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ACKNOWLEDGEMENT

Bismillahirrahmanirrahim, Alhamdulillah. Thanks to Allah the Almighty, the Most Gracious and the most Merciful for giving me the opportunity, courage and patience to accomplish ths research work. Without His help and mercy, this work will not finished especially the time to prepare this thesis, the world faced a great challenges due to pandemic Corona Virus Disease 2019 (COVID19).

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ABSTRACT

Flame synthesis of carbon nanotubes (CNTs) give advantages mainly in industrial sector as it could save overall costs in production of CNTs. The present study replicated the already available process of getting the carbon nanostructure material (CNTs) from the combustion process based on methane diffusion flame. The present study focuses on the understanding of preferable catalyst preparation method using nickel nitrate on silicon wafer for optimization purposes. Besides,the other focus is to clarify the effect of catalyst and flame preparation parameter on CNTs growth in flame-based synthesis. Furthermore, this study introduced two quite similar catalyst preparation; dipping and dropping techniques. This experimentalwork aims to establish a baseline catalyst preparation method using nickel nitrate on silicon wafer as well as to analyse the effect of catalyst preparation and flame parameter on CNTs growth in flame. The CNTs is synthesized on a silicon wafer as the substrate-supported catalyst within the flame which used designated slotted wire mesh. The synthesized carbon nanotubes in the experiment have to be further characterized in terms of physical properties via Field Emission Scanning Electron Microscope (FESEM) and Raman Spectroscopy for its crystallinity analysis. A comprehensive analysis have been done on the diameter of the produced CNTs and the analysis revealed two main stage of diameter, which are transient stage and steady stage, thus elucidating the effect of prolonged exposure in flame environment. The largest matured sized of synthesized CNTs is at 38 nm, that starting from 35 seconds to 60 seconds. The comparison has been performed to show the effect of different substrate, technique, oxidizer and synthesis method towards the growth and morphology of the CNTs. The diameter distribution of silicon wafer as the substrate are larger than nickel wire, the dipping technique reveal larger range of diameter as compared to dropping technique and the growth of CNTs more straight allignment in Chemical Vapor Deposition as compared to flame synthesis. The results of crystallinity analysis indicates insignificant difference if compared to the previous study.

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ABSTRAK

Karbon nanotube (CNTs) sistesis dalam api memberi kelebihan terutamanya dalam sektor industri kerana ianya menggunakan keseluruhan kos yang lebih rendah dalam kuantiti pengeluaran CNTs yang lebih banyak. Dalam kajian ini adalah untuk mengulangi proses yang sedia ada untuk menghasilkan bahan karbon (CNTs) berstruktur nano melalui proses pembakaran iaitu api penyebaran metana. Dalam kajian ini menekankan tentang pemahaman berkenaan cara penyediaan pemangkin yang lebih mudah menggunakan nikel nitrat diataswafer silika bagi tujuan pengoptimuman. Selain itu, fokus lainnya untuk menjelaskan kesan parameter pemangkin dan penyediaan api terhadap pertumbuhan CNTs dalam sistesis yang berasaskan api. Selanjutnya, kajian ini memperkenalkan dua kaedah penyediaan pemangkin yang agak serupa; teknik mencelup dan menitiskan. Eksperimen ini bertujuan untuk menetapkan kaedah penyediaan pemangkin asas menggunakan nikel nitrat pada wafer silika serta untuk menganalisis kesan penyediaan pemangkin dan parameter nyalaan api terhadap pertumbuhan CNTs. CNTs yang telah disintesiskan pada wafer silika yang bertindak sebagai pemangkin disokong substrat dalam api adalah menggunakan slot khas yang direka pada wire mesh. Karbon nanotube ini harus dicirikan lebih terperinci sifat fizikalnya melalui Field Emission Scanning Electron Microscope (FESEM) dan Raman Spectroscopy untuk analisis penghabluran. Analisis komprehensif telah dilakukan mengenai diameter CNTs yang dihasilkan dan analisis menunjukkan dua tahap utama iaitu tahap sementara dan tahap stabil sehingga dapat menjelaskan kesan pendedahan berpanjangan dalam persekitaran nyalaan api. Saiz CNTs matang paling besar yang dihasilkan adalah pada 38 nm yang bermula dari 35 saat sehingga 60 saat. Perbandingan juga dilakukan dalam kajian ini untuk menunjukkan kesan perbezaan substrat, cara dan pengoksidaan dan kaedah sintesis kepada pertumbuhan dan morfologi CNTs. Taburan diameter wafer silika sebagai substrat adalah lebih besar berbanding wayar nikel menunjukkan julat diameter yang lebih besar berbanding dengan teknik titisan dan pertumbuhan CNTs memberi lebih penjajaran lurus di dalam pemendapan wap kimia berbanding sintesis api. Raman spektra analisis menunjukkan sifat kristaliniti yang serupa antara penggunaan substrat wafer silikon dan substrat wayar nikel.

