UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERISATION OF CARBON NANOTUBES PREPARED USING PULSED LASER ABLATION DEPOSITION TECHNIQUE

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By

ISMAYADI BIN ISMAIL

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

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In appreciation of their love and sacrifices, this thesis is dedicated to Parents Ismail Awang and Mek Esah Awang, beloved wife Sakinah Shamsudin and my son Zafran Hakim. Not forgetting my brothers and sisters Kakak, Wani, Azli, Adik and to those who have supported me throughout my studies.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master Science

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Chairman: **Associate Professor Noorhana Yahya, PhD**

Faculty: **Institute of Advanced Technology**

Carbon nanotubes (CNTs) has been the focus of a virtual storm research, both to better understand its unique properties and to harness its potential in commercial applications such as hydrogen storage, atomic force microscopy probe, microelectronic transistor, electrical field emitter of flat panel display. There are two main premises in this research project; the first premise was to synthesis the CNTs via Pulsed Laser Ablation Deposition (PLAD) technique, and the second premise was to study the effect of Fe_2O_3 as catalyst on the magnetic properties of the deposited materials.

This work reports the formation of carbon web-like nano structure synthesized in a T-shape stainless steel chamber. ND:YAG laser with 532nm wavelength and 10.24 W power was used to ablate the target of graphite and catalyst. Fe_2O_3 and NiO were mixed separately as the catalyst with graphite (carbon) to form the target. The vacuum level was kept at 5 mtorr with argon gas flowing from bottom of the

chamber. The soot that was deposited on the glass substrate was then characterized using X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM), EDX and Vibrating Sample Magnetometer (VSM).

The SEM images confirm a web-like structure formed after the ablation. The graphite target that was ablated with laser does not form web-like structure. However, when NiO or Fe_2O_3 were introduced as the oxide catalysts, the web-like structure was formed successfully. The TEM pictures proved the web-like structure is the carbon nanotubes. Magnetic characterization via VSM was conducted after the CNTs structure was confirmed. From the magnetic characterization, we found that CNTs behaves as non-magnetic material due to the absence of the hysteresis curve. When it was filled with Fe_2O_3, the magnetic properties enhanced tremendously. It was also concluded that these Fe_2O_3 nano particles magnetic materials were trapped in the tubes. The CNTs acted as nano-wires and were able to induce the magnetization of the magnetic particles.

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

SINTESIS DAN PENCIRIAN TIUB NANO KARBON DIHASILKAN MENGGUNAKAN TEKNIK PEMENDAPAN ABLASI DENYUTAN LASER

Oleh

ISMAYADI BIN ISMAIL

Mac 2007

Pengerusi: **Profesor Madya Noorhana Yahya, PhD**

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Tiub nano karbon (CNT) telah menjadi fokus bagi banyak projek penyelidikan kini, di mana fokusnya adalah untuk memahami dengan mendalam cirri-ciri uniknya dan juga menggunakan potensinya dalam aplikasi komersial yang telah digiatkan dengan hebat seperti tempat simpanan hidrogen, prob mikroskop berskala atom, transistor mikroelektronik dan pemancar medan elektrik skrin panel rata. Ada dua objektif utama dalam projek penyelidikan ini, salah satunya adalah mengsintesiskan CNT melalui teknik pemendapan ablasi denyutan laser dan keduanya adalah mengkaji kesan Fe_2O_3 sebagai katalis terhadap sifat magnet bahan yang termendap.

Kerja projek ini melaporkan pembentukan struktur nano jaringan karbon yang disintesiskan dalam kebuk tahan karat berbentuk-T. Laser Nd:YAG dengan panjanng gelombang 532nm dan kuasa 10.24W telah digunakan untuk membakar sasaran pelet pemangkin dan grafit. Fe_2O_3 dan NiO digunakan sebagai pemangkin yang kemudiannya dicampurkan dengan grafit (karbon) bagi membentuk sasaran pelet.

