# Silicon Nanowire Sensor from Electron Beam Lithography: Design, Fabrication and Characterization

by

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A thesis submitted
in fulfillment of the requirements for the degree of
Master of Science (Microelectronic Engineering)

**School of Microelectronic Engineering**
**UNIVERSITI MALAYSIA PERLIS**

2011

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This thesis titled Silicon Nanowire Sensor from Electron Beam Lithography: Design, Fabrication and Characterization was prepared and submitted by Siti Fatimah bt Abd Rahman (Matrix Number: 0830110249) and has been found satisfactory in terms of scope, quality and presentation as partial fulfillment of the requirement for the award of degree of Master of Science (Microelectronic Engineering) in Universiti Malaysia Perlis (UniMAP).

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# DEDICATION

*Al- Fatihah to my brother, Allahyarham Harmanizam Abd Rahman, may Allah S.W.T bless you. Special dedication to my parents, Abd Rahman Aras and Hatijah Mat, and my siblings, who have supported me all the way since the beginning of my studies. May Allah S.W.T bless all of us, Amin.*

*Thank you!*

# ACKNOWLEDGEMENTS

Alhamdulillah, thanks to God Almighty for His blesses and strength that He has gave to me to finish my research. Even though I had faced many challenges during my research, but the hardest part is when the main equipment, scanning electron microscope (SEM) is shutting down for a long period which is almost 10 months. As the SEM encountered problem, indirectly the electron beam lithography (EBL) system can’t be used. Thus, I can’t proceed to produce nanowire which is the main part in this research. During that time my research progression becomes low as well as my motivation. However, thanks to Allah, finally I got the strength to move on and capable to finish my research with the support from others.

First of all, my most gratitude goes to my supervisor and co-supervisor, Prof. Dr. Uda Hashim and En. Muhammad Nuzaihan, who have providing valuable guidance and suggestions on this work. Their supervision and support truly help me to keep this research going smooth. My special thanks also go to the Dean of school Microelectronic Engineering, Assoc. Prof. Johari Adnan for his valuable support.

Many thanks also go to the Technical Staff of Microfabrication Laboratory and Nanoelectronic Laboratory, the late Mr. Phang Keng Chew, Mr Haffiz, Mr Bahari, Pn Shiela and Ms Mira, who had become the back bone of my experimental work. Without their tremendous technical support, I can’t manage to complete this project on the time.

I would like to give my sincere appreciations to my research-team, Emi Azri, who has offered his help on AutoCAD design stage. I also acknowledge Muzri for helping me to familiar with electron beam lithography system. I would also like to thank a long list of people for many enjoyable scientific discussions and personal conversations – Thikra, Kak

Emma, Kak Syud, Syidi, Fizah, Pija, Gium, Kak Mai, Seng Fatt, Cikgu Kassim, Shahrir, Naim, Azizul, Foo Kai Long and every else former and current of Nano and Microe group members. Their time, expertise and warm friendship were very much appreciated. I also want to thank Mr Anwar for kindly helping me to correct the written English.

I wish to thank National Science Fellowship (NSF) provided by the Ministry of Science, Technology and Innovation (MOSTI) for financially supported during my research work.

Finally, I want to express my appreciations to my beloved family - Mak, Ayah, Tok, Chak, Abg Chik, Kak Ayu, Adik Balqis, Adik Fatin and not forgetting Mohd Radzi for their love and encouragement. Thank you very much for supporting me every step of the way.

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# LIST OF ABBREVIATIONS

| | |
|---|---|
| 1D | One dimensional |
| Acc. Voltage | Accelerating voltage |
| AFM | Atomic Force Microscope |
| Al | Aluminum |
| Au | Aurum |
| BF | Bright Field |
| BOE | Buffered Oxide Etch |
| BOX | Buried-Oxide |
| CMOS | Complementary Metal-Oxide-Silicon |
| CVP | Chemical Vapor Deposition |
| DI | De-ionized |
| DK | Dark Field |
| DNA | Deoxyribonucleic Acid |
| dsDNA | Double-stranded DNA |
| E-beam | Electron-beam |
| EBL | Electron Beam Lithography |
| FET | Field Effect Transistor |
| GDS II Editor | Graphic Display System II Editor |
| $H_2$ | Hydrogen |
| HPM | High Power Microscope |
| ICP-RIE | Inductive Coupled Plasma-Reactive Ion Etching |
| ISFET | Ion-Selective Field Effect Transistor |
| I-V | Current-Voltage |
| KOH | Kalium Hydroxide |
| LPM | Low Power Microscope |
| MBE | Molecular Beam Epitaxy |
| NW | Nanowire |
| PMMA | Polymethyl Methacrylate |

