DEVELOPMENT OF PULSED LASER ABLATION DEPOSITION FOR SYNTHESIS OF CARBON NANOSTRUCTURE

LIEW SENG CHOY

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By

LIEW SENG CHOY

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Dedication:

Family members

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

DEVELOPMENT OF PULSED LASER ABLATION DEPOSITION FOR SYNTHESIS OF CARBON NANOSTRUCTURE

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August 2014

Chairman : Irmawati Bt. Ramli, PhD

Faculty : Institute of Advanced Technology

This thesis describes a development work for anisotropic growth of carbon nanotube. It is of interest to overcome the quenching problem found at substrate that affects the anisotropic behavior of carbon nanostructure. Temperature gradient exists between substrate and target hinders the initiation of vapor-liquid-solid (VLS) mechanism. Therefore, in this work, a suitable condition for plume formation is required to be developed first. Quality of vacuum pressure contained as well as regulating the vacuum pressure with Ar gas during pulsed laser ablation deposition (PLAD) process was address. Followed by accurate scanning of laser spot at target through the use of U-clamp designed and located at the target base. Movement of the laser spot was achieved through the wheel and axle in driving the primary focusing lens. Identification of all parameters required for vertically aligned growth of carbon nanostructure from Si wafer substrate was through literature review. A resistive heating plate designed capable heat up to 900 °C over an area of 3 cm by 1.5 cm to address the heat loss issue at substrate site during PLAD process. Al barrier layer of 30 nm thin was deposited using thermal evaporator onto n-type Si wafer as substrate with Miller indices (100). Ablation time of 20 minutes was found best for its minimal amount of amorphous carbon formed at heated substrate characterized using scanning electron microscope (SEM). Those information were used to synthesize vertically aligned carbon nanostructure by using Fe(III) catalyst synthesized using combustion method. Fast reaction of combustion method allows very pure catalyst synthesized with diameter less than 30 nm initially deposited at substrate characterized using transmission electron microscope (TEM). Optimum pressure that prevented the formation of fringes at substrate during the first step of catalyst ablation was at 0.6 torr. Follow by graphite target ablation required 2.5 torr of background pressure for substrate heated at 300 °C and above without fringe formation. Substrate temperature at 500 °C managed to burn off majority of amorphous carbon formed. Bundles of vertically aligned carbon nanotube were observed during TEM characterization. The overall tube diameter was estimated using ImageJ to be less than 20 nm. Encapsulation of catalyst in the carbon nanotube

produced confirmed using energy dispersive X-ray (EDX). The system developed managed to synthesis bundles of vertically aligned carbon nanostructure.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN PERCIKAN DENYUT LASER BAGI SINTESIS KARBON NANOSTRUKTUR