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LIST OF ABBREVIATIONS

CVD	-	Chemical Vapor Deposition
CCVD	-	Catalytic Chemical Vapor Deposition
CNT	-	Carbon Nanotubes
FESEM	-	Field Emission Scanning Electron Microscope
HAB	-	Height Above Burner
MWCNT	-	Multi-walled Carbon Nanotube
RS	-	Raman Spectroscopy
SWCNT	-	Single-walled Carbon Nanotube
TEM	-	Transmission electron Microscope

LIST OF SYMBOLS

s	-	seconds
cm	-	centimeter
μs	-	microsecond
nm	-	nanometer
ø	-	Equivalent ratio
Ω	-	Electrical resistivity
Pa	-	Pressure
$^{\circ}C$	-	Temperature
K	-	Temperature
slpm	-	Flowrate
A/cm^2	-	Electric current density
W/mk	-	Thermal conductivity
Ni	-	nickel
Fe	-	iron
N_2	-	nitrogen
Ar	-	Argon
CO	-	carbon monoxide
CH_4	-	methane
C_2H_2	-	ethylene
C_2H_4	-	acethylene
C_3H_8	-	propane

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CHAPTER 1

INTRODUCTION

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1.1 Background of Study

Carbon is a basic element that contained in various compounds. The characteristics of carbon depend on how they are bond to form the best carbon structures. The morphology, property and growth mechanism of these carbon structures are still being investigated until present. Due to the rapid advancement of nanoscience and nanotechnology, the development of carbon nanostructures in various applications becomes a well-known topic explored by numerous researchers.

Since the first discovery of Carbon Nanotubes (CNTs), the synthesis methods of producing CNTs have been developed gradually in order to establish the best quality structure of CNTs. The first synthesis method of CNTs was laser discharge method. From this method, Sumio Iijima unintentionally had discovered CNTs which was at first the aim was to yield carbon fullerences [3]. Then, development of synthesis method of CNTs is further expanded by laser ablation method, chemical vapor deposition (CVD) and flame synthesis. The flame synthesis method is currently being studied as a favourable method to produce carbon nanostructure known as carbon nanotubes. The difference in synthesis method will alter and affect the structure, quality, and properties of CNTs that being produced.

Synthesis of Carbon Nanotubes (CNTs) using diffusion flame has a significant potential to transform the available existing method to produce CNT in which the wider application of CNTs nowadays are restricted by its high production cost [4]. The needs of ideal and effortless method is very important in order to produce CNTs in a large scale to be applied in various requisition.Thus, diffusion flame have large considerable practical application as compared to premixed flame [3]. Therefore, flame synthesis of Carbon Nanotubes also can be contributed an outstanding advantages especially

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in industry area as it consume lower overall cost in CNT production with the large quantities. High demand in market for these utilization of CNTs in bulk in many application for instance in electronics, composites and energy application lead to the rapid expansion [5] in study of CNTs production among the researchers.

Apart from that, one of the vital keypoints in this study is to obtained the best structure of CNTs with optimum method of flame synthesis. Different structure and quality of CNTs have their own advantages and disadvantages in many application to be used. Hence, the experimental parameter play the important role in order to produced the best CNTs. There are two type of experimental parameter which is catalyst and carbon source parameter and flame parameter. In this experiment, diffusion flame, in which the air and fuel are mixed together after reaching the reacting flame front (heat source). The methane (CH_4) act as the carbon source for CNTs production and the precursor for CNTs formation is the nickel powder catalyst.

1.2 Problem Statement

As stated in the previous section, the motivation of this study is to produce the best quality of CNTs which contributed the high interest to conduct the experiment with development of optimum flame synthesis method. With the advantages that has in the CNTs by using flame synthesis method, increased the concerns and needs from this study to improve some issues that may arise from the previous research.

Recently, there are a few challenges to produce carbon nanotubes in flame synthesis : (i) the production of large-scale quantity with low-cost carbon nanotubes synthesis processes; (ii) to control over the pattern and structure of produced carbon nanotubes; (iii) determination of location and orientation of produced carbon nanotubes on a horizontal substrate; (iv) control over production of CNTs with several parameter; and (v) the best establish of sample preparation process.

In this study, first challenge as stated above is taking into account. The direct research on the CNTs of the diffusion flame attempts to achieve the purity of CNTs and their properties from this diffusion flame synthesis. However, there are some issues that may arise when it comes into the best structure of produced carbon nanotubes, most of the previous research need to improve the allignment of the CNTs, to reduce the amorphous carbon layer and to produce the uniform growth of CNTs in diffusion flame synthesis technique. In addditon, this study also need a generous understanding on how to playaround with several parameter in order to get the initial appearance of CNTs in diffusion flame. Nevertheless, to carry out the best and most favourable technique of sample preparation of catalyst on substrate.

