Studies on plasma assisted molecular beam epitaxial growth of GaN-based multilayer heterostructures on Si for photodetector application

Zheng, Yi

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**Studies on Plasma Assisted Molecular Beam Epitaxial
Growth of GaN-Based Multilayer Heterostructures on
Si for Photodetector Application**

Zheng Yi

School of Electrical & Electronic Engineering

A thesis submitted to the Nanyang Technological University
in partial fulfillment of the requirement for the degree of
Doctor of Philosophy

2019

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarized materials, and has not been submitted for a higher degree to any other University or Institution.

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[Zheng Yi]

Supervisor Declaration Statement

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.....
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.....
[K Radhakrishnan]

Authorship Attribution Statement

This thesis contains material from 2 papers published in the following peer-reviewed journals where I was the author and co-author.

Chapter 4 is published as Y. Zheng, M. Agrawal, S. Patwal, N. Dharmarasu and K. Radhakrishnan, A study on Ga-Si interdiffusion during (Al)GaN/AlN growth on Si by plasma assisted molecular beam epitaxy, *Applied Surface Science*, 481 (2019) 319-326.

The contributions of the co-authors are as follows:

- Prof Radhakrishnan provided the initial project direction and edited the manuscript drafts.
- I conducted the characterization and prepared the manuscript drafts.
- I co-designed the study with Dr M. Agrawal and Prof K. Radhakrishnan and performed all the growth related laboratory work at the School of Electrical and Electronic Science and Engineering. I also analyzed the data.
- Mr Patwal helped in microscopy, AFM, XRD, SEM analysis including sample preparation, in the School of Electrical and Electronic Science and Engineering.
- Dr M. Agrawal assisted in the collection and analyzation of the characterization data.
- Dr Dharmarasu helped in data analysis and amending manuscript drafts.
- Prof K. Radhakrishnan provided guidance in the growth and manuscript drafts.

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The contributions of the co-authors are as follows:

- Prof G. Bahir and Prof M. Orenstein provided the initial project direction and edited the manuscript drafts.
- Mr. B. Dror fabricated the device and conducted the device characterization and prepared the manuscript drafts.
- I co-designed the study with Dr M. Agrawal and Prof K. Radhakrishnan and performed all the growth related laboratory work at the School of Electrical and Electronic Science and Engineering. I also analyzed the data.
- All microscopy, AFM, XRD, SEM including sample preparation, was conducted by me in the School of Electrical and Electronic Science and Engineering.
- Dr M. Agrawal assisted in the collection and analyzation of the characterization data.
- Prof K. Radhakrishnan provided guidance in the device structure growth and manuscript drafts in growth part.

28/2/2019

.....
Date

ZHENG YI

.....
[Zheng Yi]

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Summary

III-Nitride materials have gathered enormous attention and undergone fast development, due to superior properties such as wide band gap, high stability, high electron motilities, high break down voltage and sensitivity to ultraviolet light. GaN ultraviolet (UV) detector can be used in fire detecting, missile's seeker, combustion process monitoring and other monitoring systems, which require UV detection in harsh environments. Tunability of band gap and the possibility of formation of heterostructures with group III-nitride materials allow the design of complex structures to detect both UV and infra-red (IR) spectral regimes (multi band) on a single chip. By growing AlGaIn/GaN multiple quantum well layers, IR detector can also be obtained, which means IR and UV detector can be achieved using the same material.

GaN based UV/IR dual band photodetectors can be grown on sapphire, Si or SiC substrate, but sapphire and SiC substrates are more expensive than Si. Moreover, development of such a technology on Si allows lower cost and opens the prospect of integration of III-nitride with Si technology. UV/IR dual band detectors grown on sapphire substrates without any optical filter has also been reported. However, growth of such photodetectors on Si (111) substrate is challenging due to large difference in thermal and lattice mismatch between Si substrate and GaN, which leads to built-in strain and cracking. In this work, plasma assisted molecular beam epitaxy (PA-MBE) is used to grow dual band photodetector heterostructures on Si substrate as well as MOCVD grown AlN/Si templates.

In this thesis, prior to the growth of complex multiple layer photodetector structures, GaN and AlGaIn growth on Si substrate as well as AlN/Si template are optimized. The effect of the AlN layer on the GaN surface morphology and crystal quality is studied. AlN layer failed to block Ga diffusion because of columnar structure or grain boundary formed, due to N-rich AlN growth in

MBE, which could act as a path for Ga diffusion into Si substrate and dissolve in Si forming Ga-Si alloy. The Ga-Si alloy would float on Ga droplets during the growth and end up in microcrystal. Separate two-step growth of AlN and GaN together makes GaN surface smooth without microcrystals. Smooth GaN/AlN grown on Si (111) by PA-MBE without microcrystals is achieved with the RMS roughness ~ 0.4 nm. Si doping concentration in GaN is found non-linear to Si cell temperature at higher doping levels.

Prior to AlN buffer layer growth, amorphous SiN_x formed on Si surface can also be used as buffer layer to achieve smooth GaN on Si substrate. Importantly, it has been confirmed that SiN_x layer acts as a blocking layer for Ga/Si diffusion across the AlN layer. Based on GaN growth, AlGaN growth in Ga-rich condition was optimized in a similar way. AlGaN layer with 30% Al-composition optimized on Si (111) substrate, AlN template deposited in-house by MOCVD and commercial AlN template showed comparable surface morphology. Si doping studies of AlGaN with Al-composition of 30% are conducted on both 4-inch Si (111) substrate and MOCVD grown AlN/Si template. The highest Si doping of $2 \times 10^{20} \text{ cm}^{-3}$ is achieved with RMS surface roughness less than 1 nm. Crack-free wafers with total GaN or AlGaN epilayer thickness up to $1.4 \mu\text{m}$ have been achieved.

Multiple layer structures for UV photodetector application are successfully grown on Si substrate and AlN/Si template. Different mesas are designed for the UV photodetectors to study the effect of device dimensions on photoresponsivity. Low dark current density in the order of 10^3 A/m^2 is observed. Photocurrent measurements under different applied bias (1 to 3 V) and wavelength of light are performed. Square photodetector showed high responsivity of 1 A/W at 3 V bias while the interdigitated detector with similar sensing area showed even higher responsivity of 5.7 A/W. In the interdigitated design, larger detecting area and shorter lateral electrodes-gap

help to improve the photoresponsivity.

AlGaIn/GaN based multiple layer structure for UV/IR dual band photodetector application is successfully grown on AlN/Si template. Interdigitated photodetector fabricated on epilayer grown on AlN/Si template show a low dark current density of 7×10^{-3} A/m² at 15 V applied bias in room temperature. Clear UV response of 0.01 A/W from the fabricated photodetectors has been demonstrated. The near-IR optical photoresponse observed is at 4.52 μ m up to about 50 K.

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List of Abbreviations

Abbreviation	Full Expression
AC	Alternating Current
AES	Auger electron spectra
AFM	Atomic Force Microscopy
DC	Direct Current
EDX	Energy Dispersive X-ray
FFT	Fast Fourier Transform
FWHM	Full-Width-Half-Maximum
HEIWIP	Heterojunction Interfacial Workfunction Internal Photoemission
HEMT	High-Electron-Mobility Transistor
HR-SEM	High Resolution Scanning Electron Microscopy
HR-TEM	High Resolution Transmission Electron Microscopy
HVPE	Hydride Vapor Phase Epitaxy
I-V	Current-Voltage
ICP-RIE	Inductively Coupled Plasma Reactive Ion Etching
IR	Infrared
LED	Light Emitting Diode
MBE	Molecular Beam Epitaxy
MOCVD	Metal Organic Chemical Vapor Deposition
MOVPE	Metal Organic Vapor-Phase Epitaxy
MSM	Metal-Semiconductor-Metal
PAC	Photo-Assisted Cryogenic
PA-MBE	Plasma Assisted Molecular Beam Epitaxy
PEC	Photo-Enhanced Electrochemical
PECVD	Plasma-Enhanced Chemical Vapor Deposition
PL	Photoluminescence
PMT	Photomultiplier Tube

RF	Radiofrequency
RHEED	Reflection High-Energy Electron Diffraction
RTP	Rapid Thermal Processing
RMS	Root Mean Square
RIE	Reactive Ion Etching
SAED	Selected Area Electron Diffraction
SEM	Scanning Electron Microscopy
SL	Super Lattice
SOI	Silicon on Insulator
TD	Threading Dislocation
TDD	Threading Dislocation Density
TEM	Transmission Electron Microscopy
UV	Ultraviolet
XRD	X-ray Diffraction
2DEG	Two Dimensional Electron Gas

Chapter 1 Introduction

GaN-based semiconductors are gaining varied interest due to their superior properties for wide applications including optical and electronic devices such as photodetector, light emitting diode (LED) and high electron mobility transistor (HEMT) [1]. Development of GaN technology on silicon allows larger size and lower substrate cost than other substrates such as silicon carbide (SiC) and sapphire. Moreover, it opens up the prospect of integration of III-nitride technology with the existing and matured Si technology. GaN and AlGa_N have wide energy bandgap and high breakdown voltage which are favorable for ultraviolet (UV) photodetector applications even in harsh environment. By tuning the bandgap offset of GaN and AlGa_N, AlGa_N/GaN based multilayer structure that are capable of photodetection in infrared (IR) range can also be realized. There are several challenges to overcome in the development of GaN-based materials on Si that include large lattice and thermal mismatch between GaN-alloys and Si, melt-back etching and interdiffusion of Si into GaN crystal and that of Ga into silicon substrate [2-4]. Thus, understanding the materials properties, growth mechanism and suitable device fabrication-processes are important.

1.1 GaN material properties

GaN and their alloys crystallize mainly in two forms of crystal structure, wurtzite and zinc blende. The wurtzite structure shown in Fig. 1.1 [5] is found to be more stable than the zinc blende structure under ambient conditions. This crystal structure is non-centrosymmetric in nature, and hence associated with properties of piezoelectric and spontaneous polarization. The polarization effect plays a large role in electronic devices such as high electron mobility transistor (HEMT), for example, in the control and formation of 2DEG (two-dimensional electron gas) at the

heterojunction interface between AlGaN and GaN. However, for photodetector applications, the bandgap energy of the nitride materials involved are more important, and hence the composition control is critical for device application in the desired wavelength.

Figure 1.1 Stick and Ball structure of GaN (wurtzite)

The physical properties of GaN are listed Table 1.1 [6,7]. As the energy gap of GaN (3.42 eV) lies in UV range, GaN UV detector can be fabricated based on the interband transition. Moreover, quantum well structures of AlGaN/GaN/AlGaN can be designed, and the intraband transition can also be used for IR detection. By modifying the Al-composition in $Al_xGa_{1-x}N$, the intraband transition energy can be controlled in a specific range. When the detector structure is properly designed, a dual band detector can be realized.

The binding energy of GaN is 2.22 eV, which makes GaN very stable, and it cannot be etched by most chemicals. As the traditional wet etching is not suitable for the device fabrication, the

alternative solution is to employ dry etching, either by Reactive Ion Etching (RIE) technique or Inductively Coupled Plasma RIE (ICP-RIE).

Table 1.1 Physical properties of GaN (Wurtzite)

Property	Value
Lattice constant (Å)	a = 3.189, c = 5.185
Binding energy (eV)	2.22
Melting point (°C)	2500
Linear expansion coefficients ($\times 10^{-6}$) (K⁻¹)	a: 5.59, c: 3.17
Static dielectric constant	8.9
Refractive index	2.85
Energy gap (eV)	3.42
Intrinsic carrier concentration (cm⁻³)	$\sim 10^{16}$
Electron effective mass	0.2m ₀
Hole effective mass	0.8m ₀
Electron mobility (300K) (cm²V⁻¹s⁻¹)	1245
Hole mobility (300K) (cm²V⁻¹s⁻¹)	< 20

During the growth, the nitrogen vacancies will act as dominant native defects, the same as donors. That's the reason why GaN is always unintentionally doped into *n*-type, and the carrier concentration is around 10^{16} cm^{-3} . GaN can also be intentionally doped into *n*-type. In GaN growth by molecular beam epitaxy (MBE) technique, Si and Mg are generally used as *n*-type and *p*-type dopants, respectively. However, the electron mobility is much higher in GaN than hole mobility; hence, for active layer or contact used for photodetector described in this thesis, *n*-GaN is preferred for better device performance.

1.2 Substrates for GaN epitaxial growth

The bulk crystal growth of GaN requires very high temperature and pressure, and a number of groups have been working on this problem worldwide for many years. Although large efforts have been made, realization of cost-effective, large sized and high quality native GaN substrates is still found to be difficult. Generally, epitaxial growth is used to obtain high quality GaN-based layers on foreign substrates such as sapphire [8], SiC [9] and Si [10]. Although the techniques of epitaxial growth of GaN on sapphire substrates is matured and the thermal conductivity of SiC substrate is much better than Si, Si still has its own superiority [11].

Si substrate is much cheaper and large diameter Si substrates are also available. The quality control on Si is matured and the control over surface polishing and wafer curvature is better. Si (100) substrate with four fold symmetry is a favorable substrate for common III-V epitaxial growth due to the wide usage in silicon technology. However, due to the six fold symmetry property in GaN, Si (111) is the preferred and more commonly used substrate for III-nitrides epitaxial growth. The four fold symmetry is more suitable for the growth of metastable cubic phase nitrides while the six fold symmetry assists in the growth of thermodynamically stable wurtzite phase. The growth of GaN on Si (100) substrate shows the epitaxial relationship of GaN [0001] $\parallel$ Si [001] and GaN $[11\bar{2}0] \parallel$ Si $[110]$ while GaN on Si (111) oriented as GaN [0001] $\parallel$ Si $[111]$, GaN $[2\bar{1}\bar{1}0] \parallel$ Si $[\bar{1}\bar{1}0]$ and GaN $[01\bar{1}\bar{0}] \parallel$ Si $[\bar{2}\bar{1}1]$ [5]. Si (111) substrate is used for the epitaxial growth GaN-based nitrides to get the stable wurtzite structure. However, the lattice mismatch between Si (111) substrate and GaN is around 17% and the thermal mismatch is nearly 54%, which makes it difficult to grow GaN on Si (111) substrate directly. A comparison of different substrates used for GaN-growth is summarized in Table 1.2 [5].

Table 1.2 Comparison of different substrates for GaN-growth

Properties	Sapphire	6H-SiC	Si (111)	GaN
Lattice constant (Å)	4.785	3.081	3.846	3.189
Lattice mismatch (%)	15	3	17	
Thermal expansion coefficient (10^{-6}K^{-1})	7.3	4.5	2.6	5.6
Thermal mismatch (%)	-30	20	54	
Thermal conductivity ($\text{Wcm}^{-1}\text{K}^{-1}$)	0.5	4.5	1.5	1.3
Wafer size (inch)	2-8	2-6	2-12	2
Cost	moderate	high	low	Extremely high

1.3 AlGaIn based heterostructure for photodetector application

GaN and AlGaIn based structures can be used to fabricate UV detector as the energy bandgap is wide and the breakdown voltage is high. UV/IR dual-band detectors can be potentially applied in various applications such as fire/flame detection, surveillance and tracking of missiles, and combustion process monitoring.

Fig 1.2 (a) and (b) [144] show the cross section and the band diagram, respectively, of the basic AlGaIn/GaN UV photodetector. The UV detector employs a Heterojunction Interfacial Work function Internal Photoemission (HEIWIP) structure, where the top and bottom *n*-GaN layers work as emitter/absorber and the free carrier absorption is used for the light detection. The interband transition is caused by the electron excitation from valence band (VB) to conduction band (CB). For interband transition, the bandgap for both GaN and AlGaIn are wide and lie in the UV range. Thus, visible light won't affect the responsivity as the energy is lower than their energy bandgap. The Al-composition can be modified to get a range of energy bandgaps, which corresponds to

different UV wavelengths. In addition to the interband transition, intraband transition also exists in the structure. Due to the bandgap offset of GaN and AlGa_N, the electrons in the CB can be excited and then collected by the *n*-GaN layer in the presence of electric field. However, the interband transition will also occur when the device is exposed to UV or visible light, and finally the signals will be mixed and collected at the absorber. For this simple structure, if IR responsivity needs to be measured, a suitable filter can be used in order to eliminate the light in the visible and UV ranges. For UV responsivity measurement, the photocurrent generated by UV light is much stronger and the signal can be easily distinguished.

Figure 1.2 (a) Schematic of AlGa_N/Ga_N HEIWIP structure and (b) band diagram under electric field

1.4 Challenges for Ga_N and AlGa_N growth on Si (111)

Realization of Ga_N-based photodetector structure requires multilayer growth. However, when the structure is grown on Si (111), due to high lattice and thermal mismatch, the resulting structure will exhibit low crystal quality, high dislocation density, strain in the material and poor surface morphology. AlGa_N based IR photodetector contains multiple quantum well structures to achieve intraband transition, and photodetector responsivity would decrease with increasing dislocation

density [12]. With high tensile strain in the material after the growth, epi-wafers would tend to bow and may even crack, particularly larger diameter wafers [13,14].

Therefore, large efforts have been made in the past to investigate AlGaIn and GaN growth on Si using both metalorganic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) techniques, which will be discussed in Chapter 2. While MOCVD growth provides GaN and AlGaIn with smooth surface morphology and high growth rate, MBE offers in-situ growth monitoring capability and provides sharp AlGaIn/GaN interface. Moreover, MBE is capable to achieve well controlled GaN and AlGaIn thickness up to monolayer level and carbon free growth condition, which enables better device performance. Thus, the main goal in this thesis is to optimize GaN and AlGaIn epilayer growth, followed by more challenging growth of AlGaIn/GaN based multiple layer structure growth on Si (111) substrates by MBE technique and device fabrication and demonstration of photodetectors for UV and IR applications.

1.5 Motivation and Objectives

The primary objective of this study is to develop AlGaIn/GaN photodetector structure on Si (111) substrates using MBE growth technique, which includes

1. Optimization of the surface morphology, *n*-type doping and crystal quality of GaN grown on Si (111), and investigation of the mechanism behind the rough GaN surface morphology with dense microcrystals.
2. Optimization of the surface morphology, *n*-type doping and crystal quality of AlGaIn and AlGaIn/GaN multiple layer structure on Si (111) for UV detector.
3. Optimization of the UV detector device structure to improve the responsivity performance.

4. Optimization of the AlGaN/GaN multiple quantum well structure grown on Si (111) substrate for UV/IR detector.
5. Investigation of the photoresponsivity in UV and IR region for the multilayer structure.

1.6 Thesis Organization

This thesis mainly focuses on the growth optimization of GaN, AlGaN/GaN, and multilayer GaN-based structures including superlattices on Si (111) substrate by MBE technique. Optimized layer structures have been used of device fabrication and demonstration of photodetectors operating in UV/ IR region.

Chapter 1 gives background of GaN material properties and epitaxial substrates. Epitaxial growth challenges and objectives of the research project are discussed in this thesis. A review of the GaN and AlGaN growth optimization to achieve smooth surface and high crystal quality is given in Chapter 2. GaN and AlGaN based UV and UV/IR dual band photodetectors with different structures and designs are also discussed in this Chapter.

Chapter 3 gives an overview of the growth, device fabrication and characterization techniques for photodetector application. GaN growth optimization on Si is discussed in Chapter 4. Two-step AlN growth together with 3D to 2D GaN growth is shown to achieve smooth GaN on Si. AlN layer has been studied to understand the GaN-microcrystals formation. Si doping in GaN has also been investigated and analysed.

The function of SiN_x layer in GaN and AlGaN growth to block Ga/Si interdiffusion is studied in Chapter 5. AlGaN growth optimization on Si with the help of amorphous SiN_x as well as AlN/Si

template is also demonstrated. Further, the effect of Si doping in AlGa_N is also investigated. Chapter 6 presents optimization of GaN/AlGa_N multilayer structure-based UV photodetector growth. The device fabrication process is discussed in detail, photodetectors with different designs have been studied to achieve high responsivity.

Chapter 7 presents GaN/AlGa_N multilayer structure-based UV/IR dual band photodetector growth optimization. Results on the UV photoresponsivity and near-IR optical absorption have been discussed in this chapter. Chapter 8 summarizes the results accomplished in this thesis and suggests possible further works to optimize device layer structure growth on Si and improve the device performance.

Chapter 2 Literature Review

2.1 AlGaN and GaN growth optimization

MBE technique has been used to grow III-V materials for more than 20 years. Both MBE [15-19] and MOCVD [20-24] can provide good quality epitaxy layers. While MOCVD provides faster deposition rate, MBE can provide sharper interface. Although MBE growth can be precisely controlled, the growth window for GaN based layers is much narrower than MOCVD. As the MBE growth takes place in an ultrahigh vacuum system, the contamination is quite low, and the growth temperature employed is also lower than MOCVD. In the plasma assisted MBE (PA-MBE), nitrogen species are provided by a plasma source using RF activated N_2 gas. Al and Ga are evaporated from the crucibles and the flux exposure is controlled by the respective shutters. The Al, Ga and nitrogen plasma reach the wafer surface and react on the substrate heated to certain temperature. Substrate heater temperature control is important as the temperature will affect the surface adhesion and atomic mobility on the growth surface. III-V ratio will determine the growth conditions and affect the epilayer quality GaN and AlGaN. There are four different growth regimes in MBE growth, defined based on Ga flux, J_{Ga} and N flux, J_N : 1) N-rich growth regime ($J_{Ga}/J_N < 1$), 2) Stoichiometric growth regime ($J_{Ga}/J_N \sim 1$), 3) Ga-rich growth regime ($J_{Ga}/J_N > 1$) and 4) Ga-droplet growth regime ($J_{Ga}/J_N \gg 1$) [25].

For N-rich growth condition, the nitrogen flux is much higher than the Ga flux, and the Ga diffusion rate is limited on the surface. In this condition, the diffusion length is shorter resulting in many stacking faults, and hence the surface becomes granular. In the stoichiometric growth regime, V-shape defects are generated resulting in pits with inverted pyramid type surface. Smooth surface is obtained in the Ga-rich growth regime which enables the growth of 2D layers of GaN and AlGaN.

The Ga atoms are abundant in this regime and so is the surface mobility of Ga [26]. AlGaIn/GaN is deposited layer by layer and the surface is smooth with lower stacking faults. Furthermore, growth under these conditions produces characteristic spiral growth hillocks around mixed- and screw- type dislocations [27]. Under this growth condition, there are still some pits but the surface is smoother than the epilayer grown under stoichiometric growth regime. The ideal growth condition corresponds to 2 ML of Ga metal maintained on the surface during the growth in order to obtain smooth surface [28]. When the Ga flux is further increased, the growth regime lies in the Ga-rich condition wherein Ga will form droplets on the surface. The surface grown in this regime should be smoother and spiral hillocks with atomic steps can be still seen. The Ga droplets can be etched by HCl process. However, if the Ga flux is too high, the surface will show some shallow pits where the droplets were removed after HCl cleaning. Thus, the optimized growth condition lies in Ga-rich regime. For different nitrogen species, the deposition rate is also different, and the growth temperature also affects the growth rate.

