CHARACTERIZATION OF CARBON NANOTUBE GROWTH REGION IN FLAME USING WIRE-BASED MACRO-IMAGING METHOD

NORIKHWAN HAMZAH

A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

School of Mechanical Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

AUGUST 2020

DEDICATION

To Nurul Adilla and Isaac Muqri

ACKNOWLEDGEMENT

Thanks to Allah, the Most Gracious, the Most Merciful, the Most Bountiful who gave me the courage and patience to accomplish this research work. Without his help and mercy, this would not have come into reality.

Completion of this work would not have been possible without the help and support of numerous people, both at UTM and in my personal life. Firstly, my sincere thanks are due to my supervisor Dr Mohd Fairus Mohd Yasin for his assistance, valuable discussion, guidance, and direction throughout my research work. His confidence in providing me with a level of independence in the experimental works has made this doctoral journey an exciting experience. I am also very thankful for the guidance and encouragement from Dr Mohd Zamri Mohd Yusop, which the discussion session that we had has helped me in many ways, especially related to materials analysis aspect of the work.

I am extremely thankful to the Hiref research group members and colleagues which have created a conducive research environment in the lab. Especially to Mr Ahmad Dairobi who always being helpful and willing to share his valuable technical skills which I have greatly benefited from. Also, to other colleagues for their valuable discussion in research and life related topics, and in their understanding and cooperation in performing our lab works. This work would have taken much longer to be completed without a supporting, helpful and friendly lab members.

I am also extremely thankful to my family for supporting me in my decision to leave the industry to become a graduate student. Thank you to my parents and my in-laws for your endless support and encouragement as I pursued my dreams. Special thanks to my wife, who never wavered in her support, patience, and confidence in me. Also, I would like to thank my son for helping me get my mind off the research when I need it the most. Most importantly, I am grateful to God for all the blessings He has bestowed on me.

ABSTRACT

Carbon nanotube (CNT) synthesis in flame has enormous potential as an energy-efficient and economical production method compared to the conventional catalytic chemical vapor deposition (CCVD) synthesis process. However, synthesis control remains a great challenge for flame synthesis due to the limited understanding on the effect of flame inlet condition toward CNT growth region in a heterogeneous flame environment and premature catalyst surface encapsulation by the amorphous carbon layer. The present study formulates a simple, yet accurate method called wire-based macro image analysis (WMA) for thorough growth region identification. The WMA method is employed to investigate the effects of reactant composition and aerodynamics on the spatial distribution of CNT growth region. Besides that, bend wire method is developed to provide cross-sectional analysis of the CNT growth region with focus on the amorphous carbon layer thickness (ACLT) at variable reactant concentration including fuel from 50% to 100% and oxygen from 19% to 27%, with addition of water vapor up to 0.14 mg/sec mass flow rate within the fuel stream. The CNT is synthesized on a 0.4 mm diameter pure nickel wire within the methane diffusion flame with a stainless-steel wire mesh placed on top and water vapor is introduced in a fuel stream using a bubbler mechanism. The CNT growth region is confined within the flame sheet, gradually shifts from flame front to flame centreline as height above the burner increases. The growth region is more sensitive towards the change in the oxygen concentration compared to that of the fuel concentration due to the significant change of flame height caused by the former. A segregation of growth region temperature with temperature difference of 100 °C that is observed between the upstream and downstream growth region is governed by the proximity with respect to the flame sheet. The ACLT reduces in lean flame due to the reduction in excess carbon concentration and the addition of water vapor remarkably reduces ACLT by 17% on average in any combination of inlet conditions due to the water-induced etching and oxidation of amorphous carbon on the catalyst surface. Development of the WMA and bend wire method leads to deeper fundamental understanding of CNT flame synthesis and further enhance possibility of highly efficient and economical CNT production process in the future.

ABSTRAK

Sintesis karbon tiub nano (CNT) menggunakan api mempunyai potensi besar sebagai kaedah pembuatan yang cekap tenaga dan ekonomik jika dibandingkan dengan proses sintesis konvensional menggunakan pemangkin deposit wap kimia pemangkin (CCVD). Walau bagaimanapun, kawalan sintesis dalam api menjadi cabaran utama disebabkan oleh persekitaran heterogen dan enkapsulasi pramatang permukaan pemangkin oleh lapisan karbon amorfus. Kajian ini mencipta satu kaedah yang mudah dan tepat yang dinamakan sebagai analisis imej makro berasaskan wayar (WMA) untuk pengenalpastian ruang pertumbuhan CNT. Analisis WMA digunakan untuk mengkaji kesan komposisi reaktan dan aerodinamik pada ruang pertumbuhan CNT di dalam api. Di samping itu, kaedah lenturan wayar dicipta untuk analisis keratan rentas ruang pertumbuhan CNT dengan memberikan tumpuan khusus kepada ketebalan lapisan karbon amorfus (ACLT) pada komposisi reaktan berbeza termasuklah kepekatan bahan bakar dari 50% hingga 100% dan oksigen dari 19% ke 27% dengan tambahan wap air sehingga 0.14 mg/s di dalam aliran bahan bakar. CNT disintesis pada dawai nikel tulen dalam api di bawah jaring keluli tahan karat dan wap air ditambah dalam aliran bahan bakar menggunakan mekanisma gelembung. Ruang pertumbuhan CNT terkurung di dalam kawasan nyalaan, secara beransur-ansur beralih dari tepi ke tengah kawasan nyalaan dengan peningkatan ketinggian di atas pembakar. Ruang pertumbuhan lebih sensitif terhadap perubahan kepekatan oksigen berbanding bahan api kerana perubahan panjang nyalaan yang disebabkan oleh kepekatan oksigen. Perbezaan suhu ruang pertumbuhan setinggi 100 °C di antara ruang pertumbuhan hulu dan hiliran nyalaan adalah disebabkan oleh kedekatan dengan lapisan nyalaan. ACLT berkurang dalam nyalaan yang kurang kerana pengurangan kepekatan karbon yang berlebihan dan penambahan wap air membantu mengurangkan ACLT sebanyak 17% dalam sebarang gabungan komposisi salur masuk. Ini disebabkan oleh pengoksidaan dan penghakisan karbon amorfus yang disebabkan oleh wap air pada permukaan pemangkin. Penciptaan kaedah analisis WMA dan lenturan wayar membantu menjadikan proses CNT sintesis di dalam api lebih difahami secara mendalam dan menambah kebarangkalian untuk proses pembuatan CNT yang sangat efisien dan ekonomik di masa hadapan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AAO	-	Anodic aluminium oxide
ACLT	-	Amorphous carbon layer thickness
AFM	-	Atomic force microscopy
CCVD	-	Catalytic chemical vapor deposition
CNF	-	Carbon nanofiber
CNT	-	Carbon nanotubes
CNO	-	Carbon nano-onion
DSLR	-	Digital single-lens reflex
EDX	-	Energy-dispersive X-ray spectroscopy
FESEM	-	Field emission scanning electron microscope
FWHM	-	Full width at half maximum
HAB	-	Height above burner
HR-TEM	-	High-resolution transmission electron microscopy
IPM	-	Interacting particle model
LED	-	Light emitting diode
m-IDF	-	Multiple-inverse diffusion flame
MWNT	-	Multiwalled carbon nanotubes
PAH	-	Polycyclic aromatic hydrocarbon
PVD	-	Physical vapor deposition
RBM	-	Radial breathing mode
SEM	-	Scanning electron microscope
STEM	-	Scanning transmission electron microscopy
SWNT	-	Single-walled carbon nanotubes
TEM	-	Transmission electron microscope
TGA	-	Thermal gravimetric analysis
WMA	-	Wire-based macro-image analysis

