GALLIUM NITRIDE NANOWIRE BY NITRIDATION OF ELECTROCHEMICALLY GROWN GALLIUM OXIDE ON SILICON

NORIZZAWATI BINTI MOHD GHAZALI

UNIVERSITI TEKNOLOGI MALAYSIA

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NORIZZAWATI BINTI MOHD GHAZALI

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ABSTRACT

GaN is a wide bandgap semiconductor with superb thermal, chemical, mechanical and electrical properties which makes it suitable for high power electronic and optoelectronic devices. Si substrate is preferable for the heterostructure growth of GaN due to its availability in large wafer size, low price and maturity. The co-integration of GaN-based devices on Si is very attractive towards the realization of advanced heterogeneous integration . A transformation of the grown Ga_2O_3 structures on Si to GaN by a so-called nitridation process is considered as a simple method to create a GaN/Si heterostructure. In the first stage, a synthesis of β-Ga_2O_3 nanostructures on Si substrate by electrochemical deposition using a mixture of Ga_2O_3, HCl, NH_4OH, and H_2O was performed. The morphologies strongly depended on the molarity of Ga_2O_3 and pH level of electrolyte. β-Ga_2O_3 nanodot-like structures were grown at low molarity of Ga_2O_3. However, Ga_2O_3 nanodot structures covered with nanorods on top of their surfaces were obtained at higher molarity, and the densities of nanorods seem to increase with the decrease of pH level. In the next stage, the nitridation of the electrodeposited Ga_2O_3 was performed. The complete nitridation was achieved at temperature of 900°C. Here, several prominent diffraction peaks correspond to hexagonal GaN (h-GaN) planes were detected with no diffraction peak of Ga_2O_3 structure. Temperature is a key parameter in a nitridation process where the deoxidization rate of Ga_2O_3 to generate gaseous Ga_2O increase with temperature. It was found that a complete transformation cannot be realized without a complete deoxidization of Ga_2O_3. A significant change of morphological structures takes place after a complete transformation of Ga_2O_3 to GaN where the original nanorod structures of Ga_2O_3 diminish, and a new nanowire-like GaN structures appear. The studied method seems to be promising in producing high-quality h-GaN nanostructures on Si.

ABSTRAK

GaN adalah bahan semikonduktor yang mempunyai sela jalur yang luas serta ciri-ciri yang luar biasa seperti ciri-ciri haba, kimia, mekanikal dan elektrik yang menjadikan ia sesuai untuk dijadikan sebagai peranti elektronik berkuasa tinggi dan peranti optoelektronik. Silikon (Si) substrat adalah lebih sesuai digunakan untuk pertumbuhan strukturhetero GaN kerana adanya saiz wafer Si yang lebih besar, harga yang murah dan kematangan teknologi berasaskan Si. Di samping itu, fabrikasi peranti berasaskan-GaN pada platform Si sangat menarik ke arah merealisasikan integrasi heterogen termaju. Pada peringkat pertama, strukturnano β-Ga_2O_3 telah disintesis pada substrat Si melalui proses pemendapan elektrokimia menggunakan campuran Ga_2O_3, HCl, NH_4OH, dan H_2O. Morfologi Ga_2O_3 yang dideposit sangat bergantung kepada molariti Ga_2O_3 dan tahap pH elektrolit. Struktur berupa nanodot Ga_2O_3 telah tumbuh diatas substrat Si pada keadaan molariti Ga_2O_3 yang rendah. Walaubagaimanapun, struktur nanodot Ga_2O_3 dilitupi dengan nanorods di atas permukaannya diperoleh pada molariti yang lebih tinggi, dan ketumpatan nanorod kelihatan meningkat dengan penurunan tahap pH. Pada peringkat seterusnya, proses penitridaan Ga_2O_3 telah dilakukan selepas melalui proses electrokimia. Pada suhu 900°C penitridaan yang lengkap telah dicapai. Pada suhu ini, beberapa puncak pembelauan utama diperolehi berpadanan dengan satah hexagon GaN (h-GaN) dikesan tanpa puncak pembelauan struktur Ga_2O_3. Suhu adalah parameter utama dalam proses peniridaan, dimana kadar penyahoksidaan bagi Ga_2O_3 untuk menjana gas Ga_2O adalah meningkat dengan suhu. Transformasi lengkap Ga_2O_3 kepada GaN tidak dapat direalisasikan tanpa penyahoksidaan Ga_2O_3 yang lengkap. Perubahan ketara morfologi berlaku selepas transformasi lengkap Ga_2O_3 kepada GaN dimana struktur asal nanorod Ga_2O_3 telah mengecil dan nanowayar GaN yang baru telah muncul. Keputusan ini menunjukkan bahawa kaedah yang dibentangkan sangat berpontensi dalam menghasilkan struktur-struktur h-GaN yang berkualiti tinggi.

