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UNIVERSITI PUTRA MALAYSIA

ELECTRICAL CHARACTERIZATION OF CARBON NANOTUBE AS GAS SENSING ELEMENT

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By

FARAH ANIZA MOHD YUSOF

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

June 2007

This work is dedicated to

My beloved husband, Ahmad Syakir Abdul Rashid

My father and mother,

Mohd Yusof Hj. Ahmad and Siti Rohani Hj. Khulan

And my brother and sister,

Farid Azwan Mohd Yusof and Farah Emalina Mohd Yusof

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

ELECTRICAL CHARACTERIZATION OF CARBON NANOTUBE AS GAS SENSING ELEMENT

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FARAH ANIZA MOHD YUSOF

June 2007

Chairman: Roslina Mohd Sidek, PhD.

Faculty: Engineering

Gas sensing is very important in order to detect dangerous gases like carbon dioxide, ammonia and acetylene, which are commonly used in industries as well as in medical applications. Carbon Nanotube is a promising candidate for gas sensing element because of their large surface area. Therefore, they offer excellent sensitivity and rapid response towards surface changes.

This work aims to investigate carbon nanotubes as gas sensing element. The growth of carbon nanotube has been done using Chemical Vapor Deposition (CVD) technique. The physical and electrical characteristics of carbon nanotube have been characterized using microscopes and source measurement unit. Sensors were fabricated and the variations of electrical resistance upon the exposure of carbon dioxide, ammonia and acetylene gas have been investigated.

The technique for growing carbon nanotubes that is called Floating Catalyst CVD has been used to produce grams of carbon nanotube. The temperature was set from

800^{o}C to 900^{o}C. For that range of temperature, grams of carbon nanotubes are produced in which the diameter is from 40 nm – 200nm and the length is in micrometer. The carbon nanotubes produced are found to have multi-layered wall in about 8nm thickness. The diameter, length and wall thickness have been measured using the Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM) and Transmission Electron Microscope (TEM). The multi-layered wall indicates that the carbon nanotubes are Multi-Walled Carbon Nanotubes (MWNTs).

Gas-sensing samples have been prepared in the forms of pellet and films. Upon exposure of carbon dioxide, ammonia and acetylene gas, the resistance of the samples increases from their steady state value. From the research, it was found that the sensor is sensitive to carbon dioxide, ammonia and acetylene gas. The sensor can be operated at room temperature with response time as fast as 0.1 to 1 second.

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

GAMBARAN SIFAT ELEKTRIK KARBON NANOTIUB SEBAGAI ELEMEN PENDERIA GAS

Oleh

FARAH ANIZA MOHD YUSOF

Jun 2007

Pengerusi: **Roslina Mohd Sidek, PhD.**

Fakulti: **Kejuruteraan**

Penderia gas penting untuk mengesan gas-gas yang bahaya seperti karbon dioksida, amonia dan asetilena yang selalu digunakan dalam industri dan juga perubatan. Karbon Nanotiub adalah bahan yang berpotensi sebagai elemen penderia gas kerana mempunyai luas permukaan yang besar.

Kajian ini berhasrat untuk mengkaji karbon nanotiub sebagai elemen penderia gas. Penghasilan karbon nanotiub dilakukan menggunakan teknik Pemendapan Wap Kimia (CVD). Ciri-ciri fizikal dan elektrikal karbon nanotiub dikaji menggunakan beberapa jenis mikroskop dan unit ukuran. Sampel disediakan dan perubahan rintangan elektrik terhadap pendedahan gas karbon dioksida, amonia dan astilena telah dikaji.

Teknik untuk menghasilkan karbon nanotiub iaitu Pemendapan Wap Kimia bermangkin terapung (FCCVD) digunakan untuk penghasilan karbon nanotiub dalam kuantiti gram yang banyak. Suhu yang digunakan adalah dari $800^{o}C$ – $900^{o}C$.

Untuk suhu sebegini, karbon nanotiub dihasilkan dalam beberapa gram yang mempunyai diameter dari 40 nm – 200nm dan panjang dalam ukuran mikron. Karbon nanotiub yang terhasil mempunyai dinding berlapis setebal 8nm. Diameter, panjang dan ketebalan dinding diukur menggunakan Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM) dan Transmission Electron Microscope (TEM). Dinding karbon nanotiub yang berlapis menunjukkan ia adalah jenis Karbon Nanotiub Dinding Berlapis (MWNTs).

Sampel penderia gas telah disediakan dalam bentuk pelet dan filem. Semasa pendedahan kepada gas karbon dioksida, amonia dan asetilena, rintangan sampel adalah lebih tinggi dari nilai rintangan asal tanpa gas. Berdasarkan kepada kajian ini mendapati bahawa penderia adalah sensitif terhadap gas karbon dioksida, amonia dan asetilena. Penderia ini boleh beroperasi pada suhu bilik dengan masa respon 0.1 ke 1 saat.

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I certify that an Examination Committee has met on 5th June 2007 to conduct the final examination of Farah Aniza Mohd Yusof on her Master of Science thesis entitled "Electrical Characterization of Carbon Nanotube As Gas Sensing Element" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the degree of Master of Science.