Oleh

LIEW SENG CHOY

Ogos 2014

Pengerusi : Irmawati Bt. Ramli, PhD

Fakulti : Institut Teknologi Maju

Tesis ini menerangkan kerja pembangunan bagi penumbuhan anisotropik tiub nano karbon. Keutamaan projek adalah untuk mengatasi masalah lindapkejut yang wujud pada substrat dan memberi kesan kepada anisotropik struktur nano karbon. Kecerunan suhu di antara substrat dan sasaran laser boleh menghalang mekanisme wap-cecair-pepejal (VLS) daripada berlaku. Oleh itu, kajian dimulakan dengan perolehan keadaan yang sesuai bagi pembentukan kepulan. Kualiti tekanan vakum dalam kebuk dan gas latar belakang, Ar yang digunakan untuk mengawal tekanan vakum semasa proses percikan denyut laser (PLAD) diatasi. Ketepatan titik laser mengimbasi sasaran laser diatasi melalui pembentukan pengapit-U yang diletakkan di dasar sasaran. Pergerakan titik laser dicapai melalui roda dan gandar untuk memacu kanta tumpuan prima. Pengenalpastian semua parameter diperlukan untuk pertumbuhan sejajar menegak struktur nano karbon dari substrat wafer Si yang diperolehi dari kajian literatur. Alat pemanas rintangan mampu memanas sehingga 900°C pada keluasan 3 cm x 1.5 cm direka bagi menangani isu kehilangan haba di permukaan substrat semasa proses PLAD. Lapisan penyekat Al senipis 30 nm dideposit menggunakan penyejat haba pada Si wafer jenis-N dengan indeks Miller (100). Masa percikan 20 minit terbaik bagi meminimumkan pembentukan karbon amorfus pada substrat panas dicirikan melalui imbasan mikroskop elektron (SEM). Maklumat-maklumat ini digunakan untuk mensintesis struktur nano karbon menegak sejajar dengan penggunaan mangkin Fe(III) yang disintesis menggunakan kaedah pembakaran. Tindak balas cepat pembakaran telah menghasilkan mangkin tulen dengan diameter kurang daripada 30 nm dipercitkan terdahulu pada substrat dan dicirikan menggunakan mikroskop elektron transmisi (TEM). Tekanan optimum yang digunakan bagi mengelak pembentukan pinggiran di substrat semasa percikan pemangkin adalah pada 0.6 torr. Ini diikuti percikan sasaran grafit memerlu 2.5 torr tekanan latar belakang bagi substrat yang dipanaskan pada 300°C dan ke atas tanpa pembentukan pinggiran. Suhu substrat pada 500°C mampu membakar majoriti karbon amorfus yang terbentuk. Gumpalan tiub nano karbon menegak sejajar didapati semasa pencirian TEM. Diameter tiub keseluruhan dianggar dengan ImageJ adalah kurang daripada 20 nm. Pengkapsulan pemangkin dalam tiub nano karbon

terhasil telah disahkan menggunakan penyerakan tenaga sinar-X (EDX). Sistem yang dibangunkan dalam kajian ini berjaya menghasilkan gumpalan struktur nanokarbon menegak sejajar.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

at %	atomic percentage
CNT	carbon nanotube
CATIA	Computer Aided Three-dimensional Interactive Application
d.c.	direct current
D/G	diamond to graphite ratio
EDX	energy dispersive X-ray
eV	electronvolt
FTIR	Fourier Transform Infrared
MWCNT	multi-walled carbon nanotube
Pa	pascal
PLAD	pulse laser ablation deposition
rpm	rotation per minute
sccm	standard cubic centimeter per minute
SEM	Scanning Electron Microscope
SWCNT	single-walled carbon nanotube
TEM	Transmission Electron Microscope
T_v	vaporization temperature
VLS	vapor-liquid-solid
wt %	weight percentage
XRD	X-ray Diffractometer
Q_{leak}	Leakage rate
I_{peak}	Laser peak intensity

CHAPTER 1

INTRODUCTION

1.1 Overview

Nanotechnologies specifically from carbon nanotube, CNT has captured tremendous attention since the discoveries of producing macroscopic quantities of fullerenes. Study of the growth process required understanding of the types of building block available. Graphite whisker was indeed found back in the 60's. Technological constrain back in the 60's postulated that catalyst required to form this carbon whisker. Bacon (1960) was among the earliest most arc discharge method reported graphite crystal sheet coiled up whisker scroll structure with high flexibility and strength. The definition of nano-object is not defined until late 2006 in Geneva, Switzerland. This happened slightly after more than a decade long since from the first observation of carbon nanotube by Sumio Iijima under high-resolution transmission electron microscopy by Iijima 1991. He was then worked in the department of electron microscopy in Hitachi. His discovery redefines the perception of carbon element physical properties. Since then, vanguard researcher from around the world developed various techniques for obtaining all possible applications from strand of carbon nanotube.

The definition of nano-object is now well acknowledged and accepted across this field of research (Figure 1.1). According to it, nano-object with at least one dimension in the nanoscale (1 nm to 100 nm) is categorized into three groups.

