Studies on GaN based HEMT heterostructures on 100-mm silicon grown by molecular beam epitaxy

Ravikiran Lingaparthi

2014

Ravikiran Lingaparthi. (2014). Studies on GaN based HEMT heterostructures on 100-mm silicon grown by molecular beam epitaxy. Doctoral thesis, Nanyang Technological University, Singapore.

<https://hdl.handle.net/10356/62076>

<https://doi.org/10.32657/10356/62076>

Studies on GaN Based HEMT Heterostructures on 100-mm Silicon Grown by Molecular Beam Epitaxy

Ravikiran Lingaparthi

School of Electrical and Electronic Engineering

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

2014

To My Beloved Parents

L.L. Ganapathi Rao

L.V.L.P. Manga Thayaru

“Imagination is more important than knowledge. For knowledge is limited, whereas imagination embraces the entire world, stimulating progress, giving birth to evolution”

- Albert Einstein

“The noblest pleasure is the joy of understanding”

- Leonardo da Vinci

Acknowledgements

I would like to express my greatest gratitude to my supervisor Assoc. Prof. K. Radhakrishnan for his constant guidance during the course of this entire work. Meetings and discussions with him have always given me invaluable inputs and kept me in the correct path of the research. I sincerely thank him for the various opportunities that he has provided me and for all his support during the difficult times in this Ph.D period. His encouragement has always been the driving force in pursuing the research.

I would also like to express my sincere thanks to my co-supervisor Dr. Dharmarasu Nethaji for his invaluable time and thoughtful guidance throughout this research period. I have learnt to a huge extent from his meticulous approach in conducting the experiments. Moreover, his planning and approach towards research projects imparted a great deal of learning experience. I am extremely grateful for his painstaking review of the report and suggestions for the improvement.

I really appreciate Dr. Sun ZhongZhe's help to train me with various characterization tools during my first year of Ph.D. I would also like to extend my sincere thanks to Dr. S. Munawar Basha who has supported me for the growth and characterization during my final year of my Ph.D.

I express my heartiest thanks to my colleague, Dr. Manvi Agrawal for the valuable and thought provoking discussions. She has been more a friend than a colleague. We explored a lot of science and tried to understand the physics behind. I really thank her for her support during the difficult period that we have faced together. I express my sincere thanks to Mr. Wong Wei Jie Terence for keeping the MBE system in good condition and helping us in performing the growth runs. He is our "Go- To Man" whenever we want to do something with the system. It was also a great fun to work with all of my other colleagues – Mr. Lin Yiding, Mrs. Namita, Ms. Aparna, Mr. Song Yang and Mr. Hidayat.

I would like to acknowledge Prof. Ng Geok Ing and the device team, led by Dr. Subramaniam Arulkumaran for their support in fabricating the devices. In this regard, I would like to extend my sincere thanks to Mr. C.M. Manoj Kumar for providing his help for fabricating ohmic contacts on the grown wafer for the buffer leakage measurements and later in annealing studies.

I would also like to appreciate the help I received from Mr. Muhd Fauzi Bin Abdullah and Ms. Seet Lye Ping in the operation of various instruments used for characterization of GaN epilayers.

My teachers at various stages of my academic career -Prof. R.S. Srinivasa, Prof. S.S. Major from IIT Bombay. Dr. V. V. Ravikanth and Dr. S.V.M. Satyanarayana from Pondicherry University have all contributed in improving the scientific aptitude in me and guided me well to pursue research as a career. I sincerely thank them all for their guidance.

My friends in Singapore have tolerated my absence from many planned events due to the busy research schedule and have been morally supporting me throughout this period. I really appreciate their understanding. Finally, I would like to thank my parents and my brother for their support and inspiration, which helped me to complete this work in an ideal environment.

Above all I bow my head before God Almighty to keep me in cheer and health during the course of this work.

Table of Contents

Acknowledgements.....	ii
Table of Contents	iv
List of Figures	viii
List of Tables.....	xiv
Abbreviations.....	xv
1. Introduction.....	1
1.1 Gallium nitride - structural properties.....	2
1.2 Nitride compounds	3
1.2.1 Ternary nitrides	3
1.2.2 Quaternary nitrides	7
1.3 GaN based HEMT heterostructures	8
1.3.1 AlGaIn/GaN HEMT heterostructure	9
1.3.2 AlGaIn/GaN/AlGaIn double heterojunction HEMT heterostructure...	11
1.3.3 Lattice matched GaN based HEMT heterostructures	12
1.4 Substrates for GaN based HEMT heterostructures	14
1.5 Challenges in the growth of GaN based HEMT heterostructures on Si(111)	16
1.6 Objectives	17
1.7 Organization of the dissertation	18
2. GaN based HEMT heterostructures on Si(111)	21
2.1 Growth techniques.....	21
2.1.1 Metal organic chemical vapor deposition	21
2.1.2 Molecular beam epitaxy	23
2.2 Stress management for GaN epitaxy on Si(111).....	28
2.3 GaN buffer resistivity control.....	31
2.4 GaN based HEMT heterostructures	35
2.4.1 AlGaIn/GaN based HEMT heterostructures on Si(111) using MBE....	36
2.4.2 Lattice matched barrier using MBE growth technique	38
3. Growth and characterization techniques.....	40
3.1 MBE growth technique	40

3.2 Characterization techniques	45
3.2.1 High resolution X-ray diffraction technique	46
3.2.2 Confocal micro-Raman spectroscopy	48
3.2.3 Atomic force microscopy	51
3.2.4 Scanning electron microscopy	53
3.2.5 Transmission electron microscopy	54
3.2.6 Secondary ion mass spectroscopy	55
3.2.7 Hall Effect.....	56
3.2.8 Capacitance-voltage measurements.....	59
 4. Stress management in AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.....	61
4.1 Growth of nitride epitaxy on Si(111) using ammonia-MBE	61
4.1.1 AlN growth.....	62
4.1.2 GaN growth.....	64
4.2 Stress mitigating layers for GaN growth	65
4.3 AlN/GaN stress mitigating layer	70
4.4 Strain states of AlN/GaN SMLs structure	72
4.4.1 Density of buried cracks in AlN/GaN SMLs.....	74
4.4.2 Density and size of voids in AlN/GaN SMLs.....	78
4.4.3 Effect of buried cracks on the residual stress in GaN buffer layers ...	79
4.5 Optimization of AlN/GaN SMLs for AlGaIn/GaN HEMT heterostructures.....	83
4.6 Optimization of 2 nd GaN for the AlGaIn/GaN HEMT heterostructures.....	84
4.7 Growth of AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.....	87
4.8 Summary.....	93
 5. Stress management in AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlGaIn/AlN/GaN SMLs.....	95
5.1 AlGaIn/AlN/GaN SMLs	95
5.1.1 In-situ stress measurements.....	96
5.1.2 Ex-situ bow measurements.....	97
5.1.3 Cross sectional TEM analysis	98
5.1.4 Crystal quality and surface morphology analysis.....	102

5.2 Effect of V/III ratio on the properties of 2 nd GaN grown using AlGa _N /Al _N /Ga _N SMLs	103
5.2.1 Surface morphology	104
5.2.2 In-situ stress measurements	105
5.2.3 Raman measurements	106
5.2.4 Ex-situ bow measurements	107
5.2.5 HR-XRD measurements	107
5.2.6 Cross sectional TEM	108
5.2.7 Surface morphology evolution with V/III ratio of 2 nd GaN	109
5.3 AlGa _N /Ga _N HEMT heterostructures grown using AlGa _N /Al _N /Ga _N SMLs ..	111
5.4 Summary	116
 6. Carbon doped GaN buffer	117
6.1 Buffer leakage current measurements	117
6.2 Sources of current conduction in GaN buffer	118
6.2.1 Secondary ion mass spectroscopy	119
6.2.2 Photoluminescence measurements	120
6.3 Parallel conductions in AlGa _N /Ga _N HEMT heterostructure	122
6.4 Carbon doping	125
6.4.1 CBr ₄ doping- HLC and VLC measurements	125
6.4.2 Structural properties of carbon doped GaN buffer	127
6.4.3 Optical properties of carbon doped GaN buffer	129
6.5 AlGa _N /Ga _N HEMT heterostructures	135
6.6 Summary	136
 7. Advanced GaN based HEMT heterostructures	137
7.1 AlGa _N /Al _N /AlGa _N double heterojunction HEMT heterostructure	137
7.1.1 Growth of AlGa _N /Ga _N /AlGa _N DH-HEMT heterostructure	138
7.1.2 Structural characterizations	138
7.1.3 Electrical characterizations	142
7.1.4 AlGa _N /Ga _N /AlGa _N DH-HEMT devices	145
7.2 Lattice matched InAl _N barrier for GaN based HEMT heterostructures	147
7.2.1 GaN growth using PA-MBE on GaN templates grown using ammonia-MBE	147

7.2.2 InAlN growth by PA-MBE	149
7.2.3 InAlN/AlN/GaN HEMT heterostructures	151
7.3 Summary	155
8. Conclusions	156
8.1 Stress management in AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.	156
8.2 Stress management in AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlGaIn/AlN/GaN SMLs	157
8.3 Carbon doped GaN buffer	158
8.4 Advanced HEMT heterostructures	159
8.4.1 AlGaIn/GaN/AlGaIn double heterojunction HEMT	159
8.4.2 Lattice matched InAlN barrier for GaN based HEMT heterostructures	160
9. Future recommendations	162
10. References	163
11. Author's Publications	182
11.1 Journal Publications	182
11.2 Conference Presentations	182

List of Figures

Fig. 1.1 Stick-ball structures of GaN in wurtzite (a) and zincblende (b) forms.	2
Fig. 1.2 Band gap energy and lattice constant variation as a function of 'Al' mole fraction in AlGaN.....	6
Fig. 1.3 Band gap energy and lattice constant variation as a function of 'In' mole fraction in InGaN.....	6
Fig. 1.4 Band gap energy and lattice constant variation as a function of 'In' mole fraction in InAlN.....	6
Fig. 1.5 Band gap energy as a function of lattice constant in AlInGaN nitride system.....	7
Fig. 1.6 Schematic of a typical AlGaN/GaN HEMT heterostructure (a) and its band diagram (b).	10
Fig. 1.7 Schematic of a typical AlGaN/GaN/AlGaN DH-HEMT heterostructure (a) and its band diagram (b).....	12
Fig. 3.1. A picture of the partial MBE system.....	41
Fig. 3.2 Functional diagram of the MBE system.	42
Fig. 3.3 BEP as a function of fore-line pressures of CBr ₄	43
Fig. 3.4 The schematic representation of a typical RHEED set-up.....	44
Fig. 3.5 Schematic of the in-situ stress measurement tool (a) and the reflected beam from the substrate is seen as an array of spots captured by the CCD in the detector box (b).	45
Fig. 3.6 The schematic of a typical optical set-up of an XRD system (a) and the diffraction phenomenon showing the diffraction of X-rays at an angle of ' θ ' for an atomic spacing of ' d ' (b).	47
Fig. 3.7 The schematic of a typical confocal micro-Raman spectroscopy set up.	49
Fig. 3.8 Intensity of Raman GaN E ₂ peak profile as a function of depth (a) and the derivative of intensity peak profile vs depth curve (b) showing the true surface of GaN that is air/GaN interface.	51
Fig. 3.9 A schematic of a typical AFM set-up.	52
Fig. 3.10 The schematic of Hall measurement set-up in Van der Pauw configuration.	57
Fig. 3.11 The schematic diagram of non-destructive mobility measurement method.	59
Fig. 3.12 Mercury probe dot-ring configuration on the semiconductor surface.	59
Fig. 4.1 Thermally cleaned Si(111) surface showing (7×7) RHEED pattern (a). AlN nucleation on Si surface showing AlN and underlying Si (1×1) RHEED patterns along (1 $\bar{1}$ 0) of Si(111) (b).	61

Fig. 4.2 RHEED patterns of AlN layer along $(11\bar{2}0)$ azimuth of samples A1 (a), A2 (b) and, A3 (c).	63
Fig. 4.3 Effect of AlN growth rate (a) and growth temperature (b) on screw type dislocation density and RMS roughness of 100 nm AlN on Si.	63
Fig. 4.4 Evolution of RHEED patterns of GaN layer as a function of its thickness (a). Microscope image showing cracking of 1 μ m thick GaN layer grown on 100 nm AlN buffer using 100-mm Si substrate (b).	64
Fig. 4.5 Epilayer structures of samples S1 (a), S2 (b) and S3 (c) grown on Si using ammonia-MBE.	65
Fig. 4.6 RHEED diffraction patterns along $(11\bar{2}0)$ direction and their corresponding AFM surface morphologies of GaN of samples S1 (a), S2 (b) and S3(c).	66
Fig. 4.7 A trend in the lateral grain size and the RMS roughness (scan area: 5 $\times$ 5 μ m ²) obtained from the AFM images of samples S1, S2 and S3.	67
Fig. 4.8 Raman shift in E ₂ phonon peak of GaN in samples S1, S2 and S3 with the vertical line indicates free standing GaN E ₂ position (567 cm ⁻¹) (a). HR-XRD rocking curves along the asymmetric $(20\bar{2}1)$ plane of samples S1, S2 and S3(b).	68
Fig. 4.9 Strain in GaN layers vs lateral grain size for samples S1, S2 and S3.	70
Fig. 4.10 Surface morphology of GaN grown on AlN/GaN SMLs with the growth rate of 0.22 μ m/h (a) and 0.7 μ m/h (b).	71
Fig. 4.11 The optical microscope image of AlGaIn/GaN HEMT heterostructures on Si substrate: the surface image (a) and the buried cracks at the interface between AlN and GaN layers in the SMLs (b).	72
Fig. 4.12 In-situ curvature plot of a typical structure with 250/250 nm 2 nd AlN /1 st GaN layers as a function of growth time.	73
Fig. 4.13 Microscopic images of buried cracks of samples A (a) and E (b).	75
Fig. 4.14 In-situ curvature plot of 1 st GaN and 2 nd AlN layers with thicknesses of 250 nm as a function of growth time. The inset shows the TEM image of a typical buried crack feature.	76
Fig. 4.15 The cross-sectional TEM images of sample A (a), sample C (b) and sample E (c) obtained using bright field mode along $(11\bar{2}0)$ zone axis. The arrow marks indicate the looping of dislocations.	77
Fig. 4.16 Raman shift in GaN-E ₂ peak of samples F and G. G1 and G2 correspond to phonon modes obtained from the close vicinity and in between the cracked regions, respectively. The insets show the epilayer structures of samples F and G.	80

Fig. 4.17 Raman shift in E_2 phonon peak of 1 st and 2 nd GaN layers of sample E.	81
Fig. 4.18 Stresses in 1 st and 2 nd GaN layers as a function of average crack spacing. The connecting lines are for the guidance to eyes only.	82
Fig. 4.19 Surface morphology of samples A (a) and E (b). Sample A shows pits on the surface with RMS roughness of 4.8 nm and sample E shows pit free surface with rms roughness of 2.06 nm.	84
Fig. 4.20 AFM Surface morphology of 1000 nm thick 2 nd GaN, grown with a growth rate of 0.7 $\mu\text{m}/\text{h}$	84
Fig. 4.21 AFM Surface morphology of 1000 nm thick 2 nd GaN layer, grown with a growth rate of 0.28 $\mu\text{m}/\text{h}$	85
Fig. 4.22 In-situ curvature measurement obtained during the growth of AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.	87
Fig. 4.23 Stress $\times$ Thickness and incremental stresses in 2 nd AlN (a) and 2 nd GaN layers (b).	87
Fig. 4.24 Schematic of AlGaIn/GaN HEMT heterostructure on Si using AlN/GaN SMLs (a). Photograph of grown HEMT heterostructure on 100-mm Si(111) wafer (b).	88
Fig. 4.25 High resolution X-ray diffraction ω -2 θ scan of AlGaIn/GaN HEMT heterostructure with aluminum composition of 29.5% and AlGaIn thickness of 27 nm (a). Reciprocal space mapping of AlGaIn/GaN HEMT heterostructure along GaN(10 $\bar{1}$ 5) direction (b).	89
Fig. 4.26 Capacitance-voltage measurements of AlGaIn/GaN HEMT structure. Sheet carrier concentration obtained by integration of C-V profile is also presented...	90
Fig. 4.27 Depth profile of carrier concentration in the AlGaIn/GaN HEMT heterostructure.	90
Fig. 4.28 Mobility and sheet carrier concentration as a function of measurement temperature for AlGaIn/GaN HEMT heterostructure.	91
Fig. 4.29 2DEG mapping of sheet resistance (a), mobility (b) and sheet carrier concentration (c) of AlGaIn /GaN HEMT heterostructures on 100-mm Si substrate grown using AlN/GaN SMLs.	92
Fig. 4.30 $I_{\text{DS}}\text{-}V_{\text{DS}}$ characteristics of AlGaIn/GaN HEMTs (a). Transfer characteristics of AlGaIn/ GaN HEMTs (b).	93
Fig. 5.1 The cross sectional view of the heterostructures on 100-mm Si substrate, using AlN/GaN (a) and AlGaIn/AlN/GaN SMLs (b).	95
Fig. 5.2 Stress $\times$ Thickness and the incremental stress as a function of thickness of samples S1 and S2.	96

Fig. 5.3 Ex-situ bow of samples S1 (a) and S2 (b) on 100-mm Si substrate.....	98
Fig. 5.4 Bright field cross sectional TEM images of heterostructures grown using AlN/GaN (a) and AlGaN/AlN/GaN SMLs (b).	99
Fig. 5.5 Cross sectional TEM images obtained in HAADF (a) and weak beam dark field modes with $g = 2\bar{1}\bar{1}0$ (b) and $g = 0002$ (c) of a heterostructure, grown using AlGaN/AlN/GaN SMLs. WBDF image with $g = 0002$ for the heterostructure with AlN/GaN SMLS (d).	101
Fig. 5.6 AFM images of $10 \times 10 \mu\text{m}^2$ scan area of 2 nd GaN of samples S1 (a) and S2 (b).	103
Fig. 5.7 AFM images obtained in the amplitude mode showing surface morphology of 2 nd GaN of samples S3 (a) S4 (b) and, S5 (c). RMS roughness as a function of 2 nd GaN V/III ratio (d).	105
Fig. 5.8 Stress $\times$ thickness and incremental stress as a function of thickness of samples S3, S4 and, S5.	106
Fig. 5.9 Stress in 2 nd GaN as a function 2 nd GaN V/III ratio, obtained from Raman measurements on samples S3, S4 and, S5.	106
Fig. 5.10 Ex-situ bow as a function of of 2 nd GaN V/III ratio.	107
Fig. 5.11 Cross sectional TEM images obtained in the weak beam dark field mode with $g = 2\bar{1}\bar{1}0$ of samples S3 (a) and S5 (b).	109
Fig. 5.12 High resolution X-ray diffraction ω - 2θ scan of AlGaN/GaN HEMT heterostructure grown using AlGaN/AlN/GaN SMLs.....	111
Fig. 5.13 Reciprocal space mapping of AlGaN/GaN HEMT heterostructure along GaN ($10\bar{1}5$) direction.	112
Fig. 5.14 Capacitance-voltage measurement of AlGaN/GaN HEMT heterostructures, grown using AlGaN/AlN/GaN SMLs. Sheet carrier concentration obtained by integration of C-V profile is also presented.....	113
Fig. 5.15 Depth profile of carrier concentration in the AlGaN/GaN HEMT heterostructure obtained from CV measurements.....	113
Fig. 5.16 Mobility and sheet carrier density as a function of temperature for AlGaN/GaN HEMT heterostructures.	114
Fig. 5.17 2DEG mapping of sheet resistance (a), mobility (b) and sheet carrier concentration (c) of AlGaN /GaN HEMT heterostructures on 100-mm Si substrate.	115
Fig. 6.1 A schematic of measurement set up for the HLC and VLC measurements on AlGaN/GaN HEMT heterostructures.....	118

Fig. 6.2 HLC and VLC as a function of voltage in the AlGa _N /Ga _N HEMT heterostructures.	118
Fig. 6.3 SIMS atomic concentration profile as a function of depth for a AlGa _N /Ga _N HEMT heterostructure grown using Al _N /Ga _N SMLs.	119
Fig. 6.4 Photoluminescence spectra of AlGa _N /Ga _N HEMT heterostructures obtained at RT (a) and as a function of a measurement temperature (b).	121
Fig. 6.5 Cross sectional view of AlGa _N /Ga _N HEMT heterostructure, showing different etch depths for parallel conduction measurements.	122
Fig. 6.6 HLC measurements as a function of etch depth in the AlGa _N /Ga _N HEMT heterostructures.	123
Fig. 6.7 Cross sectional TEM image of AlGa _N /Ga _N HEMT heterostructure.	124
Fig. 6.8 HLC measurements as a function of CBr ₄ pressure obtained from samples S1 to S5 (a). VLC measurements as a function of voltage obtained on an undoped sample - S4 and the highest carbon doped sample - S5 (b).	126
Fig. 6.9 Williamson-Hall plots of Ga _N buffers of samples S1 & S3 (a) and S4 & S5 (b). Insets show the respective rocking scans along Ga _N (30 $\bar{3}$ 2) plane.	127
Fig. 6.10 AFM images of 20 x 20 μm^2 scan area of samples S1 (a), S3 (b), S4 (c) and S5 (d) obtained using amplitude mode.	129
Fig. 6.11 RT photoluminescence spectra of samples S4 and S5.	130
Fig. 6.12 Raman spectra of samples S4 and S5, showing E ₂ modes of 2 nd Ga _N (a) and A ₁ (LO) modes with the corresponding line shape profile fitting for different depths of the heterostructures.	131
Fig. 7.1 Cross sectional view of epilayer structures of SH-HEMT (a) and DH-HEMT (b).	138
Fig. 7.2 Bow of SH-HEMT (a) and DH-HEMT (b) heterostructures grown on 100-mm Si substrate.	139
Fig. 7.3 AFM surface morphology over 10 x 10 μm^2 scan area of SH- (a) and DH- (b)-HEMT heterostructures.	140
Fig. 7.4 Reciprocal space mapping along (10 $\bar{1}$ 5) plane for SH-(a) and DH-(b) HEMT heterostructures.	141
Fig. 7.5 n _s x μH product of SH- and DH-HEMT heterostructures as a function of temperature.	143
Fig. 7.6 Capacitance-voltage measurements along with their differentiated and integrated profiles for SH-HEMT (a) and DH-HEMT (b) heterostructures.	143

Fig. 7.7 Simulated conduction band and carrier distribution profiles of SH- (a) and DH- (b) HEMT heterostructures.	145
Fig. 7.8 I_{DS} - V_{DS} (a) and transfer (b) characteristics of DH-HEMT submicron devices.	146
Fig. 7.9 Comparison of buffer breakdown characteristics of SH- and DH- HEMTs.	146
Fig. 7.10 RHEED pattern of as loaded GaN template (a), after the Ga flash off (b) and after 500 nm thick over grown GaN by PA-MBE (c).	148
Fig. 7.11 AFM images of $5 \times 5 \mu m^2$ area of ammonia-MBE grown GaN template (a) and after GaN over grown by PA-MBE (b).	148
Fig. 7.12 RHEED pattern of 1 nm AlN (a), InAlN during the growth (b), InAlN after the growth (c).	149
Fig. 7.13 Indium composition in InAlN epilayer as a function of aluminum flux obtained from HR-XRD ω -2 θ scans.	150
Fig. 7.14 HR-XRD ω -2 θ scan along GaN (0002) with the dynamic simulation on sample C.	151
Fig. 7.15 HR-XRD RSM along GaN ($10\bar{1}5$) with the dynamic simulation on sample C.	151
Fig. 7.16 Capacitance-Voltage measurement on as grown InAlN/AlN/GaN HEMT structure. The sheet carrier concentration obtained by integration of the C-V profile is also presented.	152
Fig. 7.17 Hall measurement as a function of temperature on InAlN/AlN/GaN HEMT heterostructure.	152
Fig. 7.18 HR-XRD ω -2 θ scan on InAlN/AlN/GaN HEMT heterostructure before and after annealing.	153
Fig. 7.19 Mobility and sheet carrier concentration as a function of temperature for InAlN/AlN/GaN HEMT heterostructure before and after post growth annealing.	154

List of Tables

Table 1.1. Physical properties and advantages of GaN for electronic device applications [1-4]: Comparison with conventional semiconductors.....	1
Table 1.2. Physical properties of GaN in wurtzite and zincblende crystal structures [5]. .	3
Table 1.3. Properties of sapphire, SiC and Si(111) substrates for GaN epitaxy.	15
Table 3.1 Raman active phonon modes for GaN and AlN.....	49
Table 3.2. A list of various diffraction vectors and the corresponding visibility criteria of different dislocations.	55
Table 4.1. Strain in GaN was determined by micro-Raman spectroscopy. Screw and edge type TDD in GaN and buffer ^{a)} layer of samples A, B and C was determined by HR-XRD.....	67
Table 4.2. Raman shift of GaN-E ₂ phonon peaks and their corresponding stresses in 2 nd GaN layer of samples A to E. The average crack spacing was estimated from the microscope images of the buried cracks.	74
Table 4.3. Dislocation density in sample A to E, estimated from HR-XRD rocking curve scans.	83
Table 5.1 Crystal quality and final incremental stresses in the 2 nd GaN estimated from HR-XRD rocking scans and in-situ measurements, respectively, for samples S3, S4 and S5.	108
Table 6.1. List of samples with their SMLs used and the corresponding CBr ₄ BEP.	125
Table 6.2. Raman E2 peak position and the estimated stress in 2 nd GaN. Fitting parameters of A ₁ [LO] mode at different depths are also listed.	132
Table 6.3. Electrical properties of undoped and CBr ₄ doped AlGa _{0.3} N/GaN HEMT heterostructures.....	135
Table 7.1. FWHM of XRD rocking curves along different planes of buffers in SH- and DH-HEMT heterostructures.	142
Table 7.2. Electrical properties SH- and DH-HEMT heterostructures obtained using Hall, C-V and simulation.	142

Abbreviations

2D	Two dimensional
3D	Three dimensional
2DEG	Two-dimensional electron gas
AFM	Atomic force microscopy
BEP	Beam equivalent pressure
BFM	Beam flux monitor
BLC	Buffer leakage current
CCD	Charge-coupled device
CL	Cathodoluminescence
CRT	Cathode-ray tube
DMS	Desorption mass spectroscopy
FIB	Focused ion beam
FWHM	Full width at half maximum
FE-SEM	Field emission scanning electron microscopy
HAADF	High angle annular dark field
HEMT	High electron mobility transistor
HLC	Horizontal leakage current
HR-XRD	High resolution X-ray diffraction
HVPE	Hydride vapor phase epitaxy
LED	Light emitting diode
MBE	Molecular beam epitaxy
MOCVD	Metal organic chemical vapor deposition
PA-MBE	Plasma assisted molecular beam epitaxy
PPM	Parts per million
PPB	Parts per billion
RF	Radio frequency
RGA	Residual gas analyzer
RHEED	Reflection high energy electron diffraction
RMS	Root mean square

SCCM	Standard cubic centimeter per minute
SCR	Space charge region
SEM	Scanning electron microscopy
SIMS	Secondary ion mass spectroscopy
SML	Stress mitigating layer
TEC	Thermal expansion coefficient
TEM	Transmission electron microscopy
TDD	Threading dislocation density
VLC	Vertical leakage current
WBDF	Weak beam dark field

Abstract

GaN based high electron mobility transistors (HEMTs) have attracted great attention over the last two decades for the high power and high frequency applications due to their advantages of polarization induced high electron density, high break down field and high saturation electron velocity. Development of GaN based HEMT technology on heterogeneous substrates such as SiC, sapphire and silicon is necessary due to the lack of large diameter native substrates. Low cost, excellent quality, large area availability and the unique-possibility of integrating GaN based devices with well-established silicon electronics makes silicon the substrate of choice. However, large thermal coefficient mismatch (52%) and lattice constant mismatch (17%) of GaN with Si lead to poor crystalline quality and cracking of epitaxial layers. Thus, the main challenge is to grow crack-free, thick and high quality GaN buffers for GaN based HEMT heterostructures on large diameter Si substrate. This dissertation focuses on the growth and optimization of AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) substrate using ammonia-MBE growth process. Further, the feasibility of the growth of InAlN/GaN HEMT heterostructures on Si substrate using MBE growth technique has also been explored.

Using ammonia-MBE growth process, AlN nucleation on the Si substrate was optimized to achieve lower roughness and good crystal quality 1st AlN layer. GaN buffer of 1000 nm thickness grown on the optimized 100 nm thick AlN showed heavy cracking. In order to produce crack free GaN buffers, AlGaIn SML and AlN/GaN SMLs were studied. AlN/GaN SMLs stack was successful in achieving compressive stress, lower pit density, larger grain sizes and lower TDD in the GaN buffer compared to AlGaIn SML. Hence, AlN/GaN SMLs were chosen for the development of AlGaIn/GaN HEMT heterostructure growth on 100-mm Si substrate. A typical heterostructure with AlN/GaN SMLs consists of 50 nm of 1st AlN followed by the growth of 1st GaN/2nd AlN-SMLs. The 2nd GaN buffer was grown on top of the SMLs. Heterostructures grown using AlN/GaN SMLs intrinsically contain buried cracks in them. Using in-situ curvature measurements, different stages of buried crack formation such as the crack initialization and growth of strain free 2nd AlN followed by the lateral overgrowth of the cracked AlN were identified. Increased relaxation of 1st GaN with thickness was found to enhance the buried crack density. Moreover, the residual compression in 1st GaN together with the buried cracks in the heterostructure were found to act as a stress compensating mechanism to mitigate the stress from GaN to Si substrate. Thus, it is identified that AlN/GaN SMLs stack not only helps in inducing higher compression during the growth

of 2nd GaN but also mitigates the stress from 2nd GaN to Si substrate during cool down. Using AlN/GaN SMLs, crack free AlGa_{0.2}N/GaN HEMT heterostructures with 2nd GaN up to 1000 nm thickness has been achieved on 100-mm Si substrate. AlGa_{0.2}N/GaN HEMT heterostructures grown using AlN/GaN SMLs showed a room temperature mobility of 1340 cm²/V.s and carrier concentration of 1.13×10^{13} cm⁻², which resulted in a sheet resistance of 409 Ω/sq. Good uniformity is achieved in sheet resistance, sheet carrier concentration and mobility with their standard deviation values of 2.5, 0.6 and 2.6%, respectively, across 100-mm wafers.

Even though crack free AlGa_{0.2}N/GaN HEMT heterostructures were achieved using AlN/GaN SMLs with 2nd GaN thickness of 1000 nm, the obtained wafer was tensile bowed in the range of 30-70 μm on 100-mm Si substrate. In order to achieve nearly flat bows, a new stack of AlGa_{0.2}N/AlN/GaN SMLs were studied. From in-situ stress measurements, the 2nd GaN was observed to be relaxed more for the heterostructures grown using AlGa_{0.2}N/AlN/GaN SMLs compared to AlN/GaN SMLs. However, the wafer bow was very compressive (+82 μm) for the heterostructures grown using AlGa_{0.2}N/AlN/GaN SMLs, while AlN/GaN SMLs produced tensile bow (-35 μm). Increased relaxation of 2nd GaN with high compressive bow indicates that the stress mitigation from GaN to Si is higher for the heterostructure grown using AlGa_{0.2}N/AlN/GaN SMLs compared to AlN/GaN SMLs. Using cross sectional transmission electron microscope (TEM) analysis, bending and looping of dislocation at AlGa_{0.2}N/2nd AlN and AlGa_{0.2}N/2nd GaN interfaces was attributed to the high relaxation of 2nd GaN. Weak beam dark field images showed extra tilt in the crystallites at AlGa_{0.2}N/2nd GaN interface, which was attributed to the increased stress mitigation and compressive bowing of the wafer. By increasing the 2nd GaN thickness up to 1400 nm, the compressive bowing of the wafer achieved was < +35 μm for the heterostructures using AlGa_{0.2}N/AlN/GaN SMLs. AlGa_{0.2}N/GaN HEMT heterostructures grown using this structure showed a room temperature sheet carrier concentration of 0.88×10^{13} cm⁻² and mobility of 1380 cm²/V.s, which resulted in a sheet resistance of 517 Ω/sq. Moreover, the 2DEG electrical properties across the 100-mm wafer were observed to be uniform.

Good 2DEG characteristics and control over the bowing of the AlGa_{0.2}N/GaN HEMT heterostructures on 100-mm Si substrate have been achieved. However, for various HEMT heterostructures, grown using both AlN/GaN SMLs and AlGa_{0.2}N/AlN/GaN SMLs, an average horizontal leakage current (HLC) of $\sim 1 \times 10^{-3}$ A/mm and a vertical leakage current (VLC) of $\sim 1 \times 10^{-1}$ A/cm² were obtained at 20V. Oxygen was identified as the dominant impurity in the grown HEMT heterostructures from systematic SIMS and PL

measurements. In addition, a parallel conduction channel was identified in the GaN buffer at the interface of 2nd AlN and 2nd GaN. To increase the buffer resistance, GaN buffer was carbon doped using CBr₄ source during the growth. Two and one orders of reduction in the HLC and VLC were observed, respectively for the GaN buffer doped with the maximum available CBr₄ BEP of 1.86×10^{-7} mTorr in our system. AlGaIn/GaN HEMT heterostructures grown using the carbon doped GaN buffer with 200 nm thick undoped GaN near the channel exhibited good 2DEG characteristics and reduction in the buffer leakage current by two orders of magnitude.

To improve the confinement of 2DEG in AlGaIn/GaN HEMT heterostructures, AlGaIn/GaN/AlGaIn double heterojunction HEMT (DH-HEMT) heterostructures were grown and investigated. Hall measurements on DH-HEMT structures showed a room temperature mobility of 1510 cm²/V.s with a sheet carrier concentration of 0.97×10^{13} cm⁻². Capacitance-voltage measurements showed improved confinement of 2DEG for the DH-HEMT heterostructure, which helped in the enhancement of room temperature mobility. Further, it is observed that both SH- and DH-HEMT heterostructures show similar current driving capabilities. However, DH-HEMT shows 3 times higher buffer break down voltage compared to SH-HEMT.

Having optimized the AlGaIn/GaN HEMT heterostructures on Si (111) substrate, further efforts were made to achieve strain free lattice matched InAlN barrier growth for GaN based HEMT heterostructures. A combination of ammonia-MBE and PA-MBE growth processes was explored, where the GaN buffer was grown by ammonia-MBE while PA-MBE was used for low temperature InAlN barrier growth. InAlN/AlN/GaN HEMT heterostructure with indium composition of 24% was grown. Room temperature Hall measurement showed a carrier concentration of 7.3×10^{12} cm⁻² and mobility of 140 cm²/V.s. However, post growth annealing of InAlN/AlN/GaN HEMT heterostructures showed a room temperature carrier concentration of 1.85×10^{13} cm⁻² and mobility of 512 cm²/V.s. This improvement in the 2DEG properties is attributed to the improvement in the crystal quality of InAlN barrier due to annealing process. Thus, InAlN/AlN/GaN HEMT heterostructures with reasonable 2DEG characteristics have been demonstrated for the first time on Si substrate using a combination of ammonia-MBE and PA-MBE growth processes.

1. Introduction

GaN based semiconductors have become leading materials for optoelectronic, high power, high temperature and high frequency device applications. Last two decades have witnessed an enormous development in the GaN based technology and in particular, devices such as light emitting diodes (LEDs) and high electron mobility transistors (HEMTs) have progressed from the state of the art research to the commercialization. The attractiveness of GaN over other competing semiconductors such as Si, SiC, GaAs, etc. for various applications is due to its unique material properties. Table 1.1 presents some of the important physical properties of GaN in comparison with other semiconductors and their characteristic advantages for electronic device applications.

Table 1.1. Physical properties and advantages of GaN for electronic device applications [1-4]: Comparison with conventional semiconductors.

Material Properties	Si	GaAs	InP	4H-SiC	6H-SiC	GaN	Advantages of GaN
Band gap energy (eV)	1.11	1.42	1.35	3.26	3.02	3.40	High temperature and high voltage operation
Breakdown electric field (MV/cm)	0.3	0.4	0.5	2.2	2.4	3.3	High current density and high efficiency
Saturated drift velocity ($\times 10^7$ cm/s)	1.0	2.0	1.0	2.0-2.2	2.0	2.5	High switching frequency and high current handling
Thermal conductivity (W/cm.K)	1.5	0.5	0.7	4.5	4.5	1.3	Efficient heat removal for high current applications compared to GaAs and InP

As listed in Table 1.1, GaN clearly possesses advantages over conventional Si, GaAs and InP semiconductors for high temperature, high power and high frequency applications. Even though SiC (both 4H and 6H poly types) competes well with GaN from the materials perspective, the lack of formation of heterojunction and the corresponding

improvement in carrier densities and mobilities limit SiC based device operation only to low frequency and high power applications [1].

1.1 Gallium nitride - structural properties

GaN based materials exist mainly in two types of crystal structures, wurtzite (space group: $P6_3mc$) and zincblende (space group: $F\bar{4}3m$) as shown in the Fig. 1.1. In both crystal structures, each Gallium (Ga) atom is tetrahedrally coordinated with four nitrogen atoms and vice versa. However, both structures differ in stacking with a sequence of AaBbAa... for wurtzite along (0001) direction and AaBbCc... for zincblende along (111) direction.

Fig. 1.1 Stick-ball structures of GaN in wurtzite (a) and zincblende (b) forms.

Zincblende is a cubic based crystal structure and stabilizes only for thin epitaxial layers grown over cubic substrates, such as Si(001). Some of the physical properties of GaN in wurtzite and zincblende forms are listed in Table 1.2. As may be noted, most of the physical properties are similar in both structures. However, the formation of wurtzite structure is energetically more favorable than zincblende structure and is stable at room temperature. It consists of two interpenetrating hexagonal close packed structures separated in c direction by a distance of 5/8 of the cell height. The unit cell for the wurtzite structure consists of 6 atoms of each type. Unlike zincblende structure, wurtzite structure is non-centrosymmetric in nature, which results in a surface with either metal (Ga) or nitrogen (N) polarity, indicated by [0001] and $[000\bar{1}]$, respectively. In the tetrahedral bonding of GaN, the nitrogen atom is more electro-negative, which shifts the net electron cloud towards one atom and results in the development of polarization. Thus, the developed polarization along with the lack of inversion symmetry in the wurtzite structure results in a net polarization along [0001] growth direction and

is called “spontaneous polarization”. The developed polarization further helps in the formation of two dimensional electron or hole gas at the heterojunction interface between GaN and its ternary or quaternary compounds leading to higher mobilities. This particular property of GaN in wurtzite structure makes it more widely considered over zincblende structure for the GaN based electronic applications.

Table 1.2. Physical properties of GaN in wurtzite and zincblende crystal structures [5].

Physical, thermal and mechanical properties		
	Wurtzite	Zincblende
Lattice constant (Å)	a = 3.189, c = 5.185	a = 4.511
Density (g.cm⁻³)	6.11	6.15
Melting point (°C)	2500	2500
Binding energy (eV)	2.22	~2.22*
Linear expansion coefficients (×10⁻⁶)	a: 5.59, c: 3.17	5.59
Young's modulus (GPa)	150	181
Refractive index	2.85	2.9
Optical phonon energy (meV)	91.2	87.3
Dielectric constant (static)	10.4(E c) 9.5 (E⊥c)	9.7
Electron effective mass	0.20 m ₀	0.13 m ₀
Hole effective mass	0.80 m ₀	1.3 m ₀
Electron mobility (300K) (cm²V⁻¹s⁻¹)	~1400	≤ 1000
Hole mobility (300K) (cm²V⁻¹s⁻¹)	<20	≤ 350

* based on the negligible energy difference between wurtzite and zincblende forms [6]

1.2 Nitride compounds

One of the remarkable qualities of III-nitrides is their ability to form compounds over a wide composition range. As mentioned in Section 1.1, III-nitride compounds form heterojunction with GaN and produce quantum well at the heterojunction interface in GaN based HEMT structures. The electrons confine in a two dimensional space in the quantum well and leads to high electron mobility. In general, III-nitride compound semiconductors can be divided into ternary and quaternary nitride compounds.

1.2.1 Ternary nitrides

A wide range of ternary nitrides can be obtained by compounding any two of the binary nitrides. In general the lattice constant of the ternary compound follows Vegard's law. According to this law [7],

$$a \text{ or } c_{A_xB_{1-x}N} = x(a \text{ or } c_{AN}) + (1 - x)(a \text{ or } c_{BN})\text{Å} \quad \text{Eq. 1.1}$$

Here, ' a ' and ' c ' are the lattice constants along the basal and vertical planes of a wurtzite structure, respectively. AN and BN represents two individual binary nitride compounds such as AlN, GaN and InN. Here, ' x ' represents the mole fraction of binary compound 'AN' in the ternary ' $A_xB_{1-x}N$ ' compound.

Similarly, the band gap energy of a ternary compound can be expressed as

$$E_g(A_xB_{1-x}N) = xE_g(AN) + (1 - x)E_g(BN) - bx(1 - x) \text{ eV} \quad \text{Eq. 1.2}$$

Here, $E_g(AN)$ and $E_g(BN)$ are band gap energies of individual binary compound, while $E_g(A_xB_{1-x}N)$ is the band gap energy of the resultant ternary compound. ' b ' is the bowing parameter, which indicates the deviation of the band gap energy of ternary compound from the algebraic sum of individual binary compounds. The bowing parameter, ' b ' is different for different ternary nitride compounds. The following paragraphs summarize the bow parameters of $\text{Al}_x\text{Ga}_{1-x}\text{N}$, $\text{In}_x\text{Ga}_{1-x}\text{N}$ and $\text{In}_x\text{Al}_{1-x}\text{N}$ ternary compounds and discuss the variation of lattice constant and band gap energy as a function of binary mole fraction.

The reported bowing parameter for $\text{Al}_x\text{Ga}_{1-x}\text{N}$ compound varies from -0.8 to +2.6 eV [8] that results in a wide variation of AlGa_xN band gap energy values. The maximum variation in the reported values occurs around an Al mole fraction of 0.5, which can be attributed to the poor quality of material and its effect on the band gap energy determination. Yun et al., [8] have reported a revised bow parameter value of 1 eV by fitting the experimentally observed band gap energy value to the empirical Eq. 1.2. This value has been widely considered as the acceptable bow parameter for $\text{Al}_x\text{Ga}_{1-x}\text{N}$ compound. However, a further improvement in bow parameter estimation may be possible with the improved quality of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layers. Figure 1.2 shows the band gap energy and the basal plane lattice constant values of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ across the whole composition range. An increase in the Al mole fraction in GaN lattice results in the increase of the band gap energy of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ while decreasing its lattice constant. The electrical resistance of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ was found to increase with the increase in Al mole fraction or $\text{Al}_x\text{Ga}_{1-x}\text{N}$ band gap energy. An increase in the Al mole fraction from 0 to 30 % has resulted in decreasing the carrier concentration from 10^{20} to 10^{17} cm^{-3} and increasing the carrier mobility from 10 to 30 $\text{cm}^2/\text{V.s}$ [5]. The most important application of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ in electronic devices is its usage as a barrier layer in the conventional AlGa_xN/GaN based HEMT heterostructures.

Determination of compositional dependence of band gap energy of $\text{In}_x\text{Ga}_{1-x}\text{N}$ was found to be much trickier compared to $\text{Al}_x\text{Ga}_{1-x}\text{N}$. The high miscibility gap between InN and GaN make its growth difficult and leads to partial phase separation, which contributes to the greater disparity in the measured band gap energy values. Moreover, earlier reports suggested a band gap energy value of 1.9 eV [9] for InN but later it was universally accepted as 0.7 eV [10]. This variation in the band gap energy values together with partial phase separation has resulted in a huge spread in the reported bowing parameters. The bow parameters ranging from 1.2 [11] to 8.4 eV [12] resulted in larger variation in the band gap energy values. The largest bowing parameter of 8.4 eV was obtained by considering the band gap energy value of 1.9 eV for InN. However, Wu et al., [10] have determined the composition dependence of band gap energy of $\text{In}_x\text{Ga}_{1-x}\text{N}$ using absorption, photoluminescence and photo-reflectance measurements. They have obtained a bow parameter of 1.43 eV across all the composition range using a band gap energy value of 0.77 eV for InN in their calculation. The bow parameter of ~ 1.43 eV has been found to be the most followed value currently. Figure 1.3 shows the band gap energy and lattice constant of $\text{In}_x\text{Ga}_{1-x}\text{N}$ as a function of indium mole fraction in GaN lattice. Unlike AlGaN, increased indium mole fraction results in the decreased band gap energy while its lattice constant is increased. Optoelectronic applications such as LEDs [13] and laser diodes [14] are involved in the major use of $\text{In}_x\text{Ga}_{1-x}\text{N}$ system, which allows harvesting of optical emission in the violet and blue regions of optical spectrum. Nevertheless, the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer as a back barrier for GaN based HEMTs [15] is also explored due to its inherent property of opposite polarization field to the barrier, which improves the confinement of 2DED electrons in HEMTs.

The growth of $\text{In}_x\text{Al}_{1-x}\text{N}$ is much more challenging compared to $\text{In}_x\text{Ga}_{1-x}\text{N}$ as the spinodal phase separation is a more of an issue in this nitride system [16]. Onuma et al. [17] have determined a bowing parameter of 3.1 eV for $\text{In}_x\text{Al}_{1-x}\text{N}$ by considering the band gap energy measurements from PL. However, Pelá et al. [18] have determined a compositional dependent bowing parameter of $[3.4(1-x) + 1.2]$ using LDA-1/2 calculation technique. Using this method, the band gap energy variation of $\text{In}_x\text{Al}_{1-x}\text{N}$ as a function of 'In' mole fraction is plotted in Fig. 1.4. Additionally, basal plane lattice parameter of $\text{In}_x\text{Al}_{1-x}\text{N}$ is also plotted as a function of indium mole fraction. As shown, 'In' mole fraction of 17% is lattice matched to GaN with a band gap offset of ~ 1.3 eV. This huge band gap offset creates deep a quantum well with ΔE_c of ~ 1.0 eV, which helps to confine the 2DEG. Thus the advantage of using InAlN barrier is in the development of lattice matched InAlN/GaN HEMTs.