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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 were 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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FARAH ANIZA MOHD YUSOF

Date: 13 September 2007

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LIST OF ABBREVIATIONS

A	Ampere
AFM	Atomic Force Microscope
˚C	Degree Celcius
C_2H_2	Acetylene
C_6H_6	Benzene
cm	centimeter
CNT	Carbon Nanotube
CO_2	Carbon Dioxide
CVD	Chemical Vapor Deposition
FPD	Flat Panel Display
FCCVD	Floating Catalyst Chemical Vapor Deposition
Fe	Ferum
f_o	Resonant Frequency
f_z	Zero-Reactance Frequency
G	Conductance
HRTEM	High Resolution Transmission Electron Microscope
Hz	Hertz
IV	Current Voltage
K	Kelvin
MWNT	Multi Walled Carbon Nanotube
NH_3	Ammonia
nm	Nanometer
Pd	Palladium

ppb	parts per billion
ppm	parts per million
R	Resistance
R_t	Reaction Time
R_T	Reaction Temperature
SEM	Scanning Electron Microscope
SiO_2	Silicon Dioxide
SMU	Source Measurement Unit
SPM	Scanning Probe Microscope
SWNT	Single Walled Carbon Nanotube
TEM	Transmission Electron Microscope
Tpa	Tera Pascal
V	Volt
µm	Micrometer
ε’	Permittivity
ε’’	Loss Factor
Ω	Ohm
1-D	1 Dimensional
2-D	2 Dimensional

CHAPTER 1

INTRODUCTION

This chapter introduces the carbon nanotubes and its potential to act as a gas sensing element. Besides, it will also state the objectives of the study and scope within the research.

1.1 Nanotechnology

Nanotechnology relates to the creation of devices, structures and systems whose size ranges from 1 to 100 nm and exhibits novel physical, chemical and biological properties because of their nanoscale size. *Richard Feynman [Feynmann, 1959]* had emphasized in his lecture that nanotechnology is an interdisciplinary science, engineering and biology related dimensions on the order of few nanometers. The lecture is actually meant to stimulate new discoveries and capabilities at atomic and molecular scale. The exploration of nanotechnology is realized in 1980s as the scanning tunneling microscope emerged *[Meyyapan and Srivasta, 2000]*.

Since the discovery in 1990 by Iijima, the very promising Carbon Nanotubes (CNT) have become a very potential nanotechnology materials in various applications such as interconnections, active switching element in electronic devices and gas sensing element in gas sensors due to their size in nanometer. The potential is stem from the features of carbon nanotubes structures, which are electronic, mechanical, optical and chemical characteristics *[McEuen et al., 2001 and 2002, Avouris et al., 2002, and Meyyappan and Srivasta, 2000]*.

1.2 Gas Sensing Element

As will be discussed in Chapter 2, carbon nanotubes are essentially all surface, thus they offer excellent sensitivity and rapid response towards surface changes. Furthermore, carbon nanotubes also possess a tendency to change electrical properties at room temperature in the presence of gases. Therefore, carbon nanotubes can be a good gas sensing element *[Ong et al., 2002 and Varghese et al., 2001]*. Electrical properties that are commonly used in detecting gases are resistance and impedance.

Gas sensing element to detect gases like Carbon Dioxide, Ammonia and Acetylene is needed to monitor the air quality. Ammonia sensor is important for monitoring ambient ammonia concentration since it is related to many environmental issues such as acidification, human health and climate change through particle formation. Carbon Dioxide sensors are widely used in food and medicine packages as a means of detecting spoilage *[Ong, 2002]*. Acetylene gas is widely used in chemical synthesis and also gas welding due to the high temperature of the flame produced from the combustion of Acetylene with oxygen. Therefore, the sensor is needed to detect Acetylene gas because Acetylene gas is odorless, colorless and can explode with extreme violence if the pressure of the gas exceeds 100kPa. Furthermore, inhaling Acetylene gas may cause dizziness, headache and nausea *[http://en.wikipedia.org/wiki/Acetylene]*.

1.3 Problem Statement

Research on synthesis of carbon nanotubes has started in University Putra Malaysia (UPM) since 2003 by a research group from Chemical Engineering Department, Faculty of Engineering. However, all works were emphasized on investigating the mechanical and chemical properties of carbon nanotubes. There was no work dated to electrical characterization. In this research, electrical properties of carbon nanotubes will be investigated.

The electrical properties will be investigated towards the development of gas sensing element. As known, nowadays, the gas sensing element is needed in industry for environmental analysis, medical diagnostics and other various field applications. Other researchers have proposed gas sensing elements as reviewed in Section 2.5. Common gas sensors are in the form of thick films, porous pellets or thin films. Problems encountered with these sensors, which are lack of flexibility, poor response times and operating at elevated temperature. Therefore a new gas sensing element which is small in size, high sensitivity and can operate at room temperature is needed to solve the problems. In this research, the gas sensing element can give a quick response upon exposure to the gases and operate at the room temperature.