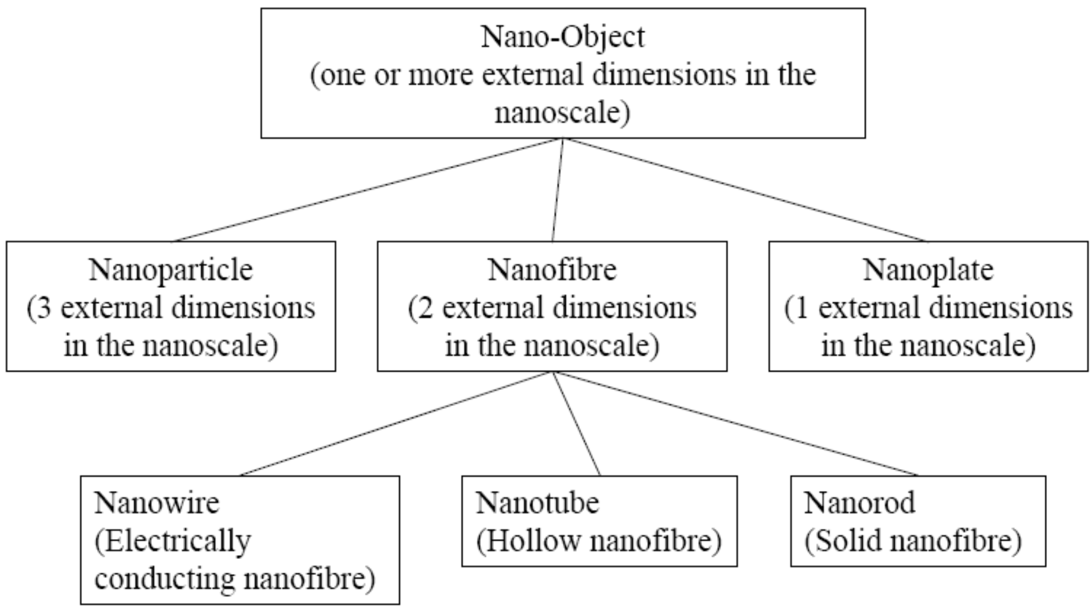

Figure 1.1: Hierarchy of terminologies set as standard for nano-object (Adapted from ISO/TS 27687 : 2008 (E) standard)

Based on standard definition, nanofibre will have a dimension significantly extends towards a finite length. Normally this third dimension is not limited to nanoscale and normally three times greater than another two dimensions. However, nanoplate has only one dimension in the nanoscale and it will be the thickness of the nano-object. However, nanoparticle is defined for object with all three lengths of dimension in the nanoscale. In three-dimensional axes, all three dimensions for nanoparticle are in nano-scale region suggesting mostly by far an oval shape or else defined as shown in Figure 1.2.

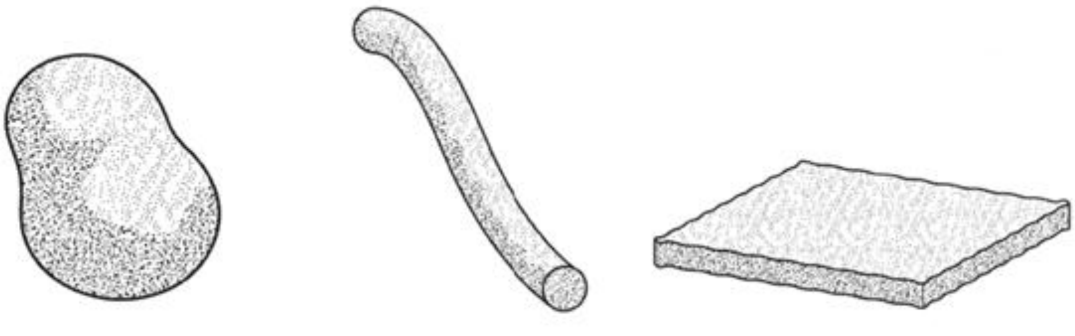

Figure 1.2: Illustration of possible nano-objects as defined. a) Nanoparticle: b) Nanofibre c) Nanoplate (Image adapted from ISO/TS 27687: 2008(E) standard.)

A sphere made of 60 atoms carbon structure at vertices. Comprise of 32 faces with 12 pentagons and 20 hexagons formed by vaporizing graphite using pulsed laser irradiation (Kroto *et al*. 1985). It was claimed as a new form of carbon (Kratschmer *et al*. 1990). Later described as a needle-like tube of finite carbon structure produced using an arc-discharge evaporation method (Iijima 1991). Formation of the tube was found feasible from sphere structure at sufficiently higher temperature of anneal with high-density carbon vapor feedstock (Guo *et al*. 1995). Carbon structure formed was described as a rolling up of the graphene sheet (White *et al*. 1993). Each graphene sheet is made up of carbon atoms bonded with sp^2 bond formed into Bravais lattice for the honeycomb (Mintmire and White 1995). Handedness or chirality of the tube therefore occurred.