Last but not least, the preparation on how to placed and hold the substrate which is silicon wafer on the arm and to make sure the tool that hold the substrate kept at the horizontal position. Because of there is a vibration and unsmooth movement of arm, it may affect by displacing or misplacing from the original position of the substrate from the arm and interrupt the centre location between the catalyst exposed on flame. Furthermore, different shape of the holder may change the shape of flame which then make it non-uniform flame shape exposed to catalyst used.

Therefore, the problems statement of this study would be the preferable catalyst preparation method using nickel nitrate on silicon wafer has to be understood for the optimization purposes and the effect of catalyst and flame preparation parameter on CNTs growth in flame still unclear.

1.3 Objective of Study

This study aims at synthesizing carbonaceous material (carbon nanotubes) from the diffusion flame. The sample (after diffusion in flame experiment) will then undergo characterization process to investigate the presence of CNTs and its properties and the impurities on it. The objectives of this study are as follows:

1. To establish a baseline catalyst preparation method using nickel nitrate on silicon wafer.
2. To analyze the effect of catalyst preparation and flame synthesis parameter on CNTs growth in flame include exposure time in flame, carbon supply rate and type of carbon source.

1.4 Scope of the Study

These research range could include a few different scopes including the preparation of nickel-based catalyst preparation method using several techniques on silicon wafer subtrate-supported for catalytic CNT growth will be done. Besides that, the growth of CNT will be done in diffusion flame environment and Chemical Vapor Deposition (CVD). Last but not least, the effect on CNT growth on catalyst parameters will be investigated.

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CHAPTER 2

LITERATURE REVIEW

2.1 Carbonaceous Material (Carbon Nanotubes)

Sumio Iijima is the first researcher who discovered carbon nanotubes in 1993 when the structure of Multi-Walled Carbon Nanotubes (MWNT) was observed [6]. Because of its remarkable and amazing properties, carbon nanotubes (CNTs) have been used widely in the thrust areas of material science and has replaced various traditional and conventional materials used in various applications. Furthermore, with certain experimental conditions, CNTs has demonstrated the ability to tune its conductance [7]. Therefore, it is important to have a great understanding on the growth mechanisms of CNTs so that its amazing characteristics and properties could be utilized for any other potential application.

2.1.1 Characteristics of Carbon Nanotubes

Carbon Nanotubes are one of the nanometer size that have a unique 1-dimensional nanostructures with carbon atoms belonging to sp^2 hybridisation make it like a beehive-shaped tube. These carbon nanostructures have around 1/50,000th of human hair thickness [8]. Figure 2.1 shows that CNTs may prevail in three different unique geometries, that are zig-zag, armchair and chiral. The chirality of the CNTs is basically determine the electrical, mechanical, optical, and different of its properties. It has been studied that the electrical properties of CNTs are affected by diameter of the tube and the angle of the chiral which show their distintive properties in conductivity either metallic or semiconducting. Consequently, it is not impossible to say that the demand of this precious behaviour is high. Nevertheless, the classification of this CNTs are divided into two configuration which are single-walled carbon nanotubes (SWNTs)

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and multi-walled carbon nanotubes (MWNTs). Figure 2.2 shows the structure of SWNTs and MWNTs.

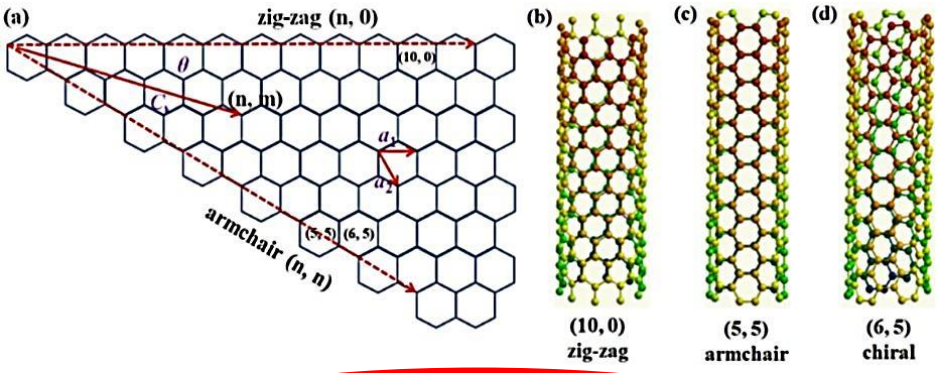

Figure 2.1 Classification of SWCNTs

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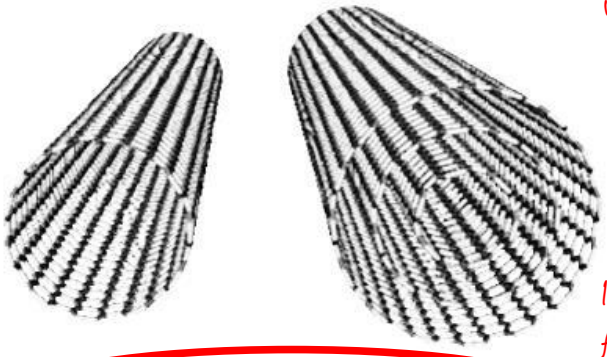

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Figure 2.2 Structure of (a) single-walled carbon nanotubes (SWCNT) and (b) multi- walled carbon nanotubes (MWCNT).

