SILICON SELF-ASSEMBLED NANODOTS FABRICATED USING A RADIO-FREQUENCY MAGNETRON SPUTTERING METHOD

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A thesis submitted in fulfilment
of the requirements for the award of the degree of
Master of Science (Physics)

Faculty of Science
Universiti Teknologi Malaysia

JANUARY 2008

Specially dedicated to my beloved parents, sisters, brother and my little cousin
Dad, Mum …… Thanks for setting me on the path toward intellectual pursuits
Yun, Chan, Xin, Yao …… Thanks for your continuing support along the way
Cute-cute Han …… Thanks for cheer me up always

ACKNOWLEDGEMENTS

I would like to express my sincerest appreciation to my project supervisor Prof. Dr. Samsudi Sakarani and co-supervisor Prof. Dr. Yusof Wahab for their advice, guidance and encouragement throughout completing this project. Especially thanks to my project supervisor who had spent a lot of time to assist and consult me in this venture.

Futhermore, this thesis would not have been possible without the very pleasant and creative working atmosphere that at the Nanostructure and Nanophysic Laboratory, Institute Ibnu Sina, UTM Skudai. My great appreciation also dedicated to my IRPA group members especially Pak Imam Sumpono and Mohd Hishamuddin. Not forgetting lab attendance Mr. Nazri and Kak Wani at IIS and also the researcher from Telekom R&D, Encik Razman. Their help has turn out to be inestimably important.

In addition, my sincere appreciation also extends to all my postgraduate friends and other who has provides assistance at various occasions. Their views and suggestions are useful indeed. Last but not least, I am grateful to all my family members that support me all the time.

ABSTRACT

Silicon nanodot is a promising nanostructured material for future single-electron devices in nanoelectronic system. The self-assembly growth of silicon nanodots on sapphire substrate was investigated, with highlights on the very early stage of nucleation and the growth process. The scope of study covers both the theoretical approach and experimental works. A classical theory of nucleation was applied to a liquid-solid phase transition, combined with high temperature supercooling to establish the expression for the net energy change in the formation of silicon nanodots. Using a computer program, the predicted parameters, such as critical radius ($r*$), critical energy ($\Delta G*$), surface energy (γ_{NS}), and free energy change per unit area (ΔG_v) were obtained and tabulated into a dome-like shape nucleus following the Volmer-Weber growth mode. Experimental works have been conducted using a radio-frequency magnetron sputtering under the varying conditions of 5-20 minutes deposition time, 100-400°C substrate temperature and 50-200 W radio-frequency power. Optimum experimental conditions for the onset of silicon nanodot were found to be at 5 minutes/400 ^{o}C/100 W setting. Characterization measurements have been done on this sample using AFM, PL, XRD and EDX. Observation from AFM indicated the presence of small islands with an average diameter of 40.81 nm. The results from PL analysis revealed the existence of a peak which corresponded to a bandgap energy of 1.78 eV. This was further confirmed by the presence of 0.48 at.% of silicon on the substrate using EDX. A further XRD analysis gave no indication of a crystallinity phase probably due to extremely small amount of silicon formed on the substrate. The results showed that the formation of dome-like silicon nanodots on sapphire substrate occurred during the first 3 minutes of deposition, ascribed by the surface energy mismatch at interface and governed by a Volmer-Weber growth mode. A further growth of silicon nanodots were found to change their properties and strongly dependent on the experimental conditions.

