



UNIVERSITI PUTRA MALAYSIA

**SYNTHESIS AND CHARACTERISATION OF CARBON
NANOMATERIAL ON SHORT CARBON FIBER BY FLUIDIZED BED
CHEMICAL VAPOUR DEPOSITION AND FABRICATION OF CARBON
NANOCOMPOSITE**

MAHTA SADEGH VISHKAEI

FK 2009 96



**SYNTHESIS AND CHARACTERISATION OF CARBON NANOMATERIAL
ON SHORT CARBON FIBER BY FLUIDIZED BED CHEMICAL VAPOUR
DEPOSITION AND FABRICATION OF CARBON NANOCOMPOSITE**

By

MAHTA SADEGH VISHKAEI

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

OCTOBER 2009



**SYNTHESIS AND CHARACTERISATION OF CARBON NANOMATERIAL
ON SHORT CARBON FIBER BY FLUIDIZED BED CHEMICAL VAPOUR
DEPOSITION AND FABRICATION OF CARBON NANOCOMPOSITE**

By

MAHTA SADEGH VISHKAEI

October 2009

Chairman : Dr. Mohamad Amran B. Mohd Salleh

Faculty : Engineering

Although there are some researches on the whiskerization of CNPs on carbon fiber, but none has studied it in the fluidized bed. This research has carried out to study the whiskerization of carbon nanoparticles on short carbon fiber by fluidized bed reactor chemical vapour deposition (FBCVD). The CNP whiskerized CF was added by appropriate amount of Polypropylene to make a composite. Some information about catalyst, nanostructural features of nanoparticles and composite discussed. Hence, in this work, experimental studies were carried out to elucidate the influential parameters and investigate the influence of (i) synthesis temperature, (ii) catalyst type (iii) carbon source gas flow rate on CNP quality in FBCVD process. The synthesis of carbon nanoparticles (CNPs) have been done by the catalytic decomposition of acetylene over Fe/CF catalysts in a fluidized bed reactor. The general growing conditions were: 40 L/min flow rate, 450-650°C synthesis temperature, atmospheric pressure and three gas compositions: 50% , 25% and 25% C₂H₂/H₂/N₂. The catalyst substrate was carbon fiber with different sizes (1mm and 2mm) and metal catalyst with different content of iron (0.23%, 1.8% and 4.92%) were used to deposit on the CF surface. Characterization of the catalysts and the products was performed by X-



ray diffraction patterns (XRD), scanning electron microscope (SEM) and thermal gravimetric analysis (TGA). An apparent relationship was found to exist between the metallic iron content of the catalysts, substrate size, and the final characteristics of the carbon products. Then the composite was prepared with the best percentage of whiskerized carbon fiber and polypropylene with concentration of 2% and 98% respectively. The mixture introduced into the mixer (The rotor speed and temperature were set at 120 rpm and 180°C, and mixing continued for 10 min). Finally the composite was analyzed by Dynamic Mechanical Analysis (DMA) for mechanical properties. This study indicates that the optimum condition for the growth of CNP on CF is 600 °C for 1.8% Fe on carbon fiber with 2mm length for a 30 minutes deposition time and 15L/min acetylene flow rate.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**SINTESIS DAN PENCIRIAN BAHAN NANO KARBON DI ATAS KARBON
FIBER PENDEK MENGGONAKAN REAKTOR LAPISAN TERBENDALIR
PENGURAIAN WAP KIMIA DAN FABRIKASI NANO KOMPOSIT KARBON**

Oleh

MAHTA SADEGH VISHKAEI

Oktober 2009

Pengerusi : Dr. Mohamad Amran B. Mohd Salleh

Fakulti : Kejuruteraan

Dalam kajian ini, satu kajian awal telah dilakukan untuk menerangkan pengaruh parameter dan mengkaji kesan (i) suhu sintesis, (ii) jenis pemangkin dan (iii) halaju gas sumber karbon ke atas kualiti CNT yang dihasilkan melalui proses FBCVD. Sintesis nano tiub karbon (CNTs) telah dijalankan melalui penguraian gas asetilena bermangkin ke atas pemangkin Fe/CF (menggunkan kaedah penyediaan impregnasi) di dalam reaktor lapisan bergerak. Keperluan pertumbuhan secara umumnya adalah seperti berikut: halaju gas 40 lit/min, suhu sintesis 450-650 °C, tekanan atmosfera dan tiga komposisi gas: 50%, 25% and 25% C₂H₂/H₂/N₂. Substrat pemangkin yang digunakan ialah gentian karbon pelbagai saiz (1mm dan 2mm) dan pelbagai komposisi besi (0.23%, 1.8% dan 4.92%) digunakan untuk dimendapkan di atas permukaan CF. Struktur pemangkin dan produk yang terhasil kemudiannya dicirikan dengan menggunakan pola X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) dan Thermal Gravimetric Analysis (TGA). Satu pertalian ketara dapat dilihat di antara kandungan besi metalik di dalam pemangkin, saiz substrat dan pencirian akhir produk karbon yang terhasil. Kemudian komposit nano disediakan dengan nano tiub berkualiti terbaik dan juga polipropilena dengan komposisi masing-masing