As described by Vanmil et al. [29], the GaN decomposition occurs when the growth temperature is higher than 750 °C. When using metastable N₂ molecular species, the thermal decomposition rate decreases a lot. Hence, the normal substrate temperature for GaN growth is between 700 °C and 750 °C when the RF plasma produces high density metastable molecular species [30]. Growth mechanisms such as Ga metal recovery can be monitored online by recording the RHEED pattern intensity. During the recovery, the RHEED intensity will increase and saturate when the residual Ga on the surface is totally desorbed. Thus, RHEED is used to confirm and maintain the Ga bilayer to ensure good crystal quality of the epitaxy layer.

2.1.1 AlN growth on Si (111)

The photodetector structure is grown on Si (111) substrate. However, due to the severe Ga/Si interdiffusion during GaN on Si growth [31], AlN together with SiN_x layer [32,33] are commonly used as buffer layer to stop Ga/Si interdiffusion and achieve smooth and high quality GaN [34-38].

AlN growth is important as it will affect the quality of the following epitaxial layer growth on it [8]. The growth conditions such as III/V ratio, growth temperature etc. are critical as AlN is highly lattice mismatched to Si (111) substrate (~19%). The normal AlN growth temperature is between 700 °C and 930 °C [28, 34, 39,40]. During the growth, the substrate temperature is kept high to get better adhesion of Al on the Si surface. However, Al and Si may diffuse into each other [33]. While low growth temperature of AlN will degrade the layer quality making it rough and inhomogeneous due to Al condensation, high growth temperature will help to reduce the roughness and make the layer homogeneous [41,42].

Prior to the growth of AlN, Si wafer is degassed in MBE growth chamber at high temperature to remove the native oxide. During the AlN deposition, nitrogen plasma reacts with the Si surface and forms SiN_x, which consequently affects the crystalline quality of AlN. To avoid the formation of SiN_x, Al is evaporated at high flux without nitrogen plasma that makes the growth surface metal-rich and inhibit the formation of the SiN_x [39, 4143-49]. Nevertheless, some groups still continue with the surface nitridation since there is no way to totally eliminate the nitridation effect [34-36, 50-53]. Also, Si diffusion from the substrate may cause the AlN and GaN layer to be slightly doped and lead to high leakage current. On the other hand, Al diffusion into the substrate may generate a *p*-type inversion layer at the AlN/Si interface. What is worse is that Al/Si interdiffusion may transfer Si from the substrate on to AlN surface if AlN growth is performed in Al-rich condition.

This will further degrade the following GaN layer grown on AlN. To avoid these complications, AlN on Si is only grown in nitrogen-rich condition at the initial stage [30,54].

Both amorphous SiN_x [31, 51,55] and β -Si₃N₄ [29, 34, 56] have been studied as a buffer layer before AlN growth. It was found that with β -Si₃N₄ can be achieved by striking nitrogen plasma with source shutter closed at a high temperature of 830 °C - 900 °C [50], and high quality AlN grown on β -Si₃N₄ with low surface roughness has been obtained [31, 57].

Although it is reported that GaN exhibiting good TEM and PL features grown on Si (111) was achieved [58], the high thermal mismatch ($\alpha_{\text{GaN}} - \alpha_{\text{Si}} = 2.9 \times 10^{-6} \text{K}^{-1}$) and lattice mismatch (~17%) between the Si substrate and GaN will result in cracks appearing after the growth when the temperature is reduced to room temperature, caused by highly tensile strained epilayer. This is the main challenge that needs to be solved for GaN growth on Si (111) substrate, particularly for larger diameter substrates. To avoid the cracks, stress mitigating layers such as SOI substrate, AlN interlayer, AlGaIn interlayer and AlGaIn/GaN superlattice (SL) structure have been introduced for the growth of GaN on Si (111) substrates [59-62].

AlGaIn/GaN superlattice layers were used by Nikishin et al. [31, 35] for the growth of GaN on 2 inch and 3 inch Si (111) substrates. In that study, two sets of AlGaIn/GaN superlattice layers were used, one set was grown on AlN buffer layer directly and the other was grown on 400 nm GaN on AlN buffer layer. It was observed that the defect propagation from the AlN/Si interface to the surface was reduced and the crystal quality improved for the set grown using superlattice layers on AlN buffer layer. It is reported by Semond et al. [34] that good GaN quality can be obtained with the AlN thickness kept higher than 120 nm when GaN is grown using ammonia MBE. To

achieve good AlN on Si layer, the Al/N ratio is critical [39,63] and grading of AlN is also found to be important to achieve better AlN quality [64].

2.1.2 GaN growth on Si (111)

Once the AlN layer growth on Si is optimized, GaN and AlGaN layers can be subsequently grown. During the growth, RHEED can be used to distinguish the polarity of GaN that can be N-polar or Ga-polar. It is particularly important to control the polarity of GaN for HEMT structures [65] in the case of electronic devices. However, the performance of a UV/IR detector depends mainly on the energy band gap, the epitaxial layer quality and surface morphology. Similar to the AlN growth optimization, great works have been done in 1980s for GaN growth using MBE [66-71]. Recently, many research studies have also been reported for GaN growth optimization on Si (111) substrate [72-76]. Epitaxial and monopolar GaN is successfully grown directly on Si (111) by PA-MBE upon nitridation of the substrate and selective N-polar nucleation under N-rich condition [77].

It is reported by Yoshizawa et al. [78] that 3D grown GaN is completely relaxed from thermal and lattice mismatch while keeping good optical quality. When the growth of GaN is done in Ga-rich condition, the growth surface becomes more compact. To obtain better surface roughness and epitaxy layer quality, the GaN growth is performed in two-steps. The growth of GaN is initiated using 3D growth mode followed by 2D growth mode in order to improve the crystal quality. The 3D grown GaN will stop the propagation of the defects on the AlN/Si surface and the 2D GaN assists the lateral growth [79]. It is also reported that initial GaN growth on AlN must be kept under N-rich condition to avoid formation of GaN microcrystals on the surface followed by GaN growth in Ga-rich condition [80].

2.1.3 $\text{Al}_x\text{Ga}_{1-x}\text{N}$ growth on Si (111)

$\text{Al}_x\text{Ga}_{1-x}\text{N}$ growth on Si (111) has been studied by many groups using MBE with either ammonia or plasma activated molecular nitrogen as group-V source. $\text{Al}_x\text{Ga}_{1-x}\text{N}$ growth using metal organic vapor-phase epitaxy (MOVPE) on GaN epilayer has also been studied. A low-temperature $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layer is grown first followed by a high temperature layer that resulted in a 0.9 μm thick crack-free $\text{Al}_{0.2}\text{Ga}_{0.8}\text{N}$ [81]. In ammonia MBE growth, the Al incorporation is a nonlinear function of the Al/Ga flux ratio. It is reported in the case of PA-MBE that when the growth temperature is kept between 750 °C – 800 °C, Al incorporates with unity incorporation probability in both the group-III and N-rich growth regimes [82]. Incorporation of Ga is controlled solely by the available nitrogen flux that is not consumed by Al. Al content estimated from Auger electron spectra (AES) is found to have a linear relation against Al/Ga flux intensity ratios at a constant substrate temperature. Al-composition increases with the increasing substrate temperature under the same Al/Ga flux ratio [83]. AlGaN layers grown on GaN templates are normally under tensile stress due to the lattice mismatch. Various experiments have led to discrepancy in determining the value of critical thickness as a function of Al-composition. The critical thickness for cracking is reported to be between 133 and 200 nm for $\text{Al}_{0.2}\text{Ga}_{0.8}\text{N}$ on GaN films, whereas $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$ on GaN layer presents a thinner critical thickness, between 66 and 100 nm [84].

To achieve good quality AlGaN with smooth surface, RHEED was used as an effective monitoring method to study the 2D growth [85-87]. Since Al-composition is affected by Al/Ga ratio during the growth, grading AlGaN layer similar to GaN grading from 3D to 2D growth cannot be used. Another method of growing AlGaN with Ga deposited on the growth surface under precise control has been developed. By controlling the ratio of the growth time and Ga desorption time, the

residual Ga on the growth surface can be just ‘used up’ to form AlGa_N without affecting Al-composition and Ga accumulation [88-94].

2.2 Si doping in GaN and AlGa_N

Several works have been reported to investigate the effect of Si doping in GaN and AlGa_N [95-99]. Mg and Be have been used for *p*-type doping [100], which is difficult [101]. The *n*-type doping in GaN and AlGa_N can be achieved by using different sources in MBE system. Silane (SiH₄) [102] has been used as gas-dopant while Si and Ge have been used as solid-dopant for *n*-type doping [103]. Efficient Si doping and low resistive Al_{*x*}Ga_{1-*x*}N layers were obtained for *x* < 0.79 whereas an exponential drop in carrier concentration was observed for *x* > 0.84 [104]. With higher Al-composition (Al_{0.7}Ga_{0.3}N), dopant compensation was also greatly reduced with reduced growth temperature [105]. It was found that Si doping would increase the surface roughness of GaN and AlGa_N at high doping level as well as produce high tensile strain [75, 76, 106]. On the other hand, Ge doping had no impact on layer stress, and smooth GaN surface was achieved with carrier concentrations up to $2.9 \times 10^{20} \text{ cm}^{-3}$ [76]. GaN and AlGa_N Raman spectra have been studied [107-112], and it was confirmed that Si-doping leads to relaxation of compressive strain in the layer. Similar results have been reported for AlN, GaN, and AlGa_N [113-116]. High Si doping in GaN helps to achieve ultra-low ohmic contact [117]. GaN/AlGa_N based multiple quantum well (MQW) structure doped with Si in GaN QW has been grown by MBE. It was found that optical properties of the Si doped QWs get improved compared with nominally undoped QWs, and it has been shown that Si dopants decrease the dislocation density and hence improve the crystalline quality of GaN layers [118-121,42]. Inter subband absorption is also affected by Si doping, lower doping level would lead to a significant reduction of the absorption line width [122]. Ge doping has a similar effect as Si doping on the dislocation inclination in *n*-type GaN [123]. Electrical and optical

properties of the Si-doped $n\text{-Al}_{0.28}\text{Ga}_{0.72}\text{N}$ epitaxial films were also affected by AlN buffer layer [124] while optical absorption in GaN and AlGaN is also reported to be affected by Si doping [125,126].

2.3 AlGaN and GaN based UV photodetector

Commercial UV detectors can be divided into two types: photomultiplier tube (PMT) and semiconductor detector. Although PMT has high sensitivity, it is large in size and requires high voltage and light filter during operation, which makes it inconvenient. However, semiconductor UV detector is smaller in size, tougher and also provides higher quantum efficiency and stability. Semiconductor UV detectors can also be divided into two types based on the material used, namely, wide bandgap and narrow bandgap detector. Si photodiode is a narrow bandgap device, and by enhancing the UV responsivity, it can be used as a UV detector [127]. However, as the bandgap is narrow, the Si UV detector is sensitive to visible light and it can't be exposed to UV light for a long time as the reliability will degrade. Different types of UV detectors have been published as detailed in the following sections.

2.3.1 Junction based UV photodetector

GaN and AlGaN based $p\text{-}n$ [128-130] or $p\text{-}i\text{-}n$ [131-141] type UV photodetectors have been widely studied, and they are capable for photodetector array integration, although not favorable for UV/IR dual band photodetection. The detection principle of this type of photodetectors is simple and solar blind UV photodetection can be easily achieved by tuning the Al-composition. Schottky junction can be formed between metal and AlGaN which can be used for UV detection as well [142-145]. Similar to $p\text{-}n$ or $p\text{-}i\text{-}n$ junction type photodetector, Schottky junction photodetector can be tuned to desired detection wavelength by tuning Al-composition and hence the Schottky barrier energy.

Based on the simple AlGaIn/GaN and metal Schottky contact, advanced metal-semiconductor-metal detector can be fabricated.

2.3.2 Metal-semiconductor-metal ultraviolet detector

Immense works have been done on Metal-Semiconductor-Metal structure on GaN epilayers [146-150] for UV detection. As shown in Fig. 2.1, the two metal electrodes are deposited on the surface of the GaN layer [151]. For MSM UV detector, two Schottky contacts are deposited on GaN forming two back-to-back series connected Schottky diodes. MSM UV detector is easy to fabricate and it is only capable for single wavelength detection [152]. The UV detection performance strongly depends on the epilayer quality [153,154], and threading dislocations are found to affect the responsivity. While the screw dislocations cause a sharp increase in responsivity, the edge dislocations are found to reduce the responsivity. To achieve high photoresponsivity, the GaN epilayer quality needs to be well controlled. Moreover, surface passivation also affects the device performance. SiO₂ can be deposited on the electrodes to passivate the surface. It is reported that density of electrodes used can affect the responsivity [155] and presence of nanoparticles on the

Figure 2.1 Top view of Ni/Au Schottky contact (yellow) GaN MSM device showing interdigitated geometry. Electrode finger length is 200 μm , width is 5 μm , and space between the electrodes is 10 μm

surface increases the photoresponsivity [156]. Photodetector responding to various wavelengths can be achieved by tuning Al-composition [157,158].

The dimension of the electrode is found to affect the responsivity at high frequency, and higher electrodes-density is reported to exhibit higher responsivity [159,160]. Thus, it can be summarized that good quality GaN and AlGa_N epilayers as well as good device design are necessary for achieving high UV detection performance. In addition, the interdigitated electrode design of the photodetector is favorable for high speed application [105, 129]. The interdigitated electrode should be well designed so that the dark current can be reduced while the responsivity and efficiency are further improved [161,162].

2.3.3 AlGa_N/GaN photoconductive ultraviolet detector

Photoconductors with simple layer structure design using AlGa_N/GaN have been used for UV detection [163,164]. Interdigitated electrode configuration has been implemented to obtain high performance photoconductive detector [165,166]. Photoconductive ultraviolet sensors fabricated on both *n*-type [167] and *p*-type [168] GaN have been demonstrated and the working kinetics have been discussed. High UV photoresponsivity was reported and huge photoconductive gain was found in such photodetectors [169, 119]. It is believed that there may be several reasons for such high gain: 1) possible trapping of minority carriers (holes) at acceptor impurities [170], 2) modulation mechanism of the conductive volume of the layer. In photoconductive detector, potential barriers generated by surface and bulk dislocation-related band bendings would separate the photogenerated carriers. Carrier recombination and capture are also controlled by such potential barriers [122, 112], 3) the avalanche process initiated by the holes with a larger impact ionization coefficient during UV detection [171], and 4) transition of electrons from deep donor states to the conduction band by photon excitation, electrons are generated on the conduction band

and leave long-lived immobile charged gap states in the active region. Such immobile charges equally affect the potential in the active region which contribute to the gain as trapped holes [135]. Therefore, the photoconductivity is substantially amplified in photoconductors.

2.3.4 Other types of UV photodetectors

GaN based HEMT structure has been used for UV detection with high photoresponsivity [172-174] though the detector fabrication is complex and difficult. GaN-based superlattice/quantum well structure has also been used for UV photodetection, it was found that quantum well design would affect the UV responsivity [175,176]. Moreover, such designs require precise control in epitaxial growth and device fabrication, which is not favorable for simple UV photodetector application.

2.4 AlGaN based ultraviolet/infrared dual band detector on Si (111)

The working principle of basic AlGaN/GaN based UV/IR photodetector has already been discussed in the introduction part (Chapter 1). However, limited by the simple structure used, it is difficult to obtain both IR- and UV-response for dual band detection. Modified on the structure described in Fig. 1.2, dual band detectors based on the group III-V materials on Si system have been previously reported as shown in Fig. 2.2, wherein detectors utilize the interband and intraband transitions of carriers in a homo- or hetero-junction structure [177-179].

Many dual band detectors have been developed for near-, mid- and far-infrared regions using homo- or hetero-junction structures based on the group III-As material systems [180-187]. Similar structures can be employed using GaN/AlGaN multiple layers for UV and IR detection, but the output signal will be a mixed one, necessitating the use of filters to separate the UV and IR

components. Since the bandgap of GaN is fixed corresponding to the wavelength of 362.5 nm, Al-composition in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ can be tuned to achieve photodetector UV response. [188].

Figure 2.2 Three contact structure for simultaneous dual-band response measurements showing (a) separate active regions for UV and IR measurements and (b) the corresponding band diagram under reverse bias configuration

As shown in Fig. 2.3 [177143], there are three GaN-doped electrode layers in the device. The top p -GaN layer and middle n -GaN layer together with intrinsic GaN layer work as p - i - n UV photodiode. The bottom multiple quantum well layers function as the IR photodetector. The UV response of the device can be explained by the interband transition. During the operation, while the electrons in the VB are excited to CB and swept to the middle n -GaN layer by the bias voltage, the holes are swept to top p -GaN layer. It may be noted that the middle n -GaN layer is the contact used for both the UV and IR region detection. The n -GaN is Si-doped with the doping concentration of $5 \times 10^{18} \text{ cm}^{-3}$, which offers low contact resistance when metal stack is deposited on it to form ohmic contact.

The GaN used for UV detection can be replaced by $\text{Al}_x\text{Ga}_{1-x}\text{N}$ with different Al-compositions corresponding to the detection of corresponding wavelengths. All the UV light will be absorbed in the top structure and no UV light will reach the bottom structure. The quantum wells formed by the AlGaN/GaN layers are used for the IR range detection, the well depth can be controlled by the

Al-composition. Since the incoming light traveling into the bottom structure is in IR range, the electrons in the quantum well are excited and moved towards the collector under the voltage bias. To increase the signal intensity, multiple quantum wells are used. In this structure, the UV and IR photocurrent can be obtained at the same time without using any filter.

The dual band photodetector structure to be discussed in Chapter 7 is the modified structure of the detector structure shown in Fig. 2.3 (a), but the principle of UV and IR photodetection is the same as described for the structure in Fig. 2.3 (b). AlGaIn/GaN quantum well structure can be used as IR detector based on the electron transition within the conduction band, and MQW structure can be designed to increase the quantum efficiency [189]. QW-based photodetectors are usually grown on sapphire or SiC substrate as it is less challenging due to lower thermal and lattice mismatch. Similar structure grown on Si (111) substrate will be cost-effective and offer larger size as mentioned before. However, further study is required to overcome the lattice and thermal mismatch issues and achieve smooth, crack-free and low defect density epilayer on Si substrate.

Figure 2.3 (a) Dual band UV/IR detector structure and (b) its band diagram

2.5 GaN and AlGaN etching

Plasma etching has been widely used to etch GaN and AlGaN but ion-induced damages and difficulty to obtain smoothly-etched sidewall are the limitations of this dry etching technique [190-194]. Many wet etching techniques [195] have been developed including photo-enhanced electrochemical (PEC) wet etch [196-199], photo-assisted cryogenic etch (PAC) [200], crystallographic wet etch [201], and photo-assisted anodic etch [202]. KOH and phosphoric acid are used for bulk GaN etching but they are not favorable for device mesa etching [203-208]. Photoenhanced wet etching is also applied for GaN etching, and different etching rates can be easily achieved. However, under-cut is a severe issue and obtaining sharp side wall is difficult [209-213]. Reactive ion etching (RIE) and inductively coupled plasma etching (ICP-RIE) based on RIE are used for device fabrication which need deep etching with high aspect ratio and sharp side wall [214]. For RIE, different plasma is generated by using SiCl_4 [215], BCl_3/SF_6 [216-219], $\text{CHF}_3/\text{C}_2\text{ClF}_5$ [220], Cl_2 [221], CF_4 [222]. However, etching-induced damage would affect the contact resistance [223] and the selectivity between GaN and the mask used. To obtain better etching surface, ICP-RIE is introduced, and different plasma can be generated using gases such as SiCl_4 [224], $\text{Cl}_2/\text{Ar}/\text{N}_2$ [225-227], Cl_2 [228,229], and $\text{BCl}_3/\text{Cl}_2/\text{Ar}$ [230-232]. Nearly vertical GaN etch profiles can be obtained using ICP-RIE due to its high etch selectivity over photoresist and the high etch rate of GaN [233]. In addition, straight sidewalls can be obtained [234,235], and compared to pure RIE technique, chemical etching can be precisely controlled in ICP-RIE [236] and deep etching can be achieved [237].

Chapter 3 Growth and Characterization Techniques

3.1 Growth technique for GaN and AlGaN

Hydride vapor phase epitaxy (HVPE) used to be the main growth technique to grow GaN-based layers, and it needs very high substrate temperature. Presently, metalorganic chemical vapor deposition (MOCVD), also known as metalorganic vapour phase epitaxy (MOVPE) and molecular beam epitaxy (MBE) are the main methods employed to grow GaN-based compound semiconductor layer structures. While MOCVD offers higher growth rate than MBE, MBE provides better interface and requires lower growth temperature. Furthermore, no organics are used during AlGaIn/GaN growth in MBE system making the growth condition carbon free. During the growth, MBE system can control the flux precisely, and hence the epilayer thickness and composition. The growth mechanism can also be understood better since the MBE system is able to monitor the growth continuously by using the reflection high energy electron diffraction (RHEED) tool, which is a surface probing technique.

In MBE there are two possible nitrogen sources: in plasma assisted MBE, nitrogen is activated by RF plasma, and in ammonia MBE, the nitrogen source is obtained by cracking the supplied ammonia gas, with the cracking rate depending mainly on the substrate temperature. Compared with ammonia-MBE, PA-MBE is simpler in service and it also has a number of features, such as possibility of low-temperature growth and absence of hydrogen on the growing surface. Moreover, it allows to increase growth rate considerably. However, for RF plasma, the generation rate of the active nitrogen species only depends on the nitrogen flow and RF power. The required metals are placed in the crucibles of separate cells. Each cell is typically provided with two heaters, one at the top zone and the other at the bottom of the crucible, to generate stable and precise flux during

evaporation. There are shutters for each cell and the epitaxy layer thickness can be controlled by the shutter opening time. In the MBE system, growth takes place in ultra-high vacuum, which reduces the interaction of atomic species on the way to the substrate, and hence keeps their thermal activated kinetic energy sufficient enough to form compounds and good crystal quality materials, even at lower temperatures. Lower growth temperatures also help in decreasing the diffusion of individual atoms leading to abrupt doping profiles. A schematic of the MBE system used in this work is shown in Fig. 3.1

Figure 3.1 Functional diagram of the MBE system

All the epilayers discussed in this thesis were grown using Veeco GEN20MZ MBE system (as illustrated in Fig. 3.2) equipped with Veeco uni-bulb as a nitrogen plasma source as well as several effusion cells for sources such as Ga, Al and Si. High power RF source up to 500 W is used to activate nitrogen plasma and high nitrogen flow up to 10 sccm can be used to achieve high growth rate.