NOMENCLATURE

T_{growth}	-	Growth region temperature
T_{fc}	-	Flame centreline temperature
T_{fs}	-	Flame sheet temperature
I_D, I_G, $I_{G'}$	-	D, G, and G' band intensity
%RH	-	Percentage of relative humidity
P_w	-	Water vapor pressure
P_{ws}	-	Saturation water vapor pressure
P_{tot}	-	Total pressure
M_w	-	Molecular mass of water
M_d	-	Molecular mass of dry methane gas
ppm_w	-	Parts per million weight
φ	-	Equivalence ratio
L_f	-	Flame length
Q_F	-	Fuel volumetric flow rate
T_∞	-	Oxidizer temperature
T_F	-	Fuel temperature
S	-	Molar stoichiometric oxidizer-fuel ratio
χo_2	-	Mole fraction of oxygen in the oxidizer
χ_{dil}	-	Diluent mole fraction in the fuel stream
m_{fuel}	-	Mass flow rate of fuel
$m_o x$	-	Mass flow rate of oxidizer
Z_{st}	-	Stoichiometric mixture fraction

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Problem Background

The discovery of a third carbon allotrope, Buckminsterfullerene C60 in 1985 by Kroto, Curl, Smalley, and co-workers marked the beginning of an era of carbon nanostructured materials [1]. With remarkable electrical, thermal, and mechanical properties, coupled with rapid development of nanoscience and nanotechnology, last three decades has seen the emergence of carbon nanostructures in various application for modern society. Nobel Prize that are awarded to Curl, Kroto, and Smalley for the work on Buckminsterfullerene, and to Geim and Novoselov for extracting single layer carbon atoms known as graphene in 1996 and 2010 respectively, are clear indications of the significant contribution of carbon nanostructured material to the society [2,3].

Combustion-based synthesis of carbon nanotube (CNT) has significant potential to revolutionize the conventional CNT synthesis process. The combination of the autothermal condition and the continuous supply of carbon sources within flame produce an energy-efficient synthesis process. Theoretically, the flame synthesis is capable to synthesize CNT at an order of magnitude faster compared to that of the catalytic chemical vapor deposition (CCVD) to significantly reduce the overall cost of the CNT production [4,5]. Furthermore, the process is technically simple to be scaled up for large volume production, proven with current production of widely used nanoparticles such as carbon black, fumed silica, and titania in the order of millions of tons valued at over 15 billion dollar per year [6,7]. These compelling fundamental advantages of flame synthesis process have driven researchers to enhance the understanding on the flame synthesis process for the last three decades. As the CNT market continues to expand each year, the search for a novel and optimized synthesis process for producing low-cost CNT remains strong [5]. Numerous research groups are currently active in exploring new possibilities to exploit the advantages of combustion-based CNT synthesis to produce an improved synthesis method through experimental works and modelling [4,8–12].

However, limited progress has been reported in the development of industrial scale of CNT synthesis process in flame. The development of a stable and well-controlled synthesis process within highly complex flame environment has been proven to be the stumbling block for mainstream acceptance of the technology [5,7]. Compared to CCVD, the degree of complexity of the CNTs growth is significantly amplified in flame environment due to the combustion process that creates dynamic gas phase kinetics with significant temperature gradient within the flame.

1.2 Problem Statement

Even though theoretically flame synthesis is capable of producing similar yield of CNTs at much higher growth rate compared to that of CCVD, none of the recorded lab-scale experiments able to achieve major breakthrough for mass production to the best of author's knowledge. The complex interrelation between fuel, oxidizer, and temperature that are coupled with other synthesis parameters like catalyst type and composition, lead to highly complex optimization parameter space which requires extensive analysis on the effect of flame inlet condition toward certain specific set of synthesis parameters toward CNT growth region distribution. Unfortunately, conventional method of CNT growth region identification using repetitive scanning electron microscopy (SEM) analysis is impractical especially for large-area heterogeneous flame synthesis environment which inhibit a comprehensive parametric study on spatial distribution of CNT growth region in flame to be performed.

Besides practical and precise growth region identification, one of the main obstacles in optimizing CNT synthesis in flame is maintaining the delicate balance of carbon supply rate and flame temperature. Oversupply of carbon will lead to the formation of the amorphous carbon layer which will encapsulate catalyst nanoparticles and caused a premature catalyst poisoning. On the other hand, the oversupply of carbon in most cases is dictated by the amount of fuel required to achieved desirable temperature within the flame for CNT inception and growth. The formation of amorphous carbon layer on catalyst nanoparticles has been extensively studied in the context of CCVD. Application of additive such as water vapor in CCVD process has been widely accepted as an effective method of minimizing formation of amorphous carbon. The same has not been systematically studied in the context of flame synthesis. Limited understanding on the effect of flame inlet condition on formation of amorphous carbon layer on catalyst nanoparticles in flame environment hinders further optimization of the CNT synthesis process in flame.

1.3 Hypothesis

CNT growth region surface is generally recognizable by its characteristically deep black colour covered with dense CNT. Utilizing concept of colour segregation, the CNT growth region is expected to produce high contrast to the surrounding substrate surface and can be visually distinguishable using high magnification image which allow for a low cost and practical growth region identification process.

Since the catalytic growth mechanism of CNT in CCVD and flame environment are similar, it is expected that reduction of amorphous carbon layer on the catalyst nanoparticles can be minimize through precise control over carbon supply rate on the catalyst nanoparticles surface. Manipulation flame inlet condition in diffusion flame will have significant effects toward the carbon supply rates in region within the flame front which will has significant effect on the distribution of CNT growth region and formation of amorphous carbon layer. Additionally, water vapor as a weak oxidizer is expected to produce etching effects on the amorphous carbon layer on the substrate-supported catalyst surface in the flame environment as has been shown in the synthesis chamber environment of CVD. However, water vapor may also inadvertently change the local temperature and species distribution in flames. Though the etching effects are desirable, an ideal temperature and carbon precursor concentration must be maintained to achieve the desired CNT synthesis characteristics in the flame.

1.4 Objectives of the Study

The objectives of the present study are:

1. To determine the effect of flame inlet condition toward the distribution of CNT growth region in methane diffusion flame using wire-based macro-image method.
2. To analyse the effect of flame inlet condition toward the thickness of amorphous carbon layer within CNT growth region using cross-sectional analysis.