adalah 2% dan 98%. Campuran itu kemudiaannya dimasukkan ke dalam pengadun (Halaju rotor dan suhu masing-masing ditetapkan kepada 120 rpm dan 180 °C dan diadun selama 10 minit). Akhirnya, analisa komposit telah dijalankan menggunakan Dynamic Mechanical Analysis (DMA) untuk menentukan sifat mekanikalnya. Kajian ini menunjukkan kondisi optimum untuk kadar pertumbuhan CNT ialah 600 °C untuk 1.8% Fe ke atas gentian karbon dengan panjang 2mm untuk 30 minit masa pengendapan dan 15 L/min halaju gas asetilena.

ACKNOWLEDGEMENTS

*In the name of God, Most gracious, Most merciful
All gratification are referred to God*

I would like to take this opportunity to express my highest gratitude and thanks to my supervisors committee, Dr. Mohamad Amran Mohd Salleh, Assoc. Prof. Dr. Robiah Yunus and Dr. Dayang Radiah Awang Biak for their time, support, advises, encouragement and constant guidance throughout the completion of my study. And most of all, for given me chance to improve myself to be a better person in real life. Special appreciation to Assoc. Prof. Dr. Robiah Yunus for serving as my thesis co-director and committee member. She provided valuable feedback and assistance as well as financial support during early stages of my program of study. I would also like to thank:

My mother, Mrs. M. Davoodi and my father, Mr. H. S. Vishkaei:

**“For all those times you stood by me
For all the truth that you made me see
For all the wrong that you made right**

My husband Mr. A. Ahmadi

**For all the joy you brought to my life
For every dream you made come true
For all the love I found in you**

**I'll be forever thankful you're the ones who held me up
Never let me fall you're the one who saw me through
Through it all”**

Finally, special thanks to my kind and respectful aunt, Mrs.H.Davoodi, my best friend in Malaysia and her respectful family, for their support and encouragement, and also my uncle Mr.D.Davoodi for lightening me when I found the going tough .



I certify that an Examination Committee met on October 2009 to conduct the final examination of Mahta Sadegh Vishkaei on her master of science thesis master thesis entitled " Synthesis of carbon nanoparticles on short carbon fiber by fluidized bed chemical vapour deposition (FBCVD) and fabrication carbon nanocomposite" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and universiti Pertanian Malaysia (High Degree) regulation 1981. The Committee recommends that the student be awarded the relevant degree.

Member of the Examination Committee are as follows:

Prof. Madaya Ir. Dr. Thomas Choong Shean Yaw
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Prof. Dr. Abdul Halim Shaari
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Dr. Suraya Abdul Rashid
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Prof. Ir. Dr. Mohd. Shobri Takriff
Faculty of Engineering
Universiti Putra Malaysia
(External Examiner)

Bujang Kim Huat, PhD
Professor/ Deputy Dean
School of Graduate studies
Universiti Putra Malaysia

Date:



This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfilment of the requirement for the degree of Master of Science.

Members of the Supervisory Committee were as follows:

Mohamad Amran B. Mohamad Salleh, Phd.

Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Assoc. Prof. Dr. Robiah Yunus, Phd.

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Dr. Dayang Radiah Awang Biak, Phd.

Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

HASANAH MOHD GHAZALI, PhD

Professor and Dean
School of Graduate studies
Universiti Putra Malaysia

Date: 11 February 2010



DEDICATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

MAHTA SADEGH VISHKAEI

Date: 11 February 2010



TABLE OF CONTENTS

	Page
ABSTRACT	ii
ABSTRACT	iv
ACKNOWLEDGEMENTS	vi
APPROVALS	vii
DEDICATION	ix
LIST OF TABLE	xii
LIST OF FIGURES	xiv
LIST OF ABBREVIATIONS	xvii
CHAPTER	
1 INTRODUCTION	
1.1 Background of study	1
1.2 Objective of the study	8
1.3 Scope of study	8
1.4 Structure of thesis	9
2 LITERATURE REVIEW	
2.1 Carbon Nanoparticles	10
2.2 Mechanism of carbon nanoparticles growth	16
2.3 Motivation for Large-Scale Synthesis of Carbon Nanoparticles	20
2.4 Carbon Nanoparticles Synthesis Techniques	21
2.4.1 Arc Method	23
2.4.2 Laser Methods	25
2.4.3 Chemical Vapour Deposition	26
2.5 Fluidized-Bed Technology	29
2.5.1 Advantages of Fluidized Beds	31
2.6 Studies of carbon nanoparticles synthesis using Fluidized-Bed Chemical Vapour Deposition (FBCVD)	34
2.7 Effect of operation parameters on CNPs growth	34
2.7.1 Effect of temperature on CNT growth	34
2.7.2 Influence of Carbon Source	36
2.7.3 Influence of catalyst	37
2.7.4 Influence of Additives	47
2.8 Whiskerization	51
2.9 Polypropylene	52
2.10 Nanocomposites	53



3	MATERIALS AND METHOD	
3.1	Introduction	55
3.2	Catalyst preparation	55
3.2.1	Cutting the carbon fiber	56
3.2.2	Making the deposit of Fe over CF	56
3.2.3	Use EDX and XRD to find the concentration the Fe over the CF	57
3.3	Synthesis process of carbon nanoparticles over the carbon fiber	58
3.3.1	Minimum velocity	59
3.3.2	Preparation the reactor	60
3.3.3	Operating condition	61
3.4	Characterization	
3.4.1	Scanning Microscopy Electron	64
3.4.2	Thermo Gravimetric Analysis (TGA)	65
3.4.3	X-ray Diffraction (XRD)	66
3.5	Composite preparation	
3.5.1	Preparation of nano composite	68
3.5.5	Specimen for testing	70
4	RESULTS AND DISCUSSION	
4.1	Introduction	73
4.2	Catalyst characterization	73
4.2.1	XRD results	74
4.2.2	EDX results	75
4.2.3	SEM results	78
4.3	Investigate the operating conditions of CNP growth	80
4.3.1	Effect of Iron concentration on CNP grown	80
4.3.2	Effect of CF size on CNP grown	85
4.3.3	Effect of Acetylene flow rate on CNP grown	87
4.3.4	Effect of temperature on CNP grown	92
4.4	Composite characterization	97
4.4.1	Effect of whiskerized carbon fiber on tensile properties of composite	101
4.4.2	Effect of coated carbon fiber on thermal property of WCF/PP Composite	104
5	CONCLUSION AND RECOMMENDATIONS	
5.1	Conclusions	114
5.1.1	Catalyst characterization (Fe over the CF)	115
5.1.2	Effect of CF size on CNP grown	115
5.1.3	Effect of Acetylene flow rate on CNP grown	115
5.1.4	Effect of temperature on CNP grown	115
5.1.5	Nanocomposite characterization	115
5.2	Recommendations	116



REFERENCES	117
APENDICES	132
BIODATA OF STUDENT	144



LIST OF TABLES

Table		Page
2.1	Specific Tensile strengths of various materials	15
2.2	Comparison of the Established Techniques for CNT Synthesis	22
3.1	Quantity of Iron Nitrate and carbon fiber	57
3.2	Different operating condition	63
3.3	Different type of composite	68
4.1	Effect of reaction temperature on CNPs production	93
4.2	Tensile results for PP, CF/PP, WCF/PP	102



LIST OF FIGURES

Figure		Page
1.1	Electron configuration of carbon atom	1
1.2	Caps to SWNT formed by cutting a fullerene C ₆₀ molecule along the equatorial plane, (a) shows the armchair cap normal to a fivefold axis while (b) shows the zigzag normal to a threefold axis	2
2.1	Eight allotropes of carbon: a) Diamond, b) Graphite, c) Lonsdaleite, d) C ₆₀ (Buckminsterfullerene or buckyball), e) C ₅₄₀ , f) C ₇₀ , g) Amorphous carbon, and h) single-walled carbon nanotube or buckytube	10
2.2	(a) Definition of the chiral vector describing the unit cell of a SWNT (b) images of armchair (n, n), zigzag (n, 0) and chiral nanotubes Details of structural parameters	12
2.3	single wall carbon nanotube	14
2.4	Schematic of the proposed interacting particle model for nanotube initiation and growth. Shading is representative of carbon concentration. Bold arrows indicate the direction of diffusive flux. Clear arrows indicate the direction of particle motion.	18
2.5	Impact of increasing gas-phase availability and associated increase in surface deposition flux and possible	19
2.6	Arc-discharge scheme. Two graphite electrodes are used to produce a dc electric arc-discharge in inert gas atmosphere.	24
2.7	Sketch of a typical fluidized-bed reactor setup. A cylindrical reactor is affixed within a high-temperature furnace with appropriate temperature, pressure, and gas flow controls, connected to a data logging system. Environmental	31