| | |
|---|---|
| PR | Photoresist |
| PVD | Physical Vapor Deposition |
| RIE | Reactive Ion Etching |
| RTA | Rapid Thermal Annealing |
| SEM | Scanning Electron Microscope |
| Si | Silicon |
| $SiH_4$ | Silane |
| SiNW | Silicon Nanowire |
| $SiO_2$ | Silicon Dioxide |
| SiOH | Silanol |
| SMU | Source Measurement Units |
| SOI | Silicon-On-Insulator |
| SPA | Semiconductor Parametric Analyzer |
| SPR | Surface Plasmon Resonance |
| ssDNA | Single-stranded DNA |
| TEM | Transmission Electron Microscopy |
| UV | Ultraviolet |
| VLS | Vapor Liquid Solid |
| WCM | Wet Cleaning Module |
| ZnO | Zinc Oxide |

# LIST OF SYMBOLS

| | |
|---|---|
| $d$ | Diameter |
| $l_c$ | Carrier mean free path |
| W | Width |
| $H^+$ | Hydrogen ion |
| I | Current (A) |
| R | Resistance (Ω) |
| V | Voltage (V) |
| S | Sensitivity |
| $R_0$ | initial resistance (Ω) |
| $\Delta R$ | change in resistance (Ω) |

# SILIKON NANOWAYAR PENDERIA MENGGUNAKAN LITOGRAFI ALUR ELEKTRON: REKABENTUK, FABRIKASI DAN PENCIRIAN

## ABSTRAK

Kajian ini menunjukkan proses pembangunan penderia SiNW yang memerlukan kedua-dua permintaan iaitu menghasilkan wayar berskala nano dan berintegrasi dengan kebiasaan proses CMOS. Sebelum proses fabrikasi yang sebenarnya, penderia SiNWs direka bentuk dengan menggunakan Elphy Quantum GDS II Editor dan AutoCAD. Sejumlah empat corak iaitu nanowayar, tanda penjajaran, petak elektrod dan kawasan penguji telah direkabentuk untuk menghasilkan peranti penderia SINWs yang lengkap. Dengan menggunakan silikon atas penebat sebagai bahan permulaan, nanowayar dihasilkan dengan menggunakan pendekatan atas-bawah yang melibatkan Mikroskop Pengimbas Elektron berasaskan kaedah Litografi Alur Elektron. Kesan pada lebar garis dan dos dedahan keatas struktur corak dikaji dengan menggunakan fotorintang negatif ma-N2403 untuk Litografi Alur Elektron. Pelbagai dos dedahan dalam lingkungan 50µC/cm$^2$ hingga 180µC/cm$^2$ pada pemecutan voltan 20kV dengan arus alur 0.075nA didedahkan ke atas fotorintang negatif tersebut. Nanowayar topeng kerintangan dihasilkan dengan sempurna iaitu mempunyai dimensi kurang daripada 100nm lebar pada dos dedahan berparameter 80µC/cm$^2$, 100µC/cm$^2$ dan 120µC/cm$^2$. Selepas itu, dua elektrod logam yang direka sebagai sumber dan salur keluar telah dibangunkan ke atas setiap nanowayar dengan menggunakan proses litografi konvensional. Ciri-ciri mofologi, elektrik dan kimia telah digunakan untuk melihat hasil peranti yang dibina. Bahagian-bahagian utama adalah untuk meneliti profil nanowayar dalam membuktikan ianya adalah berskala nano dengan menggunakan Hawk 3D-nanoprofiler dan untuk menguji prestasi peranti secara elektrik dengan menggunakan Semikonduktor Parametrik Analyzer (SPA) dalam hal berkaitan arus-voltan. SiNW terkecil dengan diameter 65nm yang dijajarkan dengan sempurna dengan pelapik elektrod telah diperoleh. Akhirnya, peranti yang dibina telah digunakan sebagai pengesanan tahap pH. Tiga jenis larutan penampan dengan pH 4, pH 7 dan pH 10 digunakan untuk menguji tindak balas elektrik peranti tersebut. SiNWs penderia menunjukkan nilai pernentangan tertinggi bagi pH 4 dan nilai penentangan terendah bagi pH 10. Dari segi kepekaan, peranti dengan nanowayar yang lebih kecil didapati lebih sensitif berbanding nanowayar yang lebih besar hasil daripada nisbah permukaan tinggi untuk isipadu.