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LIST OF ABBREVIATIONS

1D - One Dimensional
2D - Two Dimensional
AlN - Aluminium Nitride
Au - Gold
CMOS - Complementary Metal Oxide Semiconductor
CVD - Chemical Vapor Deposition
CL - Cathodoluminescence
Co - Cobalt
Cl_2 - Chloride
CO_2 - Carbon Dioxide
DC - Direct Current
EDX - Energy Dispersive X-Ray
Eu_2O_3 - Europium Oxide
Fe - Iron
FET - Field Effect Transistor
FESEM - Field-Emission Scanning Electron Microscopy
FTIR - Fourier Transform Infrared Spectroscopy
Ga - Gallium
GaAs - Gallium Arsenide
GaCl - Gallium Chloride
GaN - Gallium Nitride
$Ga(NO_3)_3$ - Gallium Nitrite Hydrate
Ga_2O_3 - Gallium Oxide
GaOOH - Gallium Oxide Hydrate

Ge - Germanium
H_2 - Hydrogen
H_2O_2 - Hydrogen Peroxide
H_2SO_4 - Sulphuric Acid
HF - Hydrofluoric Acid
HNO_3 - Nitric Acid
HVPE - Hydride Vapor Phase Epitaxy
HEMT - High Electron Mobility Transistor
LED - Light Emitting Diode
MBE - Molecular Beam Epitaxy
MESFETs - Metal-Semiconductor Field-Effect Transistor
MOCVD - Metal-Organic Chemical Vapor Deposition
MOVPE - Metal-Organic Vapor Phase Epitaxy
Nb - Neobium
Ni - Nikel
NH_3 - Ammonia
N_2 - Nitrogen
NH_4OH - Ammonia Water
PLD - Pulse Laser Deposition
PL - Photoluminescence
Pt - Platinum
RF - Radio Frequency
RCA - Radio Corporation Of America
RT - Room Temperature
Si - Silicon
SiN - Silicon Nitride
SiC - Silicon Carbide
SnO_2 - Tin Dioxide
Si-LSIs - Silicon Large Scale Integrated Circuit
SDBS - Sodium Dodecylbenzene Sulfonate
SEM - Scanning Electron Microscopy

TEM - Transmission Electron Microscopy
TMG - Trimethlygallium
Ti^{4+} - Titanium Ion
UV - Ultraviolet
V - Vanadium
XRD - X-Ray Diffraction
Zr^{4+} - Zirconium Ion

LIST OF SYMBOLS

eV - Electron volt

α - Alpha

β - Beta

γ - Gamma

δ - Delta

ε - Sigma

$^{\circ}C$ - Degree Celsius

$\mathring{A}$ - Angstrom

nm - Nanometer

Pa - Pascal

μm - Micrometer

Ωcm - Ohm Centimeter

A/cm^2 - Ampere per Square Centimeter

ml - Mililiter

J - Current Density

$^{\circ}C/min$ - Degree Celsius per Minutes

n - An integer 1, 2, 3

λ - Wavelength

d - Inter-plane Distance in Angstroms

θ - Diffraction Angle

cm - Centimeter

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Gallium nitride (GaN) is a very hard, chemically and mechanically stable wide band gap (3.4 eV) semiconductor material with high heat capacity and thermal conductivity [1] which makes it suitable to be used for some electronics and optoelectronics devices. In recent years, gallium oxide (Ga_2O_3) has been studied as the seed material for chemical synthesize of GaN by thermal transformation method [2]. A transformation of the grown Ga_2O_3 structures on Si to GaN by a nitridation of Ga_2O_3 through the utilization of a so-called ammoniating process seems to be a promising technique to create a GaN/Si heterostructure. Furthermore, the integration of GaN-based devices on Si platform seems to be very attractive for the hybrid integration towards 'More than Moore' technology [3].

Superb properties of GaN which include direct wide band gap of 3.4eV makes it suitable to be used for high power electronic devices such as a field effect transistor (FET) [4-6], sensors [7-9] and optoelectronic devices such as light emitting diode (LED) [10-12]. Up to this date, many techniques have been explored to synthesize GaN nanostructures including nanowires, nanorods, nanodots and so forth since such low dimensional nanostructures are promising for increasing the performance optoelectronic devices and the sensitivity of sensors [13,14]. For example, GaN nanorods and nanowires have been applied for chemical sensing application as reported by Wright *et al.* and Huang Y *et al.*, respectively [15,16], due to large surface to volume ratio. GaN nanodots have been used in a photodetector as reported by Kumar *et al.* [17].