Figure 3.2 A picture of the partial MBE system

3.2 Device fabrication techniques for GaN and AlGaN based photodetectors

A series of device fabrication techniques have been used for photodetector fabrication. The following section describes briefly some of the most important techniques and processes used in this study.

3.2.1 Plasma-enhanced chemical vapor deposition (PECVD)

PECVD technique was employed for oxide (SiO_2) deposition, which can be used as a hard mask in the device fabrication process. In the main chamber of PECVD system, radio frequency (RF) or alternating current (AC) frequency or direct current (DC) discharge can be used to generate plasma of the suitable reacting gas. In PECVD, deposition of thin films of materials can be performed on substrates at lower temperature compared to standard chemical vapour deposition method. For oxide deposition, N_2O and SiH_4 gas precursors are supplied, which under RF discharge excites the reactant gases into a plasma at an operating pressure of a few millitorr inducing chemical reaction. This leads to reaction product to deposit as thin layer of oxide on the substrate. During PECVD process, substrate (with epistructures) is heated up to a temperature around 300°C . Sample temperature, gas flow rate and chamber pressure are some of the important deposition parameters that will affect the film quality and stoichiometry.

3.2.2 Photolithography

Photolithography is a fabrication process technique that is used for transferring designed device pattern from mask onto photoresist. Photoresist is sensitive to UV light, and there are two types of photoresists, namely, positive photoresist and negative photoresist. After exposing to UV light, negative photoresist gets crosslinked which is difficult to dissolve in the developer. This is not an issue in the case of positive photoresist. Before photolithography, photoresist is spin-coated on the sample, and the photoresist thickness is controlled by the spinning speed and time. The exposure energy of UV light to photoresist is well controlled to achieve desired sharp pattern. After exposure, the photoresist is developed in a developer in controlled time to dissolve the area designed to be exposed. In this study, contact exposure technique is used. The mask and the photoresist are in contact tightly in order to obtain sharper exposure pattern.

3.2.3 Reactive-ion etching (RIE)

RIE is a method of dry etching technique using chemically reactive plasma. In this work, RIE is used for SiO₂ hard mask etching. To get sharp hard mask without undercut, RIE is one the best choices rather than wet etching due to the non-selective etching of SiO₂ by HF. High-energy ions from the reactive plasma is accelerated under electromagnetic field, which collide with sample surface. SiO₂ gets etched by the ions by chemical reaction and also get knocked off by the high-energy ions in the RIE process.

3.2.4 Inductively Coupled Plasma reactive-ion etching (ICP-RIE)

ICP-RIE is an advanced microfabrication dry etching technique developed based on RIE. ICP-RIE is used for GaN/AlGa_N etching as no alternative wet etching recipe is found suitable to obtain smooth and high-selective etching surface. In ICP-RIE, an ICP RF power source is incorporated into the RIE system to control the ion current and ion energy applied to the high energy ions, hence better etching surface control can be achieved. High selectivity between hard mask and etching material can be obtained using this technique in addition to deep etching possibility. Moreover, due to the high directivity of the accelerated plasma ions, there is almost no undercut of GaN/AlGa_N mesa layers.

3.2.5 E-Beam evaporation

E-Beam evaporation is a physical vapor deposition technique used for metal deposition. A filament is used to generate an electron beam and the beam get steered via electric and magnetic fields. The electron beam then strikes the material placed inside crucible in a vacuum chamber to evaporate the material locally and deposit on the target sample. Deposition rate can be precisely controlled in E-Beam evaporation using calibration-crystal. In this work, e-beam evaporation was used for ohmic contact metal deposition.

3.2.6 Rapid thermal processing (RTP)

RTP is a technique used for metal semiconductor contact annealing. In an RTP system, a special high-power lamp is used to heat up the sample to a very high temperature at high ramp up rate. RTP is conducted under vacuum or in protection gas such as nitrogen ambient. Ramping down rate is controlled to avoid generation of dislocations and epilayer cracking due to sudden thermal shock.

3.3 Characterization techniques

Different characterization tools have been used in this work for structural, electrical, surface and optical characterization of materials and devices. Some of the tools and techniques used are discussed in the following sections.

3.3.1 High resolution X-ray diffraction

High Resolution X-ray Diffraction (HR-XRD) is a widely used technique to analyze the crystal quality, composition, strain and other related properties [238]. The basic principle is that the “Bragg angle” θ and the distance between two atomic layers follows the Bragg’s Law ($n\lambda = 2d \sin \theta$) where λ is the wavelength of X-ray, d is the interplanar distance (distance between the two atomic layers), and θ is the diffraction angle. The incident X-ray is a collimated beam, which diffracts on the sample surface and is received by a high-resolution detector. As shown in Fig. 3.3, if the epitaxy layer is strained, the distance d will be different compared to the relaxed layer. According to the Bragg’s law, the change in d will make the diffraction angle θ to be different. A two-axis reciprocal space map is a set of ω - 2θ scans repeated for a sequence of steps of incident beam angle over a wide range. Reciprocal space maps in the two-axis mode represent the reciprocal data points in the diffraction space [239]. The relative positions of different epilayers in the reciprocal maps can be used for further structural information. Strain states of different epilayers,

coherent growth of barrier and the lattice matched composition of ternary and quaternary alloys can be analyzed.

Figure 3.3 X-ray incidence and diffraction

XRD can also be used to measure the crystal quality of epitaxially grown films using omega (rocking curve) scan. The distance d at the same orientation is different. Through the 2θ - ω scan, with known value of d , and by simulating the scanned data to make it match the reference, the composition of the epitaxial layer can be obtained. When the epitaxial layer thickness is higher than the critical thickness, the X-ray rocking curve shows peak-broadening due to high dislocation density. The screw (N_S) and edge (N_E) dislocation density with c-type and a-type burger vectors can be then determined by measuring the FWHM of the corresponding peak and using the following equations, respectively [240]:

$$N_S = \frac{\alpha_C^2}{4.35b_C^2} \quad \text{Eq. 3.1}$$

$$N_E = \frac{\alpha_E^2}{4.35b_E^2} \quad \text{Eq. 3.2}$$

where α_C and α_E are the tilt and twist angles obtained from the rocking curve scans, while b_C and b_E are burger vectors along c (0.5185 nm) and a (0.3189 nm) direction, respectively.

3.3.2 Atomic force microscopy (AFM)

Atomic force microscope (AFM) is used to investigate the surface morphology in a nanoscale area. In AFM, one fine tip probe is used to scan over the sample. The force between the tip and the sample surface gets affected by the surface roughness. As shown in Fig. 3.4, the tip, at the end of the cantilever, is close to the sample during the scanning, and the laser beam is aligned to the tip through mirrors. The basic principle involves the observation of deflection of the cantilever due to the forces between tip and the sample surface. Deflection of the tip is measured by the deflection in the laser spot reflected from its surface into an array of photodiodes. Scanning of the probe across the sample surface gives the contour mapping of the surface along x and y directions.

Figure 3.4 Schematic of AFM operation

There are two scan modes, namely static and dynamic modes. For dynamic mode, the tip oscillates at its resonant frequency. The dynamic mode can be further divided into two modes, non-contact mode and tapping mode. For non-contact mode, the tip oscillates during the scanning. As the surface is not perfectly smooth, the Van-der Waal attractive force between the tip and surface will increase, while the tip oscillation frequency will decrease. The frequency is kept the same through the feedback, and by calculating the tip to surface distance, the morphology of the sample surface can be obtained. For the tapping mode, the tip touches the surface and lifts up in every oscillation. In the static mode, the tip touches the surface and scans across the sample area. The tip moves up and down according to the surface morphology and the reflected laser light is collected by the system to calculate the surface morphology. Although the contact mode gives better resolution, if there are some unexpected big particles on the surface, the tip will be contaminated or even damaged.

As AFM is used to obtain high quality surface image, the sample surface needs to be cleaned thoroughly. It should be noted that the scan area is usually small and in micro meter range, so AFM image cannot represent the whole surface morphology of the sample. The only solution is to perform AFM scan in different locations. The AFM image also allows to get the size of small particles. AFM can be done on almost all kinds of materials except soft surface. During the scanning, the humidity and atmosphere inside the chamber can be also be set to desired parameters. For piezo electronics research, the contact mode can be used to apply force to the material. It is controllable and very low force can be applied.

3.3.3 Secondary ion mass spectroscopy (SIMS)

Secondary ion mass spectroscopy (SIMS) is a surface analysis technique used to quantify the composition of the material and depth profiling. One primary ionized particle beam is used to

bombard the sample surface and the surface emits secondary ions. By analyzing the mass spectroscopy, the composition of the surface layer can be obtained. During the measurement, a standard sample is analyzed at the same condition, so the SIMS is quantified with the same element and matrix material as the unknown sample to be analyzed. There are also two modes in SIMS analysis, namely statistic mode and dynamic mode. In the statistic mode, pulse beam is used to bombard the surface and the light secondary ions are likely to reach the detector followed by heavy ions. The statistic mode is surface sensitive and can quantify the composition precisely. However, in the dynamic mode, the material is sputtered and the ions are collected. It is a destructive analysis technique. The final data can be converted into composition with respect to depth, which gives a better understanding of the layer structure. The doping concentration is analyzed by dynamic SIMS using focused Cs ion beam typically with 3 kV bias voltage and a sputtering surface area of $200\text{ }\mu\text{m}^2$. Low energy can be utilized to lessen the effect of atomic mixing due to collision cascades in addition to enhancing the depth resolution

3.3.4 Micro-Raman spectroscopy

Raman spectroscopy is based on the principle of the energy shift caused by the scattering. When the incidence laser beam reaches the sample surface, there are elastic and inelastic scattering by vibration modes. The vibration-modes of the material is affected by the crystal properties, so by detecting the signal, the material strain can be obtained. During the measurement, a laser with a fixed wavelength is used as the light source. The incident light is focused by a microscope which is also used to locate the laser spot. As the laser spot is aligned under the microscope, it is easy to perform measurement at the location of interest. The elastic and inelastic scattered light are collected by a detector through the microscope. Group theory predicts eight set of phonon modes existing for wurtzite structures (C_{6v}^4) and they are $2E_2$, $2A_1$, $2E_1$ and $2B_1$. E_2 phonon mode shifts

linearly with the stress. Table 3.1 provides the standard Raman active phonon modes for GaN and AlN. For the strained epitaxy layers, the E_2 value will be slightly different from the standard value of the bulk material and hence the stress can be obtained [241,242] from the energy shift.

Table 3.1 Raman active phonon modes for GaN and AlN

	E₂(low)	A₁(TO)	E₁(TO)	E₂(high)	A₁(LO)	E₁(LO)
GaN	144	532	559	567.5	734	741
AlN	248	610	670	657	890	912

3.3.5 Scanning electron microscopy (SEM)

Scanning electron microscope technique is used to obtain the image of the sample surface in micrometer range. The principle of SEM is similar to the optical microscope except that the electrons are used instead of light and the lens are replaced by electron magnetic lens as the wavelength of visible light limits the resolution. During the scanning, an electron beam is incident on the sample surface and the surface emits electrons with wide range of energies. As the electrons are generated using different mechanisms, they can be categorized into secondary electrons, auger electrons and back scattered electrons. When the incident electrons hit the sample surface, secondary electrons are emitted from the surface due to bond-breaking, which can be collected and the signal amplified. The energy density is analyzed and the live image is displayed on the monitor. The emission of a secondary electron creates a vacancy in the atom, and the vacancy is filled. Since the electron energy is transferred from high level to low level, corresponding X-ray will be generated. For different atoms or elements, the X-ray wavelength is different. Thus, the stoichiometric ratio of the elements can also be obtained by analyzing the X-ray intensity.

Other than the secondary electrons, back scattered electrons and auger electrons are also emitted. Secondary electrons are elastically scattered electrons with large angle, and the electron energy of

Auger electron is found to be between the secondary electrons and back scattered electrons. Three dimensional (3D) image can also be obtained using SEM by placing the sample in different angles, which allows to obtain cross sectional image and the thickness. SEM combined with an energy dispersive spectroscopy (EDX) tool enables element analysis at the same time.

3.3.6 Hall-Effect measurement

Hall effect measurement is the most widely used technique for semiconductor material characterization. Fig. 3.5 shows the schematic of the Hall effect measurement based on Van-der Pauw method. Samples with dimensions of about $1\text{ cm} \times 1\text{ cm}$ are used for measurement. Ohmic contacts are made by sticking a small quantity of indium at four corners of the sample and annealing for about 3 minutes in forming gas environment. Prior to Hall measurement, the material resistivity measurement is performed without any applied magnetic field as shown in Fig 3.5. The resistivity ρ can be calculated from the voltage measured and the current applied between the contacts, and using the sample dimensions.

Figure 3.5 Schematic of Hall effect measurement

For the Hall effect measurement, a constant magnetic field B is applied perpendicular to the sample surface and current $I_{2,4}$ is applied between contact 2 and 4 while the Hall voltage $V_{1,3}$ between

contact 1 and 3 is measured, as shown in Fig 3.5. The carriers supplied by the current undergo the Lorentz force perpendicular to both the magnetic field direction and current flow. The holes and electrons will accumulate on the opposite sides of the sample, generating a potential difference between the two contacts which is termed as the Hall voltage. The Hall coefficient R can be calculated using Eq. 3.3 where r is the scattering factor for the semiconducting material and t is the sample thickness.

$$R = \frac{r \times V_{24} \times t}{I_{13} \times B} \quad \text{Eq. 3.3}$$

The hall coefficient is positive for p -type material and negative for n -type material. Thus, by calculating the Hall coefficient, the material carrier type can be obtained. The carrier concentration N_s and mobility μ can be obtained using Eq. 3.4 and 3.5 where q is the electron charge and ρ is the resistivity of the material.

$$N_s = \frac{r}{R \times q} \quad \text{Eq. 3.4}$$

$$\mu = \frac{R}{\rho} \quad \text{Eq. 3.5}$$

Measurements at several locations of a sample can be conducted to determine the uniformity of the electrical parameters across the sample.

3.3.7 Photoresponsivity measurement

The photoresponsivity measurement in this work was done using an in-house integrated system to characterize the fabricated photodetectors. Fig. 3.6 shows the schematic of the system that includes a lock-in amplifier, chopper, light source and voltage source.

The light source provides xenon light with continuously varying wavelength ranging from 200 nm to 2500 nm. The light source and the lock-in amplifier can be controlled by the computer. The

lock-in amplifier is used to collect the AC photocurrent signal, which is connected in series with the chopper. When the incident light goes through the chopper, it will be modified in to AC mode. Only the AC current will be collected during the measurement while the high dark current will be eliminated. The detector is biased using a voltage source and the voltage is fixed during each measurement. The system can measure the photocurrent under certain voltage bias and wavelength of the light that are controllable. The photocurrent is measured along with the light intensity during testing, and the photoresponsivity is determined. To calculate the photodetector photoresponsivity, one commercial photodetector sample is first calibrated to obtain the photoresponsivity $R_{detector}$ with respect to detection wavelength. The sensing area of the photodetector to be measured and the area of the incoming light spot on the detector are calculated to be A_{sample} and A_{light} , respectively. Hence, the incoming light power P_{light} can be calculated based on the photocurrent $I_{detector}$ of the commercial photodetector using Eq. 3.6:

$$P_{light} = \frac{I_{detector}}{R_{detector}} \quad \text{Eq. 3.6}$$

Then, the incoming light power P_{sample} received by the photodetector to be measured can be calculated by Eq. 3.7. Finally, the photoresponsivity R_{sample} is obtained using Eq. 3.8 based on the photocurrent I_{sample} :

$$P_{sample} = P_{light} \times \frac{A_{sample}}{A_{light}} \quad \text{Eq. 3.7}$$

$$R_{sample} = \frac{I_{sample}}{P_{sample}} \quad \text{Eq. 3.8}$$

Figure 3.6 Schematic of photoresponsivity measurement system

Chapter 4 Optimization of GaN/AlN on Si (111)

Substrate

The large lattice and thermal mismatch between GaN and Si (111) substrate results in high dislocation density and cracking in the epilayers, respectively leading to poor device performance. AlN is typically grown as a buffer layer, although the mismatch between AlN and Si (111) is large, Al can wet the Si surface better than Ga and form AlN on Si surface instead of SiN_x . AlN is only grown in N-rich condition [30, 49] to avoid Al reacting with Si. A thin AlN layer is grown on Si (111) substrate to compensate the strain and acts as a buffer layer [243]. However, during growth optimization, surface morphology was found to be rough with dense microcrystals which would affect further growth. In this chapter, GaN/AlN on Si growth is demonstrated in detail. The microcrystal issue is addressed and growth is tuned accordingly to achieve crack free smooth GaN. The effect of Si doping effect on the strain in GaN layer is also studied using PL and Raman spectroscopy.

4.1 Growth of AlN layer

In this study, before AlN growth, the surface of the Si-substrate was prepared. During growth for each growth step, all the heater temperatures are measured by a calibrated pyrometer. Firstly, Si substrate was thermal-cleaned at 975 °C for 30 minutes before heating up to 1075 °C for 10 minutes to remove the native oxide. After this step, clear (7×7) RHEED pattern is achieved, which confirms that Si (111) surface is clean and the native oxide is completely desorbed as shown in Fig. 4.1.

Figure 4.1 (7×7) RHEED reconstruction pattern of thermally cleaned Si (111) surface

This is followed by the growth of AlN, which was carried out in a two-step process at a fixed temperature of 770 °C. The two-step consists of maintaining low Al flux (LF-AlN) first, followed by high flux (HF-AlN). At the beginning of AlN deposition, streaky (1×1) RHEED pattern obtained indicates 2D AlN nucleation as in Fig. 4.2 (a), and the RHEED became spotty at the end indicating 3D growth as shown in Fig. 4.2 (b). The AlN layer thickness grown was 120 nm, which was earlier optimized [5] for relative smooth surface morphology and good crystal quality.

Figure 4.2 Streaky (1×1) RHEED pattern showing (a) AlN surface at the beginning of AlN growth, which became spotty (b) at the end

4.2 Growth optimization of GaN/AlN

4.2.1 Growth of GaN/AlN on Si (111) by PA-MBE

All the samples were grown on 2-inch Si (111) substrate. To achieve GaN growth in Ga-rich condition without too much Ga accumulation on the surface, 2 min Ga-desorption was implemented after every 20 min of growth by closing all the source shutters. This process is critical and finely controlled to allow evaporation of excess Ga on the surface but without degrading the surface quality. RF power for plasma generation was fixed at 300 W and N₂ flow was maintained at 2 sccm for all the growths. The complete growth process steps after Si substrate cleaning are detailed below:

1. AlN growth is done at the pyrometer temperature $T_P = 770$ °C.
2. After growing AlN at low Al-flux for 16 min, the flux was graded to a higher level in 5 min.
3. AlN was grown at the high flux for 15 min. (If no Al flux grading, growth time is 36 min).
4. After cooling down to GaN growth temperature of 730 °C, GaN growth was conducted in N-rich condition for 36 min.
5. Ga flux was graded to Ga-rich condition to grow GaN for 120 min (20 min growth + 2 min Ga desorption) $\times$ 6 cycles.
6. Heater is ramped down to room temperature.

A series of GaN/AlN on Si growths under different growth conditions as summarized in Table 4.1 were conducted for growth optimization. Sample 1 (V845) was grown in Ga-rich condition for GaN, and N-rich condition for AlN with fixed Al flux. Sample 2 (V849) has AlN layer, the same as sample 1 but GaN was grown with Ga flux grading from N-rich to Ga-rich condition. For sample 3 (V1010) and sample 4 (V857), both have AlN grown in N-rich condition with two-step LF-AlN

(LF = low flux) to HF-AlN (HF = high flux) growth. GaN in sample 3 (V1010) was grown in Ga-rich condition, and sample 4 (V857) was grown in N-rich condition to obtain 3D growth first, followed by Ga-rich condition (2D) at the top. For all 4 samples, at the end of AlN growth, RHEED pattern was found spotty as shown in Fig. 4.2 (b) as the growth was in N-rich condition (3D growth).

Table 4.1 GaN/AlN on Si grown under different conditions

Sample	AlN (III/V)	GaN (III/V)	AlN Thickness (nm)
1 (V845)	0.73	1.2	110
2 (V849)	0.73	LF = 0.77 HF = 1.02	110
3 (V1010)	LF= 0.18 HF= 0.26	1.14	135
4 (V857)	LF = 0.47 HF = 0.66	LF = 0.84 HF = 1.11	119

* HF = high flux, LF = low flux.

The evolution of RHEED pattern was followed for sample 4 (V857) as the growth proceeded. At the beginning of 2D GaN growth on AlN, the pattern was spotty as in Fig. 4.3 (a) due to bottom 3D growth of GaN. After 10 min of growth, the pattern was still spotty but with connected spots as shown in Fig. 4.3 (b). With further growth, GaN surface transferred from 3D to 2D growth as indicated by streaky pattern in Fig. 4.3 (c). At the end of growth, clear and improved streaky

RHEED pattern was obtained. The pattern became bright with the desorption of Ga at the end of growth as shown in Fig. 4.3 (d).

Figure 4.3 RHEED pattern of 2D GaN growth on AlN after: (a) 3 min , (b) 10 min, (c) 30 min and (d) end of growth

4.2.2 Characterization of GaN/AlN on Si (111)

It was found that GaN/AlN grown with single flux (sample 1, V845) showed very poor surface morphology as shown by the optical microscopy image in Fig. 4.4 (a). Employing either two-step growth process on GaN (sample 2, V849) or two-step growth on AlN (sample 3, V1010), showed improvement although lots of “particles” were seen on the surface as shown in Fig. 4.4 (b) and (c). Finally, crack free and smooth GaN/AlN on Si substrate (sample 4, V857) as shown in Fig. 4.4 (d)

Figure 4.4 Optical microscopy image of GaN/AlN epilayers with different Al/Ga flux ratios for samples 1 to 4

was achieved by grading AlN with Al flux in N-rich region together with grading GaN with Ga flux grading from N-rich to Ga-rich condition. Fig. 4.5 shows the AFM image of sample 4 with RMS roughness of 0.4 nm in $5 \times 5 \mu\text{m}^2$ area.