Fig. 1.2 Band gap energy and lattice constant variation as a function of 'Al' mole fraction in AlGaIn .

Fig. 1.3 Band gap energy and lattice constant variation as a function of 'In' mole fraction in InGaIn .

Fig. 1.4 Band gap energy and lattice constant variation as a function of 'In' mole fraction in InAlIn .

1.2.2 Quaternary nitrides

Quaternary nitride materials such as $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$ allow wider range of band gap energy and lattice constant selection by varying the individual compositions of In, Al and Ga in the lattice. Similar to $\text{In}_x\text{Al}_{1-x}\text{N}$ and $\text{In}_x\text{Ga}_{1-x}\text{N}$, growth of AlInGaN is challenging because of wide variation in the lattice constants, binding energies and growth temperatures of the three individual binary compounds. Lattice constants of quaternary nitride compound can be estimated using Vegard's law from the constituent-percentage of their binary compounds as presented in Eq. 1.3.

$$a \text{ or } c_{\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}} = x(a \text{ or } c_{\text{AlN}}) + y(a \text{ or } c_{\text{InN}}) + (1 - x - y)(a \text{ or } c_{\text{GaN}}) \text{ \AA}$$

Eq. 1.3

Further, the empirical formula to estimate the band gap energy of AlInGaN is expressed as

$$E_g(\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}) = xE_g(\text{AlN}) + yE_g(\text{InN}) + (1 - x - y)E_g(\text{GaN}) - b_{\text{AlGa}}x(1 - x) - b_{\text{InGa}}y(1 - y) \text{ eV}$$

Eq. 1.4

where, $E_g(\text{AlN}) = 6.2 \text{ eV}$, $E_g(\text{InN}) = 0.7 \text{ eV}$ and $E_g(\text{GaN}) = 3.4 \text{ eV}$. While x and y are AlN and InN mole fractions, $b_{\text{AlGa}} = 1 \text{ eV}$ and $b_{\text{InGa}} = 1.43 \text{ eV}$ gives the bow parameters due to AlGa and InGa mole fractions, respectively. Figure 1.5 shows the band gap energy of $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$ as a function of lattice constant.

Fig. 1.5 Band gap energy as a function of lattice constant in AlInGaN nitride system.

As shown, the area enclosed by the band gap energy vs lattice constant curves of the individual ternary nitrides represents the band gap energy of $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$. Moreover, it can also be seen that the $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$ gives an additional freedom to tune the band gap energy by keeping the lattice parameter nearly matched to GaN. This property further allows tuning of the polarization field and band offsets of the heterojunction interface independently, which can be useful for designing the devices for different applications.

1.3 GaN based HEMT heterostructures

GaN based HEMT heterostructures essentially contain a heterojunction interface between GaN buffer and a coherently grown group III- nitride based barrier epilayer. Due to the non-centrosymmetric nature of wurtzite structure, both group III-nitride barrier and GaN buffer inherently contain spontaneous polarization. In addition to that, coherently grown barrier induces additional strain at the heterojunction interface and distorts the crystal lattice along the 'c' axis. This results in the strain induced polarization known as piezoelectric polarization. The combination of spontaneous and piezoelectric polarizations develops a net polarization and results in a dipole of charges that are induced at the interface and at the surface of the barrier layer.

The net polarization induced charge density $\sigma_{POL}(x)$ at the hetero-interface is:

$$\sigma_{POL}(x) = P_{SP}(x) + P_{PE}(x) - P_{SP}(\text{GaN}) \text{ C. m}^{-2} \quad \text{Eq. 1.5}$$

where, 'x' is the mole fraction of the constituent element in the barrier, for example, 'x' represents 'Al' mole fraction in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ or $\text{Al}_x\text{In}_{1-x}\text{N}$ barrier. $P_{SP}(x)$ and $P_{PE}(x)$ are spontaneous and piezoelectric polarizations of the barrier layer, respectively. $P_{SP}(\text{GaN})$ represents the spontaneous polarization of GaN buffer. However, the contribution due to piezoelectric polarization from GaN buffer is negligible as it is almost relaxed in GaN based HEMT heterostructures.

$P_{SP}(x)$ depends upon the nitride compound that is used for the barrier layer, whereas $P_{PE}(x)$ can be expressed as,

$$P_{PE}(x) = e_{33}(x)\varepsilon_z + e_{31}(x)(\varepsilon_x + \varepsilon_y) \text{ C. m}^{-2} \quad \text{Eq. 1.6}$$

where $e_{33}(x)$ and $e_{31}(x)$ are the piezoelectric constants. ε_z and $\varepsilon_x = \varepsilon_y$, given as $\varepsilon_z = -2 \frac{C_{13}(x)}{C_{33}(x)} \varepsilon_x$ and $\varepsilon_x = \frac{a(x) - a_0(x)}{a_0(x)}$ are the elastic strains along 'c' and 'a' axis, respectively. $a(x)$ and $a_0(x)$ are the measured and relaxed lattice constants of the nitride barrier. For a coherently grown barrier layer, the measured lattice constant $a(x)$ is equal to the lattice constant of GaN buffer layer.

Substituting the above equations in the expression for P_{PE} results in

$$P_{PE}(x) = 2 \frac{a(x)-a_0(x)}{a_0(x)} \left[e_{31}(x) - e_{33}(x) \frac{C_{13}(x)}{C_{33}(x)} \right] \text{C. m}^{-2} \quad \text{Eq. 1.7}$$

Thus, the total polarization induced charge density that develops at the heterojunction interface of GaN based HEMT structures is given by

$$\sigma_{POL}(x) = P_{SP}(x) - P_{SP}(\text{GaN}) + 2 \frac{a(x)-a_0(x)}{a_0(x)} \left[e_{31}(x) - e_{33}(x) \frac{C_{13}(x)}{C_{33}(x)} \right] \text{C. m}^{-2} \quad \text{Eq. 1.8}$$

The developed polarization charge drives either electrons or holes into the quantum well at the heterojunction to create a two dimensional electron or hole gas, which further depends upon the sign of the polarization charge itself. The direction of the polarization field and consequently, the sign of polarization induced charge depends upon the polarity of GaN. Ambacher et al. [19] have studied in detail the effect of polarity of GaN on both the spontaneous and piezoelectric polarization induced charge distribution in GaN based heterostructures. Gallium polar epilayers always result in spontaneously induced polarization field towards GaN buffer layer, whereas the direction of piezoelectric polarization depends upon the piezoelectric and mechanical constants and the strain state of barrier. The directions of both the spontaneous and piezoelectric polarization fields reverse for N-polar GaN epilayers. Since HEMT heterostructures require a high density of electron gas in the quantum well, Ga polar GaN buffer layer is necessary to induce high density of positive polarization charges at the heterostructures interface. Ibbetson et al. [20] further showed that the source of 2DEG in the GaN based HEMT heterostructures is surface states, which donate electrons to the quantum well.

1.3.1 AlGaIn/GaN HEMT heterostructure

A typical AlGaIn/GaN HEMT heterostructure consists of an AlGaIn barrier grown coherently on the surface of GaN buffer as shown in Fig. 1.6 (a). Moreover, due to the spontaneous and piezoelectric polarizations, polarization induced charges develop in the AlGaIn barrier as shown in the figure. Further, the conduction band energy difference between AlGaIn and GaN develops a quantum well at the heterojunction interface as shown in Fig. 1.6 (b). Moreover, the developed positive charge at the AlGaIn/GaN interface drives electrons into the quantum well to form two dimensional electron gas (2DEG) as shown in Fig. 1.6 (b).

Fig. 1.6 Schematic of a typical AlGaIn/GaN HEMT heterostructure (a) and its band diagram (b).

The spontaneous polarization charge (P_{SP}) for the AlGaIn barrier is expressed as

$$P_{SP}(x) = (-0.052x - 0.029) \text{ C.m}^{-2} \quad \text{Eq. 1.9}$$

where, -0.029 C.m^{-2} corresponds to the spontaneous polarization charge of GaN and 'x' is the 'Al' mole fraction.

The piezoelectric polarization (P_{PE}) can be expressed as

$$P_{PE}(x) = 2 \frac{a(x) - a_0(x)}{a_0(x)} \left[e_{31}(x) - e_{33}(x) \frac{c_{13}(x)}{c_{33}(x)} \right] \text{ C.m}^{-2} \quad \text{Eq. 1.10}$$

As shown in Fig. 1.2, the lattice constant of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ barrier is always smaller than GaN buffer, which results in tensile strain generation in the barrier. Furthermore, $\left(e_{31}(x) - e_{33}(x) \frac{c_{13}(x)}{c_{33}(x)} \right) < 0$ for the whole composition range of $\text{Al}_x\text{Ga}_{1-x}\text{N}$, resulting in $P_{PE}(x) < 0$. Thus, both $P_{SP}(x)$ and $P_{PE}(x)$ are negative for Ga polar AlGaIn/GaN based

HEMT heterostructures, which are responsible for the high density of polarization induced positive charge at the hetero interface.

Thus, by substituting for $P_{SP}(x)$ and $P_{PE}(x)$ in Eq. 1.5, the total polarization induced charge density in AlGa_xN/GaN heterostructure with 'x' Al mole fraction is expressed as

$$\sigma_{POL}(x) = \left[2 \frac{a(x) - a_0(x)}{a_0(x)} \left(e_{31}(x) - e_{33}(x) \frac{C_{13}(x)}{C_{33}(x)} \right) - 0.052x \right] \text{ C. m}^{-2} \quad \text{Eq.1.11}$$

Here, $a_0(x)$, $e_{31}(x)$, $e_{33}(x)$, $C_{13}(x)$ and $C_{33}(x)$ for the Al_xGa_{1-x}N can be obtained from the linear interpolation of their individual binary values.

The two dimensional electron gas $n_s(x)$ developed at the interface between AlGa_xN/GaN heterostructure to compensate for the positive polarization induced charge is given by

$$n_s(x) = \left| \frac{\sigma_{POL}(x)}{e} \right| - \left(\frac{\epsilon_0 \epsilon(x)}{de^2} \right) (e\phi_b(x) + E_F(x) - \Delta E_C(x)) \text{ cm}^{-2} \quad \text{Eq. 1.12}$$

where $\epsilon(x)$ is the relative dielectric constant of AlGa_xN, d is the barrier layer thickness, $e\phi_b$ is the Schottky-barrier height of the gate contact and E_F is the Fermi level energy with respect to the conduction band energy of GaN.

1.3.2 AlGa_xN/GaN/AlGa_xN double heterojunction HEMT heterostructure

In AlGa_xN/GaN based HEMT heterostructures, the 2DEG is confined in the triangular quantum well at the interface as shown in Fig. 1.6. However, due to the presence of lower potential barrier at the side of GaN buffer, the 2DEG may spill over from quantum well into the GaN buffer, causing electrons to behave like three dimensional in nature. The spilling of electrons degrades the electrical characteristics and also causes the buffer punch through effect in GaN based HEMT heterostructures. In order to enhance the properties of AlGa_xN/GaN HEMT heterostructures by improving the electron confinement, growth of another heterojunction at the back of GaN channel was proposed. A typical AlGa_xN/GaN/AlGa_xN based double heterojunction HEMT (DH-HEMT) structure along with its energy band diagram are shown in Figs. 1.7(a) and (b), respectively. As shown, the introduction of AlGa_xN back barrier causes a significant upshift of GaN bands and increases the potential of the buffer layer. This helps in improving the confinement of 2DEG at the quantum well of top AlGa_xN/GaN interface. Moreover, AlGa_xN buffer in DH-HEMT might also improve the buffer resistance, and hence the overall power capability of HEMT devices.

Fig. 1.7 Schematic of a typical AlGaIn/GaN/AlGaIn DH-HEMT heterostructure (a) and its band diagram (b).

1.3.3 Lattice matched GaN based HEMT heterostructures

It is well known that lattice mismatch between AlGaIn and GaN epilayers in AlGaIn/GaN HEMTs induce high biaxial strain at the interface. Even though, the developed strain helps in generating the piezoelectric charges and consequently 2DEG, it also provides an unstable condition at the interface region. Recent studies on the reliability of AlGaIn/GaN HEMTs showed lower reliability of these devices and the cause for it has been attributed to the generation of strain induced defects in AlGaIn barrier [21, 22]. In addition, further improvement in the power capabilities of AlGaIn/GaN HEMTs requires increased current densities and hence increased sheet carrier density in the 2DEG. Enhancement of sheet carrier density can be obtained by increasing the Al mole fraction in AlGaIn barrier. But, it increases the biaxial strain further at the interface. Moreover, higher Al content induces more alloy scattering and correspondingly decreases the mobility of 2DEG [23].

Furthermore, device operation frequency in HEMTs can be increased by decreasing the channel length, which requires thinning of the barrier layer without causing any short channel effects [24, 25]. Ibbetson et al. [20] have shown that the carrier density (n_s) of

2DEG in GaN based HEMT heterostructures depends upon the barrier thickness (d) according to Eq. 1.13:

$$n_s = \frac{1}{e} [\sigma_{POL} (1 - d_{CR}/d)] \quad \text{Eq. 1.13}$$

where the critical barrier thickness, $d_{CR} = (E_D - \Delta E_C)\epsilon/e\sigma_{POL}$. Here, ΔE_C and ϵ are the conduction band offset with respect to Fermi level and dielectric constant of the barrier layer, respectively and E_D is the donor level of the surface states in GaN based HEMT heterostructures. As can be observed from Eq. 1.13, decreasing the barrier thickness reduces the sheet carrier concentration considerably due to the depletion of polarization induced charges. In case of AlGaIn/GaN HEMTs, reduction of barrier thickness below 25 nm results in decreased sheet concentration [26], which further limits the scaling of AlGaIn/GaN HEMTs and hence their frequency of operation.

1.3.3.1 In_{0.17}Al_{0.83}N/GaN HEMT heterostructures

In order to address the above mentioned limitations with AlGaIn/GaN HEMTs, Kuzmik [27] has proposed the usage of lattice matched In_{0.17}Al_{0.83}N as a barrier layer for GaN HEMTs. Lattice matched In_{0.17}Al_{0.83}N barrier eliminates the stress development at the interface, which consequently improves the long term stability of these devices. The spontaneous polarization (P_{SP}) for In_xAl_{1-x}N barrier is expressed as

$$P_{SP}(x) = (0.049x - 0.081) \text{ C.m}^{-2} \quad \text{Eq. 1.14}$$

For the lattice matched composition of 17%, $P_{SP}(0.17) = -0.0727 \text{ C.m}^{-2}$ and $P_{PE}(x) = 0$.

Thus,

$$\sigma_{POL}(x) = P_{SP}(x) - P_{SP}(GaN) \quad \text{Eq. 1.15}$$

$$\sigma_{POL}(In_{0.17}Al_{0.83}N/GaN) = -0.0439 \text{ C.m}^{-2} \quad \text{Eq. 1.16}$$

The lattice matched In_{0.17}Al_{0.83}N barrier induces higher polarization charge than AlGaIn barrier due to its increased mole fraction of AlN, which results in higher sheet carrier density (2DEG) of $n_s = 2.8 \times 10^{13} \text{ cm}^{-2}$. Consequently, enhanced device current and power densities can be achieved. In AlGaIn/GaN HEMT heterostructures, the donor level related to surface states lies 1.9 eV below the conduction band whereas it is just 0.4 to 0.6 eV [28] below the conduction band for the InAlN barrier. Shallow surface donor levels for InAlN allows thinner barriers, without depleting the polarization induced surface charges. This provides negligible reduction in the sheet carrier density of 2DEG with the

decrease in barrier thickness. This effect has been experimentally demonstrated by achieving a sheet carrier concentration of $1.7 \times 10^{13} \text{ cm}^{-2}$ for InAlN barrier with a thickness less than 10 nm. Thinner barrier layer also decreases the parasitic capacitance occurring at the gate-source and gate drain junctions, which improves the frequency of operation of GaN based HEMT devices.

1.3.3.2 $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}/\text{GaN}$ HEMT heterostructures

Similar to $\text{In}_{0.17}\text{Al}_{0.83}\text{N}$, it is possible to grow lattice matched barrier using $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$ as mentioned in Section 1.2.2. Moreover, by inserting GaN in the lattice of InAlN, the growth temperature of barrier can be increased, which further improves the crystal quality of the barrier. Lim et al. [29] have increased growth temperature of barrier from 420 °C (for the AlInN barrier) to 580 °C (for the AlInGaN barrier) and observed an improvement in the electrical characteristics in terms of reduced sheet resistance for the quaternary barrier [30]. For the lattice matched composition of $\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}$, the piezoelectric polarization (P_{PE}) is zero, whereas the spontaneous polarization can be expressed as a linear interpolation of its ternary compositions, which can further be simplified as follows:

$$P_{SP}(\text{Al}_x\text{In}_y\text{Ga}_{1-x-y}\text{N}) = xP_{SP}(\text{AlN}) + yP_{SP}(\text{InN}) + (1-x-y)P_{SP}(\text{GaN}) + \\ b_{\text{AlGa}}(x)(1-x-y) + b_{\text{InGa}}(y)(1-x-y) + \\ b_{\text{AlIn}}(x)(y) \text{ C.m}^{-2} \quad \text{Eq. 1.17}$$

It can be inferred from Eq. 1.17 and Eq. 1.4 that the quaternary nitride barrier allows designing and controlling of the band gap energy offsets and the polarization fields independently by keeping the lattice matching condition to GaN. This flexibility allows designing of the HEMT heterostructures for depletion mode [31] and enhancement mode [32] device operation.

1.4 Substrates for GaN based HEMT heterostructures

GaN substrates for homoepitaxial growth of HEMT heterostructures are available only in smaller sizes and are expensive. This is because of the difficulty in the growth of bulk GaN crystals from the stoichiometric liquids with high melting temperature (~ 2500 °C) and high equilibrium N_2 over pressure ($\sim 45,000$ bar). This leaves the only choice of using heteroepitaxial growth for the growth of GaN based device structures. Among various substrates investigated, sapphire, SiC and Si are widely used for the growth of GaN based electronic and optoelectronic device heterostructures. Properties of these substrates are compared in the Table. 1.3.

Table 1.3. Properties of sapphire, SiC and Si(111) substrates for GaN epitaxy.

Property	Sapphire (Al₂O₃)	6H-SiC	Si(111)
Lattice constant (Å)	4.785	3.0806	3.846
Thermal expansion coefficient ($\times 10^{-6}$) K⁻¹	9.03 (c axis) 5.0 ($\perp$ c axis)	4.16 (c axis) 4.46 ($\perp$ c axis)	2.616
Thermal conductivity (W/cm.K)	0.23 (c axis) 0.25 ($\perp$ c axis)	4.5	1.5
Epitaxial relationship with GaN	GaN:Sapphire [0001]:[0001] $[\bar{1}2\bar{1}0]:[\bar{1}100]$ (30° in-plane rotation)	GaN:SiC [0002]:[0006] [11 $\bar{2}$ 0]:[11 $\bar{2}$ 0]	GaN:Si [0001]:[111] [2 $\bar{1}\bar{1}$ 0]:[011]

As can be observed from Table 1.3, the sapphire substrate offers higher lattice mismatch of 15% and a thermal mismatch of -30% to GaN resulting in a compressive strain development. However, the high lattice mismatch along with an in plane rotation of 30° in GaN induces high dislocation density in the epitaxial layers and degrades the operation of GaN based devices. Further, the major disadvantage of using sapphire substrates for GaN growth for electronic applications is its poor thermal conductivity, which results in self-heating of the devices during the operation and reduces their performance. SiC is more suitable for GaN heteroepitaxial growth due to its low lattice mismatch of ~ 3% and thermal mismatch of 20%. Unlike the growth on sapphire, GaN on SiC follows epitaxial relation without any rotation of the crystallites. This helps in producing low dislocation density and good crystal quality epitaxial layers, suitable for electronic device applications. Even though the thermal mismatch of 20% between GaN and SiC results in tensile strain in GaN during cool down, it can be compensated during the growth by using an AlN buffer. Though there are advantages, the main limitations of SiC substrate are that it is only available in smaller diameter and is expensive.

Availability of large diameter wafers up to 12", comparatively lower prices and better crystal quality substrates due to its mature technology make silicon substrate a better option for GaN epitaxial growth. Moreover, the establishment of high quality GaN growth on silicon substrate opens the unique opportunity to integrate matured silicon technology with GaN devices. However, growth of GaN on Si(111) substrate is challenging due to high lattice mismatch of 17% and thermal mismatch of 52% , which

induce high tensile strain during the growth and cool down, resulting in cracking of the epilayers. Moreover, higher lattice mismatch induces high dislocation density in GaN layers, which hampers the device performances. Nevertheless, intense efforts are being made to develop GaN-on-Si growth technology by research groups world-wide to fully exploit the advantages of using Si substrates.

1.5 Challenges in the growth of GaN based HEMT heterostructures on Si(111)

GaN growth on Si(111) faces some particular challenges such as melt back etching, nucleation issues, high tensile strain generation and cracking, poor crystal quality and leaky GaN buffer. The details of these issues are discussed as follows:

Due to the large lattice and thermal mismatch between GaN and Si, growth of nucleation layer on Si substrate is crucial. The growth of GaN directly on Si results in melt back etching and formation of Si_xN_y amorphous layer at the interface leading to polycrystalline nature of GaN layer [33]. Growth of an AlN epilayer on Si substrate avoids the direct contact of Ga with silicon and results in reduced melt back etching. Higher bonding energy of AlN (2.88 eV) further avoids the formation of amorphous Si_xN_y layer. Moreover, AlN layer additionally reduces the tensile stress in GaN. However, typical growth temperatures for obtaining good quality AlN are in the range of 700 to 930 °C in the molecular beam epitaxy (MBE) growth process. At these high temperatures, there is an increased probability of aluminum to diffuse into the silicon substrates as well as silicon to diffuse into AlN and GaN epilayers. The former causes *p*-type inversion layer [33] in Si substrate whereas the later causes the reduction in resistance of buffer layer and increases the GaN buffer leakage. Thus, the optimization of AlN nucleation is necessary to produce good quality GaN based heterostructures.

As mentioned, another important challenge in the growth of GaN on Si(111) is the cracking of epitaxial layers during the cool down of the substrate from the growth temperature to room temperature. High lattice mismatch (~17%) and thermal expansion coefficient mismatch (~52%) between these materials result in tensile strain and formation of cracks in the epilayers eventually. Further, higher lattice mismatch also induces higher dislocation density in the GaN epilayers, which not only increases the buffer conduction [34] but also affects the 2DEG properties of HEMT heterostructures [35]. In order to overcome the tensile strain and cracking issues, several groups introduced stress mitigating layers such as AlN interlayer [36, 37], AlGaIn interlayer [38, 39] and AlGaIn/GaN superlattices (SL) [40] for the growth of GaN on Si substrates. Thus,

the optimization of stress mitigation layer growth is essential to produce crack free thick GaN layers with good crystalline quality. GaN material is intrinsically *n*-type in nature. Moreover, un-intentional dopants such as nitrogen vacancies, oxygen and silicon increase the conduction in GaN layers. Thus, the reduction of back ground carrier concentration or compensation with deep acceptor dopants such as Fe [41] or C [42] are necessary to produce semi-insulating GaN layer to achieve sharp pinch off for GaN based HEMTs. Furthermore, it is extremely important to have reproducible Ga polarity- GaN buffer layers to obtain 2DEG in GaN based HEMT heterostructures.

Continuous efforts are being made by research groups to address the various issues associated with GaN based HEMT heterostructure growth on Si substrate by metal organic chemical vapor deposition (MOCVD) and MBE growth techniques. Lower growth temperatures, sharp interfaces, hydrogen free growth environment and in-situ monitoring capabilities are some of the advantages of using MBE growth technique. In particular, MBE growth using ammonia as a nitrogen precursor offers wide growth window and reproducible Ga polarity, which are important for GaN based HEMT heterostructures. Good quality AlGaIn/GaN based HEMT heterostructures have been demonstrated on 50 mm diameter Si(111) substrates by ammonia-MBE [37]. In this report, growth and characterization of AlGaIn/GaN based single and double heterojunction HEMT structures on 100mm-Si(111) substrates using ammonia-MBE are studied in detail. Moreover, there are no reports of lattice matched HEMT heterostructure growth on Si(111) by MBE growth technique. Thus, in order to address the above mentioned challenges in the group III- nitride research, this thesis is devoted to the growth and optimization of crack free, thick and high resistive GaN buffer for AlGaIn/GaN based SH- and DH-HEMT heterostructure on 100-mm Si (111) by ammonia-MBE. Further, the growth and the optimization of lattice matched InAlN HEMT heterostructure on Si(111) has also been attempted in this thesis.

1.6 Objectives

The study involves the growth of AlGaIn/GaN based HEMT structures on 100-mm Si(111) using ammonia-MBE and InAlN/GaN based HEMT heterostructures on Si substrates using PA-MBE.

1. High tensile bowing of the wafer and cracking of GaN epitaxial layers on larger diameter Si substrate occur majorly during the cool down process due to the thermal expansion coefficient mismatch between these materials. Higher residual compressive stress in GaN layers during the growth or the mitigation of

stress from GaN to Si during cool down avoids the cracking. Thus, an objective is set to investigate these properties in the heterostructures with different stress mitigating layers using characterization tools such as in-situ curvature measurement technique, Raman spectroscopy and ex-situ bow measurements to achieve crack-free, lower bow thick GaN buffers on Si substrate.

2. Impurities such as oxygen and silicon or the crystalline defects such as nitrogen vacancies and dislocations can cause higher conduction in GaN buffer. Thus, the second objective is to investigate and identify the source of conduction in GaN buffer using characterization techniques such as secondary ion mass spectroscopy, photoluminescence and buffer leakage measurements. Carbon doping of GaN buffers is generally used to compensate the donor impurities and conduction paths to achieve higher resistance of GaN buffers. Investigation of buffer resistance, structural quality and surface morphology of carbon doped GaN buffers using CBr₄ source is also a part of the second objective of this work.
3. Uniformity in electrical characteristics across the substrate diameter depends upon the growth technique and growth parameters that are used. Thus, the third objective is the study of AlGaIn/GaN HEMT heterostructures on 100-mm Si substrate to demonstrate uniform 2DEG properties across 100-mm wafer using MBE growth technique.
4. InAlN/GaN HEMT heterostructure growth on Si substrate is challenging due to non-equilibrium growth conditions that are required for InAlN growth and the relatively poorer quality of GaN buffers on silicon. Establishment of a combined growth processes involving GaN buffer growth using ammonia-MBE and InAlN growth using PA-MBE and demonstration of InAlN/GaN HEMT heterostructure on Si is set as the last objective of the study.

1.7 Organization of the dissertation

The dissertation mainly focuses on the growth optimization of AlGaIn/GaN HEMT heterostructures on 100-mm Si substrate using ammonia-MBE growth process. Further, it also addresses the growth of advanced HEMT heterostructures such as AlGaIn/GaN/AlGaIn double heterojunction HEMT and InAlN/AlN/GaN HEMT heterostructures.

In chapter 1, Characteristic advantages of GaN in comparison with other semiconductors such as GaAs, InP, SiC and Si are summarized followed by the description of physical properties of GaN in wurtzite and zincblende forms. Band gap energy and lattice

constant variation of ternary and quaternary nitrides are further discussed. Analytical equations describing the polarization in GaN based HEMT heterostructures are presented. Finally, challenges involving the growth of GaN based HEMT heterostructures on Si substrate are discussed followed the objectives set for the thesis.

Chapter 2 primarily reviews the literature on the growth of GaN based HEMT heterostructures on Si substrate. Growth of GaN using MOCVD and MBE growth techniques are discussed. Stress management techniques for the growth of GaN on Si are presented followed by the discussion on the GaN buffer conduction. Finally, a brief review of literature on GaN based HEMT heterostructures grown by MBE is presented.

Chapter 3 describes the details of various modules and components of the MBE growth system used. It also discusses in-situ monitoring tools such as reflection high energy electron diffraction (RHEED) and in-situ curvature measurement tool that are integrated with the MBE. This chapter further describes various structural, optical and electrical characterization tools such as HR-XRD, AFM, Raman, Hall and CV that are used to study GaN based HEMT heterostructures.

Chapter 4 discusses the growth and characterization of AlGaIn/GaN HEMT heterostructures on Si substrate using AlN/GaN stress mitigating layers. Nucleation of AlN on Si substrate is discussed followed by the investigation on AlGaIn and AlN/GaN stress mitigating layers. Stress mitigation property of AlN/GaN SMLs is studied using in-situ stress and Raman measurements. The optimization of the growth of 2nd GaN is presented followed by the demonstration of AlGaIn/GaN HEMT heterostructure on Si substrate.

In Chapter 5, growth and characterization of AlGaIn/GaN HEMT heterostructures on Si substrate using AlGaIn/AlN/GaN stress mitigating layers is discussed. The effect of this stack of SMLs on the 2nd GaN is presented in comparison with AlN/GaN SMLs. Stress mitigation effect has been discussed using various characterization techniques. Optimization of the surface morphology of 2nd GaN is also discussed. Finally, electrical measurements including two dimensional electron gas characteristics are presented.

In Chapter 6, horizontal and vertical leakage current measurements on the HEMT heterostructures are discussed. SIMS and PL measurements are studied to investigate and identify the dominant impurity in GaN buffer. This study is followed by the investigation on parallel conduction in the heterostructure. In order to increase the buffer resistance, carbon doping of GaN buffers using CBr₄ source is investigated.

Finally, the electrical characteristics of AlGaN/GaN HEMT heterostructures with carbon doped GaN buffers are presented.

Chapter 7 discusses the growth and characterization of advanced HEMT heterostructures such as AlGaN/GaN/AlGaN double heterojunction HEMT heterostructures to improve the confinement of 2DEG and the buffer breakdown voltage. InAlN/AlN/GaN HEMT heterostructures have also been studied towards achieving strain-free barrier and high 2DEG in the channel.

Chapter 8 gives the conclusions of this dissertation, while the possible future scope of work using MBE growth technique is described in Chapter 9.

2. GaN based HEMT heterostructures on Si(111)

GaN based HEMT heterostructure technology gained a lot of momentum over the last decade and is in the initial phases of commercialization for high power and high frequency applications. One of the main driving factors for the continuous research and potential commercial success of this technology is the improved material properties of GaN based heterostructures coupled with its development on Si(111) substrates. The possibility of growing GaN based HEMT heterostructures on large diameter Si substrates not only reduces the cost of production but also possibly avails the use of silicon foundries. However, as discussed in Chapter 1, growth on Si(111) is a challenging issue that requires careful optimization to develop reproducible and high performance HEMT technology. Growth of GaN based HEMT heterostructures on Si was developed mainly using MOCVD technique and MBE growth techniques. Following is the discussion on the MOCVD and MBE growth methods for the GaN growth.

2.1 Growth techniques

2.1.1 Metal organic chemical vapor deposition

Typical growth process in the MOCVD technique involves the transport of group III organometallics such as, tri-methyl gallium ($(CH)_3Ga$), indium ($(CH)_3In$) and aluminum ($(CH)_3Al$) to the high temperature growth surface, where they react with the hydrides like ammonia (NH_3) to form III-nitrides through pyrolysis process. Unlike the case on sapphire, low temperature GaN buffer grown on silicon has resulted in poor and pitted surface morphologies [43, 44]. Out diffusion of Si [45] and Ga-Si alloy reaction with corresponding melt back etching [43] were attributed to the observed poor surface morphologies.

AlN nucleation layer was found to be more successful for GaN growth, as it prevents direct contact of Ga with Si and avoids melt back etching. It also induces compressive stress in GaN epilayers, which helps in the mitigation of thermal stress and prevents cracking of the epilayers. However, optimization of the growth of the first AlN layer on silicon substrate and its nucleation is extremely important to achieve high crystal quality GaN buffer. Formation of amorphous SiN_x layer due to the reaction of nitrogen precursors like ammonia with Si is one of the major reasons for the degradation of GaN crystal quality. Deposition of few monolayers of 'Al' on silicon was found to prevent the nitridation of Si surface [46]. Moreover, the time of predeposition of aluminum is identified as an important parameter [47]. While higher 'Al' predeposition resulted in

rougher surface morphology, lower or no 'Al' predeposition resulted in island growth of AlN layers. Amano et al. [48] have first time demonstrated the growth of low temperature AlN buffer for the GaN epitaxy on sapphire and showed improved quality of epitaxial layers. Dadgar et al. [49] have used deposition of a few mono layers of Al, followed by LT-AlN nucleation layer on Si (111) and achieved best quality GaN layers.

Reacted compounds involving 'Ga' in MOCVD are weaker electron acceptors compared to 'Al' related compounds and resulted in their easy dissociation and lesser incorporation in GaN lattice. Thus, GaN growth using MOCVD technique requires much higher V/III ratios (typically >1000), which consequently increase the abundance of Ga related compounds and their subsequent incorporation. Higher V/III ratio also helps to provide high nitrogen partial pressures during the growth of nitride epitaxy, which provides its near equilibrium growth atmosphere. The quality and properties of GaN in MOCVD growth process can be changed by tuning several growth parameters such as V/III ratio, growth temperature, growth pressure and growth mode during the early stages of GaN growth. As mentioned, increase in the V/III ratio of GaN not only increases its growth rate but also decreases the impurity incorporation. It was also reported that the increased V/III ratio was found to have improved the surface morphology [50]. Moreover, higher V/III is required for the growth at higher temperatures to obtain smoother surface morphologies. Hence, V/III ratio was found to play a pivotal role in the structural, surface and growth properties of GaN using MOCVD growth technique.

Reactor pressure of MOCVD growth chamber is another crucial parameter in determining the properties of GaN. An increased reactor pressure from 100 to 300 Torr has resulted in decreasing the growth rate by 7 times [51]. The decrease in the growth rate has been attributed to the increased pre-reaction between tri-methyl gallium and ammonia. Surface morphology of GaN was also found to have more roughened with the increased reactor pressure [52]. The smoother surface morphologies obtained for low pressure growth was attributed to the increased island density during early stages of the growth and their subsequent faster coalescence to 2D growth mode. However, smoother surface morphologies were found to have associated with more dislocation density. Numerous dislocations generated at the coalescence boundaries have been attributed for the increased dislocation density in the samples grown with lower pressure. Thus, by tailoring the growth pressure, it is possible to initiate the growth with low island density followed by the rapid transformation to 2D growth mode. This process ensures both lower dislocation density and smoother surface morphologies in

GaN [53]. Thus, using MOCVD technique and optimizing the growth parameters as discussed, good quality GaN buffer layer with high growth rate, lower impurity incorporation and smoother surface morphology can be obtained.

2.1.2 Molecular beam epitaxy

Even though GaN epilayer and heterostructures growth can be achieved on larger diameter wafers with high growth rates using MOCVD growth technique, MBE growth technique presents several advantages over MOCVD. They include atomically sharp interfaces, lower temperature growth regime, in-situ diagnostic techniques, impurity-free growth environment and low source consumption. In this growth method, high purity materials are heated in effusion cells and the evaporated materials are projected on to the substrate surface, where they react and form compound materials. By controlling the shutters of the effusion cells, atomically sharp interfaces with good uniformity can be achieved using MBE growth process. Therefore, MBE is a very suitable growth technique for precisely controlling the growth parameters and to gain the insight into the growth kinetics at every growth step to develop a deeper understanding of the growth process.

Another advantage of MBE is that the growth process generally takes place in ultra-high vacuum conditions, which reduces the interaction of atomic species on their way to the substrate and hence keeps their thermal activated kinetic energies sufficient enough to form compounds even at lower temperatures. This property of MBE makes it particularly attractive over MOCVD to grow materials in thermodynamically non equilibrium conditions such as low temperature growth regime for Indium-based nitrides. Moreover, lower growth temperatures in MBE growth process also help in decreasing the diffusion of individual atoms leading to abrupt doping profiles. The use of ultra-high vacuum further enables employing in-situ monitoring techniques such as RHEED, reflectance spectroscopy, desorption mass spectroscopy (DMS) and cathodoluminescence (CL), which not only aid in the monitoring of the growth process but also help in controlling the growth. Usage of high purity materials for the growth along with UHV conditions reduces the contamination in the epitaxial growth process and improves the electrical and optical properties of the grown materials. These advantages put forward MBE as a favorable growth method for III-nitride growth.

Growth of nitride based materials using MBE can be achieved through two different processes, namely, plasma assisted molecular beam epitaxy (PA-MBE) and ammonia molecular beam epitaxy (ammonia-MBE). While plasma-cracked nitrogen source is used

as the nitrogen precursor in the PA-MBE, ammonia is used as the nitrogen precursor in ammonia-MBE growth process. The detailed growth mechanisms of each of these techniques are explained in sections 2.1.2.1 and 2.1.2.2.

2.1.2.1 Plasma assisted molecular beam epitaxy

In the PA-MBE growth process, N_2 gas is used as a nitrogen source. However, owing to the high bonding energy (9.2 eV) of N_2 [54], it cannot be dissociated by thermal means. Hence, N_2 gas is passed through a plasma source, which dissociates the gas source into different kinds of atomic and molecular species such as atomic nitrogen (N), excited state of molecules (N_2^*), ionized atoms (N^+) and molecules (N_2^+) [55, 56]. Among these different kind of molecules, the excited N_2^* molecule was found to have longer life times and hence contributed majorly in the growth process [57]. Typical growth mechanism in PA-MBE growth process involves the exothermic dissociation of N_2^* molecule on the growth surface and chemisorption into the lattice by reaction with group III elements. However, along with the excited N_2^* molecules, increase in the nitrogen atoms (N) was also found to have increased the nitride growth rates [58, 59], establishing the importance of nitrogen atoms in the growth process. The composition of different nitrogen species in the N_2 plasma can be controlled by the combination of RF power, nitrogen flow and PBN aperture of plasma cell with different hole diameter and hole number [60-63].

Growth on Si(111) using PA-MBE is usually initiated with an AlN nucleation layer. However, similar to the MOCVD growth, optimization of AlN nucleation layer is extremely important to achieve good quality epitaxial GaN in PA-MBE growth process. Al-Si inter diffusion can occur during the growth of AlN that may result in etch pit formation on Si surface via Al-Si alloy eutectic transformation [64]. These etch pits can also act as nucleation centers for GaN crystallites with different facets [65]. Further, diffusion of Al in Si can cause p-type conduction layer resulting in parallel conduction in the heterostructures. Moreover, Si diffusion in GaN epitaxial layer leads to the reduction of its buffer resistance [66, 67]. In order to counter these problems, several groups have followed different growth conditions for AlN epitaxial growth on Si substrate. However, most of them have adopted N-rich nucleation and growth conditions for AlN growth [68, 69] on Si substrate.

The underlying principle in adopting the N-rich growth conditions is to prevent the diffusion of Si from the substrate into GaN epilayers. However, Radhakrishnan et al. [70] have adopted a two layer growth technique. In their growth process, during the

nucleation step, formation of SiN and Al-Si eutectic alloy formation were avoided by exposing the Si surface to nitrogen plasma as well as Al flux simultaneously by maintaining the growth temperature below 600 °C. A few nanometer thick AlN nucleation layer was grown, followed by the growth of 120 nm thick HT-AlN, grown at 850 °C.

Different growth regimes of GaN using PA-MBE was studied by Heying et al. [71] and identified three major growth windows, namely, Ga-rich, intermediate and, N-rich growth regimes. The surface morphology of GaN in N-rich growth regime was found to be rough while the layers showed a lot of surface pits in the intermediate growth regime. However, layers grown in Ga-rich growth conditions were found to be atomically flat with lower RMS roughness values. Thus, the GaN layer growth using PA-MBE in Ga-rich growth conditions is found to be advantageous, which is in complete contrast with the N-rich growth regime of MOCVD. In PA-MBE growth of GaN in Ga-rich growth conditions, surplus Ga on the growth surface accumulates in the form of Ga droplets, which can be removed by dipping the grown wafer in HCl solution. Investigation of GaN growth rate as function of growth temperature was conducted on the GaN epilayers grown using different nitrogen species and with different polarities [72]. For the Ga polar GaN epilayers, grown using metastable molecules, the growth rate was found to be constant in the temperature range of 650 to 750 °C, beyond which growth rate was observed to be decreased due to the desorption of GaN. Thus, the optimized growth window used for GaN growth using PA-MBE growth process is in the metal rich growth regime with growth temperatures ranging from 710 to 740 °C to produce atomically smooth surfaces.

In order to improve the crystal quality of grown HEMT heterostructures by PA-MBE growth process, a two-step growth approach was adopted [73, 74]. In the first step, by pulsing the Ga shutter during the initial stages of the growth, coalescence of 3D islands was achieved, followed by the growth of nominal GaN in the 2nd step in the metal rich growth conditions. This process results in improved crystal quality and smoother surface morphologies. However, final metal rich growth conditions result in Ga accumulation and droplet formation. To desorb the excess gallium, growth with growth interruption has been implemented by various groups [68-70, 75]. During the growth interruption, accumulated 'Ga' gets evaporated and provides with smooth GaN surface for the subsequent growth. AlGaIn/GaN HEMT heterostructures with good electrical characteristics on Si(111) have been demonstrated for the growths using lower temperature (<740 °C) and metal rich growth regime [68-70]. Other than these growth

regimes for the PA-MBE growth process, high temperature (765-810 °C) and N-rich growth window ($Ga/N < 0.8$) for the GaN growth was also identified [76]. In this growth window, AlGaIn/GaN HEMT heterostructures with good crystal quality and RMS roughness of less than 1 nm on sapphire and SiC templates were demonstrated.

2.1.2.2 Ammonia-molecular beam epitaxy

Growth of GaN using ammonia-MBE proceeds through pyrolysis technique, where the atomic nitrogen obtained from thermally cracked ammonia on the substrate surface reacts with Ga flux to form GaN. The formation of GaN through the reaction of gallium and ammonia is represented by the Eq. 2.1.

As can be seen from the equation, increased ammonia flow or increased growth temperature enhances the cracking of ammonia and consequently the growth rate of GaN, provided sufficient Ga flux is supplied. In order to understand this behavior, ammonia cracking studies were performed as a function of growth temperature and ammonia flow in the presence of Ga flux [77, 78]. Ammonia cracking was found to be negligibly low below 350 °C. However, significant cracking was observed beyond 500 °C of the substrate temperature. Ammonia cracking increased linearly with the increase of growth temperature from 500 °C to 650 °C and became almost saturated at around 750°C. The maximum cracking efficiency of ammonia is 4% at 700 °C. The cracking efficiency was found to have increased in the presence of Ga flux, which was attributed to the incorporation of cracked and available N in the GaN lattice. Cracking experiments were also performed on GaN and AlN surfaces and the cracking was found to be higher on these surfaces compared to sapphire [79]. From these experiments, it can be seen that the deposition of GaN below 500 °C leads to very low growth rates owing to the insufficient amount of nitrogen. Hence, the optimized growth region for obtaining stable growth conditions is the saturated part of the ammonia cracking curve, which is beyond 750 °C, to provide nitrogen rich growth condition for GaN growth.

In the ammonia-MBE growth process, nitride growth on Si surface suffers from the unintentional nitridation due to the residual ammonia in the growth chamber [80]. In order to overcome this, several groups have adopted two completely different approaches and they are intentional nitridation of the growth surface [81, 82] and pre-depositing the surface with Al metal [80]. A systematic study was conducted to investigate the effect these two different nucleation techniques [83]. In the case of

intentional nitridation of Si surface using ammonia exposure faster nucleation of AlN and subsequent faster transformation to 2D growth mode was observed. On the other hand, the initial Al pre-deposited surface resulted in slower nucleation of AlN followed by 3D growth mode. Faster transformation of RHEED pattern from 3D to 2D growth mode in the AlN growth resulted in dislocation looping and good crystal quality in the subsequent GaN [80]. Moreover, partial nitridation of silicon surface is unavoidable in the ammonia-MBE growth process [80, 84]. These results indicate that the growth initiation with controlled intentional nitridation is advantageous for nitride epitaxy using ammonia-MBE growth process.

Growth kinetics of GaN grown using ammonia-MBE were studied to understand the variation of growth rate with ammonia flow by keeping Ga flux and temperature constant [77, 85]. A trend of increasing growth rate with the increasing ammonia flow is observed up to a certain flow rate after which the growth rate almost saturates. At low ammonia flow rates, the availability of atomic nitrogen is less, which allows more unreacted Ga to remain on the growth surface that subsequently gets desorbed producing lower growth rates. However, another argument suggests that the 'Ga' accumulation in the Ga-rich growth conditions reduces the growth rate by 'Ga' blocking the chemisorption sites of atomic nitrogen [86]. With the increase of ammonia flow, sufficient atomic nitrogen atoms become available for the formation of GaN, which reduces desorption of Ga and produces higher growth rates. The growth rate increases with the increase of ammonia flow and the corresponding growth regime is called ammonia-limited or Ga-rich growth regime. When the flow was increased beyond the ammonia-limited growth regime, the growth rate saturates. Non availability of sufficient Ga atoms to grow GaN has been attributed for the observed saturation of growth rate, and hence the growth regime is called 'Ga' limited or ammonia-rich growth regime. The separation between these two growth regimes occurs at $V/III \sim 1$ [77]. A clear RHEED intensity oscillations were observed for the GaN growth in the Ga limited growth regime at lower temperatures (<650 °C), indicating two dimensional island growth. However, beyond 850 °C, no RHEED oscillations were observed, suggesting step flow growth mode. Similarly, no RHEED oscillations were observed for the growth in the ammonia-limited growth regime, where epilayers showed much smoother surfaces [86]. Material characteristics were found to have improved for the GaN epilayers grown in N-rich growth conditions [87]. Moreover, unintentional impurity incorporation in nitride epitaxial layers reduced drastically for the epitaxial layers grown in the N-rich growth

conditions [88]. These studies indicate that the N-rich growth conditions are advantageous for the nitride epitaxial growth by ammonia- MBE.

