UNIVERSITI PUTRA MALAYSIA

SELECTIVE AREA FABRICATION OF CARBON NANOSTRUCTURES USING ALCOHOL CATALYTIC CHEMICAL VAPOR DEPOSITIONAND SPIN ON GLASS

AISHAH BINTI FAUTHAN

ITMA 2015 7

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By

AISHAH BINTI FAUTHAN

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

May 2015

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

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By

AISHAH BINTI FAUTHAN

May 2015

Chair: Associate Professor Mohd Nizar Hamidon, PhD
Faculty: Institute of Advanced Technology

The thesis describes an investigation on selective area fabrication of carbon nanostructures as a technique for sensor application. Nowadays, the researchers are using spraying technique for selective area of carbon nanostructure. However due to adhesion issues, different fabrication processes are investigated to overcome the problem. The alcohol catalyst chemical vapor deposition (ACCVD) has been chosen as the technique to synthesis the carbon nanostructure. Different parameters of carbon nanostructures process synthesis using ACCVD are tested. Catalyst preparation method and temperature are varied to produce the best parameter for carbon nanostructures growth. It was evident that using impregnation method of catalyst and mixed of ethanol gives a good result in carbon nanostructures growth. The temperature effect is investigated by varying the temperature value using 750°C, 800°C and 850°C. The growth of carbon nanostructures showed that the quality of yield increased with increasing temperature. For the selective area fabrication, two materials had been investigated as the layer for the carbon nanostructure growth which are resist and spin on glass. The ability of materials to withstand the high temperature phase is considered in the beginning of selection for the selective layer fabrication. The etching method and lithography process is proposed as a method to solve the problems in selective area fabrication of carbon nanostructure. The SOG is more suitable compared to resist AZ1500 in ACCVD nanostructures synthesis method due to high temperature requirement. The pretreatment processes method in hydrogen gas at 425°C for SOG is necessary for high quality layer. This method is known as annealing process to make the SOG harder, tougher and more stable in high temperature condition in synthesis process. The thickness and etching rate time of SOG is also investigated for the selective area fabrication. The samples of 0.42µm SOG is immersed in buffered oxide etch solution for five minutes to create a selective layer.

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

FABRIKASI TERPILIH BAGI PERTUMBUHAN KARBON NANOSTRUKTUR MENGGUNAKAN WAP ALKOHOL SEBAGAI PEMANGKIN DAN PUTARAN KACA

Oleh

AISHAH BINTI FAUTHAN

Mei 2015

Pengerusi: Professor Madya Mohd Nizar Hamidon, PhD
Fakulti: Institut Teknologi Maju

Matlamat kajian ini adalah untuk memahami struktur nanokarbon dan seterusnya memfabrikasi kawasan tertentu bagi tujuan aplikasi sensor. Pada masa ini, para penyelidik menggunakan teknik semburan bagi menghasilkan kawasan terpilih karbon nanostruktur. Walau bagaimanapun disebabkan isu lekatan, proses fabrikasi berbeza disiasat untuk mengatasi masalah ini. Kajian ini dimulakan dengan kajian mengenai cara menghasilkan nanokarbon menggunakan wap alkohol sebagai pemangkin (*ACCVD).* Sepanjang kajian ini, cara penyediaan pemangkin dan suhu yang berbeza telah dikaji bagi menghasilkan pertumbuhan nanokarbon yang baik. Penyediaan pemangkin dengan cara *impregnation* beserta suhu yang mencukup memberikan hasil yang dikehendaki. Suhu sintesis pula diubah menggunakan 750 °C, 800 °C dan 850 °C. Perubahan suhu ini membuktikan bahawa kualiti hasil nanokarbon meningkat dengan peningkatan suhu. Bagi fabrikasi kawasan terpilih, dua bahan telah dikaji yang akan bertindak sebagai lapisan semasa proses pertumbuhan nanokarbon. Bahan tersebut adalah *resist* dan putaran kaca (SOG). Kemampuan bahan untuk menahan suhu yang tinggi harus dipertimbangkan sebelum bahan tersebut digunakan di dalam proses sintesis nanokarbon. Kaedah punaran dan proses litografi digunakan sebagai kaedah fabrikasi dalam menyelesaikan masalah fabrikasi bagi kawasan terpilih nanokarbon. Kaedah sintesis nanokarbon memerlukan suhu yang tinggi, oleh itu kajian ini menunjukkan SOG adalah bahan yang lebih sesuai digunakan berbanding *resist*. Proses pra-perawatan di dalam laluan gas hidrogen pada suhu 425 °C diperlukan dalam fabrikasi SOG untuk menghasilkan lapisan berkualiti tinggi. Proses pra-perawatan ini bertujuan untuk menghasilkan lapisan SOG yang lebih keras dan stabil dalam keadaan suhu yang lebih tinggi semasa pertumbuhan nanokarbon. Kadar ketebalan dan masa punaran SOG dikaji bagi tujuan fabrikasi terpilih ini. Hasil kajian menunjukkan, kadar punaran adalah selama lima minit di dalam larutan penimbal oksida dengan ketebalan SOG setebal 0.42µm bagi menghasilkan paten dikawasan terpilih.