# SILICON NANOWIRE SENSOR FROM ELECTRON BEAM LITHOGRAPHY: DESIGN, FABRICATION AND CHARACTERIZATION

## ABSTRACT

This study demonstrates the process development of silicon nanowires (SiNWs) sensor requires both the fabrication of nanoscale diameter wires and standard integration to CMOS process. Prior to actual fabrication process, the SiNWs sensor is designed via Elphy Quantum GDS II Editor and AutoCAD. A total of four designs namely nanowire, alignment mark, electrode pad and test channel are designed in order to create a complete SiNWs sensor device. By using silicon-on-insulator (SOI) wafer as a starting material, the nanowires is fabricated using a top-down approach which involved Scanning Electron Microscope (SEM) based Electron Beam Lithography (EBL) method. The effect of line width and exposure dose on the pattern structure is investigated experimentally using the negative photoresist ma-N2403 for EBL. The exposure doses for the resist layer are varied in the range of 50µC/cm$^2$ to 180µC/cm$^2$ at 20 kV accelerating voltage with a beam current of 0.075nA. The nanowires resist masks are well developed with dimension less than 100 nm in width for the dose exposure parameters of 80µC/cm$^2$, 100µC/cm$^2$ and 120µC/cm$^2$. Subsequently, the two metal electrodes which are designated as source and drain are fabricated on top of individual nanowire using conventional lithography process. Morphological, electrical and chemical characteristics have been proposed to verify the outcome of the fabricated device. The major parts are to observe the nanowire profile in order to meet the nano-scale dimension by using Hawk 3D-nanoprofiler and to test the device performance electrically by using Semiconductor Parametric Analyzer (SPA) in terms of I-V relations. It is found that, the smallest SiNW with diameter of 65nm is well aligned with electrode pads have been obtained. Finally, the fabricated device is performed as pH level detection. Three types of standard aqueous pH buffer buffer solutions which are pH 4, pH 7 and pH 10 are used to test the electrical response of the device. The SiNWs sensor show the highest resistance value for pH 4 and the lowest resistance value for pH 10. In terms of sensitivity, the device with smaller nanowire is found to be more sensitive than larger nanowire as a result of the high surface-to-volume ratio.

# CHAPTER 1

# BACKGROUND

## 1.1 Introduction

This introductory chapter starts a brief explanation of the background and roadmap of nanotechnology, followed by the overview of the nanowire sensor device. Next, the objectives and scopes of this research are described in detail. Lastly, the organization of dissertation is addressed.

## 1.2 Nanotechnology

Nanotechnology entails the control of matter at the scale of 1 to 100 nanometers to create the functional materials, devices and systems (Wang, 2005). The word “nano” means $10^{-9}$, so a nanometer (nm) is one billionth of a meter which in comparison states that a typical dimension of 10nm is 1000 times smaller than the diameter of a human hair (Hunt & Mehta, 2006). Discovery of these nanoscale things such as carbon nanotubes and nanowires, had provided researchers with an opportunity to construct electronic interfaces with components which sizes are comparable to the size of biological molecules (Noy, Artyukhin, & Misra, 2009) such as proteins, viruses, deoxyribonucleic acid (DNA), ions and molecules as illustrated in Fig. 1.1.

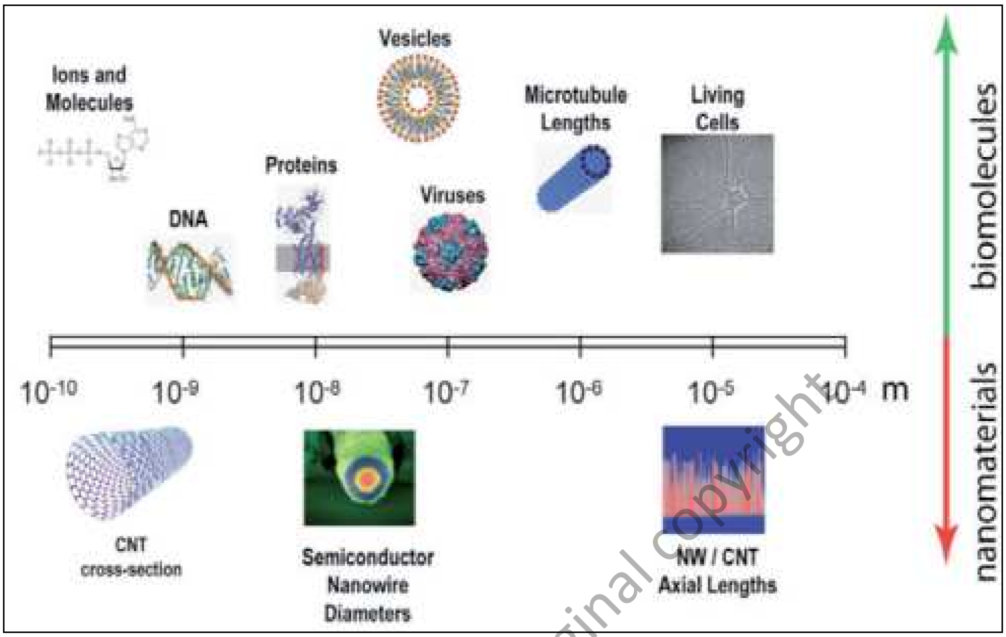

Figure 1.1: Nanometer scale chart. Comparison of natural things and manmade things in terms of dimensions (Noy et al., 2009).