Effect of growth temperature on the properties of GaN was studied and three regions were identified for GaN layer growth [78, 89]. Growths performed below 740 °C, called region 1, has resulted in 3D growth mode and poor crystal quality. Similarly, 3D growth mode was observed for the growths performed beyond ~810 °C (region 3). However, excellent crystal quality was achieved for the films grown in this region. GaN growth in region 2 has resulted in two dimensional growth and also resulted in good crystal quality. While three dimensional growth in the regime 3 has been attributed to GaN dissociation at higher growth temperatures, two dimensional growth mode in regime 2 has been attributed to screw type dislocation mediated step flow growth mechanism [90, 91]. Nucleation of GaN in the regime 2 originates at a screw type dislocation, which proceeds through the step flow growth leading to mound type surface morphology [90]. The step size depends upon the diffusion of Ga on the growth surface, which is a function of residual compression in GaN. Reduction in the residual compression with increased thickness leads to the kinetic roughening of the grown mounds. However, studies indicate that the roughening of mound decreases with the increase of ammonia flow at a particular growth temperature [92]. These results also suggest that the higher ammonia pressure might not only improve the quality of GaN layer but also its surface roughness.

AlGaIn/GaN HEMT heterostructures, grown in the growth regime 3 with lower ammonia flow rates exhibit rough surface morphology with lower buffer resistance but good crystal quality [89, 93]. However, AlGaIn/GaN HEMT heterostructures, grown in the growth regime 2 with high ammonia flow rates resulted in high resistive buffer layers with smoother surface morphologies [82, 92]. Carbon doping was found to be necessary for the layers grown in the growth regime 3 for improving the buffer resistances [94]. Thus, GaN growth in regime 2 with high ammonia overpressure might be advantageous due to its smoother surface morphologies and sharper interfaces for the HEMT heterostructure growth using ammonia-MBE growth process.

2.2 Stress management for GaN epitaxy on Si(111)

One of the major challenges in the growth of GaN on Si(111) using both MOCVD and MBE techniques is to overcome the high tensile strain generation, which cracks GaN epilayers. A large thermal mismatch of 52% and a lattice mismatch of 17% between GaN and Si has been attributed to the high tensile strain generation and cracking. Cracking

occurs along $[1\bar{1}00]$ and its equivalent directions [95] when the tensile stress in the GaN layer exceeds 800 MPa. Tensile stress develops in GaN epilayers grown on Si substrate during cool down from the growth temperature. In addition to that tensile stress also develops during growth due to the grain coalescence mechanism [96]. However, Krost et al. [97] have proposed that the origin of tensile stress in GaN during the growth on Si cannot be attributed solely to the grain coalescence mechanism. Moreover, they have suggested that the tensile stress generation during growth can be attributed to the grain growth [98] and its consequent free volume elimination at the grain boundaries [99]. Thus, it is important to induce compressive stress in GaN to compensate the tensile stress generated during the growth and cool down to achieve crack free thick GaN layer on Si substrate. In order to achieve this, various groups have been using different stress mitigating layers such as low temperature-AlN (LT-AlN) interlayers [100, 101], AlGaIn SML [102, 103], super lattice SML [104-106] and AlN/GaN SMLs [23, 107] for GaN growth on Si(111).

Amano et al. [100, 101] have first time demonstrated the growth of crack free AlGaIn on GaN using LT-AlN interlayers. Later, LT-AlN intermediate layers were utilized for the stress compensation in GaN growth on Si substrate to achieve crack free GaN epilayers [108, 109]. LT-AlN acts as a de-coupler of stress between its top and bottom GaN layers and produces crack free GaN layers [45]. Superlattice stress mitigating layers (SL-SML) were also attempted in obtaining crack free GaN epitaxial growth on Si(111) substrate. SL-SML such as AlGaIn/GaN SL [104] and AlN/GaN SL [105, 106] have been found to reduce the tensile strain in GaN growth, and crack free 2 to 2.5 μm thick GaN buffer layers have been reported [110].

Other SMLs reported include AlGaIn- based layers for the growth of GaN on Si substrate. Crack free 1 μm thick GaN [111, 112] was demonstrated on Si substrate using AlGaIn-SML, which showed improved performance of light emitting diodes (LEDs) [113]. Instead of a single AlGaIn SML, by using a step graded AlGaIn SML (grading all the way from AlN nucleation layer to GaN), 2 μm thick crack free GaN on Si substrate was successfully achieved [114]. Crack free AlGaIn/GaN HEMT heterostructures using MOCVD were also successfully demonstrated on Si-substrate using a single and step graded AlGaIn SMLs [103, 115, 116]. AlGaIn SML was also employed for the stress mitigation in GaN growth on Si substrate by MBE growth. Effect of thicknesses of AlGaIn/AlN-SMLs on the crack density of 1 μm thick GaN buffer, grown on 2-inch Si substrate using ammonia-MBE was reported [95]. The optimized thickness of AlGaIn and AlN layers in the SML was found to be 120 and 250 nm, respectively with Al composition

in the range of 0.3 to 0.6 for the growth of crack free 1 μm thick GaN on Si substrate. Moreover, Agrawal et al. [117] have also studied the effect of AlGaIn growth temperature and AlN layer thickness on the strain states of GaN on 100-mm Si(111), grown using ammonia-MBE. The optimized growth temperature of AlGaIn SML and AlN thickness to achieve comparatively lower tensile strain and two dimensional growth mode of GaN are 820 $^{\circ}\text{C}$ and 200 nm, respectively. However, the tensile strain in GaN layer was still too high to implement the AlGaIn SML for the growth of GaN based HEMT heterostructures using ammonia-MBE.

GaN growth by AlN/GaN SMLs has been studied intensely for the growth using ammonia-MBE on Si substrate. Semond et al. [81, 82] have utilized a thick (250/250 nm) AlN/GaN SMLs on the top of the AlN nucleation layer to grow crack free GaN with a thickness up to 3 μm on 2-inch Si substrate. In-situ measurements performed on such a layer scheme revealed that the first GaN layer only compensated the high tensile strain generated due to AlN growth on Si. However, the 2nd GaN layer produced higher negative curvatures compared to the first GaN and compensated the thermal stress generated during cool down to produce crack free heterostructures [118]. It was also identified that the growth of 2nd GaN at 820 $^{\circ}\text{C}$ instead of 800 $^{\circ}\text{C}$ resulted in quick saturation of negative curvature during the growth and lead to high positive curvature during cool down and eventual cracking [84]. Faster relaxation of 2nd GaN through dislocation looping at higher growth temperatures was attributed to the saturation of negative curvature. Thus, by maintaining the growth temperature of GaN at 800 $^{\circ}\text{C}$, growth of thick crack free GaN epilayers on 2- inch diameter silicon substrates was achieved using AlN/GaN SMLs for ammonia-MBE growth process.

Most of the stress mitigating layer structures that were discussed so far have been utilized for the GaN growth using MOCVD and ammonia-MBE. However, using PA-MBE growth process, Radhakrishnan et al. [70, 119] have successfully demonstrated for the first time crack free AlGaIn/GaN HEMT heterostructures without using any stress mitigating layers on 100-mm Si(111) substrate. They have grown GaN buffer thickness up to 1500 nm in their heterostructures. Similarly, several groups have obtained AlGaIn/GaN HEMT heterostructures on Si substrate without any stress mitigating layers [68, 69]. Recently, GaN buffer thickness up to 4200 nm was also reported by using PA-MBE growth process on 100-mm Si substrate [69]. In this study, growth of GaN involved two step growth process where the first step was grown in the slightly higher than stoichiometric growth conditions and the second step was grown in Ga droplet regime. They have further suggested that the GaN growth during the first step results

compressive stress that may compensate the tensile strain during cool down. Moreover, relatively lower growth temperatures used in PA-MBE also induce lesser thermal strain ($\sim 0.2\%$) compared to ammonia-MBE ($\sim 0.25\%$) and MOCVD ($\sim 0.3\%$). Metal rich growth conditions in PA-MBE growth process induce sufficient compression in GaN layer and compensate relatively lower thermal strain generated during cool down to produce crack free thick GaN HEMT heterostructures on Si substrate.

2.3 GaN buffer resistivity control

Realization of high resistive GaN buffer is extremely important for the proper operation of HEMT devices. Lower resistive GaN buffer leads to high leakage currents, poor pinch off characteristics with degradation of device RF performances at high frequencies. Impurities and defects that are responsible for high conduction in GaN layers need to be well studied and growth conditions have to be optimized to produce high resistive GaN buffers for HEMT applications. Oxygen, silicon [120], intrinsic defects such as nitrogen vacancies [121] and gallium interstitials [122] are in general considered as the shallow impurities in GaN. It may be noted that wide range of values has been reported for the binding energy of various impurities in the literature. Thus, the identification of an individual impurity using various measurement techniques is important to reduce its density or compensate.

Nitrogen related vacancy has been attributed as one of the major factors for the unintentional n-type nature of GaN layer [121]. Deep level transient spectroscopy (DLTS) measurements performed on HVPE grown GaN layers showed a defect level at $E_c - 0.25$ eV and was attributed to the nitrogen vacancy related complex [123]. Moreover, DLTS measurements performed on MOCVD, PA-MBE and ammonia-MBE grown GaN layers have also shown the presence of similar defect level [124]. The concentration of nitrogen vacancy related complex is lower in MOCVD grown GaN. However, it is the highest in ammonia-MBE grown GaN with low V/III ratio. The defect complex concentration for the GaN grown with higher V/III ratio in ammonia-MBE was found to be lower compared to GaN grown by PA-MBE. Moreover, increased V/III ratio in ammonia-MBE GaN has resulted in decreased concentration of defect complex level at $E_c - 0.25$ eV by almost six fold. This behavior signifies that the trap is associated with nitrogen vacancy related complex. Thus, growth using N-rich conditions is advantageous in reducing unintentional nitrogen vacancy formation in GaN. Nevertheless, energy of formation of native defects is much higher in n-type GaN

compared to the extrinsic defects and hence the majority of impurity states in GaN can be attributed to the extrinsic donor states such as silicon and oxygen [125].

Hall measurements performed by Götz et al. have attributed the binding energy of 17 meV to Si related donor level [126]. In contrast, magneto-optical studies indicated the binding energy of Si as 29 meV [127], whereas PL measurements have revealed a binding energy of 34.5 meV to Si [128]. Thus, a wide range of binding energies (17 to 34.5 meV) were reported by different characterization techniques all attributing to Si related donor level. Unlike the other techniques, high resolution PL measurement has revealed a split in the donor bound exciton levels and the split has been attributed to the presence of two different shallow impurities [129]. Similarly, Meyer et al. [125] have investigated low temperature (7K) PL measurements with high resolution spectroscopy and obtained a doublet of donor bound exciton levels with binding energies of 30.4 and 34.5 meV. The binding energy of 30.4 meV was attributed to Si related donor level [130]. Similarly, Frietas et al. [131] have observed two donor bound exciton peaks at 3.4714 and 3.4723 eV by PL measurements and attributed them to oxygen and Si related impurity states, respectively. The assignment of impurity state was further confirmed by the PL measurements on Si doped MOCVD grown GaN films and oxygen containing MBE grown GaN layers [132]. Thus, high resolution-low temperature measurement is useful to identify both silicon and oxygen related impurity states in GaN with their binding energy levels at 30.4 and 34.5 meV, respectively. Thus, with these relatively lower binding energies, both oxygen and Si act as shallow impurities in GaN, resulting in significant conduction.

In addition to point defects and impurities, pure screw and mixed dislocations have also been identified as the leakage paths in GaN using conductive-AFM measurements [34]. Metastable trap states, both acceptor and donor-like, present at the screw type of dislocation were found to be responsible for high conduction. Moreover, it was identified that the annealing of ohmic metallic contact on the surface can cause the diffusion of metal atoms from the contact into the dislocation cores and also act as direct conduction paths [133]. Thus, dislocations, in particular, screw type dislocations were found to behave as a source of conduction in GaN layers. Reduction in the reverse leakage current by three orders of magnitude for the GaN layer without dislocations compared to the one with dislocations confirms the effect of dislocations on the conduction properties of GaN [134].

In addition to the vertical conduction through dislocation, there is also a possibility of parallel conduction in GaN layers. Studies on sequential etching of GaN on sapphire substrate and Hall measurements after etch step revealed higher carrier concentration and near metallic conduction at the GaN-sapphire interface [135, 136]. The high carrier concentration and the corresponding parallel conduction was attributed to the diffusion of oxygen from the substrate through dislocations around the interface and its segregation at the interface. Similarly, a gradient of electron concentration in free standing GaN from N-polar surface to Ga-polar surface was observed through micro Raman measurements [137]. In this case also, gradient of oxygen was found to be the reason for carrier concentration gradient in the layers. Parallel conduction was also observed at GaN/AlN interface for the PA-MBE grown GaN based HEMT heterostructures on SiC substrate [67]. However, based on SIMS measurements, the observed parallel conduction was attributed to the diffusion of Si from the SiC substrate and its segregation at the interface. Thus, as observed from these studies, a channel of highly doped GaN with impurities such as oxygen and silicon can cause parallel conduction in GaN based HEMT heterostructures. Moreover, native defect induced parallel conduction with localized high dislocations is also possible in GaN [135, 136].

Some or all of these different conduction mechanisms such as native defect, unintentional impurities, and the dislocation mediated parallel and vertical conduction can be the reason for conduction in GaN buffer layer of HEMT heterostructures. However, it is extremely important to identify, reduce or compensate them for achieving high resistive GaN buffer layers. One of the ways to reduce the buffer leakage in GaN is the compensation of impurity states and leakage paths with acceptor states. Several acceptor impurities such as Be [138], Zn [139], Fe [140] and carbon [42] have been investigated. Among them, doping using 'Fe' and 'C' have been widely studied. Heikman et al. [140] have used Fe doping in GaN layers grown by MOCVD and observed semi-insulating nature of GaN layers. However, 'Fe' memory effect and uncontrolled diffusion profiles were observed, leading to lesser control over the doping profiles in heterostructures. Moreover, Fe-doping resulted in the precipitation of Fe related phases in the PA-MBE growth process and was found to have limited success [141]. These disadvantages of Fe limit its usage for the doping of GaN to produce high resistive buffer. Carbon as the compensating acceptor is more successful for different growth techniques including PA-MBE [42, 75], ammonia-MBE [142] and MOCVD [143].

In carbon doping of GaN, if carbon occupies the nitrogen site (C_N), it creates an acceptor state, which either helps to capture free electron or to compensate them. Theoretical

calculations by Wright et al. showed that the formation of acceptor state C_N is more favorable in Ga-rich growth conditions whereas, donor state C_{Ga} was found to be favorable in N-rich growth conditions when Fermi level is near to the valence band [144]. Moreover, self-compensation effect of acceptor state is also possible with the formation of donor state during carbon doping. In particular, under Ga-rich growth conditions interstitial carbon (C_i) acts as a compensating donor state whereas it is C_{Ga} under N-rich growth conditions. Carbon incorporation in nitrogen site was found to have resulted in less lattice strain, whereas its incorporation in Ga (C_{Ga}) and interstitial sites (C_i) lead to high strain in the lattice. It was also suggested that the yellow luminescence observed in PL measurements of GaN sample was due to carbon C_N - C_i transitions. Seager et al. [143] have performed theoretical and experimental studies and have suggested that the effective p-type doping of carbon in GaN occurs primarily under Ga-rich growth conditions. However, recent theoretical studies using hybrid functional theory [145] showed that the C_N acts rather as a deep acceptor in GaN than shallow acceptor as proposed by earlier theoretical calculations [144]. They have further suggested that the yellow luminescence is due to the transition between C_N^0 and C_N^- states. The theoretical calculations using hybrid functional theory are in accordance with the experimental results of Ogino et al. [146], which also showed carbon as a deep acceptor with an energy state 0.86 eV above the valence band.

Metal-organic precursors used in MOCVD growth may act as un-intentional carbon dopants produce high resistive GaN epilayers [143]. However, for the MBE growth process, different carbon sources such as CCl_4 [147, 148], methane cracked in plasma [142], electron beam evaporated graphite and CBr_4 [42] have been studied. Among the different sources, CCl_4 and electron beam evaporated plasma were found to have compensated the n-type doping. However, the former was found to etch the GaN layer during the growth process, whereas the latter was found to be uncontrolled. CBr_4 source [42] was successfully used to dope GaN, grown using PA-MBE growth process. CBr_4 gas was introduced into the growth chamber and was cracked at the high temperature substrate surface. A linear increase in the carbon concentration with the increase of CBr_4 pressure was observed. However, carbon incorporation was found to have reduced with the increase in the growth temperature. Good control in carbon incorporation levels and no degradation in GaN crystal quality was observed using CBr_4 doping source. In accordance with the theoretical studies, the carbon incorporation was observed to be higher in Ga-rich growth conditions and lower in N-rich growth conditions. Good quality

and higher resistive GaN buffer was demonstrated for CBr₄ doped AlGa_{0.3}N/GaN HEMT heterostructures on SiC [75, 149, 150] and Si(111) [69] substrates.

Carbon doping of GaN using ammonia-MBE growth process was achieved using cracked methane by saddle field ion gun [151]. PL measurements performed on corresponding carbon doped GaN showed complete quenching of the band edge emission [94]. Similar observation was observed for CBr₄ doped GaN in PA-MBE process [42]. This phenomenon was attributed to the trapping of excitons in carbon doped GaN buffer. In addition, carbon doped samples also showed an increase in the yellow luminescence for both PA-MBE and ammonia-MBE grown GaN layers [42, 94]. These two observations from PL measurements have been attributed as the signature of carbon incorporation in GaN lattice. Moreover, recent studies on carbon doped GaN using methane cracked source showed slight degradation in the surface morphology of ammonia-MBE grown GaN layers [152]. They have observed pit formation on the surface of the carbon doped samples with pits. However, semi-insulating GaN layers were achieved with resistivities in the order of 10⁶ to 10⁸ ohm-cm and AlGa_{0.3}N/GaN HEMT heterostructures were demonstrated on sapphire [142], SiC [89] and Si(111) [153] substrates. Thus, carbon doping can be employed to improve the buffer resistance of GaN for GaN based HEMT heterostructures.

2.4 GaN based HEMT heterostructures

Crack free, thick and, high resistive GaN buffer is required for GaN based HEMT heterostructure. Khan et al.[154, 155] have first demonstrated the 2DEG formation at the AlGa_{0.3}N/GaN interface with a room temperature electron mobility of 834 cm²/V.s and a carrier concentration 1×10¹¹ cm⁻² on sapphire substrates by MOCVD growth technique. Even though initial demonstration of GaN based HEMT heterostructure was done on the sapphire substrate, with all its commercial applications, it has been increasingly replaced with Si and SiC substrates as they offer improved device performances and better thermal management capabilities. To enhance the properties of 2DEG at the AlGa_{0.3}N/GaN interface, new concepts were proposed such as AlN spacer growth between AlGa_{0.3}N and GaN layers [156]. Using AlN spacer layer, a high 2DEG mobility of 1540 cm²/V.s with a carrier density of 1.48 × 10¹³ cm⁻² was achieved on SiC substrate. The higher mobility obtained was attributed to the reduced alloy scattering due to the prevention of carrier penetration into the AlGa_{0.3}N layer. Further, a high mobility of 25000 cm²/V.s was achieved at 15 K with the 2DEG density of 1×10¹³ cm⁻² by using an AlN spacer on HEMT growth using AlN /Sapphire templates [157]. To improve

the confinement of 2DEG at the AlGaN/GaN heterojunction interface, a double heterojunction structure with AlGaN/GaN/AlGaN layer sequence was proposed. It resulted in improving the carrier confinement of 2DEG in the quantum well and yielded higher mobility of 8100 cm²/V.s at 4.2 K, compared to 5700 cm²/V.s for usual AlGaN/GaN HEMT heterostructure [158]. Usage of thin In_{0.1}Ga_{0.9}N layer as a back barrier for AlGaN/GaN double hetero junction HEMT heterostructure was also studied to improve the confinement of 2DEG in the channel and achieved a high f_T of 153 GHz and f_{max} of 230 GHz [15].

With the improved characteristics of AlGaN/GaN HEMT heterostructures and owing to the advantages of GaN epitaxial growth on silicon substrate, several groups have demonstrated AlGaN/GaN HEMT heterostructures on Si(111) substrate using MOCVD growth technique. HEMT with high electron mobility exceeding 900 cm²/V.s was initially demonstrated by Schremer et al. [159]. AlGaN/GaN based HEMT devices with 25 GHz cut off frequency and near unity f_{max}/f_T ratio were also demonstrated as initial set of results on Si substrate [160]. From then, with the advanced stress mitigating techniques and improvement in the growth technology, AlGaN/GaN HEMT heterostructure growth using MOCVD has been improved enormously over the years [116, 161, 162]. However, owing to the advantages such as low temperature growth conditions, UHV background pressure, precise control over interfaces and the usage of in-situ monitoring techniques, GaN based HEMT heterostructures were grown on Si substrate using MBE growth technique and the details are discussed in the following section.

2.4.1 AlGaN/GaN based HEMT heterostructures on Si(111) using MBE

AlGaN/GaN based HEMT heterostructures were first demonstrated using ammonia-MBE growth process by Semond et al. on 2-inch Si(111) substrate [81, 82]. As discussed in the sections 2.3, using AlN/GaN SMLs, 3 µm thick GaN buffer was grown on Si substrate and achieved a high room temperature mobility of 1600 cm²/V.s. The mobility increased to 7500 cm²/V.s at 20 K. Typical RMS roughnesses achieved for GaN based HEMT heterostructures on Si by ammonia-MBE are in the range of 3 to 7 µm. The higher roughness observed was attributed to the growth mechanism involved in ammonia-MBE growth process. In this process, the initial stage of the growth follows diffusion driven screw type dislocation mediated step flow growth via BCF mechanism [91] that results in growth spirals and mounds [90]. As the thickness of the epilayer increases, relaxation

of GaN increases and causes kinetic roughening of the grown mounds and increasing of the RMS roughness.

Subsequently, AlGaIn/GaN HEMT heterostructure growth was also demonstrated on high resistive 2-inch Si(111) substrate using AlN/GaN SMLs scheme using ammonia-MBE [163]. A maximum mobility of $\sim 1850 \text{ cm}^2/\text{V.s}$ for a 2DEG carrier concentration of $4 \times 10^{12} \text{ cm}^{-2}$ was achieved with 25% Al and 30 nm thick AlGaIn barrier. An inverted bell shape behavior of mobility as a function of carrier concentration was also observed. Moreover, 2DEG concentration and mobility as a function of Al composition was investigated and observed that the carrier concentration increased linearly with Al%. However, the mobility was found to reduce for Al content of more than 25% [164]. The reduction in the peak value of the mobility for higher Al% was attributed to the increased mismatch and consequent degradation of the quality of the interface. Using ammonia-MBE growth process, advanced HEMTs such as AlGaIn/GaN/AlGaIn DH-HEMT heterostructure was demonstrated by the same group [165] on the Si substrate and observed an improved 2DEG carrier confinement at the AlGaIn/GaN interface. These results suggest the feasibility and control of the growth of AlGaIn/GaN based HEMT heterostructures on Si substrate using ammonia-MBE growth process.

As discussed in section 2.2, AlGaIn/GaN HEMT heterostructures with 1.5 μm thick GaN buffer using PA-MBE were demonstrated on 100-mm Si(111) with the room temperature mobility and carrier concentration of $1100 \text{ cm}^2/\text{V.s}$ and $9 \times 10^{12} \text{ cm}^{-2}$, respectively by Radhakrishnan et al.[70]. For these heterostructures, sub nanometer roughness values were achieved. Growth of GaN in gallium rich growth conditions such that a constant adlayer of metallic gallium always present on the growth surface without forming droplets [119] can be attributed to the observed lower RMS roughnesses. Hoke et al. [68] have also demonstrated AlGaIn/GaN HEMT heterostructures with 1.7 μm thick GaN buffer on 100-mm Si substrates using a thin AlN nucleation layer of 50 nm thickness, grown in N-rich growth conditions. Aidam et al. [69] have recently reported GaN HEMT heterostructures with GaN buffer thickness up to 4.3 μm on 100-mm Si substrate with 130 nm thick AlN, grown in N-rich growth conditions and at high growth temperature of 950 °C. Hall measurements performed on their samples showed mobility values in the range of 1230 - 1350 $\text{cm}^2/\text{V.s}$ with corresponding carrier concentration in the range of $6.5 - 7.0 \times 10^{12} \text{ cm}^{-2}$. Thus, AlGaIn/GaN HEMT heterostructures have been successfully demonstrated using both ammonia-MBE and PA-MBE growth processes on Si substrate.

2.4.2 Lattice matched barrier using MBE growth technique

As discussed in the Section 1.3.3 of Chapter 1, lattice matched barrier provides strain free condition at the barrier/buffer interface, which improves the reliability of GaN based HEMT heterostructures. Moreover, due to their high spontaneous polarization and shallow surface state position in the band gap, it is possible to achieve thinner barriers without much compromise on the concentration of 2DEG. This allows scaling of the HEMT devices, which simultaneously increases their frequency of operation by keeping relatively higher operating powers. Lattice matched barriers include ternary compounds such as InAlN and quaternary compounds such as AlInGaN as discussed in Sections 1.3.3.1 and 1.3.3.2, respectively. Wide difference in the binding energies of binary alloys, InN (1.98 eV), GaN (2.20 eV) and, AlN (2.80 eV) makes the compound formation of InN with other nitrides difficult. Thus, the growth of 'In' related compounds requires non-equilibrium growth conditions. This can be attained by PA-MBE process at lower growth temperatures to grow lattice matched barriers for GaN based HEMT heterostructures.

So far, lattice matched barriers such as InAlN and InAlGaN alloys for GaN based HEMT heterostructures on Si(111) have not been demonstrated by MBE growth technique. However, they have been demonstrated on sapphire [166], SiC [167] and MOCVD grown GaN templates [168]. Different growth regimes of InAlN growth using PA-MBE process were investigated and identified that the indium (In) incorporation is negligible beyond 550 °C [169]. Even though, 'In' incorporation occurs in the lattice below 550 °C, phase separation of grown material was also observed. To avoid the phase separation of InAlN into InN and AlN phases, growth of InAlN below 480 °C was suggested [166]. Moreover, recent report identified that higher growth rates (~ 350 nm/h) could lead to the phase separation of single layer into different layers along the growth direction, whereas no phase separation was observed with the reduced growth rates (~ 100 nm/h) [168]. Thus, from these studies, the optimized growth conditions for InAlN growth were identified as lower growth temperatures below 480 °C and low growth rates (~ 100 nm/h).

Higashiwaki et al. [166] have, for the first time, demonstrated the growth of InAlN barrier with 1 nm AlN spacer on GaN/sapphire at a growth temperature of 400 °C. The AlN spacer provides the protection to the growth surface and helps in screening the alloy scattering. They have obtained a near lattice matched ($\text{In}_{0.15}\text{Al}_{0.85}\text{N}$) barrier without any phase separation. A 30 nm thick ternary barrier resulted in a 2DEG carrier concentration of $2 \times 10^{13} \text{ cm}^{-2}$ with the mobility of $654 \text{ cm}^2/\text{V.s}$. Similarly,

$\text{In}_{0.12}\text{Al}_{0.88}\text{N}/\text{GaN}$ based HEMT heterostructures were grown on SiC substrates at a growth temperature of 445 °C, which resulted in a 2DEG carrier concentration of $1.96 \times 10^{13} \text{ cm}^{-2}$ [170]. However, a high sheet resistivity value of 980 Ω/sq was reported, signifying very low mobility of 2DEG achieved for these heterostructures.

Lattice matched $\text{In}_{0.17}\text{Al}_{0.83}\text{N}$ barrier was also successfully grown using PA-MBE growth process at 450 °C on MOCVD grown sapphire templates [171]. A high sheet carrier concentration of $2.68 \times 10^{13} \text{ cm}^{-2}$ with a room temperature mobility of 1080 $\text{cm}^2/\text{V.s}$ was achieved for a barrier thickness of 20 nm, which consequently resulted in a low sheet resistance value of 215 Ω/sq . The higher mobility obtained in their growth condition was attributed to the optimized AlN spacer thickness and avoiding the roughening of the growth surface by turning off the plasma during the ramp down. Similarly, lattice matched InAlN/GaN HEMT heterostructures were grown using PA-MBE on MOCVD grown GaN templates on sapphire substrates at 420 °C [30]. However, a high sheet resistance value of 3810 Ω/sq was obtained for these heterostructures. By using a triplet spacer layer [$\text{AlN}(0.6\text{nm})/\text{GaN}(1.1 \text{ nm})/\text{AlN}(0.7 \text{ nm})$] instead of 1nm AlN, the sheet resistivity value was reduced to 650 Ω/sq with the carrier concentration and mobility of $1.7 \times 10^{13} \text{ cm}^{-2}$ and 570 $\text{cm}^2/\text{V.s}$, respectively. The improved mobility value with the triplet spacer has been attributed to the increased separation of 2DEG from the spacer, which lead to the reduction of alloy scattering.

Overall, the lower mobilities obtained for InAlN/GaN HEMT heterostructures have been attributed to the interface roughness scattering, occurring due to the strain field variation [172] that is driven by the immiscibility gap between InN and AlN layers [173, 174]. Growth of quaternary alloys decreases the miscibility gap [173] and improves the mobility of GaN based HEMT heterostructures [174]. Lim et al. [29] have grown near lattice matched quaternary barrier of $\text{In}_{0.07}\text{Al}_{0.40}\text{Ga}_{0.53}\text{N}$ on GaN templates by PA-MBE growth process. They have used a triplet spacer layer between the quaternary barrier and GaN layer. Due to increased Ga percentage in the quaternary compound, the growth temperature was increased up to 580 °C. High mobility of 1460 $\text{cm}^2/\text{V.s}$ with a carrier concentration of $1.9 \times 10^{13} \text{ cm}^{-2}$ was reported for the HEMT heterostructures grown on MOCVD grown GaN templates on SiC substrates. These studies signify that the growth of quaternary lattice matched barrier by PA-MBE growth process is more advantageous in terms of relatively higher growth temperatures to obtain better crystal quality and improved mobilities for GaN based HEMT heterostructures.

3. Growth and characterization techniques

Nitride epitaxial growth on Si substrate is achieved using MBE growth technique. Following the growth, the grown epiwafers are characterized for their structural, optical, morphological and electrical characteristics using various characterization tools. This chapter discusses the growth and characterization tools that are used in this study.

3.1 MBE growth technique

AlGaIn/GaN HEMT heterostructures on 100-mm Si substrate and InAlN/GaN HEMT heterostructures investigated in this dissertation were grown using molecular beam epitaxy system (Veeco-GEN20MZ). The picture of the partial MBE system is illustrated in Fig. 3.1. This MBE system is capable of operating both as ammonia-MBE and PA-MBE. This unique capability of the system enables to grow nitrides in two mutually opposite growth conditions that is nitrogen-rich high temperature growth conditions in ammonia-MBE growth process and metal-rich low temperature growth conditions in PA-MBE growth process. A combination of these two growth processes has also been explored in order to exploit the advantages of both these techniques.

The MBE system consists of several modules, namely load lock and preparation, storage and growth modules as shown in Fig. 3.2. Each module has its own vacuum systems that are controlled independently to minimize the cross contamination from load lock to growth module as shown in the figure. The modules serve their own purpose in carrying out the growth from a bare wafer substrate to the end epitaxial heterostructures growth. The substrate on which epitaxial growth needs to be carried out is loaded into the load lock chamber, where it gets degassed at a lower temperature to outgas any water vapor or organic compounds present on the surface. The loaded wafer is subsequently transferred to the preparation chamber where it is degassed at a much higher temperature. The high temperature degassing makes the substrates epiready for the growth process, which are subsequently stored in the storage module. A batch of 8 wafers can be simultaneously stored in the storage module. The wafer from the storage module is transferred to the substrate holder of the growth module where the actual growth takes place.

Growth module is equipped with multiple vacuum systems such as a turbo molecular pump (TMP), cryo-pump and a cryo-jacket that surrounds the growth chamber. All these vacuum systems ensure a high level of vacuum (2 to 4×10^{-11} Torr) in the growth module prior to the growth, which ensures relatively lesser impurity incorporation in

the epitaxial layers. The growth module is equipped with multiple effusion “SUMO” cells for the source metals such aluminum, gallium and indium. A low temperature gas source for the ammonia gas is placed in one of the cell ports, which is used as the nitrogen source during the ammonia-MBE growth process. It is maintained at 200 °C to avoid the condensation of the ammonia. A Uni-bulb nitrogen plasma source that is assembled in another cell port acts as a nitrogen source in PA-MBE growth process. The Uni-bulb plasma source operates at a high frequency of 13.56 MHz with an output power up to 500 W.

Growth of nitrides in MBE proceed through a non-equilibrium growth process where the elemental fluxes from the sumo effusion cells react with the activated nitrogen species obtained either from cracking of ammonia or from plasma source on the substrate to form III-nitrides. The growth chamber is equipped with a substrate heater capable of heating up to 1200 °C, which heats the substrate in a radiative manner during the growth. Moreover, a temperature variation of only ± 3 °C across the 100-mm diameter of the wafer ensures a uniform growth.

Fig. 3.1. A picture of the partial MBE system.

Fig. 3.2 Functional diagram of the MBE system.

Carbon doping of HEMT heterostructures may be necessary to increase the GaN buffer resistance. Carbon is supplied into the growth chamber using a CBr₄ delivery system (Fig. 3.2) that is connected through a low temperature gas source. The solid CBr₄ source is sublimed and its vapor is delivered through a set of four fore-line valves with different orifice diameters and a leak valve into the growth chamber. The amount of CBr₄ entering into the chamber can be controlled by adjusting the pressure in a single or a combination of fore-line valves. The supplied CBr₄ thermally cracks on the grown wafer surface at the high growth temperature and incorporates carbon into the epilayers. Figure 3.3 shows the BEP as a function of fore-line pressure of CBr₄ for the four different orifices. As can be seen, all the orifices show similar trend of BEP with the fore-line pressure. The BEP of CBr₄ increases with the increase in the fore line pressure for all the orifices with different diameters. Thus, by selecting a combination of orifices, CBr₄ BEP ranging from 0.02 to 1.86×10^{-7} Torr can be achieved to dope layers.

Fig. 3.3 BEP as a function of fore-line pressures of CBr₄.

Additionally, the growth chamber is also equipped with the process monitoring and analytical instruments such as vacuum gauges, beam flux monitor (BFM), residual gas analyzer (RGA), BandiT temperature measurement instrument, RHEED and in-situ curvature measurement tools. Vacuum gauges measure the vacuum state of the growth module while the beam flux monitor is used to measure the BEPs of different fluxes from the effusion cells. RGA is used to monitor the residual gas pressures in the growth chamber before and during the growth process. The temperatures of the growth process are determined using the black body radiation obtained from the silicon substrate at high growth temperatures. The obtained black body data is analyzed by the BandiT measurement tool to display the growth temperature.

The schematic of a typical RHEED system is shown in the Fig. 3.4. The electron gun in the RHEED system operates at 15 kV and generates an electron beam that strikes the growth surface at a grazing angle. The diffracted electron beam from the growth surface forms diffraction patterns on the phosphor screen. A camera records the diffraction pattern and gives feedback to a computer. A sharp streaky RHEED pattern generally indicates 2D growth mode of grown of nitride epilayers while the spotty RHEED pattern corresponds to 3D growth mode. In addition to the growth mode, RHEED diffraction patterns also give details of surface reconstruction and polarity of the epilayer grown. A KSA-400 RHEED system is used to collect and analyze the RHEED diffraction patterns.

Fig. 3.4 The schematic representation of a typical RHEED set-up.

The nitride epitaxial growth on Si substrate results in the generation of huge stress in the layer during the growth and while cool down. An in-situ curvature measurement tool installed in the growth module provides details of the curvature of the substrate and stresses in the epitaxial layers at various stages of the growth. The schematic of the in-situ stress measurement tool is shown in Fig. 3.5 (a). It consists of an optical source box and a detector box. The optical source box comprises of a laser source with multiple lenses and etalons. The laser beam from its source is focused by a lens while the etalons splits a single beam into a parallel set of beams with equal distance as shown in the figure. Further, the parallel beams are aligned to focus at the center of the substrate. The beams that are incident on the substrate get reflected back at the same angle as that of the incident beam. A camera with a CCD arrangement in the detector box detects the reflected beams as an array of spots as shown in Fig. 3.5 (b). An average spacing (d_0) between two array points is a direct measure of the stress induced curvature of the substrate.

The change in the curvature due to stress generated in the epitaxial layers can be expressed as :

$$\kappa - \kappa_0 = \left(\frac{\Delta d}{d_0} \right) \frac{\cos \alpha}{2L} \quad \text{Eq. 3.1}$$

where $\kappa - \kappa_0$ is the increase in the curvature and Δd is the increment in the array spacing from its initial value of d_0 . The distance from the sample to the CCD in the camera is 'L' and the angle between beam array and the sample normal is α , as shown in figure.

Using the modified Stoney's equation (Eq. 3.2) [69] with a correction factor, 'c', the curvature ' $\kappa - \kappa_0$ ' can be expressed as [175]:

$$\kappa - \kappa_0 = 6c \frac{M_{\text{III-N}}}{M_{\text{Si}}} \frac{h_{\text{III-N}}}{h_{\text{Si}}^2} \epsilon \quad \text{Eq. 3.2}$$

where $M_{\text{III-N}}$ & M_{Si} and $h_{\text{III-N}}$ & h_{Si} are the biaxial module and the thicknesses of grown nitride epilayer and Si substrate, respectively. Here, 'ε' represents the developed strain and the corresponding to a stress of $\sigma_{\text{III-N}}$ in the epitaxial layer.

From the curvature, the stress-thickness product can be written as

$$\sigma_{\text{III-N}} \times h_{\text{III-N}} = (\kappa - \kappa_0) \left[6c \frac{1}{M_{\text{Si}}} \frac{1}{h_{\text{Si}}^2} \right]^{-1} \quad \text{Eq. 3.3}$$

Fig. 3.5 Schematic of the in-situ stress measurement tool (a) and the reflected beam from the substrate is seen as an array of spots captured by the CCD in the detector box (b).

The mean stress in the heterostructures can be obtained by taking the ratio of stress-thickness product and the thickness. To obtain the incremental stress in the epitaxial layers, the stress-thickness product and the thickness curve is fitted with various polynomials such that the least square regression for the fitting is as high as possible. The derivative of the fitted polynomial results in the incremental stress in a particular epitaxial nitride layer. KSA-MOS in-situ curvature tool has been used to measure the in-situ curvature during the nitride epitaxial growth on silicon substrate.

3.2 Characterization techniques

Various characterization techniques have been employed to study the structural, electrical, optical and surface morphology properties of the grown epilayers. The key

characterization tools used are described and most of the characterization tools that described are capable of handling full four inch wafers.

3.2.1 High resolution X-ray diffraction technique

High resolution X-ray diffraction (XRD) technique is a versatile characterization technique that has been used to study the structural properties such as crystal quality, thickness, composition and strain states of the grown nitride epilayers. In this technique, a well collimated beam of X-rays is projected on to the epilayer surface and the rays that are diffracted, following Bragg's diffraction condition ($n\lambda = 2d \sin \theta$) are detected using a detector with the highest angular resolution. Here, ' λ ' is the wavelength of the X-rays, ' θ ' is the diffraction angle (Bragg angle) and ' d ' is the atomic spacing for a particular set of planes. Figures 3.6 (a) and (b) show the schematic of a typical optical set up of a HR-XRD system and the diffraction phenomenon showing the diffraction of X-ray beams at an angle of ' θ ' for an atomic spacing of ' d '. Panalytical X'pert pro HR-XRD with a monochromic Cu-K α source has been used with a wavelength of 1.54056 Å to study the structural quality of the grown epilayers.

Mainly, three types of HR-XRD scans have been analyzed, namely, ω -2 θ scan, rocking curve scans and two axis reciprocal space mapping. In a typical ω -2 θ scan, both the source and the detector simultaneously move such that the step size for the detector, $\Delta 2\theta$ is twice that of the step size of the incident beam angle, $\Delta \omega$ ($\Delta 2\theta = 2 * \Delta \omega$). Typically ω -2 θ scans along GaN (0002), (0004) and (0006) planes have been considered for the composition and thickness estimation of barrier layer of HEMT heterostructures. The composition and thickness of barrier has been estimated from the barrier peak position with respect to GaN and its peak profile in the ω -2 θ scan, respectively.

Fig. 3.6 The schematic of a typical optical set-up of an XRD system (a) and the diffraction phenomenon showing the diffraction of X-rays at an angle of ' θ ' for an atomic spacing of ' d ' (b).

In addition to the composition measurements, XRD can also be used to study the crystal quality of grown epilayers by rocking curve scans. In this mode, the position of the detector (2θ) is fixed for a particular plane and the XRD scan is performed by rocking the incident beam angle (ω). For a perfect crystal lattice, the rocking curve scan results in very narrow curves. However, dislocations in the epilayers significantly broaden the rocking curves of the epitaxial layers. Thus, by measuring the FWHM of the broadened rocking curves obtained along symmetric and asymmetric planes, screw (N_S) and edge (N_E) type of dislocation density can be quantitatively estimated using the Eq. 3.4 and 3.5, respectively [176].

$$N_S = \frac{\alpha_C^2}{4.35b_C^2} \quad \text{Eq. 3.4}$$

$$N_E = \frac{\alpha_E^2}{4.35b_E^2} \quad \text{Eq. 3.5}$$

Here, 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.

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. By using the relative positions of different epilayers in the reciprocal maps, information such as strain states of different epilayers, coherent growth of barrier and the lattice matched composition of ternary and quaternary alloys have been verified.

3.2.2 Confocal micro-Raman spectroscopy

Raman spectroscopy involves the observation of inelastic scattering of light by vibrational modes of the material. Since the vibrational modes get affected by the residual stress, electron concentration and crystalline quality of the material, Raman spectroscopy can be employed to estimate all these parameters. A schematic of a typical confocal micro-Raman spectroscopy set up is shown in Fig. 3.7. As shown, a laser light of fixed wavelength is incident normally on the sample through a microscope objective. All the reflected, elastically scattered and inelastically scattered Raman signals that travel normal to the surface are collected by the microscope and passed through a pin hole in front of the detector. The collected signal that goes through the pin hole is directed on to a pair of notch filters in the detector set up. These filters allow the inelastically scattered light on to the detector and attenuate completely the reflected and elastically scattered light. A monochromatic grating in the detector resolves the signal and is sent to a charge coupled device (CCD) to obtain the Raman spectrum.

Fig. 3.7 The schematic of a typical confocal micro-Raman spectroscopy set up.

According to group theory, wurtzite structures (C_{6v}^{4v}) possess eight types of phonon modes and they are $2E_2$, $2A_1$, $2E_1$ and $2B_1$. Among these eight, two E_2 sets, one A_1 set and one E_1 set are Raman active, whereas B_1 modes are silent [177]. A list of standard phonon modes with their wave numbers for freestanding GaN and AlN are presented in Table 3.1. Among these listed phonon modes, only E_2 and $A_1(LO)$ phonon modes appear [178] for the nitride epitaxial layers grown in the c-axis direction.

Table 3.1 Raman active phonon modes for GaN and AlN

	Phonon wave numbers (cm^{-1})					
	$E_2(\text{low})$	$A_1(\text{TO})$	$E_1(\text{TO})$	$E_2(\text{high})$	$A_1(\text{LO})$	$E_1(\text{LO})$
GaN	144	532	559	567.5	734	741
AlN	248	610	670	657	890	912

Raman measurements start with the observation of Raman signal from a standard strain free Si(111) substrate, which gives the $O(\Gamma)$ phonon mode of Si (111) at 520.5 cm^{-1} . The spectrometer is aligned with respect to the standard silicon peak before measuring the GaN epitaxial layers on Si(111) substrate. Planar stress in the nitride epitaxial layers shifts the E_2 phonon mode in the Raman spectrum from its free standing position. Raman shift of E_2 phonon mode of GaN ($\Delta\omega$) with respect to its free standing position follows a linear relation with residual stress (σ_{xx}), which is given by $\Delta\omega = 4.3\sigma_{xx}$ [179].

Confocal resolution of Raman set up depends upon the size of the pin hole and microscope objective. For the current experimental set up, a microscope objective of 100X with a numerical aperture (NA) of 0.9 is used. Based on the optics theory of diffraction limit, the spatial and depth resolution of a microscope is given by Eq. 3.6 and 3.7.

$$\text{Spatial resolution} = \frac{0.61\lambda}{NA} \quad \text{Eq. 3.6}$$

$$\text{Depth resolution} = \frac{2.2n\lambda}{\pi(NA)^2} \quad \text{Eq. 3.7}$$

Here, λ and n are the wavelength of the laser light used (532 nm) and refractive index of air (1), respectively. Using Eq. 3.6 and 3.7, with the current microscope set up, a maximum spatial resolution of 0.35 μm and depth resolution of 0.46 μm can be achieved. However, in practice using a pin hole size of 50 μm , a depth resolution of only slightly less than 1 μm has been achieved.

In a multilayer heterostructure containing various GaN epilayers, it is important to identify individual epilayers and collect the Raman shift data from them. In order to achieve this, the true surface of GaN and the depth calibration into the heterostructure using micro-Raman set up is necessary. Figure 3.8 (a) shows the intensity of Raman GaN E_2 peak profile as a function of depth. The true surface of GaN that is air/GaN interface is identified as the position of the first peak in the derivative of the intensity peak profile vs depth curve as shown in Fig. 3.8 (b) [180]. Having identified the surface of GaN, the depth calibration was achieved by focusing the microscope at the buried crack position in the heterostructure. It is to note that the buried cracks in the heterostructure lies at the interface of 2nd AlN/1st GaN. The details of the heterostructure and the buried cracks are discussed in detail in Chapter 3. The difference in the microscope position at the surface and the buried cracks and the corresponding thickness observed using TEM gives a depth calibration factor of 1.7 in the heterostructures. This value is closer to the reported factor of 2 obtained using similar objective lens of 100X with NA of 0.9 [180]. Thus, using this depth calibration, strain values in different epitaxial GaN layers of the heterostructures were obtained.

Fig. 3.8 Intensity of Raman GaN E₂ peak profile as a function of depth (a) and the derivative of intensity peak profile vs depth curve (b) showing the true surface of GaN that is air/GaN interface.