Figure 4.5 AFM image of sample 4 (V857) with RMS roughness of 0.4 nm in $5 \times 5 \mu\text{m}^2$ area

It was found that similar particle-formation have also been reported during AlGaN/GaN/AlN on Si growth, and this issue was solved by using similar two-step AlN growth grading from 3D AlN to 2D AlN followed by GaN layer growth [60]. The particles shown in Fig. 4.4 (a) to (c) were further studied by SEM. As shown in the optical microscopy image of Fig. 4.6 (a), there are some triangular-like features under microcrystals, which will be described later in this section. However, these features cannot be seen under SEM as shown in Fig. 4.6 (b), which means that these triangular-like features are not on the GaN surface. Instead of triangular-like features, some circular features are found under higher magnification in SEM as shown in Fig. 4.6 (c). Since the

Figure 4.6 (a) Microscopy images, (b, c) SEM images of the surface of GaN/AlN shown in Fig. 4.4 (a) to (c), and (d) AFM of the surface in between these microcrystals

wafer was cleaned by HCl to etch away the residual Ga droplets after growth which is a normal cleaning process, these circular features are located where there were droplets before the cleaning. In between these microcrystals, the surface was found smooth as shown by AFM image in Fig 4.6 (d). The RMS roughness measured in this area is only 0.55 nm. TEM specimens were prepared using a focused ion beam (FIB) tool (FEI NOVA nanolab system) using a 30 KV Ga⁺ source. They were examined by JEOL JEM-2100F and JEOL JEM-ARM spherical aberration corrected scanning transmission electron microscope (STEM) operated at 200 kV while composition profiles were studied by energy dispersive X-Ray analysis (EDX).

Fig. 4.7 (a) shows FE-SEM image of the surface of sample 1, which exhibits the presence of microcrystals and voids as indicated by the arrows. Microcrystals were found grown together closely to form isolated clusters, and these microcrystals were confirmed to be made of GaN as determined from EDX analysis. The microcrystals were further investigated by performing cross-sectional TEM along [11-20] zone axis as shown in Fig. 4.7 (b). The presence of void indicates that no growth ever occurred underneath the microcrystal. Figs. 4.7 (c) and (e) show high-angle annular dark-field imaging STEM (HAADF-STEM) images of the regions below the microcrystal. The spot EDX scan was measured on the layer deposited on top of GaN (marked in red) as shown in Fig. 4.7 (d). The spectra showed major presence of Si and a small signal corresponding to N while negligible signal was obtained for Ga. Fig. 4.7 (f) shows the EDX line profile of Ga along GaN/AlN/Si, indicated by dotted line in red in Fig. 4.7 (e). The inset profile of Fig. 4.7 (f) shows the magnified EDX line scan signal at the AlN/Si interface. As can be seen, an increase in the peak intensity of Ga K α at the AlN/Si interface signifies the presence of Ga at the interface.

The growth of AlN using III/V < 1 results in insufficient surface migration or diffusion of Al atom due to inadequacy of Al metal. The reduced diffusion results in 3D growth with the formation of

Figure 4.7 (a) FE-SEM image of the GaN surface in sample 1, (b) cross-sectional TEM image of the microcrystal, (c, e) HAADF-STEM images of the region below the microcrystal in sample 1, (d) and (f) corresponding spot and line EDX spectra

columnar like structures similar to the widely reported morphology for GaN grown under similar conditions [244, 245]. The columns, elongated along the growth direction, are also found to be separated from columns nearby [246]. It is likely that the columnar structure of AlN grown under $\text{III/V} < 1$ or threading dislocation assisted pipe diffusion may allow the migration of Ga metal to Si substrate leading to the interdiffusion. The threading dislocation assisted Ga-Si diffusion can be eliminated as the diffusion was found to occur regardless of the dislocation density (screw dislocation density: $3 \times 10^9 - 10^{10} \text{ cm}^{-2}$). Thus, Ga may possibly diffuse through the spacing between the adjacent AlN columns and interact with silicon inducing meltback etching. In order to confirm this mechanism, a spot EDX scan was measured on the AlN layer of sample 1 as marked in red. The EDX spectra in Fig. 4.8 (b) shows the presence of both Ga as well as Si atoms in AlN layer, which proved the diffusion of Ga via AlN layer.

Figure 4.8 (a) HAADF-STEM image of the region below the microcrystal of sample 1 and (b) spot EDX spectrum on AlN layer, marked in red in (a)

Fig. 4.9 (a) shows the magnified HAADF STEM image of the microcrystals after HCl cleaning while Fig. 4.9 (b) and (c) show the spot EDX scan of the 1 and 2 locations marked in red in Fig. 4.9 (a). The EDX spectra in Fig. 4.9 (b) and (c) confirms the formation of GaN and existence of

reacted Ga-Si alloy. The interdiffusion of Ga into silicon via AlN layer undergoes eutectic transformation thereby forming Ga-Si alloy. The alloy together with the Ga metal and nitrogen supplied during the growth forms GaN microcrystals. It can be concluded that HCl-cleaning of the sample removed the excess Ga metal leaving the void, reacted Ga-Si alloy (Fig. 4.9 (b)) and GaN (Fig. 4.9 (c)) behind.

Figure 4.9 (a) HAADF-STEM image of the microcrystal of sample 1, and (b), (c) show the spot EDX spectra, marked in red in (a)

Thus, the excessive Ga during the initial growth of GaN of sample 1 diffuses through the spacing between the AlN columns as indicated in the schematic Fig. 4.10 (a) and leads to the formation of microcrystals. For direct 2D GaN grown on AlN layer, when Ga flux was set at Ga-rich condition at the beginning of the growth, not much Ga could diffuse through AlN layer or the Ga diffusion time is not long enough. With the growth going on, Ga metal accumulates on GaN surface, and with longer Ga interdiffusion time, the Ga/Si interdiffuses finally through the AlN layer. Hence, during GaN growth, Ga-Si alloy together with nitrogen formed such microcrystals only inside GaN

layer or above instead of at the GaN/AlN interface. Similar but less dense microcrystals are formed for sample 2 even when the growth of GaN is performed in two steps. GaN grown under $\text{III/V} < 1$ condition also exhibits columnar morphology similar to 3D grown AlN. The excess Ga from the part of GaN grown using $\text{III/V} > 1$ is found to diffuse through these columns followed by AlN leading to the interdiffusion and microcrystal formation. AlN of samples 3 and 4 are likely to exhibit two different column sizes corresponding to the two-step growth with different III/V ratios as the lateral correlation length or the average grain size of AlN is found to decrease with reducing III/V ratio [247]. The excess metal during the growth of GaN of sample 3 diffused through the AlN layer and formed microcrystals. However, clean surface was achieved when GaN was grown using two-step growth process as observed in sample 4. As AlN layer has different AlN grain sizes and there will be more boundary branches in-between which leads to longer Ga diffusion length and makes it difficult for Ga to diffuse through AlN layer as shown in Fig. 4.10 (b). For sample 3, graded AlN was intentionally used for blocking the Ga diffusion, but top GaN was grown in Ga-rich condition, hence graded AlN failed to block the diffusion of massive Ga. GaN layer for sample 4 is similar to sample 2 and some part of Ga diffused through 3D GaN layer to the GaN/AlN

Figure 4.10 Schematic of Ga diffusion through AlN layer of (a) sample 1 and 2, (b) sample 3

interface. However, since the quantity of Ga on GaN/AlN interface was much less compared with sample 1 and 3, graded AlN layer was believed to block the rest of the residual diffused Ga.

In order to further evaluate the formation of nanocolumns, AFM scans were done on two samples, namely, samples A and B with single AlN layer grown using a fixed III/V ratio of 0.3 and 0.6, respectively. Fig. 4.11 (a) and (b) show the AFM images and the corresponding line profile across the surface of sample A and B, respectively. An average column size of 30 nm and 60 nm was measured for sample A and B, respectively. The column size increased with increasing the III/V ratio of AlN which is very well in accordance with the findings that the lateral correlation length or the average grain size of AlN varies linearly with the III/V ratio [247].

Figure 4.11 (a) and (b) AFM images with the corresponding line profiles across the surface of samples A and B, respectively.

Thus, it can be inferred that the AlN grown using two-step growth process with different III/V ratios, for the top and bottom AlN layers, will exhibit two different column sizes one on top of the other, as proposed schematically in Fig. 4.10.

To study the Ga reaction on AlN layer at GaN growth temperature, two AlN samples (Sample 5 and 6) grown by MBE done under different growth conditions and two AlN on Si MOCVD grown templates (samples 7 and 8) were considered as summarized in Table 4.2. In this experiment, Ga metal was deposited on AlN with a flux of 6.95×10^{-7} Torr followed by Ga desorption for 10 cycles, and in each cycle, the deposition and desorption times were kept at 2.5 min and 2 min, respectively. After AlN growth and before Ga deposition/desorption, nitrogen plasma was cut off and the chamber was pumped down to avoid any GaN formation. The same experiment was repeated on sample 7 that was loaded into MBE chamber to compare. Cracked AlN on Si template (sample 8) was used for GaN growth, and GaN was grown directly in Ga-rich condition following the same procedure mentioned in 4.2.1 on the growth of GaN/AlN on Si (111).

Table 4.2 GaN/AlN on Si for Ga/Si diffusion study

Sample	AlN growth temperature (°C)	AlN (III/V)	GaN growth temperature (°C)	AlN Thickness (nm)
5 (V1133)	773	0.19-0.26	715	74
6 (V1136)	774	0.19-0.26	715	203
7 (V1134)	N/A	N/A	715	247
8 (V1118)	N/A	N/A	715	245

AlN was grown with different thicknesses (Table 4.2), and similar triangular-pattern as in Fig. 4.6 (a) can be seen under the microscope on sample 5 and 6 after unloading from the MBE chamber as shown in Fig. 4.12 (a) and (d). The corresponding higher magnification surface morphology obtained using SEM for samples 5 and 6 are shown in Fig. 4.12 (b, c) and (e and f), respectively. In the SEM image of Fig. 4.12 (b) and (e), no triangular-pattern can be seen, but only circular pattern wherever there was Ga droplet before HCl cleaning. As shown in Fig. 4.12 (c) and (f),

voids under the Ga droplet are the etch pits which are formed due to the Ga-Si interdiffusion and are likely to be filled by Ga-Si alloy. The presence of these etch pits after Ga deposition/desorption indicate that Ga metal will diffuse into Si substrate through AlN layer if there is excess Ga on AlN at GaN growth temperature.

Figure 4.12 Microscopy image of AlN epilayer surface after Ga deposition/desorption with different AlN growth conditions (a) sample 5, and (d) sample 6. SEM image (b,c) sample 5 and (e,f) sample 6 after HCl cleaning

For comparison, the same experiment was repeated on MOCVD grown AlN template (sample 7) having similar thickness as MBE grown AlN sample before Ga deposition. AFM analysis on sample 7 showed smooth surface with RMS roughness of 0.4 nm in $5 \times 5 \mu\text{m}^2$ area as in Fig. 4.13 (a). After Ga deposition/desorption, “pits-like” features can be observed under AFM and optical microscopy images as depicted in Fig. 4.13 (b), and the AlN surface is found with the RMS roughness increased to 1.5 nm. However, only the surface roughness increased, but no triangular-pattern is seen in the optical microscopy image. Further, the AFM image shows that pits are shallow and do not penetrate AlN layer. Although AlN grown by MBE and MOCVD have similar

Figure 4.13 Surface morphology of sample 7 (a) before and (b) after Ga deposition/desorption

thickness, MOCVD grown AlN succeeded in blocking Ga/Si interdiffusion. Sample 8 was GaN grown in Ga-rich condition directly on cracked AlN template. Cracks are clearly seen in SEM image in Fig. 4.14 and microcrystals were still formed but only along the cracks. This observation confirms that only where such diffusion path exists in AlN template can Ga metal diffuse through AlN into Si substrate.

Figure 4.14 (a) SEM image of sample 8 with microcrystals appearing only along the cracks and (b) magnified image of (a)

Table 4.3 lists dislocation density in AlN and GaN obtained using HR-XRD. As can be seen, sample 1 and 2 show similar AlN dislocation density of $3.5 \times 10^9 \text{ cm}^{-2}$, which is better than sample 4 with the value of $9.7 \times 10^9 \text{ cm}^{-2}$, because sample 1 and 2 used higher Al flux than sample 4 while

sample 3 used the lowest Al flux and ended up with worst AlN dislocation density of $3.1 \times 10^{10} \text{ cm}^{-2}$. Sample 1, 2 and 4 show similar GaN quality while sample 3 has one order higher dislocation density that may be caused by low quality bottom AlN layer. Since AlN needs to be grown in N-rich condition ($\text{III/V} < 1$), quality of AlN is limited by Al/N ratio, and hence GaN-quality is also limited. It can be concluded that to achieve good GaN/AlN on Si, there is a trade-off with respect to surface morphology and crystal quality

Table 4.3 Dislocation density in AlN and GaN of sample 1 - 4 with different growth conditions

Sample	AlN screw dislocation density ($\times 10^9 \text{ cm}^{-2}$)	GaN screw dislocation density ($\times 10^9 \text{ cm}^{-2}$)	GaN edge dislocation density ($\times 10^{10} \text{ cm}^{-2}$)
1 (V845)	3.5308	3.0448	2.1547
2 (V849)	3.5308	1.5093	1.2022
3 (V1010)	31.650	14.232	10.620
4 (V857)	9.6547	2.6772	1.7580

4.3 Si doping of GaN layer

Based on the optimized GaN growth of sample 4 (V857) listed in Table 4.1, a series of Si doped *n*-GaN was grown on high resistive Si substrates at different Si cell temperatures. All the Si doped GaN was grown with a thickness of 500 nm on high-resistive Si substrate (resistivity $\sim 6000 \Omega \times \text{cm}$). Hall-effect measurement results of these samples are summarized in Table 4.4. As shown in Fig. 4.15, doping of Si is not linear with respect to Si cell temperature. It can be contributed to the nonlinear relation between the Si flux and the Si cell temperature, similar trend was also observed for Ga and Al flux. The curve can be divided into two regions with two incorporation efficiencies. When Si cell temperature is lower than 1200 °C, carrier concentration is linear as a function of Si cell

temperature. It is noted that the highest carrier concentration of $\sim 2 \times 10^{20} \text{ cm}^{-3}$ was achieved at Si cell temperature of 1310 °C.

Table 4.4 Si doping concentration vs. Si cell temperature

Samples	Si cell temperature (°C)	N ($\times 10^{19} \text{ cm}^{-3}$)
V876	No doping	Resistive sample
V880	1170	0.24 - 0.38
V877	1180	0.47 - 0.52
V878	1190	0.52 - 0.59
V875	1200	1.0 - 1.5
V895	1230	2.1 - 2.3
V896	1280	7.8 - 9.2
V898	1310	17.0 - 18.0

Figure 4.15 Si doping concentration vs. Si cell temperature

At the end of growth, RHEED pattern was checked. Similar streaky RHEED pattern as shown in Fig. 4.16 (a) were observed for all the samples grown with Si cell doping temperatures upto 1280 °C (samples V876 to V896). However, at the end of sample growth for V898 which was doped at the

highest Si cell temperature of 1310 °C, RHEED pattern was very slightly spotty which indicates that the surface morphology may be getting poorer as in Fig. 4.16 (b).

Figure 4.16 RHEED pattern of sample (a) V896 and (b) V898 at the end of growth

Fig. 4.17 shows microscopy image of GaN doped with different Si concentration. All samples surfaces are smooth without microcrystals. AFM image showed that samples with Si doping at lower concentration have similar RMS roughness. Samples V880, V875 and V896 were doped with Si concentration of $3 \times 10^{18} \text{ cm}^{-3}$, $1 \times 10^{19} \text{ cm}^{-3}$ and $9 \times 10^{19} \text{ cm}^{-3}$ and their corresponding RMS roughness in $5 \times 5 \text{ } \mu\text{m}^2$ are 0.4 nm, 0.5 nm and 0.6 nm, respectively. However, at the highest Si doping concentration of $1.8 \times 10^{20} \text{ cm}^{-3}$, RMS roughness increased to 1.1 nm, which is also matching with the poor RHEED pattern shown in Fig. 4.16 (b). It was reported that high Si doping level leads to increase in the density of grains on the surface of Al(Ga)N layers and the threading dislocation density [102]. Due to high Si doping, the compressive strain in the GaN

layer was relaxed, and leads to the rough surface morphology [126]. The overall surface morphology is still acceptable for the following epitaxial growth.

Figure 4.17 Optical microscopy and AFM image (inset) of Si doped GaN at different Si cell temperatures: (a) V880, (b) V875, (c) V896 and (d) V898

Temperature dependent hall-effect measurement was performed on samples V896 and V898 as illustrated in Fig. 4.18 (a) and (b), respectively, to understand the effect of temperature on mobility and carrier concentration. While carrier concentration remained nearly unchanged as a function of temperature for both the samples, the carrier mobility was found to exhibit slightly higher at low temperatures. It can also be observed that, as expected, the mobility is found to be lower for the highly doped sample, V898. From the temperature dependent hall-effect measurement, the activation energy values of 0.25 meV and 0.05 meV were obtained for samples V896 and V898,

respectively. Decreasing of activation energy with the increasing of donor concentration has been observed before [248], and it can be explained by the interaction of donor wave functions which reduces ionization energies, as has been observed for other semiconductors, e.g., Si and Ge [249]. These low values indicate that all the silicon atoms are activated and the carrier concentration determined represents the dopant concentration [250].

Figure 4.18 Temperature dependent Hall-effect measurement on sample (a) V896 (a) and (a) V898

Photoluminescence measurement was conducted on Si doped GaN samples to understand the doping effect. As can be observed from the PL spectra of all samples shown in Fig. 4.19 (a), it can be observed that initially the PL peak shifts towards lower energy with doping. However, with further doping, the PL peak energy shifts towards higher energy. It can be assumed that the reduction of bandgap with the doping can be attributed to bandgap narrowing (BGN) [251], and further increase in optical bang gap upon higher doping can be attributed to Moss-Burstein shift (MBS) [252] as shown in Fig. 4.19 (b). BGN can be explained by Eq. 4.1 where N and a_B in Eq. 4.2 to 4.5 are carrier density and effective Bohr radius, respectively.

$$\Delta E_{BGN} = \Delta E_{ee} + \Delta E_{ei} \quad \text{Eq. 4.1}$$

Figure 4.19 (a) Photoluminescence measurement on Si doped GaN with different carrier concentration and (b) PL peak intensity vs. doping concentration

$$\Delta E_{ee} = -\frac{e^2 k_f}{2\pi^2 \kappa \epsilon_o} - \frac{e^2 k_{TF}}{8\pi \kappa \epsilon_o} \left[1 - \frac{4}{\pi} \tan^{-1} \left(\frac{k_f}{k_{TF}} \right) \right] \quad \text{Eq. 4.2}$$

$$\Delta E_{ei} = -\frac{e^2 N}{\kappa \epsilon_o a_B k_{TF}^3} \quad \text{Eq. 4.3}$$

where ΔE_{ee} and ΔE_{ei} are electron-electron interaction and electron ion interaction, respectively.

The constant a_B is the effective Bohr radius of the electron, e is the elementary charge. k is the wave factor and ϵ_0 is the vacuum dielectric constant. The Fermi vector k_f in Eq. 4.2 is given as:

$$k_f = (3\pi^2 N)^{\frac{1}{3}} \quad \text{Eq. 4.4}$$

where N is the number of particles (doped Si). The estimated inverse Thomas-Fermi screening length

k_{TF} in Eq. 4.2 is given by:

$$k_{TF} = 2\sqrt{\frac{k_f}{\pi a_B}} \quad \text{Eq. 4.5}$$

Hence, the band gap varies with $N^{1/3}$. The fitting with the data in Fig. 4.19 (b) gives a BGN factor of 1.33×10^{-8} eV. Similar value was obtained earlier and attributed to lower compensating films (n to p compensation) [251]. Band gap filling can be described by MBS, and ΔE_{MBS} is given as

$$\Delta E_{MBS} = \frac{\hbar^2 k_f^2}{2m_{Dep}^*} \quad \text{Eq. 4.6}$$

The $\hbar$ is the reduced Planck constant and m_{Dep}^* is the density-of-states mass. The Band gap variation due to strain ΔE_{str} is given as a sum of conduction band variation ΔE_c and valence band variation ΔE_v :

$$\Delta E_{str} = \Delta E_c + \Delta E_v \quad \text{Eq. 4.7}$$

The band gap narrowing curves shown in Fig. 4.20 match with PL peak energy showed in Fig. 4.19 (b). The strain effect on band gap is considerably lower compared to BGN and MBS effects.

Figure 4.20 Band gap narrowing (BGN), Moss-Burstein shift (MBS) and strain induced BGN vs. doping concentration

At around $9 \times 10^{18} \text{ cm}^{-3}$, the MBS effect overtakes the BGN effect while Strain induced band gap narrowing does not change much. The Peak position of the second peak in PL is 3.33 – 3.35 eV. According to the reported data [253], the second peak is close to Y4, Y5 and Y6 group, and it can be attributed to excitation from structural defect at the surface or surface donor–acceptor pair or the combination. It was reported that high Si doping level leads to increase in the threading dislocation density [102]. At high doping level, MBS effect overtakes the BGN effect and the MBS effect measured may be lower than expected.

XRD scan was performed along (002) orientation on GaN doped samples with different Si concentration. AlN and GaN wurtzite peaks were observed for undoped to the highest Si doped GaN samples and no other orientation was observed (Fig. 4.21).

Figure 4.21 XRD ω scan along GaN (002) plane on GaN with different Si doping concentration

GaN-doped samples were analysed by Raman spectroscopy to study the effect of doping on the strain in the sample. As shown in Fig. 4.22, there is a large Raman red shift of E_2 peak for the highest doped sample compared to the undoped. Correspondingly, the tensile strain value

determined from Raman measurements is the highest for this sample as listed in Table 4.5. In the case of undoped sample, a small peak appears near 733 cm^{-1} , which corresponds to GaN A₁ [LO] peak. The annihilation of this GaN A₁ [LO] peak for the heavily doped sample with the dopant concentration of $\sim 2 \times 10^{20}\text{ cm}^{-3}$ is attributed to the phonon–plasmon coupled modes mechanism [250, 254]. The films appeared to be “relaxed” because the introduced tensile strain by Si doping compensated the remnant compressive strain at a particular thickness and at a certain doping concentration. The strain seemed to have saturated and even decreased for higher doping concentrations. As reported, the strain should always increase with the Si doping concentration [123].

Table 4.5 Stress of Si doped GaN calculated by Raman shift

Sample	Doping ($\times 10^{19}\text{ cm}^{-3}$)	Stress (GPa)
V899	Undoped	-0.016
V896	7.8 - 9.2	+0.006
V898	21-22	+0.322

Figure 4.22 Raman shift of E₂ peak on samples listed in Table 4.5

4.4 Summary

In this study, it was found that only AlN grown in N-rich condition graded from low Al flux to high flux followed by GaN grading from 3D growth (N-rich) to 2D growth (Ga-rich) showed smooth surface without any microcrystal. Since the top GaN layer was grown in Ga-rich condition and there were Ga metal droplets on the surface during growth. AlN layer failed to block Ga diffusion because gap or grain boundary formed, due to N-rich AlN growth in MBE, could act as a path for Ga diffusion into Si substrate and dissolve in Si. Ga-Si alloy would form and float on Ga droplets during the growth. The following GaN growth would continue on the Ga-Si alloy and form dense GaN microcrystals. Ga/Si diffusion would always happen regardless of AlN thickness. However, no Ga/Si inter diffusion was found on MOCVD grown AlN/Si template. As a result, GaN growth must be done in N-rich condition at GaN/AlN interface with no residual Ga on surface. During the following GaN growth, Ga flux could be increased to Ga-rich condition on the thin GaN grown already on AlN. Bottom two-step grown AlN could stop rest of the Ga from diffusing into bottom Si substrate.