mitigation systems are incorporated to remove entrained solid particles in the off-gas before venting to atmosphere.

2.8	Circulating fluidized bed reactor	33
2.9	Synthesis of CNTs from substrate-bound catalyst particles: (a) growth mechanism by incorporation of carbon at catalyst particle which remains at base of CNT, (b) critical steps of catalyst preparation, annealing, and CNT nucleation and growth	41
3.1	Universal Cutting Mill Machine Pulverisette 19	56
3.2	Measure the differential pressure based on flow rate	59
3.3	Fluidized bed reactor set up	61
3.4	Thermo Haake PolyDrive with Rheomix R600/610 blending machine	69
3.5	Toyoseiki Mini Test Hydraulic Hot and Cold Press	70
3.6	Q800 TA Instrument DMA Q800 V7.1 Build 116. Dynamic mechanical analyzing machine	71
4.1	Sample XRD result for decomposition of iron on carbon fiber surface	74
4.2	Typical EDX spectrum of Fe nanoparticles (a) and composition as measured by EDX in SEM (b), at 0.23% Fe over 2mm CF.	76
4.3	EDX measured 1.8% Fe over 1mm CF	77
4.4	EDX measured 4.92% Fe over 2mm CF	77
4.5	SEM image of Fe catalyst decomposed on 1mm carbon fiber with 1.8%Fe	79
4.6	SEM image of Fe catalyst decomposed on 1mm carbon fiber with 4.92%Fe	79



4.7	SEM image for CNT with 0.23% Fe-CF	81
4.8	SEM image for CNT with 1.8% Fe-CF	82
4.9	SEM image for CNT with 4.92% Fe-CF	82
4.10	TGA result for 0.23%, 1.8% and 4.92% Fe-CF	84
4.11	CNT produced at different iron concentration per 2 g of catalyst	85
4.12	TGA result for different size of CF (1mm and 2mm)	86
4.13	SEM image for 10L/min acetylene flow rate	88
4.14	SEM image for 20L/min acetylene flow rate	89
4.15	TGA result for different acetylene flow rate	90
4.16	CNT produced for different acetylene flow rate	92
4.17	SEM images of CNT grown on Fe/CF at a) 600°C and b) 450°C	94
4.18	SEM images CNT grown at a) 550°C and b) 500°C	95
4.19	CNT produced at different temperature	96
4.20	XRD result for CNT grown at 650°C	97
4.21	XRD results of CNT growth with various growing temperatures	98
4.22	TGA results of CNTs growth processed at various reaction temperatures	100
4.23	SEM images of CF after Fe nanoparticle deposition on it (a) and CF surface after growing carbon naotube(b)	101
4.24	TGA curves of PP, CF/PP and CNT/PP composite	105
4.25	DTA-TGA result for 2% coated CF with nanoparticles /PP composite	106
4.26	Storage modulus (E') of unfilled PP, CF/PP and CNT/PP composite	108



4.27	Loss modulus (E'') of unfilled PP, CF/PP and CNT/PP composite	110
4.28	Tangent δ of unfilled PP, CF/PP and CNT/PP composite	112



LIST OF ABBREVIATIONS

ARNT	Array of Carbon Nano Tube
ASTM	American Society for Testing Materials
β	Maximum of the XRD peak
CAT	Catalyst
CCVD	Catalyst Chemical Vapour Deposition
CVD	Chemical Vapour Deposition
CNT	Carbon Nano Tube
CNP	Carbon Nano Particle
CNM	Carbon Nano Material
DC	Direct Current
DMA	Dynamic Mechanical Analysis
DNA	Deoxyribo Nucleic Acid
DSC	Differential scanning Calorimetry
TDA	Thermal Differential Analysis
E'	Storage Modulus (MPa)
E''	Loss Modulus (MPa)
EDX	Energy Dispersive X-ray
EDS	Energy Dispersive Spectroscopy
EDXRF	Energy Dispersive X-Ray Fluorescence
FBCVD	Fluidized Bed Chemical Vapour Deposition
HIPCO	High Pressure Carbon Monoxide
ID	Internal Diameter