Despite the current excitement and recent waves of visionary predictions, nanotechnology is not entirely a new area (P. Wagner, 2005). The reality is back in 29th December 1959, Nobel Laureate Richard P. Feynman had given a talk at the annual meeting of the American Physical Society in the California Institute of Technology, which had become one of the twentieth century's classic science lecturers. In the talk which titled "There's Plenty of Room at the Bottom", he presented a technological vision off extreme miniaturization several years before the word "chip" became part of the lexicon (Bhushan, 2006; Mansoori, 2005). He also discussed about the problem of manipulating and controlling things on a small scale. Extrapolating from known physical laws, a technology using the ultimate toolbox of nature which building nanoobjects atom by atom or molecule by molecule was envisioned by the renown scientist, Feynman (Bhushan, 2006).

Consequently in 1974, Norio Taniguchi, a Japanese researcher had first coined the term ‘nanotechnology’ which in engineering at length scales less than a micrometer (Prasad, 2008). However it is noted that a futurist K. Eric Drexler has been widely credited for popularizing the term in the mainstream. In his book Engines of Creation published in 1986, Drexler envisioned a world in which tiny machines are able to build other structures with exquisite precision by physically manipulating individual atoms (Drexler, 2006). Thus, since 1980’s has led to many on inventions and discoveries in the fabrication of nanoobjects which have become a demonstration to Feynman’s vision (Bhushan, 2006).

In 1991, carbon nanotubes were discovered by Sumio Iijima at NEC Fundamental Research Laboratories in Tsukuba, Japan (L. Williams & Adams, 2007). Then, scanning probe microscopes which is capable of imaging and manipulating surface-bound molecules with atomic-scale precision, have been around for about 15 years and now commonly used in all fields such as material science (P. Wagner, 2005). Nanotechnology is considered to be a general purpose technology which still has most of its experts located in academia and government-funded basic research centers. Table 1.1 summarizes the chronicles of major nano events since 1960s in nanotechnology development.

In nanotechnology, there are two commonly techniques that become approach for fabrication process which known as bottom-up approach and top-down approach. The bottom-up approach attempts to build nanodevices from atomic or molecular component, meanwhile the top-down approach is basically based on the lithographic techniques to pattern bulk material (Parak, Simmel, & Holleitner, 2008). In this research work, the top-down approach was chosen to form resist mask and fabricate the nanoscale structures. The differences between these two nanotechnology fabrications approaches will be summarized in Chapter 2, Section 2.2.3.

Table 1.1: The roadmap of nanotechnology adapted from (Frederick, 2005).

| Years | Events |
|---|---|
| 1959 | Speech given by physicist Richard Feynman entitled “There’s Plenty of Room at the Bottom” |
| 1968 | Alfred Y. Cho and John Arthur of Bell Laboratories invented molecular beam epitaxy (MBE) that can deposit single atomic layers on a surface. |
| 1974 | Japanese researcher Norio Taniguchi coined the word ‘nanotechnology’ |
| 1981 | Invention of scanning tunneling microscope (STM) by Gerd K. Binnig and Heinrich Rohrer. |
| 1985 | Richard Smalley, Robert Curl and Harold Kroto discovered buckminsterfullerenes, also known as C60 and the diameter was about one nanometer. |
| 1986 | Invention of atomic force microscope (AFM) by Binnig, Quate and Gerber. |
| 1991 | Discovery of carbon nanotubes by Sumio Iijima of NEC, Japan. |
| 1996 | Curl, Kroto and Smalley awarded Nobel prize in chemistry – discovery of Buckminsterfullerene (or fullerene). |
| 1998 | Cees Dekker’s group from Delft University of Technology, Netherlands created a carbon nanotube-based transistor. |

## 1.3 Development of Nanowire Sensor Device

Nanowires (NWs) and other one-dimensional (1D) nanostructures has introduced to be as a suitable base for sensing element as well as maintaining the historical trend of scaling down the electronic devices (Frederick, 2005). These materials are attractive because they have very narrow diameters and provide a link between molecular and solid state physics (Frederick, 2005; Gorton, 1993). Due to quantum confinement effects and their high surface area-to-volume ratios , NWs can be proposed as chemical and biological sensing element (Sheriff, 2009).