Direction of rolling of graphene sheet for a lattice point superimposed onto the origin is identified by an index *A(n, m)* (Hamada *et al*. 1992). Hamada Index is the superimposed coordinate base on hexagonal Bravais lattice system. Second digit of the index indicates the number of repeating hexagonal unit along second axis. Index with equal number *A(n, n)* denoted a vector index with armchair silhouette repeating along circumference of tube. Zigzag silhouette along the brim of the tube formed with second digit of index *A(n, 0)* to be zero. Tubes formed are categorized to be chiral for index with both digits to be an integral also behaves as semiconductors (Iijima and Ichihashi 1993). Diameter of the tube hence could be estimated by using the vector index of the tube. This index format generally used to indicate types of electronics band structure formed. It was also found that curvature effect becomes dominant by alteration of the fiber diameter (Saito *et al*. 1992). It however changes the electronics energy gap between semiconducting and metallic for zigzag fiber. Band gap energy decreases in inverse proportion with diameter and approaches zero for planar graphene (Yakobson and Smalley 1997). It was estimated that the lowest possible limit of the tube diameter to be around 4 Å (Sawada and Hamada 1992).

Application of carbon nanotube, CNT has also been very well defined over the past two decades of extensive research. Realizing the vision of having commercial products make from CNT is still afar. This is caused by still technological gap to be bridged. Research data parameters from various methods are gathered to enable a system to be designed into such a manner to study the growth dynamics for better understanding of the causes. Bamboo liked horizontal growth carbon nanotube, CNT observed previously indicate that there is a tendency of capping at the tip (Beh 2006; Ismail 2006). The random capping of the tube occurred suggested that heat treatment at substrate play a crucial role during the growth mechanism.

1.2 Future for Protruding Structure

Carbon nanotubes growth condition has been successfully found over the past two decades using various methods purposed for numerous applications. The tiny strand of structure made from carbon found to have several advantages over conventional carbon structure, graphene or diamond. Its superior mechanical properties such as tremendously high break strain of up to 50 times higher than steel considering of its lightweight structure (Goze *et al*. 1999; Salvetat *et al*. 1999). This finding makes this new piece of technology very promising to promote it as an ideal candidate for aerospace. One of the currently technological constrained vision for space elevator using carbon nanotube as finite cable was proposed (Clarke 2001). Interlinking of carbon nanotubes under pressure induced were found best to enhance mechanical properties (Yildirim *et al*. 2000). Desirable strength-to-weight ratio of vertically aligned carbon nanotube further enhanced by pulling into nano-yarn served as artificial muscle (Lima *et al*. 2012). Torsional actuation of the nano-yarn achieved by coating the nanotube with paraffin wax. Contraction and expansion of the nano-yarn occurred once the wax is subjected to heat source from white-light of 100W.

Study on the optical absorption for the total darkness found has been proved to be very useful coating for terahertz laser power calibration tool by National Institute of Standard and Technology, NIST (Yang *et al*. 2008). The arrays of 1.5 mm thick coating of vertically aligned carbon nanotube were found to be the blackest man made material. Coated on Silicon surface could make it feasible to transfer to the laser power detector. It is thick enough to absorb wide range wavelength of light perfectly from infrared to microwave range (Mizuno *et al*. 2009). Thermal properties possess enable dissipating the heat quick enough as compared to other black coating. It allows laser power meter operate in a more responsive and rapid signal detection. Array of these vertically aligned multi-walled carbon nanotube were found to have even lower reflectance, VAMWCNT (Lehman *et al*. 2010). Coating it on pyroelectric material of lithium tantalite will produce current once light is absorbed efficiently as heat and signal transferred used at laser power meter. It is practical use to make precision measurement of laser power for advanced technologies such as solar energy conversion, satellite-borne sensors and optical communications (Ost 2010).