Several vapor-phase techniques have been reported for growing GaN nanostructures directly on Si with high quality which include molecular beam epitaxy (MBE) [18], metal-organic chemical vapor deposition (MOCVD) [19] and hydride vapor phase epitaxy (HVPE) [20]. However, these vapor-phase techniques are too expensive and their growth parameters are quite complicated. In recent years, a nitridation of Ga_2O_3 nanostructures seems to be one of the alternative promising techniques [21] to form GaN nanostructures on Si. A transformation of the grown Ga_2O_3 structures on Si to GaN by a so-called nitridation seems to be a simple method to create a GaN/Si heterostructure. For example, there are several studies reporting the formation of GaN nanostructures by annealing the sputtered Ga_2O_3 layer on metal-coated Si substrates in ammonia [22-25]. Unfortunately, these reports demonstrate that Ga_2O_3 nanostructures cannot be grown without the assistance of metal catalyzers.

Recently, the growth of Ga_2O_3 nanostructures directly on Si without any assistance of metal catalyzer by using a simple electrochemical deposition is reported [26]. This liquid phase technique provides several advantages such as high controllability of thickness and morphologies of Ga_2O_3 nanostructures due to less number of growth parameters. In this research, the formation of GaN nanostructures by ammoniating the electrochemically deposited β-Ga_2O_3 nanostructures on Si substrate was investigated. The effects of the nitridation times and temperatures were also studied.

1.2 Research Motivation

Recently, GaN on silicon carbide (SiC) or sapphire substrates have been widely used for several electronic applications due to specific requirements. However, these substrates are expensive and not available in large wafer size [27]. According to Kukushkin et al., Si substrate seems to be more preferable for the heterostructure growth of GaN due to the availability of Si in large wafer size, the low price of Si and the maturity of Si-based technology [28]. In addition, the integration of GaN based devices on Si platform seems to be very attractive for the hybrid integration towards “More than Moore” technology [3].

However, the integration of GaN nanostructures directly on Si still becoming a challenging issue due to the large lattice mismatch between GaN and Si which is 16 % and thermal mismatch of 54% as reported by Kukushkin *et al* [28]. According to Guha *et al.*, they are the first researchers who developed GaN LED on Si substrate by MBE technique, however, the grown films had several cracks and the fabricated LED had poor efficiency [1]. From this point of view, the major problem is to prevent direct growth of GaN on Si substrate. One of the ways to solve this problem is by introducing a buffer layer such as aluminium nitride (AlN), SiC, silicon nitride (SiN) and etc. on the Si substrate before the growth of GaN to accommodate the lattice mismatch between the Si substrate and GaN [27,28]. Many researchers combined two or three buffer layer in order to improve the quality of the grown GaN which can be considered as a complicated process [29-33]. Generally, depending on the growth technology, the fabrication of GaN can be realized.

A transformation of the grown gallium oxide (Ga_2O_3) structures on Si to GaN by a so-called nitridation seems to be a simple method to create a GaN/Si heterostructure. In this study, Ga_2O_3 nanostructures were deposited on Si substrate before the growth of GaN. Ga_2O_3 seems to be promising materials for chemical synthesis by thermal transformation method due to the relatively low lattice mismatch between Ga_2O_3 and GaN which is only 2.6% [34]. In this work, two-step process of the growth of GaN was conducted, first part of this work is formation of Ga_2O_3 by an electrochemical technique and then followed by nitridation of the electrochemically deposited Ga_2O_3 through the utilization of a so-called ammoniating process. The formation of GaN nanostructures by ammoniating the electrochemically deposited β-Ga_2O_3 on Si substrate was investigated.

1.3 Research Objectives and Scopes

The objectives of this study are as follows:

i. To synthesize Ga_2O_3 nanostructures on Si substrate deposited by electrochemical technique at room temperature and to characterize the deposited nanostructures in term of morphologies and composition properties.

ii. To study the formation of GaN through ammoniating the electrochemically deposited Ga_2O_3 on Si substrate by single zone CVD furnace and to characterize the properties of the ammoniated sample.