2.1.1.1 Single Walled Carbon Nanotube (SWCNT)

Make sure you write the full term before using the short form or abbreviation.

Single-walled carbon nanotubes (SWCNTs) was observed by Oberin in 1973. Most structures of the SWCNTs have diameter between 0.4 and > 3 nm, while the length can be several in million times the diameter [8]. In addition, the structure of SWCNTs is reported as a tube consisting of a graphene sheet rolling on to form a cylinder, usually closed by hemispheric domes at the tips. In various ways (chirality) a graphene sheet can be wrapped to form a seamless cylinder from a physical point of view [3]. Normally, a few SWCNTs that attached together may appears in the form of

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bundles and a crystal-like structure formation are usually arranged in bundles form [6].

Table 2.1 Similarity and Differences of Single-Walled Carbon Nanotubes(SWCNTs) and Multi-Walled Carbon Nanotubes (MWCNTs)

SWCNT	MWCNT
Single layer of graphene	Multi layers of graphene
Low purity	High purity
Increased the risks of defects during functionality	Lesser risks of defects, once occur it is difficult to recover
Increased the risks of defects during functionality	Complicated of structure
Less body aggregation	Greater body aggregation
More flexible and easy to twist	Hard to twist
Difficult bulk synthesis growth and condition of atmospheric	Synthesis of bulk is easy

2.1.1.2 Multi-Walled Carbon Nanotube (MWCNT)

As mention in the above section, MWCNT was discovered by Sumio Iijima [6]. Most of the method of producing carbon nantubes found in the form of Multi-Walled Carbon Nanotube. The space between the graphite sheets is about the same as that between the graphite sheets, which is 3.4 Å. For these MWCNTs, its diameter is in between 1.4 to at least 100 nm [8]. Table 2.1 also indicates the similarity and differences between two types of carbon nanotubes. MWCNTs have better characteristic as compared to SWCNTs.

2.1.2 Properties of Carbon Nanotubes

Carbon Nanotubes (CNTs) are a prodigious material due to their remarkable properties. CNTs are reported to have an outstanding and promising properties

especially in mechanical, electrical and thermal properties. Due to their ability to improve the existing applications, the demand for a large amount of CNTs has continuously increased. Nevertheless, several factors may also contribute to all these impressive properties such as high aspect ratio, quantum size effect and unique structure [3]. Therefore, it gives a motivation to researchers both theoretically and experimentally with a variety procedures to prove it. To add with all these properties, single-walled carbon nanotubes (SWCNTs) is better than multi-walled carbon nanotubes (MWCNTs). The expectation of the properties of these carbon nanotubes is mainly due to their strength and light weight, which is helpful in the application of material.

2.1.2.1 Mechanical Properties

In terms of tensile strength and Young Modulus, carbon nanotubes are known to be one of the strongest and most stable materials. This happens because of the bonding of covalent sp^2 between the different carbon atoms [9]. The nature of flexibility of CNTs [10] does not affect its capabilities to withstand buckling under compression. With the low density for a solid, allow the CNTs to have high strength to weigh ratio structure.

Carbon Nanotubes posses a good mechanical properties in terms of their high tensile strength and elastic modulus [11]. According to [11], the paper reported that the tensile strength and Young‘s modulus of produced carbon nanotubes are in the range of 13 to 52 GPa and 320 to 1470 GPa respectively for single-walled carbon nanotubes (SWCNTs) whereas the range from 11 to 63 GPa and from 270 to 950 GPa respectively are for produced multi-walled carbon nanotubes (MWCNTs). Suprisingly, if these value are compared with tensile strength and Young Modulus of stainless steel is roughly in 2 GPa and 200 MPa respectively [12]. From that, these properties shows that when compared to stainless steel, carbon nanotubes offer better endurance.

will give TEM images that used to estimate the diameters of a single and bundles of CNT. According to [8], high-magnification images of TEM also give numerical data of measurement of the spacing in several bundles between fringes.

2.5.3 Raman Spectroscopy (RS)

The Raman Spectroscopy (RS) is one of characterization technique which is non-destructive, non-invasive, quick and simple. The instrumentation can be performed at pressure and room temperature and mostly available to a wide range of user communities. This technique is extremely sensitive to examine changes in the properties of nanotubes that are synthesized using different conditions and procedures. With the incident laser power, the Raman signal intensity for the sample increases, providing detailed and accurate electronic and structural characterization.