3.2.3 Atomic force microscopy

Atomic force microscopy (AFM) is a standard technique that has been used to study the surface morphology of the grown heterostructures. AFM consists of a sharp tip (probe) at the end of a cantilever of about 100-200 μm long and 0.5-5 μm thick. 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. The schematic of a typical AFM set-up is shown in Fig. 3.9.

AFM can be operated typically in two modes, namely static and dynamic modes. In the static mode of operation (contact mode), AFM tip is in contact with the surface and the

static deflection observed is used to probe the surface morphology. In this mode of operation, there is a high probability of the tip to get attracted to the sample surface and damage.

Fig. 3.9 A schematic of a typical AFM set-up.

The dynamic mode of operation is more feasible for various kinds of surfaces. In this mode of operation, the tip oscillates with its resonance frequency. During the AFM scan, the resonant frequency of the tip changes due to the attractive force of the surface. This change in resonant frequency is balanced by adjusting the distance between the sample and tip by using the feedback mechanism and the piezo electric sensor under the sample holder. Measuring the average distance between tip and the sample surface allows the formation of the topographic image of the sample surface. The dynamic mode of operation can be further divided into non-contact mode and tapping mode. In non-contact mode, the amplitude of oscillation is in the range of 10 nm for measuring the short range forces. In ambient conditions, where some moisture develops on the surface, probing short range forces without the tip touching the surface is not possible. However, in tapping mode of operation, the oscillation amplitudes are in the range of 100-200 nm, where the tip goes very near to the surface, and hence its amplitude of oscillations changes. The distance between the tip and surface can be adjusted accordingly to bring back the amplitude. This mode of operation is intermediate between the contact and non-contact modes, and hence provides good sensitivity for surface morphology mapping without much damage to the sample surface and tip. The tapping mode of operation has been used for scanning the surface morphology of GaN based HEMT

heterostructures. Two AFM models were used for the purpose of investigation in this study, namely Shimadzu SPM-9500J2 and Cypher-MFP-3D.

3.2.4 Scanning electron microscopy

Scanning electron microscope (SEM) is a microscopic technique that uses electrons instead of photons (light). The advantage of using electrons is to decrease the wavelength of the probing wave and to improve the resolution. In SEM, high energy electrons are incident on to the sample surface, which leads to the emission of electrons with different energies from the sample. Broadly, the emitted electrons can be divided into three categories, namely secondary electrons, auger electrons and back scattered electrons. The secondary electrons are those that are loosely bonded to the sample and are emitted from the conduction band of the sample. Back scattered electrons are large angle elastically scattered electrons, which have the same energy as that of incident electrons, whereas auger electrons are electrons that have an energy intermediate between secondary electrons and back scattered electrons. Among these three categories, secondary electrons are the most useful for imaging the surface morphology. The basic principle of operation of SEM involves the observation of secondary electrons emitted from the sample surface during the raster scan of incident beam. Clear contrast can be observed for peak and valley points as the number of electrons reaching the detector are different per unit scan time. The detected signal is amplified and synchronized with the CRT tube raster scan to provide surface morphology pictures. JSM-7500F field emission-SEM (FE-SEM) has been mainly used to determine the thicknesses of different epilayers to estimate the growth rates of nitride epitaxial layers.

Energy dispersive spectroscopy (EDX): EDX is an add-on to the SEM machines. When high energy electrons interact with the sample surface, electrons are ejected from some of the core atomic levels leaving behind the vacant places. This allows the transfer of electrons from high energy level to this internal core level and ejects X-rays from the sample. The wavelength or energy of the emitted electrons is a characteristic of a particular atom or element, and hence analyzing different X-rays emitted from the sample gives the information on the elements present in the sample. Comparison of intensities of different X-rays give the stoichiometric ratio of elements present in the sample. In this report, EDX has been mainly used to cross verify the indium incorporation and estimation of composition in InAlN layers grown by MBE technique.

3.2.5 Transmission electron microscopy

Transmission electron microscope (TEM) also works on the advantage of low wavelength generation by high velocity electrons for the imaging purpose. However, unlike SEM, the TEM captures the image using the transmitted electrons that travel through a thin material. Thinner samples for the TEM analysis are obtained by using focused ion beam (FIB) technique, where a beam of Ga atoms is used to thin the material to obtain the cross sectional TEM samples. TEM images on as grown AlGaIn/GaN HEMT heterostructures were obtained in three different modes. They are bright field (BF), weak beam dark field (WBDF) and high angle annular dark field (HAADF) imaging modes.

BF mode: In this mode, the direct un-scattered beam of electrons through the specimen is only analysed by placing the aperture such that it blocks all other diffracted beams. Thus, in this imaging mode, diffraction contrast and mass-thickness play a major role. This mode is extensively used to study the different interfaces in the grown HEMT heterostructures and to measure the precise thickness of the barrier layer. Microstructure features like buried cracks, voids and dislocations in the HEMT heterostructures have also been analyzed from the images taken in the BF mode.

WBDF mode: In this mode, the aperture is placed such that the specific scattered beams that obey the Bragg's condition are only selected while the un-scattered light is blocked by the aperture. In this way, only those parts of a sample which satisfy certain Bragg's condition can only be seen in the image. Further, by selecting the large deviation parameter ' s ', most of the sample does not satisfy the Bragg's condition, while the local bending of the planes around the dislocations satisfy Bragg's condition and make dislocations to appear in the image. Only those dislocations with burger vector (b) that satisfy the Bragg condition with certain diffraction vector (g) are visible in the images. Table 3.2 lists various diffraction vectors and the corresponding visibility criteria of different dislocations. As listed in the table [181], the diffraction condition which satisfies $g \cdot b \neq 0$ are only visible, while the rest are invisible. Thus, diffraction vectors with $g = 0002$ and $g = 11\bar{2}0$ have been selected to analyze the screw and edge type of dislocation densities in this thesis.

Table 3.2. A list of various diffraction vectors and the corresponding visibility criteria of different dislocations.

$\mathbf{g} \backslash \mathbf{b}$	$\frac{1}{3}[\bar{2}\bar{1}\bar{1}0]$	$\frac{1}{3}[\bar{1}\bar{2}\bar{1}0]$	$\frac{1}{3}[\bar{1}\bar{1}\bar{2}0]$	$[0001]$
0002	0	0	0	2
0110	0	1	-1	0
1120	1	1	-2	0

HAADF mode: In HAADF mode, high-angled scattered electrons from the sample are detected by the annular dark field detector and form the image. The contrast of HAADF image is primarily sensitive to the average atomic number of the sample. So, the HAADF imaging mode can be used for elemental mapping. In this study, sharpness of the interfaces of different AlN/GaN epilayers in the AlGaIn/GaN HEMT heterostructure has been studied by HAADF mode. TEM study was done by using JEOL-2100F and Tecnai F20 TEM models operating at 200 V.

3.2.6 Secondary ion mass spectroscopy

Secondary ion mass spectroscopy (SIMS) works on the principle of analysis of secondary ions emitted from a sample when bombarded with a beam of primary heavy particles. Using this technique, composition, phase and dopant levels in a material can be quantitatively estimated. The ionization yield of the secondary ions of positive and negative charges can be controlled by the choice of the different primary ion beams. In order to increase the yield of positive secondary ions, a beam of ions with higher electron affinity can be used. Implantation of higher electron affinity ions capture the electrons from the elements and leaving them positively charged. Oxygen is the most common primary ion beam source to increase the yield of positive secondary ions. Similarly cesium is the most common primary ion source to increase the yield of negative secondary ions.

SIMS in general operates in static and dynamic modes. The static mode is more of a surface analysis technique, which uses a time of flight (TOF) detector. In this mode, a pulsed beam of primary ions bombard on the surface and release the secondary ions with various masses. The lighter ions reach the detector first followed by the heavier ions. Thus, based on the time of flight of different ions to the detector, a mass spectrum can be constructed. Thus, each pulse of primary ions gives a full mass spectrum of the

secondary ions. Sample destruction is very minimal in this technique and can be primarily used to obtain the surface composition of materials.

The dynamic SIMS technique is more of a bulk technique where the primary ion beam source sequentially etches the sample surface by sputtering. The fraction of sputtered beam that are ionized will be driven to the detector. Either a magnetic sector or a quadrupole mass filter is used as the detector in dynamic SIMS operation. As the name suggests, the former uses magnetic field to separate the secondary ions based on charge to mass ratio, while the later uses RF electric field. Thus, by measuring the depth of etched sample and corresponding concentration of elements, atomic concentration depth profiles can be constructed. Dynamic SIMS mode is extremely sensitive and capable of detecting compositions in the range of parts per million (PPM) to parts per billion (PPB).

The ionic yield of a particular element depends upon the element itself and the matrix it contained. Hence, the absolute quantification of concentration is not directly possible from the obtained secondary ion data. However, calibration of concentration can be achieved by using a standard sample with the same matrix and known concentrations. Thus, using dynamic SIMS mode with well quantified standard samples, elemental concentrations of oxygen, silicon, carbon have been obtained as a function of depth in the grown AlGaIn/GaN HEMT heterostructures. CAMECA-IMS 3f with magnetic sector has been used to study the samples using SIMS technique.

3.2.7 Hall Effect

3.2.7.1 Van der Pauw method

The Hall Effect is one of the most commonly used techniques for electrical characterization of semiconductor materials. For Hall measurements, samples with dimensions of about 1 cm × 1 cm are prepared. Indium contacts are placed at the four corners of the sample and annealed to make them ohmic. Figure 3.10 shows the schematic diagram of Van der Pauw set up to measure the electrical properties of GaN based HEMT heterostructures. Prior to the Hall measurements, the sheet resistance (R_s) of the samples is obtained using Van der Pauw formula (Eq. 3.8).

$$e^{-\frac{\pi R_{12,34}}{R_s}} + e^{-\frac{\pi R_{23,41}}{R_s}} = 1 \quad \text{Eq. 3.8}$$

Here, $R_{12,34}$ and $R_{23,41}$ are resistances measured along the edges of the sample.

Fig. 3.10 The schematic of Hall measurement set-up in Van der Pauw configuration.

During the Hall measurements, a constant magnetic field of B is applied perpendicular to the sample surface and an electrical current of ' I ' is injected across the two non-adjacent contacts as shown in the Fig. 3.10. The current ' I ' gives the carriers a drift velocity of v_d such that the carriers are subjected to a Lorentz force perpendicular to both the magnetic field and current directions. Due to this force, the carriers are forced to move towards either one of the remaining two contacts (1 or 3), which results in the generation of a potential difference between them. The generated potential is called the Hall voltage V_H . The Hall voltage, V_H can be expressed using the Eq. 3.9.

$$V_H = \frac{I \times B}{q \times n_s} \quad \text{Eq. 3.9}$$

Here, n_s is the sheet carrier concentration, which is 2DEG concentration in case of HEMTs. The mobility of 2DEG can be obtained using Eq. 3.10.

$$\mu_H = \frac{1}{n_s \times q \times R_s} \quad \text{Eq. 3.10}$$

In addition to the room temperature measurements, the Hall measurement technique has been used to measure the electrical characteristics of GaN based HEMT heterostructures as a function of temperature from 90 to 400 K. A Bio-rad HL 5500 system was used in this investigation.

3.2.7.2 Contact less Hall measurement system

A contact less Hall measurement system has been used to check the uniformity in the electrical characteristics of AlGaIn/GaN HEMT heterostructures across 100-mm diameter. The measurement system consists of an electromagnet coupled with a wave guide network to transmit and receive the microwaves. The measurement technique involves sending a microwave (TE_{10}) operating at 10 GHz on to the sample surface. The reflected wave with the same polarization (TE_{10}) as that of the incident wave is detected by the wave guide network and measured. The ratio of the forward and reflected powers has been used to determine the sheet resistivity of the samples under measurement.

In the presence of magnetic field during the Hall measurements, the incident TE_{10} mode generates an additional reflected mode of TE_{11} with 90° rotation, which can be attributed to the applied perpendicular magnetic field. The reflected power of the rotated TE_{11} mode is also measured by the wave guide network. Figure 3.11 shows the schematic diagram of contact less Hall measurement method showing incident and reflected waves on the sample surface. The figure also shows the waveguides of TE_{10} and 90° rotated TE_{11} modes.

A software module in the system converts the measured microwave powers into reflection coefficient to develop the components of conductivity sensor, σ_{xx} and σ_{xy} . Using this conductivity sensor, electrical characteristics such as carrier concentration, mobility and sheet resistivity are extracted. Moreover, by measuring the reflected power at various magnetic fields, multi carrier analysis can also be done [182]. A Leighton contact less electrical measurement mapping system is used in this study.

Fig. 3.11 The schematic diagram of non-destructive mobility measurement method.

3.2.8 Capacitance-voltage measurements

Semiconductor characterization using CV measurements is a well-established technique to study the doping profiles and impurity concentrations. A Schottky and an ohmic contact are required in general for the CV measurement. However, in order to perform non-destructive electrical characterization of as grown GaN based HEMT heterostructures, a mercury probe system is used. Mercury-Probe employs a unique dot-ring configuration on the semiconductor surface as shown in Fig. 3.12.

Fig. 3.12 Mercury probe dot-ring configuration on the semiconductor surface.

The dot works as a Schottky contact on the surface while the ring works as an ohmic contact. A DC voltage, V is applied to the Schottky contact, which will provide a space charge region of width W in the semiconductor. The applied DC voltage is overlapped with an AC voltage of small amplitude and high frequency. Due to the applied AC voltage, 'v' an increment in the charge (dQ_s) in the space charge region occurs, which is given by

$$dQ_s = -qN(w)Adw \quad \text{Eq. 3.11}$$

Here, A is the cross-sectional area of semiconductor, N(w) is the doping density at width w, dw is the slight increase in the space charge region (SCR) width due to the applied voltage.

Further, the differential capacitance is defined as

$$C = \frac{-dQ_s}{dV} \quad \text{Eq. 3.12}$$

Substituting Eq. 3.11 and Eq. 3.12 in the equation for a parallel plate capacitance,

$C = \frac{k\epsilon A}{w}$ and differentiating Eq. 3.12 with respect to the voltage leads to

$$N(w) = \frac{-C^3}{qAk\epsilon\left(\frac{dC}{dV}\right)} \quad \text{Eq. 3.13}$$

Thus, using Eq. 3.13 the doping concentration in a semiconductor at a depth of W can be determined by taking the slope of CV curve, $\frac{dC}{dV}$. Doping concentration vs depth profile has been used to determine the 2DEG position and the back ground carrier concentration in this study. Further, the concentration of 2DEG has also been obtained by integrating the CV profile from the zero bias to pinch off (V_o) position.

4. Stress management in AlGaN/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs

GaN based HEMT heterostructures were grown on 100-mm diameter Si(111) substrate using ammonia-MBE growth technique. Details of the growth of nitride epitaxy on Si substrate, investigation and optimization of stress mitigating layers and demonstration of AlGaN/GaN HEMT heterostructures are discussed in this chapter.

4.1 Growth of nitride epitaxy on Si(111) using ammonia-MBE

A typical growth process using ammonia as nitrogen precursor starts by loading the Si substrate into the load lock of the MBE system, where it gets degassed at 200 °C for 3 hours to remove the surface related contamination such as organic and water vapor. Following the initial degassing step, the Si substrate is transferred to the preparation chamber, where it undergoes a thermal cleaning process involving the degassing of the substrate at 800 °C for 60 minutes. Thermal cleaning process ensures to desorb the oxide and produce a clean Si(111) surface. Subsequently, the substrate is transferred to the growth chamber, where a (7×7) RHEED reconstruction pattern is observed as shown in Fig. 4.1 (a), indication of epi-ready Si(111) surface without the oxide layer.

Fig. 4.1 Thermally cleaned Si(111) surface showing (7×7) RHEED pattern (a). AlN nucleation on Si surface showing AlN and underlying Si (1×1) RHEED patterns along $(1\bar{1}0)$ of Si(111) (b).

GaN growth on Si requires an AlN nucleation layer to overcome the issues like melt back etching and the growth of poly crystalline layers as discussed in Section 1.5 of Chapter 1. Thus, it is extremely important to optimize the nucleation of AlN layer, which determines the properties and quality of subsequent epilayers. The growth of AlN

nucleation layer was initiated by ramping up the temperature of the thermally cleaned Si substrate to 660 °C and was exposed to ammonia flow of 2 SCCM corresponding to a BEP of 1.32×10^{-6} Torr for 30 seconds to intentionally nitridate the Si surface. Intentional nitridation ensures uniform coverage of Si growth surface with $\beta\text{-Si}_3\text{N}_4$ [183]. Aluminum (Al) predeposition on the nitradated surface and the subsequent annealing at 920 °C resulted in two (1×1) RHEED patterns as shown in Fig. 4.1(b), indicative of AlN nucleated layer and the underlying Si surface. The process of obtaining the AlN nucleation layer by converting $\beta\text{-Si}_3\text{N}_4$ into AlN is more favorable over nucleation using Al predeposition over Si surface [80]. The later process may result in a parasitic parallel conduction path in the Si substrate by Al diffusion and may also result in non-uniform nucleation due to unintentional nitridation of the Si surface.

AlN layer was subsequently grown on the nucleated surface, which was used as the buffer layer for GaN growth. However, optimization of AlN growth was necessary to ensure good crystal quality and smooth surface morphology and is discussed in detail in section 4.1.1. Moreover, unlike the case with PAMBE, where metal rich growth conditions are considered to be optimized for GaN growth, growth of GaN layers in ammonia-MBE are performed in N-rich growth conditions [184]. N-rich growth conditions of ammonia-MBE offer distinctive advantages such as wider growth window, reproducible Ga-polarity surface and possibly improved device uniformity. Grandjean et al. [184] have shown that the higher V/III ratios in ammonia-MBE improve the surface morphology and simultaneously reduce the incorporation of unintentional impurities resulting in resistive buffer layers for GaN based electronics. Further, Tang et al. [185] have grown GaN layers at various growth temperatures, ranging from 550 to 870 °C and found that the growth window for 2D growth mode is between 720 to 810 °C. Based on these studies, GaN growth is described in section 4.1.2.

4.1.1 AlN growth

AlN growth was optimized with respect to the growth rates and growth temperatures by keeping the ammonia flow at a constant value of 280 SCCM. Two sets of samples were grown. The first set of samples, set I comprised of samples A1, A2 and, A3 were grown at growth rates of 0.08, 0.10, 0.13 $\mu\text{m/h}$, respectively by keeping the growth temperature at 920 °C. The second set of samples, set II comprised of samples A4, A5 and, A6 were obtained by growing AlN layer at different growth temperatures of 890, 905 and, 920 °C, respectively by keeping the growth rate of AlN at 0.13 $\mu\text{m/h}$. Sample A6 is similar to that of sample A3 in the first set. The increase of the growth rate from sample A1 to A3 by increasing the Al flux has resulted in the improvement of RHEED pattern as shown in

Fig. 4.2. This indicates faster transition from three dimensional (3D) nucleation to two dimensional (2D) growth mode with the increased growth rate. Moreover, higher growth rate has also resulted in the improvement of AlN crystal quality and the surface RMS roughnesses as shown in the Fig.4.3 (a). Samples A4 to A6 that were grown as a function of growth temperature has resulted in almost similar dislocation density as shown in Fig. 4.3 (b), moreover, samples A5 and A6 have demonstrated sub nanometer surface roughnesses compared to sample A4.

Fig. 4.2 RHEED patterns of AlN layer along $(11\bar{2}0)$ azimuth of samples A1 (a), A2 (b) and, A3 (c).

Faster transition of the growth mode from (3D) to (2D) at a growth rate of $0.13 \mu\text{m/h}$ of sample A3 has resulted in the improved crystal quality of AlN epilayer on Si substrate [80]. Higher growth rate has resulted in the improved wetting and nucleation of the growth surface, which consequently enables faster coalescence of nuclei, leading to the 2D growth mode and lesser dislocation generation. At lower growth rates, 3D growth mode and subsequent coalescence of grains has resulted in the increased threading dislocation generation at the coalescent boundaries.

Fig. 4.3 Effect of AlN growth rate (a) and growth temperature (b) on screw type dislocation density and RMS roughness of 100 nm AlN on Si.

Keeping the growth rate constant at $0.13 \mu\text{m/h}$ and increasing the growth temperatures from 890 to 920°C might have resulted in a similar wetting and coalescence of nuclei.

Thus, it has resulted in the comparable crystal quality of the epilayers. However, higher temperature might have increased the mobility of adatoms on the growth surface and produced sub nano-meter roughnesses of AlN layers. Thus, a growth rate of 0.13 $\mu\text{m/h}$ and a growth temperature of 920 $^{\circ}\text{C}$ has been fixed as the optimized growth parameters for the AlN growth using ammonia-MBE.

4.1.2 GaN growth

GaN growth was performed at a growth temperature of 800 $^{\circ}\text{C}$ and using a high V/III (BEP) ratio of 950 on the optimized AlN of 100 nm thickness. During the growth the ammonia flow of 280 SCCM and Ga flux of 2.3×10^{-7} Torr were maintained, and a growth rate of 0.22 $\mu\text{m/h}$ was achieved. The growth of GaN on AlN buffer was started with the spotty RHEED pattern and recovered slowly to a streaky pattern as shown in Fig. 4.4 (a).

Fig. 4.4 Evolution of RHEED patterns of GaN layer as a function of its thickness (a). Microscope image showing cracking of 1 μm thick GaN layer grown on 100 nm AlN buffer using 100-mm Si substrate (b).

Thus, the growth was initiated with the 3D growth mode and as the thickness increased, 3D islands got coalesced, leading to the streaky RHEED pattern after a thickness of 800 nm of GaN layer. RHEED pattern showed (2 $\times$ 2) surface reconstruction at the end of the growth after the substrate was cooled down to 300 $^{\circ}\text{C}$, signifying that the grown GaN epilayers were Ga-polar in nature. Further, optical microscope investigation on the

grown wafer (Fig. 4.4(b)) showed high density of cracks, indicative of higher tensile strain that developed in the GaN. The stress in GaN layer was quantitatively measured using micro-Raman measurements, which showed a red shift of GaN E_2 peak (563.8 cm^{-1}) with respect to the E_2 (567.5 cm^{-1}) peak of free standing GaN, indicating the tensile nature of the GaN layers. By considering the relation between Raman shift and stress in epitaxial GaN layers [179], a tensile stress value of 590 MPa has been obtained for $1 \mu\text{m}$ thick GaN layers. Since it is reported that the cracking of GaN layer occurs at a tensile stress of 800 MPa, the obtained tensile stress is the residual stress in GaN layers after the cracking. Thus, stress mitigating layer (SML) is necessary to compensate the tensile stress to obtain thick crack free GaN on Si substrate.

4.2 Stress mitigating layers for GaN growth

Among several types of SMLs [23, 102, 103, 107, 186-189] studied so far, AlGaN [102, 103] and AlN/GaN SMLs [23, 107] have been widely used for GaN growth on Si(111). However, optimization of these stress mitigating layers for GaN growth on Si using ammonia-MBE have been reported only on 50-mm diameter Si substrates. As the bowing of the GaN/Si wafer depends upon the substrate diameter, it is necessary to study the effect of both AlGaN and AlN/GaN SMLs on the stress and structural properties of GaN on 100-mm Si substrate. Three samples S1, S2, and S3 were grown and the corresponding epilayer structures are shown in Fig. 4.5 (a), (b) and, (c), respectively.

	1 st GaN 500nm	2 nd GaN 500nm
	Al _{0.45} Ga _{0.55} N 400nm	2 nd AlN 250nm
1 st GaN 500nm	1 st AlN 50nm	1 st GaN 250nm
1 st AlN 50nm	Si (111)	1 st AlN 50nm
Si (111)		Si (111)
(a)	(b)	(c)

Fig. 4.5 Epilayer structures of samples S1 (a), S2 (b) and S3 (c) grown on Si using ammonia-MBE.

As shown, a 50 nm thick 1st AlN layer at the bottom of the structure and a 500 nm thick GaN layer at the top were kept constant in samples S1, S2 and S3 to study only the influence of different SMLs on GaN properties. Sample S1 was grown on 50 nm thick AlN layer without the use of SML for comparison, while samples S2 and S3 were grown with Al_{0.45}Ga_{0.55}N (400 nm) and 2nd AlN (250 nm)/1st GaN (250 nm) SMLs, respectively. Growth rates of 0.22 and 0.40 $\mu\text{m}/\text{h}$ were maintained in that order for GaN and AlGaN

epilayers. While GaN and AlN layers were grown at the substrate temperature of 800 and 920 °C, respectively, AlGaIn layer was grown at an intermediate temperature of 840 °C.

RHEED patterns and the corresponding AFM surface morphologies of GaN of samples S1, S2, and S3 are shown in Figs. 4.6(a), 4.6(b), and 4.6(c), respectively. Sample S1 shows spotty RHEED pattern, indicative of 3D growth mode. Moreover, AFM image of this sample shows rough surface morphology with columnar type grains and high pit density. While samples S2 and S3 show streaky RHEED patterns indicative of two-dimensional (2D) growth mode, sample S2 shows smaller grain sizes and higher pit density compared to sample S3. Surface morphology of sample S3 typically appears as mound type, consistent with the morphologies observed for similar type of structures grown by ammonia-MBE [23, 107]. As shown in Fig. 4.7, the average lateral grain size observed from AFM images increases from sample S1 to S3, while the surface roughness (scan area 5×5 μm²) decreases from sample S1 to S2 and increases in sample S3.

Fig. 4.6 RHEED diffraction patterns along $[11\bar{2}0]$ direction and their corresponding AFM surface morphologies of GaN of samples S1 (a), S2 (b) and S3(c).

Fig. 4.7 A trend in the lateral grain size and the RMS roughness (scan area: $5 \times 5 \mu\text{m}^2$) obtained from the AFM images of samples S1, S2 and S3.

Table 4.1. Strain in GaN was determined by micro-Raman spectroscopy. Screw and edge type TDD in GaN and buffer ^{a)} layer of samples A, B and C was determined by HR-XRD.

Sample/ buffer layer	Strain at RT by micro- Raman* (%)	TDD** in GaN** (cm^{-2})		TDD** in buffer layer ^{a)} (cm^{-2})	
		Screw type ($\times 10^{10}$)	Edge type ($\times 10^{11}$)	Screw type ($\times 10^{10}$)	Edge type ($\times 10^{11}$)
S1 / 1 st AlN	+0.24	0.38	0.39	4.60	***
S2 / Al _{0.45} Ga _{0.55} N	+0.22	0.55	2.56	1.63	2.71
S3 / 2 nd AlN	+0.03	0.39	0.66	1.15	0.71

* '+' sign indicates tensile strain

** TDD was estimated from the HR-XRD rocking curves along (0002) and ($20\bar{2}1$) planes

*** Weak signal

a) Immediate bottom layer on which 500 nm thick GaN was grown is considered buffer layer in this study

The shift in E_2 phonon peak in Raman spectrum was used to determine the strain states of GaN [190] of samples S1 to S3 as listed in Table 4.1. As shown in Fig. 4.8 (a), E_2 peaks of GaN for both the samples S1 and S2 are largely red-shifted with respect to standard E_2

peak of free standing GaN ($\sim 567.5 \text{ cm}^{-1}$), indicating high tensile nature of these GaN layers. However, E_2 peak of GaN of sample S3 is very close to standard GaN E_2 peak position, indicating almost relaxed GaN layer. Figure 4.8 (b) shows the peak profiles of HR-XRD rocking curves obtained along the asymmetric $(20\bar{2}1)$ plane of the samples S1, S2, and S3. The XRD peak width is found to be lower for sample S1 compared to sample S2. The peak profile of sample S3 is closer to that of sample S1 but with a slightly higher peak width. Similar trend was observed for the measurements performed along the symmetrical (0002) plane. Broadening of the XRD peak profile indicates the deterioration in the crystal quality, which is found to be in the order of samples S1, S3, and S2. In order to quantify the crystal quality further, the rocking curves obtained along (0002) and $(20\bar{2}1)$ planes were used to estimate the screw and edge type threading dislocation densities, respectively in GaN and the buffer layer [191]. It should be noted that the immediate epilayer on which 500 nm thick GaN was grown is considered as the buffer layer for all these samples. As listed in Table 4.1, the 3D growth mode in sample S1 resulted in GaN with the lowest screw and edge type threading dislocation densities. However, when the samples grown using different SMLs are compared, sample S3 grown using AlN/GaN SMLs produced lower screw and edge type threading dislocation densities than sample S2 with AlGaIn SML.

Fig. 4.8 Raman shift in E_2 phonon peak of GaN in samples S1, S2 and S3 with the vertical line indicates free standing GaN E_2 position (567 cm^{-1}) (a). HR-XRD rocking curves along the asymmetric $(20\bar{2}1)$ plane of samples S1, S2 and S3(b).

The growth temperature and the V/III ratio of GaN were kept constant for all the samples. Thus, the variation in the surface morphologies obtained for samples S1 to S3 can be primarily attributed to the differences in the buffer layer on which GaN was grown, GaN nucleation conditions and the successive growth. Spotty RHEED pattern and columnar grain type surface morphology of sample S1 indicate that the growth preceded

more in the vertical than in the lateral direction. Lateral diffusion of Ga adatoms on the growth surface bridges the junction between two isolated nuclei and transforms the growth mode to 2D [192]. As listed in Table 4.1, the higher dislocation density in the buffer layer (40 nm thick AlN layer) of sample S1 and/or non-coalesced grains of thin (40 nm) AlN might have reduced the mobility of Ga adatoms on the growth surface and limited the coalescence of initial nucleated grains, leading to its rough and columnar type surface morphology with high pit density. However, the reduced dislocation density in the AlGaN buffer layer of sample S2 along with its smoother surface morphology possibly enhanced the surface diffusion and the coalescence of nucleated grains. This has resulted in 2D growth mode with lower surface roughness and lesser pit density for GaN of sample S2. These results further suggest that the initial nucleation and the successive growth proceeded in Volmer-Weber growth mechanism in samples S1 and S2. Furthermore, lower threading dislocation density (TDD) of the 2nd AlN and higher coherent strain ($\sim 2.5\%$ for GaN/AlN) in sample S3 might have increased the lateral diffusion of Ga adatoms on the growth surface and assisted in the formation of mounds through screw type dislocation-mediated step flow growth mechanism [90, 91]. Thus, with AlN/GaN SMLs, the growth mechanism was found to change from Volmer-Weber growth to screw type dislocation mediated step flow growth. Moreover, the lower pit density in GaN of sample S3 can be attributed to higher Ga adatoms diffusion. However, the slightly increased surface roughness can be due to the kinetic roughening of the grown mounds [90].

Continuous 3D growth of GaN in sample S1 helped in the annihilation of dislocations[193] and consequently resulted in lower TDD. The annihilation of dislocations in GaN occurs through the phenomenon of bending and looping of dislocations [194], which is driven by the initial compressive stress present in the layer [195, 196]. Compressive stress generated in GaN of sample S2 is lower due to its reduced lattice mismatch at AlGaN/GaN interface compared to the AlN/GaN interfaces of sample S3. Thus, the lower compressive stress in GaN of sample S2 might not have led to the annihilation of the dislocations through looping and hence resulted in higher TDD. On the other hand, the reduced TDD observed in sample S3 compared to sample S2 can be attributed to their annihilation at two subsequent AlN/GaN interfaces.

The overall residual stress in GaN after the growth depends upon various parameters such as the total lattice mismatch induced compressive stress generated during the growth of GaN, different relaxation mechanisms involved and the tensile stress generated during cool down due to the thermal mismatch between GaN and Si substrate.

The phenomenon of bending and looping of dislocations not only annihilates the dislocations but also relaxes the compressive stress in GaN. However, an intrinsic relationship between the lateral grain sizes and the residual compressive strain in GaN at the growth temperature was observed for all the samples. Similar to Vezian et al. [90], the residual compressive strain in GaN at the growth temperature was estimated by subtracting a constant tensile strain (0.23%) due to the thermal mismatch between GaN and Si from the strain at room temperature, determined from Raman measurements. As shown in Fig. 4.9, the residual compressive strain in GaN at the growth temperature increases with the increase of the lateral grain size from samples S1 to S3. This trend can possibly be attributed to the decrease in the tensile strain generation due to the grain coalescence [197]. However, the lattice mismatch induced compressive stress and its relaxation by dislocation looping in samples S1 to S3 might also play a role in the observed strains of GaN. Hence, overall, higher residual compressive strain during the growth, lower pit density and better crystal quality were observed for GaN of sample S3, compared to the GaN of sample S2, which shows that AlN/GaN SMLs has proved to be a better stress mitigating layer than AlGaIn SML.

Fig. 4.9 Strain in GaN layers vs lateral grain size for samples S1, S2 and S3.

4.3 AlN/GaN stress mitigating layer

The surface morphology of GaN of sample S3 with AlN/GaN SMLs exhibits pits on the surface as shown in Fig. 4.10(a), which are undesirable for good quality GaN HEMT heterostructures. It has been reported that the increased GaN growth rate has been found to enhance the lateral growth and quicker the transformation from 3D to 2D growth mode [198].

Fig. 4.10 Surface morphology of GaN grown on AlN/GaN SMLs with the growth rate of 0.22 $\mu\text{m}/\text{h}$ (a) and 0.7 $\mu\text{m}/\text{h}$ (b).

In this study, GaN growth rate was increased by increasing the Ga flux to 7.1×10^{-7} Torr and ammonia flow to 1000 SCCM simultaneously such that the V/III ratio (NH_3/Ga BEP) was kept constant at ~ 950 . However, increase in the Ga flux is an absolute parameter but increase in the nitrogen flux depends upon the cracking of ammonia on the growth surface. Ammonia cracking studies [107] showed that the cracking increased with the increase of ammonia flow up to 600 SCCM, beyond which it was found to have almost saturated. The increased cracking with increase of ammonia flow was attributed to the availability of more ammonia molecules and the subsequent saturation of cracking was attributed to the limited surface area of the substrate. Moreover, the presence of Ga flux was further found to have enhanced the cracking of ammonia on the growth surface and was attributed to the reaction between Ga and NH_3 molecules, which results in the incorporation of Ga and N species to form GaN [185].

Thus, GaN growth rate was increased from 0.22 to 0.70 $\mu\text{m}/\text{h}$ and was found to improve the surface morphology with pit-free surface and reduced RMS roughness of 2 nm as shown in Fig. 4.10(b). The improvement of the surface morphology can be attributed to the enhanced lateral over growth for the samples with higher GaN growth rate. The increased growth rate, obtained by the simultaneous increase of Ga flux and ammonia flow might have enhanced the cracking of ammonia and resulted in the incorporation of more Ga and N atoms. Incorporation of growth species in the lattice generally takes place at the step edges following the diffusion of Ga adatoms. This leads to the step flow or two dimensional growth mode and improved surface morphologies for the higher growth rate samples. However, it should also be noted that the diffusion of Ga adatoms to the step edges depends on the growth temperature [199], residual compressive stress [200, 201], ammonia over pressure and the formation of weak metallic Ga-Ga

bonds on Ga-polar GaN surface [202] [203] . As the growth temperature was kept constant at 800 °C, either one of these mentioned different parameters or a combination of all of them might have reduced the Ga adatom diffusion barrier and make them available for the enhanced step flow growth mode resulting in smooth surface morphologies with increased growth rates.

4.4 Strain states of AlN/GaN SMLs structure

AlN/GaN SMLs have resulted in compressively strained GaN layers with good crystalline quality and improved surface morphologies to grow AlGaIn/GaN HEMT heterostructures using ammonia-MBE. The optical microscope image of Fig. 4.11(a) shows the surface morphology of a heterostructure with 1 μm thick 2nd GaN grown using AlN/GaN SMLs. As shown in the figure, crack free and smooth surface morphology was obtained. However, buried cracks are found at the interface between AlN and GaN layers in the SMLs as shown in Fig. 4.11 (b). Observation of buried cracks in samples requires shifting the focus of the microscope from the sample surface to the interface of 2nd AlN and 1st GaN layers. Buried cracks have been also observed for the metal organic chemical vapor deposition (MOCVD) grown AlGaIn layers and ammonia-MBE grown AlN layers on GaN templates [204]. Brittle relaxation through crack formation has been referred as the reason for buried crack formation in nitride semiconductors due to the inability of forming misfit dislocation by Mathew-Blakeslee mechanism [205].

Fig. 4.11 The optical microscope image of AlGaIn/GaN HEMT heterostructures on Si substrate: the surface image (a) and the buried cracks at the interface between AlN and GaN layers in the SMLs (b).

MOCVD grown AlGaIn layers with good structural quality were demonstrated by the method of overgrowth of cracked AlGaIn layers [204]. In the structures grown using AlN/GaN SMLs, cracks are formed to relax the tensile strain in AlN layer. Continuation of the AlN growth after cracking leads to the lateral over growth of cracked AlN and results in smooth surface morphology. The laterally overgrown AlN acts as a buffer layer and

induce higher compression in the 2nd GaN, which helps in overcoming the tensile strain during the cool down[82, 107]. Tang et al. [206] have also accounted the buried cracks in AlN/GaN SMLs structure to the stress compensation mechanism during the cool down. Hence, buried cracks play a critical role in GaN heterostructures and determine their strain states and structural quality on Si substrate.

In order to understand the variation in strain states of AlN/GaN SMLs and their effect on the density of buried cracks, five structures were grown with varied thicknesses of 2nd AlN/1st GaN layers. Table 4.2 lists the thicknesses of 2nd AlN/1st GaN layers of samples A to E. The thickness of 1st AlN and 2nd GaN were kept constant at 50 and 500 nm, respectively for all the samples. The streaky RHEED patterns observed during the growth of different epilayers in samples A to E indicated that the growths proceeded in two-dimensional mode for all the samples. In-situ curvature data obtained during the growth of a typical structure with AlN/GaN SMLs (with thicknesses of 250/250 nm) is shown in Fig. 4.12.

Fig. 4.12 In-situ curvature plot of a typical structure with 250/250 nm 2nd AlN / 1st GaN layers as a function of growth time.

The slope of the curvature becomes positive during AlN growth and negative during GaN growth, indicative of tensile and compressive stresses, respectively. Further, the increment in the curvature is found to be steeper for 2nd GaN compared to 1st GaN layer indicative of its higher residual stress and slower relaxation rate. Raman measurements

were also performed on samples A to E at room temperature to measure the strain states of 2nd GaN layer by observing the shift in GaN-E₂ phonon mode. The focus of confocal microscope in Raman measurements was adjusted on to the sample surface (2nd GaN layer) to collect the signal. The true surface of the sample was determined by differentiating the GaN E₂ peak intensity with respect to the depth [207], as presented in chapter 3 and section. 3.2.2. As the peak of phonon modes in Raman measurements follows the Lorentzian profile [208], the exact peak positions of E₂ mode were determined by Lorentzian fit. Further, the stress in GaN layers were estimated using the relation between biaxial stress and Raman shift [179]. Table 4.2 lists the Raman shift in E₂ peak position and the corresponding stress value for samples A to E. A red shift in E₂ peak was observed from samples A to E with decreased compression in 2nd GaN layer.

Table 4.2. Raman shift of GaN-E₂ phonon peaks and their corresponding stresses in 2nd GaN layer of samples A to E. The average crack spacing was estimated from the microscope images of the buried cracks.

Sample	2 nd AlN /1 st GaN Thicknesses (nm)	Raman shift in E ₂ peak (cm ⁻¹)	Stress in 2 nd GaN (MPa)*	Average crack spacing of buried cracks (μm)
A	150/120	569.14	-395	1.55
B	250/120	568.93	-319	1.58
C	250/250	568.43	-200	1.37
D	250/350	568.00	-153	1.25
E	370/470	567.06	+132	1.04

* '- ve' and '+ ve' signs indicate compressive and tensile strains, respectively.

4.4.1 Density of buried cracks in AlN/GaN SMLs

Surface morphology of samples A to E, observed using an optical microscope, presented crack free surface for all the samples except E. The cracking of sample E coincides with the observation of nearly relaxed 2nd GaN in Raman measurements, where the tensile strain generated in GaN layer might have relaxed by cracking. Figures 4.13 (a) and (b) show the buried crack features of samples A and E, respectively.

Fig. 4.13 Microscopic images of buried cracks of samples A (a) and E (b).

As can be seen, the buried cracks are oriented in several crystallographic directions and the crack density is higher in sample E compared to sample A. Quantification of the buried crack density is difficult as the cracks are oriented in different crystallographic directions. However, a numerical representation of the buried crack density was obtained by taking the statistical average of crack spacing between two parallel cracks in different directions from the microscope image. Table 4.2 lists the average spacing of buried cracks for samples A to E.

From Table 4.2, results for samples B, C and, D show that the average crack spacing of buried cracks decreases with the increase of 1st GaN layer thickness, while the thickness of 2nd AlN layer is kept constant at 250 nm. However, for a constant 1st GaN layer thickness of 120 nm, the crack spacing is found to be almost independent of the thickness of 2nd AlN when its thickness is varied from 150 to 250 nm (samples A and B). These observations suggest that the density of buried cracks respond more to the thickness of 1st GaN layer than to the thickness of 2nd AlN layer.

Buried cracks are formed at the interface between 2nd AlN and 1st GaN layers to relax the lattice mismatch induced strain during the growth. In-situ stress measurements offer critical details of the strain states of the epilayer during the growth. Hence, the formation of buried cracks and their behavior were observed using an in-situ curvature measurement set-up. Figure 4.14 shows a ‘close-look’ of the in-situ curvature data during the growth of 2nd AlN/ 1st GaN layers with the thickness of 250/250 nm. As can be seen, the curvature data is divided into 6 segments from point 1 to 7. The segment between ‘1’ to ‘2’ indicates the in-situ wafer curvature during the growth of 1st AlN layer. The segments between ‘2’ to ‘4’ and ‘4’ to ‘7’ indicate the curvature during the growth of 1st GaN and 2nd AlN layers, respectively. The overall in-situ curvature measured is a

result of stresses induced and simultaneous relaxation processes during the growth. Moreover, the slope of the curvature becomes negative from point '2' to '3' of GaN growth, indicating the development of net compressive stress. However, the curvature changes its sign, and its slope becomes positive at point '3', signifying that the relaxation process overtakes the compression at this point. Based on the curvature data obtained from repeated growths, the typical thickness determined for the relaxation is around 110-130 nm.

The relaxation of GaN layer occurs through dislocation bending and looping as described by Cantu et al. [209] and modeled by Romanov and Speck [210]. Plastic relaxation ($\bar{\epsilon}_{pl}$) through dislocation bending and looping follows Eq. 4.1, where 'b' is the magnitude of the burger vector, ' ρ_{TD} ' is the threading dislocation density, 'h' is the layer thickness of dislocation looping and ' α ' is the inclination angle.

$$\bar{\epsilon}_{pl} = \frac{1}{2} b \rho_{TD} h \tan(\alpha) \quad \text{Eq. 4.1}$$

Fig. 4.14 In-situ curvature plot of 1st GaN and 2nd AlN layers with thicknesses of 250 nm as a function of growth time. The inset shows the TEM image of a typical buried crack feature.

It was observed that the compressive stress relaxation of GaN grown in 2D growth mode using AlGaN SML was mainly due to the dislocation looping mechanism [211]. Similarly, as shown in Fig. 4.12, the steeper increment in the curvature of 2nd GaN layer with thickness can be attributed to its lower compressive stress relaxation due to the lesser dislocation density and their consequent annihilation compared to 1st GaN layer. To

understand the relaxation process of 1st GaN layer, the dislocation behavior in the 1st GaN layer was observed using cross sectional TEM images of Fig. 4.15.

Fig. 4.15 The cross-sectional TEM images of sample A (a), sample C (b) and sample E (c) obtained using bright field mode along $(11\bar{2}0)$ zone axis. The arrow marks indicate the looping of dislocations.

As indicated by the arrow marks in the figure, clear bending and looping of dislocations occur in samples C and E. However, sample A showed relatively lesser looping as most of the dislocations appeared to be travelling to the top layers. The layer thickness of dislocation looping, ' h ' is found to be around 110 nm, which is well matched with the critical thickness of relaxation from the curvature data of Fig. 4.14. Hence, the sudden relaxation of 1st GaN layer in the curvature measurements can be attributed to the intense bending and looping of dislocations around this thickness. However, as can be seen Fig. 4.15 (b) and (c), dislocation activity continues in the 1st GaN layer throughout its thickness to further relax the residual compressive strain. Sahonta et al.[212] have also observed similar behavior of compressive stress relaxation in GaN layers with increased thickness and attributed it to dislocation bending and looping mechanism. The increase in relaxation of 1st GaN layer increases its lattice mismatch with 2nd AlN, leading to higher tensile strain generation. The higher tensile strain in AlN layer further favors the formation of high density of cracks to relax the strain. Thus, the observed trend of decreased crack spacing with the increased 1st GaN thickness in samples B to D can be attributed to the enhanced relaxation of GaN layer with thickness. Moreover, the

highest crack spacing observed in sample B with 1st GaN thickness of 120 nm might be due to its partially relaxed GaN layer, resulted from the lesser dislocation looping.

The curvature data also provides an insight into the effect of 2nd AlN layer on the density of buried cracks. As shown in Fig. 4.14, the positive slope of curvature at point '4' signifies the growth of tensile strained 2nd AlN on 1st GaN layer. The curvature subsequently becomes flat at point '5' and saturates with a slight negative slope towards point '6', indicating the relaxation through the formation of cracks and growth of relaxed AlN layer, respectively. Here, the negative slope observed from point 5 to 6 might be due to the induced compression in the underlying GaN layer, which is explained in detail in the following section. After the initial relaxation through cracking, development of tensile strain is observed at point '6' of AlN layer growth. As shown in the TEM image for the sample C of Fig. 4.14, point '6' corresponds to the AlN thickness of ~70 nm, where the lateral overgrowth of cracked regions initiates. The process of lateral overgrowth might have resulted in tensile strain generation in AlN layer [197, 213]. Thus, from the in-situ curvature measurements, different stages of 2nd AlN growth such as the initial tensile strain generation followed by the relaxation through buried crack formation and the lateral overgrowth can be observed. Furthermore, it can also be seen that the relaxation of AlN through crack formation is not observed beyond point '5'. This signifies that the cracking and formation of buried cracks is not observed anymore during 2nd AlN layer growth up to 250 nm of its thickness. This justifies the observation of similar crack spacing for samples A and B with AlN layer thickness of 150 and 250 nm, respectively. Thus, the density of buried cracks is found to be independent of 2nd AlN layer thickness (up to 250 nm) and depends primarily on the relaxation of 1st GaN layer and hence on its epilayer thickness.