This research study involves a two-step process, firstly synthesize the Ga_2O_3 nanostructures by electrochemical technique and, secondly the nitridation of electrochemically deposited Ga_2O_3 to convert Ga_2O_3 into GaN. In electrochemical process, pH value of the electrolyte and Ga_2O_3 concentration is the main parameter to control the morphologies of deposit Ga_2O_3 on Si substrate. Secondly, nitridation processes start with the setting-up of the single zone CVD furnace. Parameters such as nitridation time and temperature were controlled during the nitridation process to study suitable parameter for transformation of Ga_2O_3 into GaN.

1.4 Originality of This Work

There are several studies that have been reported on the growth of GaN nanostructures by nitridation of the deposited Ga_2O_3. But, not much research has been reported on the growth of Ga_2O_3 nanostructures by liquid phase technique as the seed for the nitridation. Examples of the liquid phase technique are hydrothermal and electrochemical technique. These liquid phase technique looked like promising techniques because Ga_2O_3 nanostructures can be directly grown on the substrate without using any catalyzer. Moreover, there are several studies reporting the formation of GaN nanostructures by annealing the sputtered Ga_2O_3 layer on metal-coated Si substrates in ammonia [22-25]. To our knowledge, the nitridation of the electrochemically deposited Ga_2O_3 structures on bare Si substrates to form GaN nanostructures without the assistance of metal catalyzers does not appear in the published literature.

According to Li *et al.*, they successfully synthesized GaN nanorods using a two-step process of deposited gallium oxide hydrate (GaOOH) by the microwave hydrothermal method prior to the nitridation process [35]. They were reported that simple heat treatment of GaOOH under the flow of ammonia (NH_3) gas leads to the formation of GaN nanostructures at temperature as low as 800°C. However, at 800°C, the transformation of Ga_2O_3 to GaN is not complete and it still contains Ga_2O_3 materials in the sample. When the sample was annealed at 900°C and 1000°C, all the diffraction pattern of the XRD can be indexed as hexagonal wurtzite GaN and no peak of Ga_2O_3 was observed.

Another research that has been reported on the growth of Ga_2O_3 nanostructures by liquid phase technique is electrophoresis technique, by Yang Li *et al.* [36]. This technique also involved two step processes for synthesizing GaN which are electrophoresis and ammoniating of Ga_2O_3. In this technique, the deposited Ga_2O_3 was ammoniated in an open tube furnace at a temperature of 950°C for 15 minutes. From the XRD analysis, Ga_2O_3 was successfully transformed into GaN where they found that three strong diffraction peak was observed corresponding to hexagonal wurtzite GaN.

Other than liquid phase technique, most of the researchers deposited Ga_2O_3 by magnetron sputtering prior to the nitridation process. According to Qin Lixia *et al.*, they successfully synthesized GaN nanowire by ammoniating the deposited Ga_2O_3 film using radio frequency (RF) magnetron sputtering on Cobalt (Co) thin films, deposited on a Si substrate [22]. Co thin films were deposited as the buffer layer or catalyzer to assist the growth of Ga_2O_3 on Si substrate. Few studies have been reported on nitridation of Ga_2O_3 to GaN on Si substrate, but most of them using a metal catalyst to assist the growth of GaN prior to the nitridation.

From past studies, only few researches have been reported on the growth of Ga_2O_3 by liquid phase technique. Up to this date, no such similar work is reported where a combination of liquid-phase such as electrochemical technique and vapor-phase methods is utilized to form GaN/Si heterostructure. Therefore, in this work we focus on the formation of GaN nanostructures by ammoniating the electrochemically deposited β-Ga_2O_3 nanostructures on Si substrate.

1.5 Research Activities

The implementation of this study has been summarized into a flowchart as shown in Figure 1.1. This study is focused on the formation of GaN nanostructures by nitridation of the electrochemically deposited Ga_2O_3 nanostructures on Si substrates. This technique involved two step processes, firstly, depositing of Ga_2O_3 nanostructures on Si substrate by the electrochemical technique, and then followed by a nitridation process to convert Ga_2O_3 into GaN.

The formation of Ga_2O_3 nanostructures on Si substrate is carried out by a simple set-up of the two terminal electrochemical deposition cells. Here, the Ga_2O_3 molarities and pH value of the electrolyte were varied. Then the morphological, elemental, crystallinity, composition and photoluminescence of the deposited Ga_2O_3 were characterized by FESEM, EDS, XRD, FTIR and PL respectively. After characterization, the electrochemically grown Ga_2O_3 nanostructures on Si were nitrided in order to transform Ga_2O_3 into GaN. Finally, once again the morphological, elemental and crystallinity of the nitride samples were characterized by FESEM, EDS and XRD respectively to study the effect of ammoniating time and temperature.