Paras vakum dibiarkan pada 5mtorr dengan gas Argon mengalir dari bawah kebuk. Jelaga terhasil termendak di atas substrat kaca yang kemudiannya dicirikan dengan XRD (Serakan Sinar-X), SEM (Mikroskop Imbasan Elektron), TEM (Mikroskop Transmisi Elektron), EDX (Pembelauan Elektron Sinar-X) dan VSM (Sampel Tergetar Magnetometer).

Mikrograf SEM mengesahkan struktur jaringan terbentuk selepas ablasi. Sasaran pelet grafit dibakar dengan laser tidak membentuk struktur jaringan. Bagaimanapun, selepas dicampurkan dengan NiO atau Fe_2O_3 sebagai pemangkin oksida, struktur jaringan terhasil. Imej TEM telah membuktikan struktur jaringan tersebut sebagai tiub nano karbon. Pencirian magnet dengan VSM dilakukan selepas struktur tiub nano karbon disahkan. Dari pencirian magnet, didapati CNT tidak bersifat bahan magnet disebabkan tiada graf histeresis terhasil. Apabila ia diisi dengan Fe_2O_3, sifat magnetnya meningkat secara mendadak. Terdapat juga bahawa bahan magnet berpartikel nano Fe_2O_3 ini terperangkap di dalam tiub. Tiub nano karbon bertindak sebagai wayar-nano dan dapat menghasilkan pemagnetan kepada partikel magnet ini.

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The acknowledgement should also be given to the fruitful discussions from my interaction with our nanotechnology research group member, such as Shamsul Ezzad, Ramadhan Al-Habashi, Beh Hoe Guan, Samaila Bawa Waje, and Hashim Saad.

Special thanks to Ma and Abah, wife (Sakinah Shamsudin) and son (Zafran Hakim), brothers and sisters who have helped so much to endure my difficult moments during my studies.

Finally, thank you to the Ministry of Science, Technology and Innovation (MOSTI) of Malaysia under IRPA grant, vot 09-02-04-0855-EA001 for the financial support.

I certify that an Examination Committee has met on 23rd March 2007 to conduct the final examination of Ismayadi Bin Ismail on his Master of Science thesis entitled "Synthesis and Characterisations of Carbon Nanotubes (CNTs) Prepared Via Pulse Laser Ablation Deposition Technique" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

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

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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LIST OF ABBREVIATIONS

Nd:YAG	Neodymium Aluminium Garnet
PLAD	Pulsed Laser Ablation Deposition
CNTs	Carbon Nanotubes
SWNT	Single Walled Carbon Nanotube
MWNT	Multi Walled Carbon Nanotube
XRD	X-Ray Diffraction
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
EDX	Energy Dispersive X-ray
VSM	Vibrating Sample Magnetometer
At%	Atomic percent
$\Delta L/L_o$	Thermal expansion
E	Young's modulus
T_m	Melting point
LIPSS	Laser-Induced Periodic Surface Structures
CVD	Chemical Vapor Deposition
d_{hkl}	Lattice spacing
hkl	Miller indices
r.p.m	Rotation per minute
Ar	Argon
Hc	Coercive force
Bs	Saturation induction
G	Gauss
Oe	Oested
MSDS	Material Safety Data Sheet

CHAPTER 1

INTRODUCTION

1.0 Introduction of Carbon Nanotubes

Carbon nanotubes (CNTs) are tubular carbon molecules with properties that make them potentially useful in extremely small scale electronic and mechanical applications. They exhibit unusual strength and unique electrical properties, and extremely efficient conductors of heat.

A carbon nanotubes has a structure similar to a fullerene, but where a fullerene's carbon atoms form a sphere, a carbon nanotube is cylindrical and each end is capped with half a fullerene molecule. Their name derives from their size, carbon nanotubes are on the order of only a few nanometres wide (on the order of one ten thousandth the width of a human hair), and their length can be millions of times greater than their width.

Carbon nanotubes is composed entirely of sp^2 bonds, similar to graphite. Stronger than sp^3 bonds found in diamond, this bonding structure provides them with their unique strength. They can naturally align themselves into "ropes" held together by Van der Waals force. Under high pressure, carbon nanotubes can merge together, trading some sp^2 bonds for sp^3 bonds, giving great possibility for producing strong, unlimited-length wires. (Yildrim *et al*., 2000).