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CHAPTER 3

METHODOLOGY

3.1 Research Outlines

The information on the experiment facilities used in this study will be discussed in this chapter. A description of the sample preparation on subtrate-supported catalyst, followed by the diffusion flames produced by a system of burner, production process of CNTs, as well as the sampling process for characterization of CNTs such as Field Emission Scanning Electron Microscopy (FESEM), Transmission Electron Microscopy (TEM) and Raman Spectroscopy (RS). Several technique and parameter will be controlled in this experiment to analyze the CNTs growth in flame environment. Last but not least, some of the precautionary measures to perform the test will also be considered. Some of the previous study of CNTs will be used as guidelines and references to study and analyze the outcomes of this experiment. In addition, in the present work also consists of flame synthesis and chemical vapor deposition.

3.1.1 Methodology Structure

Figure 3.1 shows flowchart that illustrates the process involves in the present study. The literature review and information is done after the problems and requirement of this study have been identified. After that, the subtrate-supported catalyst is prepared on silicon wafer substrate with several technique including drops, spray, spin coater and using heating element.

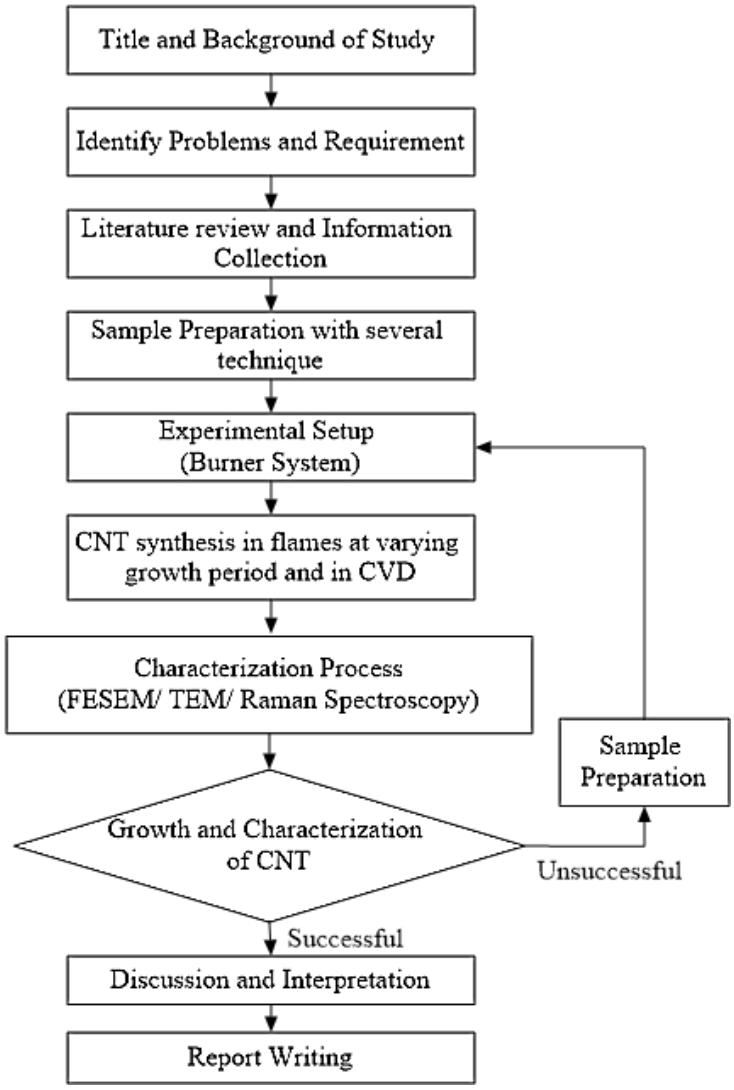

Figure 3.1 Flowchart of Methodology structure

Then, the experimental system of burner will be setup followed by the proper setting of positioning system. These to ensure that the catalyst is exposed at the desired location and coordinates on the mesh stand before run the experiment. Parametric study is done in this stage before the synthesis of the CNTs through the diffusion flame process and some of the sample will be done in CVD for comparison. The synthesis of CNT is done at varying exposure time. The sampling process will then be conducted on the basis of synthesis.

The samples will then be prepared and sent for characterization process analysis of FESEM, TEM, and Raman Spectroscopy. Experiment will be conducted and any

CHAPTER 4

RESULTS AND DISCUSSION

In this section, the physical properties as well as the growth of synthesized CNTs will be presented. The effect of catalyst preparation and flame parameter on CNTs growth in flame will be further analyzed.

4.1 Characterization of FESEM

A nano tube-shaped material that made up of carbon called carbon nanotube is usually measured in nanometer scale on its diameter. A nanometer is one-billionth of a meter, or about one ten-thousandth of the thickness of a human hair. In order to study the type of nanomaterials produced in the growing region, series of analysis using FESEM and Raman Spectroscopy shall be performed on this synthesis materials. The analysis also give the information to determine the differences of the synthesized nanomaterials morphology, homogeneity and characteristic with vary of experimental conditions and synthesis parameters for instance, exposure time.