Electronic properties of metallic or semiconducting depends much on the tube chirality. Metallic type conducting carbon nanotube served as nano-size gas detector. Ueda *et al*. (2009) found a way to detect nitrogen monoxide, NO gas at ambient temperature by using conducting single-walled carbon nanotube. NO gas molecule detached from the nanotube surfaces changes the resistances at carbon nanotube are detected once activated by using UV light irradiation. This method avoids the use of heating while increases six times of its sensitivity to 50 ppb in detecting the presents of NO gas. It helps to provide cheap and low power consumption sensor. Extremely large surface area-to-volume ratio of porous carbon nanotube has proven to be best technology for miniaturizing microchip. Different alignment of carbon nanotube affected the sensitivity of the sensor probe. It altered the overall density of the nanotube array as sensor in relation to gas permeability. Recovery time of sensor is however governed by Arrhenius absorption energy that is best function at high temperature (Hoa *et al*. 2009).

High aspect ratio of carbon nanotube is another unique physical properties besides high electrical-thermal conductivities and mechanical strength being exploited. Vertically array of conducting carbon nanotube are suited to application for cathode ray device that are consuming huge amount of energy during operation. Tailoring the density of array growth it is possible to reduce the turn-on threshold for field emission found for aligned array of carbon nanotube. Low densities of catalyst dots (0.5 μm) were found best to avoid screening effect from the adjacent conducting array of carbon nanotube for higher resolution display (Neupane *et al*. 2012). Application for the similar property as field emission was discovers beginning of the 20^{th} century. Durability and high-stability of single strand carbon nanotube prepared using electron microscope is promising yet repeatable. It served as source tip inside high-resolution electron microscope in getting better resolution image than 10 nm barrier (Kim *et al*. 2003).

1.3 Pulsed Laser as Energy Source

A Light amplification by stimulation emission radiation (LASER) device was employed during the experiment for generating a thin film of catalyst and carbon at the substrate. This device emits coherent light source through amplification of light. A process involved a thermally non-equilibrium state of atom that favors stimulated emission radiation process than spontaneous emission. This naturally uncommon state of atom was stimulated by first introducing a state of atom in already excited state. A flash lamp will be used to pump up the crystal rod made of Neodymium doped Yttrium Aluminum garnet ($Nd{:}Y_3Al_5O_{12}$) or in short Nd:YAG lasing compound. Rare earth Nd with triply ionized oxidation number enable the amplification of light that leads to stimulated emission. Doping amount of Nd of around 1% by weight into the host crystal varies according to its application. Heavily doped of laser rod meant for pulsed laser is optically distinguishable for its pink-purplish color of Nd. Laser rod for continuous wave output is almost white (Golap and Maheshwar 2010).

Pumping up of laser rod by Kr flash lamp was used to achieve the non-equilibrium state very efficiently. It enables the amount of excited state atoms out-number the amount of lower energy state atoms. Absorption of photon by electrovalence from the flash lamp will excite an atom from ground state into excited state. However its life cycle is limited to 10^{-8} s during excited state. It eventually emits photon once de-excited back into ground state. This process is spontaneously occurs in nature to maintain a thermal equilibrium state of matter. An optical resonator is used to prevent this spontaneous emission. A pair of mirror is place at either end of the laser rod. One end of the rod is cover with higher reflection coefficient than the other end. These mirrors bounce off photon within the cavity at the same time it induced those already excited state of atom to decay from meta-stable state through resonant effect. This process continuous and causes the light amplification. Two photons that have the same frequency and phase will be produced.

Number of excited state of atoms will be greater than those in the ground state over a period of time. One of the mirrors with least reflection often as little as 1% will allow partial transmit of those photon generated through stimulated emission to escape through the cavity. A laser pulse is generated by Q-switch mode 20 MW act as shutter for obtaining pulse width 10 ns at the opening of cavity. This high-energy beam source of monochromatic light is coherent caused of the same photon source being populated from the same transition state. It is therefore has same energy and color traveling parallel to the laser rod in a narrow exist.

One drawback to the Nd:YAG pulsed laser is the huge amount of heat generated during the population inversion process. Decay of electron into meta-stable level between ground state and excited state propagate as heat than photon radiation. A versatile cooling system is required to withdraw heat from the system to prevent a meltdown of the device. Schematic of the laser cavity was adapted from the device manufacture website and is attached in Appendix B.

