UNIVERSITI PUTRA MALAYSIA

EFFECTS OF TEMPERATURE, DEPOSITION TIME AND CATALYST LOADING ON THE SYNTHESIS OF CARBON NANOTUBES IN A VERTICAL CVD REACTOR

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BY

HENGAMEH HANAEI

Thesis Submitted to the School of Graduate Studies, University Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

June 2011

DEDICATION

This thesis is dedicated to my parents, ***Amir Hanaei and Homa Kazemi****.*

Thank you for all your love, patience and sacrifice and thanks for all your supports.

Abstract of thesis presented to the Senate University Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

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Carbon nanotubes (CNTs), were discovered by Ijima in 1991. They have special electrical, mechanical and electronic properties that make them attractive for a lot of applications. However, there is no clear relation between synthesis parameters and CNTs growth. Therefore more data investigations are required for Vertical CVD synthesis of CNTs. This research intend to investigate the effects of some synthesis parameters, namely, reaction temperature, catalyst loading, deposition time on Vertical CVD growth of CNTs. In present study, CNTs were produced through decomposition of acetone over a prepared catalyst Iron and Molybdenum supported on Alumina (Fe-Mo/AL_2O_3). The effects of those parameters on Vertical CVD synthesis of CNTs were studied using analysis of variance (ANOVA). After each run the product, including deposited carbon and catalyst, was characterized using

different analysis techniques. The morphology of as grown CNTs was observed by Scanning Electron Microscope (SEM). Thermo Gravimetric Analysis (TGA) was applied as this technique can reveal different forms of the carbon deposits (eg. amorphous and CNTs) according to their different thermal stabilities. Besides that, the effects of parameters on carbon deposit yield as well as their interactions were statistically studied using ANOVA. Finally, the appropriate values for parameters under study towards effective Vertical CVD synthesis of CNTs were determined full factorial design of experiments method. This research demonstrated that by increasing the reaction temperature to 950°C, more amorphous carbon was observed due to deactivation of catalyst at high temperature. It was also observed that by increasing the duration time, the yield of CNTs had decreased and by decreasing the catalyst loading the yield and quality of CNTs had increased. It was proved that there is a significant interaction between parameters under observation. In other words, synthesis parameters are not independent variable. As a result of that the quality and yield of the CNTs were appropriate achieved at 750°C reaction temperature, 40 min deposition time and 5 gr catalysts loading.

Abstrak tesis yang dikemukakan kapada Senat Universiti Putra Malaysia sebagi memenuhikeperluan untuk Ijazah Master of Sains

KESAN SUHU, MASA PEMENDAPAN DAN PEMUATAN PEMANGKIN KE ATAS SINTESIS NANOTIUB KARBON DI DALAM MENEGAK CVD REAKTOR

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Nanotiub karbon (CNTs), yang telah ditemui oleh Ijima pada 1991, mempunyai keistimewaan sifat-sifat elektrik, mekanikal dan elektronik menjadikan ia menarik untuk pelbagai kegunaan. Pemendapan wap kimia di dalam lapisan terbendalir (Vertical CVD) muncul sebagai kaedah yang menjanjikan sintesis nanotiub karbon dalam skala yang tinggi, kos yang rendah, dan hasil yang tinggi. Sebelum ini, tidak ada hubungan yang jelas antara parameter-parameter sintesis (suhu tindak balas, pemuatan pemangkin dan masa pemendapan) dengan pertumbuhan nanotiub karbon. Maka, banyak penyiasatan diperlukan terhadap sintesis nanotiub karbon. Penyelidikan ini menerangkan sintesis nanotiub karbon melalui penguraian aseton ke atas pemangkin yang disediakan, iaitu Fe-Mo/AL_2O_3 melalui reaktor lapisan terbendalir. Pada mulanya, sebelum aseton dimasukkan ke dalam reaktor, cecair aseton ditukar menjadi wap oleh aliran gas argon yang telah dipanaskan terlebih dahulu

menggunakan badan induk relau. Penyelidikan ini menumpukan kepada penyiasatan kesan utama dan kesan interaksi parameter-parameter sintesis ke atas pertumbuhan nanotiub karbon. Tambahan lagi, keadaan terbaik untuk sintesis nanotiub karbon telah ditentukan. Bersamaan itu, kesan utama dan kesan interaksi telah disiasat berdasarkan *one-way* dan *two-way analysis of variance (ANOVA), Scanning Electron Microscope (SEM) and Thermo Gravimetric Analysis (TGA)* dalam eksperimen rawak, sementara keadaan terbaik telah ditentukan berdasarkan kaedah rekabentuk eksperimen *full factorial, three-way ANOVA* dan *TGA.* Penyelidikan ini menunjukkan bahawa dengan meningkatkan suhu kepada 950°C, lebih banyak karbon amorfus telah diperhatikan kerana ketiadaaan pemangkin pada suhu tinggi. Sementara itu, telah diperhatikan bahawa dengan mengurangkan tempoh masa, hasil nanotiub karbon berkurang dan dengan mengurangkan pemuatan pemangkin, hasil dan kualiti nanotiub karbon telah meningkat. Penyiasatan ke atas kesan interaksi telah menunjukkan bahawa interaksi darjah kedua dan ketiga antara parameter-parameter di atas mempunyai kesan penting ke atas hasil karbon dan nanotiub karbon. Juga, hasil dari pelbagai keadaan sintesis yang telah disiasat untuk menentukan keadaan terbaik nanotiub karbon, telah didapati bahawa kualiti dan hadil nanotiub karbon adalah terbaik pada suhu tindakbalas pada 750°C, masa pemendapan selama 40 min dan pemuatan pemangkin sebanyak 5 gram.

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This thesis was submitted to the senate of University Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the supervisory committee are as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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HENGAMEH HANAEI
Date: 23 June 2011

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