L_c	Mean Crystalline Size (along the c-axis)
λ	Wavelength of X-rays
M	Weight
MWCNT	Multi Wall Carbon Nano Tube
PECVD	Plasma Enhanced Chemical Vapour Deposition
PP	Poly Propylene
SEM	Scanning Electron Microscopy
SWCNT	Single Wall Carbon Nano Tube
TAN δ	Tangent Delta
SCF	Short Carbon Fiber
TGA	Thermal Gravimetric Analysis
T_{onset}	Degradation Temperature
T_m	Melting temperature
T_g	Glass Transition temperature
θ	Angle corresponding to the peak of XRD
U_{mf}	Minimum Velocity
U	Velocity
VPSEM	Scanning Electron Microscope Environmental
XRF	X-ray fluorescence
XRD	X-ray diffraction



CHAPTER 1

INTRODUCTION

1.1 Background of study

Carbon, with an electronic ground state arrangement of $1s^2 2s^2 2p^2$, has the ability to form four valence orbitals: $2s$, $2p_x$, $2p_y$, and $2p_z$ (Figure 1.1). The mixing of the $2s$ and $2p$ atomic orbitals is known as hybridization. Three possible hybridization states (sp^1 , sp^2 , and sp^3) have resulted in many important carbon structures, including carbon nano particles (CNPs). Before the discovery of fullerenes, two solid elemental carbons were believed to exist in only two crystalline phases: graphite and diamond, exhibiting sp^2 and sp^3 hybridized bonding, correspondingly. The structure of large carbon clusters with >40 carbon atoms remained unsolved until Kroto et al. synthesized fullerene C_{60} in 1985 and postulated its structure as that of a shortened icosahedrons (Chee et al., 2007).

Element	Total Electrons	Orbital Diagram				Electron Configuration
		1s	2s	2p	3s	
C	6					$1s^2 2s^2 2p^2$
N	7					$1s^2 2s^2 2p^3$
Ne	10					$1s^2 2s^2 2p^6$
Na	11					$1s^2 2s^2 2p^6 3s^1$

Figure 1.1. Electron configuration of carbon atom

(Source: <http://intro.chem.okstate.edu/1215/Lecture/Chapter11/Fri112098.html>)

There are different types of CNTs, due to the fact that the graphitic sheets can be rolled in many ways. The three types of CNTs are Zigzag, Armchair, and Chiral. It is possible to recognize zigzag, armchair, and chiral CNTs just by following the pattern across the diameter of the tubes, and analyzing their cross-sectional structure (Michael et al., 2002). Figure 1.2 explains two types of CNTs.

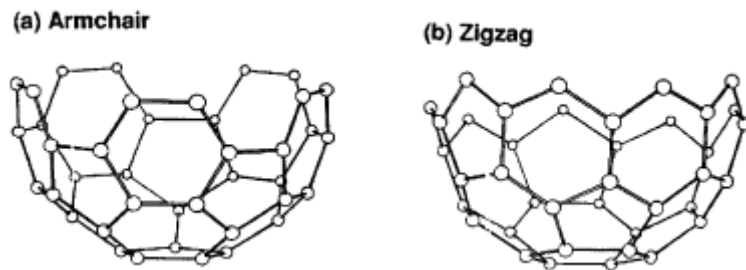


Figure 1.2: Caps to SWNT formed by cutting a fullerene C₆₀ molecule along the equatorial plane, (a) shows the armchair cap normal to a fivefold axis while (b) shows the zigzag cap normal to a threefold axis (Source: Dresselhaus, 1992)

Carbon nanotubes (CNTs) have been investigated for a multitude of applications including energy storage, field emission devices, and composite material strengthening, because of their exclusive mechanical, optical, and electrical properties. Both multiwalled (MWCNTs) and single-walled (SWCNTs) carbon nanotubes can be used, depending on the value-added functionality required in the product. On the other hand, the use of CNTs in both research and end-user applications is currently limited by production throughput. Of the three main techniques used for CNT synthesis, that is, laser ablation, arc discharge, and chemical vapour deposition (CVD), the latter is recognized as having the most potential for large-scale, economically viable CNT production (Venegoni et al., 2002).