4.4.2 Density and size of voids in AlN/GaN SMLs

AlN/GaN SMLs structure contains voids in the 1st GaN layer along with the buried cracks in the structure as shown in the cross-sectional TEM images of Fig. 4.15. Cracks in the 2nd AlN layer propagates to the 1st GaN layer due to the strain field that develops at the crack edge of 2nd AlN layer [204]. Tang et al. [206] have reported that the formation of voids in GaN layer occurs due to the decomposition of GaN in cracked regions and diffusion along the crack opening at high growth temperature of AlN. As shown in Fig. 4.15, no voids were observed in TEM image of sample A with thinner AlN/GaN layers, indicating very low void density. However, samples C and E clearly showed the presence of voids with their density and size increasing from sample C to E. The increased growth time of 2nd AlN layer from sample A to C to E led to the continuous decomposition of GaN

in the cracked regions, which enhanced the probability of void formation and increased the size of the existing voids. The material decomposed during this process fills in the buried cracks of 2nd AlN layer and the upper portion of voids in the 1st GaN layer. EDX measurements performed on the material in the buried crack regions of AlN showed the presence of Al and Ga, indication of possible formation of AlGa₂N. Similarly, Bethoux et al. [204] have also observed GaN domains in the buried crack regions by EDX analysis, which confirms the mass transport. Further, they have also reported that the depth of the crack in GaN layer, over which AlGa₂N layer was grown, increases as its thickness is increased. The increased crack depth in GaN might have also exposed more cracked area to higher temperature during the subsequent AlN growth leading to higher decomposition and increased void dimensions. Thus, the thicknesses of both 2nd AlN as well as 1st GaN layers might play a critical role in the variation of density and size of voids in AlN/GaN SMLs structure.

4.4.3 Effect of buried cracks on the residual stress in GaN buffer layers

The relaxation of 2nd AlN layer due to cracking can be visualized from the in-situ curvature measurements. However, the curvature measurement does not show the effect of cracking on the strain states of 1st GaN layer. Two samples namely F and G, shown as insets in Fig. 4.16 were grown to study the effect of cracking on the 1st GaN layer. Sample G with the growth sequence of 2nd AlN/1st GaN/1st AlN showed the presence of buried cracks in the microscope image, whereas sample F with only 1st GaN/1st AlN was found to be completely crack free (microscope images are not presented here). Fig. 4.16 shows the normalized GaN-E₂ phonon peaks obtained from both the samples. A high spatial resolution of < 0.35 μm of the confocal microscope allowed us to obtain Raman spectra from the close vicinity of the crack and from the center of the two parallel cracks of sample G, as represented in Fig. 4.16 as G1 and G2, respectively. The solid vertical line at 567.5 cm⁻¹ in the Raman spectra indicates the E₂ position of free standing GaN.

Fig. 4.16 Raman shift in GaN- E_2 peak of samples F and G. G1 and G2 correspond to phonon modes obtained from the close vicinity and in between the cracked regions, respectively. The insets show the epilayer structures of samples F and G.

The red shift in GaN E_2 peak of sample F with respect to the free standing GaN E_2 peak indicates its tensile nature, whereas the blue shift of GaN E_2 (G2) peak of sample G indicates its compressive nature. This observation clearly suggests that the growth of 2nd AlN layer induces compression in the 1st GaN layer, changing its strain state from tensile to compression. Further, the slight negative slope of curvature observed during the growth of the relaxed AlN layer from point 5 to 6 in Fig. 4.14 can be attributed to the induced compression in the 1st GaN layer by the growth of 2nd AlN layer. Steude et al. have also observed similar behavior, where the growth of AlGaIn layers was found to induce compression in the bottom GaN layer [214]. Moreover, the separation of GaN E_2 peaks at positions G1 and G2 in Fig. 4.16 shows that the formation of buried cracks relaxes the induced compression in GaN layer. Thus, the growth of 2nd AlN layer induces compression in the 1st GaN layer, which gets partially relaxed through crack formation at the interface.

The variation in the crack spacing of samples A to E (Table 4.2) indicates different compression states of the 1st GaN layer. To determine the effect of strain states of the 1st GaN layer on the 2nd GaN layer, Raman measurements were performed on the 1st and 2nd GaN layers of samples A to E. The focus of the confocal microscope was adjusted to the sample surface and to the 1st GaN layer to obtain the spectra from the 2nd and 1st GaN layers, respectively. Focusing of the 1st GaN layer was achieved by adjusting the microscope focus on to the interface containing buried cracks. In the case of 1st GaN

layer, Raman spectrum was collected from the center of the two parallel cracks to determine its average compression. Further, to confirm the results obtained from the confocal microscopy on the 1st GaN layer, 2nd GaN layer was etched away using reactive ion dry etching and observed that the stress values using Raman measurements are closer to the ones obtained using confocal microscopy. A typical plot of normalized GaN E₂ peaks of 1st and 2nd GaN layers obtained from sample E is shown in Fig. 4.17. The separation of GaN E₂ peak positions indicates the different strain states of these two layers.

Fig. 4.17 Raman shift in E₂ phonon peak of 1st and 2nd GaN layers of sample E.

Stresses obtained from Raman measurements of GaN layers in samples A to E are plotted as a function of buried crack spacing in Fig. 4.18. It can be seen that the residual compression in the 1st and 2nd GaN layers decreases with the decrease of the buried crack spacing. As discussed, relaxation of the 1st GaN layer through crack formation was observed in Raman measurements on sample G at position G1 as shown in Fig. 4.16. Hence, the decrease in the residual compression in 1st GaN with the decrease of crack spacing can be attributed to the increased relaxation through cracking.

Fig. 4.18 Stresses in 1st and 2nd GaN layers as a function of average crack spacing. The connecting lines are for the guidance to eyes only.

However, the formation of buried cracks at the interface of 1st GaN/2nd AlN layer may not directly affect the strain states of the 2nd GaN layer. Using variable temperature high resolution X-ray diffraction measurements, Tang et al. have shown that AlN/GaN SMLs compensates the thermal strain during cool down and attributed it to the buried cracks in the structure [206]. But, our observation from Fig. 4.18 indicates that the increase in the buried crack density is found to decrease the residual compression of the 2nd GaN layer. Moreover, a trend of decreased compression of 2nd GaN layer with the decrease in compression of 1st GaN layer is observed. These two observations suggest that the stress compensation observed in AlN/GaN SMLs structure during cool down may be related to the compressive nature of 1st GaN layer together with buried crack density. Compression in the 1st GaN together with the buried cracks act as a stress compensating mechanism for the tensile stress generated during cool down and hence reduces the residual compression in the 2nd GaN layer with reduction in the compression of 1st GaN. Thus, the strain states of GaN layer during the growth of AlN/GaN SMLs influence the density of buried cracks, which further affect the residual strain in GaN buffer layers. These results also point out that AlN/GaN SMLs not only helps in inducing higher compression during the growth of GaN buffer but also compensates the thermal tensile strain generated due to GaN heteroepitaxy on Si during cool down, where the compressive strain in 1st GaN together with the buried cracks act as a compensation mechanism for the thermal strain.

4.5 Optimization of AlN/GaN SMLs for AlGaIn/GaN HEMT heterostructures

AlN/GaN SMLs of sample A with thicknesses of 120/150 nm showed higher compression for 500 nm thick GaN layer as shown in the Fig. 4.18. However, it should be noted that the selection of the optimized structure for GaN growth on Si should not only be dependent on their efficiency in compensating the tensile strain but also on its ability to produce lower dislocation density and smooth surface morphology. Thus, samples A to E were investigated for their structural quality and surface morphology using HR-XRD and AFM techniques, respectively. HR-XRD rocking curve scans along GaN (0002), (0004) and (0006) planes were used to generate Williamson-Hall plots and obtain the screw type dislocation density, and FWHM of GaN (30 $\bar{3}$ 2) scans were used to estimate the edge type dislocation density in GaN layers [176]. Even though samples D and E, grown with thicker AlN/GaN SMLs combination showed comparatively lower dislocation densities as listed in Table 4.3, sample D showed lower compression for 500 nm thick GaN layer (Fig. 4.18) and sample E showed cracks on the surface of the epilayer structure.

Table 4.3. Dislocation density in sample A to E, estimated from HR-XRD rocking curve scans.

Sample	dislocation density ($\times 10^{10} \text{ cm}^{-2}$)	
	Screw type	Edge type
A	0.39	5.32
B	0.41	6.18
C	0.56	5.05
D	0.46	4.21
E	0.37	4.45

Among samples A, B and C, samples A and C showed similar dislocation density but are lower compared to sample B. Moreover, the surface morphology of samples A and C were further investigated to obtain the optimized AlN/GaN SMLs layer structure. As shown in Fig. 4.19, both samples A and C showed mound type surface morphology. However, sample A shows higher pit density on the growth surface with an RMS roughness value of 3.6 nm on $5 \times 5 \mu\text{m}^2$ scan area, whereas sample C shows pit-free surface morphology with an RMS roughness of 2.1 nm. Higher pit density on the surface

of sample A might be possible due to the incomplete recovery of 2nd AlN layer surface, owing to its lower thickness of 120 nm. Thus, AlN/GaN SMLs with thicknesses of 250/250 nm yielded compressively strained GaN layers with smooth surface morphology and good crystal quality. Hence, this structure was considered for the development of GaN based HEMT heterostructures on Si using ammonia-MBE in this study.

Fig. 4.19 Surface morphology of samples A (a) and C (b). Sample A shows pits on the surface with RMS roughness of 4.8 nm and sample C shows pit free surface with RMS roughness of 2.06 nm.

4.6 Optimization of 2nd GaN for the AlGaIn/GaN HEMT heterostructures

On the optimized 250/250 nm AlN/GaN SMLs, 2nd GaN was grown with a thickness up to 1000 nm. Surface morphology of the grown heterostructure was investigated by AFM and is shown in the Fig. 4.20.

Fig. 4.20 AFM Surface morphology of 1000 nm thick 2nd GaN, grown with a growth rate of 0.7 μm/h.

Mound type surface morphology was observed with some of the mounds exhibiting roughening behavior. An increased surface roughness of 4.2 nm was obtained, which is higher compared to 2.06 nm obtained for a 500 nm thick GaN buffer. The increased

roughness can be attributed to the kinetic roughening of the grown mounds with the increasing thickness [90] .

However, increasing the V/III ratio of 2nd GaN from 950 to 2070 by the reduction of the Ga flux alone and keeping the ammonia flow at 1000 SCCM has resulted in the improvement of the surface morphology with the reduced RMS roughness of 3 nm. Figure 4.21 shows the AFM surface morphology of 2nd GaN, grown with a V/III ratio of 2070. Here it should be noted that the V/III ratio change leads to a change in the growth rate of GaN from 0.70 to 0.28 $\mu\text{m}/\text{h}$.

Fig. 4.21 AFM Surface morphology of 1000 nm thick 2nd GaN layer, grown with a growth rate of 0.28 $\mu\text{m}/\text{h}$.

In-situ curvature measurement obtained during the growth of GaN based HEMT heterostructure with the 2nd GaN thickness of 900 nm is presented in Fig. 4.22. The V/III ratio of 2nd GaN is kept at 2070. Steeper increment in the negative curvature during the growth of 2nd GaN indicates its lower relaxation rate of induced compression compared to the 1st GaN layer. Further, incremental stresses in the 2nd AlN and 2nd GaN layers were calculated using the modified Stoney's equation (Eq. 4.2) [69] with a correction factor, 'c' obtained from nonlinear theory [175]:

$$k = 6c \frac{M_{III-N}}{M_{Si}} \frac{h_{III-N}}{h_{Si}^2} \epsilon \quad \text{Eq.4.2}$$

The stress-thickness product is given as:

$$\sigma_{III-N} \cdot h_{III-N} = k \left(6c \frac{1}{M_{Si}} \frac{1}{h_{Si}^2} \right)^{-1} \quad \text{Eq.4.3}$$

where, 'k' is the relative change in curvature, M_{III-N} & M_{Si} and h_{III-N} & h_{Si} are the biaxial modulus values and the thicknesses of grown nitride epilayer and Si substrate, respectively while ' ϵ ' and σ_{III-N} represents developed strain and the corresponding

stress in the epitaxial layer. Incremental stresses were obtained by differentiating the stress thickness product curve with the thickness.

Figure 4.23 (a) and (b) shows the stress-thickness product and the corresponding incremental stresses in 2nd AlN and 2nd GaN layers as a function of their thicknesses. As shown in Fig. 4.23 (a), 2nd AlN layer starts with a high tensile strain of ~ 11.9 GPa, nearly consistent with the 11.4 GPa of tensile strain developed due to 2.5% lattice mismatch between relaxed GaN and AlN layers. However, as the growth proceeds, cracking and subsequent coalescence lead to an end tensile stress of 1.6 GPa for 250 nm of AlN thickness. Further, initial incremental stress in the 2nd GaN layer grown over the 2nd AlN layer gives an induced compressive stress of 8.85 GPa. Considering the tensile strain at the end of 2nd AlN layer (1.6 GPa) and 2.5% lattice mismatch between GaN and AlN layers, theoretical value of induced compressive stress is ~ 9 GPa, indicating a good match between them. Moreover, the induced compressive stress relaxes with the increased thickness such that the residual compressive stress in 2nd GaN layer is 1.54 and 0.8 GPa, respectively for 500 and 900 nm of its thickness.

Kinetic roughening of the mound type morphology has been attributed to the reduction in the surface diffusion of Ga adatoms with thickness [90]. As shown in the Fig. 4.23(b), with the increase in the thickness of 2nd GaN, relaxation of induced compression occurs. This reduces the diffusion of Ga adatoms on the surface and results in the roughening of the grown mounds. However, it was also found that the process of roughening was V/III ratio or incident Ga flux dependent where the constant ammonia flow and increased V/III ratio by reducing Ga flux alone led to smoother surface morphologies. Thus, not only diffusion of Ga adatoms but also a combination of adsorption and incorporation mechanisms might play a role in the developed morphologies of GaN. To investigate the factors that influence the surface morphology, a systematic study of the effect of V/III ratio by changing the Ga flux alone on the GaN surface morphology is investigated and is discussed in detail in Section 5.2.7 of Chapter 5. Thus, the optimization of 2nd GaN growth using AlN/GaN SMLs involves increasing the V/III ratio up to 2070 by keeping the ammonia flow constant at 1000 SCCM.

Fig. 4.22 In-situ curvature measurement obtained during the growth of AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.

Fig. 4.23 Stress × Thickness and incremental stresses in 2nd AlN (a) and 2nd GaN layers (b).

Figure 4.22 also shows that the curvature changes sharply to a positive value of 0.01 m^{-1} during the cool down to 200°C , which should be associated with the strain generated due to the thermal mismatch between the nitride epilayer and the Si substrate. For several growth runs using AlN/GaN SMLs, room temperature tensile bow values ranging from $30\text{-}70 \text{ }\mu\text{m}$ were observed, which requires further improvement.

4.7 Growth of AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs

AlGaIn/GaN HEMT heterostructures were grown using the optimized $250/250 \text{ nm}$ AlN/GaN SMLs and 2nd GaN of thickness up to 1000 nm . A typical schematic diagram of AlGaIn/GaN HEMT heterostructure grown on 100-mm Si(111) substrate is shown in the

Fig. 4.24 (a) and the corresponding grown wafer is shown in the photograph of Fig.4.24(b).

Fig. 4.24 Schematic of AlGaIn/GaN HEMT heterostructure on Si using AlN/GaN SMLs (a). Photograph of grown HEMT heterostructure on 100-mm Si(111) wafer (b).

Structural characterization of the AlGaIn/GaN HEMT heterostructures was performed using HR-XRD measurements. The increase in the 2nd GaN layer thickness from 500 to 1000 nm was found to improve the crystal quality of GaN layers due to reduced screw type dislocation density from 5.5×10^9 to 1.75×10^9 cm⁻² and edge type dislocation density from 5×10^{10} to 3.8×10^{10} cm⁻². Further, a typical ω -2 θ scan of AlGaIn/GaN HEMT heterostructure with an aluminum composition of 29.5% and AlGaIn thickness of 27 nm is shown in Fig.4.25 (a). The figure also presents the dynamic simulation of HR-XRD data to obtain the composition and thickness of AlGaIn barrier layer. Observation of pendullösung fringes in the ω -2 θ scan indicates good structural perfection and abrupt interface of AlGaIn/GaN HEMT heterostructure along c-direction. Moreover, reciprocal space mapping was performed using HR-XRD along GaN (10 $\bar{1}$ 5) direction to check the coherence of AlGaIn barrier layer. As shown in Fig. 4.25 (b), a perfect alignment of AlGaIn barrier with GaN buffer layer indicates the coherent growth without any relaxation.

Fig. 4.25 High resolution X-ray diffraction ω - 2θ scan of AlGaN/GaN HEMT heterostructure with aluminum composition of 29.5% and AlGaN thickness of 27 nm (a). Reciprocal space mapping of AlGaN/GaN HEMT heterostructure along GaN(10 $\bar{1}$ 5) direction (b).

Capacitance-voltage measurements were performed on as grown AlGaN/GaN HEMT heterostructures using non-destructive mercury probe measurement system. A typical C-V plot of HEMT heterostructure with 27 nm thick $\text{Al}_{0.295}\text{Ga}_{0.705}\text{N}$ barrier is shown in Fig. 4.26. As shown, a plateau of capacitance followed by its sharp pinch off represents the presence of 2DEG at AlGaN/GaN interface.

Fig. 4.26 Capacitance-voltage measurements of AlGaIn/GaN HEMT structure. Sheet carrier concentration obtained by integration of C-V profile is also presented.

The concentration of 2DEG was obtained by integrating the area under C-V curve, which resulted in value of $1.1 \times 10^{13} \text{ cm}^{-2}$. Further, the capacitance values obtained after pinching off the 2DEG represents the background carrier concentration in GaN buffer layer. A typical depth profile of carrier concentration obtained using C-V measurements is presented in Fig. 4.27. The initial high carrier concentration represents the 2DEG and its corresponding depth indicates the combined thickness of GaN cap and AlGaIn barrier layer. The background carrier concentration in the GaN buffer layer was found to be lower ($\sim 10^{15} \text{ cm}^{-3}$).

Fig. 4.27 Depth profile of carrier concentration in the AlGaIn/GaN HEMT heterostructure.

Hall measurements performed in Van der Pauw configuration on AlGaIn/GaN HEMT heterostructure showed a carrier concentration of $1.13 \times 10^{13} \text{ cm}^{-2}$ and a mobility of $1340 \text{ cm}^2/\text{V.s}$, which resulted in a sheet resistance of $409 \Omega/\text{sq}$. The 2DEG concentration obtained was almost consistent with the value obtained from CV measurements. Moreover, Hall measurements performed as a function of temperature (Fig. 4.28) showed that the carrier concentration was insensitive to the temperature while the mobility increased up to $4290 \text{ cm}^2/\text{V.s}$ at 90K.

Fig. 4.28 Mobility and sheet carrier concentration as a function of measurement temperature for AlGaIn/GaN HEMT heterostructure.

The insensitivity of carrier concentration to temperature confirms the existence of 2DEG, while the improvement in the mobility with decreasing temperature indicates reduced phonon scattering due to good structural quality of AlGaIn/GaN HEMT heterostructures. Uniformity in 2DEG electrical characteristics of AlGaIn/GaN HEMT heterostructures across 100-mm Si substrate was obtained using a non-contact Hall measurement system. As shown in Fig. 4.29 (a), (b) and (c), a good uniformity is achieved in sheet resistance, mobility and sheet carrier concentration with their standard deviation values of 2.9, 3.6 and 3.0%, respectively. Moreover, a good run-to-run growth reproducibility was observed with a standard deviation of 3.6 and 2.6% for mobility and sheet carrier concentration, respectively.

Fig. 4.29 2DEG mapping of sheet resistance (a), mobility (b) and sheet carrier concentration (c) of AlGaIn /GaN HEMT heterostructures on 100-mm Si substrate grown using AlN/GaN SMLs.

HEMT devices were fabricated using grown AlGaIn/GaN heterostructures on 100-mm Si using a T-gate with a gate length and width of 0.3 and $2 \times 142 \mu\text{m}$, respectively. The gate stack used was Ni/Au with their thicknesses of 50 and 400 nm, respectively. Fig. 4.30 shows the $I_{\text{DS}}-V_{\text{DS}}$ (a) and transfer characteristics (b) of HEMT devices, which exhibited a maximum drain current (I_{Dmax}) of 768 mA/mm at $V_{\text{g}} = +1\text{V}$ and a maximum transconductance (g_{mmax}) of 190 mS/mm at $V_{\text{D}} = 6\text{V}$. The devices showed good pinch off characteristics with a threshold voltage of -4.53 V.

Fig. 4.30 $I_{\text{DS}}-V_{\text{DS}}$ characteristics of AlGaIn/GaN HEMTs (a). Transfer characteristics of AlGaIn/GaN HEMTs (b).

4.8 Summary

Nucleation of AlN on Si substrate using ammonia-MBE growth is studied followed by the optimization of AlN epilayer to achieve smooth surface morphology and good crystal quality. GaN epilayer of 1000 nm thickness grown on AlN was found heavily cracked. AlGaIn SML and AlN/GaN SMLs were investigated to achieve crack free GaN. High residual compressive stress at the growth temperature, larger grain sizes, lower pit density and good crystalline quality indicated that the AlN/GaN SMLs served as efficient stress mitigating layers compared to AlGaIn SML. Moreover, AlN/GaN SMLs were found to be not only successful in inducing the compressive stress in 2nd GaN but also mitigate the stress transfer during cool down. Hence, they were considered for the development of GaN based HEMT heterostructures. The surface roughness of GaN buffer layer of 1000 nm thickness grown on AlN/GaN SMLs showed a surface roughness of 4.2 nm for a scan area of $5 \times 5 \mu\text{m}^2$. However, it was reduced to 2.8 nm by the increased V/III ratio of 2nd GaN from 950 to 2070. AlGaIn/GaN HEMT heterostructures on 100-mm Si (111) resulted in crack free wafers with tensile bowing in the range of 30-70 μm . The HEMT

heterostructure showed a room temperature mobility of $1340 \text{ cm}^2/\text{V.s}$ and carrier concentration of $1.13 \times 10^{13} \text{ cm}^{-2}$, which resulted in a sheet resistance of $409 \text{ } \Omega/\text{sq}$. A good uniformity across 100-mm wafer was achieved with standard deviation of 2.9, 3.6 and 3.0% for the sheet resistance, mobility and sheet carrier concentration, respectively.

5. Stress management in AlGaN/GaN HEMT heterostructures on 100-mm Si(111) using AlGaN/AlN/GaN SMLs

GaN based HEMT heterostructures with a GaN buffer thickness of $\sim 1 \mu\text{m}$ have resulted in a tensile bow of 30-70 μm for the growths using AlN/GaN SMLs on 100-mm Si substrate as discussed in Chapter 4. Even though the grown epiwafers were crack free, it is important to reduce the tensile bow. The reduction of tensile bow requires either to mitigate the stress from 2nd GaN to Si or to induce high compression during its growth. In order to achieve this, stress mitigating layers with an epilayer stack of AlGaN/AlN/GaN were investigated and reported in this Chapter.

5.1 AlGaN/AlN/GaN SMLs

Compared to AlN/GaN SMLs, the proposed SMLs have an additional $\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$ epilayer grown between 2nd AlN and 2nd GaN. Figure 5.1 shows the cross sectional view of grown heterostructures on 100-mm Si substrate using AlN/GaN (a) and AlGaN/AlN/GaN SMLs (b).

2 nd GaN 900 nm		2 nd GaN 900 nm	
2 nd AlN 250 nm		$\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$ 250 nm	250 nm
1 st GaN 250 nm		2 nd AlN 250 nm	250 nm
1 st AlN 50 nm		1 st GaN 250 nm	250 nm
Si(111)		1 st AlN 50 nm	50 nm
(a)		Si(111)	
		(b)	

Fig. 5.1 The cross sectional view of the heterostructures on 100-mm Si substrate, using AlN/GaN (a) and AlGaN/AlN/GaN SMLs (b).

The growth of $\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$ is proposed to reduce the lattice mismatch with 2nd AlN to decrease the relaxation of residual compressive stress in 2nd GaN during the growth. Lower relaxation and higher residual compression in the 2nd GaN helps to decrease the tensile bow of the wafer during cool down. In order to investigate the effect of AlGaN/AlN/GaN SMLs on the structural properties of 2nd GaN, a comparative study was made between the AlN/GaN SMLs and AlGaN/AlN/GaN SMLs. Two samples were grown, namely S1 and S2 with 2nd GaN thickness of 900 nm on 100-mm Si substrate using AlN/GaN SMLs and AlGaN/AlN/GaN SMLs as shown in Figs. 5.1 (a) and (b), respectively. An optimized V/III ratio of 2070, which results in a growth rate of 0.28 $\mu\text{m}/\text{h}$ obtained from the study of AlN/GaN SMLs (Section 4.6 of Chapter 4) was used for the growth of 2nd GaN of samples S1 and S2.

5.1.1 In-situ stress measurements

The behavior of wafer curvature and the evolution of stresses in the grown epilayers are expected to be similar up to 2nd AlN which are discussed in Chapter 4. Figure 5.2 shows the stress-thickness product and the incremental stress as a function of thickness of 2nd GaN in both the samples S1 and S2. It is to note that the 2nd GaN layer in sample S2 represents the combined effect of this layer with $\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$. Thus, the data for the first 250 nm represents the stress thickness product and incremental stress of the $\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$ epilayer.

Fig. 5.2 Stress × Thickness and the incremental stress as a function of thickness of samples S1 and S2.

The incremental stress is observed to be relaxed by 68% in the first 250 nm of 2nd GaN of sample S1, whereas it is relaxed by 64% in the $\text{Al}_{0.065}\text{Ga}_{0.935}\text{N}$ epilayer of sample S2.

Subsequently, a relaxation of 82% and 91% is observed in the 2nd GaN of sample S1 at 500 and 900 nm thicknesses, respectively. The stress relaxation is 88% and 97% at similar thicknesses in sample S2. However, at the end of 2nd GaN in sample S2, the residual compressive stress in 2nd GaN is almost relaxed with a relaxation value of 99.4%. Thus, AlGa_N/AlN/GaN SMLs in sample S2 have resulted in more relaxation of 2nd GaN compared to AlN/GaN SMLs in sample S1. This behavior is opposite to the predicted lower relaxation of 2nd GaN with AlGa_N/AlN/GaN SMLs.

The stress thickness-product values in Fig. 5.2 show high compressive nature of heterostructures of samples S1 and S2. This indicates that the mean stress (stress-thickness product/thickness) is still compressive in nature. However, this is in contradiction to the incremental stresses, which show relaxation of 2nd GaN in both the samples S1 and S2. Ideally, the relaxed 2nd GaN should result in reduced or no net compressive mean stress. However, relaxation of 2nd GaN with higher mean compressive stress signifies the stress mitigation property of these two heterostructures in mitigating the stress from 2nd GaN to Si. Based on the stress-thickness product, the mean stress of the overall heterostructure is higher in sample S2 compared to S1. The higher mean compressive stress in the heterostructure of sample S2 along with its higher relaxation of 2nd GaN indicates that the stress mitigation property is enhanced in the heterostructure grown using AlGa_N/AlN/GaN SMLs.

The high compressive mean stress also result in high convex bowing during the growth. Based on the wafer curvature (stress-thickness product), the wafer bow during the growth is estimated as follows. High tensile stress during 2nd AlN growth resulted in a high concave bow of -75 μm . Growth of AlGa_N epilayer compensated the concave bow and resulted in a convex bow of +105 μm . Thus, during 250 nm AlGa_N layer, a total convex bow of +180 μm was generated. During the subsequent growth of 900 nm 2nd GaN layer, a total convex bow of +95 μm is generated, which resulted in an end wafer compressive_bow of +200 μm .

5.1.2 Ex-situ bow measurements

Ex-situ bow measurements obtained on samples S1 and S2 are shown in Figs. 5.3 (a) and (b), respectively. Opposite bowing was observed with a concave bow (tensile) of -35 μm and a convex bow (compressive) of +82 μm for samples S1 and S2, respectively.

Fig. 5.3 Ex-situ bow of samples S1 (a) and S2 (b) on 100-mm Si substrate.

The high compressive mean stress in sample S2, which resulted in a convex bow of +200 μm at the growth temperature might have compensated the thermal stress during cool down and resulted in a convex bow of +82 μm . As discussed in Section 5.1.1, the 2nd GaN is more relaxed in sample S2 compared to S1. However, convex ex-situ bow is observed for samples S2, while it is concave for sample S1. These results further emphasize the stress mitigation property of the heterostructure grown using AlGa_{0.2}N/AlN/GaN SMLs.

5.1.3 Cross sectional TEM analysis

In order to understand the stress relaxation process in the 2nd GaN of samples S1 and S2, cross sectional TEM analysis was done on the two heterostructures, grown using AlN/GaN SMLs and AlGa_{0.2}N/AlN/GaN SMLs and the corresponding images are shown in Figs. 5.4 (a) and (b), respectively. Here, the heterostructures considered for TEM analysis are similar to S1 and S2, but they are not exactly the same. However, since the study concentrates on the relaxation behavior of 2nd GaN, it is important to investigate the different interfaces in the heterostructures grown using AlN/GaN SMLs and AlGa_{0.2}N/AlN/GaN SMLs. Hence the following TEM analysis can be applied to samples S1 and S2.

Fig. 5.4 Bright field cross sectional TEM images of heterostructures grown using AlN/GaN (a) and AlGaIn/AlN/GaN SMLs (b).

Heavy misfit and dislocation looping is observed within 200 nm from the interface of 2nd GaN/2nd AlN in the heterostructure grown using AlN/GaN SMLs as shown in Fig. 5.4(a). But, relatively lower misfit and dislocation looping is observed at the Al_{0.065}Ga_{0.935}N / 2nd AlN interface as revealed in Fig.5.4 (b). The relative lower lattice mismatch of Al_{0.065}Ga_{0.935}N / 2nd AlN interface might be responsible for the lower misfit dislocation generation and looping. This further resulted in lower relaxation rate of Al_{0.065}Ga_{0.935}N epilayer of sample S2 in the in-situ stress measurements.

Bending and looping of dislocations are found to be higher near the interface of 2nd GaN and Al_{0.065}Ga_{0.935}N, as indicated with white arrow marks in Fig. 5.4 (b) for the heterostructure grown using AlGaIn/AlN/GaN SMLs. However, the dislocation bending and looping effect is relatively lower in the 250 to 500 nm of 2nd GaN grown using AlN/GaN SMLs as shown with the arrow marks in Fig. 5.4 (a). Thus, it is evident from these images that the AlGaIn/2nd GaN heterojunction interface grown using AlGaIn/AlN/GaN SMLs provides additional impetus for the dislocations to bend and loop. As shown in Fig. 5.2, the observed increase in the rate of relaxation of residual compressive stress in the thickness range between 250 to 500 nm of sample S2 compared to S1 can be attributed to the bending and looping of dislocations near the AlGaIn and 2nd GaN interface. Moreover, overall lower residual stress and higher relaxation of 2nd GaN in sample S2, grown using AlGaIn/AlN/GaN SMLs can be attributed to the numerous dislocation generation, their bending and looping at two the subsequent interfaces of 2nd AlN/AlGaIn and AlGaIn/2nd GaN.

Even though 2nd GaN is more relaxed, high mean stress and compressive bow of sample S2 grown using AlGa_N/Al_N/Ga_N SMLs indicate the increased stress mitigation from Ga_N to Si substrate. Stress mitigation that was observed in the heterostructure using Al_N/Ga_N SMLs has been attributed to the compression in the 1st Ga_N together with the buried cracks as discussed in the Section 4.4.3 of Chapter 4. However, increased mitigation of the stress from Ga_N to Si in the heterostructure using AlGa_N/Al_N/Ga_N SMLs should be due to the growth of AlGa_N between 2nd Ga_N and 2nd Al_N epilayers. To understand this behavior, AlGa_N/2nd Al_N and 2nd Ga_N/AlGa_N interfaces were studied using TEM in high angle annular dark field (HAADF) mode and weak beam dark field mode (WBDF) taken with $g = 2\bar{1}\bar{1}0$ and $g = 0002$, as shown in Figs. 5.5 (a), (b) and, (c), respectively. Figure 5.5 (d) shows the WBDF cross sectional TEM image of a sample with Al_N/Ga_N SMLs, taken with $g = 0002$ to compare the heterostructure with AlGa_N/Al_N/Ga_N SMLs. Consistent with the bright field TEM image of Fig. 5.4 (b), the generation and looping of dislocations near the AlGa_N / 2nd Al_N and 2nd Ga_N/AlGa_N interfaces are clearly observed in Fig. 5.5 (a) to (c). As discussed, the dislocation looping has been attributed to the compressive stress relaxation of 2nd Ga_N. In addition, the generation of additional dislocations at the AlGa_N and 2nd Ga_N interface is also observed in Figs. 5.5 (a) and (b), which leads to the increased dislocation density in the 2nd Ga_N and decreasing its crystalline quality.

Sharp interfaces are observed in HAADF image and WBDF image with $g = 2\bar{1}\bar{1}0$ as shown in Figs. 5.5 (a) and (b), respectively. However, WBDF image taken with $g = 0002$ in Fig. 5.5 (c) shows that AlGa_N/2nd Al_N interface and 2nd Al_N/1st Ga_N interfaces are not sharp and are bright in contrast. In addition, near the interface at AlGa_N and 2nd Ga_N there exist bright horizontal misfit lines, indicated by arrows in the Fig. 5.5 (c). The heterostructure with Al_N/Ga_N SMLs [Fig. 5.5 (d)] also presents not sharp and bright 2nd Al_N/1st Ga_N interface. However, the interface at 2nd Ga_N/2nd Al_N is relatively sharper and not bright compared to the AlGa_N/2nd Al_N interface in the heterostructure with AlGa_N/Al_N/Ga_N SMLs [Fig 5.5(c)]. Moreover, no additional horizontal misfit lines are observed in the 2nd Ga_N of the heterostructure with Al_N/Ga_N SMLs [Fig. 5.5 (d)]. Bright features in the WBDF images appear only if the extinction condition $g \cdot b \neq 0$ is satisfied, where b is burger's vector. Hence, in this case with $g = 0002$, the direction of b should be along 'c' direction, indicating the tilt in the crystallites. This shows that crystalline tilt is occurring at various interfaces in the heterostructure. The tilt observed at 2nd Al_N/1st Ga_N interface can be attributed to the formation of buried cracks and misfit dislocations, which is common in both the heterostructures with Al_N/Ga_N and

AlGaN/AlN/GaN SMLs. However, the crystalline tilt observed near the 2nd GaN/AlGaN interface and at AlGaN/2nd AlN interfaces might have been additional crystalline defects that originate in the heterostructure growth with AlGaN/AlN/GaN SMLs.

Fig. 5.5 Cross sectional TEM images obtained in HAADF (a) and weak beam dark field modes with $g = 2\bar{1}\bar{1}0$ (b) and $g = 0002$ (c) of a heterostructure, grown using AlGaN/AlN/GaN SMLs. WBDF image with $g = 0002$ for the heterostructure with AlN/GaN SMLS (d).

Based on the observations from TEM analysis, the following explanation can be hypothetically presented to understand the increased stress mitigation property of AlGaN/AlN/GaN SMLs as discussed in Sections 5.1.1 and 5.1.2. In any multilayer heterostructure, the bow of the overall heterostructure is a resultant of equilibrium of forces and moments of individual epilayers [215]. The stress that generates or relaxes in one epilayer causes its effect in the subsequent epilayers and following in the substrate such that the net force is at equilibrium in the entire heterostructure. Thus, for every change in the generated or relaxed stress in an epilayer there is a consequent effect in the substrate to maintain the equilibrium of forces. However, if the generated stress in one layer is partly mitigated by the elastic distortion in the crystalline defect regions at

various interfaces, the net effect of the generated stress is reduced in the subsequent epilayers as well as in the substrate.

In the heterostructures grown using AlN/GaN SMLs, the elastic distortion of the buried cracks [206] together with the compressive nature of 1st GaN has been attributed to its stress mitigation property. In addition to these factors, extra crystalline tilt at 2nd GaN/AlGaIn and AlGaIn/2nd AlN interfaces in the heterostructure using AlGaIn/AlN/GaN SMLs might be undergoing elastic distortion and contributing to the increased stress mitigation from GaN to Si substrate. Thus, even though 2nd GaN is more relaxed with thickness, the stress mitigation property of AlGaIn/AlN/GaN SMLs might be responsible for mitigating the stress to Si and allowing the wafer to have high compressive mean stress and compressive bow during the growth (Fig. 5.2). This higher compressive mean stress might have compensated the tensile stress during cool down and resulted in the compressive bow at room temperature as shown in Fig. 5.3(b) for sample S2, whereas relatively lower stress mitigation property of the AlN/GaN SML leading to lower compressive mean stress at growth temperature might be responsible for the tensile bow of sample S1 at the room temperature as in Fig. 5.3(a).

5.1.4 Crystal quality and surface morphology analysis

Crystal quality of 2nd GaN of both the samples S1 and S2 was investigated by using HR-XRD. HR-XRD rocking curve scans along GaN (0002), (0004) and (0006) planes were used to generate Williamson-Hall plots and obtain the screw type dislocation density, and FWHM of GaN (30 $\bar{3}$ 2) scans were used to estimate the edge type dislocation density in GaN layers. The estimated screw type dislocation density from the XRD rocking curve is 2.09×10^9 and 3.71×10^9 cm⁻² for the 2nd GaN of samples S1 and S2, respectively. Whereas the edge type dislocation density is 3.89×10^{10} and 6.08×10^{10} cm⁻² in the 2nd GaN of samples S1 and S2, respectively. Hence, these measurements indicate that the overall dislocation density is higher in the 2nd GaN of sample S2 grown using AlGaIn/AlN/GaN SMLs compared to S1 using AlN/GaN SMLs. The additional dislocation generation in 2nd GaN near the interface of AlGaIn and 2nd GaN, as observed from the TEM images of Figs. 5.5 (a) to (c) could be the reason for the increased dislocation density.

Surface morphology of samples S1 and S2 was investigated by AFM. AFM images of 10×10 μm^2 scan area of 2nd GaN surface of samples S1 and S2 are shown in Figs. 5.6 (a) and (b), respectively. AFM images were obtained in the amplitude mode.

Fig. 5.6 AFM images of $10 \times 10 \mu\text{m}^2$ scan area of 2nd GaN of samples S1 (a) and S2 (b).

Mound type surface morphology [90] is observed for both the samples S1 and S2. The surface morphology of 2nd GaN of sample S2 shows higher roughening compared to that of sample S1 as shown in Fig. 5.6. The insets in Fig. 5.6 are the zoomed images showing close look of a mound. As shown, sample S2 exhibits columnar feature at the center of the mound unlike the mounds in sample S1. The observation of columnar features on the mounds indicates the kinetic roughening of the growth process in 2nd GaN of sample S2. As the growth conditions of GaN and the underlying epilayers were similar in both the heterostructures, the observed kinetic roughening of the 2nd GaN surface in S2 can be attributed to its higher compressive stress relaxation. Decrease in the residual compressive stress reduces the diffusion of Ga adatoms on the growth surface and results in higher kinetic roughening [90]. Thus, from these studies, it can be concluded that AlGa_xN_{1-x}/AlN/GaN SMLs have been successful in mitigating the stress from 2nd GaN to Si and resulting in the compressive bow of the wafer, however, the surface morphology is found to be rough.

5.2 Effect of V/III ratio on the properties of 2nd GaN grown using AlGa_xN_{1-x}/AlN/GaN SMLs

Heterostructures grown using AlGa_xN_{1-x}/AlN/GaN SMLs have resulted in compressive bow of the wafers. However, the surface morphology was found to be rough. In order to improve the surface morphology, growth parameters of 2nd GaN were tuned. V/III ratio was found to have affected the surface morphology of 2nd GaN, grown using AlN/GaN SMLs as discussed in the Section 4.6 of Chapter 4. Similarly, in order to investigate the

effect of V/III ratio on the surface morphology of 2nd GaN, grown using AlGa_{0.065}N/GaN SMLs, three samples namely S3, S4 and, S5 were grown. V/III ratios were maintained at 1020, 2070 and, 4050 for the 2nd GaN of samples S3, S4 and, S5, respectively. The variation in the V/III ratio of 2nd GaN was obtained by keeping the ammonia flow rate at 1000 SCCM and changing the Ga flux only. Moreover, V/III ratios of 939, 1890 and, 3712 were maintained for the growth of Al_{0.065}Ga_{0.935}N epilayer in samples S3, S4 and, S5. In general, the discussion about V/III ratio in the following sections would represent both 2nd GaN and Al_{0.065}Ga_{0.935}N V/III ratios. Further, it should be noted that the growth conditions for the underlying structure were kept constant in all the samples as that of heterostructure grown using AlN/GaN SMLs. Thickness of 2nd GaN was maintained between 1.15 to 1.35 μm while keeping the Al_{0.065}Ga_{0.935}N thickness at ~ 250 nm in all the samples.

5.2.1 Surface morphology

Figures 5.7 (a), (b) and, (c) show the surface morphology of samples S3, S4 and S5, respectively, obtained using the AFM amplitude mode. Figure 5.7 (d) shows the RMS roughness obtained from $20 \times 20 \mu\text{m}^2$ scan area as a function of V/III ratio. As shown in the figure, a systematic decrease in the surface roughness is observed with increase in the V/III ratio from 1020 to 4050. The lowest roughness of 4.8 nm is obtained for sample S5, grown with a V/III ratio of 4050. As the residual stress in the 2nd GaN plays a major role in the evolution of surface morphology of GaN, in-situ curvature measurements of 2nd GaN of samples S3 to S5 are further analyzed.

Fig. 5.7 AFM images obtained in the amplitude mode showing surface morphology of 2nd GaN of samples S3 (a) S4 (b) and, S5 (c). RMS roughness as a function of 2nd GaN V/III ratio (d).

5.2.2 In-situ stress measurements

Figure 5.8 shows the in-situ measured stress-thickness product and incremental stress as a function of thickness of 2nd GaN (including AlGaIn) of samples S3, S4, and, S5. As shown in the figure, all the samples show similar stress-thickness products and incremental stresses below a thickness of 100 nm. This indicates that the rate of relaxation is similar up to a thickness of 100 nm. However, at a thickness of 250 nm, that is at the end of AlGaIn epilayer, the stress relaxation is found to be 64.5%, 64% and, 57% for the samples S3, S4 and S5, respectively. The rate of relaxation is continued to be lower for sample S5 compared to that of samples S3 and S4 throughout the growth of 2nd GaN. At the end of 2nd GaN, samples S3 and S4 are found to have shown tensile nature with final incremental stresses of +10 and +100 MPa, respectively. However, sample S5 shows compressive stress with a value of -434 MPa. Thus, from these studies, it can be clearly seen that the rate of relaxation is almost similar in samples S3 and S4 except for sample S5 that shows lower rate of relaxation.

Fig. 5.8 Stress $\times$ thickness and incremental stress as a function of thickness of samples S3, S4 and, S5.

5.2.3 Raman measurements

Residual stress in 2nd GaN was measured by Raman spectroscopy at the room temperature for samples S3 to S5. Figure 5.9 shows the residual stress as a function of V/III ratio of 2nd GaN.

Fig. 5.9 Stress in 2nd GaN as a function 2nd GaN V/III ratio, obtained from Raman measurements on samples S3, S4 and, S5.

Higher tensile stress is observed in 2nd GaN of sample S3 compared to sample S4. This trend is contradicting with the in-situ stress measurements, where the tensile stress in sample S4 is found to be higher compared to sample S3. Raman measurement on sample S5 shows compressive stress in 2nd GaN. These results suggest that for the

heterostructures grown using AlGaIn/AlN/GaN SMLs, compressive residual stress in 2nd GaN is achieved only when the V/III ratio > 3300.

5.2.4 Ex-situ bow measurements

Ex-situ bow measurements were performed on samples S3 to S5. The bow as a function of 2nd GaN V/III ratio is shown in Fig. 5.10. All the samples showed convex bowing, indicating compressive natured bow.

Fig. 5.10 Ex-situ bow as a function of of 2nd GaN V/III ratio.

Even though the in-situ stress measurements and Raman measurements show tensile nature of 2nd GaN of samples S3 and S4, the obtained convex bow of these samples further demonstrates the stress mitigation property of AlGaIn/AlN/GaN SMLs. Unlike the in-situ stress measurements, where sample S4 showed higher tensile stress compared to sample S3, Raman and ex-situ bow measurements indicate a systematic decrease in the residual stress in the 2nd GaN and increase in the convex bow from sample S3 to S5. This contradiction between the results obtained from in-situ and ex-situ measurements for samples S3 and S4 suggest that the stress mitigation during cool down might not be similar in all the heterostructures.

5.2.5 HR-XRD measurements

HR-XRD measurements were performed to investigate the crystal quality and dislocation density in 2nd GaN of samples S3 to S5. HR-XRD rocking curve scans along GaN (0002), (0004) and (0006) planes were used to generate Williamson-Hall plots to obtain the screw type dislocation density, while FWHM of GaN obtained from scans along (30 $\bar{3}$ 2) were used to estimate the edge type dislocation density in GaN layers. Table 5.1 lists the screw and edge type of TDD. Almost similar density of screw type TDD

was observed in 2nd GaN for all the samples. However, the edge type TDD was observed to be the highest in sample S4 and is lowest in sample S5. Thus, no trend in the TDD is observed with the V/III ratio of 2nd GaN. However, by comparing the edge type TDD with the final incremental stresses in the 2nd GaN (listed in Table 5.1), a trend of reduced final incremental stress with increased dislocation density is observed. Thus, higher V/III ratio has resulted in both lower dislocation density and higher residual compressive stress of 2nd GaN.