Smooth GaN/AlN grown on Si (111) by PA-MBE without microcrystals was achieved with the RMS roughness ~ 0.4 nm. Si doping concentration is found non-linear to Si cell temperature at higher doping levels. Surface morphology was not affected by Si doping at lower doping levels while very high doping level ($> 10^{20} \text{ cm}^{-3}$) caused surface roughness to increase to 1.1 nm. Clear band gap narrowing effect on Si-doping of GaN has been observed, and also MBS effect is found to overtake BGN effect. With Si doping concentration increasing from undoped to $2 \times 10^{20} \text{ cm}^{-3}$, strain in GaN changed from compressive to tensile strain.

Chapter 5 Optimization of AlGa_N/AlN/SiN_x on Si

(111) Substrate by PA-MBE

Unlike GaN/AlN growth on Si (111), GaN grading from 3D to 2D growth could not be used for AlGa_N/AlN on Si (111) as Al-composition would be affected by Ga/N ratio. In this study, 2D GaN growth on AlN by MBE was optimized first, followed by introducing Al for AlGa_N growth using similar flux in Ga-rich condition. During the 2D growth of AlGa_N on AlN/Si, surface nitridation effect on AlGa_N surface morphology was studied and optimized. It was previously demonstrated that covering Si substrate with a thin amorphous layer of 1-2 nm thickness was found to suppress the Si out-diffusion, with the amorphous layer acting as a diffusion barrier to prevent the Al-Si interdiffusion [255]. Single crystalline AlN can be grown on amorphous like SiN_x layer, and it is believed that the crystal orientation of the Si substrate is transferred to the AlN layer through the crystallized area or the short-range tetrahedral coordination of each atom is maintained to support the epitaxial relation [256, 257].

Besides, there is also the possibility that AlN seeds nucleate through this non-continuous thin SiN_x layer and a planar growth develops laterally to form an almost single-crystalline AlN epitaxial layer for further growth [258]. Similar to GaN, AlGa_N with smooth surface morphology was also achieved using SiN_x as the nucleation layer. It should be noted that the growth of AlGa_N using the two-step process is not possible as that will result in a varying Al/Ga composition in the AlGa_N layer. Thus, the use of SiN_x as a nucleation layer allows the growth of AlGa_N having fixed Al/Ga composition with smooth surface morphology. Thus, the amorphous like SiN_x layer, during the growth, assisted as a masking or a barrier layer that prevented any Ga-Si interdiffusion, thereby

circumventing the process of melt-back etching. In this study, AlN/Si (111) grown by MOCVD was also used as template for AlGaIn growth and doping investigation.

5.1 Growth of AlGaIn layer by PA-MBE

5.1.1 Surface nitridation of Si

Prior to AlN growth, Si substrate was thermally cleaned. This was confirmed by the observation of clean 7×7 RHEED pattern (Fig. 4.1). After plasma-striking successfully, RF power was fixed at 300 W and N_2 flow was graded down from 10 sccm to 2 sccm within 5 min for all the nitridation process. Following procedures were used for the Si surface nitridation:

1. Si (111) wafer was cleaned by heating up and maintaining at 830 °C for 30 mins followed by 910 °C for 10 mins. RHEED pattern of 7×7 on Si surface was observed.
2. Heater was ramped down to AlN growth temperature of 772 °C, plasma was initiated and the RF source shutter was opened.
3. The RF source shutter was kept open for 5 min to form amorphous SiN_x (β - SiN_x is formed if the shutter is closed).

RHEED pattern was taken to confirm the formation of SiN_x after exposing to nitrogen plasma for 5 min. Fig. 5.1 (a) shows no meaningful (amorphous-like) pattern while (8×8) pattern was observed, as shown in Fig. 5.1 (b) which matches the β - SiN_x .

To optimize SiN_x growth time and method, a series of GaN growths was done using the recipe as summarized in Table 5.1. As indicated, 6 growths were done, and it was found that amorphous SiN_x was formed when the RF plasma source shutter was kept open while β - SiN_x was formed

when the shutter was closed. Of all the growths, 2D GaN growth using Recipe 3 for surface nitridation provided the best quality GaN layer.

Figure 5.1 RHEED reconstruction pattern of (a) amorphous SiN_x (no pattern) and (b) $\beta\text{-SiN}_x$

Table 5.1 Surface nitridation using different condition

Recipe	Nitridation type	RF power (W)	N_2 flow (sccm)	Time (min)
1	$\beta\text{-SiN}_x$	500	10	10
2	$\beta\text{-SiN}_x$	300	10-2	5
3	amorphous SiN_x	300	10-2	5
4	amorphous SiN_x + Close shutter	300	10-2	3+2
5	amorphous SiN_x	300	10-2	5
6	amorphous SiN_x	300	10-2	20

5.1.2 AlN growth

AlN growth of sample 4, V857 (Table 4.1) was repeated on both amorphous SiN_x and $\beta\text{-SiN}_x$ formed on Si substrates. Since the Si substrate condition has changed from Si to SiN_x and AlN was

grown on SiN_x , RHEED pattern obtained for AlN was totally different from what was observed in Fig. 4.2 in Chapter 4.

Fig. 5.2 shows the RHEED patterns of AlN grown on SiN_x using amorphous SiN_x and $\beta\text{-SiN}_x$. The observed RHEED pattern of AlN on amorphous SiN_x was blurry as shown in Fig. 5.2(a), but (1×1) pattern can still be seen at the beginning (3 minutes into the growth) and at the end of AlN growth, as in Fig. 5.2(b), although not so bright as Fig. 4.2 (b) in Chapter 4. However, AlN grown on $\beta\text{-SiN}_x$ showed slightly different RHEED pattern, still blurred but spots can be seen with light rings connecting the spots in Fig. 5.2(c). At the end of AlN growth, RHEED was bright and spotty, and rings can be observed clearly as in Fig. 5.2(d).

Figure 5.2 RHEED pattern (a) 3 min into AlN growth on amorphous SiN_x , (b) end of AlN growth on amorphous SiN_x and (c) 3 min into AlN growth on $\beta\text{-SiN}_x$ (d) end of AlN growth on $\beta\text{-SiN}_x$

5.1.3 GaN and AlGaN growth

GaN growth was continued on both AlN grown on amorphous SiN_x and β -SiN_x formed on Si substrates following the same growth procedure with optimized growth/desorption time as described in Chapter 4. 2D GaN growth was optimized and AlGaN growth was done based on the optimized GaN growth recipe using fine-tuned Al and Ga flux. To obtain even smoother GaN and AlGaN, AlN/Si template grown by MOCVD was also used. For the samples grown on AlN/Si templates, AlN cleaning was conducted before growth by Ga-flashing using the following steps:

1. AlN template was heated up and maintained at 650 °C for 2 min before Ga was deposited on the template with a flux of 1.05×10^{-7} Torr.
2. After closing the Ga source shutter, AlN template was heated up and maintained at 850 °C for 5 min to fully desorb Ga on the surface.
3. AlN template was ramped down to 650 °C, and steps 1 to 3 were repeated for another 2 times.

This was followed by 300 nm GaN growth using the following steps:

1. Heater was cooled down to GaN growth temperature of 725 °C.
2. GaN growth was done in Ga-rich condition for 120 min (20 min growth + 2 min Ga desorption) $\times$ 6 cycles.
3. Heater ramped down to room temperature.

The same recipe as described above was used for AlGaN growth on AlN template with higher AlGaN growth temperature at 735 °C. Bright streaky (1 $\times$ 1) RHEED pattern of AlN was observed after the cleaning process as shown in Fig. 5.3 (a) indicating good AlN crystal quality [259]. Unlike the spotty RHEED pattern in Fig. 4.2 (b) of Chapter 4 and Fig. 5.2 (b), RHEED pattern observed

during AlGa_{0.7}N growth was streaky throughout the growth as shown in Fig. 5.3 (b), since the growth was started on the smooth AlN surface at the bottom.

Figure 5.3 RHEED pattern of (a) AlN after cleaning and (b) at the end of AlGa_{0.7}N growth

A series of AlGa_{0.7}N growths with different Al flux values were conducted to calibrate the flux required to grow AlGa_{0.7}N layer with a composition of 30%. The growths were done under Ga-rich growth conditions at a constant Ga-flux, and Al-composition was determined using XRD rocking curve analysis. Al-composition as a function Al-flux used during the growth is plotted in Fig. 5.4, and it can be observed that Al-composition increases linearly with Al flux.

Figure 5.4 AlGa_{0.7}N composition vs. Al flux, grown in Ga rich condition

5.1.4 Characterization of AlN on Si (111) with surface nitridation

Fig. 5.5 shows AFM images of (a) amorphous SiN_x with RMS roughness of 0.5 nm and (b) $\beta\text{-SiN}_x$ with RMS roughness of 1.4 nm. The surface morphology of amorphous SiN_x was observed to be the same as that of the Si substrate. However, typical hexagon features with gaps in between them are observed for $\beta\text{-SiN}_x$.

Figure 5.5 (a) AFM image of amorphous SiN_x and (b) $\beta\text{-SiN}_x$ on Si substrate

Fig. 5.6 shows cross-sectional TEM image of AlN grown on amorphous SiN_x layer on Si substrate. The thickness of amorphous SiN_x is approximately 2-3 nm. It can be seen that AlN layer, growing initially as an amorphous layer, becomes quickly single crystalline when the growth is continued. Bright spotty SAED (selective area diffraction) pattern shown as inset in Fig 5.6 confirms that AlN grown was single crystalline. It was also found that according to different SiN_x growth in Table 5.1, 3 min was sufficient for SiN_x to form and with time increasing no obvious improvement was observed on surface AFM morphology.

Figure 5.6 Cross-sectional HR-TEM image of AlN/SiN_x/Si interface and SAED pattern of AlN layer in inset

5.1.5 Characterization of 2D GaN and AlGaN on AlN/Si (111)

GaN/AlN grown on β -SiN_x shows rough surface with microcrystals and triangular-patterns as can be seen in Fig. 5.7 (a), but the same GaN/AlN structure grown on amorphous SiN_x is found smooth as in Fig. 5.7 (b). This can be attributed to the same issue of Ga/Si interdiffusion as discussed in detail in Chapter 4, and β -SiN_x is not effectively acting as a blocking layer for Ga and Si. β -SiN_x film is not dense when it was formed during the surface nitridation of Si substrate. In the β -SiN_x layer, large gaps exist in between 3D β -SiN_x crystals as shown in Fig. 5.5 (b), and the layer is also not thick or strong enough to withstand Ga diffusion into Si at high temperature. But, for the amorphous SiN_x, microcrystals are formed in this layer in different directions and this dense and thicker film blocks Ga diffusion. Hence, no microcrystal and triangular-pattern are seen on GaN surface as discussed in Chapter 4. Although no microcrystal was observed in 100×100 μm^2 AFM image shown as inset Fig. 5.7 (b), it clearly indicates that there are clusters of GaN, and such GaN

clusters can be fully eliminated by tuning the growth/recovery time for the fixed Ga flux during growth, indicating such clusters were caused by Ga accumulation on the surface during GaN growth.

Figure 5.7 Microscopy image of GaN/AlN grown on (a) β -SiN_x, with the magnified image in inset and (b) amorphous SiN_x (inset is 100×100 μm² AFM image)

The growth time was tuned down to 2.5 min so that excess Ga metal covering the surface within this period will not be too much and can be desorbed. Recovery time was also tuned to 55s accordingly so that no residual Ga remains on the surface before next GaN growth cycle starts to avoid formation of GaN clusters. This growth/desorption ratio was fine-tuned at fixed growth temperature of 725 °C and metal/gas flux ratio. Finally, smooth 2D GaN/AlN on amorphous SiN_x was achieved without any GaN clusters. Fig. 5.8 shows RMS roughness value of 0.4 nm obtained over 5×5 μm², and similar roughness even over a larger area of 100×100 μm² showing uniform surface morphology.

Figure 5.8 AFM image of optimized GaN/AlN grown on amorphous SiN_x

Cross-section TEM image was scanned on optimized 2D GaN/AlN grown on amorphous SiN_x. As shown in Fig 5.9, EDX line scan was conducted at several locations along the direction from Si to GaN layer. There is a sharp interface at each layer and no Ga/Si interdiffusion was found. Thus, it can be concluded that amorphous SiN_x rather than β-SiN_x is acting as an effective blocking layer to successfully achieve smooth microcrystal-free 2D GaN grown on Si.

Figure 5.9 EDX line scan conducted along the direction from Si to GaN layer and the corresponding EDX spectra on the right

Based on the optimized 2D GaN growth recipe, AlGaN with the same thickness of 300 nm was grown using the same recipe by introducing Al during GaN growth. Al-composition was confirmed by XRD rocking curve analysis. Since it is difficult to use normal scan for accurately determining Al-composition due to strain, reciprocal space mapping scan (RSM) [260, 261] was conducted and AlN was selected as relaxed substrate. To achieve clear RSM image, which is affected by AlN thickness, one AlGaN sample was grown for this purpose with the AlN thickness increased from 120 nm to 220 nm, keeping the AlGaN layer growth condition unchanged. As shown in Fig. 5.10, by simulating the peak position of AlN and AlGaN, Al-composition and relaxation can be easily obtained, and the values found are 300 nm thickness of AlGaN layer, 30% Al-composition and a relaxation of 44%.

Figure 5.10 XRD RSM mapping of 30% AlGaN on Si

AFM study was conducted on the 30% AlGaN sample optimized with smooth surface on 120 nm AlN buffer layer on amorphous SiN_x on Si substrate. As indicated in Fig. 5.11, 5×5 μm² AFM image shows RMS roughness of 0.8 nm. Since 30% AlGaN was intended for photodetector application, it is critical to improve the AlGaN crystal quality as well as surface morphology. Thus, AlN/Si template grown by MOCVD was chosen as substrate for 30% AlGaN growth.

Figure 5.11 Microscopy image of 30% AlGaIn/AlN/Si (111) grown by PA-MBE, inset is $5 \times 5 \mu\text{m}^2$ AFM image

Two types of 4 inch AlN/Si templates from different sources were used. One was commercially available AlN/Si template provided by a vendor, and the other was high-quality AlN template deposited in-house by MOCVD system. For easy comparison, as indicated in Table 5.2, AlN template from the vendor is named template A and the in-house deposited is named template B. Template B has already been used to study the effect of AlN in blocking Ga/Si interdiffusion in Chapter 4. As shown in Fig. 4.13 (a), template B surface is smooth with RMS roughness of 0.4 nm in $5 \times 5 \mu\text{m}^2$ area, and the thickness was around 230 nm to 240 nm.

Table 5.2 AlN from different sources

Growth type	Thickness (nm)	AlN (002) FWHM (arcsec)
MBE	105	1700
Template A	200	3528
Template B	240	2168

Fig. 5.12 (a) and (b) show the AFM images of optimized 30% AlGaN grown on template B with $5 \times 5 \mu\text{m}^2$ area and $100 \times 100 \mu\text{m}^2$ area, respectively and the RMS roughness measured was 0.8 nm. As shown in Fig. 5.12 (c), the surface morphology of template A was found rough with RMS roughness of 1.7 nm before the growth, and the value reduced to 0.8 nm after the growth as shown in Fig. 5.12 (d), similar to AlGaN grown on template B.

Figure 5.12 AFM image of (a), (b) 30% AlGaN grown on template B, (c) pristine template A and (d) 30% AlGaN grown on template A

AlN grown by MBE shows the best quality with thickness of only 105 nm. Template A and B have little difference in AlN-thickness. Template A is 200 nm thick, and AlN (002) XRD-FWHM measured was 3528 arcsec. However, template B has a thickness of 240 nm, and AlN (002) XRD-FWHM obtained was 2168 arcsec, which is much less than that of template A. It is clear that locally deposited AlN template is better than commercial one. Also, AlGaN grown on template B

shows better AFM surface morphology than that on AlN grown by MBE. Although with similar RMS roughness, template B is 4 inch in size which is preferred for mass production, while the MBE grown samples were on 2 inch Si in size. Hence, template B was chosen for the following Si doping study and for the growth of UV/IR dual band photodetector structures in Chapter 7.

5.2 Si doping of AlGaIn layer

With AlGaIn growth optimized on 2 inch Si substrate and 4 inch AlN/Si template, Si doping study as described in Chapter 4 for GaN was repeated for 30% AlGaIn grown using 4 inch AlN template on high resistive Si substrate. Hall-effect measurement was used to determine the dopant density in the Si doped AlGaIn samples. Fig. 5.13 shows that Si doping concentration in AlGaIn is also not linear with respect to Si cell temperature as was the case for GaN. Similar to Si doping in GaN, it can be divided into two regions with two incorporation efficiencies. At Si cell temperature of 1310 °C, the highest carrier concentration of $\sim 2 \times 10^{20} \text{ cm}^{-3}$ was achieved. To the best of our knowledge, for 30% AlGaIn on Si substrate grown by PA-MBE, the doping level achieved in this study is relatively high [262, 263]. During the AlGaIn and GaN doping growth, the growth condition was

Figure 5.13 Si doping concentration vs. Si cell temperature

tuned for AlGa_N growth. Growth temperature was increased 20 °C higher than Ga_N. Hence, the Si doping efficiency or the Si solubility in Ga_N and AlGa_N growth cannot be compared. Moreover, the growth rates for Si doped AlGa_N and Ga_N growth are different. With Si flux kept the same, the actual doped Si level of AlGa_N is observed to be higher than that for Ga_N.

Fig. 5.14 shows the AFM images of surface morphology of 30% AlGa_N with different doping concentration under the same growth conditions. As observed, when Si doping concentration is $1 \times 10^{19} \text{ cm}^{-3}$ and $5 \times 10^{19} \text{ cm}^{-3}$, RMS roughness in $5 \times 5 \text{ }\mu\text{m}^2$ area is 0.89 nm and 0.86 nm, respectively. When the doping concentration was increased to $1.4 \times 10^{20} \text{ cm}^{-3}$, RMS roughness decreased to 0.8 nm which may be within the measurement variation. For the highest carrier

Figure 5.14 AFM image of 30% AlGa_N on AlN template with different carrier concentration of (a) $1 \times 10^{19} \text{ cm}^{-3}$, (b) $5 \times 10^{19} \text{ cm}^{-3}$, (c) $1.4 \times 10^{20} \text{ cm}^{-3}$ and (d) $2 \times 10^{20} \text{ cm}^{-3}$

concentration of $2 \times 10^{20} \text{ cm}^{-3}$, RMS roughness increased to 0.94 nm which is still considerably smooth. Similar to Si doping effect on GaN surface morphology, AlGaN surface roughness increased with the increase of doping concentration, but is still under control when the epilayer is grown in Ga-rich condition. No clear observation was obtained on the effect of low Si doping on surface morphology. The AFM surface RMS roughness is roughly between 0.8 nm – 0.95 nm which may be because the AlGaN surface is slightly rougher than GaN and Si doped AlGaN is also slightly rougher. There would be some variation between different locations and the difference is small between different doping levels. The highest doped sample shows the highest RMS roughness of 0.94 nm which is relatively higher than the other 3 samples.

Temperature dependent hall-effect measurement was conducted on Si doped AlGaN samples with the carrier concentration of $6 \times 10^{19} \text{ cm}^{-3}$ and $2 \times 10^{20} \text{ cm}^{-3}$. Fig 5.15 shows the electron mobility and carrier concentration of these two samples as a function of temperature. Carrier concentration is found almost constant with the change in temperature, and the mobility is found slightly higher at low temperatures similar to GaN samples. From the temperature dependent hall-effect measurement, the activation energy values of $< 0.14 \text{ meV}$ was obtained for the doped samples.

Figure 5.15 Temperature dependent Hall-effect measurement on Si doped AlGaN with carrier concentration of (a) $6 \times 10^{19} \text{ cm}^{-3}$ and (b) $2 \times 10^{20} \text{ cm}^{-3}$

Considering that absolute strain calculation in AlGa_N by Raman measurement is difficult [264], high resolution XRD was utilized to calculate the strain developed in AlGa_N layer due to Si doping. Fig. 5.16 shows 2θ-ω XRD scan performed along AlGa_N (004) and AlGa_N (105) and the strain (%) in the layer was calculated using Eq. 5.1:

$$\text{Strain} = \frac{a - a_0}{a_0} \quad \text{Eq. 5.1}$$

where a_0 is lattice-relaxed constant and a is the measured strained lattice constant. For Al_xGa_{1-x}N, a_0 can be calculated by Vegard's law using Eq. 5.2:

$$a_0 = xa_{\text{AlN}} + (1 - x)a_{\text{GaN}} \quad \text{Eq. 5.2}$$

where a_{AlN} is 3.113 Å and a_{GaN} is 3.1899 Å, x is 0.3 for 30% AlGa_N. Strained lattice constant c can be calculated using θ taken from 2θ-ω XRD scan along AlGa_N (004) using Eq. 5.3:

$$c = \frac{4 \times 1.54}{2 \sin \theta} \quad \text{Eq. 5.3}$$

The lattice constant c can be converted to lattice constant a using Eq. 5.4:

Figure 5.16 XRD 2θ-ω scan performed along (a) AlGa_N (004) and (b) AlGa_N (105) plane

$$a = \frac{4}{\left(\frac{1}{d^2} - \frac{25}{c^2}\right) \times 3} \quad \text{Eq. 5.4}$$

With Si doping concentration increasing, the strain inside AlGaN layer will change from compressive to tensile strain. The value of d is calculated using θ taken from 2θ - ω XRD scan along AlGaN (105) by Eq. 5.3. As can be observed in Fig. 5.17, AlGaN is in compressive strain under low doping concentration and in tensile strain under high doping concentration. In between, there is a balance point making the layer stain-free, which can be used in future for strain control.

Figure 5.17 Strain in Si doped AlGaN calculated by XRD vs. Si doping concentration

5.3 Summary

2D GaN was grown on SiN_x formed by exposing Si substrate to nitrogen plasma at growth temperature. Different-quality SiN_x is formed depending on the nitridation method employed. If Si substrate is exposed to nitrogen plasma directly, the amorphous SiN_x will form while indirect exposing, by closing the source shutter, will lead to β - SiN_x . Amorphous SiN_x is found to be an effective blocking layer to Ga diffusion during 2D GaN growth, and β - SiN_x is not dense enough to stop Ga diffusion. GaN grown on amorphous SiN_x in Ga-rich condition was optimized to

achieve smooth surface with RMS roughness of 0.4 nm. Based on GaN growth, AlGaIn growth in Ga-rich condition was optimized in a similar way. AlGaIn layer with 30% Al-composition was grown and optimized on Si (111) substrate, AlN template deposited in-house by MOCVD and commercial AlN template. All AlGaIn layer growths were optimized to achieve smooth surface morphology with similar RMS roughness of 0.8 nm regardless of the bottom substrate.