4.1.1 Diameter of Carbon Nanotubes in Flame Synthesis

Diameter is one of the interesting CNTs features that can be analysed. The present study is done to investigate the relationship between size of diameter with prolonged exposure time.

allignment of CNTs due to its uniformity condition. Based on Figure 4.10, the size is dictated more by the catalyst preparation rather than synthesis environment in terms of average diameter nevertheless the CNTs from diffusion flame shows higher number of average diameter as compare to CVD method. To be noted that, the diffusion flame's sample of synthesized CNTs from exposure time for 60 seconds in flame. The sample preparation is same as explained in section 3.4.1.To get the normalized axis in Figure 4.10 (b), all the actual value of y-axis and x-axis is divided by 20 and 52 respectively. Theoretically, the allignment can be effected by the carbon supply rate that reduce the CNTs growth rate [3]. Therefore, in flame synthesis, the curly allignment of CNTs due to the carbon supply rate is much higher than its diffusion rate whereas the straight allignment of CNTs in CVD because of the carbon supply rate is approximately same with its diffusion rate into catalyst used resulted the optimum growth rate of CNTs. Possibly, the maximum carbon supply rate in CVD could be similar to argon rate supply. When the sample is inserted into the hot furnace, it gained energy to cut the nanoparticles bonding. Noted that when the carbon source comes in contact with catalyst nanoparticles, some of the carbon atom is needed to eliminate other elements such as hydrogen (H) and oxygen (O_2) via elimination method and not all will diffuse into catalyst particle.

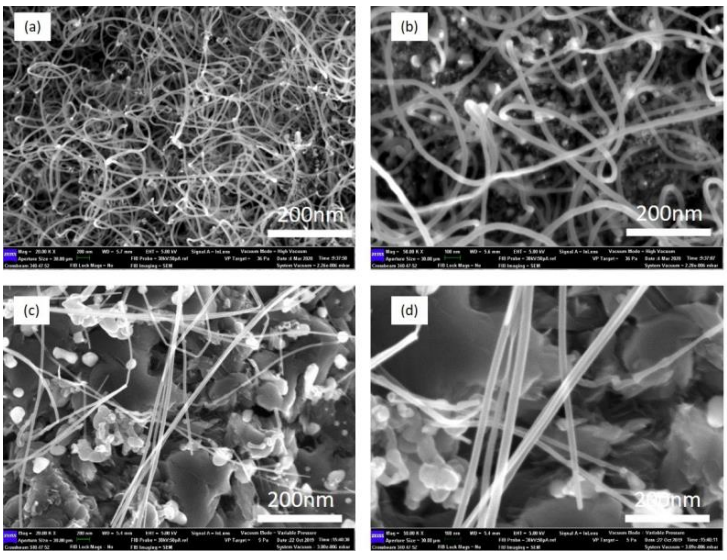

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Figure 4.9 FESEM images of synthesized CNTs that using method of (a) Diffusion flame at 20K magnification, (b) Diffusion flame at 50K magnification, (c) Chemical Vapor Deposition (CVD) at 20K magnification and (d) Chemical Vapor Deposition (CVD) at 50K magnification.

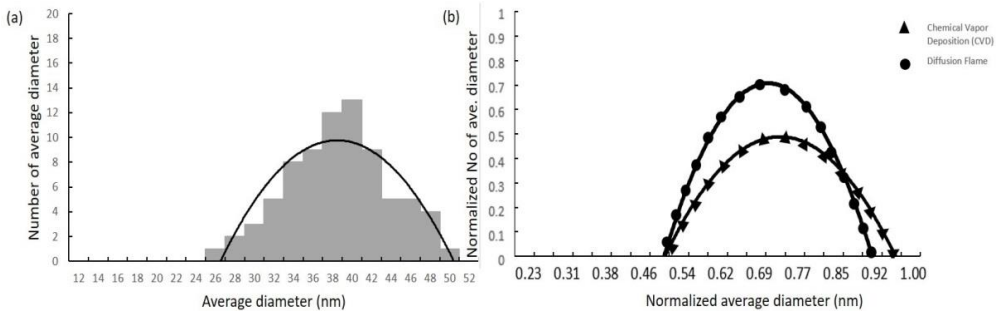

Figure 4.10 (a) the distribution curve and histogram of Chemical Vapor Deposition (CVD) with unnormalized axis (b) the combination distribution curve of average diameter of CNTs from both Diffusion flame and Chemical Vapor Deposition (CVD) with normalized axis.

4.4 The Effect of Different Techniques and Oxidizer Towards the Growth of CNTs

Besides substrate that give the effect on growth of CNTs, study on different sample preparation techniques also have done to investigated if there is some effect or not. The technique of this present study which is dipping technique was compared with dropping technique. Both techniques are quiet similar to each other but for the dropping technique, the sample preparation are followed as explained in section 3.3.1. The other differences in experimental condition between dipping and dropping technique, both are using air and oxygen as their oxidizer respectively. Furthermore, the exposure time of the sample from dropping technique is 3 minutes and compared with Figure 4.1 (i).