1.5 Significance of the Study

The present study analyses the effect of flame inlet conditions toward spatial distribution of CNT growth region and formation of amorphous carbon layer. The objectives will be achieved through development of a novel CNT growth region identification method using wire-based macro-image analysis (WMA) method and amorphous carbon layer analysis using cross-sectional imaging technique through bend wire method. The simplification of growth region identification using WMA method allow in depth and thorough growth region distribution analysis in flame with varied inlet condition. Whereas the bend wire method reveals the cross-sectional condition within the growth region and enable extensive study on the effect of flame condition toward the formation of amorphous carbon layer on the substrate surface. Furthermore, the work also explores effects of water vapor in fuel stream toward CNT growth region, temperature, and formation of amorphous carbon layer. The analyses will provide fundamental understanding on the etching effect of water on catalytic growth of CNTs in flame. It is envisaged that the develop methodologies and findings in the present study will be the foundation toward further development of effective manipulation of flame parameters especially for enhancement of the yield, growth rate, and the quality of CNTs produced in flame synthesis in the future.

1.6 Research Scope

The present study is an experimental-only work on CNT growth region characterization and amorphous carbon formation analysis using wire-based macro-image analysis and bend wire method respectively within methane diffusion flame. Pure methane gas is employed to ensure consistency and significant size of growth region for measurement reliability. Dilution of methane is done through addition of pure nitrogen gas to the fuel stream. Mixture of pure oxygen and nitrogen gas were utilized to provide desired concentration in the oxidizer stream, whereas in the oxidizer flow rate experiments, compressed dry air is employed for economical purposes. Pure nickel wire with 0.4 mm diameter without any pre-treatment is used as catalyst. The utilization of the wire provide consistency in terms of catalyst preparation and eliminate any contributing factor in catalyst preparation toward the effect of CNT growth. The established flame characterization done through direct image capture and temperature measurement. The growth region is characterized through measurement of spatial distribution and temperature. The synthesized CNT is analysed using FESEM, TEM, EDX, and Raman spectra analysis only.

REFERENCES

[1] Kroto HW, Heath JR, O'Brien SC, Curl RF, Smalley RE. C 60: Buckminsterfullerene. *Nature*, 1985. 318: 162.

[2] The Nobel Prize in Chemistry 1996. The Nobel Prize Website. http://www.nobelprize.org/nobel_prizes/chemistry/laureates/1996/. Published 1996. Accessed November 1, 2017.

[3] The Nobel Prize in Physics 2010. The Nobel Prize Website. http://www.nobelprize.org/nobel_prizes/physics/laureates/2010/. Published 2010. Accessed November 1, 2017.

[4] Okada S, Sugime H, Hasegawa K, et al. Flame-assisted chemical vapor deposition for continuous gas-phase synthesis of 1-nm-diameter single-wall carbon nanotubes. *Carbon N Y*, 2018. 138: 1-7.

[5] Hamzah N, Yasin MFM, Yusop MZM, Saat A, Subha NAM. Rapid production of carbon nanotubes: a review on advancement in growth control and morphology manipulations of flame synthesis. *J Mater Chem A*, 2017. 5(48): 25144-25170.

[6] Li S, Ren Y, Biswas P, Tse SD. Flame aerosol synthesis of nanostructured materials and functional devices: Processing, modeling, and diagnostics. *Prog Energy Combust Sci*, 2016. 55: 1-59.

[7] Haynes BS. Combustion research for chemical processing. *Proc Combust Inst*, 2019. 37(1): 1-32.

[8] Suzuki S, Mori S. Synthesis of carbon nanotubes from biofuel as a carbon source through a diesel engine. *Diam Relat Mater*, 2018. 82(May 2017): 79-86.

[9] Padilla O, Gallego J, Santamaría A. Using benzene as growth precursor for the carbon nanostructure synthesis in an inverse diffusion flame reactor. *Diam Relat Mater*, 2018. 86: 128-138.

[10] Suzuki S, Mori S. Considerations on the key precursor for the growth of carbon nanotubes using a diesel engine as a reactor. *Chem Eng Sci*, 2018. 186: 62-73.

[11] Zhao T, Li X, Yan H. Metal catalyzed preparation of carbon nanomaterials by hydrogen–oxygen detonation method. *Combust Flame*, 2018. 196: 108-115.

[12] Wu J, Liang K, Yang C, Zhu J, Liu D. Synthesis of carbon nanotubes on metal

mesh in inverse diffusion biofuel flames. *Fullerenes Nanotubes and Carbon Nanostructures*, 2019. 27(1): 77-86.

[13] Tessonnier J-P, Su DS. Recent progress on the growth mechanism of carbon nanotubes: a review. *ChemSusChem*, 2011. 4(7): 824-847.

[14] Jourdain V, Bichara C. Current understanding of the growth of carbon nanotubes in catalytic chemical vapour deposition. *Carbon N Y*, 2013. 58: 2-39.

[15] Merchan-Merchan W, Saveliev A V., Kennedy L, Jimenez WC. Combustion synthesis of carbon nanotubes and related nanostructures. *Prog Energy Combust Sci*, 2010. 36(6): 696-727.

[16] Baker RTK. Catalytic growth of carbon filaments. *Carbon N Y*, 1989. 27(3): 315-323.

[17] Dai H, Rinzler AG, Nikolaev P, Thess A, Colbert DT, Smalley RE. Single-wall nanotubes produced by metal-catalyzed disproportionation of carbon monoxide. *Chem Phys Lett*, 1996. 260: 471-475.

[18] Height MJ, Howard JB, Tester JW, Vander Sande JB. Carbon nanotube formation and growth via particle-particle interaction. *J Phys Chem B*, 2005. 109(25): 12337-12346.

[19] Amelinckx S, Zhang XB, Bernaerts D, Zhang XF, Ivanov V, Nagy JB. A formation mechanism for catalytically grown helix-shaped graphite nanotubes. *Science*, 1994. 265: 635-639.

[20] Pan C, Liu Y, Cao F, Wang J, Ren Y. Synthesis and growth mechanism of carbon nanotubes and nanofibers from ethanol flames. *Micron*, 2004. 35(6): 461-468.

[21] Unrau CJ, Axelbaum RL, Fraundorf P. Single-walled carbon nanotube formation on iron oxide catalysts in diffusion flames. *J Nanoparticle Res*, 2010. 12(6): 2125-2133.

[22] Unrau CJ, Axelbaum RL, Lo CS. High-yield growth of carbon nanotubes on composite Fe/Si/O nanoparticle catalysts: A Car-Parrinello molecular dynamics and experimental study. *J Phys Chem C*, 2010. 114(23): 10430-10435.

[23] Suzuki S, Mori S. Flame synthesis of carbon nanotube through a diesel engine using normal dodecane/ethanol mixing fuel as a feedstock. *J Chem Eng Japan*, 2017. 50(3): 178-185.

[24] Chong CT, Tan WH, Lee SL, Chong WWF, Lam SS, Valera-Medina A. Morphology and growth of carbon nanotubes catalytically synthesised by

premixed hydrocarbon-rich flames. *Mater Chem Phys*, 2017. 197: 246-255.