ABSTRAK

Nanobintik silikon merupakan bahan nanostruktur yang berpotensi untuk peranti transistor elektron tunggal dalam sistem nanoelektronik pada masa hadapan. Pertumbuhan swa-terhimpun nanobintik silikon di atas substrat alumina telah dikaji dan tumpuan diberikan kepada penukleusan peringkat awal dan proses pertumbuhan. Skop kajian adalah meliputi teori dan eksperimen. Teori penukleusan klasik telah diaplikasikan untuk peralihan fasa cecair-pepejal serta gabungan suhu penyejukkan lampau yang tinggi bagi menghasilkan persamaan perubahan tenaga yang diperlukan untuk membentuk nanobintik silikon. Dengan menggunakan pengatucaraan komputer, parameter ramalan seperti jejari genting ($r*$), tenaga genting ($\Delta G*$), tenaga permukaan (γ_{NS}), perubahan tenaga per unit luas (ΔG_v) telah digunakan untuk menghasilkan nukleus yang berbentuk macam-kubah dengan mengikut mod pertumbuhan Volmer-Weber. Eksperimen telah dijalankan dengan menggunakan teknik percikan pemagnetan frekuensi radio berikut dengan perubahan pelbagai parameter pemendapan seperti masa pemendapan antara 5 - 20 minit, suhu substrat antara 100 ^{o}C- 400 ^{o}C dan kuasa frekuensi radio dalam julat 50 W-200 W. Keadaan eksperimen yang optimum untuk pertumbuhan nanobintik silikon telah dikenalpasti dan didapati berlaku pada persekitaran 5 minit/400 ^{o}C/100 W. Pengukuran pencirian terhadap sampel telah dilakukan dengan menggunakan AFM, PL, XRD dan EDX. Pemerhatian daripada AFM menunjukkan wujudnya pulau kecil yang mempunyai diameter purata dalam 40.81 nm. Analisis PL menunjukkan kewujudan puncak yang jurang tenaganya bernilai 1.78eV. Ini telah dibuktikan dengan analisis EDX, iaitu 0.48% atom silikon telah terbentuk di atas substrat. Lanjutan daripada analisis XRD menunjukkan bahawa tiada fasa penghabluran dikesan kemungkinan disebabkan oleh kuantiti silikon yang sangat kecil termendap di atas substrat. Keputusan eksperimen menunjukkan nanobintik silikon yang berbentuk macam-kubah telah terjadi seawal masa pemendapan 3 minit disebabkan oleh ketidaksepadanan tenaga permukaan dan mematuhi mod pertumbuhan Volmer-Weber. Pertumbuhan nanobintik silikon yang seterusnya didapati berubah sifat-sifatnya dan sangat bergantung kepada keadaan eksperimen yang ditetapkan.

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LIST OF ABBREVIATIONS / SYMBOLS

a_x - The lateral size of the nanodots

at.% - Atomic percentage

a-Si - Amorphous Silicon

AFM - Atomic Force Microscope

Al_2O_3 - Aluminium Oxide or Sapphire

Ar - Argon

B - Magnetic field

c - Velocity

ΔC_p - Difference in specific heat

CVD - Chemical vapour deposition

DC - Direct Current

eV - Electron volt

E_e - Energy of the electron

E - Electric Field

EDX - Energy Dispersive X-Ray Spectroscopy

F-M - Frank-van der Merwe

FESEM - Field Emission Scanning Electron Microscope

FTM7 - Film Thickness Monitor

FWHM - Full Wave Half Maximum

ΔG - Net change of energy (Gibbs Work)