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I certify that a Thesis Examination Committee has met on 21 May 2015 to conduct the final examination of Aishah binti Fauthan on her thesis entitled "Selective Area Fabrication of Carbon Nanostructures Using Alcohol Catalyst Chemical Vapor Deposition and Spin on Glass" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

s	-	s-orbital
p	-	p-orbital
C_{60}	-	Buckminsterfullerene
CNT	-	Carbon Nanotubes
CNS	-	Carbon Nanostuctures
MWCNTs	-	Multi wall Carbon Nanotubes
SWCNTs	-	Single wall Carbon Nanotubes
TEM	-	Transmission Electron Microscopy
nm	-	Nano meter
C_h	-	Chirality Vector
$\vec{a}_1, \vec{a}_2$	-	Nearest-neighbor carbon distance
n, *m*	-	Lattice Parameters
C	-	Carbon
CVD	-	Chemical Vapor Deposition
ACCVD	-	Alcohol Catalytic Chemical Vapor Deposition
$<$	-	Less Than
g/cm^3	-	Density
Å	-	Armstrong
eV	-	Electron Volt
SOG	-	Spin on Glass
BAWs	-	Bulk Acoustics Waves
SAWs	-	Single Acoustics Waves
MEMS	-	Microelectromechanical System
HF	-	Hydrogen Fluoride
XeF_2	-	Xenon Diflouride
IDT	-	Integrated Digital Terminal
RCA	-	Radio Corporation of America
UV	-	Ultra Violet
rpm	-	rotation per minutes
Id	-	intensity for D band
Ig	-	intensity for G band

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CHAPTER 1

INTRODUCTION

1.1 An Overview of Selective Growth of Carbon Nanostructures

Research on the carbon nanostructures actively began when Harry Kroto discovered C_{60} molecules in 1985 [1]. Many researchers around the world are thoroughly investigating carbon nanostructure capability in various applications. Carbon nanostructures can be used such as energy application, healthcare, sensor technologies, the environment, and effecting material. It also has been applied for sensors due to its electrical and mechanical properties. The capability of carbon nanostructures that withstand temperatures of up to 950°C [2] meets the requirement for the sensors for high-temperature applications. Therefore, the combination of carbon nanostructures, silicon based micro fabrication, and the micromachining process will further develop nanosensors technology. This technology of sensors provides advantages in nanosensors in high sensitivity, low power consumption, compactness, high yield and low cost. The critical part is the application of nanostructures on selective growth, diameter control, and quality contacts for electronic devices.

Carbon nanostructures can be synthesized using several conventional methods, such as laser ablation and electric arc discharge [3]. Nowadays, researchers prefer to use the chemical vapor deposition (CVD) method overother conventional techniques. The synthesis of carbon nanostructures in ACCVD required a high temperature within a range of 750°C to 950°C [3], [4]. In this method, the researchers can control precisely the orientation, length, diameter, purity, and density of carbon nanostructures by adjusting the synthesis parameters.