Si doped AlGaIn layers exhibited free carrier concentration of 1×10^{19} , 5×10^{19} , 1×10^{20} and 2×10^{20} cm^{-3} , for the Si cell temperatures 1190 °C, 1250 °C, 1280 °C and 1310 °C, respectively. To the best of our knowledge, the value of 2×10^{20} cm^{-3} obtained is much higher than reported free carrier concentration achieved in Si doped 30% AlGaIn layer on Si by PA-MBE technique. Study on strain in doped AlGaIn found that lower Si doping produced 0.4% tensile strained layer and the highest doped layer resulted with 0.6% compressive strain.

Chapter 6 AlGaN based UV photodetector

Tunability of band gap and the possible heterostructure formation of group III-nitride materials allow the design of complex structures for UV detection. Development of such a technology on Si allows lower cost and opens the prospect of integration of III-nitride with Si technology. This chapter describes the growth of crack free AlGaN/GaN multiple structure on Si substrate with low dark current for UV photodetector applications. The same structure is also designed to be capable of working in near infrared region.

AlGaN/GaN multilayer structures have been grown on sapphire substrates with low Al-composition of 2.6% for UV photodetector, and the highest photoresponsivity achieved was 0.6 mA/W [85, 175]. Higher Al-composition in such layer structure is necessary to obtain photodetector with deeper UV detection region. However, realization of good quality layer structures with smooth surface is challenging due to increased Al-composition. Furthermore, to achieve the target of lower cost and better integration with Si technology, the whole epilayer device structure has to be heteroepitaxially grown on Si (111) substrate, making the whole growth even more complex and challenging.

In this Chapter, the AlGaN based UV photodetector structures, simulated and designed by Prof. A. G. Unil Perera's [265] group from Georgia State University, have been grown in-house by MBE in NTU. In addition to epitaxial growth, all the materials characterization of the grown structures as well as the device fabrication and characterization of photodetector for UV applications were conducted in NTU.

As shown in Fig. 6.1, the epilayer structure consists of n^+ -GaN-top contact/un-doped $\text{Al}_x\text{Ga}_{1-x}\text{N}$ / n^+ -GaN emitter/graded $\text{Al}_x\text{Ga}_{1-x}\text{N}$ / n^+ -GaN-bottom contact. The Al-composition in the un-doped $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layer was optimised to tune the cut-off wavelength.

Figure 6.1 AlGa_xN/GaN multilayer epistructure ($\text{Al}_x\text{Ga}_{1-x}\text{N}$ graded from $x = 0.23$ at the top to $x = 0.14$ at the bottom)

For better understanding of the UV detector, the energy band diagram simulated using Poisson-Schrodinger simulation is shown in Fig 6.2. For UV light, the electrons in CB will be excited to the VB under either positive or negative bias voltage, and these excited electrons will be collected at top or bottom n -Ga N . Incident UV radiation with the energy greater than the band gap of the AlGa N barrier is absorbed, generating an electron-hole pair. Excited carriers are then collected by the applied electric field. The interband response threshold corresponds to the band gap of the AlGa N barrier. Hence, using different AlGa N composition, the interband response can be tailored to different wavelength regions for detection of different UV radiation.

Figure 6.2 Unbiased energy band simulation of $\text{Al}_{0.18}\text{Ga}_{0.82}\text{N}$ multilayer structure

6.1 Growth of AlGaN/GaN multi-layer structure for UV photodetector applications

Based on the GaN growth optimization discussed in Chapter 4, 500 nm thick bottom GaN layer was grown on 2-inch Si substrate using two-step AlN growth followed by 3D to 2D GaN growth to achieve smooth bottom GaN layer. For the complete structure shown in Fig. 6.1, during the growth, nitrogen plasma RF power was fixed at 300 W and N_2 flow was maintained at 2 sccm. All the GaN layers were Si doped with a doping concentration of $5 \times 10^{18} \text{ cm}^{-3}$ using Si cell temperature of 1190 °C. The complete structure on Si (111) substrate was grown following the growth steps listed below:

1. Si (111) substrate was heated up and maintained at 975 °C for 30 minutes. The temperature was then ramped up to 1075 °C and kept for 10 minutes to remove the native oxide. Appearance of clear (7×7) RHEED pattern confirms that Si (111) surface is clean and the native oxide is completely desorbed (Fig. 4.1).
2. The substrate was cooled down to $T_p = 777$ °C and AlN was then grown at a low flux with Al/N ratio of 0.47 for 16 min.

3. Al flux was graded to higher level with Al/N ratio of 0.66 with the grading time of 5 min before the growth of AlN for 15 min.
4. The substrate was cooled down to GaN growth temperature $T_P = 715^\circ \text{C}$. Growth of GaN in N-rich condition was performed with the Ga/N ratio of 0.48 for 36 min with Si cell at 1190°C .
5. Ga/N ratio of 1.11 was used for GaN growth in Ga-rich condition with Si cell at 1190°C . The total growth time was 120 min, consisting of 6 cycles, each with 20 min of GaN growth followed by 2 min of Ga desorption.
6. AlGaIn layer growth: Al flux was graded from 1.38×10^{-8} to 2.36×10^{-8} Torr in 24 min.
7. Si doped GaN growth with Si cell at 1190°C for 22 min followed by 2.5 min of Ga desorption.
8. AlGaIn growth for 120 min with different Al-flux to achieve Al-compositions of 15%, 17% or 23% as described in Chapter 5 (Fig. 5.4).
9. Si doped GaN growth with Si cell at 1190°C for 60 min, consisting of 3 cycles, each with 20 min GaN growth followed by 2 min of Ga desorption.
10. Heater was ramped down to 200°C .

As shown in Fig. 6.3, at the end of bottom 3D GaN growth, spotty RHEED pattern with connected spots (a) was observed, typical of 3D growth mode. RHEED pattern became streaky throughout the entire growth as shown at different stages: at the end of bottom 2D GaN (b); end of graded AlGaIn (c); end of middle GaN (d); end of top AlGaIn (e) and end of top GaN (f). AlGaIn layers were grown without recovery as the bottom graded AlGaIn should be continuous and the top AlGaIn is sensitive to Ga flux, which would make it difficult to control the desorption time if Ga desorption were to be inserted during the growth.

Figure 6.3 RHEED pattern at the end of growth of (a) 3D GaN, (b) bottom GaN, (c) graded AlGaIn, (d) middle GaN, (e) top AlGaIn, and (f) after the growth

The complete structure was also grown on AlN/Si MOCVD grown-template for performance comparison following the growth steps listed below:

1. AlN template was heated up and maintained at 650 °C. Ga metal was deposited for 2 min on AlN template with flux of 1.05×10^{-7} Torr.
2. Ga source shutter was closed and the AlN template was heated up and maintained at 850 °C for 5 min to fully desorb Ga on the surface.
3. AlN template was then ramped down to 650 °C. Steps 1-3 were repeated for another 2 times.
4. AlN template temperature was ramped up to GaN growth temperature $T_P = 715$ °C.

After step 4, further growth proceeded similar to steps 5 to 10 as listed for the growth on Si (111) substrate.

During the growth, RHEED patterns obtained were streaky and similar to what had been observed for the growth on Si (111) substrate as illustrated in Fig. 6.3 (b to f). Table 6.1 lists the four optimized complete structure for AlGaIn based UV photodetector. These samples were grown with different top AlGaIn compositions (Fig. 6.1) of 17%, 23% and 15% on both 2-inch Si (111) substrate and 4-inch MOCVD grown AlN/Si template. Complete structures with Al-compositions

of 17% and 23% were grown on 2-inch Si substrate while complete structure with Al-compositions of 23% and 15% were grown on 4-inch AlN/Si template. All the growth on same substrates were following same recipe and RHEED was used to control each growth to repeat same RHEED pattern.

Table 6.1 Samples with different Al-compositions grown on different substrates

Sample	Al-composition	Substrate
1	17%	2" Si
2	23%	2" Si
3	23%	4" AlN/Si template
4	15%	4" AlN/Si template

6.2 Characterization of AlGaN/GaN multilayer structure

AlGaN/GaN multilayer structure were optimized to achieve smooth surface for different Al-compositions and substrates. Fig. 6.4 shows microscopy and AFM (inset) images of the optimized growth with smooth surface morphology. For AlGaN/GaN multilayer structure with the compositions of 17% and 23% on 2-inch Si substrate, similar surface RMS roughness of 0.7 nm was obtained. For multilayer structure with the compositions of 15% and 23% on AlN template the surface RMS roughness was less than 0.6 nm, showing better surface morphology.

Figure 6.4 Microscopy image and AFM image in the inset of AlGaIn UV detector structure of (a) sample 1, (b) sample 2, (c) sample 3 and (d) sample 4

To confirm the Al-composition, XRD scan was performed on each wafer with different Al-composition as shown in Fig. 6.5. As shown in Fig. 6.1 the designed Al-composition in the bottom AlGaIn should be increasing from 14% to 23% and the Al-composition in top AlGaIn should be 15%, 17% and 23%, respectively. According to the measured XRD profile in Fig. 6.5, the actual Al-composition is confirmed to be the same as designed. The Al-composition in the graded AlGaIn layer is measured and simulated by XRD to be 14% to 25%, 14% to 23% and 14% to 26.5% individually. Although sample 1 and 2 were grown on Si and sample 4 was grown on AlN template, XRD didn't show much difference on GaN and AlN layer.

Figure 6.5 XRD ω -2 θ rocking curve measured and simulated along (002) plane of the complete epistructure

Si-doping in GaN was calibrated as discussed in Chapter 4 (Table 4.4). To confirm the Hall results on Si-doping concentration, SIMS analysis was conducted on sample 1 (Table 6.1) and the SIMS profile is shown in Fig. 6.6. It is noted that the doping concentration determined by SIMS is close to the value obtained using Hall effect measurement. As shown in Table 4.4, for sample V878 with the Si-cell temperature of 1190 °C during growth, the corresponding carrier concentration should be around $5.5 \times 10^{18} \text{ cm}^{-3}$, which matches closely with the SIMS data of $6 \times 10^{18} \text{ cm}^{-3}$. The SIMS profile also reveals that Si-doping in the three GaN layers is quite uniform. The Al-profile also shows that AlGaN composition is constant in the top AlGaN layer while it is graded linearly in the

bottom AlGaIn layer. The thickness of each layer is also found to match with the designed layer thickness.

Figure 6.6 SIMS analysis of the AlGaIn/GaN multilayer structure shown in Fig. 6.1

To have better understanding on the strain in the epilayer for device fabrication, bow measurement was conducted immediately after the growth. Bow values were measured on AlGaIn/GaN multilayer structures with different Al-composition grown on 2-inch Si substrates and the bow values are listed in Table 6.2. Prior to the bow measurement, the same piece of Si (111) substrate before the epilayer growth was measured as a reference. All the bow values are found to be negative, which means the entire epilayer is under tensile stress. The stress was calculated automatically by the bow measurement system using the relation

$$\sigma_f = \frac{M_s h_s^2}{6 h_f R} \quad \text{Eq. 6.1}$$

where σ_f is the average stress on the plane of the film, M_s is the biaxial elastic modulus of the substrate, h_s is the substrate thickness, h_f is the film thickness, and $1/R$ is the system curvature [266]. From the bow result, it can be observed that the entire epilayer stress keeps reducing from 240 MPa to 129 MPa with Al-composition increasing from 15% to 23%. With highest Al-composition of 23%, the bow value measured is 11 μm , which is acceptable for device fabrication without cracking.

Table 6.2 Bow measurement data of AlGaIn/GaN multilayer structure with different Al-compositions on 2-inch Si substrate and the corresponding stress

<i>Sample</i>	<i>Al (%)</i>	<i>Bow (μm)</i>	<i>Stress (MPa)</i>
V887	15	-21.17	240.4
V888	17	-15.56	232.6
V890	18	-11.66	143.9
V893	23	-11.03	128.5

6.3 Device fabrication of UV photodetector

Two types of photodetector designs, namely, square detector and interdigitated detector were used to study the effect of photodetector configuration on its performance including photoresponsivity. The square detector has a mesa with a simple design area of $400 \times 400 \mu\text{m}^2$ as shown in Fig. 6.7 (a). The electrode width was fixed at 80 μm while the lateral gap between top and bottom electrodes was fixed at 60 μm . Interdigitated-type electrodes were used for the interdigitated detector as shown in Fig. 6.7 (b). Differing from planar structure of typical MSM-PD with metal-semiconductor Schottky contact, vertical device structure design with ohmic contact was employed for the photodetector in this study. It was designed with fixed interdigitated mesa width of 20 μm and fixed electrode width of 2 μm . The number of electrodes and gap between each top and bottom electrode pairs were varied to study the effect of device design on photoresponsivity. As shown in Fig. 6.7 (c), for both types of detector, the mesa contains two GaN layers and two

AlGa_xN layers. The structure was etched down to bottom *n*-Ga_{1-x}N layer and metal-stack was deposited on both the top and bottom *n*-Ga_{1-x}N to form electrodes.

Figure 6.7 Photodetector with (a) square and (b) interdigitated design, and (c) cross section of the device of AlGa_xN/GaN multilayer structure

The photodetector device fabrication process flow is shown in Fig. 6.8, which consists of 9 process steps. Two sets of masks were used for mesa etching and metal deposition. The device fabrication process is kept the same for both the square and interdigitated detectors. Sample 1 and 2 in Table 6.1 grown on 2-inch Si substrates were diced into 1 cm × 1 cm squares for device fabrication while the samples 3 and 4 that were grown on 4-inch AlN/Si substrate were diced into 1-inch × 1-inch squares for fabrication. Organic cleaning was performed by dipping the sample in boiling acetone for 10 min followed by ultrasonic cleaning for 5 min, and repeating the same for 3 times. The acetone was then changed to IPA and the same process was repeated followed by DI water flushing.

Figure 6.8 Device fabrication process flow

6.3.1 SiO₂ deposition by PECVD

Before PECVD, samples were organic cleaned and dried by baking on hot plate. The thickness of the wafer to be etched is 800 nm before ohmic metal deposition. Initially, photoresist was used for mesa etching protection. However, due to the etching selectivity and by product generated by photoresist, the etching surface was found poor and etching depth could not reach 800 nm. Thus, to protect the mesa, 500 nm SiO₂ layer was used as hard mask for etching instead of photoresist as shown in Fig. 6.9. SiO₂ was deposited on the sample surface using PECVD with N₂O and SiH₄ as reacting species under RF plasma at 300 °C.

Figure 6.9 SiO₂ deposition by PECVD

6.3.2 Photolithography of mesa pattern on SiO₂

SiO₂ absorbs water easily and the adhesion of the photoresist to the SiO₂ surface becomes very poor. Thus, developing of the photoresist becomes non-uniform and the patterned mesa is not precisely transferred in addition to poor adhesion of pattern on the SiO₂ surface during developing. In order to solve this issue, the sample was first organic cleaned to remove the particles followed by baking at 275 °C on a hot plate for 30 min. Once the sample cooled down to room temperature, it was spin coated with photoresist AZ5214E at a rotating speed of 4000 rpm for 30 seconds. The sample was then soft baked at 105 °C for 2 min. The photoresist thickness was kept at 1.4 μm. The UV exposure power during lithography was 500 mW for mesa mask. The AZ developer was diluted with DI water (the developer and water in the ratio of 1:1) and the developing time was set to 30 seconds. After developing, the mesa pattern transferred onto SiO₂ is shown in Fig.6.10.

Figure 6.10 Photolithography of mesa pattern on SiO₂

6.3.3 RIE of SiO₂ for hard mask and cleaning of photoresist

During the RIE etching of SiO₂ hard mask, the photoresist will also be etched at the same time. To reduce the photoresist etching rate, the sample was hard baked for another 2 min after developing. SiO₂ was etched by RIE using a mixture of gases, 30 sccm CF₄ and 30 sccm CHF₃, under RF forward power of 170 W with chamber pressure of 150 Torr at the room temperature of 20 °C. The etching rate was 100 nm/cycle, with each cycle was lasting 3 min. The total etching time used was 6 cycle × 3 min, and the RIE chamber was flushed by Ar and pumped down to remove etching by products in between each etching cycles. The 6 etching cycles was found to ensure that no residual SiO₂ was left on the exposed area and sharp SiO₂ hard mask is achieved for the next processing step. After removing the photoresist using organic cleaning, the sample was cleaned by ozone plasma for another 20 min. SiO₂ hard mask was obtained as shown in Fig. 6.11.

Figure 6.11 RIE of SiO₂ for hard mask and cleaning of photoresist

6.3.4 ICP-RIE for detector mesa

Since 500 nm thick SiO₂ layer was used as a hard mask, long time dry etching can be applied. Since no reliable wet etching process was available for fabricating AlGaN/GaN mesa, ICP-RIE was used to achieve fast and smooth etching. The etching recipe was optimized under different conditions of chamber pressure, substrate temperature as well as gas flow. The final optimized recipe used was a mixture of 10 sccm of Cl₂ and 40 sccm of BCl₃ under RF forward power of 60

W and ICP power of 300 W with the chamber pressure of 5 Torr at 20 °C. Etching rate was 40nm/min, each cycle was 5 min. The total etch time was 4 cycle $\times$ 5 min, and the ICP-RIE chamber was flushed by Ar and pumped down in between each etching cycle to remove etching by products . Finally, the mesa covered with SiO₂ was obtained as shown in Fig.6.12.

Figure 6.12 ICP-RIE for detector mesa

6.3.5 Hard mask removal by BOE

The SiO₂ hard mask was removed by etching in buffer oxide etchant (BOE) for 3 min, BOE is a buffered HF containing NH₄F and HF. After BOE etching, the sample was organic cleaned and dried. The device cross section after the hard mask cleaning is shown in Fig. 6.13, Mesa height was measured using a surface profiler before and after SiO₂ etching to confirm the presence of SiO₂ mask layer as well as to determine the mesa height of the multilayer structure.

Figure 6.13 Hard mask removal by BOE

6.3.6 Photolithography for metal contacts

The sample was spin coated with AZ5214E negative photoresist at a rotating speed of 4000 rpm for 30 seconds to realize a photoresist thickness of 1.4 μm . The sample was then soft baked at 105 $^{\circ}\text{C}$ for 2 minutes. After the first UV exposure with the power of 50 mW using electrode mask, the sample was baked on a hot plate at 120 $^{\circ}\text{C}$ for 2 min. The sample was then flood-exposed under UV power of 500 mW. Diluted AZ developer was used similar to mesa processing. The cross-sectional schematic of the structure after pattern transfer is shown in Fig.6.14.

Figure 6.14 Photolithography for metal contacts

6.3.7 Metal deposition for electrodes by eBeam evaporation

For contact electrode, electron beam evaporation was techniques was employed to deposit multilayer metal stack of Ti/Al/Ti/Au (30nm/80nm/120nm/200nm) (Fig. 6.15). For all the metal-layer deposition, the electron beam voltage was kept at 5 kV and the deposition rate was controlled

Figure 6.15 Metal deposition by eBeam evaporation

by current. The initial Ti layer was deposited at a low rate to achieve good contact with the top/bottom GaN layer. The deposition rate was thus kept at 0.3 nm/min for the first 3 nm and then the rate was increased to about 1 nm/min. Al deposition rate is fast and difficult to control as Al melting point is low. To get good Al layer, the deposition should be controlled at low beam current lower than 30 mA. For the middle Ti layer, the deposition rate was adjusted to high rate of 2 - 3 nm/min. Finally, Au layer was used to protect the Ti/Al/Ti layer below and prevent any oxidization. The Au deposition rate was controlled to 3 - 4 nm/min to avoid overheating of the photoresist, which otherwise would be difficult to remove.

6.3.8 Photoresist lift off

Fig. 6.16 shows the structure after photoresist lift off. After eBeam deposition, the sample was kept in acetone for several hours to let the photoresist fully dissolve or AZ stripper was used to remove the photoresist before ultrasonic cleaning. Since the electrode pattern is small, ultrasonic power is too strong to be applied. In this case, acetone injector was used to flush away photoresist after soaking the sample in the acetone. The needle tube was modified to have smaller tube diameter to improve flow pressure. This was found to be the best method to remove photoresist covered on extremely thin interdigitated electrodes without any damage.

Figure 6.16 Cross-section of detector after photoresist lift off

6.3.9 Annealing for ohmic contact

To achieve ohmic contacts on GaN layer, the samples after photoresist lift off were organic cleaned and annealed using rapid thermal processing (RTP). During annealing, the sample was heated up to 800 °C within 1.5 min and maintained at that temperature for 2 min. Nitrogen gas was used during RTP to avoid oxidization, and finally, the sample was cooled down to room temperature with a low ramp down rate.

6.3.10 Metal deposition for wire bonding by eBeam evaporation

Sample was mounted to IC package and wire bonding was done between the package-pad and electrodes. To achieve good bonding between the gold wire and annealed contact pad, a second layer of 300 nm Au was deposited by ebeam evaporation on the first annealed contact pad (Fig. 6.17). The photolithography procedure adopted for the second gold layer is the same as the first contact layer described.

Figure 6.17 Gold deposition for wire bonding by eBeam evaporation

After a series of wafer processing, photodetectors with different designs were achieved. As shown in Fig. 6.18, (a) square photodetector and (b) interdigitated photodetector with clean mesa and smooth bottom contact layer were achieved, gold contact is rough due to annealing process. For interdigitated photodetector, the electrodes were aligned and placed at the center of the crossfinger mesa to make full use of the mesa area.

Figure 6.18 Photodetector with different designs: (a) square detector and (b) interdigitated detector

6.4 Device characterization of UV photodetector

Devices with different designs fabricated on both the epistructures, grown on Si and AlN/Si template, were characterized. Dark current was measured at room temperature and devices with similar dark current density and effective electrical/optical mesa area were chosen for photoresponsivity measurement as listed in Table 6.3.