ΔG^* - Critical energy

ΔG_{hom} - Net change of energy for homogeneous nucleation

$\Delta G^{*}_{\mathrm{hom}}$ - Critical energy for homogeneous nucleation

ΔG_{het} - Net change of energy for heterogeneous nucleation

ΔG^{*}_{het} - Critical energy for heterogeneous nucleation

ΔG_{v} - Free energy change per unit volume

GIXRD - Grazing Incidence X-ray Diffraction

GaN - Gallium Nitride

Δh_{v} - The changes in enthalpy

ΔH_{f} - Enthalpy of fusion

h - Planck constant = $6.6260755 \times 10^{-34}$ Js

H_2 - Hydrogen

HRTEM - High Resolution Transmission Electron Microscopy

Hz - Hertz

k - Boltzmann constant

L_a - Lattice constants for Silicon

L_b - Lattice constants for Sapphire

L_f - Latent heat of fusion of the material per unit volume

LPCVD - Low-Pressure Chemical Vapour Deposition

LSI - Large-Scale Integration

m - Mass of electron

n - Principal Quantum Number

N_2 - Nitrogen

p - Vapour pressure

p_e - Saturation vapor pressure

PECVD - Plasma-Enhanced Chemical Vapour Deposition

PL - Photoluminescence

PVD - Physical Vapour Deposition

Q - The scattering vector

r - The radius of the nucleus

r^* - Critical radius

$r^*_{\hom}$	-	Critical radius for homogeneous nucleation
r^*_{het}	-	Critical radius for heterogeneous nucleation
R	-	Gas constant
RF	-	Radio Frequency
RMS	-	Root mean square
S	-	Supersaturation ratio
Δs_v	-	The changes in entropy
SET	-	Single Electron Transistor
SEM	-	Scanning Electron Microscope
Si	-	Silicon
SiH_4	-	Silane
SiO_2	-	Silicon dioxide
S-K	-	Stranski-Krastanov
T	-	Temperature
T_m	-	Solidification temperature
ΔT	-	Undercooling temperature
v_o	-	Atomic volume
V-W	-	Volmer-Weber
wt.%	-	Weight percentage
XPS	-	X-Ray Photoelectron Spectroscopy
XRD	-	X-ray Diffraction
λ	-	The Peak Wavelength Emission
π	-	Pi = 3.1415926
α	-	Incidence angle
β	-	Exit angle
γ	-	Surface energy per unit area
γ_{LN}	-	Surface energy per unit area at liquid-nucleus interface
γ_{NS}	-	Surface energy per unit area at nucleus-solid interface

γ_{LS}	-	Surface energy per unit area at liquid-solid interface
θ	-	Contact angle
ε	-	Lattice mismatch
ϑ	-	Number of monolayers deposited

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

The emerging field of nanoscience aims to create new materials with length scales of approximately 1-100 nanometers, ranging from macromolecules to nanoscale solid, and to understand the fundamental of new phenomena displayed by these nanoscale materials. It has stimulated interdisciplinary research at the interfaces of chemistry, physics, biology, and engineering sciences, and it is also the cornerstone for the discovery and development of revolutionary nanotechnology[1].

Semiconductor technology is one of the fields that dominated by the revolutionary of nanotechnology now. The trend toward miniaturization, dominating the technological progress of the past decades, corresponds to the demands for faster operation and less heat dissipation. Furthermore, the rapid development of the semiconductor technology, including epitaxy techniques for preparation of even single-molecular layers of materials, lithography methods, and advances in controlling self-aggregation processes, results in quite new possibilities for artificial creation of ultra small physical system with controlled properties[2].

Silicon is at the center of today's modern electronics consumer industry. Since there is a trend towards ever smaller electronic devices, microelectronics may eventually go over to nanoelectronics and maybe the key technology in the 21st century[3]. Silicon becomes the preferred material for single-electron and quantum-electronic devices from a practical point of view are due to the reason that mature technologies can be used during the fabrication process[4]. This is a definite advantage in developing commercial devices. Research into the practical uses of silicon single-electron and quantum-electronic devices began in the early 1990s when the fabrication of nanostructures in silicon became possible owing to the continuous reduction of device size in LSI circuits for higher integration and performance.

Silicon nanodots, also known as silicon quantum dots or silicon nanocrystal, consist of 100s-1000s of atoms and are approximately one billionth of a meter in size. It becomes the interest of research work due to its specific structure, electronic and optical properties. The latter was stimulated by the discovery of efficient light emission in different forms of silicon nanostructures[5] and by the demonstration of a silicon-based light-emitting device prototype integrated into conventional microelectronic circuitry[6]. Hence, the interest in reliable fabrication of silicon-based nanostructures with control over the nanocrystal size, shape, and crystallographic orientation has been growing continuously the last decade. Recently, the application of silicon nanocrystals in electronic devices was suggested and proved by the demonstration of a silicon nanocrystal nonvolatile memory and other devices utilizing the coulomb bloackage effect[7,8].

In general, there are two philosophically distinct approaches for creating silicon nanostructure, which can be characterized as top down and bottom up. In the conventional top-down approach, nanostructures are patterned in bulk materials by a combination of lithography, etching and deposition to form functional devices. This approach has greatly pushed nanoscience research forward during the past few decades, although efforts to push the resolution down to a few nanometers will face scientific challenges intrinsic to the top-down approach. Recently the bottom–up approach to form nanometer- scaled silicon structures with silicon nanodots are attracting us more particularly for future nanoelectronics and quantum information device applications.