The aim of this study is to grow carbon nanostructures and fabricate the selective area on the devices using direct growth method using gallium phosphate as a substrate. Gallium phosphate offers remarkable thermal stability up to 950°C and is suited to the temperature of carbon nanostructure synthesis techniques. Besides that carbon nanostructures and gallium phosphate are suitable for the sensors application where requires a high temperature condition[5].

Catalyst is needed for synthesis process of carbon nanostructures. There have been many types of transition metal used as the catalyst in recent research. However, iron nitrate has been chosen as a catalyst for synthesis of carbon nanostructures process. Iron nitrate was the most effective catalyst for carbon nanostructures[6], [7].

Several materials have been investigated as a masking layer to suit the high temperature requirement. There are many processes involved in fabricating materials

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on the substrate such as lift off, etching, photolithography, and curing process. The fabrication processes are investigated and analyzed to get the most suitable parameters for the carbon nanostructures synthesis purposes.

1.2 Problem Statement

Currently, the spraying technique is used for the selective area of carbon nanostructures. However, this technique is restricted by the strength of carbon nanostructures adhesion on the substrate. The parameters of carbon nanostructures growth on selective area have to be explored to determine the parameters which solve the complexity of the problem between the sensor and material as a substrate.

ACCVD is the most popular technique because of its parameter flexibility to control the structure of carbon nanostructures produced. The properties of carbon nanostructures may be examined with a suitable measurement instrument for the characterization of carbon nanostructures. Therefore, if adequate parameters of carbon nanostructures synthesis can be found, it can be applied for selective carbon nanostructures synthesis. Understanding the characteristics of carbon nanostructures is important for sensor applications.

Meanwhile, in the fabrication process, the selection of materials has to be made for the protective layer. The material chosen has to withstand the synthesis process, which requires high temperatures of up to 950°C. Gallium phosphate will be selected as the substrate where, IDT pattern is deposited on it as part of sensor system.

1.3 Research Objectives

The main research objective is to fabricate the selective growth area for carbon nanostructures using the direct growth method. In order to achieve this, the following analyzes are outlined:

i. To investigate and characterize carbon nanostructures using the ACCVD.
ii. To determine suitable materials for the layer to produce a selective pattern in fabrication process using resist and SOG.
iii. To fabricate the pattern for selective area on the gallium phosphate as a substrate for synthesis purposes.

1.4 Scope of Study

This thesis presents a sub-topic of a post-doctoral student studying the development of a surface acoustic wave resonator system for gas sensing applications. The sub-topic of the study is the synthesis of a selective area of carbon nanostructures on the system. Therefore, in this thesis, a detailed investigation on selective area fabrication of carbon nanostructures will be presented. The focus is the investigation of the

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synthesis and fabrication of the selective area of carbon nanostructures using a direct growth method to produce good adhesion on the substrate. Temperature and rotation per minute are the process parameters used in the carbon nanostructures synthesis process. The contribution is the selection of a material as a masking layer in the fabrication process for carbon nanostructure synthesis. However, it faces a difficulty where the small size of the existing gallium phosphate (2 mm x 3 mm) is used. The problem faced in fabrication process is that the substrate has to spin at a high rotational speed to produce uniform dispersion of materials on the substrate.

1.5 Layout of Thesis

This thesis begins with Chapter 2, in which the introduction, synthesis process, and preparation of carbon nanostructures growth are reviewed. The chapter also presents a fabrication of selective carbon nanostructures used in this study. The methodology and experimental set-up used in this work are presented in this chapter. Chapter 3 compares the characterization of each method used in this work. The chapter also discusses the parameter used based on the pattern of the results. In Chapter 4, the results and discussion are presented. Comparisons between the suitable materials for the selective area are elaborated. Finally, recommendations for future work are given in Chapter 5.

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