Table 5.1 Crystal quality and final incremental stresses in the 2nd GaN estimated from HR-XRD rocking scans and in-situ measurements, respectively, for samples S3, S4 and S5.

Sample	TDD in 2 nd GaN ($\times 10^{10} \text{ cm}^{-2}$)		Final incremental stress in 2 nd GaN* (MPa)
	Screw type	Edge type	
S3	0.36	6.3	+10
S4	0.37	8.5	+100
S5	0.34	5.4	-434

* '+' and '-' indicate tensile and compressive stresses, respectively

5.2.6 Cross sectional TEM

In order to understand the observed relation between the stress relaxation in the 2nd GaN and dislocations, cross-sectional TEM measurements were performed on samples S3 and S5. Since the V/III ratio of sample S4 is intermediate between samples S3 and S5 and it showed almost similar relaxation behavior as that of sample S3 in the in-situ stress measurements, sample S4 was not considered for the TEM analysis. Figure 5.11 (a) and (b) show the cross sectional weak beam dark field (WBDF) TEM images of samples S3 and S5, respectively. Generation of new dislocations is indicated by white arrow marks and the looping of dislocations is indicated by white inverted triangular-shape mark in the TEM images of Fig. 5.11. As shown in the figure, generation of new dislocations as well as the bending and looping of dislocations is observed to be higher in the 2nd GaN of sample S3 compared to sample S5. Higher dislocation generation in the sample with lower V/III ratio (S3) increased the overall dislocation density as observed in the HR-XRD measurements. Moreover, increased looping of dislocations might have resulted in the enhanced compressive stress relaxation in the 2nd GaN of sample S3

compared to sample S5. Hence, higher relaxation rate is observed of 2nd GaN of sample S3 in the in-situ stress measurements.

Fig. 5.11 Cross sectional TEM images obtained in the weak beam dark field mode with $g = 2\bar{1}10$ of samples S3 (a) and S5 (b).

5.2.7 Surface morphology evolution with V/III ratio of 2nd GaN

In order to understand the observed V/III ratio effect on the surface morphology of samples S3 to S5 (Fig. 5.7), following explanation has been given. Typical growth process in the ammonia rich growth regime of ammonia-MBE proceeds through the absorption of Ga adatoms on the growth surface followed by their diffusion to the step edges and incorporation into the lattice [185]. Ga sticking coefficient can be assumed to be close to unity at a growth temperature of ≤ 800 °C [185]. This assumption is particularly applicable in the ammonia rich growth regime. Moreover, diffusion of Ga adatoms on the growth surface is dependent on temperature [199], ammonia over pressure and residual compressive stress [200, 201]. At a constant growth temperature and ammonia flow, the relaxation of residual compressive stress in GaN increases with the increase of epilayer thickness. This results in the reduction of diffusion of Ga adatoms to the step edges. Reduced diffusion leads to the formation of fresh nucleation sites on the mound morphology and results in the increased kinetic roughening. Hence, the increased kinetic roughening of the surface morphology of the 2nd GaN, grown using AlGaIn/AlN/GaN SMLs compared to AlN/GaN SMLs (Fig. 5.6) has been attributed to the enhanced relaxation of its compressive stress. Similarly, the lower stress relaxation rate and higher residual compression of 2nd GaN in sample S5, as observed from the in-situ measurements (Fig. 5.8), might be the main reason for its smooth surface morphology as seen in Fig. 5.7 (c) compared to samples S3 and S4.

However, between samples S3 and S4, the increased roughness in sample S3 compared to S4 cannot be explained solely on the basis of residual stress relaxation. As shown in the Fig. 5.8, the relaxation is observed to be similar in the 2nd GaN of both the samples S3 and S4 up to 900 nm of its thickness, beyond which sample S4 was found to have relaxed more. These results indicate that in addition to residual compression, adsorption and incorporation of 'Ga' on the growth surface might also be responsible for the increased roughening of sample S3. Similarly, the increased roughening of the surface morphology for the samples with lower V/III ratio in the heterostructures grown using AlN/GaN SMLs was also observed in Section. 4.6 of Chapter 4.

The roughening of the surface with the decreased V/III ratio can be explained based on the kinetic behavior of the growth process, where the competition among rate of adsorption, diffusion and incorporation of Ga adatoms play a major role, considering that the desorption is negligible in the ammonia rich growth environment. Under the constant ammonia flow, when the residual compressive stress is lower, the diffusion of Ga adatoms to the step edges reduces. This leads to the increase in the time of replenishment of the step edges leading to lower incorporation of Ga and N atoms at step edges. When the Ga flux is lower, the adsorbed adatom may have sufficient time to go to the step edge and incorporate into the lattice before the next Ga adatom sticks to the surface. However, when the Ga flux is higher under similar diffusion conditions, all the adsorbed Ga adatoms may not have sufficient time to incorporate into the step edges. This leads to an increase in the nucleation sites on the mound morphology, leading to the roughening of the surface. Thus, the increased roughening of the surface morphology in sample S3 compared to sample S4 can be attributed to the increased rate of adsorption of Ga adatoms compared to their rate of diffusion and incorporation at the step edges. From this understanding, it can also be seen that with the increased residual compression, smoother surface morphologies can be achieved at higher Ga flux and lower V/III ratios. This is possibly the reason for the smoother surface morphologies achieved for the 2nd GaN, grown with relatively higher V/III ratio of 2070 using AlN/GaN SMLs, while a high V/III ratio of 4050 is required in the case of AlGaIn/AlN/GaN SMLs. With this, both residual compression and V/III ratio were found to play a major role in the surface morphology evolution of GaN in ammonia-MBE growth process. Overall, the smooth surface morphology with reduced dislocation density is achieved for the 2nd GaN grown using AlGaIn/AlN/GaN SMLs with a V/III ratio of 4050, which enhances the 2DEG characteristics of AlGaIn/GaN HEMT heterostructures.

5.3 AlGa_N/Ga_N HEMT heterostructures grown using AlGa_N/Al_N/Ga_N SMLs

AlGa_N/Ga_N HEMT heterostructures were grown using AlGa_N/Al_N/Ga_N SMLs on 100-mm Si substrate with the optimized 2nd Ga_N V/III ratio of 4050. HR-XRD ω -2 θ scan of the AlGa_N/Ga_N HEMT heterostructure along with the dynamic simulation is presented in Fig.5.12.

Fig. 5.12 High resolution X-ray diffraction ω -2 θ scan of AlGa_N/Ga_N HEMT heterostructure grown using AlGa_N/Al_N/Ga_N SMLs.

The dynamic simulation shows that the composition of Al in AlGa_N barrier is 25.5% with a thickness of 22 nm. Pendullösung fringes in the ω -2 θ scan indicate structural perfection and abrupt interface of the grown AlGa_N/Ga_N HEMT heterostructure along the c-direction. Figure 5.13 shows the reciprocal space mapping (RSM) measurement data of HEMT heterostructure along Ga_N (10 $\bar{1}$ 5) plane. Bottom Al_{0.065}Ga_{0.835}N epilayer is also observed in the RSM mapping and is indicated with an arrow mark. Coherent growth of AlGa_N barrier with Ga_N is clearly observed in the grown HEMT heterostructure.

Fig. 5.13 Reciprocal space mapping of AlGaN/GaN HEMT heterostructure along GaN ($10\bar{1}5$) direction.

Capacitance-voltage measurement performed on the as-grown AlGaN/GaN HEMT heterostructures using mercury probe measurement system is shown in Fig. 5.14. A plateau of capacitance followed by its pinch off represents the presence of 2DEG at AlGaN/GaN interface. Integration of capacitance-voltage curve from zero bias to pinch off gives a 2DEG carrier concentration of $0.75 \times 10^{13} \text{ cm}^{-2}$. Depth profile of carrier concentration obtained using C-V measurements is also presented in Fig. 5.15. The depth of 2DEG gives the combined thickness of barrier and GaN cap, which is 23 nm. The background carrier concentration in the GaN buffer layer is around 10^{15} cm^{-3} .

Fig. 5.14 Capacitance-voltage measurement of AlGaIn/GaN HEMT heterostructures, grown using AlGaIn/AlN/GaN SMLs. Sheet carrier concentration obtained by integration of C-V profile is also presented.

Fig. 5.15 Depth profile of carrier concentration in the AlGaIn/GaN HEMT heterostructure obtained from C-V measurements.

Hall measurements performed in Van der Pauw configuration on AlGaIn/GaN HEMT heterostructure grown using AlGaIn/AlN/GaN SMLs showed a carrier concentration of $0.88 \times 10^{13} \text{ cm}^{-2}$ and a mobility of $1380 \text{ cm}^2/\text{V.s}$. The sheet resistance obtained was $517 \Omega/\text{sq}$. Fig. 5.16 shows the sheet carrier concentration and mobility as a function of temperature obtained from Hall measurement.

Fig. 5.16 Mobility and sheet carrier density as a function of temperature for AlGaIn/GaN HEMT heterostructures.

The insensitivity of carrier concentration with temperature and improvement in the mobility confirm the presence of 2DEG. The higher mobility of 5530 cm²/V.s at 90K indicates the reduced phonon scattering. The obtained value of 2DEG mobility is comparatively higher than that for the heterostructure grown using AlN/GaN SMLs. The higher mobility can be attributed to the decreased alloy scattering due to lower Al% in the barrier. However, the improved mobility was obtained at the expense of decreased carrier concentration leading to increased sheet resistance.

The uniformity in electrical characteristics of AlGaIn/GaN HEMT heterostructures across 100-mm Si substrate were measured by a nondestructive mobility measurement method. Figs. 5.17 (a), (b) and (c) show the mapping of the sheet resistance, 2DEG mobility and 2DEG sheet carrier concentration, respectively. Reasonably good uniformity was observed for the HEMT heterostructures with the standard deviation of 7%, 1% and, 8% for the sheet resistance, carrier concentration and mobility, respectively, across the 100-mm substrate. Moreover, for all the HEMT samples studied using AlGaIn/AlN/GaN SMLs with 2nd GaN thickness of 1400 nm, a convex bow of <+34 μm was achieved. Thus, AlGaIn/GaN HEMT heterostructures with good 2DEG properties have been achieved on 100-mm Si(111) substrate using AlGaIn/AlN/GaN SMLs with relatively lower wafer bow values.

(a)

Sheet resistance 443 (ohm/sq,
STD Dev.: 7.11%

(b)

Mobility 1409 cm²/V.s,
STD Dev.: 8.0%

(c)

Sheet carrier density
 $1.0 \times 10^{13} \text{ cm}^{-2}$,
STD Dev.: 1.35%

Fig. 5.17 2DEG mapping of sheet resistance (a), mobility (b) and sheet carrier concentration (c) of AlGaIn /GaN HEMT heterostructures on 100-mm Si substrate.

5.4 Summary

An AlGa_N layer was added to the AlN/GaN SMLs in order to reduce the tensile bowing of AlGa_N/Ga_N HEMT heterostructures on 100-mm Si substrates. The in-situ curvature measurements showed that the new AlGa_N/AlN/GaN SMLs resulted in higher relaxation of 2nd Ga_N while the overall heterostructure showed higher compressive mean stress. It resulted in an overall compressive bow of +82 μm for a 2nd Ga_N thickness of 900 nm. Cross sectional TEM measurements revealed that the bending and looping of dislocations at 2nd AlN/AlGa_N and AlGa_N/2nd Ga_N interfaces is the reason for the increased relaxation. Moreover, WBDF TEM images suggested that the elastic distortion due to additional tilt at the 2nd Ga_N/AlGa_N and AlGa_N/2nd AlN interfaces could have caused the increased stress mitigation leading to compressive bow. AlGa_N/AlN/GaN SMLs were also resulted in an increased RMS roughness of 8.4 nm for a scan area of $10 \times 10 \mu\text{m}^2$, while it was only 3.8 nm for the 2nd Ga_N grown using AlN/GaN SMLs. Increased V/III ratio of 2nd Ga_N from 2070 to 4050 resulted in an improved RMS roughness of 4.1nm for a Ga_N of thickness 1150 nm. The ex-situ wafer bow was reduced to $\leq +35 \mu\text{m}$ by increasing the 2nd Ga_N thickness up to 1400 nm. AlGa_N/Ga_N HEMT heterostructures grown using AlGa_N/AlN/GaN SMLs showed a room temperature sheet resistance of 517 $\Omega/\text{sq.}$, mobility of 1380 $\text{cm}^2/\text{V.s}$ and a carrier concentration of $0.88 \times 10^{13} \text{ cm}^{-2}$ with their respective standard deviations of 7%, 8% and 1%, respectively, across the 100-mm substrate.

6. Carbon doped GaN buffer

AlGaIn/GaN HEMT heterostructures with good 2DEG characteristics have been demonstrated on 100-mm Si substrate using both AlN/GaN and AlGaIn/AlN/GaN SMLs as discussed in chapters 4 and 5. However, high resistive GaN buffers for GaN based HEMT heterostructures are extremely important to achieve good device isolation and improved RF performances. Crystalline defects such as nitrogen vacancies, threading dislocations and the incorporation of unintentional impurities such as silicon and oxygen causes higher back ground carrier concentration in GaN buffers [123, 216, 217]. Thus, the optimization of the growth is necessary to achieve high resistive GaN buffers with low leakage current for HEMT applications.

6.1 Buffer leakage current measurements

In order to measure the leakage currents in the buffer of grown HEMT heterostructures, ohmic contacts (Ti/Al/Ni/Au) in the form of a geometric square shape with an edge dimension of 55 μm have been fabricated. Buffer leakage current (BLC) measurements were obtained in two ways. They are horizontal leakage current (HLC) and vertical leakage current (VLC) measurements. HLC was obtained on the grown HEMT heterostructures by dry etching the AlGaIn barrier and the GaN channel between the two ohmic contacts that are separated by 5 μm distance. An etch depth of 100 nm into the epilayer structure is considered to measure the HLC. VLC measurements were performed on the AlGaIn/GaN HEMT heterostructures by measuring the current conduction between the ohmic contact and the bottom of the substrate. Measurement set up for the HLC and VLC measurements is shown in Fig. 6.1. The measured HLC and VLC of a typical AlGaIn/GaN HEMT heterostructure are shown in Fig. 6.2. For various HEMT heterostructures, grown using AlN/GaN and AlGaIn/AlN/GaN SMLs, an average HLC value of $\sim 1 \times 10^{-3}$ A/mm and a VLC value of $\sim 1 \times 10^{-1}$ A/cm² were obtained at 20V. Increase in the buffer resistance will further improve the performance of the AlGaIn/GaN HEMT heterostructures. In order to achieve that, identification, compensation and reduction of impurity sources and parasitic conduction paths in the heterostructures are necessary.

Fig. 6.1 A schematic of measurement set up for the HLC and VLC measurements on AlGaN/GaN HEMT heterostructures.

Fig. 6.2 HLC and VLC as a function of voltage in the AlGaN/GaN HEMT heterostructures.

6.2 Sources of current conduction in GaN buffer

Nitrogen vacancy has been widely attributed to the unintentional n-type nature of GaN based materials [123, 218, 219]. In ammonia-MBE growth process, the deep level concentration of nitrogen vacancy related states were found to respond strongly with

the ammonia flow and it decreases almost six fold from 1.5×10^{14} to $2.7 \times 10^{13} \text{ cm}^{-3}$ by increasing the beam equivalent pressure (BEP) ratio (ammonia BEP/ gallium BEP) from 1000 to 1850 [220]. With a BEP ratio of 2070 in the current growth conditions (growth rate of $0.28 \text{ }\mu\text{m/h}$ at a high ammonia flow of 1000 SCCM with a BEP of $6.88 \times 10^{-4} \text{ Torr}$) of GaN, the probability of formation of high density of nitrogen vacancies and their consequent donor impurity states can be considered as relatively minimal in the as-grown AlGaIn/GaN HEMT heterostructures. Thus, it is necessary to investigate the other possible dominant donor impurities, which are responsible for the leakage current conduction in GaN buffers.

6.2.1 Secondary ion mass spectroscopy

Concentrations of other unintentional impurities such as silicon, oxygen and, carbon in the AlGaIn/GaN HEMT heterostructures were determined using SIMS, operating in dynamic mode with magnetic sector. A SIMS atomic concentration profiles of Si, O and, C as a function of depth for an AlGaIn/GaN HEMT heterostructure grown using AlN/GaN SMLs is shown in Fig. 6.3. It should be noted that the concentration of elements in silicon is not calibrated.

Fig. 6.3 SIMS atomic concentration profile as a function of depth for an AlGaIn/GaN HEMT heterostructure grown using AlN/GaN SMLs.

The concentration of silicon is found to be higher in the 1st AlN and it drastically decreases to the SIMS detection limit in the subsequent epilayers. It further confirms

that the diffusion of silicon from the substrate to the epitaxial layers is minimum in the established growth process. Oxygen concentration is lower compared to silicon at the interface of 1st AlN and silicon substrate. However, the concentration of oxygen is relatively higher for the subsequent layers. Moreover, oxygen peaks are seen at heterostructure interfaces such as 2nd GaN / 2nd AlN, 2nd AlN / 1st GaN and, 1st AlN / 1st GaN. Carbon concentration is found to be lower compared to oxygen in all epitaxial layers and interfaces except in the major portion of 2nd GaN layer, where it is almost comparable. These results suggest that with its overall higher concentration levels, oxygen might possibly be acting as the dominant donor impurity in the AlGaIn/GaN HEMT heterostructures. Further, oxygen peaks at various interfaces might also cause parasitic conduction paths in the AlGaIn/GaN HEMT heterostructures.

6.2.2 Photoluminescence measurements

Photoluminescence measurements were performed on as-grown AlGaIn/GaN HEMT heterostructures using He-Cd laser source with a wavelength of 325 nm to investigate the active impurity states in GaN buffer. Figure 6.4 (a) shows the typical room temperature PL spectrum obtained on as-grown AlGaIn/GaN HEMT heterostructures. Significant band edge (BE) emission at 3.43 eV, consistent with the band gap of GaN at room temperature [221] was observed. Moreover, the absence of yellow luminescence emission (YL) indicates the good structural quality of grown GaN buffer. PL measurements were also performed as a function of measurement temperature and are presented in Fig. 6.4 (b).

As shown in the Fig. 6.4 (b), the peak at 3.473 eV in the 10K measurement can be attributed to the free exciton (X_A) emission. This is based on the fact that the corresponding peak is present in the spectra at all the measurement temperatures. Similarly, the peak at 3.493 eV can be attributed to the free exciton emission, X_C . However, the presence of the peak at 3.466 eV in only lower measurement temperatures (≤ 150 K) indicates that this peak corresponds to donor bound exciton emission (I_2). Further, the peak at 3.418 eV corresponds to the first phonon replica of X_A and its presence confirms good structural quality of grown GaN buffer. The peak position of X_A exciton at 10K (3.473 eV) with respect to the standard X_A of strain free GaN (3.47 eV) [222] indicates the presence of negligible tensile strain of 0.0004 in the studied GaN buffer. Raman measurements performed on the same sample showed a tensile strain value of 0.0005, suggesting a good match between the two measurements. A continuous red shift of the X_A peak with increasing measurement temperature indicates the shift in GaN band gap energy with temperature.

Fig. 6.4 Photoluminescence spectra of AlGaIn/GaN HEMT heterostructures obtained at RT (a) and as a function of a measurement temperature (b).

The separation of 7 meV between X_A and I_2 emission peaks in the PL spectrum at 10 K gives the localized energy of the donor bound exciton (E_L). The binding energy of the corresponding donor state (E_D) was obtained as 35 meV by considering the applicability of Haynes rule [223, 224] in GaN ($E_L = \alpha E_D$) and assuming $\alpha = 0.2$ [225]. Binding energies of 30 and 34.5 meV were reported for two donor bound exciton states, obtained using high resolution PL measurements of GaN grown on 6H-SiC substrate [125]. Similarly, PL measurements on HVPE grown GaN have also resulted in two donor bound exciton peaks with their peak positions at 3.4714 and 3.4723 eV [132]. Moreover, PL measurements on MBE grown GaN and Si doped MOCVD grown GaN layers have also revealed donor bound exciton peaks with their peak positions at 3.4714 and 3.4723 eV [132]. From these studies, the former peak was attributed to the oxygen related donor state, while the later was attributed to silicon related donor state. Hence, the oxygen related donor state shows slightly higher localized binding energy compared to silicon state. Thus, the presence of I_2 peak in PL measurements with a binding energy

of 35 meV can be attributed to the oxygen related donor state in the GaN buffer, grown using ammonia-MBE. Relatively higher oxygen atomic concentrations in SIMS profiles along with the presence of the oxygen related donor states in PL measurements indicate that oxygen might be the dominant impurity in GaN. Moreover, oxygen peaks at interfaces might possibly be creating the localized parallel conduction channels at the interfaces of AlGaN/GaN HEMT heterostructure.

6.3 Parallel conductions in AlGaN/GaN HEMT heterostructure

In order to observe the parallel conduction in the grown AlGaN/GaN HEMT heterostructures, an experiment containing sequential dry etching to different etch depths and the subsequent measurement of HLC after each etch step was performed. The parallel conduction studies were conducted on the same sample for which SIMS measurements were obtained. Ohmic contacts were made on the surface of the grown AlGaN/GaN HEMT heterostructures with a contact gap of 5 μm as mentioned in Section 6.1. By considering ohmic contacts as metal masks, reactive ion etching (RIE) was performed on the samples to etch the region between the two contacts. Figure 6.5 shows the epilayer structure that was etched to different etch depths before parallel conduction measurements were performed. For each etch depth, at least 5 ohmic contact pairs were measured to check the consistency of the measurements.

Fig. 6.5 Cross sectional view of AlGaN/GaN HEMT heterostructure, showing different etch depths for parallel conduction measurements.

Fig. 6.6 shows the HLC obtained as a function of etch depth in the AlGaN/GaN HEMT heterostructure. As shown in the figure, at an etch depth of 130 nm, the HLC is 5.7×10^{-4} A/mm and it represents the HLC of the entire structure. No significant decrease in the HLC is observed with an increase in the etch depth up to 230 nm. At an etch depth of 460 nm that is approximately $\frac{2}{3}$ rd of the 2nd GaN thickness, the HLC is reduced by an

order. However, the HLC is reduced drastically by three orders of magnitude at an etch depth of 640 nm. Further, etching of the entire 2nd GaN and up to a thickness of 20 nm of the 2nd AlN results in the lowest HLC of 4.3×10^{-8} A/mm. Hence, from these measurements, it can be inferred that the top 500 nm thick GaN is lesser conducting compared to the bottom 250 nm of the 750 nm thick GaN buffer. This further suggests that a highly conducting parallel conduction channel lies in the bottom 250 nm of 2nd GaN layer and is majorly responsible for the observed HLC of the grown AlGaIn/GaN HEMT heterostructures. The one order reduction in the BLC during the initial 460 nm of 2nd GaN is due to the removal of comparatively higher resistive material. Further etching of the entire 2nd AlN layer and top few nm of 1st GaN layer has surprisingly resulted in an increase in the HLC. Nevertheless, HLC is reduced by an order of magnitude upon etching of the grown epitaxial layers up to silicon substrate.

Fig. 6.6 HLC measurements as a function of etch depth in the AlGaIn/GaN HEMT heterostructures.

In a similar study, electron concentration as a function of etch depth was obtained using Hall measurements for GaN epilayers grown on sapphire substrate [136]. An increase in the electron concentration with consequent enhancement of the parallel conduction was observed near the interface of GaN and the sapphire. The increase in the electron concentration was attributed to the dislocation looping near the interface that provided easy lateral diffusion paths for the oxygen from the substrate and caused parallel conduction. Likewise, as shown in Fig. 6.7, majority of dislocations are found to loop in

the initial 200 nm of 2nd GaN in a typical AlGaIn/GaN HEMT heterostructure. The looping of dislocations might have provided easy diffusion paths, leading to the segregation of the oxygen near the 2nd GaN/2nd AlN interface. A sharp peak of oxygen observed in the SIMS measurements (Fig. 6.3) may be attributed to this phenomenon and could be the major reason for the observed parallel conduction. However, the origin of the oxygen that accumulates at the interface is not clear. Observation of increase in the HLC with increase in the growth time of GaN further suggests that the residual oxygen from the growth chamber is likely the source of impurity. Further, inherent doping action of the looped dislocation [136] might also possibly be contributing to the observed parallel conduction. Thus, the localized high oxygen impurity incorporation or the formation of native defects due to dislocation looping near the interface might have led to the formation of high density donor impurity states, resulting in parallel conduction paths at the interfaces.

Fig. 6.7 Cross sectional TEM image of AlGaIn/GaN HEMT heterostructure.

The observed increase in the HLC after the complete etching of 2nd AlN and top few layers of 1st GaN is contrary to the expected trend. From the SIMS measurements (Fig. 6.3), the presence of high oxygen concentration in this layer indicates higher impurity levels. Thus, etching of few layers of 1st GaN should show further reduction rather than increase in the HLC. This anomaly could be due to the presence of trap states at the interface of 1st GaN and the 2nd AlN. As shown in Fig. 6.7, voids are present at the interface of 2nd AlN and 1st GaN. These voids associate with the formation of misfit dislocations near the interface in the 1st GaN [204]. The formed dislocations might have acted as trap centers of electrons and resulted in lower HLC. Thus, etching of a few layers of 1st GaN near the interface might have removed these trap centers and led the

observed increase in HLC. However, further analysis is required to confirm the exact source of trap centers at this interface. Overall, the parallel conduction at the interface of 2nd GaN and 2nd AlN was found to be the major conduction channel that is responsible for the observed HLC in the grown AlGaIn/GaN HEMT heterostructures. Thus, in order to reduce the BLC, it is required to reduce the impurity incorporation or compensate them to increase the resistance of the GaN buffer.

6.4 Carbon doping

Carbon tetrabromide (CBr₄) source was used as carbon source to decrease the buffer leakage current in AlGaIn/GaN HEMT heterostructures. The CBr₄ source was successfully used for the carbon doping in PA-MBE grown GaN buffer and achieved higher buffer resistance for AlGaIn/GaN HEMT heterostructures [42, 69]. Five samples, S1 to S5 were grown in order to study the effect of CBr₄ doping in ammonia- MBE grown GaN buffer. As listed in Table 6.1, samples S1 to S3 were grown using AlN/GaN SMLs, while samples S4 and S5 were grown using AlGaIn/AlN/GaN SMLs. The thickness of 2nd GaN was kept constant at 1 μm for all the samples. Samples S1 and S4 were undoped while the rest of the samples were doped with carbon. The samples were carbon doped from 1st GaN all the way to the 2nd GaN except the top GaN channel region and the AlGaIn barrier. The thickness of undoped GaN channel was kept at 200 nm for samples S2 and S3, while its thickness was kept at only 40 nm for the sample S5. Table 6.1 also lists the CBr₄ BEP used for the doping in GaN buffer.

Table 6.1. List of samples with their SMLs used and the corresponding CBr₄ BEP.

Sample	SMLs	CBr ₄ BEP ($\times 10^{-8}$ mTorr)
S1	AlN/GaN	0
S2		0.51
S3		6.2
S4	AlGaIn/AlN/GaN	0
S5		18.6

6.4.1 CBr₄ doping- HLC and VLC measurements

Ohmic contacts were fabricated as shown in Fig. 6.1 to measure the leakage current in samples S1 to S5. The space between the contacts was dry etched up to a depth of 100 nm to measure the HLC, whereas, VLC measurements were performed by measuring the current between the contact and the substrate. Figure 6.8 (a) shows the measured HLC on samples S1 to S5 as a function of CBr₄ BEP. As shown, the undoped samples, S1 and S4 show HLC values of $\sim 1 \times 10^{-3}$ A/mm and $\sim 2 \times 10^{-3}$ A/mm, respectively. The HLC is

found to have almost unchanged for the sample S2 doped with the lowest CBr_4 pressure. However, the HLC is decreased by 5 times for the sample S3, doped with intermediate value of CBr_4 BEP. The HLC of sample S5, doped with highest CBr_4 BEP that is available with the current system is found to have reduced by two orders in magnitude when compared to undoped sample, S4. VLC measurements on samples S2 and S3 didn't result in any significant improvement compared to sample S1. However, measurements on higher carbon doped sample, S5 (Fig. 6.8(b)) shows one order reduction in the VLC compared to undoped sample, S4.

Fig. 6.8 HLC measurements as a function of CBr_4 pressure obtained from samples S1 to S5 (a). VLC measurements as a function of voltage obtained on an undoped sample - S4 and the highest carbon doped sample - S5 (b).

Thus, carbon doping with the lower CBr_4 BEP ($\leq 6.2 \times 10^{-8}$ mTorr) has resulted in a very minimal change in the HLC and almost no change in the VLC. However, significant change in the HLC and VLC values were observed for the GaN buffer of sample (S5) doped with the highest possible CBr_4 BEP of 1.86×10^{-7} mTorr. Ramdani et al. [152] have performed carbon doping in nitrogen rich ammonia-MBE grown GaN heterostructures on silicon substrate using saddle field methane cracker. Their reported VLC values are comparable to the present observations. On the other hand, semi insulating GaN buffer using ammonia-MBE was achieved using carbon doping with similar cracked methane source [153, 226]. However, their experimental growth conditions involve lower ammonia flows, which resulted in a three dimensional growth mode with rough surface morphology [89]. This signifies that the ammonia-rich growth environment inhibits the incorporation and possibly the activation of carbon in GaN. Theoretical studies performed by Wright et al. [144] also suggest that the formation energy for C_N is higher in N-rich growth conditions.

6.4.2 Structural properties of carbon doped GaN buffer

6.4.2.1 Crystal quality analysis by HR-XRD

The crystal quality of GaN buffer doped with carbon was studied using HR-XRD technique. Fig. 6.9 (a) shows the Williamson-Hall (W-H) plot of GaN layers of samples S1 and S3. As shown in the figure, GaN buffer with carbon doping (S3) shows higher slope in the W-H plot compared to the undoped GaN buffer of sample S1. Similar trend was observed for the samples S4 and S5, where sample S5 shows higher slope in the W-H plot (Fig. 6.9 (b)).

Fig. 6.9 Williamson-Hall plots of GaN buffers of samples S1 & S3 (a) and S4 & S5 (b). Insets show the respective rocking scans along GaN (3032) plane.

The slope of W-H plot along c direction indicates the tilt in the lattice [176]. Hence, the samples doped with carbon were found to have more tilt in the crystallites compared to undoped samples. The estimated screw type dislocation density obtained from the tilt is

1.75×10^9 and $3.38 \times 10^9 \text{ cm}^{-2}$ for the undoped samples S1 and S4, respectively. The increased tilt for the carbon doped samples, S3 and S5 has increased their dislocation density by 35% when compared to their respective undoped samples. Insets of Figs. 6.9 (a) and (b) show the XRD rocking curve scans along GaN (30 $\bar{3}$ 2) plane for both the undoped and carbon doped samples. The FWHM of XRD rocking curve along GaN (30 $\bar{3}$ 2) plane can be considered as the twist in the GaN crystallite [227]. Near overlapping of GaN (30 $\bar{3}$ 2) peaks of samples S1 & S3 and S4 & S5 indicate that the change in the crystalline twist is considerably lower after carbon doping for both the structures grown using AlN/GaN SMLs and AlGaIn/GaN SMLs. Moreover, the edge component of dislocation density was estimated as 3.66×10^{10} and $5.97 \times 10^{10} \text{ cm}^{-2}$ for the GaN buffer of samples S3 and S5, respectively, which is $\leq 10\%$ higher than the undoped samples S1 and S4. Thus, carbon doping using CBr₄ was found to have majorly affected the tilt than the twist in the GaN crystallite.

6.4.2.2 Surface morphology analysis by AFM

Surface morphology of samples S1, S3, S4 and, S5 was investigated by AFM and the corresponding images obtained in the amplitude mode are shown in the Fig. 6.10 (a), (b), (c) and (d), respectively. Carbon doping by CBr₄ did not seem to have much effect on the kinetics of the adatoms on the growth surface as the mound shape and feature size looks similar to the corresponding undoped samples. However, relatively higher pit density was observed in the samples doped with carbon (S3 and S5) compared to the undoped samples (S1 and S4). Similar behaviour of pit formation in the carbon doped samples was also observed by Ramdani et al. [152]. Pits on the growth surface have been attributed to the open core screw type dislocations [228]. Hence, the increased pit density observed in the AFM images of carbon doped samples can be attributed to the increased tilt and the corresponding screw type dislocation density as estimated from the XRD measurements. Thus, carbon doping of GaN using CBr₄ in ammonia-MBE growth process has resulted in increased tilt in GaN crystallites along with the associated pit formation on the surface.

Fig. 6.10 AFM images of $20 \times 20 \mu\text{m}^2$ scan area of samples S1 (a), S3 (b), S4 (c) and S5 (d) obtained using amplitude mode.

6.4.3 Optical properties of carbon doped GaN buffer

Optical properties of carbon doped GaN buffer was investigated by photoluminescence and Raman measurements, which are complementary to the BLC measurements. Undoped (S4) and the highest carbon doped samples (S5) were further investigated in this regard as the intermediate carbon doped samples (S2 and S3) did not show much of a reduction in the buffer leakage current measurements.

6.4.3.1 Photoluminescence measurements

Figure 6.11 shows the photoluminescence spectra obtained at room temperature on samples S4 and S5. As shown in the figure, undoped sample (S4) shows sharp exciton emission at 3.43 eV with no yellow luminescence (YL). However, carbon doped sample (S5) shows YL with decreased BE.

Fig. 6.11 RT photoluminescence spectra of samples S4 and S5.

Similar PL results were reported for the carbon doped GaN, grown using both PA-MBE [42] and ammonia-MBE [94]. The decrease in the intensity of free exciton emission in carbon doped GaN is attributed to the capturing of excitons by carbon impurities [94]. In another way, free electrons in the conduction band of GaN get trapped by the deep impurity states of carbon which results in the reduction of near band edge emission. Further, YL of GaN has been associated with two different types of mechanisms, one is due to the electron transition involving $V_{Ga}-O_N$ complex [229-231] and the other is carbon related impurities [143, 146]. However, YL was also observed for semi insulating GaN, where the formation energy of V_{Ga} is very high [232, 233]. Nevertheless, the reported observations of increased YL with the increased carbon doping attribute carbon more to the YL than $V_{Ga}-O_N$ complex [143, 234]. Experimental studies based on the shift in the YL position with temperature indicated the acceptor energy level of carbon related impurity (C_N) is at 0.86 eV above the valence band. More recently, hybrid functional theory studies suggested that the carbon acts as deep acceptor with its position at 0.90 eV above the valence band [145]. These studies further attributed the YL to the transition between the neutral charge state, C_N^0 and the negative charge state, C_N^- of carbon acceptor. Thus, the decreased exciton emission with increased YL of carbon doped sample (S5) can be attributed to the incorporation and activation of carbon in GaN buffer layers. The exciton emission in carbon doped sample with highly resistive GaN layers was reported to be completely quenched [42, 94]. Thus, the low intensity exciton emission indicates the presence of some residual free electron concentration even in the highest carbon doped GaN buffer.

6.4.3.2 Raman measurements

Raman measurements were performed on samples S4 and S5 using a confocal micro-Raman spectroscope, attached with a laser of wavelength 532 nm. Using the high penetration depth of 532 nm laser and the confocal set up, Raman measurements were performed as a function of depth in the epilayer structure of samples S4 and S5. As discussed in the Section 3.2.2 of Chapter 3, the true surface of GaN layers was identified by the first peak of the derivative of GaN- E_2 intensity vs depth profile. The measurement depth was calibrated based on the position of buried crack in the structure. Figure 6.12 (a) shows the E_2 phonon peaks of 2nd GaN of samples S4 and S5 with their corresponding peak positions. The stress values determined are listed in Table 6.2.

Fig. 6.12 Raman spectra of samples S4 and S5, showing E_2 modes of 2nd GaN (a) and A_1 (LO) modes with the corresponding line shape profile fitting for different depths of the heterostructures.

The 2nd GaN of carbon doped sample was found to be tensile strained, whereas the undoped sample is more relaxed in nature. Thus, the carbon doping resulted in the generation of tensile strain in GaN, which can be associated with the increased tilt and twist of GaN crystallites.

Figure 6.12 (b) shows the peaks corresponding to A₁ [LO] phonon-plasmon coupled modes of GaN at different depths of samples S4 and S5. In semiconductors, the plasmon modes of free electrons can couple with longitudinal optical phonon modes of crystal and result in phonon-plasmon coupled modes. The position of these coupled modes is strongly dependent upon the plasmon frequency and hence on the free electron density. The coupled mode positions, represented by A3 and B3 in the figure are obtained from the surface of 2nd GaN in both undoped and carbon doped samples, respectively. However, A2 and B2 represents the peaks obtained from the 2nd GaN, near the GaN/AlGaIn (buffer) interface, while A1 and B1 represents the coupled modes obtained in the 1st GaN of undoped and carbon doped samples, respectively.

Table 6.2. Raman E2 peak position and the estimated stress in 2nd GaN. Fitting parameters of A₁ [LO] mode at different depths are also listed.

Sample	GaN – E ₂	Stress * (M Pa)	Position	Γ (cm ⁻¹)	γ (cm ⁻¹)	ω_p (cm ⁻¹)	Carrier concentration (cm ⁻³)
S4	567.8	-67	A3	9.27	308.63	10.97	<1.0E16
			A2	9.71	366.07	47.70	3.0E+16
			A1	10.23	377.08	57.83	4.4E+16
S5	566.7	+190	B3	9.66	302.06	2.12E-7	<1.0E16
			B2	10.14	337.18	3.44E-7	
			B1	11.60	419.42	-35.55	1.7E16

* – sign represents the compression and '+' sign represents the tensile nature

Early studies on coupled phonon-plasmon A₁ [LO] modes showed that its shift in peak position with biaxial stress is only -0.8 cm⁻¹ GPa⁻¹ [235]. However, recent studies [236]

revisited the stress related shift in A_1 [LO] and obtained the relation as $-2.14 \pm 0.28 \text{ cm}^{-1}$. Ab-initio calculations on A_1 [LO] modes also suggest a shift of -1.91 cm^{-1} with 1 GPa biaxial stress [237]. These values are consistent with the reported values by Demangeot et al. [238]. However, all different reports considered that the stress free position of A_1 [LO] is $\sim 734 \text{ cm}^{-1}$. Thus, even though the stress values in 2nd GaN of both the samples are close to the relaxed value, the positions of obtained A_1 [LO] coupled modes were corrected for the stress in both the samples S4 and S5 based on the relation $\Delta\omega_{A1} = -1.91\sigma_{xx} \text{ cm}^{-1}\text{GPa}^{-1}$.

As can be observed from Fig. 6.12 (b), there is a continual blue shift in the position of A_1 [LO] from the surface of 2nd GaN (position A3) to the interface of 2nd GaN and AlGaN buffer (A2) to the 1st GaN (A1). The blue shift in the coupled modes can be attributed to the increased plasmon frequency, which is a consequence of increased electron density [239]. The plasmon frequency and the approximate carrier density at A1 to A3 can be estimated from the line-shape fitting analysis of A_1 [LO] phonon mode, derived based on a semi classical model involving the deformation potential and electro-optical mechanism [240, 241]. The line shape profile is presented in Eq. 6.1,

$$I(\omega) = KA(\omega) \text{Im} \left[\frac{-1}{\epsilon(\infty) \left[1 + \frac{\omega_L^2 - \omega_T^2}{\omega_L^2 - \omega^2 - i\omega\Gamma} - \frac{\omega_p^2}{\omega(\omega + i\gamma)} \right]} \right], \quad \text{Eq. 6.1}$$

Here, K is the proportionality factor and $A(\omega)$ is given by,

$$A(\omega) = 1 + \frac{2C\omega_T^2[\omega_p^2\gamma(\omega_T^2 - \omega^2) - \omega^2\Gamma(\omega^2 + \gamma^2 - \omega_p^2)]}{\Delta} + C^2 \left(\frac{\omega_T^4}{\Delta(\omega_L^2 - \omega_T^2)} \right) \{ \omega_p^2[\gamma(\omega_L^2 - \omega_T^2) + \Gamma(\omega_p^2 - 2\omega^2)] + \omega^2\Gamma(\omega_p^2 + \gamma^2) \} \quad \text{Eq. 6.2}$$

Where, Δ is given by

$$\Delta = \omega_p^2\gamma \left[(\omega_T^2 - \omega^2)^2 + (\omega\Gamma)^2 \right] + \omega^2\Gamma(\omega_L^2 - \omega_T^2)(\omega^2 + \gamma^2) \quad \text{Eq. 6.3}$$

$$\omega_P = \sqrt{\frac{4\pi ne^2}{m^*\epsilon\epsilon_0}} \quad \text{Eq. 6.4}$$

Here, ω_L and ω_T are the frequencies of longitudinal optical and transverse optical phonon modes, respectively. Further, n, γ, Γ and ω_p are the carrier concentration, plasmon damping constant, phonon damping constant and plasmon frequency, respectively. For fitting Eq. 6.1 to the measured GaN-A₁ [LO] modes, frequencies of $\omega_L = 533 \text{ cm}^{-1}$ and $\omega_T = 735 \text{ cm}^{-1}$ were considered. The Faust-Henry coefficient, $C = 0.48$ and the GaN constants, $\epsilon(\infty) = 5.35$ and $m^* = 0.2m_0$ were adopted [242]. Table 6.2 lists the fitting parameters, Γ, γ and ω_p , and the corresponding estimated electron concentration obtained from Eq. 6.4. As listed in the table, the plasmon frequency, ω_p , as well as the calculated carrier concentration was found to be lower near the surface of undoped 2nd GaN (A3). However, their values are found to be increased at the position A2, which confirms the parallel conduction near the interface of 2nd GaN and AlGaIn buffer. Moreover, ω_p at A1 was found to be higher compared to A2, which leads to an increased carrier concentration of $4.4 \times 10^{16} \text{ cm}^{-3}$ in the 1st GaN.

Similarly, fitting was performed for the coupled modes obtained from the surface of 2nd GaN [B3], interface of 2nd GaN/AlGaIn buffer [B2] and the 1st GaN [B1] of carbon doped sample, S5. Fig. 6.12 (b) shows the Raman A₁ [LO] phonon modes obtained at positions B1 to B3 and their corresponding peak profile fitting. As shown in the figure, the coupled modes of carbon doped sample are red shifted when compared to their equivalent modes in the undoped sample. It shows a clear indication of reduction of carrier density and carbon activation. Further, Table 6.2 lists the corresponding fitting parameters with the estimated electron concentration. As can be observed from the table for positions B3 and B2, ω_p values are lower and the corresponding electron concentration are below the estimated range ($<10^{16} \text{ cm}^{-3}$). However, for the position B1, the electron concentration is found to be $1.7 \times 10^{16} \text{ cm}^{-3}$, which shows a reduction of only ~ 2.6 times, when compared to the undoped sample.

Thus, from both PL and Raman measurements, it can be seen that carbon doping using the highest possible CBr₄ available with the current system has compensated partially the electron concentration in GaN buffer and resulted in only two orders of reduction in the HLC of AlGaIn/GaN HEMT hetero structures. However, carbon doping performed in ammonia-MBE grown GaN with lower ammonia flows resulted in semi insulating nature [94] but with rough surface morphology. These results suggest that the ammonia rich growth environment in the growth of AlGaIn/GaN HEMT heterostructures might be the cause for lower incorporation and activation of carbon in GaN layers. On the other hand, decreasing of the ammonia flow would lead to the three dimensional growth mode [89, 184], which consequently degrades the HEMT 2DEG characteristics [153]. Thus,

there exists a trade-off between smooth surface morphologies and efficient carbon doping in GaN buffer for ammonia-MBE growth process. Hence, in order to preserve the 2DEG characteristics of HEMT heterostructures, ammonia flow in the growth process was not reduced.

6.5 AlGaIn/GaN HEMT heterostructures

Undoped and carbon doped AlGaIn/GaN HEMT heterostructures with almost similar aluminum compositions and barrier layer thicknesses are listed in Table 6.3. GaN channel thickness was kept at 200 nm for all the carbon doped AlGaIn/GaN HEMT heterostructures.

Table 6.3. Electrical properties of undoped and CBr₄ doped AlGaIn/GaN HEMT heterostructures.

	AlGaIn barrier		Sheet resistance ($\Omega/\text{sq.}$)	Mobility ($\text{cm}^2/\text{V.s}$)	Sheet carrier concentration $\times 10^{13}$ (cm^{-2})
	Al (%)	AlGaIn thickness (nm)			
Undoped	31.0	24.2	436	1190	1.19
	33.0	24.0	430	1180	1.23
	32.4	23.0	527	1010	1.17
Carbon doped	31.3	23.0	544	1112	1.02
	32.0	22.6	530	1160	1.02

As listed in the table, a slight degradation in the sheet carrier concentration and mobility was observed for carbon doped samples, which resulted in the increase of sheet resistance of AlGaIn/GaN HEMT heterostructures. Similar results of degradation in the electrical characteristics were reported for the carbon doped GaN buffer in the AlGaIn/GaN HEMT heterostructures [152]. The degradation was attributed to the trapping effects of additional dislocations that were generated due to the carbon doping. Similar mechanism might be the cause in this study for the observed degradation of electrical properties in the carbon doped AlGaIn/GaN HEMT heterostructures. In summary AlGaIn/GaN HEMT heterostructures with reasonably good 2DEG characteristics have been demonstrated with two orders of reduction in the HLC by using carbon doped GaN buffers. This is the first report on CBr₄ based carbon doping in GaN buffers using ammonia-MBE.

6.6 Summary

Buffer leakage measurements performed on AlGaIn/GaN HEMT heterostructures grown using both AlN/GaN SMLs and AlGaIn/AlN/GaN SMLs resulted in an average HLC of $\sim 1 \times 10^{-3}$ A/mm and a VLC of $\sim 1 \times 10^{-1}$ A/cm² at 20V. SIMS and PL measurements suggested that the dominant impurity in the heterostructure is oxygen. Moreover, SIMS measurements also showed peaks of oxygen at various interfaces. Sequential dry etching of different epilayers in the heterostructure and the measurement of HLC after each etch step revealed a notable parallel conduction channel at the interface of 2nd AlN and 2nd GaN. Accumulated oxygen at the interfaces as observed in SIMS was attributed to the parallel conduction. Carbon doping of AlGaIn/GaN HEMT heterostructures using CBr₄ source resulted in two and one orders of reduction in the HLC and VLC, respectively. However, carbon doped GaN buffers resulted in an increased screw type dislocation density as observed from HR-XRD and associated increased pit density on the surface as observed from AFM. C-doped AlGaIn/GaN HEMT heterostructures with 200 nm thick undoped channel showed only a slight degradation in the 2DEG carrier concentration and mobility compared to undoped HEMT heterostructures.