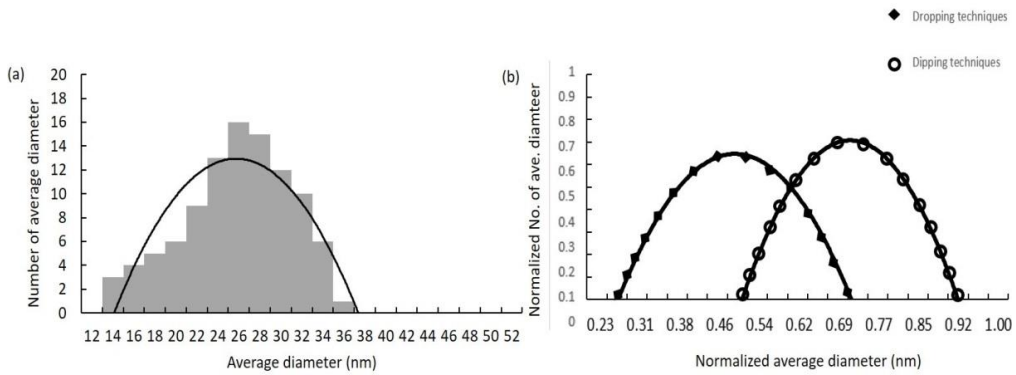

Figure 4.11 (a) the distribution curve and histogram of dropping technique with unnormalized axis (b) the combination distribution curve of average diameter of CNTs from both Dipping and Dropping techniques with normalized axis.

Figure 4.11 shows the analysis from FESEM images from respective technique that are reach a complete growth of CNTs in flame environment. Moreover, the average diameter from dropping techniques is smaller which the higher number is at 26 nm as compare with dipping technique at 38 nm. This smaller value of diameter may cause by the usage of oxidizer that contain oxygen. The benefit of this oxygen, it can accelerate pyrolysis reaction which then can deactive the catalyst particles from growing into CNTs [29], [31], so that it can reach the matured CNTs more faster than the usage of air as oxidizer. Therefore, it is found that for complete growth of CNTs by using dropping technique that used wire mesh and oxygen in oxidizer, the synthesized CNTs is much smaller.In order to get the normalized axis as shown in Figure 4.11 (b), all the actual value of y-axis and x-axis is divided by 20 and 52 respectively.

4.5 Crystallinity Analysis of CNTs

Spectroscopic analysis is found to be more useful to indirectly characterize nanomaterial structure and properties representative of the bulk sample. Analysis of vibrational modes in CNTs structures by Raman Spectroscopy has proved to be a great utility in resolving structural properties of carbon nanotubes.

4.5.1 Graphitic Properties

Through the whole process of flame synthesis, the synthesized CNTs produces nearly same trends intensity of Raman Spectroscopy of the peaks and full width half maximum (FWHM) of the peak bands. Figure 4.12 shows an example of a standard CNTs spectra study of Raman synthesized in the present work. Since the MWCNT is made up of concentric graphene sheets, the Raman spectra peaks of the first order are similar to other graphite-like materials which are D peak and G peak around 1359 cm^{-1} and 1593 cm^{-1} respectively. The intensity of D peaks is representative of defects on the MWCNT walls for instances impurities with sp^3 bonding and fractured sp^2 bonding on the side walls [32], [33]. While the G peaks intensity shows the graphitic nature ofthe sample for example pristine arrangement and crystallinity of the carbon atoms [33]. As

shown in Figure 4.12, the second-order Raman, visible in all samples tested which are G‘peak around 2711.7 cm^{-1} and G+D peak around 2926.02 cm^{-1} . The G‘peak band is the representative of long-range order of the sample produced by the two-phonon, second-order scattering [34].

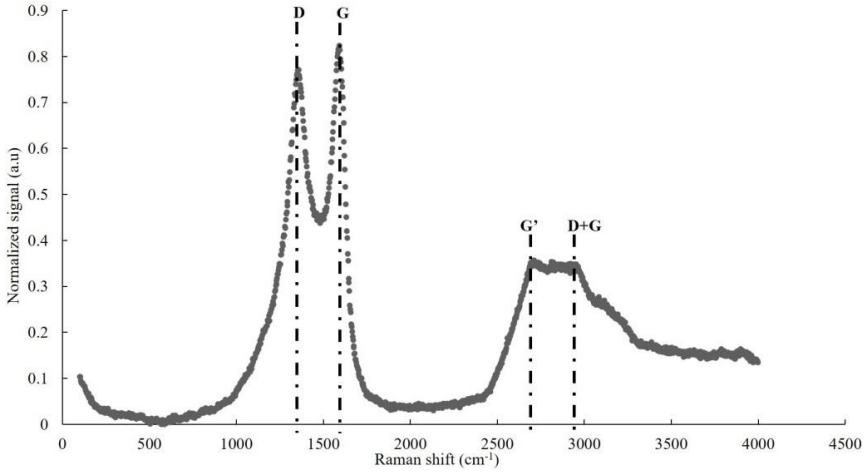

Figure 4.12 Raman spectra of CNT synthesized in diffusion flame.

