM	Radial breathing mode
PE	Polyethylene
D-band	Defect-band
G-band	Graphite-band
hkl	Miller indices
C	Carbon
N	Nitrogen
H	Hydrogen
O	Oxygen
Au	Gold
Cu	Copper
ITO	Indium Tin Oxide
CNF	Carbon nanofibers

CHAPTER 1

INTRODUCTION

1.1 Introduction to carbon

Carbon is a nonmetallic and one of the nature most abundant elements, which allotropes such as graphite, diamond, nanotubes and graphene. Carbon has the ability to react with many organic elements to form compounds. Nanotechnology deals with manipulation of materials at least one dimension of about 1 nm - 100 nm in to form larger structures. Research in nanotechnology in recent years has attracted various inter-disciplinary scientists around the world due to its unique degree of functionality at various nanomaterials, and its nanoscale dimensions in different fields of applications. And the concept of nanotechnology was brought about by an American physicist, Richard Feynman as far back as 1959 and has tremendous impact on the new research area from the scientific communities (Mamalis *et al.*, 2004).

Carbon-based nanomaterials are the most promising group of nanostructure materials, contribution by Kroto *et al* in 1985 has great impact to the era of carbon science (Kroto et al., 1991). These nanomaterials are basically fullerene, diamond, graphite, carbon nanotubes and graphene. The carbon nanostructures have proven its strong potentials in revolutionizing wide range of applications globally and they can be classified as zero-dimensional fullerenes (0D), one-dimensional carbon nanotubes (1D), two-dimensional graphene (2D) and three-dimensional graphite (3D) which comprises of carbon as their building block nanomaterial. Figure 1.1 depicted the various carbon nanostructures classification (Chen, 2014). Carbon nanomaterials is the hallmark for increasing research interest on carbon nanotechnology globally, due to its ability to offer insights on material properties at nanoscale, and has significant role in its area of potential applications.

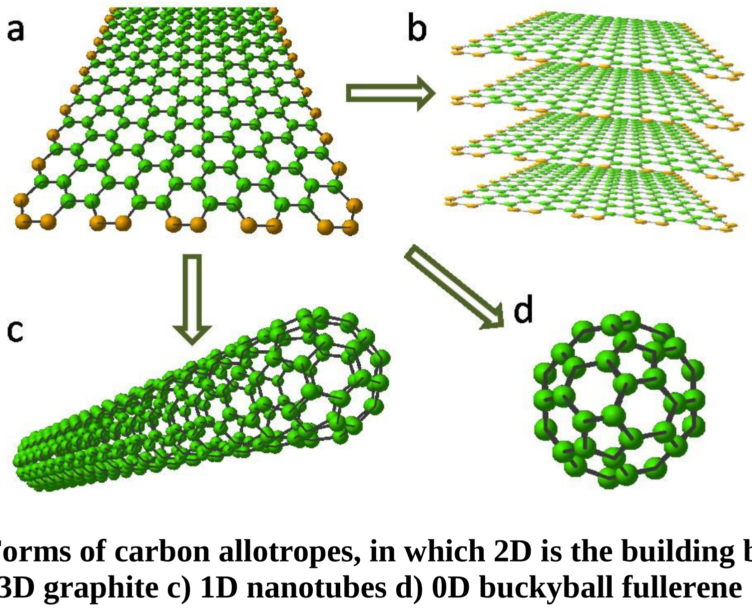

Figure 1.1 Forms of carbon allotropes, in which 2D is the building block. a) 2D graphene b) 3D graphite c) 1D nanotubes d) 0D buckyball fullerene (Chen, 2014)

Carbon nanotubes (CNTs) are tubular carbon molecules with structure similar to a fullerene. Although, fullerene carbon atoms form sphere shape while in CNTs the carbon atoms form a hollow concentric shape and each end is capped with half fullerene molecule which composed of hexagonal rings carbon atoms held together by van der Waals force (Iijima *et al.*, 1993). CNTs entirely consists of hexagonal network of sp^2 bonds of carbon atoms similar to graphite. CNTs are among the strongest materials due to carbon-carbon bonds. And CNTs are categories mainly into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs).