7. Advanced GaN based HEMT heterostructures

High 2DEG density obtained in the grown AlGa_N/Ga_N HEMT heterostructures may spill over from the quantum well to the Ga_N buffer during the high voltage operation of the devices and result in the reduction of peak currents and RF power densities [243]. Improvement of carrier confinement in the channel is essential to achieve high power performance of HEMT heterostructures. In order to achieve this, it is required to increase the electric field in the channel. In this Chapter, the improvement of 2DEG carrier confinement has been demonstrated using AlGa_N/Ga_N/AlGa_N double heterojunction HEMT (DH-HEMT) heterostructure growth on Si(111) using ammonia-MBE growth process. Another advancement in the Ga_N based HEMT technology is to grow lattice matched barrier. Lattice matched barrier alleviate the strain at the heterostructure interface and improve the reliability of Ga_N based HEMT devices. Epilayer growth, composition optimization and, preliminary HEMT demonstration of nearly lattice matched InAl_N barrier layer by MBE have also been discussed in this chapter.

7.1 AlGa_N/Al_N/AlGa_N double heterojunction HEMT heterostructure

In the case of a single heterojunction AlGa_N/Ga_N HEMT (SH-HEMT) heterostructure, the electric field in the quantum well is majorly controlled by the presence of positive polarization charges at the AlGa_N/Ga_N heterojunction interface. By designing a double heterojunction such as AlGa_N/Ga_N/AlGa_N based DH-HEMT heterostructure, the electric field in the channel can be increased due to the combined effect of positive polarization charge that develops at the initial AlGa_N/Ga_N interface and the negative polarization charges at the second Ga_N/AlGa_N interface [158]. This increase in the electric field further improves the confinement of 2DEG. The improvement in the carrier confinement and the consequent enhancement of carrier mobility has been demonstrated successfully for AlGa_N/Ga_N/AlGa_N DH-HEMTs grown using MOCVD on Si substrate [161, 162]. Using ammonia-MBE growth process, DH-HEMT heterostructure growth was demonstrated on 50-mm diameter Si substrate [244]. Growth of DH-HEMT heterostructure on 100-mm Si substrate using ammonia-MBE growth process is presented in this chapter. In the following sections, detailed analysis of the surface, structural and electrical characteristics of the DH-HEMT heterostructure were performed and the comparative advantage of DH-HEMT over the SH-HEMT heterostructures has been demonstrated. Moreover, submicron HEMT devices are

fabricated on grown DH-HEMT heterostructures and corresponding I_{DS} - V_{DS} and transfer characteristics are also presented.

7.1.1 Growth of AlGaIn/GaN/AlGaIn DH-HEMT heterostructure

Figures 7.1 (a) and (b) show the cross sectional view of typical epilayer stack of AlGaIn/GaN SH- and DH-HEMT heterostructures, respectively grown on 100-mm Si substrate. In both heterostructures, AlN/GaN SMLs were used as stress mitigation layers to grow 1 μ m thick (Al)GaIn buffer. In the DH-HEMT heterostructure, a thin layer of GaN (40 nm) acts as channel. AlGaIn barrier with the aluminum mole fractions of 31.7 and 32.5% were grown for the SH-HEMT and DH-HEMT heterostructures, respectively followed by the growth of GaN cap layer. The thickness of AlGaIn barrier was 27 and 28.5 nm for the SH-HEMT and DH-HEMT heterostructures, respectively. Al composition of AlGaIn buffer in the DH-HEMT heterostructure is 10%.

Fig. 7.1 Cross sectional view of epilayer structures of SH-HEMT (a) and DH-HEMT (b).

7.1.2 Structural characterizations

Room temperature ex-situ bow measurements conducted on as-grown SH-HEMT and DH-HEMT heterostructures revealed mutually opposite bowing as shown in Figs. 7.2 (a) and (b), respectively. The SH-HEMT heterostructure showed a concave bow of -44 μ m, while DH-HEMT heterostructure showed a convex bow of +120 μ m. High convex bowing was also observed for the heterostructures grown using AlGaIn/AlN/GaN SMLs (Section 5.1.2 of Chapter 5). The AlGaIn/2nd AlN interface is one of the reasons that were attributed to the mitigation of stress from GaN to Si. Similarly, bottom AlGaIn/AlN interface in the DH-HEMT heterostructure might be mitigating the stress from GaN to Si substrate and resulting in high convex bowing compared to SH-HEMT heterostructure.

In spite of completely opposite bowing observed, the microscopic investigation revealed crack free surfaces for both of these heterostructures on 100-mm Si substrate.

Fig. 7.2 Bow of SH-HEMT (a) and DH-HEMT (b) heterostructures grown on 100-mm Si substrate.

Surface morphology of SH-HEMT and DH-HEMT heterostructures was investigated by AFM and the corresponding images are shown in Figs. 7.3 (a) and (b), respectively. Mound type surface morphology was observed for both the heterostructures. However, mound features appeared to be larger in the lateral direction for the DH-HEMT compared to SH-HEMT heterostructure. While an RMS roughness value of 2.2 nm was obtained for the DH-HEMT, it was 2.9 nm for the SH-HEMT heterostructure for a scan area of $10 \times 10 \mu\text{m}^2$. Streaky RHEED patterns observed during the growth of different epilayers of SH- and DH-HEMT heterostructures indicate that the growth proceeded through two dimensional growth mode in both the heterostructures. However, the larger mound size and lower RMS roughness indicate that the lateral growth is enhanced in the buffer of DH-HEMT heterostructure. Similarly observation has been reported by Cordier et al. [244] with lower RMS roughness for the DH-HEMT compared to the SH-HEMT heterostructure.

Fig. 7.3 AFM surface morphology over $10 \times 10 \mu\text{m}^2$ scan area of SH- (a) and DH- (b)-HEMT heterostructures.

Enhancement of the lateral growth and the consequent improvement in the surface morphology in the ammonia-MBE growth process can be attributed to two parameters. They are enhanced surface diffusion of metallic adatoms [84] and the increased ammonia cracking, leading to the enhanced incorporation of metal and nitrogen atoms at the step edges [89] as discussed in the Section 4.3 of Chapter 4. As observed from the in-situ stress measurements of AlGa_{0.10}N/GaN SMLs in the Section 5.1.1 of Chapter 5, the reduced lattice mismatch between 2nd AlN and Al_{0.10}Ga_{0.90}N buffer leads to its lower relaxation and higher residual compression. This might have enhanced the surface diffusion of metal adatoms during the growth of AlGa_{0.10}N buffer [200, 201]. Moreover, additional metal flux available in the form of aluminum during the growth of AlGa_{0.10}N buffer might have also contributed in the cracking of more ammonia at the step edges leading to the replenishment of the adsorption sites to incorporate more atoms into the lattice. Thus, the combined effect of increased adatom diffusion followed by the improved incorporation of metal atoms at the step edges might possibly have enhanced the 2D growth mode and resulted in smoother surface morphology for the DH-HEMT heterostructure.

Structural properties of the HEMT heterostructures grown were investigated by HR-XRD technique. In order to investigate the strain states and relaxation properties of different epilayers, reciprocal space mapping (RSM) was performed along GaN (10 $\bar{1}$ 5) plane. The results are presented in Figs. 7.4 (a) and (b) for SH-HEMT and DH-HEMT samples, respectively.

Fig. 7.4 Reciprocal space mapping along $(10\bar{1}5)$ plane for SH-(a) and DH-(b) HEMT heterostructures.

While 2nd AlN, AlGaIn barrier and 2nd GaN are clearly observed in the RSM of SH-HEMT heterostructure, AlGaIn buffer and GaN channel, instead of 2nd GaN are observed in the case of DH-HEMT sample. GaN channel (40 nm thick) in the DH-HEMT was found to be under high residual compressive stress with only a parallel mismatch of 1334 ppm with respect to Al_{0.10}Ga_{0.90}N buffer in the RSM.. However, AlGaIn barrier was found to have grown coherently with GaN buffer for the SH-HEMT and GaN channel for the DH-HEMT. Coherent growth ensures the development of piezoelectric polarization (P_{PE}) to induce 2DEG in the quantum well at the heterojunction interfaces.

In order to investigate the crystal quality, XRD rocking curve scans were performed along (0002) , $(10\bar{1}2)$ and $(30\bar{3}2)$ planes of GaN and AlGaIn buffers of SH- and DH-HEMT heterostructures, respectively and the corresponding FWHM values obtained are listed in Table 7.1. The FWHM of (0002) plane shows slight degradation in the screw type of dislocation density in the DH-HEMT buffer compared to the buffer in the SH-HEMT. However, the FWHM of $(30\bar{3}2)$ plane shows improvement in the edge type of dislocation density for the buffer in DH-HEMT compared to SH-HEMT. The overall dislocation density as observed from the FWHM of $(10\bar{1}2)$ scan is almost similar in both the

heterostructures. This further signifies the fact that the growth of AlGaIn buffer in the DH-HEMT did not contribute to any overall degradation in the crystal quality.

Table 7.1. FWHM of XRD rocking curves along different planes of buffers in SH- and DH-HEMT heterostructures.

Buffer	FWHM (arcsec)		
	(0002)	(10 $\bar{1}$ 2)	(30 $\bar{3}$ 2)
GaN (SH-HEMT)	1003	1652	2540
AlGaIn (DH-HEMT)	1185	1640	2376

7.1.3 Electrical characterizations

Electrical measurements on the as grown SH- and DH-HEMT heterostructures were conducted using Van der Pauw Hall measurement and mercury probe capacitance-voltage (C-V) techniques. The obtained electrical properties of both the heterostructures are listed in Table 7.2. DH-HEMT heterostructure shows lower carrier concentration compared to SH-HEMT. However, the room temperature mobility of 2DEG in the DH-HEMT heterostructure is considerably higher compared to that in the SH-HEMT heterostructure.

Table 7.2. Electrical properties SH- and DH-HEMT heterostructures obtained using Hall, C-V and simulation.

Heterostructure	Electrical properties				
		Hall	C-V	Simulation	
	R_s ($\Omega/\text{sq.}$)	μ_H ($\text{cm}^2/\text{V.s}$)	n_s ($\times 10^{13} \text{ cm}^{-2}$)		
SH-HEMT	434	1310	1.09	1.04	1.15
DH-HEMT	420	1510	0.97	0.97	1.09

Hall measurements were also performed as a function of temperature from 400 to 90K on SH- and DH-HEMT heterostructures. Figure 7.5 shows the product of carrier concentration and the mobility ($n_s \times \mu_H$) as a function of temperature for both SH- and DH-HEMTs. The ($n_s \times \mu_H$) product yields almost close values for both SH- and DH-HEMT heterostructures. This may indicate that the current driving capabilities are similar. However, the slight deviation obtained in the product at lower temperatures is due to the saturation of mobility in the DH-HEMT heterostructure, which can be attributed to its lower 2DEG carrier concentration and its consequent lesser screening effect.

Fig. 7.5 $n_s \times \mu_H$ product of SH- and DH- HEMT heterostructures as a function of temperature.

Figure 7.6 (a) and (b) show the capacitance-voltage (C-V) measurements obtained on the SH- and the DH-HEMT heterostructures, respectively. Nearly flat plateau of capacitance followed by a sharp pinch off characteristic indicates the improved confinement of 2DEG in the DH-HEMT compared to the SH-HEMT heterostructure.

Fig. 7.6 Capacitance-voltage measurements along with their differentiated and integrated profiles for SH -HEMT (a) and DH-HEMT (b) heterostructures.

The pinch off characteristics of capacitance were further analyzed by taking the derivative of capacitance-voltage data (dC/dV) and plotting as dotted peak profiles in Figs. 7.6 (a) and (b). The sharp dC/dV profile shows the improvement in the confinement of 2DEG in DH-HEMT heterostructure compared to SH-HEMT

heterostructure. Further, the integration of capacitance as a function of voltage from zero bias to pinch off was used to estimate the 2DEG carrier concentrations in the heterostructure, which resulted in a carrier concentration of $1.04 \times 10^{13} \text{ cm}^{-2}$ and $0.97 \times 10^{13} \text{ cm}^{-2}$ for SH-HEMT and DH-HEMT heterostructures, respectively. These values are closer to the obtained n_s value from Hall measurements. It was also observed that the capacitance after pinch off was lower for DH-HEMT, indicative of its decrease in the background carrier concentration compared to SH-HEMT. Thus, the overall electrical characterization indicates that the DH-HEMT heterostructure provides improved confinement of 2DEG with slightly lower carrier concentration and improved mobility.

To understand the behavior of 2DEG and its confinement in the quantum well for both the SH-HEMT and DH-HEMT heterostructures, theoretical simulations using self-consistent 1D Schrodinger-Poisson solver [245] were performed. For the simulations, epilayer structure from the top GaN cap layer to the 2nd AlN layer was only considered, assuming layers below 2nd AlN may not have significant effect on the 2DEG. Further, the Fermi level in the 2nd AlN layer was assumed pinned to the centre of the band gap considering high resistive nature of AlN. Moreover, all the epilayers in both heterostructures were considered to be undoped for the simulation. Polarization values for the ternary alloy were obtained from their respective binary alloys and both the spontaneous and piezoelectric polarizations were considered for the simulation. The required mechanical and piezoelectric constants were obtained from the literature [246]. Figure 7.7 shows the simulated conduction band profiles and carrier distribution in the quantum well for the SH- (a) and the DH- (b) HEMT heterostructures.

As expected, the quantum well becomes more triangular for the DH-HEMT compared to the SH-HEMT. Moreover, the confinement of 2DEG is also improved for the DH-HEMT, as indicated by the sharp carrier concentration profile. This improvement in the confinement was also observed as near flat capacitance plateau followed by sharp pinch off in the CV measurements. The area under the carrier concentration profile was further used to obtain the sheet carrier concentration and the corresponding values are presented in Fig. 7.7 (a) and (b) and are also listed in Table 7.2. The simulated sheet carrier concentration values were found to be slightly higher compared to measured values. The slight deviation might be due to the variation in values of different physical constants considered for the simulation. However, consistently lower sheet carrier concentration of 2DEG was observed for the DH-HEMT compared to the SH-HEMT heterostructures in the calculated and measured values. The decrease in value in case of DH-HEMT heterostructure can be attributed to the negative polarization that develops

at the bottom GaN/AlGa_N interface. Moreover, the increase in the electron mobility of the 2DEG may be associated with their increased confinement in the quantum well.

Fig. 7.7 Simulated conduction band and carrier distribution profiles of SH- (a) and DH-(b) HEMT heterostructures.

7.1.4 AlGa_N/Ga_N/AlGa_N DH-HEMT devices

HEMT devices with submicron gate were fabricated using the grown DH-HEMT heterostructure. The T-gate used was with a gate length and width of 0.27 and $2 \times 150 \mu\text{m}$, respectively. The gate metal stack used was Ni/Au with the thickness of 50/400 nm. Figures 7.8 shows the preliminary results of $I_{\text{DS}}-V_{\text{DS}}$ and transfer characteristics of HEMT devices, which exhibited a maximum drain current (I_{Dmax}) of 806 mA/mm at $V_{\text{g}} = +1\text{V}$ and a maximum transconductance (g_{mmax}) of 135 mS/mm at $V_{\text{D}} = 10\text{V}$. The devices further showed good pinch off characteristics with a threshold voltage of -5.1 V. Thus, as predicted from Hall measurements, almost similar I_{DS} was observed in the Section 4.7 of Chapter 4 for the SH-HEMT.

Fig. 7.8 I_{DS} - V_{DS} (a) and transfer (b) characteristics of DH-HEMT submicron devices.

In addition, the buffer breakdown voltage has also been measured between two ohmic contacts with the gap of 10 μm and a width of 50 μm . Figure 7.9 shows the breakdown characteristics of SH- and DH-HEMTs. With respect to the SH-HEMT heterostructure, the DH-HEMT heterostructure exhibited 3 times higher buffer breakdown voltage. In fact, the breakdown voltage could be even higher at the usual reference current of 1 mA/mm. The increase of buffer breakdown can be attributed to the AlGaIn buffer in the DH-HEMT heterostructure, which is consistent with its observed lower capacitance sub threshold region of CV measurements.

Fig. 7.9 Comparison of buffer breakdown characteristics of SH- and DH- HEMTs.

Thus, DH-HEMT heterostructure was proved to be a better option for the high power applications of GaN based HEMTs due to its improved confinement of 2DEG and breakdown voltages but similar current driving capabilities as that of SH-HEMT.

7.2 Lattice matched InAlN barrier for GaN based HEMT heterostructures

AlGaIn/GaN based SH-HEMT and DH-HEMT heterostructures have produced excellent 2DEG properties on Si substrate. The developed 2DEG in these heterostructures is a result of both spontaneous and piezoelectric polarizations. The tensile strain in the AlGaIn barrier contributes to piezoelectric polarization. However, it is also a concern for the reliability of AlGaIn/GaN HEMT heterostructures [21, 22]. To alleviate the strain in the barrier, Kuzmick [247] proposed the usage of lattice matched $\text{In}_{0.17}\text{Al}_{0.83}\text{N}$ barrier for the GaN based HEMT applications. Growth of InAlN is challenging due to the high binding energy difference between InN (2.2 eV) and AlN (2.8 eV), leading to the high miscibility gap. Hence, the growth of InAlN material requires non-equilibrium growth conditions to stabilize the InAlN phase. The low temperature growth regime of PA-MBE has been successfully used for the growth InAlN epilayers [26, 30, 171]. This section of the chapter deals with the growth, optimization and characterization of nearly lattice matched $\text{In}_{0.18}\text{Al}_{0.82}\text{N}$, grown using PA-MBE growth process. Further, demonstration of InAlN/AlN/GaN HEMT heterostructure is also discussed.

7.2.1 GaN growth using PA-MBE on GaN templates grown using ammonia-MBE

Growth of GaN buffer on Si substrate has been well optimized using ammonia-MBE growth process, which resulted in uniform structural, morphological and electrical characteristics across the 100-mm diameter of Si substrate. The as grown buffers have been utilized as templates for the growth of InAlN by PA-MBE growth process. Either a two layer structure (40 nm AlN/200 nm GaN) on Si substrate or 1200 nm thick GaN grown using AlGaIn/AlN/GaN SMLs has been utilized as templates for various growth runs. Diced 1" × 1" samples from 100-mm ammonia-MBE grown GaN wafers were used for the growth in this study. Prior to the growth, the diced samples underwent organic and chemical cleaning.

Ammonia-MBE grown GaN templates on Si substrate prior to the growth showed bright streaky RHEED (1 × 1) pattern as shown in Fig. 7.10 (a). Subsequently, the GaN template went through three cycles of gallium (Ga) deposition and desorption at temperatures of 670 and 770 °C, respectively. This process ensures an oxygen-free surface prior to the PA-MBE growth. However, it also resulted in spotty RHEED pattern as shown in Fig. 7.10 (b). PA-MBE growth of GaN started at a growth temperature of 710 °C. A 500 nm thick GaN layer was grown in slightly metal rich growth conditions (III/V > 1). GaN layer with

a thickness of 500 nm resulted in near streaky RHEED pattern as shown in Fig. 7.10 (c). The improved RHEED pattern shows that the surface has recovered after the initial ‘Ga’ deposition and desorption cycles.

Fig. 7.10 RHEED pattern of as loaded GaN template (a), after the Ga flash off (b) and after 500 nm thick over grown GaN by PA-MBE (c).

Surface morphology of ammonia-MBE grown GaN template and 500 nm thick PA-MBE over grown GaN samples were investigated by AFM, which are shown in Figs. 7.11 (a) and (b), respectively. The images are taken in the amplitude mode of AFM.

Fig. 7.11 AFM images of 5 x 5 μm² area of ammonia-MBE grown GaN template (a) and after GaN over grown by PA-MBE (b).

It can be seen that the PA-MBE growth follows mound type morphology of ammonia-MBE grown GaN template. However, the morphology appears to be more coalesced for PA-MBE over grown GaN epilayers. In addition, more pits are observed on the regrown surface, which can be attributed to the screw type dislocations. HR-XRD measurements on ammonia-MBE grown GaN templates showed screw and edge type dislocation densities of 3.15×10^9 and $3.23 \times 10^{10} \text{ cm}^{-2}$, respectively. However, the overgrown PA-MBE grown GaN resulted in screw and edge type dislocation densities of 2.61×10^9 and $1.83 \times 10^{10} \text{ cm}^{-2}$, respectively. These measurements clearly indicate that the screw type of dislocation density is not increased for the PA-MBE overgrown GaN epilayers. Thus,

the appearance of surface pits for the over grown layers can be attributed to the opening of dislocations on the growth surface. Moreover, from HR-XRD measurements, it was found that the overall dislocation density reduced for 500 nm thick over grown layers. The reduction of dislocation density with the increase in GaN thickness on Si substrate has been reported [70]. Similarly, the reduction in the dislocation density can be attributed to the overall increase in the thickness of GaN. These optimized GaN buffers were further used for InAlN/GaN HEMT heterostructure growth.

7.2.2 InAlN growth by PA-MBE

In a PA-MBE growth process, the incorporation of indium into the lattice is found to be minimal for the growth temperatures above 560 °C [248, 249] and the phase separation in the InAlN layer was observed for a growth temperature of 480 °C [26]. Hence, the temperature window for InAlN growth is 400 to 450 °C to grow InAlN/GaN HEMT heterostructures by PA-MBE [26, 30, 171]. Based on these observations, a growth temperature of 420 °C was chosen to grow InAlN on PA-MBE over grown GaN layers. However, InAlN growth at 420 °C requires temperature ramp down from the GaN growth temperature, which results in a growth interruption. To protect the GaN surface during the ramping down process, an AlN epilayer of 1 nm thickness was proposed on GaN [26]. AlN layer not only protects the surface of GaN during ramp down but also acts as a spacer layer to decrease the alloy scattering of InAlN barrier layer. Hence, prior to the InAlN growth an AlN spacer was grown on GaN at the GaN growth temperature. Bright and near streaky RHEED pattern was obtained for the AlN growth as shown in Fig. 7.12 (a). During the temperature ramp down, nitrogen plasma shuttered was closed to protect the growth surface from roughening due to high energy plasma particles [171].

Fig. 7.12 RHEED pattern of 1 nm AlN (a), InAlN during the growth (b), InAlN after the growth (c).

In order to optimize the composition of InAlN to the lattice matched value of 17%, three samples, namely A, B and C were grown by keeping the indium flux at 1.72×10^{-8} Torr, while increasing the aluminum flux from 6.10 to 7.93×10^{-8} Torr. InAlN was grown in a

slightly metal rich growth conditions and its thickness was kept at ~ 60 nm for all the samples. InAlN growth on AlN spacer started with streaky RHEED pattern. However, after few monolayers of InAlN growth, RHEED become slightly spotty and remained spotty till the end of the growth as shown in Fig. 7.12(b). RHEED pattern became bright after the growth at 300°C as shown in Fig. 7.12 (c).

Composition of InAlN in samples A, B and, C was estimated using HR-XRD ω - 2θ scans. Figure 7.13 shows the indium composition as a function of 'Al' flux for samples A to C for a given In flux of 1.72×10^{-8} Torr. As shown, the indium composition of InAlN decreases with the increase of aluminum flux. A composition of 18 % is achieved for an aluminum flux of 7.93×10^{-8} Torr, which corresponds to nearly lattice matching with GaN.

Fig. 7.13 Indium composition in InAlN epilayer as a function of aluminum flux obtained from HR-XRD ω - 2θ scans.

Figure 7.14 shows the HR-XRD ω - 2θ scan along GaN (0002) plane of sample C along with the dynamic simulation indicating the In mole fraction of 18%. There is no indication of any phase separation or indium segregation in the grown InAlN lattice. Sample C with the near lattice matched composition of 18% was further investigated by HR-XRD RSM mapping along GaN (10 $\bar{1}$ 5) plane. As shown in Fig. 7.15, 60 nm thick InAlN layer is found to have coherently grown with GaN layer, confirming the near lattice matched composition of grown InAlN layer.

Fig. 7.14 HR-XRD ω -2 θ scan along GaN (0002) with the dynamic simulation on sample C.

Fig. 7.15 HR-XRD RSM along GaN ($10\bar{1}5$) with the dynamic simulation on sample C.

7.2.3 InAlN/AlN/GaN HEMT heterostructures

An InAlN/AlN/GaN HEMT heterostructure with similar InAlN composition as that of sample A was grown to check the feasibility InAlN based HEMT growth on ammonia-MBE grown GaN templates. The epilayer stack consists of a 500 nm thick PA-MBE overgrown GaN layer on ammonia-MBE grown GaN on Si template. This is followed by 1 nm AlN spacer and a 15 nm thick $\text{In}_{0.24}\text{Al}_{0.76}\text{N}$ barrier. CV measurement (Fig. 7.16) shows plateau of capacitance followed by pinch off, indicating the presence of 2DEG. However, the high capacitance observed after pinch off indicates the conduction in buffer GaN. Integration of capacitance from zero bias to pinch off voltage results in a 2DEG carrier concentration of $4.5 \times 10^{12} \text{ cm}^{-2}$.

Fig. 7.16 Capacitance-Voltage measurement on as grown InAlN/AlN/GaN HEMT structure. The sheet carrier concentration obtained by integration of the C-V profile is also presented.

Hall measurements on as grown InAlN HEMT heterostructures showed a room temperature carrier concentration of $7.3 \times 10^{12} \text{ cm}^{-2}$ and a mobility of $140 \text{ cm}^2/\text{V.s}$, which resulted in a sheet resistivity of $4142 \text{ } \Omega/\text{sq}$. Hall measurement as a function of measurement temperature (Fig. 7.17) shows that the carrier concentration is invariant below 250 K. However, it shows an increasing trend beyond 250 K. This behavior indicates that the sample consists of a combination of 2DEG and buffer conduction. This behavior is consistent with the observation from CV measurements. Thus, the observed mobility is also a resultant of both 2DEG and buffer conduction effects. The buffer conduction in the epitaxial layers could be due to the regrowth interface between PA-MBE and ammonia-MBE GaN layers.

Fig. 7.17 Hall measurement as a function of temperature on InAlN/AlN/GaN HEMT heterostructure.

The 2DEG carrier concentration determined appears low for InAlN/AlN/GaN HEMT heterostructures. The degradation of 2DEG concentration is either due to the regrown PA-MBE GaN or the poor quality of InAlN barrier. In order to separate these two effects, an AlGaIn/GaN HEMT heterostructure was grown on the regrown PA-MBE GaN with an AlN spacer thickness of 1nm. HR-XRD ω -2 θ scans on as grown AlGaIn/GaN HEMT heterostructures showed an aluminum composition of 28 % and AlGaIn thickness of 18 nm. Hall measurements on this sample showed a sheet carrier concentration of $9 \times 10^{12} \text{ cm}^{-2}$ and a mobility of $568 \text{ cm}^2/\text{V.s}$. The carrier concentration is consistent with the 'Al' composition of AlGaIn barrier. Hence, it can be concluded that the regrown GaN might not have much effect on the obtained lower 2DEG carrier concentration in InAlN/AlN/GaN HEMT heterostructure. This suggests that the quality of InAlN barrier might be the cause for the observed results and further investigation is required.

7.2.3.1 Post growth annealing of InAlN/AlN/GaN HEMT heterostructures

In order to improve the quality of as grown InAlN barrier, post growth furnace annealing was done for InAlN/AlN/GaN HEMT heterostructures. To protect the epitaxial surface during the annealing process, 120 nm thick Si_3N_4 was deposited on the sample surface using plasma enhanced chemical vapor deposition (PECVD) technique. The annealing was done in nitrogen ambient at 750°C for 1 hr. After annealing, Si_3N_4 from the surface of the sample was removed by treating the samples with buffer oxide etchant (BOE). Figure 7.18 shows the HR-XRD ω -2 θ scan on the sample before and after annealing.

Fig. 7.18 HR-XRD ω -2 θ scan on InAlN/AlN/GaN HEMT heterostructure before and after annealing.

As shown in the figure, after annealing the indium composition is reduced compared to as grown InAlN layer. With Si₃N₄ on the surface of InAlN, there might not be any desorption of indium from the sample. Thus, the variation could be due to the rearrangement of indium atoms inside the lattice.

Hall measurements performed on the annealed sample showed a room temperature carrier concentration of $1.85 \times 10^{13} \text{ cm}^{-2}$ and mobility of $512 \text{ cm}^2/\text{V.s}$. Figure 7.19 shows Hall measurements as a function of temperature from 90 to 400 K for the HEMT sample before and after annealing. Hollow triangles and circles represent the 2DEG mobility and carrier concentration, respectively before annealing. The filled triangles and circles represent the corresponding values after annealing.

Fig. 7.19 Mobility and sheet carrier concentration as a function of temperature for InAlN/AlN/GaN HEMT heterostructure before and after post growth annealing.

At lower temperatures, both the 2DEG carrier concentration and mobility increased by about 3 times and 4 times, respectively, after the annealing process, as shown in figure. The improvement in the 2DEG characteristics can be attributed to the improved quality of InAlN barrier by the annealing process. This assumption is based on the fact that the as-grown AlGaIn/GaN HEMT sample showed good 2DEG characteristics in spite of using regrown GaN buffer for the layer growth.

Thus, InAlN/AlN/GaN HEMT heterostructures with good 2DEG characteristics are demonstrated on Si substrate in this study using a combination of ammonia-MBE and PA-MBE growth processes. This is the first demonstration of InAlN based HEMT heterostructure growth on Si substrate by MBE. However, further structural and optical analyses are required to study the annealing effect on the properties of InAlN layers and

progress in successful development of lattice matched InAlN/AlN/GaN HEMT heterostructures.

7.3 Summary

AlGaIn/GaN/AlGaIn DH-HEMT heterostructure was grown on 100-mm Si substrate using ammonia-MBE with 'Al' composition of 10% for the AlGaIn buffer. Surface roughness was observed to be decreased for the DH-HEMT heterostructure compared to SH-HEMT heterostructure while the buffers of both the heterostructures showed similar crystal quality. Hall measurements on DH-HEMT heterostructure showed a room temperature mobility of 1510 cm²/V.s and sheet carrier concentration of 0.97×10^{13} cm⁻². Capacitance-voltage measurements confirmed the improved confinement of 2DEG in the DH-HEMT heterostructure compared to the SH-HEMT heterostructure. Low temperature hall measurements and dc characteristics of the HEMT devices showed similar current carrying capability of both the heterostructures. However, DH-HEMT showed 3 times higher buffer break down voltages compared to SH-HEMT.

InAlN/AlN/GaN heterostructures on Si substrate were grown using MBE growth technique. InAlN layer was grown using PA-MBE growth process while ammonia-MBE was used for GaN buffer growth. By maintaining an InAlN growth temperature of 420 °C and slightly metal rich growth conditions, nearly lattice matched (18%) 60 nm thick InAlN was grown without any phase separation or indium segregation. InAlN/AlN/GaN HEMT heterostructures with 'In' composition of 24% and barrier thickness of 15 nm was found to result in a room temperature 2DEG carrier concentration of 7.3×10^{12} cm⁻² and a mobility of 140 cm²/V.s. However, post growth annealing of the as-grown InAlN/AlN/GaN HEMT heterostructures at 750 °C for 1 hour resulted in improved 2DEG concentration of 1.85×10^{13} cm⁻² and mobility of 512 cm²/V.s. Improved the quality of barrier with the annealing was attributed to the improved 2DEG concentration.

8. Conclusions

This chapter summarizes the inferences drawn from several systematic studies conducted to achieve crack free, low wafer-bow, good crystal quality and smooth surface morphology AlGa_N/Ga_N HEMT heterostructures on 100-mm Si substrate. A summary of the studies conducted on AlGa_N/Ga_N/AlGa_N based DH-HEMT and InAlN/AlN/GaN based HEMTs are also described.

8.1 Stress management in AlGa_N/Ga_N HEMT heterostructures on 100-mm Si(111) using AlN/GaN SMLs.

AlN growth on Si substrate was optimized for a growth rate of 0.13 $\mu\text{m/h}$ and a growth temperature of 920 °C to achieve smooth surface morphologies and good crystal quality. However, Ga_N grown on the optimized AlN showed heavy cracking. AlGa_N-SML and AlN/GaN-SMLs were investigated to achieve crack free Ga_N. Ga_N buffer grown with a growth rate of 0.22 $\mu\text{m/h}$ showed 2D growth mode in both the heterostructures. However, the growth of Ga_N on AlGa_N-SML was observed to follow Volmer-Weber mechanism, while AlN/GaN-SMLs resulted in screw type dislocation mediated step flow growth phenomenon. Moreover, high residual compressive stress in 500 nm thick 2nd Ga_N, larger grain sizes, lower pit density and good crystalline quality indicate that the AlN/GaN-SML stack is an efficient stress mitigating layer compared AlGa_N-SML. Thus, AlN/GaN-SMLs were chosen for HEMT heterostructure growth.

A typical heterostructure with AlN/GaN-SMLs consists of 50 nm of 1st AlN followed by the growth of 1st Ga_N/2nd AlN-SMLs and finally 2nd Ga_N buffer. Increased growth rate of Ga_N to 0.7 $\mu\text{m/h}$ by the simultaneous increase of Ga flux and ammonia flow resulted in the improvement of the surface morphology of 500 nm thick 2nd Ga_N. This was primarily attributed to the enhanced 2D growth mode due to increased cracking of ammonia at the step edges.

AlN/GaN-SMLs intrinsically contained buried cracks in them. The variation of buried crack density as function of thicknesses of 1st Ga_N/2nd AlN layers was investigated. While the increase in the 1st Ga_N thickness increased the buried crack density, the increase in the 2nd AlN thickness did not show any effect on the buried crack density up to 250 nm of its thickness. With higher buried crack density, compression in the 1st Ga_N was found to have reduced, which further decreased the compression in the 2nd Ga_N. This study indicates that the compression in the 1st Ga_N together with the buried cracks

act as a compliant buffer and mitigate the stress from GaN to Si during sample cool down. Thus, AlN/GaN-SMLs not only induced the compressive stress successfully in 2nd GaN but also mitigated the stress transfer during cool down. Further, AlN/GaN-SMLs with the thicknesses of 250/250 nm yielded compressively strained GaN layers with smooth surface morphology and good crystal quality. Hence, they were considered for the development of GaN based HEMT heterostructures on Si using ammonia-MBE.

The increased relaxation of 2nd GaN with its increased thickness up to 1000 nm resulted in kinetic roughening of the mounds and led to high RMS roughness of 4.2 nm for a scan area of 5×5 μm^2 . Increasing the V/III ratio of 2nd GaN from 950 to 2070 resulted in decreased kinetic roughening, and an RMS roughness value of 2.8 nm was achieved for a scan area of 5 ×5 μm^2 . AlGaIn/GaN HEMT heterostructures, grown on such a buffer, showed a room temperature mobility of 1340 $\text{cm}^2/\text{V.s}$ and carrier concentration of $1.13 \times 10^{13} \text{ cm}^{-2}$, which resulted in a sheet resistance of 409 Ω/sq . Good uniformity in electrical characteristics across 100-mm wafer with the minimal standard deviation of 2.5, 0.6 and 2.6% for the sheet resistance, sheet carrier concentration and mobility, respectively was obtained. However, as-grown HEMT heterostructures grown using AlN/GaN SMLs on 100-mm Si (111) resulted in crack free wafers with tensile bowing in the range of 30-70 μm .

8.2 Stress management in AlGaIn/GaN HEMT heterostructures on 100-mm Si(111) using AlGaIn/AlN/GaN SMLs

In order to reduce the tensile bowing of grown AlGaIn/GaN HEMT heterostructures, a new stack of AlGaIn/AlN/GaN-SMLs was studied. The new SMLs contained an additional AlGaIn on top of the AlN/GaN-SMLs. In-situ stress measurements on 2nd GaN grown using AlGaIn/AlN/GaN-SMLs showed higher relaxation compared to GaN grown using AlN/GaN-SMLs. However, the former heterostructure showed higher mean compressive stress. In accordance with the high mean stress, the heterostructure grown using AlGaIn/AlN/GaN-SMLs has also resulted in compressive bow of +82 μm for a 2nd GaN thickness of 900 nm. However, for a similar thickness of 2nd GaN, AlN/GaN-SMLs exhibited a tensile bow of -35 μm . Thus, the increased relaxation of 2nd GaN with compressive bow of the wafer showed increased stress mitigation from GaN to Si in the case of AlGaIn/AlN/GaN-SMLs.

Cross sectional TEM images obtained in the bright field mode showed that the bending and looping of dislocations occur at both 2nd AlN/AlGaN and AlGaN/2nd GaN interfaces. The relaxation of residual compressive stress at these two interfaces was attributed to increased relaxation of 2nd GaN as observed in the in-situ stress measurements. Moreover, WBDF images of TEM obtained with $g = 0002$ has revealed additional tilt at the 2nd GaN/AlGaN and AlGaN/2nd AlN interfaces. Stress mitigation through elastic distortion at such interfaces could have caused the increased stress mitigation in the heterostructures grown using AlGaN/AlN/GaN SMLs.

Increased RMS roughness of 8.4 nm for a scan area of $10 \times 10 \mu\text{m}^2$ was obtained for 2nd GaN grown using AlGaN/AlN/GaN SMLs. However, an RMS roughness of only 3.8 nm was obtained for the 2nd GaN grown using AlN/GaN SMLs. The increased RMS roughness was attributed to the increased relaxation of 2nd GaN in the heterostructures grown using AlGaN/AlN/GaN SMLs. In order to improve the surface morphology, the V/III ratio of 2nd GaN was varied from 1020 to 4050. Smooth surface morphologies with RMS roughness of 4.1 nm for $10 \times 10 \mu\text{m}^2$ scan area was achieved for the sample grown with higher V/III ratio of 4050 and a thickness of 1150 nm. Moreover, a residual compressive stress of ~ 200 MPa in 2nd GaN and a compressive bow of $+97 \mu\text{m}$ was observed at room temperature in these heterostructures. The ex-situ wafer bow was reduced from $+97 \mu\text{m}$ to $<+35 \mu\text{m}$ by increasing the 2nd GaN thickness up to 1400 nm. AlGaN/GaN HEMT heterostructures grown using AlGaN/AlN/GaN-SMLs showed a room temperature sheet carrier concentration of $0.88 \times 10^{13} \text{ cm}^{-2}$ and mobility of $1380 \text{ cm}^2/\text{V.s}$, which resulted in a sheet resistance of $517 \Omega/\text{sq}$. Reasonably good uniformity in electrical characteristics was observed for as grown HEMT heterostructures with their standard deviation of 7%, 1% and, 8% for the sheet resistance, carrier concentration and mobility across the 100-mm substrate.

8.3 Carbon doped GaN buffer

AlGaN/GaN HEMT heterostructures grown using both AlN/GaN SMLs and AlGaN/AlN/GaN SMLs showed an average HLC of $\sim 1 \times 10^{-3} \text{ A/mm}$ and a VLC of $\sim 1 \times 10^{-1} \text{ A/cm}^2$ at 20V. Comparably higher oxygen concentration was found than silicon in the epitaxial layers using SIMS measurements. Moreover, the presence of oxygen peaks was also observed at various interfaces of the heterostructure. PL measurements as a function of temperature showed a donor bound exciton peak with a binding energy of 35 meV, which can be attributed to the oxygen related impurity. These results suggest that oxygen might be the dominant impurity in these heterostructure. An experiment

containing sequential dry etching to different etch depths and the subsequent measurement of HLC after each etch step revealed that a notable parallel conduction channel exists in the GaN buffer at the interface of 2nd AlN and 2nd GaN.

In order to decrease the back ground carrier density in the AlGaIn/GaN HEMT heterostructures and improve the buffer resistance, carbon doping of GaN buffer using CBr₄ source was investigated. Two and one orders of reduction in the HLC and VLC were observed, respectively, for the GaN buffer doped with the highest available CBr₄ BEP of 1.86×10^{-7} mTorr. Increased pit density was observed on the surface of the carbon doped GaN buffer, which was attributed to the increased screw type of dislocation density measured using HR-XRD. Carbon doped AlGaIn/GaN HEMT heterostructures with 200 nm thick undoped channel showed only a slight degradation in the 2DEG carrier concentration and mobility compared to undoped HEMT heterostructures. Thus, AlGaIn/GaN HEMT heterostructures with good 2DEG characteristics and two orders reduction in the HLC was achieved by carbon doped GaN buffers using CBr₄ source.

8.4 Advanced HEMT heterostructures

To improve the 2DEG characteristics of GaN based HEMT heterostructures, AlGaIn/GaN/AlGaIn double heterojunction HEMT (DH-HEMT) and InAlN/AlN/GaN HEMT heterostructures were investigated.

8.4.1 AlGaIn/GaN/AlGaIn double heterojunction HEMT

In order to improve the confinement of 2DEG in the channel, AlGaIn/GaN/AlGaIn double heterojunction HEMT was grown on 100-mm Si(111) substrate using ammonia-MBE. The composition of Al in the AlGaIn buffer was kept at 10%. As-grown DH-HEMT showed higher compressive wafer bow and smoother surface morphology compared to SH-HEMT heterostructure. HR-XRD measurements revealed that the buffers of both SH-HEMT and DH-HEMT heterostructures are of similar crystal quality. Hall measurements on DH-HEMT heterostructure showed a room temperature mobility of 1510 cm²/V.s and sheet carrier concentration of 0.97×10^{13} cm⁻². Capacitance-voltage measurements showed that the confinement of 2DEG is improved for the DH-HEMT heterostructure. The enhancement in the room temperature mobility was attributed to the improved confinement of the 2DEG. Both SH-HEMT and DH-HEMT heterostructures showed similar current driving capabilities. However, DH-HEMT showed 3-times higher buffer break down voltage compared to SH-HEMT. Thus, DH-HEMT heterostructure was found to have improved the characteristics of AlGaIn/GaN HEMT heterostructures.

8.4.2 Lattice matched InAlN barrier for GaN based HEMT heterostructures

In order to alleviate the tensile strain in the barrier, growth of lattice matched InAlN barrier for GaN based HEMT heterostructures was attempted. A combination of PA-MBE and Ammonia-MBE growth processes was explored, where the GaN buffer layer on Si substrate was grown by ammonia-MBE and InAlN was grown by PA-MBE. The growth temperature of InAlN was kept at 420 °C and the growths were performed in slightly metal rich growth conditions. As-grown InAlN epilayer of 60 nm thickness showed no phase separation and indium segregation. The mole fraction of indium in the InAlN barrier was optimized to 18%, which is nearly lattice matched to GaN. Further, InAlN/AlN/GaN HEMT heterostructures were grown with the indium mole fraction of 24% and InAlN thickness of 15 nm. Hall measurements showed a room temperature 2DEG carrier concentration of $7.3 \times 10^{12} \text{ cm}^{-2}$ and a mobility of $140 \text{ cm}^2/\text{V.s}$. The obtained 2DEG concentration for InAlN/GaN HEMT heterostructure is too low. Low quality of grown InAlN barrier was identified as the issue. Post growth annealing of the as-grown InAlN/AlN/GaN HEMT heterostructures at 750 °C for 1 hour was found to improve the 2DEG concentration to $1.85 \times 10^{13} \text{ cm}^{-2}$ and mobility to $512 \text{ cm}^2/\text{V.s}$. Improved quality of the barrier layer with the annealing might have improved the 2DEG properties. Thus, InAlN/AlN/GaN HEMT heterostructures with good 2DEG characteristics were achieved on Si substrate by MBE growth technique.

Thus, overall, in this study, nucleation and the growth of nitride epilayers on Si substrate was established using ammonia-MBE growth process. AlN/GaN SMLs were identified as the better stress mitigating layers to achieve crack -free 1 μm thick GaN buffer with good crystal quality and relatively smoother surface morphology on 100-mm Si substrate. Since the as-grown wafers showed high tensile bowing in the range of 30 to 70 μm , optimised AlGaN/AlN/GaN SMLs was used to reduce the bowing to < 34 μm and increase the GaN buffer thickness up to 1400 nm. AlGaN/GaN HEMT heterostructures that were grown using AlN/GaN and AlGaN/AlN/GaN SMLs showed excellent 2DEG characteristics with a good uniformity across the 100-mm wafer. Further, the confinement of the 2DEG in the channel was improved by growing AlGaN/GaN/AlGaN DH-HEMT heterostructure. DH-HEMT heterostructures showed improved 2DEG mobility. Thus, crack free, low wafer bow, good crystal quality smooth surface morphology AlGaN/GaN HEMT heterostructures with good confinement of 2DEG was achieved on 100-mm Si substrate. Further, Growth of InAlN for the lattice matched barrier for GaN based HEMT technology was also explored. InAlN barrier was

successfully grown without any phase separation and indium segregation. The 2DEG characteristics of the as-grown HEMT heterostructures were poor. However, post growth annealing showed enormous improvement in the 2DEG characteristics of InAlN/AlN/GaN HEMT heterostructures on Si substrate. Thus, for the first time, InAlN/AlN/GaN HEMT heterostructures on Si substrate have been demonstrated using MBE growth technique.

9. Future recommendations

In this dissertation, DH-HEMT heterostructure with a thick AlGa_N buffer layer has been explored to achieve the improved confinement of 2DEG in the channel. However, there is an alternative way to improve the confinement of 2DEG in the AlGa_N/Ga_N HEMT heterostructure, which involves the growth of a very thin InGa_N barrier at the back of a Ga_N channel. The polarization field of InGa_N raises the conduction band energy of Ga_N channel region and improves the confinement of 2DEG in the quantum well. Improved mobility of 2DEG and frequency of operations can be achieved using InGa_N back barrier for AlGa_N/Ga_N based HEMT heterostructures. Similar to the growth of InAlN/AlN/GaN HEMT heterostructures, a combination of PA-MBE and ammonia-MBE growth processes can be used to develop AlGa_N/Ga_N/InGa_N HEMT heterostructures.