Bottom-up, or self-assembly, approaches is the nanofabrication technique which use chemical or physical forces operating at the nanoscale to assemble basic units into larger structures[9]. The latter has two subdisciplines, physical vapor-deposition (PVD) and chemical vapor deposition (CVD). The difference between the last two is that in PVD the actual active species are directly evaporated or injected into the gas phase; in CVD, a precursor is used that, on transporting into the vapour space, is chemically decomposed into the required species[1]. The key advantage of bottom-up approach is that critical nanoscale features are defined during synthesis, and this can yield uniform structures at the atomic scale.

Despite the research history for silicon nanodots is still short, but we believe that the unique properties of silicon nanodots will make it a strong candidate for a major application of nanotechnology in the future including the single electron transistor. Thus, in order to understand on how silicon nanodot is being formed, an in depth knowledge of nucleation, i.e. an early stage of the formation of stable nuclei from the gas/liquid/solid phases, its growth process, and the control of fabrication process become a pre-requisite requirement for those working in this area.

1.2 Research Objectives

- To study the theory of nucleation and growth of silicon nanodots and predict the related parameters.
- To set up the Radio Frequency (RF) Magnetron Sputtering system for the fabrication of samples.
- To determine the optimum experimental conditions for the fabrication of silicon self-assembled nanodots.
- To analyze and interpret the results obtained from the characterization measurements.

1.3 Research Scopes

Recently the bottom–up approach to form nanometer- scaled silicon structures with silicon nanodots are attracting researchers for future nanoelectronics and quantum information device applications. In this research, the very early stage of nanodots formation known as nucleation and the assembly of nano-sized islands have been investigated theoretically and experimentally. The sapphire has been chosen as substrate. Samples of silicon nanodots are fabricated using a RF magnetron sputtering under different experimental conditions, and further investigation on the optimum operational parameters. The following setting parameters are identified: Deposition times vary from 5 to 20mins, substrate temperatures between 100 ^{o}C to 400 ^{o}C and finally the RF powers ranging from 50-200W. Characterization measurements are performed using Atomic Force Microscope (AFM), Photoluminescence Spectroscopy (PL) and X-Ray Diffraction (XRD). In addition, analysis using Energy Dispersive X-ray Spectrometry (EDX) has been conducted to confirm the contents of nanodots.

1.4 Research Problems

Self-assembly has become very effective and promising approach to synthesize a wide range of novel nanoscale materials due to unexpected physical properties that the self-aggregated nanoparticles show. Research on silicon nanostructures has a great attention with the excitement that the size range of silicon nanostrutures start exhibiting novel properties that are both quantitatively and qualitatively different from those of their respective bulk materials and from the discrete atoms or molecules which they are derived.

Thus, the fabrication of silicon nanodots have been investigated in many research works which will be discussed in Chapter 2. It is found that the study for early stage growth of silicon nanodots still less been reported. A lot of works has been concentrated to study the nucleation and growth of GaN on sapphire based on the large lattice mismatch that evokes the Volmer-Weber growth. It is observed that the large lattice mismatch also appear between silicon and sapphire. In order to investigate the self-assembly growth of silicon nanodots following the Volmer-Weber growth mode in the early stage of film growth, the sapphire substrate has been chosen as substrate. The RF sputtering system has been chosen as the technique to fabricate the silicon nanodots on sapphire in this research since it has been reported as promising technique to produce silicon nanocrystals in many research works.

In order to better understand the nucleation and growth mechanisms of the silicon nanodots, the cappilarity approximation of nucleation theory has been studied. A computing program has been developed based on the quantitative description of nucleation theory. The influences of temperature, surface energy and contact angle to the critical size and critical energy have been predicted theoretically.

In the following, the morphology and microstructural evolution of the silicon nucleation layer as the function of growth time also been reported in this research. The effect of substrate temperature and rf power to the surface morphology, silicon composition, photoluminescence and X-ray diffraction results have been investigated.

1.5 Layout of Thesis

This thesis is comprised of 5 chapters. The first contains an overview of research background and specified the aim of studies, choice of system, and outline of the thesis plan. Chapter 2 discusses the previous work done, theory of self assembled growth, nucleation and fabrication techniques. The following chapter 3 covers the simulation setup, sample preparation, setup of the system, design of the experiment and also methods of measurement and characterization. Chapter 4 presents the simulation and experimental results obtained in this work with the details discussion. Finally, the conclusion of the project is made in chapter 5. It also includes the summarization of the whole project and some recommendations for future work.

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