[25] Tan WH, Lee LS, Ng J-H, Chong WWF, Chong CT. Characterization of carbon nanotubes synthesized from hydrocarbon-rich flame. *Int J Technol*, 2016. 2: 343-351.

[26] Oulanti H, Laurent F, Le-Huu T, Durand B, Donnet JB. Growth of carbon nanotubes on carbon fibers using the combustion flame oxy-acetylene method. *Carbon N Y*, 2015. 95: 261-267.

[27] Hou S-S, Chen K-M, Yang Z-Y, Lin T-H. Enhanced synthesis of carbon nanomaterials using acoustically excited methane diffusion flames. *Materials (Basel)*, 2015. 8(8): 4805-4816.

[28] Han W, Chu H, Ya Y, Dong S, Zhang C. Effect of fuel structure on synthesis of carbon nanotubes in diffusion flames. *Fullerenes Nanotubes and Carbon Nanostructures*, 2019. 27(3): 265-272.

[29] Zheng L, Bao C, Lei S, et al. In situ growing CNTs encapsulating nickel compounds on Ni foils with ethanol flame method as superior counter electrodes of dye-sensitized solar cells. *Carbon N Y*, 2018. 133: 423-434.

[30] Xiong, Zhao, Ren, Cui, Ji. Flame-synthesis of carbon nanotube forests on metal mesh structure: Dependence, morphology, and application. *Nanomaterials*, 2019. 9(9): 1188.

[31] Hong H, Memon NK, Dong Z, Kear BH, Tse SD. Flame synthesis of gamma-iron-oxide (y-Fe_2O_3) nanocrystal films and carbon nanotubes on stainless steel substrate. *Proc Combust Inst*, 2019. 37(1): 1249-1256.

[32] Memon NK, Kear BH, Tse SD. Transition between graphene-film and carbon-nanotube growth on Nickel alloys in open-atmosphere flame synthesis. *Chem Phys Lett*, 2013. 570: 90-94.

[33] Memon NK, Xu F, Sun G, Dunham SJB, Kear BH, Tse SD. Flame synthesis of carbon nanotubes and few-layer graphene on metal-oxide spinel powders. *Carbon N Y*, 2013. 63: 478-486.

[34] Wang Y, Gao X, Qian H-J, et al. Quantum chemical simulations reveal acetylene-based growth mechanisms in the chemical vapor deposition synthesis of carbon nanotubes. *Carbon N Y*, 2014. 72: 22-37.

[35] Hu W-C, Lin T-H. Ethanol flame synthesis of carbon nanotubes in deficient oxygen environments. *Nanotechnology*, 2016. 27(16): 1-10.

[36] Hu W-C, Hou S-S, Lin T-H. Transition of carbon nanostructures in heptane

diffusion flames. *J Nanoparticle Res*, 2017. 19(2): 82.

[37] Sun BM, Cao WH, Guo YH, Wang Y, Luo JT, Jiang P. Flame pyrolysis synthesis of self-oriented carbon nanotubes. *AIP Adv*, 2013. 3: 1-7.

[38] Sun Y, Sun B, Kang Z-Z, Guo Y-H, Jia X-W. Impact of support calcination and competitive adsorbate in Fe/Mo-Al_2O_3 catalyst for synthesis of carbon nanotubes by V-flame. *Mater Res Express*, 2018. 5: 1-9.

[39] Guo Y, Zhai G, Ru Y, et al. Effect of different catalyst preparation methods on the synthesis of carbon nanotubes with the flame pyrolysis method. *AIP Adv*, 2018. 8: 1-11.

[40] Gore JP, Sane A. Flame synthesis of carbon nanotubes. In: Siva Yellampalli, ed. *Carbon Nanotubes: Synthesis, Characterization, Applications*. InTech Open; 2011:121-146.

[41] Vander Wal RL, Hall LJ, Berger GM. Optimization of flame synthesis for carbon nanotubes using supported catalyst. *J Phys Chem B*, 2002. 106(51): 13122-13132.

[42] Richter H, Treska M, Howard JB, et al. Large scale combustion synthesis of single-walled carbon nanotubes and their characterization. *J Nanosci Nanotechnol*, 2008. 8(11): 6065-6074.

[43] Xu F, Liu X, Tse SD. Synthesis of carbon nanotubes on metal alloy substrates with voltage bias in methane inverse diffusion flames. *Carbon N Y*, 2006. 44(3): 570-577.

[44] Hou S-S, Huang W-C. Influence of oxygen concentration, fuel composition, and strain rate on synthesis of carbon nanomaterials. *J Nanoparticle Res*, 2015. 17(2): 1-11.

[45] Kimura H, Goto J, Yasuda S, et al. The infinite possible growth ambients that support single-wall carbon nanotube forest growth. *Sci Rep*, 2013. 3: 1-6.

[46] Vander Wal RL, Ticich TM, Curtis VE. Diffusion flame synthesis of single-walled carbon nanotubes. *Chem Phys Lett*, 2000. 323: 217-223.

[47] Diener MD, Nichelson N, Alford JM. Synthesis of single-walled carbon nanotubes in flames. *J Phys Chem B*, 2000. 104(41): 9615-9620.

[48] Hall B, Zhuo C, Levendis YA, Richter H. Influence of the fuel structure on the flame synthesis of carbon nanomaterials. *Carbon N Y*, 2011. 49(11): 3412-3423.

[49] Suzuki S, Mori S. Carbon nanotube-like materials in the exhaust from a diesel engine using gas oil/ethanol mixing fuel with catalysts and sulfur. *J Air Waste*

Manage Assoc, 2017. 67(8): 873-880.

[50] Vander Wal RL, Ticich TM. Flame and furnace synthesis of single-walled and multi-walled carbon nanotubes and nanofibers. *J Phys Chem B*, 2001. 105(42): 10249-10256.

[51] Yuan L, Saito K, Hu W, Chen Z. Ethylene flame synthesis of well-aligned multi-walled carbon nanotubes. *Chem Phys Lett*, 2001. 346: 23-28.

[52] Yuan L, Saito K, Pan C, Williams FA, Gordon AS. Nanotubes from methane flames. *Chem Phys Lett*, 2001. 340: 237-241.

[53] Hou SS, Huang WC. Flame Synthesis of carbon nanotubes in a rotating counterflow. *J Nanosci Nanotechnol*, 2009. 9: 4826-4833.

[54] Hou SS, Huang WC. Flame synthesis of carbon nanotubes and nano-onions in a rotating opposed-jets flow. *Appl Mech Mater*, 2013. 284-287(8): 310-314.

[55] Hu W-C, Hou S-S, Lin T-H. Analysis on controlling factors for the synthesis of carbon nanotubes and nano-onions in counterflow diffusion flames. *J Nanosci Nanotechnol*, 2014. 14(7): 5363-5369.

[56] Hou SS, Chung DH, Lin TH. High-yield synthesis of carbon nano-onions in counterflow diffusion flame. *Carbon N Y*, 2008. 47(4): 938-947.

[57] Li TX, Kuwana K, Saito K, Zhang H, Chen Z. Temperature and carbon source effects on methane-air flame synthesis of CNTs. *Proc Combust Inst*, 2009. 31: 1855-1861.