Table 6.3 AlGaIn/GaN multilayer structure grown on different substrates with different designs for UV detector

Sample No.	Photodetector Design	Mesa dimension (μm^2)	Number of Electrodes	Distance between electrodes (μm)	Al composition	Substrate	Responsivity (A/W)
1	Square	400×400			17%	2" Si	1.9
2	Square	400×400			22%	2" Si	0.4
3	Square	400×400			23%	4" AlN/Si template	0.5
4	Interdigitated		10	15	23%	4" AlN/Si template	5.7
5	Interdigitated		10	15	15%	4" AlN/Si template	4.7
6	Interdigitated		10	25	15%	4" AlN/Si template	1.8
7	Interdigitated		20	25	15%	4" AlN/Si template	2.2
8	Interdigitated		30	25	15%	4" AlN/Si template	3.6
9	Interdigitated		10	20	15%	4" AlN/Si template	3.5
10	Interdigitated		10	30	15%	4" AlN/Si template	1.5

6.4.1 I-V measurement

I-V measurement was done using Agilent B1500A semiconductor device analyzer. Dark current was measured for all the devices listed in Table 6.3. Fig. 6.19 shows the dark current characteristics when positive bias was applied to the top electrode and bottom electrode was grounded. The asymmetric dark current is caused by the asymmetric barrier designed in this detector structure [187, 267]. Samples 1 to 10 were grown with different Al-composition on different substrates and

the devices were fabricated with different device designs, dark current density ranging between 10^1 to 10^3 A/m² was achieved for devices with similar mesa area.

Figure 6.19 I–V characteristics of AlGaIn/GaN multilayer photodetector with square mesa (sample 1-3) and interdigitated mesa (4-10) design

6.4.2 UV Photoresponsivity measurement

Photoresponsivity of the devices were measured using a home-made measurement system. Responsivity data with respect to wavelength was obtained with the wavelength increasing from 270 nm to 400 nm in steps of 2 nm. The measurement was conducted at different positive bias voltage applied to top electrode while keeping the bottom electrode grounded. The sample surface area illuminated is 5 mm × 5 mm covering the whole detector. Calibrated Si photodetector was used to determine the incident light power density P_{Beam} , which is the ratio of the Si detector photocurrent I_{Si} and the product of light beam size A_{Beam} and known value of Si-detector responsivity R_{Si} , as given by Eq. 6.2:

$$P_{Beam} = \frac{I_{Si}}{A_{Beam} \cdot R_{Si}} \quad \text{Eq. 6.2}$$

Then, the device responsivity R_{Device} can be calculated using the measured device photocurrent I_{Device} , effective area illuminated A_{Device} and light power density P_{Beam} as given in Eq. 6.3:

$$R_{Device} = \frac{I_{Device}}{A_{Device} \cdot P_{Beam}} \quad \text{Eq. 6.3}$$

Fig. 6.20 shows the photoresponsivity spectra of sample 1, grown directly on Si-substrate (Table 6.3) with square detector design, measured at different applied bias voltages. The first peak lies around 330 nm which matches with the designed bandgap corresponding to $\text{Al}_{0.17}\text{Ga}_{0.83}\text{N}$. The second peak near 360 nm matches with the bandgap of the GaN. From the plot, it can be clearly seen that the background noise is much lower than the two peaks, there is a peak in between 360 nm and 370 nm which is caused by grating changing during measurement. The responsivity of the detector is found to increase as the applied bias voltage is increased. For comparison on the effect of design and different substrates used on the photoresponsivity, all the subsequent measurements were conducted at a constant bias of 3 V.

Figure 6.20 Photoresponsivity at different bias voltages of sample 1

Fig. 6.21 (a) shows the comparison of photoresponsivity spectra of square detector samples 1 and 2 on Si substrate with Al-compositions of 17% and 23%, respectively. They show similar

responsivity with responsivity peak shifting to shorter wavelength for the sample with higher Al-composition. Fig. 6.21 (b) shows the comparison of photoresponsivity spectra of samples 2 and 3 with similar Al-composition (22% and 23%, respectively) grown on Si substrate and AlN template, respectively. They exhibit similar responsivity of about 0.45 A/W. Thus, for the square detector design, the substrate used for the growth do not seem to affect the photoresponsivity, and this may also indicate that the quality of the epilayer grown directly on Si is comparable to that on AlN template.

Figure 6.21 Photoresponsivity of detector on different substrates of Si (sample 1 and 2) and AlN template (sample 3)

Fig. 6.22 (a) compares the photoresponsivity spectra of sample 3 with square detector design and sample 4 with interdigitated design with the same device structure grown AlN template. It can be observed that with same Al-composition and same substrate-type, sample 4 exhibits much higher responsivity than sample 3, which means that interdigitated detector provides higher efficiency. Samples 4 and 5 with the same interdigitated electrode design and same substrate, but grown with different Al-composition, reveals responsivity curve shifting to longer wavelength when the Al-composition is decreased from 23 to 15%, as expected, Fig. 6.22 (b). The value of responsivity

determined for both the samples is close to 5 A/W. Hence, for interdigitated electrode design the detection wavelength shifts to shorter range with higher Al-composition which is similar to the trend observed for sample 1 and 2.

Figure 6.22 Photoresponsivity of detector with square (sample 3) and interdigitated (sample 4 and 5) mesa design

Fig. 6.23 compares the photoresponsivity spectra of samples 6, 7 and 8 which have the same lateral distance between the top and bottom electrodes but with different number of electrodes (10, 20

Figure 6.23 Photoresponsivity of interdigitated detector with different electrode numbers and distance

and 30). With the number of electrodes increasing in the interdigitated detector design, it offers higher responsivity. While for samples 5, 6, 9 and 10 which have the same number of electrodes, but different lateral distances between the top and bottom electrodes, higher responsivity is achieved when the electrode-gap is decreased.

The higher responsivity obtained for interdigitated detectors fabricated on Si substrates compared to the reported value on sapphire substrate can be attributed to the internal gain. GaN grown on sapphire has already been optimized, however GaN grown on Si is still far from reaching lower defect density. These defects such as traps and acceptor impurities can trap the minority carriers (holes) which maybe the cause of the photoconductivity gain mechanism [268 - 271]. The lower responsivity of square detector may be because of the absorption of incident light outside the region in which photogenerated carriers can be collected by the electrodes [272]. Hence, with similar detecting area, more electrode pairs help to increase the collection efficiency. For interdigitated detectors, according to the simple theory of photoconductivity, the voltage responsivity R_V can be expressed by Eq. 6.4:

$$R_V = \frac{q\lambda}{hc} \frac{\eta\tau\mu_e V_b R_D}{L^2} \left(1 + \frac{1}{b}\right) \quad \text{Eq. 6.4}$$

where q is the electron charge, λ is the wavelength, μ_e is electron mobility, τ is the effective life time, η is the quantum efficiency, b is the electron-to-hole mobility ratio, L is the length between electrodes, V_b is the bias voltage, and R_D is the detector resistance. Therefore, for a given structure, shorter electrode gap results in higher responsivity, and higher bias voltage would also increase the responsivity [273], which match with the results observed for samples 1 to 10.

6.5 Summary

Two-step AlN growth together with 3D to 2D growth transition helped to optimize the bottom GaN layer in the multilayer detector structure grown on Si substrate. Optimized crack free AlGaIn/GaN multilayer structures for photodetector application were successfully grown on 2-inch Si substrate as well as 4-inch AlN/Si template grown by MOCVD with similar RMS surface roughness of less than 1 nm. Three AlGaIn/GaN multilayer structure photodetectors with Al-compositions of 15%, 17% and 23% were grown to achieve corresponding cutoff wavelengths. All these structures exhibited smooth regardless of the Al-composition.

Two different photodetector designs of square detector and interdigitated detector were fabricated on different AlGaIn/GaN multilayer structures grown by MBE on both Si substrate and AlN/Si template. Fabrication process was optimized and SiO₂ hard mask rather than photoresist was used for dry etching GaN and AlGaIn. Modified ICP-RIE recipe was used to achieve clean etching-surface of AlGaIn/GaN for ohmic contact.

Photodetectors showed dark current density of $10^1 - 10^3$ A/m² under 5 V bias regardless of detector design (square and interdigitated) and the substrate-type (Si substrate or AlN template). It was confirmed that, for the same detector design, the photoresponsivity was found to be the same for structures grown on both Si substrate and AlN/Si template. Square photodetector showed improved responsivity of 1 A/W under 3 V bias which is three orders higher in magnitude than the previously reported results [177, 178] while the interdigitated detector showed even higher responsivity of 5.7 A/W. Considering the fact that the reported photodetector was grown on sapphire substrate, it was not difficult to achieve such structure with good crystal quality and smooth surface morphology, due to the less thermal mismatch and lattice mismatch. It may be noted that the

photodetector described in this chapter was grown on Si substrate and still exhibited lower dark current and much higher responsivity.

As per design, compared to square design structure with similar detecting area, the interdigitated detector shows higher efficiency. For the interdigitated detectors which have the same lateral distance between the top and bottom electrodes but with different electrode numbers, when the number of electrodes is increased, the detectors tend to exhibit higher responsivity. For interdigitated detectors which have same electrode numbers but with different lateral distances between the top and bottom electrodes, when the electrode gap is decreased, the detectors also tend to show higher responsivity. The photoresponsivity tend to be higher for an interdigitated detector with more detecting area and short lateral electrodes gap.

Chapter 7 AlGaN based UV/IR Dual Band

Photodetector

Optimized crack free AlGaN/GaN multilayer structures for UV photodetector application have been successfully grown on 2-inch Si substrate as well as 4-inch AlN/Si template grown by MOCVD. Two different UV photodetector designs of square detector and interdigitated detector were fabricated. It was found that interdigitated photodetector design gives better UV photoresponsivity. Based on the optimized growth recipe of GaN and AlGaN used in Chapter 6, more advanced AlGaN/GaN based multilayer structures have been grown on Si substrate in this work. Moreover, AlGaN based UV/IR dual band photodetector can be realized based on intraband/interband transition mechanism explained in Chapter 2. It is considerably challenging to heteroepitaxially grow such complex structure on Si (111) substrate as well as to design the photodetector structure to achieve the best photoresponse.

GaN and other III-nitrides (N) are the most appealing materials for monolithic integration with Si electronics, as the epitaxial growth of GaN over Si substrates is an established and mature technology both in power electronics [274] and optoelectronics[275]. Among the potential monolithic integrated detectors, Si and GaN interband photodetectors are limited by their band gap energy- wavelengths shorter than 1.1 μm and 350 nm, respectively. Longer wavelength infrared (IR) photodetectors, especially imaging detector arrays are commonly implemented by the conventional hybrid approach in which silicon circuitry and III-V or II-VI infrared sensor (InGaAs, InSb, HgCdTe) are fabricated separately, and then combined by flip chip bonding[276], indium bump technology [277] or wafer bonding [278]. This approach is limited by interconnect size and

losses, which affects performance, restricts the minimum pixel and chip sizes and augments the cost and complexity [279-282]. Another method is GeSn photodetectors grown on silicon, which have been reported to extend the detection wavelength up to 2.4 μm [283], yet further extension of the operating wavelength is challenging [284].

Optoelectronic devices that can be epitaxially grown on silicon wafers and operate at a wide range of wavelengths using the same material system are very much desired. This can be met by using GaN-based intersubband transition (ISBTs) devices. ISBT is the optical excitation of an electron in a quantum well (QW) from its ground state to a higher energy state [285], the transition energy can be easily and precisely tuned by proper design of the well-width and barrier height. As a result, ISBT photodetectors can be implemented over a very wide-spectrum, by using the same material system and growth process, but only modifying the width of the layers and the barrier composition. Indeed, III-N ISBT detectors have been demonstrated at a spectral range from terahertz radiation [286] to near infrared [287]. Moreover, ISBT is an excellent technology for ultrafast optoelectronics due to the strong electron-phonon interaction which results in extremely short absorption recovery times [288,289]. Overall, epitaxial growth of III-N based heterostructures enables realization of integrated photonic devices, which will be fast, reliable, high-yield and cheap, operating over a very wide spectral range.

Most common ISBT devices are quantum well infrared photodetectors (QWIPs) [290] and quantum cascade detectors (QCDs) [291,292], both consist of periodic unit cells with an active absorption QW and an electron transport stage where photoexcited electrons are transferred across the device. In QCD, the excitation-transport process gives rise to photovoltage at zero bias. The transfer of photoexcited electrons in the extractor stage is achieved via resonant or phonon-assisted tunneling. The transport is optimized by proper alignment of the levels, which is imperative, and

is achieved by careful design of the widths and compositions of the QWs. For III-N devices, we can exploit the strong polarization-induced internal electric field to design an efficient AlGaN electron extractor where the energy levels are separated by the longitudinal optic (LO) phonon energy, such as in the first III-N QCD demonstrated by Vardi et al [293].

In this Chapter, the AlGaN/GaN based UV/IR dual band photodetector structures, simulated and designed by Prof. Gad Bahir and Prof. Meir Orenstein's [294] group from Technion University, have been grown in-house by MBE in NTU. In addition to epitaxial growth, all the materials characterization of the grown structures were carried out in NTU. While the device fabrication and characterization of photodetector for UV applications were conducted in NTU, the photodetector fabrication and device characterization for IR applications were performed in Technion. As shown in Fig. 7.1 (a), the photodetector structure on Si substrate consists of top and bottom Si doped AlGaN as electrodes and N periods of AlGaN/GaN superlattice (SL) structure in between these electrodes. In each SL, the active layer is a GaN-QW whose width is 3.2 nm (12 ML) with Si doping of $2 \times 10^{18} \text{ cm}^{-3}$. There are 3 GaN-extractors in SL, each one has a width of 1.5 nm, and the AlGaN-barrier layer width is set to 1.5 nm (6 ML). The conduction band diagram of the active periodic structure, calculated by a Schrödinger- Poisson solver [295], is shown in Fig. 7.1 (b) together with the squared envelope functions of the bound states. The vertical red arrow indicates ISBT from the ground state (e_1) to the upper state (e_2), corresponding to the transition energy of 267 meV (wavelength of 4.49 μm). The diagonal gray arrows indicate the direction of electron flow between the bound levels in the extractor ($e_2 \rightarrow e_3 \rightarrow e_4 \rightarrow e_5 \rightarrow e'_1$), which is facilitated by LO phonon assisted tunneling. Macroscopically, the excitation-transport process generates photovoltage across the device.

Figure 7.1 (a) Photodetector layer structure on Si substrate and (b) Calculated conduction band profile of one and a half period of the QCD's active area and shifted squared envelope functions (e_i , $i = 1 \dots 5$) of the electronic bound states. The red vertical arrow indicates optical transition, the gray arrows indicate transport direction of the electrons in the extractor.

7.1 Growth of AlGaN/GaN multilayer structure for UV/IR dual band photodetector

The AlGaN growth on Si-substrate and AlN/Si template have already been optimized as discussed in Chapter 5. In this Chapter, the growth and characterization of photodetector structure (Fig 7.1) grown on both 4-inch Si and 4-inch AlN/Si template have been discussed. During the growth, RF power for the nitrogen plasma was fixed at 300 W and N_2 flow was maintained at 2 sccm. However, during growth, the heater temperature was kept at 725 °C for all GaN and AlGaN layers. Growth rate for AlN, GaN and AlGaN at 725 °C was recalibrated to achieve accurate growth control during SL growth. Separately, single layers of Si doped GaN and AlGaN were grown at 725 °C based on GaN and AlGaN growth optimization described in Chapter 5, and smooth surface morphology and

precise doping concentration were obtained for the grown *n*-GaN and *n*-AlGaN (Fig. 7.1). Al and Ga fluxes were maintained constant throughout growth without any adjustment. The following steps were employed for the growth of complete structure on AlN/Si template:

1. AlN template was heated up and maintained at 650 °C for 2 min before depositing Ga with a flux of 1.05×10^{-7} Torr for 2 min.
2. After closing the Ga source, AlN template was heated up and maintained at 850 °C for 5 min to fully desorb Ga on surface.
3. AlN template was ramped down to 650 °C, and steps 1 to 3 were repeated for another 2 times.
4. AlN template was ramped up to AlGaN growth temperature $T_P = 725$ °C, and Al and Ga fluxes were brought to 9.28×10^{-8} Torr and 7.98×10^{-7} Torr, respectively.
5. AlGaN layer was grown in Ga-rich condition with Si cell at 1250 °C for 37.5 min, (2.5 min growth + 55 sec Ga desorption) $\times$ 15 cycles.
6. SL growth was performed under Ga-rich condition with GaN and AlGaN growth time of 12.8 sec and 12.1 sec, respectively. After each period of growth, all the source shutters were closed for 15 sec for Ga desorption process to complete.
7. Repeat SL for N cycles, followed by AlN growth for 25.6 sec.
8. AlGaN was grown in Ga-rich condition with Si cell at 1230 °C for 20 min (2.5 min growth + 55 sec Ga desorption) $\times$ 8 cycles.
9. Heater was ramped down to 200 °C.

The same structure was also grown on 4-inch Si substrate, and the growth process followed the same recipe based on AlGaN optimized growth on Si as described in Chapter 5, and the only difference is the growth process employed for the bottom AlN and AlGaN that includes formation

of amorphous SiN_x to prevent Ga/Si interdiffusion. The remaining growth process steps are the same as steps 4 to 9 used for the growth on AlN/Si template. All AlGa_xN and GaN growth steps were maintained in Ga-rich conditions, and bright streaky RHEED patterns were observed. Ga-desorption during growth is critical to achieve smooth surface.

7.2 Characterization of AlGa_xN/GaN multilayer structure

Initially, a series of complete AlGa_xN/GaN multilayer structure with 30 periods of superlattice was grown on AlN/Si template with different recovery time as listed in Table 7.1. Since this growth was unoptimized (growth recipe as described above), the AFM image of sample 1 showed AlGa_xN clusters and pits on top of AlGa_xN surface with RMS roughness of 6.4 nm in 100 × 100 μm² as shown in Fig. 7.2 (a).

Table 7.1 Recovery time during the growth of AlGa_xN/GaN multilayer structure with 30 periods SL on AlN/Si template

Samples	1	2	3	4
Top AlGa _x N Recovery time (sec)	55	N.A.	55	80
SL Recovery time (sec)	15	15	12	12

To locate in which layer these AlGa_xN clusters were formed, the same growth was repeated without top AlGa_xN layer (sample 2). It turned out that the surface had no AlGa_xN clusters, and the pits became much denser as in Fig. 7.2 (b). The RMS roughness has also reduced to 1.8 nm. It was found out that such pits were formed in SL layer since the bottom AlGa_xN was already optimized with smooth surface as described in Chapter 5. Since the pits were formed due to insufficient Ga, Ga desorption time during SL growth was decreased to avoid pits by keeping sufficient Ga on surface. Various manually controlled growths were tried to decide the Ga desorption time during SL growth based on RHEED pattern changing from streaky to spotty-like pattern. However, most

ended up with the poor surface with pits as shown in Fig. 7.2 (b). Finally, during the various growth, the Ga desorption time was tuned to be 12 sec to achieve pits free SL surface. The desorption time during SL growth is critical and the window is very narrow of only 12 sec. RHEED pattern observation was not taken as reference for SL-growth control since RHEED pattern changing from streaky to spotty would take 3 to 5 sec, which is close to the narrow Ga desorption window of 12 sec. It means that during growth, the growth condition changing from Ga-rich to N-rich may take only 3 to 5 sec. If the Ga desorption time during SL growth is longer or shorter than the desorption window, it may result in rough surface morphology with either pits or AlGa_N clusters.

Figure 7.2 AFM image of AlGa_N/Ga_N multilayer structure grown on AlN/Si template with different recovery times used in each layer as listed in table. 7.1

The complete structure (sample 3) was grown with optimized bottom AlGaIn layer and SL layer. The top AlGaIn layer growth still needed further fine tuning because the AFM image revealed rough surface with AlGaIn clusters, but without pits as shown in Fig. 7.2 (c). The RMS roughness measured was 2.0 nm. It was clear that such AlGaIn clusters were formed due to excess Ga during top AlGaIn growth. Ga flux was fixed during growth to avoid any growth interruption which may lead to degradation of the AlGaIn/GaN interface. Hence, the Ga desorption time was increased to 80 sec during the growth of top AlGaIn layer to achieve smooth surface as shown in Fig. 7.2 (d), and the RMS roughness measured was 0.66 nm.

Figure 7.3 AFM image of AlGaIn/GaN multilayer structure with (a) 40 SL grown on Si substrate and (b-c) 20 SL, 30 SL, 40 SL grown on AlN/Si template

Complete structures with SL periods of $N = 20, 30$ and 40 were optimized. As shown in Fig. 7.3, AlGaIn/GaN multilayer structure with 40-period SL grown on 4 inch Si substrate showed RMS

roughness of 0.8 nm in $5 \times 5 \mu\text{m}^2$ and the same structure grown on AlN/Si template with different SL periods of 20 SL, 30 SL and 40 SL showed similar RMS roughness of 0.78 nm, 0.78 nm and 0.76 nm, respectively. Although growths on both substrates showed similar surface roughness, further studies were conducted only on AlN/Si templates as described in Chapter 5.

It was found that Ga-rich condition is necessary during growth to achieve crack-free epilayer on 4-inch AlN template. As shown in Fig. 7.4 (a) and (b), very shallow cracks can be observed, and the AFM image shows pits together with AlGaN clusters, indicating insufficient Ga during SL growth. However, when growth was maintained even in Ga-rich condition, multilayer structures

Figure 7.4 (a) Microscopy and (b) AFM image of the epilayer with 30 SL as well as (c) microscopy and (d) SEM image of same structure with 40 SL

with 40 SL still cracked several days after the growth. Fig. 7.4 (c) shows the optical microscopy image of shallow but dense cracks and SEM picture in Fig. 7.4 (d) clearly confirms that the crack is on the epilayer surface, but not inside the layer. In order to achieve better epilayer quality and crack-free surface, SL period was reduced to 30 for further growth optimization of the detector structure.

As shown in Fig. 7.5, the detector structure with 30 SL grown in Ga-rich condition exhibited bow of $4.5\ \mu\text{m}$ (a) and the other sample grown with insufficient Ga (b) showed bow of $12\ \mu\text{m}$. This indicates the importance of maintaining precise Ga flux during growth for strain control. To avoid any crack and also achieve smooth surface morphology, the growth time and recovery time in the recipe were fine tuned. Finally, crack free complete structure with 30 periods SL was successfully

Figure 7.5 Bow measurement of multilayer structures with 30 SL grown in (a) Ga-rich condition and (b) Ga insufficient condition

Figure 7.6 AFM image of the epistructure with 30 SL

grown with RMS roughness of only 0.46 nm as shown in Fig. 7.6, which will be used for further device fabrication and device characterization.

HR-XRD scan of ω -2 θ rocking curve along (002) plane of the epilayer structure was performed to determine the quantum well thickness. Fig. 7.7 shows the measured and simulated symmetric rocking curves for the structure with 30 period SL. It can be clearly observed that there are several strong peaks including satellite peaks generated in the XRD scan corresponding to GaN, AlN, and AlGa_{0.3}N peaks matching the designed composition of 30 %. The simulation curve also shows that well controlled AlGa_{0.3}N thickness of 1.5 nm is obtained.