Furthermore, Iijima discovered CNTs in 1991 which consists of concentric graphitic layers with 0.34 nm - 0.36 nm interlayer spacing corresponding to a typical graphite interlayer spacing and carbon-carbon bond length of 0.14 nm (Iijima, 1991; Mamalis *et al*., 2004). Carbon-based materials serves vital role in nanotechnology and they have exhibit distinctive properties at nanoscale levels compared to its bulk materials.

The exceptional electrical, mechanical, thermal and electrochemical properties exhibited by CNTs, make it to have great potentials in wide range of applications either in its unfunctionalized (pure CNTs) or functionalized (CNTs bonded with other materials) forms. Such applications are sensor, energy storage, field emission, composite materials, microelectronics, drug delivery and superconductive electrode (Ajayan *et al*., 2001; Collins, 2000; Cott *et al*., 2013; Gannon et al., 2007; Lee *et al*., 2001; Mahajan *et al*., 2015; Peretz *et al*., 2012; Ren *et al.*, 2013; Tang *et al*., 2001; Wang et al., 2004; Yang *et al*., 2015).

The techniques employed by researchers in synthesizing CNTs such as arc discharge evaporation which was the first technique to synthesize CNTs (Iijima, 1991), laser ablation (Kroto *et al.*, 1991), thermal chemical vapor deposition (TCVD) (Jung *et al.*, 2001), plasma enhanced chemical vapor deposition (PECVD) employed use plasma as it source of heat rather than electric heating coil as in TCVD (Chhowalla *et al*., 2001), alcohol catalytic chemical vapor deposition (ACCVD) (Maruyama, 2002) and floating catalyst chemical vapor deposition (Atiyah *et al*., 2011).

1.2 Problem statement

The constant quest in understanding the nature (properties), growth mechanism, and application of CNTs by researchers, thus attract the need to synthesize this material. Several techniques have been proposed by researchers to synthesize CNTs as mentioned earlier. The drawback in CNTs synthesis in spite of the proposed syntheses techniques, is the lack of developing techniques which are economical and less time consuming. In this study, an attempt was made to develop a technique that can synthesize CNTs economically and less time consuming by using commercial microwave oven in a batch process of synthesis. The plasma techniques offer volumetric heating which heat the whole samples at the same time rapidly compare to others based on conventional heating (with samples temperature gradient from hotter to colder region). The challenge of the present study is to control the synthesis parameters such as pressure (atmospheric pressure) to generate plasma, carbon source which decomposes easily when irradiated with microwave, catalyst as active growth layer and ambient temperature which control the CNTs growth.

1.3 Aim and objectives

The present study ultimate aim is to develop a plasma technique which generate the temperature for carbon decomposition using commercial microwave oven with operating power of 600 W at 2.45 GHz capable of synthesizing CNTs. In order to achieve this aim, the following objectives will be embarked upon in this study:

1. To develop a plasma technique using commercial microwave oven to synthesize CNTs due to its volumetric heating.
2. To study the appropriate carbon source, catalyst, ambient temperature and pressure for CNTs synthesis.
3. To characterize the carbon quality, morphology, purity and structure of CNTs.

1.4 Scope of study

This study is devoted to develop a plasma technique capable of synthesizing CNTs via commercial microwave oven operating at microwave power of 600 W at 2.45GHz. The synthesis process is carried out under natural air environment. Lastly, to characterize the CNTs using microscopy and spectroscopy techniques. Raman Spectroscopy, FESEM, HRTEM, EDX and XRD were used to characterize the CNTs.

1.5 Thesis content

The layout of the thesis is presented as follows:
Chapter 1 presents an introduction to carbon nanotechnology, outline of carbon nanotubes based on their structure, properties, application and syntheses, problem statement, aim, objectives and scope.

Chapter 2 explains critical literature review concerning subjects related to the structure and properties of CNTs, application, growth mechanism, syntheses techniques and characterization of CNTs. Furthermore, plasma and microwave heating are also reviewed. Other works related to elevated temperature synthesis of CNTs are described.

Chapter 3 describes mainly the methodology of the study and characterization techniques.

Chapter 4 explains and discussed the experimental results from the characterization techniques applied such as Raman spectroscopy, FESEM, HRTEM, EDX and XRD.

Chapter 5 summarizes the conclusion of this study and provides recommendation for future study.

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