[58] Li TX, Zhang HG, Wang FJ, Chen Z, Saito K. Synthesis of carbon nanotubes on Ni-alloy and Si-substrates using counterflow methane-air diffusion flames. *Proc Combust Inst*, 2007. 31: 1849-1856.

[59] Xu F, Zhao H, Tse SD. Carbon nanotube synthesis on catalytic metal alloys in methane/air counterflow diffusion flames. *Proc Combust Inst*, 2007. 31: 1839-1847.

[60] Saveliev A V., Merchan-Merchan W, Kennedy LA. Metal catalyzed synthesis of carbon nanostructures in an opposed flow methane oxygen flame. *Combust Flame*, 2003. 135: 27-33.

[61] Merchan-Merchan W, Saveliev A V., Kennedy LA. High-rate flame synthesis of vertically aligned carbon nanotubes using electric field control. *Carbon N Y*, 2004. 42(3): 599-608.

[62] Merchan-Merchan W, Saveliev A V., Kennedy LA. Flame nanotube synthesis in moderate electric fields: From alignment and growth rate effects to structural

variations and branching phenomena. *Carbon N Y*, 2006. 44: 3308-3314.

[63] Vander Wal RL. Flame synthesis of substrate-supported metal-catalyzed carbon nanotubes. *Chem Phys Lett*, 2000. 324: 217-223.

[64] Liu Y, Fu Q, Pan C. Synthesis of carbon nanotubes on pulse plated Ni nanocrystalline substrate in ethanol flames. *Carbon N Y*, 2005. 43(11): 2264-2271.

[65] Arana CP, Puri IK, Sen S. Catalyst influence on the flame synthesis of aligned carbon nanotubes and nanofibers. *Proc Combust Inst*, 2005. 30(2): 2553-2560.

[66] Chung DH, Lin TH. Nitrogen dilution effect on flame synthesis of carbon nanostructures with acoustic modulation. *J Phys Chem C*, 2011. 115: 16287-16294.

[67] Bao Q, Pan C. Electric field induced growth of well aligned carbon nanotubes from ethanol flames. *Nanotechnology*, 2006. 17: 1016-1021.

[68] Zhang J, Pan C. Magnetic-field-controlled alignment of carbon nanotubes from flames and its growth mechanism. *J Phys Chem C*, 2008. 112(35): 13470-13474.

[69] Du X, Liu HY, Xu F, Zeng Y, Mai YW. Flame synthesis of carbon nanotubes onto carbon fiber woven fabric and improvement of interlaminar toughness of composite laminates. *Compos Sci Technol*, 2014. 101: 159-166.

[70] Du X, Liu HY, Zhou C, Moody S, Mai YW. On the flame synthesis of carbon nanotubes grafted onto carbon fibers and the bonding force between them. *Carbon N Y*, 2012. 50(6): 2347-2350.

[71] Du X, Xu F, Liu H-Y, Miao Y, Guo W-G, Mai Y-W. Improving the electrical conductivity and interface properties of carbon fiber/epoxy composites by low temperature flame growth of carbon nanotubes. *RSC Adv*, 2016. 6(54): 48896-48904.

[72] Yuan L, Li T, Saito K. Growth mechanism of carbon nanotubes in methane diffusion flames. *Carbon N Y*, 2003. 41(10): 1889-1896.

[73] Yuan L, Li T, Saito K. Synthesis of multiwalled carbon nanotubes using methane/air diffusion flames. *Proc Combust Inst*, 2002. 29(1): 1087-1092.

[74] Pan C, Xu X. Synthesis of carbon nanotubes from ethanol flame. *J Mater Sci Lett*, 2002. 21(24): 1207-1209.

[75] Pan C, Bao Q. Well-aligned carbon nanotubes from ethanol flame. *J Mater Sci Lett*, 2002. 21(24): 1927-1929.

[76] Hou SS, Huang WC, Lin TH. Flame synthesis of carbon nanostructures using mixed fuel in oxygen-enriched environment. *J Nanoparticles Res*, 2012. 14: 1-11.

[77] Lee GW, Jurng J, Hwang J. Formation of Ni-catalyzed multiwalled carbon nanotubes and nanofibers on a substrate using an ethylene inverse diffusion flame. *Combust Flame*, 2004. 139: 167-175.

[78] Lee GW, Jurng J, Hwang J. Synthesis of carbon nanotubes on a catalytic metal substrate by using an ethylene inverse diffusion flame. *Carbon N Y*, 2004. 42(3): 682-685.

[79] Vander Wal RL, Hall LJ, Berger GM. The chemistry of premixed flame synthesis of carbon nanotubes using supported catalysts. *Proc Combust Inst*, 2002. 29: 1079.

[80] Gopinath P, Gore J. Chemical kinetic considerations for postflame synthesis of carbon nanotubes in premixed flames using a support catalyst. *Combust Flame*, 2007. 151: 542-550.

[81] Okuno H, Issi JP, Charlier JC. Catalyst assisted synthesis of carbon nanotubes using the oxy-acetylene combustion flame method. *Carbon N Y*, 2004. 43(4): 864-866.

[82] Nakazawa S, Yokomori T, Mizomoto M. Flame synthesis of carbon nanotubes in a wall stagnation flow. *Chem Phys Lett*, 2005. 403: 158-162.

[83] Woo SK, Hong YT, Kwon OC. Flame synthesis of carbon nanotubes using a double-faced wall stagnation flow burner. *Carbon N Y*, 2008. 47: 912-916.

[84] Woo SK, Hong YT, Kwon OC. Flame-synthesis limits and self-catalytic behavior of carbon nanotubes using a double-faced wall stagnation flow burner. *Combust Flame*, 2009. 156: 1983-1992.

[85] Mohd Syahir MS, Chong CT. Synthesis of carbon nanotubes from rich premixed propane/air flame. *Appl Mech Mater*, 2014. 699: 136-140.

[86] Tan WH, Lee SL, Chong CT. TEM and XRD analysis of carbon nanotubes synthesised from flame. *Key Eng Mater*, 2016. 723: 470-475.

[87] Merchan-merchan W, Saveliev A, Kennedy LA, Fridman A. Formation of carbon nanotubes in counter-flow, oxy-methane diffusion flames without catalysts. *Chem Phys Lett*, 2002. 354: 20-24.

[88] Moisala A, Nasibulin AG, Kauppinen EI. The role of metal nanoparticles in the catalytic production of single-walled carbon nanotubes - a review. *J Phys -*

Condens Matter, 2003. 15: 3011-3035.

[89] Wen JZ, Richter H, Green WH, et al. Experimental study of catalyst nanoparticle and single walled carbon nanotube formation in a controlled premixed combustion. *J Mater Chem*, 2008. 18: 1561.

[90] Height MJ, Howard JB, Tester JW, Vander Sande JB, Sande JB Vander. Flame synthesis of single-walled carbon nanotubes. *Carbon N Y*, 2004. 42(11): 2295-2307.