Carbon nanotubes (CNTs) are attractive materials with a wide range of potential applications. Various CNT based electronic devices, such as transistors (Weitz et al., 2007; Tans et al., 1998) logical circuits (Bathold et al., 2001) nano electromechanical devices (Ke and Espinosa , 2006; Rueckes et al., 2000) sensors (Brian et al., 2007; Park et al., 2006) have been established. In 1995 field emission from CNTs was reported (Rinzler et al., 1995, Deheer et al., 1995) and nanotubes becomes promising candidates as a field electron emitters. The high aspect ratio (length to diameter) of the CNTs results in a high field improvement factor, useful to field emission. These applications demand an unusual combination of material properties, such as structural rigidity, electric conductivity and small density, possessed only by CNTs. Electronic devices should also resist high temperatures caused by Joule heating and high tensile stresses to keep away from device failure (Wong et al., 2003, Pan et al., 2001).

One of the most promising uses of CNTs is in the development of nano composites, where the CNTs are used as novel fillers and binders to improve their mechanical, electrical and thermal properties. CNTs have great potential applications due to their very large aspect ratio (1000–10,000), low density, high rigidity (Young's modulus of the order of 1 TPa), and high tensile strength (up to 60 GPa). In addition, the excellent electrical conductivity (106 S/m at 300 K for single-walled CNT (SWCNT) and $>10^5$ S/m for multi-walled CNT (MWCNT)) and thermal conductivity (6600 W/mK for an individual SWCNT and >3000 W/mK for an individual MWCNT) make them suitable candidates in preparing nanocomposites with new functional properties (Kumaria et al., 2008).



It has been established in recent years that polymer based composites reinforced with a small percentage of strong fillers can significantly improve the mechanical, thermal and barrier properties of pure polymer matrix (Mahfuz et al., 2004). When these fillers are rod-shaped, the surface area per particle will lead to high mechanical strength and stiffness in axial direction and because of this they are considered the most interesting fillers for advanced applications composites. Carbon fiber reinforced composites have all the ideal properties, leading to their rapid development and successful use for many applications over the last decade (Chung, 1994). Although the carbon fiber polypropylene composite yields catastrophically, it bears a higher load than the glass fiber polypropylene composite, resulting in higher flexural strength. The effect of carbon fiber content and fiber length on mechanical and thermal properties of SCF/PP has already been studied (Fateme, 2006).

The good composite performance often depends on the degree of adhesion between the fiber and the resin binder. Adhesion is usually controlled by chemical bonding due to functional groups and by mechanical interlocking due to surface morphology and this leads researchers to develop a number of surface treatments that could improve the fiber matrix polymer interfacial bonding. Whiskerization treatment on carbon fibers was an effective way to increase the shear strength of carbon-epoxy composite materials by 400%. Whiskerization involves carbon nanoparticles growth on the surface of carbon fibers. The total surface area would increase, and enhance the mechanical interlocking between fibers and matrixes (Rebouillat, 1984).



Since nanoparticles can be grown by different methods with different parameters, the selection of a suitable type of CNP synthesis for a selective application is challenging. The CNTs can be prepared by various methods such as arc discharge, laser ablation, chemical vapour deposition (CVD) and others with CVD the most widely used method of production. Among the different techniques that have been applied for the processing of carbon nanotubes (CNTs) (Journet and Bernier, 1998) catalytic chemical vapour deposition (CCVD) appears promising in opinion due to its relatively low cost and potential high yield production. Definitely, this method, originally used to prepare carbon filaments (Rodriguez, 1993) or vapour-grown carbon fibers (De and Geus, 2000; Endo, 1998) also could be effective for the growth of nanotubes. In fact, precursors of filaments or vapour-grown carbon fibers were evidenced as having nanotubular structure (Toan et al., 1999; Serp and Figueiredo, 1996; Endo et al., 1995). The CCVD technique has been applied both in the absence and in the presence of a substrate. The earlier is a gas phase homogeneous process, the latter is a heterogeneous process. Although more scarce studies on homogeneous processes have demonstrated, that it was possible to produce selectively multiwalled carbon nanotubes (MWNTs) (Marangoni et al., 2001), single-walled carbon nanotubes (SWNTs) (Satishkumar et al., 1998, Cheng et al., 1998), or arrays of carbon nanotubes (ARNTs) (Rao et al., 1998).

Heterogeneous processes have been explored more intensively and MWNTs (Piedigosso et al., 2000; Ivanov et al., 1995), SWNTs (Colomer et al., 2000; Hafner et al., 1998) have been produced on supported or bulk catalysts. Currently, the use of bulk metal catalyst leads mainly to the production of carbon filaments, also called

