UNIVERSITI TEKNOLOGI MARA

SYNTHESIS AND CHARACTERISATION OF CARBON NANOTUBE FROM CAMPHOR OIL USING TWO-STAGE FLOATED-CATALYTIC CHEMICAL VAPOUR DEPOSITION

MUHAMMAD SALLEH BIN SHAMSUDIN

Thesis submitted in fulfillment of the requirements for the degree of Master of Science

Faculty of Applied Sciences

June 2016

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work as in author's profile. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Muhammad Salleh Bin Shamsudin			
Student I.D. No.	:	2010277976			
Programme	:	Master of Science (Research) - AS 780			
Faculty	:	Applied Sciences			
Thesis Title	:	Synthesis and Characterisation of Carbon			
		Nanotube from Camphor Oil Using Two-Stage			
		Floated-Catalytic Chemical Vapour Deposition			
Signature of Student	:	~			
Date		June 2016			

ABSTRACT

Nanomaterials have become an important part of the materials research and development. For the last couple of decades, emerging low-dimensional carbon materials, in particular, carbon nanotube (CNT) has received considerable attention in the literature in the broad field of nanoscience and nanotechnology. More recently, CNT appears to be a very appropriate candidate to become the advanced nanomaterial of the 21st century. Indeed, today, CNT has gained significant attention at the forefront of the carbon nanomaterial development, and has become one of the most widely investigated materials. In general, CNT is a cylindrically-shaped atom thin laver(s) of graphene or carbon crystal with an identical and high aspect ratio of its length over to diameter. In this thesis, the aim of this project was to explore the deposition/growth process using two-stage floated-catalytic chemical vapour deposition (CVD) technique and its intrinsic properties using advances nano-metrology analysis based on these CNTs. The current state of art on CNT growth by two-stage floated-catalytic CVD technique has been examined; emphasizing the fundamental processes that distinguish CNT growth from conventional crystal growth. The epitaxial iron-filled CNT growth from camphor oil precursor has attracted considerable interest because of its extraordinary characteristics and ability to grow by synthesised on the lab-scale. Although the quality of CNT over this technique has improved, there are still obstacles such as structural non-uniformity, that limit applications in a wide range of truly current technologies. The key elements of CNT growth using this technique are highlighted, and discussed with regard to impacts on structural uniformity, and physico-chemical properties. The parameters of specific components such as i) influence of synthesis temperature, ii) influence of synthesis time, iii) influence of catalyst to hydrocarbon ratio, iv) influence of thermal annealing atmosphere, and v) influence of post thermal annealing time were investigated in an effort to provide a pathway for future advancements in CNT synthesis. The CNTs were mainly characterised by field emission scanning electron microscopy, micro-Raman spectroscopy, and thermogravimetric analysis to determine physico-chemical properties of CNT. The findings of the study were supported by the highly resolution transmission electron microscopy. The iron-filled spherical-like graphene sheet to almost catalyst-free CNT nucleation-growth mechanism has been proposed for the CNT growth. Narrow diameter size distribution, low amorphous carbon content, defect-free, high thermal stability and low catalyst remained in CNT is observed for the optimised parameter of high quality of CNT. Finally, the major impact of CNT on materials science, challenging issues, and key future directions for research in CNT are briefly discussed.

TABLE OF CONTENT

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENTS	v
TABLE OF CONTENTS	vi
LIST OF TABLES	x
LIST OF FIGURES	xi
LIST OF SYMBOLS	xv
LIST OF ABBREVIATIONS	xvi

CHAPTER ONE: INTRODUCTION

1.1	Spectrum in Nano Landscape	1
1.2	Carbon Nanotube - Time to Think Out the Box	3
1.3	Problem Statement	4
1.4	Objectives of the Research	6
1.5	Scope of the Research	6
1.6	Contributions of the Research	7
1.7	Thesis Organisation	8

CHAPTER TWO: LITERATURE REVIEW

2.1	Introduction	9			
2.2	General Perspective of Camphor Oil as a Precursor in the Synthesis	9			
	of CNT				
2.3	Carbon Nanotube: Correlation Between of Electrical Field and	10			
	Structural Properties				
2.4	Carbon Nanotube: Mechanics	11			
2.5	Carbon Nanotube: Chemistry	12			
2.6	The Potential Application of CNT for Next Decades	13			

	2.6.1	Field Emission	13
	2.6.2	Energy Production and Storage	14
2.7	Conclu	ding Remarks	16

CHAPTER THREE: METHODOLOGY

3.1	Introduction				
3.2	Experimental Apparatus of the Two-Stage Floated-Catalytic CVD				
3.3	Carbon Nanotube Growth				
	3.3.1	Influence of Synthesis Temperature on the Formation of	19		
		Carbon Nanotube Using Titanium Dioxide as a Catalyst			
	3.3.2	Influence of Synthesis Temperature on the Formation of	20		
		Carbon Nanotube Using Ferrocene as a Catalyst			
	3.3.3	Influence of Synthesis Time on the Formation of Carbon	21		
		Nanotube Growth on Silicon Substrate			
	3.3.4	Influence of Synthesis Time on the Formation Substrate-	23		
		Free of Carbon Nanotube Growth			
	3.3.5	Influence of Catalyst/Carbon-Source Ratio on the	24		
		Formation of Carbon Nanotube			
	3.3.6	Influence of Thermal Annealing Treatment Atmosphere on	25		
		the Formation of Carbon Nanotube			
	3.3.7	Influence of Post-Thermal Annealing Treatment Time on	26		
		the Formation of Carbon Nanotube			
3.4	Charact	erisation	27		
	3.4.1	Field Emission Scanning Electron Microscope	27		
	3.4.2	High Resolution Transmission Electron Microscope	28		
	3.4.3	Micro-Raman Spectrometer	29		
	3.4.4	Thermogravimetric Analyzer	30		
3.5	Conclue	ding Remarks	31		

CHAPTER FOUR: RESULTS AND DISCUSSION

4.1	Influer	ice of Synthesis Tempe	rature on	the	Formation	of	Carbon	33
	Nanotube Using Titanium Dioxide as a Catalyst							
	4.1.1	Structural Analysis						33