[91] He M, Amara H, Jiang H, et al. Key roles of carbon solubility in single-walled carbon nanotube nucleation and growth. *Nanoscale*, 2015. 7: 20284-20289.

[92] Duan HM, McKinnon JT. Nanoclusters produced in flames. *J Phys Chem*, 1994. 98(49): 12815-12818.

[93] Das Chowdhury K, Howard JB, VanderSande JB. Fullerenic nanostructures in flames. *J Mater Res*, 1995. 11(02): 341-347.

[94] Bang JJ, Guerrero PA, Lopez DA, Murr LE, Esquivel EV. Carbon nanotubes and other fullerene nanocrystals in domestic propane and natural gas combustion streams. *J Nanosci Nanotechnol*, 2004. 4(7): 716-718.

[95] Singh N, Bharj R. Experimental investigation on the role of indigenous carbon nanotube emulsified fuel in a four-stroke diesel engine. *Proc Inst Mech Eng Part C J Mech Eng Sci*, 2016. 230(12): 2046-2059.

[96] Swanson JJ, Febo R, Boies AM, Kittelson DB. Fuel sulfur and iron additives contribute to the formation of carbon nanotube-like structures in an internal combustion engine. *Environ Sci Technol Lett*, 2016. 3(10): 364-368.

[97] Merchan-Merchan W, Saveliev A V., Kennedy LA. Carbon nanostructures in opposed-flow methane oxy-flames. *Combust Sci Technol*, 2003. 175(12): 2217-2236.

[98] Height MJ, Howard JB, Tester JW, Vander Sande JB. Flame synthesis of single-walled carbon nanotubes. *Carbon N Y*, 2004. 42(11): 2295-2307.

[99] Nishino H, Yasuda S, Namai T, et al. Water-assisted highly efficient synthesis of single-walled carbon nanotubes forest from collodial nanoparticle catalysts. *J Phys Chem C*, 2007. 111(48): 17961-17965.

[100] Chen G, Sakurai S, Yumura M, Hata K, Futaba DN. Highly pure, millimeter-tall, sub-2-nanometer diameter single-walled carbon nanotube forests. *Carbon N Y*, 2016. 107: 433-439.

[101] Chen G, Davis RC, Futaba DN, et al. A sweet spot for highly efficient growth

of vertically aligned single-walled carbon nanotube forests enabling their unique structures and properties. *Nanoscale*, 2016. 8: 162-171.

[102] Hu W, Yuan L, Chen Z, Gong D, Saito K. Fabrication and characterization of vertically aligned carbon nanotubes on silicon substrates using porous alumina nanotemplates. *J Nanosci Nanotechnol*, 2002. 2(2): 203-207.

[103] Teo KBK, Singh C, Chhowalla M, Milne WI. Catalytic synthesis of carbon nanotubes and nanofibers. *Encycl Nanosci Nanotechnol*, 2003. X: 1-22.

[104] Bartholomew CH. Catalysis reviews : Science and reforming and methanation carbon deposition in steam reforming and methanation. *Catal Rev Sci Eng*, 1982. 24(1): 67-112.

[105] Camacho J, Choudhuri AR. Effects of fuel compositions on the structure and yield of flame synthesized carbon nanotubes. *Fullerenes, Nanotubes and Carbon Nanostructures*, 2007. 15(2): 99-111.

[106] Hamzah N, Yasin MFM, Yusop MZM, Saat A, Subha NAM. Growth region characterization of carbon nanotubes synthesis in heterogeneous flame environment with wire-based macro-image analysis. *Diam Relat Mater*, 2019. 99: 107500.

[107] Nguyen NH, Henning R, Wen JZ. Molecular dynamics simulation of iron nanoparticle sintering during flame synthesis. *J Nanoparticle Res*, 2011. 13(2): 803-815.

[108] Vander Wal RL, Ticich TM. Comparative flame and furnace synthesis of single-walled carbon nanotubes. *Chem Phys Lett*, 2001. 336: 24-32.

[109] Chen T, Cai Z, Qiu L, et al. Synthesis of aligned carbon nanotube composite fibers with high performances by electrochemical deposition. *J Mater Chem A*, 2013. 1(6): 2211-2216.

[110] Unrau CJ, Axelbaum RL, Biswas P, Fraundorf P. Synthesis of single-walled carbon nanotubes in oxy-fuel inverse diffusion flames with online diagnostics. *Proc Combust Inst*, 2007. 31: 1865-1872.

[111] Unrau CJ, Axelbaum RL. Gas-phase synthesis of single-walled carbon nanotubes on catalysts producing high yield. *Carbon N Y*, 2010. 48: 1418-1424.

[112] Manciu FS, Camacho J, Choudhuri AR. Flame synthesis of multi-walled carbon nanotubes using CH_4-H_2 fuel blends. *Fullerenes, Nanotubes and Carbon Nanostructures*, 2008. 16(4): 231-246.

[113] Vander Wal RL, Ticich TM, Curtis VE. Flame synthesis of metal-catalyzed

single-wall carbon nanotubes. *J Phys Chem A*, 2000. 104(31): 7209-7217.

[114] Geurts FWAH, Sacco A. Relative rates of the Boudouard reaction and hydrogenation of CO over Fe and Co foils. *Carbon N Y*, 1992. 30(3): 415-418.

[115] Park C, Rodriguez NM, Baker RTK. Carbon deposition on iron–nickel during interaction with carbon monoxide–hydrogen mixtures. *J Catal*, 1997. 169(1): 212-227.

[116] Nolan PE, Lynch DC, Cutler AH. Carbon deposition and hydrocarbon formation on group VIII metal catalysts. *J Phys Chem B*, 1998. 102(21): 4165-4175.

[117] Jablonski GA, Geurts FWAH, Sacco Jr A. Carbon deposition over Fe, Ni, and Co foils from $CO-H_2-CH_4-CO_2-H_2O$, $CO-CO_2$, CH_4-H_2, and $CO-H_2-H_2O$ gas mixtures: II. Kinetics. *Carbon N Y*, 1992. 30(1).

[118] Kumar M, Ando Y. Chemical vapor deposition of carbon nanotubes: A review on growth mechanism and mass production. *J Nanosci Nanotechnol*, 2010. 10(6): 3739-3758.

[119] MacKenzie KJ, Dunens OM, Harris AT. An updated review of synthesis parameters and growth mechanisms for carbon nanotubes in fluidized beds. *Ind Eng Chem Res*, 2010. 49(11): 5323-5338.

[120] Liu C, Cheng H-M. Carbon nanotubes: Controlled growth and application. *Mater Today*, 2013. 16: 19-28.

[121] Liu QX, Ouyang Y, Zhang L, Xu Y, Fang Y. Effects of argon flow rate and reaction temperature on synthesizing single-walled carbon nanotubes from ethanol. *Phys E Low-Dimensional Syst Nanostructures*, 2009. 41(7): 1204-1209.

[122] Hata K, Futaba DN, Mizuno K, Namai T, Yumura M, Iijima S. Water-assisted highly efficient synthesis of impurity-free single-walled carbon nanotubes. *Science*, 2004. 306(5700): 1362-1364.