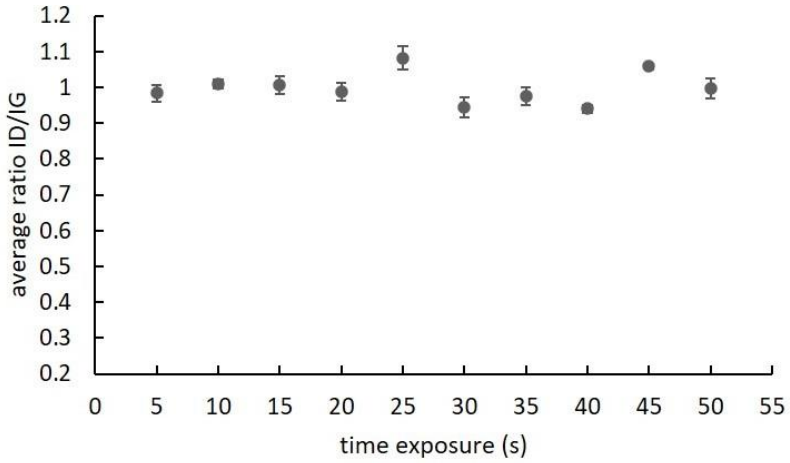

Figure 4.13 Overall Raman Spectra analysis increment of time exposure for 60 seconds.

To make sure the sampling method follow the consistensy for the Raman analysis, a key measurable factor tested such the effect of time exposure in flame toward the measured Raman spectroscopy was analysed. Table 4.2 shows The summarized result of the research done by Raman analysis on ten set of samples. For each sample, the analysis of Raman is performed in the middle of the CNT growth region. The ID / IG ratio indicates continuity around the board. The accuracy of the measurement

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

Flame-based synthesis of carbon nanotube has tremendous potential for large scale production. This is because the synthesis process of carbon nanotube in flame environment is a simple and economical process as compared to the common conventional method of CVD method. However, up to this moment, there is a great challenge faced by researchers for controlling a repetitive good quality of synthesized carbon nanotubes in flame due to the physical behaviour of combustion flame. At the premature stage, deactivation of the catalyst encapsulation of nanoparticle catalysts by amorphous carbon layer affected by carbon oversupply is one of the main factors that prevent effectiveness of the flame environment to produce carbon nanotube. In addition, the preferable catalyst preparation method using nickel nitrate on silicon wafer has yet to be understood for the optimization purpose. Hence, to further develop the optimization process, the main objective of the present study is to establish a baseline catalyst preparation method using nickel nitrate on silicon wafer. As stated in Section 4.1.1, the established sample preparation has proven the existence of synthesized CNTs in flame environment using silicon wafer as a substrate-supported catalyst. Another factor that contributed towards this baseline condition was the size of the substrate silicon wafer used to expose the sample in methane diffusion flame that was affected by the restricted size of concentric stainless steel tube as explained in section 3.3.2. Based on Figure 3.2, the optimum size of silicon wafer can be varied from 1cm x 0.5cm to 1cm x 1 cm in square shape.

The effects of catalyst and flame preparation parameter on CNTs growth in flame were directly observed while the synthesized CNTs morphology was analyzed using FESEM and Raman spectra. For the catalyst preparation method, this study used different substrate and technique which are by comparing the silicon wafer subsrate

and nickel wire as well as dipping and dropping technique respectively. For the flame parameter, this study also is done at varying exposure time for each samples with increment of 5 seconds for 60 seconds. As a result, the diameter increasing with time at first 35 seconds before reached its steady state onwards. In the meantime, silicon wafer revealed a larger size of diameter compared to nickel wire and dipping process revealed a larger size of diameter compared to dropping. The present work also have been extended by comparing the method of synthesized CNTs in diffusion flame and CVD. There are no significant difference in terms of diameter size from both method but an interesting observation is found at the allignment between both method. CVD shows better visual allignment as compared to methane diffusion flame. Last but not least, CNTs are among the most significant materials in contemporary nanoscience and nanotechnology, including molecular role in the evolution of this interdisciplinary area.

5.2 Recommendation for Future Research on CNTs Synthesis in Flame

Based on the findings the progress made in the present study, the goal of the following remarks is to include a guide to further explore the full potential of the methane diffusion flame for future work:

1. Detail analysis on the properties of these nanostructures like density, surface area and weight need to be done.
2. Further refinement of experiment on the concentration of the nickel nitrate solution to be used on the surface of silicon wafer substrate to explore the optimum concentration that may covered all the surface in terms of improvement on morphology and catalyst lifetime.
3. Development and utilization of fixed substrate holder especially for silicon wafer instead of using wire mesh. A proper designed of substrate holder have been suggested and made using 3-Dimensional model in Solidwork. This leads the sample to be firmly positioned in the slot and to ensure a fixed horizontral position of the sample. The used of stainless steel plate of thickness 0.5 mm are highly recommended.

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ABSTRACT

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