Figure 7.7 The experimental and simulated XRD ω -2 θ rocking curves along (002) plane of the epistructure shown in Fig. 7.1

Cross section Aberration-Corrected (AC) TEM was used to analyze the grown detector structure. As shown in Fig. 7.8, the SL structure can be clearly observed and the thickness measured matches

with the result simulated by the XRD scan. Sharp AlGa_N/Ga_N interface in the superlattice can also be seen in higher magnification. The dark layer seen is AlGa_N while the bright layer corresponds to Ga_N.

Figure 7.8 Cross-section TEM image of multilayer structure with 30 SL

7.3 Device characterization of UV/IR dual band photodetector

UV/IR dual band photodetector was fabricated following the same procedure used for UV detector fabrication described in Chapter 6. Interdigitated design was used for UV detection of dual band detector. Interdigitated detector fabricated has 30 electrodes with electrode-width of 2 μm . Lateral distance kept between the two electrodes is 15 μm and electrode length is 400 μm . The reason for using this dimension-combination is based on the UV responsivity results obtained in Chapter 6. As described, more electrodes and less lateral electrode gap would lead to higher photoresponsivity. All the fabrication process was the same except ICP-RIE etching depth that was changed to 600 nm to reach the bottom contact layer.

7.3.1 I-V measurement

The inset in Fig. 7.9 shows the optical microscopy image of the photodetector fabricated with clear etching surface and metal deposition is well aligned on interdigitated mesa. *I-V* measurement was conducted, and the dark current was measured with positive bias applied to the top electrode while the bottom electrode was kept grounded. Fig. 7.9 shows the highest dark current density of 7×10^3 A/m² at 15 V bias.

Figure 7.9 I-V characteristics of AlGaIn/GaN multilayer photodetector, inset is the microscopy image of photodetector

Although device design with relatively more electrode pairs and longer electrode length was used (compared to the UV photodetector in Chapter 6), dual band photodetector still showed much lower dark current density at 5 V bias than that of UV detector described in Chapter 6. The lower dark current may be caused due to the multiple superlattice structure, which is acting as barrier layer and decreasing the dark current dramatically. Although the expected dark current should be

asymmetric due to the asymmetric structure design, the dark current density curve obtained symmetric at room temperature. Thermionic emission scheme is reported to dominate the dark current at room temperature and the ground state carriers partially jump up to the quasi-continuum state, and elimination of the internal electric fields at the step barrier layers results in the symmetric I-V characteristic curve [184]. It was also found that room temperature resistance is about 40Ω , while increased by 3-4 order of magnitude when cooled to 10K. It suggests that the inter-well tunneling may be the reason of the dark current which can be suppressed by increasing the barrier height [296].

7.3.2 UV Photoresponsivity measurement

Fig. 7.10 (a) shows the photocurrent spectra with respect to the applied bias voltage. The peak lies around 330 nm and it matches with the designed bandgap corresponding to $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$. From the photoresponsivity spectra shown in Fig. 7.10 (b), it can be clearly observed that the background noise is lower. With the bias voltage increasing, the responsivity also increased, and the highest UV responsivity was achieved at 3 V applied bias with a responsivity of 0.01 A/W. Compared

Figure 7.10 (a) UV photocurrent and (b) responsivity spectra vs. bias voltage

with the UV detector investigated in Chapter 6, UV photoresponsivity in this study is lower as well as the dark current density. It is believed that either multiple superlattice layers act as barrier and reduce the UV photocurrent collected [167] or due to the complexity of the multiple layer structure employed. With multiple optical interface formed in this complex structure, UV incident light was reflected and not fully absorbed during measurement compared to the simple UV photodetector structure investigated in Chapter 6.

7.3.3 IR response measurement of photodetector

IR photodetector was fabricated using normal square mesa design in Technion, Israel [297]. IR photodetector was fabricated using normal square mesa design in Technion, Israel. For device fabrication and characterization, the 4 inch wafer was diced into $7 \times 7 \text{ mm}^2$ pieces. Standard optical photolithography and dry inductive plasma (ICP) in Cl_2 etching processes were employed to form active mesas of $700 \times 700 \text{ }\mu\text{m}^2$. Ti/Al 30/200 nm bi-metallic electrical contacts were deposited by e-beam evaporation on the top and bottom doped $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$ contact layers, as illustrated in Fig.

Figure 7.11 A schematic illustration of the QCD structure with metal contacts. The inset shows the conduction band energy in the top AlGaN/AlN/GaN interface.

7.11. Rapid thermal annealing was applied to achieve ohmic contact. The center of the top surface of the mesas was kept unmetallized to allow front illumination.

Optical measurements of the devices were conducted using Bruker Equinox 55 Fourier transform spectrometer (FTIR) in rapid-scan mode and a silicon carbide globar source. First, the *s*-polarized and *p*-polarized absorption of an unprocessed sample in a multi-pass waveguide fashion were measured as illustrated in the inset of Fig. 7.12. The measured absorption coefficient is shown in Fig. 7.12 along with the absorbance per pass per QW, which equals to $\alpha \times W$. The maximum value of absorption coefficient $\alpha = 2330 \text{ cm}^{-1}$, measured at the wavelength of $4.48 \text{ }\mu\text{m}$, which is comparable to the theoretically calculated center wavelength of $4.49 \text{ }\mu\text{m}$ [298].

Figure 7.12 Absorption coefficient α and the absorbance per pass per QW. Peak absorption is at $4.48 \text{ }\mu\text{m}$, while the expected is $4.49 \text{ }\mu\text{m}$. The broadening is 44 meV ($0.72 \text{ }\mu\text{m}$). The inset shows the multi-pass waveguide configuration of the transmission measurement.

The photocurrent measurement was performed by directing the incident beam on a 30° polished wedge of the processed sample, achieving 45° propagation angle in the active area and efficient light coupling. The sample was mounted on a copper plate attached to the cold finger of a cryostat

for which the temperature was varied from room temperature to 18 K. The detector signal was collected using a low noise trans-impedance amplifier. The resulting spectral response is shown in Fig. 7.13. The prominent CO₂ absorption dip at 4.3 μm makes it difficult to determine the peak wavelength, thus a Lorentzian fit of the spectra was made to analyze the peak wavelength and broadening at each temperature. The peak wavelength of 4.14 μm is obtained at 18 K and the broadening (FWHM) is 1.26 μm. The peak wavelength red shifts as the temperature is elevated and reaches 4.5 μm at 150 K. The peak response, achieved at 18 K, degrades with the increase of temperature as shown in the inset of Fig. 7.13. The photosignal is not detectable above 150 K probably due to increased noise.

Absolute responsivity was measured using a calibrated 1000 K blackbody source illuminating the device through the cryostat optical window, with the device placed at $d = 4.5$ cm away from the blackbody aperture and cooled to 18 K. Photocurrent was measured using lock-in-amplifier and low noise trans-impedance preamplifier. The responsivity R_i is calculated by

$$R_i = \frac{I_{sig}}{P_{opt} \times M} \quad \text{Eq. 7.1}$$

where M is a factor representing the p -polarized portion of the light (taken to be 0.5) and the reflection from the device, calculated by Fresnel's equation for p -polarized light using a refractive index of 3.42 ($M = 0.5 \times 0.7$). I_{sig} is the photocurrent, and P_{opt} is the blackbody's incident optical power [299]. The measured photocurrent measured was 162 pA and the corresponding responsivity is 44 μA/W.

Figure 7.13 Spectral response of the device. The 4.3 μm dip is due to CO_2 absorption. At 18 K the peak wavelength equals 4.14 μm and the broadening is 1.26 μm . In the inset the temperature dependency of the signal is shown, peak signal is achieved at 18 K.

7.4 Summary

In this chapter, successful growth of crack-free AlGaIn/GaN multiple layer structure for UV/IR dual band photodetector application on full 4-inch Si (111) and AlN/Si templates have been investigated. The complete structure was grown at 725 °C, and uniform Si doping concentration of $2 \times 10^{18} \text{ cm}^{-3}$ and high doping with the carrier concentration up to $2 \times 10^{19} \text{ cm}^{-3}$ have been achieved for the activation layer and AlGaIn contact layers, respectively. The complete structure was grown with high III/V ratio under Ga-rich condition while surface recovery time of 12 sec and 80 sec were introduced for superlattice and top AlGaIn contact layer growth, respectively, such that the surface was not overexposed or left with residual Ga in order to maintain 2D growth without any defects. Several samples with SL period varying from 20 to 40 were investigated. The

successful device structure grown with 30 period SL was in compressive stress with a low wafer bow of $4.5\ \mu\text{m}$ and the surface was found to be smooth with the RMS roughness of $0.46\ \text{nm}$ over an area of $5 \times 5\ \mu\text{m}^2$. HR-XRD scan of ω - 2θ rocking curve performed along (002) plane of the epilayer to determine the thickness of superlattice layers show a well-controlled AlGaIn thickness of $1.5\ \text{nm}$. TEM analysis revealed the sharp interfaces achieved in the growth and also confirmed the thickness results obtained using XRD analysis.

Interdigitated photodetector was fabricated on epilayer grown on AlN/Si template, and a low room temperature dark current density of $7 \times 10^3\ \text{A/m}^2$ was achieved at $15\ \text{V}$ applied bias. The highest UV responsivity of $0.01\ \text{A/W}$ was demonstrated at $3\ \text{V}$ bias. The first mid-infrared GaN/AlGaIn QCD epitaxially grown on silicon have been designed, modeled, and fabricated. At $19\ \text{K}$, the device exhibits a responsivity of $44\ \mu\text{A/W}$ and detectivity of 2×10^8 Jones at $\lambda = 4.14\ \mu\text{m}$. The responsivity and detectivity can be improved through optimizing the trade-off between absorption, extraction efficiency and resistance. Improved design can reduce unwanted diagonal transitions from the ground state to the extractor QWs, thus increasing the resistance and decreasing the dark current. Further improvement can be achieved by higher doping which will increase the electron density in the QW and hence enhance the absorption. The successful implementation of GaN ISBT optoelectronic device on Si is promising to integrated-optoelectronics technology, showing great potential for ultra-fast operation at a very wide spectral range.

Chapter 8 Conclusions and Recommendations

8.1 Conclusions

In this thesis, growth and characterization studies on AlN, GaN, AlGaIn/GaN and GaN-based multilayer structures grown on Si (111) substrate using plasma assisted molecular beam epitaxy have been reported. Si doping effect on GaN and AlGaIn on the surface and crystalline quality have been studied. Some of the optimized layer structures were used for the fabrication of photodetectors for UV/IR applications, and performance of the photodetectors are reported.

AlN grown on Si (111) substrate in N-rich condition graded from low Al flux to high flux followed by GaN growth grading from 3D growth (N-rich) to 2D growth (Ga-rich) showed smooth surface without any microcrystal. Ga/Si interdiffusion would always happen regardless of AlN thickness in MBE grown samples of GaN/AlN on Si. However, such issue was not observed when MOCVD grown AlN/Si template was used for GaN growth. AlN grown by MBE could not stop Ga/Si interdiffusion. The gap or grain boundary formed due to N-rich AlN growth by MBE could act as a path for (liquid) Ga atoms to diffuse into Si substrate and dissolve Si, allowing Si to penetrate AlN and reach the surface. This issue was addressed in this study by growing a thin layer of GaN in N-rich condition at GaN/AlN interface such that no residual Ga was present on top of the AlN surface. Then, the following GaN growth was performed under Ga-rich condition to obtain high quality GaN layer. Using this method, Ga diffusion into the bottom of AlN layer and Si substrate was impeded by the thin GaN layer grown already on AlN

Smooth GaN/AlN grown on Si (111) by PA-MBE with the RMS surface roughness ~ 0.4 nm was achieved without microcrystals. Si doping concentration in GaN was found to be non-linear with

respect to Si cell temperature at higher doping level. Surface morphology was not affected by Si doping concentration at low doping level while high doping level ($> 10^{20} \text{ cm}^{-3}$) caused surface RMS roughness to increase to 1.1 nm. In GaN doping investigation, clear band gap narrowing effect was observed, and with the increase in Si doping concentration, compressive strain in GaN was changed to tensile strain.

2D GaN was grown on SiN_x formed by exposing Si substrate to nitrogen plasma at growth temperature. In this work, both $\beta\text{-SiN}_x$ and amorphous SiN_x have been studied and it was found that $\beta\text{-SiN}_x$ could not block Ga/Si interdiffusion effectively, while amorphous SiN_x suppressed interdiffusion successfully and solved the microcrystal formation issue. Hence, amorphous SiN_x was used for the subsequent growths of GaN and AlGaIn on Si. GaN grown on amorphous SiN_x directly in Ga-rich condition was optimized to achieve RMS roughness of 0.4 nm. Similarly, AlGaIn growth in Ga-rich condition was also optimized using amorphous SiN_x , and AlGaIn with 30% Al-composition was achieved with RMS roughness of 0.8 nm. AlGaIn grown on 4 inch AlN/Si template was also optimized to achieve smooth surface with similar RMS roughness.

AlGaIn-doping study was conducted to determine the carrier concentration as a function of Si-cell temperature for contact layer development in photodetectors. The carrier concentration increases exponentially with the increase in Si cell temperature. A value of $2 \times 10^{18} \text{ cm}^{-3}$ was determined at Si-cell temperature of 1310 °C, which is the highest free carrier concentration level achieved in Si doped 30% AlGaIn layer on Si substrate by PA-MBE to the best of our knowledge. While the surface morphology was not affected by Si doping, lower Si doped layers were found to be 0.4% tensile strained and the highest doped layer was in 0.6% compressive strain, which is similar to that observed in GaN doping.

Optimized crack free AlGaIn/GaN multilayer structures for UV photodetector application were successfully grown on 2 inch Si substrate as well as 4 inch AlN/Si template grown by MOCVD with similar RMS roughness less than 1nm in $5 \times 5 \mu\text{m}^2$. Three AlGaIn/GaN multilayer structures with Al-compositions of 15%, 17% and 23% were grown to achieve corresponding cutoff wavelengths for photodetector.

Photodetector designs of square detector and interdigitated detector were fabricated on different AlGaIn/GaN multilayer structures grown by MBE on both Si substrate and AlN/Si template. Photodetectors (grown on Si substrate and also AlN template) showed similar dark current density of 10^1 to 10^3 A/m^2 at 5 V bias for both square and interdigitated detectors. Square photodetector showed responsivity of 1 A/W at 3 V bias which is higher than the previously reported results [177 - 179]. The interdigitated detector performed even better with the responsivity of 5.7 A/W. Higher photoresponsivity obtained for interdigitated detector is attributed to the larger detecting area and short-gap lateral electrodes. Optical transmittance spectrum was scanned and near-IR absorption was found in multilayer structure due to intraband transition.

AlGaIn/GaN multiple layer structure for UV/IR dual band photodetector application was grown on full 4-inch Si (111) and AlN/Si grown by MOCVD template. The complete structure grown was in compressive stress with a wafer bow of 4.5 μm and the surface is found to be smooth with the RMS roughness of 0.8 nm over an area of $5 \times 5 \mu\text{m}^2$. Interdigitated photodetector fabricated showed a low dark current density of $7 \times 10^3 \text{ A/m}^2$ at 15 V bias at room temperature. The highest UV responsivity achieved was 0.01 A/W at 3 V bias, and near-IR optical absorption was observed at 4.52 μm with FWHM of 1.47 μm . AlGaIn/GaN based multilayer structure grown by MBE are found promising for UV/IR dual band photodetector applications.

8.2 Recommendations for Further Work

Growth optimization of AlN, GaN, AlGaIn/GaN and GaN-based multilayer structures on Si (111) by MBE has been studied. However, surface and crystalline quality of AlGaIn and GaN used in the device structures can be improved further. Although AlGaIn/GaN based UV and UV/IR dual band photodetector have been demonstrated in this thesis, the photodetectors can be further improved to achieve better performance. There are still more device structures can be optimized and fabricated for more applications.

8.2.1 AlN buffer layer

AlN buffer layer for GaN and AlGaIn growth was optimized in N-rich condition to prevent excess Al metal, which would otherwise cause severe Al/Si interdiffusion issue. AlN thickness can be increased to achieve better quality with lower dislocation density. Also, the same method of 3D to 2D transition during GaN growth can be applied to achieve better AlN quality in Al-rich growth condition. To achieve better AlN crystalline quality together with better surface morphology, two-step AlN growth currently used can be optimized to have an additional of 100 nm AlN grown in Al-rich condition and at higher growth temperature. The high-temperature grown AlN is expected to improve the quality of AlN. Since AlN layer was actually grown on amorphous SiN_x on Si substrate, it is necessary to study the SiN_x/AlN interface to further improve AlN nucleation on amorphous SiN_x.

8.2.2 GaN/AlGaIn growth optimization on AlN

Although GaN and AlGaIn growth on AlN have been optimized, the growth window for Ga flux control is still very narrow. Flux measurement before each growth is necessary to get a close control on the excess Ga metal on surface. Growth time for each AlGaIn growth can be further decreased and also the desorption time with fixed Ga flux. Since less Ga would be deposited during

each growth period and the amount of excess Ga metal would be lower and, hence Ga flux window would be larger and easier to control.

In this thesis, AlGa_N growth and doping were only optimized for Al-composition of 30% and below. Considering the fact that to achieve even shorter wavelength for UV photodetection and even larger GaN/AlGa_N band gap offset to form deeper quantum well for IR detection, AlGa_N with higher Al-composition needs to be grown on Si substrate. Therefore, AlGa_N growth requires further optimization at higher Al-composition due to different growth temperature. Also, when Al-composition increases, high Si doping concentration will be tougher to incorporate for ohmic contact with low resistance. Si doping for higher Al-composition also needs to be further studied for better understanding.

8.2.3 UV photodetector with thin SiN_x

Device processing for photoconductive UV photodetector can be further improved. Surface passivation which was widely used in HEMT fabrication can be introduced to reduce the reflection on GaN surface. SiN_x is commonly used for surface passivation which can be easily deposited by PECVD. The SiN_x thickness needs to be calculated and optimized to obtain minimum reflection on top GaN surface. The thin SiN_x layer also helps to reduce the light escaping from photoconductive layer. In this case, the device fabrication process can be modified to add one step of SiN_x deposition and a new mask needs to be designed.

8.2.4 UV/IR dual band photodetector with three electrodes design

Although UV/IR responsivity has been demonstrated for the dual band photodetector demonstrated in Chapter 7, there is still some space for improvement compared with photoconductive UV photodetector described in Chapter 6 which showed high responsivity. Although the near-IR absorption has been observed for dual band photodetector in Chapter 7, the device fabrication is

quite challenging. To achieve better UV responsivity as well as IR responsivity, the function layers for different wavelength detection need to be separated.

Fig. 8.1 (a) shows the modified detector structure. As can be observed, there are three electrodes namely top, middle and bottom electrode. Although similar design has been reported on similar devices grown on sapphire substrate [177 - 179], the UV responsivity was found lower to our results. In the proposed design, a modified 3-electrode design is introduced. As shown in Fig. 8.1, there are two mesas, namely mesa 1 and mesa 2. Mesa 1 is controlled by top and middle electrodes while mesa 2 is controlled by middle and bottom electrodes. As indicated in Fig 8.1 (b), mesa 1

Figure 8.1 Photodetector with (a) square and interdigitated design on AlGaN/GaN multilayer structure and (b) cross section of the device

contains n-GaN/AlGaIn/n-GaN for photoconductive UV detection. Mesa 2 contains n-GaN/SL/n-GaN for intraband near-IR detection. Interdigitated electrodes design is used for mesa 1 to achieve high UV responsivity similar to what have been achieved in Chapter 6. In mesa 2, normal square electrode is used for bottom contact. For device operation, middle contact can be grounded, and positive bias can be applied to top and bottom contacts or middle contact is applied to a fixed voltage and top/bottom is set at different voltage levels. During light detection, mesa 1 will only absorb UV light and near-IR light will be by passed. In this situation, the two photodetectors are isolated and with the top photodetector acting as a UV filter. Once the mesa 1 thickness is well designed, no UV light will be leaked to bottom detector. Two signals can be collected from this pancaked photodetector separately.

In addition to UV/IR dual band photodetection, the design shown in Fig. 8.1 can also be extended for other applications such as UV/UV or IR/IR dual band photodetectors in which mesa 1 needs to be designed to detect wavelength shorter than mesa 2. The functional layer should also be well designed so that incident light with desired detection wavelength is fully absorbed in mesa 1.

List of Publications

Journal Articles :

1. **Y. Zheng**, M. Agrawal, N. Dharmarasu and K. Radhakrishnan and S. Patwal, “A study on Ga-Si interdiffusion during (Al)GaN/AlN growth on Si by plasma assisted molecular beam epitaxy”, Applied Surface Science, vol. 481, pp. 319-326, 2019.
2. Ben Dror, **Y. Zheng**, M. Agrawal, K. Radhakrishnan, Meir Orenstein, Gad Bahir, “Mid Infrared GaN/AlGaIn Quantum Cascade Detector Grown On Silicon”, IEEE Electron Device Letters, vol.2, pp. 263-266, 2018.
3. M. Agrawal, L. Ravikiran, N. Dharmarasu, K. Radhakrishnan, G. S. Karthikeyan, and **Y. Zheng**, “Stress evolution of GaN/AlN heterostructure grown on 6H-SiC substrate by plasma assisted molecular beam epitaxy”, AIP Advances 7, 015022 (2017).

Conference Proceedings:

1. **Yi Zheng**, Manvi Agrawal, K. Radhakrishnan, N. Dharmarasu, G. S. Karthikeyan, A. G. Unil Perera, Annalisa Bruno, Cesare Soci, “AlGaIn-based UV-photodetector heterostructure on Si (111) substrate grown by PA-MBE”, in 2017 International Workshop on UV Materials and Devices (IWUMD), Nov. 2017.
2. **Y. Zheng**, M. Agrawal, N. Dharmarasu, and K. Radhakrishnan, “Growth of AlGaIn/GaN multiple quantum well structure on 4-inch Si by plasma assisted molecular beam epitaxy for infrared photodetector”, in 2017 International Conference on Intersubband Transitions in Quantum Wells (ITQW), Sep. 2017.
3. **Yi ZHENG**, K. RADHAKRISHNAN, Dharmarasu NETHAJI, Manvi AGRAWAL, “Si Doped Al_{0.3}Ga_{0.7}N Grown on 4-inch Si (111) Substrate by Plasma Assisted Molecular Beam Epitaxy (PA-MBE)”, in 2017 International Conference on Materials for Advanced Technologies (ICMAT), Jun. 2017.
4. **Yi ZHENG**, Ravikiran LINGAPARTHI, K. RADHAKRISHNAN, Dharmarasu NETHAJI, Manvi AGRAWAL, Unil PERERA, “Group III-nitride Heterojunction Based Multi-Band Detector on Si (111) Substrate Grown by Plasma Assisted Molecular Beam

Epitaxy (PA-MBE)”, in 2016 International Conference on Electronic Materials (ICEM),
Jul. 2016.

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