[123] Cho W, Schulz M, Shanov V. Growth and characterization of vertically aligned centimeter long CNT arrays. *Carbon N Y*, 2014. 72: 264-273.

[124] Zhu L, Xiu Y, Hess DW, Wong CP. Aligned carbon nanotube stacks by water-assisted selective etching. *Nano Lett*, 2005. 5(12): 2641-2645.

[125] Patole SP, Park JH, Lee TY, Lee JH, Patole AS, Yoo JB. Growth interruption studies on vertically aligned 2-3 wall carbon nanotubes by water assisted chemical vapor deposition. *Appl Phys Lett*, 2008. 93(11): 2008-2010.

[126] Ran M, Sun W, Liu Y, Chu W, Jiang C. Functionalization of multi-walled carbon nanotubes using water-assisted chemical vapor deposition. *J Solid State Chem*, 2013. 197: 517-522.

[127] Liu S, Zhang Y, Lin Y, Zhao Z, Li Q. Tailoring the structure and nitrogen content of nitrogen-doped carbon nanotubes by water-assisted growth. *Carbon N Y*, 2014. 69: 247-254.

[128] Futaba DN, Goto J, Yasuda S, Yamada T, Yumura M, Hata K. General rules governing the highly efficient growth of carbon nanotubes. *Adv Mater*, 2009. 21(47): 4811-4815.

[129] Hasegawa K, Noda S. Moderating carbon supply and suppressing Ostwald ripening of catalyst particles to produce 4.5-mm-tall single-walled carbon nanotube forests. *Carbon N Y*, 2011. 49(13): 4497-4504.

[130] Yamada T, Maigne A, Yudasaka M, et al. Revealing the secret of water-assisted carbon nanotube synthesis by microscopic observation of the interaction of water on the catalysts. *Nano Lett*, 2008. 8(12): 4288-4292.

[131] Little RB. Mechanistic Aspects of carbon nanotube nucleation and growth. *J Clust Sci*, 2003. 14(2): 135-185.

[132] Yoshihara N, Ago H, Tsuji M. Chemistry of water-assisted carbon nanotube growth over Fe-Mo/MgO catalyst. *J Phys Chem C*, 2007. 111(31): 11577-11582.

[133] Amama PB, Pint CL, McJilton L, et al. Role of water in super growth of single-walled carbon nanotube carpets. *Nano Lett*, 2009. 9(1): 44-49.

[134] Seung Min Kim, Cary LP, Placidus BA, et al. Evolution in catalyst morphology leads to carbon nanotube growth termination. *J Phys Chem Lett*, 2010. 1(6): 918-922.

[135] Noda S, Hasegawa K, Sugime H, et al. Millimeter-thick single-walled carbon nanotube forests: Hidden role of catalyst support. *Jpn J Appl Phys*, 2007. 46: L399-L401.

[136] Pint CL, Pheasant ST, Parra-vasquez ANG, et al. Investigation of optimal parameters for oxide-assisted growth of vertically aligned single-walled carbon nanotubes. *J Phys Chem C*, 2009. 113: 4125-4133.

[137] Amama PB, Pint CL, Mirri F, Pasquali M, Hauge RH, Maruyama B. Catalyst-support interactions and their influence in water-assisted carbon nanotube carpet growth. *Carbon N Y*, 2012. 50(7): 2396-2406.

[138] Hasegawa K, Noda S, Sugime H, Kakehi K, Maruyama S, Yamaguchi Y. Growth window and possible mechanism of millimeter-thick single-walled carbon nanotube forests. *J Nanosci Nanotechnol*, 2008. 8(11): 6123-6128.

[139] Wyss RM, Klare JE, Park HG, Noy A, Bakajin O, Lulevich V. Water-Assisted growth of uniform 100 mm diameter SWCNT arrays. *ACS Appl Mater Interfaces*, 2014. 6(23): 21019-21025.

[140] Hasegawa K, Noda S. Millimeter-tall single-walled carbon nanotubes rapidly grown with and without water. *ACS Nano*, 2011. 5(2): 975-984.

[141] Ren F, Kanaan SA, Majewska MM, et al. Increase in the yield of (and selective synthesis of large-diameter) single-walled carbon nanotubes through water-assisted ethanol pyrolysis. *J Catal*, 2014. 309: 419-427.

[142] Chen Z, Kim DY, Hasegawa K, Noda S. Methane-assisted chemical vapor deposition yielding millimeter-tall single-wall carbon nanotubes of smaller diameter. *ACS Nano*, 2013. 8(7): 6719-6728.

[143] Vander Wal RL. Fe-catalyzed single-walled carbon nanotube synthesis within a flame environment. *Combust Flame*, 2002. 130: 37-47.

[144] Kim MS, Rodriguez NM, Baker RTK. The Interplay between sulfur adsorption and carbon deposition on cobalt catalysts. *J Catal*, 1993. 143: 449-463.

[145] Tibbetts GG, Bernardo CA, Gorkiewicz DW, Alig RL. Role of sulfur in the production of carbon fibers in the vapor phase. *Carbon N Y*, 1994. 32(4): 569-576.

[146] Hu JL, Yang CC, Huang JH. Vertically-aligned carbon nanotubes prepared by water-assisted chemical vapor deposition. *Diam Relat Mater*, 2008. 17(12): 2084-2088.

[147] Stadermann M, Sherlock SP, In J-B, et al. Mechanism and kinetics of growth termination in controlled chemical vapor deposition growth of multiwall carbon nanotube arrays. *Nano Lett*, 2009. 9: 738-744.

[148] Futaba DN, Hata K, Yamada T, Mizuno K, Yumura M, Iijima S. Kinetics of water-assisted single-walled carbon nanotube synthesis revealed by a time-evolution analysis. *Phys Rev Lett*, 2005. 95(5): 056104.

[149] Merchan-Merchan W, Saveliev A V., Nguyen V. Opposed flow oxy-flame synthesis of carbon and oxide nanostructures on molybdenum probes. *Proc Combust Inst*, 2009. 32(2): 1879-1886.

[150] Hamzah N, Yasin MFM, Zainal MT, Rosli MAF. Identification of CNT growth

region and optimum time for catalyst oxidation : Experimental and modelling studies of flame synthesis. *Evergr Jt J Nov Carbon Resour Sci Green Asia Strateg*, 2019. 06(01): 85-91.

[151] Medjo RE. Characterization of carbon nanotubes. *Physical and Chemical Properties of Carbon Nanotubes*. InTech Open; 2013:171-193.

[152] Heitor M V., Moreira ALNN. Thermocouples and sample probes for combustion studies. *Prog Energy Combust Sci*, 1993. 19(3): 259-278.

[153] Friedman R. Measurement of the temperature profile in a laminar flame. In: *Laminar Combustion and Detonation Waves*. Vol 148. The Combustion Institute; 1953:259-263.

[154] Wagner W, Pruss A. The IAPWS Formulation 1995 for the Thermodynamic Properties of Ordinary Water Substance for General and Scientific Use. *J Phys Chem Ref Data*, 2002. 31(2): 387-535.