Table 1.1: Summary for system specification

Parameter	Dimension
Pulsed Laser	Nd:YAG 532 nm, 10.24 Watt, 5 kHz
Target Holder rotations (13 mm diameter Al stud)	$500 – 4{,}000 \pm 10\%$ rpm
Backing and Ablation Pressure	0.3 mPa – 333 ± 13 Pa ($\sim 2.0\times10^{-6} - 2.5 \pm 0.1\ Torr$)
Target-substrate distance	$10 – 50 \pm 4$ mm
Electric Plate Heater	$1000 \pm 10\%$ °C

1.4 Problem Statement

Cross-chamber developed previously is not suitable for anisotropic growth of carbon nanostrcture. The previous setup experienced huge temperature gradient between laser hot zone and substrate (Yahya *et al* 2007). Quenching effect causes formation layer of web-liked carbon nanotube that will hinders the vapor-liquid-solid, VLS mechanism required for vertical alignment purpose. Formation of the aligned carbon nanostructure required the initially carbon in vapor phase result from laser ablation. Catalyst in nanosize as a platform to initiate the growth of carbon nanostructure activated for the metallofullerene formation while in liquid phase. Further incoming of carbon atom in vapor phase combined with the liquid phase of metallofullerene forms a nanosize tube in solid phase once cooled. Develop a pulsed laser ablation, PLAD system capable to achieve the growth mechanism is intended in this research. It enables to get nanotube application in becoming reality. Major obstacle would be to stabilize the plume generated using PLAD that has to be also an environment suitable for synthesis of vertically aligned of carbon nano-structure on n-type silicon wafer of (100) plane. Developing this tool enable the study of the established theory for carbon to form protruding structure following VLS mechanism. Re-design of the available cross-chamber are required to comply with the VLS mechanism for vertical growth of carbon structure. Once the system is commissioned, the growth mechanism will be investigated by altering parameters involved. The purpose is determine the suitable parameter for anisotropic growth behavior of carbon nano-structure by the well-acknowledged VLS growth mechanism under controlled environment during PLAD process on a standard silicon wafer substrate.

1.5 Research Objectives

The main objective of this study is to achieve anisotropic growth of carbon nanostructure that will be divided into the following:

Objective 1: To investigate, and develop a suitable condition for plume formation inside the cross-chamber.

Objective 2: To identify and optimize the overall parameters of PLAD chamber required for carbon nanostructure growth.

Objective 3: To synthesize and develop a methodology to characterize the anisotropic growth behavior of carbon nano-structure.

1.6 Chapter Summary

There are a total of six major chapters in this dissertation. Research background reasons and its terminology in accordance to ISO/TS 27687:2008 (E) standardization were also highlighted are explained in Chapter 1. Techniques in achieving the protruding nature for carbon structures from various methods were referred and reported in Chapter 2. A good understanding of various available methods can aid the understanding of this research about aligned nano-object as literatures review. The fundamental theories specifically in chamber upgrading required for the formation of individually vertical-align carbon structure relative to its substrate is also discussed. The term protruding will be used throughout this dissertation. The essence of the dissertation is presented in Chapter 3. This chapter emphasized on methodologies to realize the individual protruding carbon structure. The methodology involved the customization of new device introduced into the cross-chamber consistent with literature for preparation of protruding carbon nanotube. The ultimate goal for this system capable in producing individual protruding carbon structure will be discussed and showed in Chapter 4. Finally, Chapter 5 summarizes the research outcome as well as some suggestions cater for future work on this available system. All references cited, calculation involved and designing draft of substrate heater is arranged in Appendices to aid the reading of this dissertation, followed by the author biography and list of publications.

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LIST OF PUBLICATIONS

1. SengChoy, L., Irmawati, R., Noorhana, Y., A. Halim, S., (2013). "Thermal Decomposition of Iron Citrate into Nano-sized Iron(III) Oxide Deposited on Si Wafer by using Pulsed Laser Ablation" Journal of Applied Science Research 9(9): 5497-5501. ISSN 1819-544x

2. SengChoy, L., Irmawati, R., Noorhana, Y., A. Halim, S., (2014). "Encapsulation of Iron(III) Oxide in Carbon Nanotube bundles" Advances in Natural and Applied Science 8(2): 69-74. ISSN:1995 - 0772