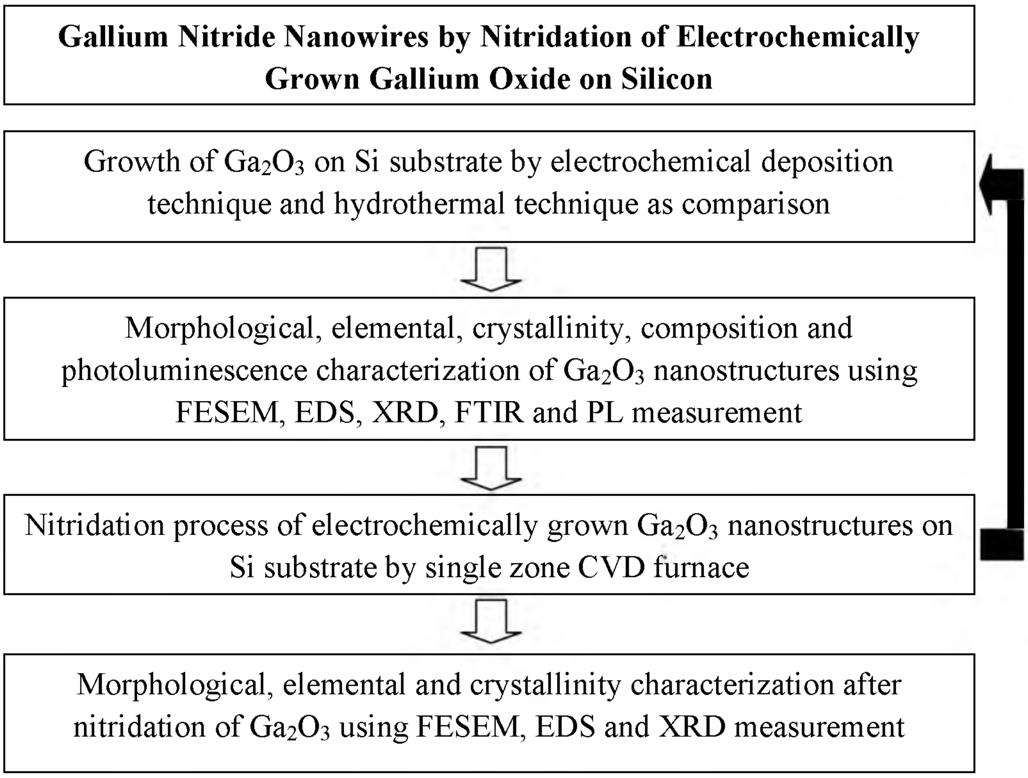

Figure 1.1 Research activities

1.6 Overview of Thesis Organization

This thesis is organized into 6 chapters. Chapter 1 gives an overview of the research background and motivation of the study. The originality, objectives, scopes and research activities of the present work are also presented.

In chapter 2, an overview of the basic properties of Ga_2O_3 and also GaN is provided. This chapter discusses a brief description of the method used to grow GaN and also nitridation of Ga_2O_3 from past work. The potential application of Ga_2O_3 and GaN are also discussed in this chapter.

In chapter 3, the experimental procedures and characterization techniques that have been used in this work to synthesize and characterize Ga_2O_3 nanostructures by electrochemical technique are described. The nitridation process of Ga_2O_3 to transform Ga_2O_3 into GaN by CVD furnace is also described.

In chapter 4, the properties of the synthesized Ga_2O_3 by electrochemical deposition technique at different Ga_2O_3 molarities and pH value of the electrolyte are discussed. A basic study of the formation of Ga_2O_3 as well as investigation on the surface morphology, elemental, crystallinity, composition and photoluminescence is performed in order to have an understanding of the formation of Ga_2O_3 nanostructures on Si substrate. The properties of the grown Ga_2O_3 by hydrothermal technique are compared. The possible growth mechanism and chemical reaction are discussed in details.

In chapter 5, the properties of the synthesized GaN through nitridation of Ga_2O_3 by a single zone CVD furnace is discussed. The nitridation time and temperatures are varied and the effect of the nitridation time and temperature are analyzed. The morphologies, elemental, and crystalline properties of the structures before and after nitridation are discussed in details. The possible growth mechanism and chemical reaction involved in this study are also explained in this chapter.

Finally, chapter 6 concludes the contributions of the present work and discusses future research direction.

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