[155] Turns SR. *An Introduction to Combustion: Concepts and Applications*. 2nd ed. McGraw-Hill; 2000.

[156] Lehman JH, Terrones M, Mansfield E, Hurst KE, Meunier V. Evaluating the characteristics of multiwall carbon nanotubes. *Carbon N Y*, 2011. 49(8): 2581-2602.

[157] Suenaga K, Wakabayashi H, Koshino M, Sato Y, Urita K, Iijima S. Imaging active topological defects in carbon nanotubes. *Nat Nanotechnol*, 2007. 2(6): 358-360.

[158] Homma Y, Suzuki S, Kobayashi Y, Nagase M, Takagi D. Mechanism of bright selective imaging of single-walled carbon nanotubes on insulators by scanning electron microscopy. *Appl Phys Lett*, 2004. 84(10): 1750-1752.

[159] Nojeh A, Shan B, Cho K, Pease RFW. Ab initio modeling of the interaction of electron beams and single-walled carbon nanotubes. *Phys Rev Lett*, 2006. 96(5): 1-4.

[160] Dileo RA, Landi BJ, Raffaelle RP, Dileo RA. Purity assessment of multiwalled carbon nanotubes by Raman spectroscopy. *J Appl Phys*, 2007. 101(6): 1-5.

[161] Aitkaliyeva A, Shao L. The production of amorphous regions in carbon nanotubes by 140 keV He ion irradiation. *Carbon N Y*, 2012. 50(12): 4680-4684.

[162] Glassman I, Yaccarino P. The effect of oxygen concentration on sooting diffusion flames. *Combust Sci Technol*, 1980. 24(October 2013): 107-114.

[163] Baukal CE. *Oxygen-Enhanced Combustion*.; 1998.

[164] Love A, Middleman S, Hochberg AK. The dynamics of bubblers as vapor delivery systems. *J Cryst Growth*, 1992. 129: 119-133.

[165] Vicariotto M, Dunn-Rankin D. Temperature profiles and extinction limits of a coflow water-vapor laden methane/air diffusion flame. *Exp Fluids*, 2018. 59(9): 1-10.

[166] Padilla RE, Escofet-Martin D, Pham T, Pitz WJ, Dunn-Rankin D. Structure and behavior of water-laden CH4/air counterflow diffusion flames. *Combust Flame*, 2018. 196: 439-451.

[167] Wakeland RS, Keolian RM. Measurements of resistance of individual square-mesh screens to oscillating flow at low and intermediate Reynolds numbers. *J Fluids Eng Trans ASME*, 2003. 125(5): 851-862.

[168] Cao S, Ma B, Giassi D, Bennett BA V., Long MB, Smooke MD. Effects of pressure and fuel dilution on coflow laminar methane–air diffusion flames: A computational and experimental study. *Combust Theory Model*, 2018. 22(2): 316-337.

[169] Lin K-C, Faeth GM. Hydrodynamic suppression of soot emissions in laminar diffusion flames. *J Propuls Power*, 1996. 12(1): 10-17.

[170] Lock A, Briones AM, Aggarwal SK, Puri IK, Hegde U. Liftoff and extinction characteristics of fuel- and air-stream-diluted methane-air flames. *Combust Flame*, 2007. 149(4): 340-352.

[171] Gülder ÖL, Snelling DR, Sawchuk RA. Influence of hydrogen addition to fuel on temperature field and soot formation in diffusion flames. *Symp Combust*, 1996. 26(2): 2351-2358.

[172] Zainal MT, Yasin MFM, Wahid MA. Investigation of the coupled effects of temperature and partial pressure on catalytic growth of carbon nanotubes using a modified growth rate model. *Mater Res Express*, 2016. 3(10): 105040.

[173] Schünemann C, Schäffel F, Bachmatiuk A, et al. Catalyst poisoning by amorphous carbon during carbon nanotube growth: Fact or fiction? *ACS Nano*, 2011. 5(11): 8928-8934.

[174] Kharissova O V., Kharisov BI. Variations of interlayer spacing in carbon nanotubes. *RSC Adv*, 2014. 4(58): 30807-30815.

[175] Schäff F, Rümmeli MH, Kramberger C, et al. Tailoring the diameter, density and number of walls of carbon nanotubes through predefined catalyst particles.

Phys Status Solidi Appl Mater Sci, 2008. 205(6): 1382-1385.

[176] Rebelo SLH, Guedes A, Szefczyk ME, Pereira AM, Araújo JP, Freire C. Progress in the Raman spectra analysis of covalently functionalized multiwalled carbon nanotubes: Unraveling disorder in graphitic materials. *Phys Chem Chem Phys*, 2016. 18(18): 12784-12796.

[177] Bokobza L, Zhang J. Raman spectroscopic characterization of multiwall carbon nanotubes and of composites. *Express Polym Lett*, 2012. 6(7): 601-608.

[178] Han W, Ya Y, Chu H, Cao W, Yan Y, Chen L. Morphological evolution of soot emissions from a laminar co-flow methane diffusion flame with varying oxygen concentrations. *J Energy Inst*, 2020. 93(1): 224-234.

LIST OF PUBLICATIONS

The following articles were published based on the research reported on this thesis:

Journal with Impact Factor

1. N. Hamzah, M.F.M. Yasin, M.Z.M. Yusop, A. Saat, and N.A.M. Subha, "Rapid production of carbon nanotubes: a review on advancement in growth control and morphology manipulations of flame synthesis," *J. Mater. Chem. A*, vol. 5, no. 48, pp. 25144–25170, 2017. (**Q1, IF:9.93**)

2. N. Hamzah, M.F.M. Yasin, M.Z.M. Yusop, A. Saat, and N.A.M. Subha, "Growth region characterization of carbon nanotubes synthesis in heterogeneous flame environment with wire-based macro-image analysis," *Diam. Relat. Mater.*, vol. 99, pp. 107500, 2019. (**Q2, IF:2.29**)

3. N. Hamzah, M.F.M. Yasin, M.Z.M. Yusop, M.A.Z.M. Haniff, M.F. Hasan, K.F. Tamrin, and N.A.M. Subha, "Effect of fuel and oxygen concentration toward catalyst encapsulation in water-assisted flame synthesis of carbon nanotubes," *Combustion and Flame*, vol. 220, pp. 272-287, 2020. **(Q1, IF:4.57)**

Indexed Journal

1. N. Hamzah, M. F. M. Yasin, M. T. Zainal, and M. A. F. Rosli, "Identification of CNT growth region and optimum time for catalyst oxidation : Experimental and modelling studies of flame synthesis," *Evergr. Jt. J. Nov. Carbon Resour. Sci. Green Asia Strateg.*, vol. 06, no. 01, pp. 85–91, 2019. (**Indexed by Scopus**)

Paper presented in international conferences

1. N. Hamzah, M. F. M. Yasin, M. T. Zainal, and M. A. F. Rosli, "Identification of CNT growth region and optimum time for catalyst oxidation : Experimental and modelling studies of flame synthesis," The 10th International Meeting on Advances in Thermofluids, Bali, Indonesia (IMAT2018). **Best Paper Award**.