InAlN barrier of 15 nm thickness has resulted in high 2DEG density in the lattice matched InAlN/GaN HEMT heterostructures. Thinning of the InAlN barrier thickness to much lower values (< 5nm) would result in the reduction of 2DEG concentration. However, ultrathin barriers are required to decrease the device sizes for high power and high speed applications. AlN barrier with ultra-small thickness can simultaneously provide low device dimensions and high 2DEG density of $6 \times 10^{13} \text{ cm}^{-2}$. High spontaneous and piezoelectric polarizations of AlN/GaN system are responsible for high sheet carrier density. High current densities with high frequencies can be achieved using AlN barrier. Using PA-MBE growth process, by optimizing the growth conditions, ultrathin AlN barriers can be grown in a controlled manner on Ga_N buffers.

GaN based HEMT heterostructures that are studied in this dissertation are of Ga polar in nature. However, by reversing the polarity of epitaxial layers, N polar Ga_N based HEMT heterostructures can be obtained. N polar HEMTs have several advantages such as the lower ohmic resistance of contacts, possibility of development of enhancement mode devices and highly scaled transistors. Moreover, high sensitive nature of N polar surface further allows the usage of these HEMT heterostructures in sensor applications. N polar epilayers can be achieved by performing the epitaxial growth on C-face of SiC substrate. Thus, by using MBE growth technique, novel epitaxial heterostructures can be explored in future for a variety of applications.

10. References

- [1] U.K. Mishra, L. Shen, T.E. Kazior, W. Yi-Feng, Proceedings of the IEEE 96/2 (2008) 287.
- [2] L. Liu, J.H. Edgar, Materials Science Engineering R 37/3 (2002) 61.
- [3] J.C. Brice, A.D. Prins, S. Adachi, K. Haruna, D.J. Dunstan, H. Maeta, Properties of indium phosphide. EMIS datareviews series no.6, INSPEC, London, UK, 1991, p. 1.
- [4] G.S. Patrick, Journal of Physics D: Applied Physics 40/20 (2007).
- [5] H. Morkoç, Handbook of Nitride Semiconductors and Devices, Wiley-VCH Verlag GmbH & Co. KGaA, 2009, p. 1.
- [6] B. Paulus, S. Fa-Jian, H. Stoll, Journal of Physics: Condensed Matter 9/13 (1997) 2745.
- [7] A. Zoroddu, F. Bernardini, P. Ruggerone, V. Fiorentini, Physical Review B (Condensed Matter and Materials Physics) 64/4 (2001) 045208.
- [8] F. Yun, M.A. Reshchikov, L. He, T. King, H. Morkoc, S.W. Novak, L. Wei, Journal of Applied Physics 92/8 (2002) 4837.
- [9] A.G. Bhuiyan, A. Hashimoto, A. Yamamoto, Journal of Applied Physics 94/5 (2003) 2779.
- [10] J. Wu, W. Walukiewicz, K.M. Yu, J.W. Ager, E.E. Haller, H. Lu, W.J. Schaff, Y. Saito, Y. Nanishi, Applied Physics Letters 80/21 (2002) 3967.
- [11] A.F. Wright, K. Leung, M. van Schilfgaarde, Applied Physics Letters 78/2 (2001) 189.
- [12] E. Dimakis, A. Georgakilas, M. Androulidaki, K. Tsagaraki, G. Kittler, F. Kalaitzakis, D. Cengher, E. Bellet-Amalric, D. Jalbert, N.T. Pelekanos, Journal of Crystal Growth 251/1-4 (2003) 476.
- [13] S. Nakamura, T. Mukai, M. Senoh, Applied Physics Letters 64/13 (1994) 1687.

- [14] S. Nakamura, M. Senoh, S. Nagahama, x, ichi, N. Iwasa, T. Yamada, T. Matsushita, H. Kiyoku, Y. Sugimoto, T. Kozaki, H. Umemoto, M. Sano, K. Chocho, *Applied Physics Letters* 72/16 (1998) 2014.
- [15] T. Palacios, A. Chakraborty, S. Heikman, S. Keller, S.P. DenBaars, U.K. Mishra, *Electron Device Letters*, 27/1 (2006) 13.
- [16] S. Nakamura, M. Senoh, S. Nagahama, N. Iwasa, T. Yamada, T. Matsushita, H. Kiyoku, Y. Sugimoto, *Japanese Journal of Applied Physics*, 35/1B (1996) 74.
- [17] T. Onuma, S.F. Chichibu, Y. Uchinuma, T. Sota, S. Yamaguchi, S. Kamiyama, H. Amano, I. Akasaki, *Journal of Applied Physics* 94/4 (2003) 2449.
- [18] R.R. Pelá, C. Caetano, M. Marques, L.G. Ferreira, J. Furthmüller, L.K. Teles, *Applied Physics Letters* 98/151907 (2011).
- [19] O. Ambacher, J. Smart, J.R. Shealy, N.G. Weimann, K. Chu, M. Murphy, W.J. Schaff, L.F. Eastman, R. Dimitrov, L. Wittmer, M. Stutzmann, W. Rieger, J. Hilsenbeck, *Journal of Applied Physics* 85 (1999) 3222.
- [20] J.P. Ibbetson, P.T. Fini, K.D. Ness, S.P. DenBars, J.S. Speck, U.K. Mishra, *Applied Physics Letters* 77 (2000) 250.
- [21] J. Jungwoo, J.A. del Alamo, 2006 International Electron Devices Meeting, 11-13 Dec. 2006, IEEE, Piscataway, NJ, USA, 2006, p. 4 pp.
- [22] M. Faqir, G. Verzellesi, G. Meneghesso, E. Zanoni, F. Fantini, *IEEE Transactions on Electron Devices* 55, The Institution of Engineering and Technology (2008) 1592.
- [23] F. Semond, P. Lorenzini, N. Grandjean, J. Massies, *Applied Physics Letters* 78 (2001) 335.
- [24] Y. Awano, M. Kosugi, K. Kosemura, T. Mimura, M. Abe, *IEEE Transactions on Electron Devices* 36 (1989) 2260.
- [25] G.H. Jessen, R.C.J. Fitch, J.K. Gillespie, G. Via, A. Crespo, D. Langley, D.J. Denninghoff, M.J. Trejo, E.R. Heller, *IEEE Transactions on Electron Devices* 54 2007, The Institution of Engineering and Technology (2007) 2589.
- [26] M. Higashiwaki, T. Matsui, *Japanese Journal of Applied Physics*, 43 (2004) 1147.

- [27] J. Kuzmik, IEEE Electron Device Letters 22 (2001) 510.
- [28] F. Medjdoub, J.F. Carlin, M. Gonschorek, E. Feltin, M.A. Py, D. Ducatteau, C. Gaquiere, N. Grandjean, E. Kohn, 2006 International Electron Devices Meeting, IEDM, December 10, 2006 - December 13, 2006, Institute of Electrical and Electronics Engineers Inc., San Francisco, CA, United states, 2006, p. IEEE Electron Devices Society.
- [29] T. Lim, R. Aidam, L. Kirste, P. Waltereit, R. Quay, S. Müller, O. Ambacher, Applied Physics Letters 96/25 (2010) 252108.
- [30] T. Lim, R. Aidam, L. Kirste, P. Waltereit, S. Müller, O. Ambacher, physica status solidi (c) 7/7-8 (2010) 1958.
- [31] T. Lim, R. Aidam, P. Waltereit, W. Pletschen, R. Quay, L. Kirste, Ambacher, Oliver, physica status solidi (c) 8/7-8 (2011) 2439.
- [32] B. Reuters, A. Wille, N. Ketteniss, H. Hahn, B. Holländer, M. Heuken, H. Kalisch, A. Vescan, Journal of Electronic Materials 42/5 (2013) 826.
- [33] E. Calleja, M.A. Sanchez-Garcia, D. Basak, F.J. Sanchez, F. Calle, P. Youinou, E. Munoz, J.J. Serrano, J.M. Blanco, C. Villar, T. Laine, J. Oila, K. Saarinen, P. Hautojarvi, C.H. Molloy, D.J. Somerford, I. Harrison, Physical Review B (Condensed Matter) 58/3 (1998) 1550.
- [34] E.G. Brazel, M.A. Chin, V. Narayanamurti, Applied Physics Letters 74/16 (1999) 2367.
- [35] D. Jena, A.C. Gossard, U.K. Mishra, Applied Physics Letters 76/13 (2000) 1707.
- [36] E. Feltin, B. Beaumont, M. Laugt, P. de Mierry, P. Vennegues, H. Lahreche, M. Leroux, P. Gibart, Applied Physics Letters 79/20 (2001) 3230.
- [37] F. Semond, Y. Cordier, N. Grandjean, F. Natali, B. Damilano, S. Vezian, J. Massies, Fourth International Conference on Nitride Semiconductors, 16-20 July 2001, Wiley-VCH, Germany, 2001, p. 501.
- [38] N.H. Zhang, X.L. Wang, Y.P. Zeng, H.L. Xiao, J.X. Wang, H.X. Liu, J.M. Li, Journal of Crystal Growth 280/3-4 (2005) 346.
- [39] J.W. Johnson, E.L. Piner, A. Vescan, R. Therrien, P. Rajagopal, J.C. Roberts, J.D. Brown, S. Singhal, K.J. Linthicum, IEEE Electron Device Letters 25/7 (2004) 459.

- [40] S.A. Nikishin, N.N. Faleev, V.G. Antipov, S. Francoeur, L. Grave De Peralta, G.A. Seryogin, H. Temkin, T.I. Prokofyeva, M. Holtz, S.N.G. Chu, *Applied Physics Letters* 75/14 (1999) 2073.
- [41] S. Heikman, S. Keller, T. Mates, S.P. Den Baars, U.K. Mishra, *Journal of Crystal Growth* 248 (2003) 513.
- [42] D.S. Green, U.K. Mishra, J.S. Speck, *Journal of Applied Physics* 95/12 (2004) 8456.
- [43] H. Ishikawa, K. Yamamoto, T. Egawa, T. Soga, T. Jimbo, M. Umeno, *Journal of Crystal Growth* 189–190/0 (1998) 178.
- [44] K. Takemoto, H. Murakami, T. Iwamoto, Y. Matsuo, Y. Kangawa, Y. Kumagai, A. Koukitu, *Japanese Journal of Applied Physics, Part 2: Letters* 45/17-19 (2006) L478.
- [45] A. Krost, A. Dadgar, *International Workshop on Nitride Semiconductors (IWN 2002)*, 22-25 July 2002, Wiley-VCH, Germany, 2002, p. 361.
- [46] J.W. Yang, A. Lunev, G. Simin, A. Chitnis, M. Shatalov, M.A. Khan, J.E. Van Nostrand, R. Gaska, *Applied Physics Letters* 76/3 (2000) 273.
- [47] P. Chen, R. Zhang, Z.M. Zhao, D.J. Xi, B. Shen, Z.Z. Chen, Y.G. Zhou, S.Y. Xie, W.F. Lu, Y.D. Zheng, *Journal of Crystal Growth* 225/2–4 (2001) 150.
- [48] H. Amano, N. Sawaki, I. Akasaki, Y. Toyoda, *Applied Physics Letters* 48/5 (1986) 353.
- [49] A. Dadgar, M. Poschenrieder, J. Blasing, O. Contreras, F. Bertram, T. Riemann, A. Reiher, M. Kunze, I. Daumiller, A. Krtischil, A. Diez, A. Kaluza, A. Modlich, M. Kamp, J. Christen, F.A. Ponce, E. Kohn, A. Krost, *Journal of Crystal Growth* 248 (2003) 556.
- [50] O. Briot, J.P. Alexis, S. Sanchez, B. Gil, R.L. Aulombard, *Topical Workshop on III-V Nitrides*, 21-23 Sept. 1995, Elsevier, UK, 1997, p. 315.
- [51] L. Considine, E.J. Thrush, J.A. Crawley, K. Jacobs, W. van der Stricht, I. Moerman, P. Demeester, G.H. Park, S.J. Hwang, J.J. Song, *Metalorganic Vapour Phase Epitaxy 1998. Ninth International Conference*, 31 May-4 June 1998, Elsevier, Netherlands, 1998, p. 192.
- [52] P. Fini, X. Wu, E.J. Tarsa, Y. Golan, V. Srikant, S. Keller, S.P. Denbaars, J.S. Speck, *Japanese Journal of Applied Physics*, 37/8 (1998) 4460.

- [53] S. Li, J. Su, G. Fan, Y. Zhang, S. Zheng, H. Sun, J. Cao, *Journal of Crystal Growth* 310/16 (2008) 3722.
- [54] J. Almlöf, B.J. Deleeuw, P.R. Taylor, C.W. Bauschlicher, P. Siegbahn, *International Journal of Quantum Chemistry* 36/S23 (1989) 345.
- [55] A.N. Wright, C.A. Winkler, *Active Nitrogen*, Academic press, New York, 1968.
- [56] D.I. Slovetskii, *Mechanisms of Chemical Reactions in Nonequilibrium Plasma*, Nauka, Moscow, 1980.
- [57] K. Ohkawa, A. Ueno, T. Mitsuyu, *Journal of Crystal Growth* 117/1–4 (1992) 375.
- [58] A.V. Blant, O.H. Hughes, T.S. Cheng, S.V. Novikov, C.T. Foxon, *Plasma Sources, Science and Technology* 9/1 (2000) 12.
- [59] M.A.L. Johnson, Z. Yu, C. Boney, W.C. Hughes, J.W. Cook, J.F. Schetzina, H. Zhao, B.J. Skromme, J.A. Edmond, *MRS Online Proceedings Library* 449 (1996).
- [60] T.H. Myers, M.R. Millecchia, A.J. Ptak, K.S. Ziemer, C.D. Stinespring, *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures* 17/4 (1999) 1654.
- [61] W.E. Hoke, P.J. Lemonias, D.G. Weir, *Journal of Crystal Growth* 111/1–4 (1991) 1024.
- [62] J.M. Van Hove, G.J. Cosimini, E. Nelson, A.M. Wowchak, P.P. Chow, *Journal of Crystal Growth* 150 (1995) 908.
- [63] R.P. Vaudo, J.W. Cook, J.F. Schetzina, *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures* 12/2 (1994) 1232.
- [64] P. Glaser, D. Sawicka, *Acta Physica Hungarica* 56/1-4 (1984) 111.
- [65] M. Agrawal, Ph.D Thesis, NOVITAS, School of EEE, Nanyang technological university, Singpaore, 2013.
- [66] W.E. Hoke, A. Torabi, J.J. Mosca, R.B. Hallock, T.D. Kennedy, *Journal of Applied Physics* 98/8 (2005) 084510.

- [67] C. Poblentz, P. Waltereit, S. Rajan, U.K. Mishra, J.S. Speck, P. Chin, I. Smorchkova, B. Heying, *Journal of Vacuum Science Technology B* 23/4 (2005) 1562.
- [68] W.E. Hoke, T.D. Kennedy, J.J. Mosca, A.J. Kerr, A. Torabi, S. Davis-Hearns, J.R. LaRoche, *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures* 29/3 (2011) 03C107.
- [69] R. Aidam, E. Diwo, N. Rollbühler, L. Kirste, F. Benkhelifa, *Journal of Applied Physics* 111/11 (2012) 114516.
- [70] K. Radhakrishnan, N. Dharmarasu, Z. Sun, S. Arulkumaran, G.I. Ng, *Applied Physics Letters* 97 (2010).
- [71] B. Heying, R. Averbeck, L.F. Chen, E. Haus, H. Riechert, J.S. Speck, *Journal of Applied Physics* 88/4 (2000) 1855.
- [72] B.L. VanMil, H. Guo, L.J. Holbert, K. Lee, T.H. Myers, T. Liu, D. Korakakis, *Journal of Vacuum Science Technology B* 22/4 (2004) 2149.
- [73] M.A. Sánchez-García, E. Calleja, F.J. Sanchez, F. Calle, E. Monroy, D. Basak, E. Muñoz, C. Villar, A. Sanz-Hervas, M. Aguilar, J.J. Serrano, J.M. Blanco, *Journal of Electronic Materials* 27/4 (1998) 276.
- [74] E. Calleja, M.A. Sánchez-García, F.J. Sánchez, F. Calle, F.B. Naranjo, E. Muñoz, S.I. Molina, A.M. Sánchez, F.J. Pacheco, R. García, *Journal of Crystal Growth* 201–202/0 (1999) 296.
- [75] C. Poblentz, P. Waltereit, S. Rajan, S. Heikman, U.K. Mishra, J.S. Speck, *Journal of Vacuum Science & Technology B* 22/3 (2004) 1145.
- [76] G. Koblmüller, F. Wu, T. Mates, J.S. Speck, S. Fernández-Garrido, E. Calleja, *Applied Physics Letters* 91/22 (2007).
- [77] M. Mesrine, N. Grandjean, J. Massies, *Applied Physics Letters* 72/3 (1998) 350.
- [78] H. Tang, Z.Q. Fang, S. Rolfe, J.A. Bardwell, S. Raymond, *Journal of Applied Physics* 107/10 (2010) 103701.
- [79] V.K. Gupta, K.L. Averett, M.W. Koch, B.L. McIntyre, G.W. Wicks, *Journal of Electronic Materials* 29/3 (2000) 322.

- [80] S.A. Nikishin, V.G. Antipov, S. Francoeur, N.N. Faleev, G.A. Seryogin, V.A. Elyukhin, H. Temkin, T.I. Prokofyeva, M. Holtz, A. Konkar, S. Zollner, *Applied Physics Letters* 75/4 (1999) 484.
- [81] F. Semond, Y. Cordier, N. Grandjean, F. Natali, B. Damilano, S. Vézian, J. Massies, *physica status solidi (a)* 188/2 (2001) 501.
- [82] F. Semond, P. Lorenzini, N. Grandjean, J. Massies, *Applied Physics Letters* 78/3 (2001) 335.
- [83] A. Le Louarn, S. Vézian, F. Semond, J. Massies, *Journal of Crystal Growth* 311/12 (2009) 3278.
- [84] N. Baron, Y. Cordier, S. Chenot, P. Vennéguès, O. Tottereau, M. Leroux, F. Semond, J. Massies, *Journal of Applied Physics* 105/3 (2009) 033701.
- [85] W. Kim, Ö. Aktas, A.E. Botchkarev, A. Salvador, S.N. Mohammad, H. Morkoç, *Journal of Applied Physics* 79/10 (1996) 7657.
- [86] P.I. Cohen, A.M. Johnston, A.M. Dabiran, D.E. Crawford, R. Held, *Surface Review and Letters* 05/03n04 (1998) 913.
- [87] N. Grandjean, J. Massies, *Materials Science and Engineering B: Solid-State Materials for Advanced Technology* 59/1-3 (1999) 39.
- [88] N. Grandjean, M. Leroux, J. Massies, M. Mesrine, M. Laugt, *Japanese Journal of Applied Physics*, 38/2A (1999) 618.
- [89] H. Tang, S. Rolfe, F. Semond, J.A. Bardwell, J.M. Baribeau, *Journal of Crystal Growth* 311/7 (2009) 2091.
- [90] S. Vezian, F. Natali, F. Semond, J. Massies, *Physical Review B (Condensed Matter)* 69/, *The Institution of Engineering and Technology* (2004) 125329.
- [91] W.K. Burton, N. Cabrera, C. Frank, *Philosophical Transactions of the Royal Society of London. Series A (Mathematical and Physical Sciences)* 243/866 (1951) 299.
- [92] A.L. Corrión, C. Pöblenz, F. Wu, J.S. Speck, *Journal of Applied Physics* 103/9 (2008).
- [93] H. Tang, J.B. Webb, J.A. Bardwell, S. Rolfe, T. MacElwee, *Solid-State Electronics* 44/12 (2000) 2177.

- [94] H. Tang, J.B. Webb, J.A. Bardwell, S. Raymond, J. Salzman, C. Uzan-Saguy, *Applied Physics Letters* 78/6 (2001) 757.
- [95] N.H. Zhang, X.L. Wang, Y.P. Zeng, H.L. Xiao, J.X. Wang, H.X. Liu, J.M. Li, *Journal of Crystal Growth* 280/3–4 (2005) 346.
- [96] T. Böttcher, S. Einfeldt, S. Figge, R. Chierchia, H. Heinke, D. Hommel, J.S. Speck, *Applied Physics Letters* 78/14 (2001) 1976.
- [97] A. Krost, A. Dadgar, J. Bläsing, A. Diez, T. Hempel, S. Petzold, J. Christen, R. Clos, *Applied Physics Letters* 85/16 (2004) 3441.
- [98] P. Chaudhari, *Journal of Vacuum Science Technology* 9/1 (1972) 520.
- [99] S. Raghavan, J.M. Redwing, *Journal of Applied Physics* 98/2 (2005) 023514.
- [100] H. Amano, M. Iwaya, T. Kashima, M. Katsuragawa, I. Akasaki, J. Han, S. Hearne, J.A. Floro, E. Cheson, J. Figiel, *Japanese Journal of Applied Physics*, 37/12B (1998) 1540.
- [101] K.E. Waldrip, J. Han, J.J. Figiel, H. Zhou, E. Makarona, A.V. Nurmikko, *Applied Physics Letters* 78/21 (2001) 3205.
- [102] N.H. Zhang, X.L. Wang, Y.P. Zeng, H.L. Xiao, J.X. Wang, H.X. Liu, J.M. Li, *Journal of Crystal Growth* 280/3-4 (2005) 346.
- [103] K. Cheng, M. Leys, S. Degroote, J. Derluyn, B. Sijmus, P. Favia, O. Richard, H. Bender, M. Germain, G. Borghs, *Japanese Journal of Applied Physics* 47/3 (2008) 1553.
- [104] A. Dadgar, J. Christen, T. Riemann, S. Richter, J. Bläsing, A. Diez, A. Krost, A. Alam, M. Heuken, *Applied Physics Letters* 78/15 (2001) 2211.
- [105] E. Feltin, B. Beaumont, M. Laügt, P. de Mierry, P. Vennéguès, M. Leroux, P. Gibart, *physica status solidi (a)* 188/2 (2001) 531.
- [106] E. Feltin, B. Beaumont, M. Laügt, P. de Mierry, P. Vennéguès, H. Lahrèche, M. Leroux, P. Gibart, *Applied Physics Letters* 79/20 (2001) 3230.
- [107] N. Dharmarasu, K. Radhakrishnan, M. Agrawal, L. Ravikiran, S. Arulkumaran, K.E. Lee, N.G. Ing, *Applied Physics Express* 5 (2012) 091003.

- [108] A. Dadgar, J. Blasing, A. Diez, A. Alam, M. Heuken, A. Krost, *Japanese Journal of Applied Physics*, 39/11B (2000) 1183.
- [109] A. Dadgar, C. Hums, A. Diez, J. Bläsing, A. Krost, *Journal of Crystal Growth* 297/2 (2006) 279.
- [110] S.L. Selvaraj, T. Suzue, T. Egawa, *Electron Device Letters*, 30/6 (2009) 587.
- [111] H. Ishikawa, G.Y. Zhao, N. Nakada, T. Egawa, T. Soga, T. Jimbo, M. Umeno, *physica status solidi (a)* 176/1 (1999) 599.
- [112] H. Ishikawa, Z. Guang-Yuan, N. Nakada, T. Egawa, T. Jimbo, M. Umeno, *Japanese Journal of Applied Physics*, 38/5A (1999) 492.
- [113] T. Egawa, B. Zhang, N. Nishikawa, H. Ishikawa, T. Jimbo, M. Umeno, *Journal of Applied Physics* 91/1 (2002) 528.
- [114] M.-H. Kim, Y.-G. Do, H.C. Kang, D.Y. Noh, S.-J. Park, *Applied Physics Letters* 79/17 (2001) 2713.
- [115] K. Cheng, M. Leys, J. Derluyn, S. Degroote, D.P. Xiao, A. Lorenz, S. Boeykens, M. Germain, G. Borghs, *Journal of Crystal Growth* 298/0 (2007) 822.
- [116] S. Tripathy, V.K.X. Lin, S.B. Dolmanan, J.P.Y. Tan, R.S. Kajen, L.K. Bera, S.L. Teo, M.K. Kumar, S. Arulkumaran, G.I. Ng, S. Vicknesh, S. Todd, W.Z. Wang, G.Q. Lo, H. Li, D. Lee, S. Han, *Applied Physics Letters* 101/8 (2012) 082110.
- [117] M. Agrawal, N. Dharmarasu, K. Radhakrishnan, L. Ravikiran, *Thin Solid Films* 520/24 (2012) 7109.
- [118] Y. Cordier, N. Baron, F. Semond, J. Massies, M. Binetti, B. Henninger, M. Besendahl, T. Zettler, *Journal of Crystal Growth* 301–302/0 (2007) 71.
- [119] N. Dharmarasu, K. Radhakrishnan, Z.Z. Sun, M. Agrawal, *physica status solidi (c)* 8/7-8 (2011) 2075.
- [120] W. Seifert, R. Franzheld, E. Butter, H. Sobotta, V. Riede, *Crystal Research and Technology* 18/3 (1983) 383.
- [121] H.P. Maruska, J.J. Tietjen, *Applied Physics Letters* 15/10 (1969) 327.

- [122] P. Boguslawski, E.L. Briggs, J. Bernholc, *Physical Review B* 51/23 (1995) 17255.
- [123] Z.-Q. Fang, D.C. Look, J. Jasinski, M. Benamara, Z. Liliental-Weber, R.J. Molnar, *Applied Physics Letters* 78/3 (2001) 332.
- [124] A.R. Arehart, A. Corrión, C. Poblenz, J.S. Speck, U.K. Mishra, S.P. DenBaars, S.A. Ringel, *physica status solidi (c)* 5/6 (2008) 1750.
- [125] B.K. Meyer, D.M. Hofmann, H. Alves, *Materials Science and Engineering: B* 71/1–3 (2000) 69.
- [126] W. Götz, N.M. Johnson, C. Chen, H. Liu, C. Kuo, W. Imler, *Applied Physics Letters* 68/22 (1996) 3144.
- [127] Y.J. Wang, R. Kaplan, H.K. Ng, K. Doverspike, D.K. Gaskill, T. Ikeda, I. Akasaki, H. Amano, *Journal of Applied Physics* 79/10 (1996) 8007.
- [128] S. Fischer, D. Volm, D. Kovalev, B. Averboukh, A. Graber, H.C. Alt, B.K. Meyer, *Materials Science and Engineering: B* 43/1–3 (1997) 192.
- [129] D. Volm, K. Oettinger, T. Streibl, D. Kovalev, M. Ben-Chorin, J. Diener, B.K. Meyer, J. Majewski, L. Eckey, A. Hoffmann, H. Amano, I. Akasaki, K. Hiramatsu, T. Detchprohm, *Physical Review B* 53/24 (1996) 16543.
- [130] U. Kaufmann, M. Kunzer, H. Obloh, M. Maier, C. Manz, A. Ramakrishnan, B. Santic, *Physical Review B* 59/8 (1999) 5561.
- [131] J.A. Freitas, W.J. Moore, B.V. Shanabrook, G.C.B. Braga, S.K. Lee, S.S. Park, J.Y. Han, *Physical Review B* 66/23 (2002) 233311.
- [132] M. Murthy, J.A. Freitas Jr, J. Kim, E.R. Glaser, D. Storm, *Journal of Crystal Growth* 305/2 (2007) 393.
- [133] C.-Y. Hsu, W.-H. Lan, Y.S. Wu, *Applied Physics Letters* 83/12 (2003) 2447.
- [134] P. Kozodoy, J.P. Ibbetson, H. Marchand, P.T. Fini, S. Keller, J.S. Speck, S.P. DenBaars, U.K. Mishra, *Applied Physics Letters* 73/7 (1998) 975.
- [135] C. Mavroidis, J.J. Harris, R.B. Jackman, I. Harrison, B.J. Ansell, Z. Bougrioua, I. Moerman, *Journal of Applied Physics* 91/12 (2002) 9835.

- [136] C. Mavroidis, J.J. Harris, M.J. Kappers, N. Sharma, C.J. Humphreys, E.J. Thrush, *Applied Physics Letters* 79/8 (2001) 1121.
- [137] E. Frayssinet, W. Knap, S. Krukowski, P. Perlin, P. Wisniewski, T. Suski, I. Grzegory, S. Porowski, *Journal of Crystal Growth* 230/3–4 (2001) 442.
- [138] D.F. Storm, D.S. Katzer, S.C. Binari, E.R. Glaser, B.V. Shanabrook, J.A. Roussos, *Applied Physics Letters* 81/20 (2002) 3819.
- [139] N.I. Kuznetsov, A.E. Nikolaev, A.S. Zubrilov, Y.V. Melnik, V.A. Dmitriev, *Applied Physics Letters* 75/20 (1999) 3138.
- [140] S. Heikman, S. Keller, S.P. DenBaars, U.K. Mishra, *Applied Physics Letters* 81/3 (2002) 439.
- [141] A. Corrion, F. Wu, T. Mates, C.S. Gallinat, C. Poblenz, J.S. Speck, *Journal of Crystal Growth* 289/2 (2006) 587.
- [142] S. Haffouz, H. Tang, J.A. Bardwell, E.M. Hsu, J.B. Webb, S. Rolfe, *Solid-State Electronics* 49/5 (2005) 802.
- [143] C.H. Seager, A.F. Wright, J. Yu, W. Götz, *Journal of Applied Physics* 92/11 (2002) 6553.
- [144] A.F. Wright, *Journal of Applied Physics* 92/5 (2002) 2575.
- [145] J.L. Lyons, A. Janotti, C.G. Van de Walle, *Applied Physics Letters* 97/15 (2010) 152108 (3 pp.).
- [146] T. Ogino, M. Aoki, *Japanese Journal of Applied Physics* 19/12 (1980) 2395.
- [147] D.J. As, U. Köhler, *Journal of Physics: Condensed Matter* 13/40 (2001) 8923.
- [148] R. Armitage, Q. Yang, H. Feick, E.R. Weber, *Journal of Crystal Growth* 263/1–4 (2004) 132.
- [149] S. Rajan, A. Chakraborty, U.K. Mishra, C. Poblenz, P. Waltereit, J.S. Speck, *High Performance Devices, 2004. Proceedings. IEEE Lester Eastman Conference on, 2004*, p. 108.

- [150] R. Aidam, P. Waltereit, L. Kirste, M. Dammann, R. Quay, *physica status solidi (a)* 207/6 (2010) 1450.
- [151] J.B. Webb, H. Tang, S. Rolfe, J.A. Bardwell, *Applied Physics Letters* 75/7 (1999) 953.
- [152] M.R. Ramdani, M. Chmielowska, Y. Cordier, S. Chenot, F. Semond, *Solid-State Electronics* 75/0 (2012) 86.
- [153] S. Haffouz, H. Tang, S. Rolfe, J.A. Bardwell, *Applied Physics Letters* 88/25 (2006).
- [154] M.A. Khan, J.N. Kuznia, J.M. Van Hove, N. Pan, J. Carter, *Applied Physics Letters* 60/24 (1992) 3027.
- [155] M. Asif Khan, A. Bhattarai, J.N. Kuznia, D.T. Olson, *Applied Physics Letters* 63/9 (1993) 1214.
- [156] L. Shen, S. Heikman, B. Moran, R. Coffie, N.Q. Zhang, D. Buttari, I.P. Smorchkova, S. Keller, S.P. DenBaars, U.K. Mishra, *Electron Device Letters, IEEE* 22/10 (2001) 457.
- [157] M. Miyoshi, H. Ishikawa, T. Egawa, K. Asai, M. Mouri, T. Shibata, M. Tanaka, O. Oda, *Applied Physics Letters* 85/10 (2004) 1710.
- [158] N. Maeda, T. Saitoh, K. Tsubaki, T. Nishida, N. Kobayashi, *Applied Physics Letters* 76/21 (2000) 3118.
- [159] A.T. Schremer, J.A. Smart, Y. Wang, O. Ambacher, N.C. MacDonald, J.R. Shealy, *Applied Physics Letters* 76/6 (2000) 736.
- [160] E.M. Chumbes, A.T. Schremer, J.A. Smart, Y. Wang, N.C. MacDonald, D. Hogue, J.J. Komiak, S.J. Lichwalla, R.E. Leoni, III, J.R. Shealy, *Electron Devices, IEEE Transactions on* 48/3 (2001) 420.
- [161] K. Cheng, M. Leys, J. Derluyn, K. Balachander, S. Degroote, M. Germain, G. Borghs, *physica status solidi (c)* 5/6 (2008) 1600.
- [162] K. Cheng, H. Liang, M. Van Hove, K. Geens, B. De Jaeger, P. Srivastava, X. Kang, P. Favia, H. Bender, S. Decoutere, J. Dekoster, J.I. Del Agua Borniquel, S.W. Jun, H. Chung, *Applied Physics Express* 5/1 (2012) 011002.

- [163] Y. Cordier, F. Semond, P. Lorenzini, N. Grandjean, F. Natali, B. Damilano, J. Massies, V. Hoël, A. Minko, N. Vellas, C. Gaquière, J.C. DeJaeger, B. Dessertene, S. Cassette, M. Surrugue, D. Adam, J.C. Grattapain, R. Aubry, S.L. Delage, *Journal of Crystal Growth* 251/1–4 (2003) 811.
- [164] Y. Cordier, M. Hugues, F. Semond, F. Natali, P. Lorenzini, Z. Bougrioua, J. Massies, E. Frayssinet, B. Beaumont, P. Gibart, J.P. Faurie, *Journal of Crystal Growth* 278/1–4 (2005) 383.
- [165] Y. Cordier, F. Semond, M. Hugues, F. Natali, P. Lorenzini, H. Haas, S. Chenot, M. Laügt, O. Tottereau, P. Vennegues, J. Massies, *Journal of Crystal Growth* 278/1–4 (2005) 393.
- [166] M. Higashiwaki, T. Matsui, *physica status solidi (c)* 2/7 (2005) 2598.
- [167] L. Kirste, T. Lim, R. Aidam, S. Müller, P. Waltereit, O. Ambacher, *physica status solidi (a)* 207/6 (2010) 1338.
- [168] J.M. Manuel, F.M. Morales, J.G. Lozano, D. González, R. García, T. Lim, L. Kirste, R. Aidam, O. Ambacher, *Acta Materialia* 58/12 (2010) 4120.
- [169] Ž. Gačević, S. Fernández-Garrido, D. Hosseini, S. Estradé, F. Peiró, E. Calleja, *Journal of Applied Physics* 108/11 (2010) 113117.
- [170] D.S. Katzer, D.F. Storm, S.C. Binari, B.V. Shanabrook, A. Torabi, L. Zhou, D.J. Smith, *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures* 23/3 (2005) 1204.
- [171] K. Jeganathan, M. Shimizu, H. Okumura, Y. Yano, N. Akutsu, *Journal of Crystal Growth* 304/2 (2007) 342.
- [172] Y. Cao, D. Jena, *Applied Physics Letters* 90/18 (2007) 182112.
- [173] T. Takayama, M. Yuri, K. Itoh, T. Baba, J.S. Harris Jr, *Journal of Crystal Growth* 222/1–2 (2001) 29.
- [174] W. Ronghua, L. Guowang, J. Verma, B. Sensale-Rodriguez, F. Tian, G. Jia, H. Zongyang, O. Laboutin, Y. Cao, W. Johnson, G. Snider, P. Fay, D. Jena, H. Grace Xing, *Electron Device Letters*, 32/9 (2011) 1215.
- [175] R. Clos, A. Dadgar, A. Krost, *Physica Status Solidi A* 201/11 (2004) 75.

- [176] T. Metzger, R. Hopler, E. Born, O. Ambacher, M. Stutzmann, R. Stommer, M. Schuster, H. Gobel, S. Christiansen, M. Albrecht, H.P. Strunk, *Philosophical Magazine A (Physics of Condensed Matter: Structure, Defects and Mechanical Properties)* 77 (1998) 1013.
- [177] H. Siegle, G. Kaczmarczyk, L. Filippidis, A.P. Litvinchuk, A. Hoffmann, C. Thomsen, *Physical Review B (Condensed Matter)* 55/11 (1997) 7000.
- [178] M. Kuball, *Surface and Interface Analysis* 31 (2001) 987.
- [179] S. Tripathy, S.J. Chua, P. Chen, Z.L. Miao, *Journal of Applied Physics* 92/7 (2002) 3503.
- [180] V.V. Strelchuk, V.P. Bryksa, K.A. Avramenko, M.Y. Valakh, A.E. Belyaev, Y.I. Mazur, M.E. Ware, E.A. DeCuir, G.J. Salamo, *Journal of Applied Physics* 109/12 (2011) 123528.
- [181] J.L. Rouviere, M. Arlery, B. Daudin, G. Feuillet, O. Briot, *Materials Science and Engineering: B* 50/1–3 (1997) 61.
- [182] W.A. Beck, J.R. Anderson, *Journal of Applied Physics* 62/2 (1987) 541.
- [183] A. Le Louarn, S. Vezian, F. Semond, J. Massies, *Journal of Crystal Growth* 311 (2009) 3278.
- [184] N. Grandjean, M. Leroux, J. Massies, M. Mesrine, M. Laugt, *Japanese Journal of Applied Physics*, 38 (1999) 618.
- [185] H. Tang, Z.Q. Fang, S. Rolfe, J.A. Bardwell, S. Raymond, *Journal of Applied Physics* 107/10 (2010) 103701 (12 pp.).
- [186] A. Dadgar, P. Veit, F. Schulze, J. Blasing, A. Krtschil, H. Witte, A. Diez, T. Hempel, J. Christen, R. Clos, A. Krost, *Thin Solid Films* 515/10 (2007) 4356.
- [187] B.S. Zhang, M. Wu, J.P. Liu, J. Chen, J.J. Zhu, X.M. Shen, G. Feng, D.G. Zhao, Y.T. Wang, H. Yang, A.R. Boyd, *Journal of Crystal Growth* 270/3-4 (2004) 316.
- [188] L. Kung-Liang, E.Y. Chang, H. Yu-Lin, H. Wei-Ching, L. Tien-Tung, W. Yuen-Yee, L. Tingkai, T. Doug, C. Chen-Hao, *Journal of Vacuum Science Technology B* 28/3 (2010) 473.

- [189] Z. Chen, Y. Pei, S. Newman, R. Chu, D. Brown, R. Chung, S. Keller, S.P. Denbaars, S. Nakamura, U.K. Mishra, *Applied Physics Letters* 94/11 (2009) 112108.
- [190] M. Azize, T. Palacios, *Journal of Applied Physics* 108/Copyright 2010, The Institution of Engineering and Technology (2010) 023707 (4 pp.).
- [191] J. Xiong, J. Tang, T. Liang, Y. Wang, C. Xue, W. Shi, W. Zhang, *Applied Surface Science* 257/4 (2010) 1161.
- [192] J. Chen, S.M. Zhang, B.S. Zhang, J.J. Zhu, X.M. Shen, G. Feng, J.P. Liu, Y.T. Wang, H. Yang, W.C. Zheng, *Journal of Crystal Growth* 256/3–4 (2003) 248.
- [193] M.J. Manfra, N.G. Weimann, J.W.P. Hsu, L.N. Pfeiffer, K.W. West, S.N.G. Chu, *Applied Physics Letters* 81/8 (2002) 1456.
- [194] S.L. Sahonta, M.Q. Baines, D. Cherns, H. Amano, F.A. Ponce, *Physica Status Solidi C* 234 (2002) 952.
- [195] P. Cantu, F. Wu, P. Waltereit, S. Keller, A.E. Romanov, U.K. Mishra, S.P. DenBaars, J.S. Speck, *Applied Physics Letters* 83 (2003) 674.
- [196] S. Raghavan, X. Weng, E. Dickey, J.M. Redwing, *Applied Physics Letters* 88/4 (2006) 1.
- [197] R.W. Hoffman, *Thin Solid Films* 34/2 (1976) 185.
- [198] S.A. Nikishin, N.N. Faleev, V.G. Antipov, S. Francoeur, L.G. de Peralta, G.A. Seryogin, H. Temkin, T.I. Prokofyeva, M. Holtz, S.N.G. Chu, *Applied Physics Letters* 75/14 (1999) 2073.
- [199] T. Kimoto, H. Matsunami, *Journal of Applied Physics* 78/5 (1995) 3132.
- [200] M. Schroeder, D.E. Wolf, *Surface Science* 375/1 (1997) 129.
- [201] C. Ratsch, A. Zangwill, *Applied Physics Letters* 63/17 (1993) 2348.
- [202] R. Held, D.E. Crawford, *Surface Review and Letters* 5 (1998) 913.
- [203] T. Zywiets, J. Neugebauer, M. Scheffler, *Applied Physics Letters* 73 (1998) 487.

- [204] J.M. Bethoux, P. Vennegues, F. Natali, E. Feltin, O. Tottereau, G. Nataf, P. De Mierry, F. Semond, *Journal of Applied Physics* 94/10 (2003) 6499.
- [205] J.W. Matthews, A.E. Blakeslee, *Journal of Crystal Growth* 27/1 (1974) 118.
- [206] H. Tang, J.M. Baribeau, G.C. Aers, J. Fraser, S. Rolfe, J.A. Bardwell, *Journal of Crystal Growth* 323/1 (2011) 413.
- [207] V.V. Strelchuk, V.P. Bryksa, K.A. Avramenko, M.Y. Valakh, A.E. Belyaev, Y.I. Mazur, M.E. Ware, E.A. DeCuir, Jr., G.J. Salamo, *Journal of Applied Physics* 109/12 (2011) 123528 (8 pp.).
- [208] G.G. M. Cardona (Ed.), *Light Scattering in Solids II: Basic concepts and Instrumentation*, Springer-Verlag, 1982.
- [209] P. Cantu, F. Wu, P. Waltereit, S. Keller, A.E. Romanov, U.K. Mishra, S.P. DenBaars, J.S. Speck, *Applied Physics Letters* 83/4 (2003) 674.
- [210] A.E. Romanov, J.S. Speck, *Applied Physics Letters* 83/13 (2003) 2569.
- [211] M. Agrawal, N. Dharmarasu, K. Radhakrishnan, L. Ravikiran, *Journal of Crystal Growth* 378/0 (2013) 283.
- [212] S.L. Sahonta, M.Q. Baines, D. Cherns, H. Amano, F.A. Ponce, *physica status solidi (b)* 234/3 (2002) 952.
- [213] W.D. Nix, B.M. Clemens, *Journal of Materials Research* 14/8 (1999) 3467.
- [214] G. Steude, B.K. Meyer, A. Goldner, A. Hoffmann, A. Kaschner, F. Bechstedt, H. Amano, I. Akasaki, *Japanese Journal of Applied Physics*, 38/5 (1999) 498.
- [215] G.H. Olsen, M. Ettenberg, *Journal of Applied Physics* 48/6 (1977) 2543.
- [216] D.C. Look, D.C. Reynolds, J.W. Hemsky, J.R. Sizelove, R.L. Jones, R.J. Molnar, *Physical Review Letters* 79/12 (1997) 2273.
- [217] W.J. Moore, J.A. Freitas, G.C.B. Braga, R.J. Molnar, S.K. Lee, K.Y. Lee, I.J. Song, *Applied Physics Letters* 79/16 (2001) 2570.
- [218] D.W. Jenkins, J.D. Dow, M.H. Tsai, *Journal of Applied Physics* 72/9 (1992) 4130.

- [219] J. Neugebauer, C.G. Van de Walle, *Physical Review B* 50/11 (1994) 8067.
- [220] A.R. Arehart, A. Corrion, C. Poblentz, J.S. Speck, U.K. Mishra, S.A. Ringel, *Applied Physics Letters* 93/11 (2008) 112101.
- [221] W. Grieshaber, E.F. Schubert, I.D. Goepfert, R.F. Karlicek, M.J. Schurman, C. Tran, *Journal of Applied Physics* 80/8 (1996) 4615.
- [222] H. Lahrèche, M. Leroux, M. Laügt, M. Vaille, B. Beaumont, P. Gibart, *Journal of Applied Physics* 87/1 (2000) 577.
- [223] J.R. Haynes, *Physical Review Letters* 4/7 (1960) 361.
- [224] J.R. Haynes, *Physical Review Letters* 17/16 (1966) 860.
- [225] R.E. Halsted, M. Aven, *Physical Review Letters* 14/3 (1965) 64.
- [226] J. Webb, H. Tang, J.A. Bardwell, S. Rolfe, Y. Liu, J. Lapointe, P. Marshall, T.W. MacElwee, *physica status solidi (a)* 188/1 (2001) 271.
- [227] M.E. Vickers, M.J. Kappers, R. Datta, C. McAleese, T.M. Smeeton, F.D.G. Rayment, C.J. Humphreys, *Journal of Physics D: Applied Physics* 38/10A (2005) A99.
- [228] W. Qian, M. Skowronski, K. Doverspike, L.B. Rowland, D.K. Gaskill, *Journal of Crystal Growth* 151/Copyright 1995, FIZ Karlsruhe (1995) 396.
- [229] J. Neugebauer, C.G. Van de Walle, *Applied Physics Letters* 69/4 (1996) 503.
- [230] K. Saarinen, T. Laine, S. Kuisma, J. Nissilä, P. Hautojärvi, L. Dobrzynski, J.M. Baranowski, K. Pakula, R. Stepniewski, M. Wojdak, A. Wyszomolek, T. Suski, M. Leszczynski, I. Grzegory, S. Porowski, *Physical Review Letters* 79/16 (1997) 3030.
- [231] A. Sedhain, J. Li, J.Y. Lin, H.X. Jiang, *Applied Physics Letters* 96/15 (2010) 151902.
- [232] R. Armitage, W. Hong, Q. Yang, H. Feick, J. Gebauer, E.R. Weber, S. Hautakangas, K. Saarinen, *Applied Physics Letters* 82/20 (2003) 3457.
- [233] F. Reurings, F. Tuomisto, *Gallium Nitride Materials and Devices II*, 22 Jan. 2007, SPIE - The International Society for Optical Engineering, USA, 2007, p. 64730.

- [234] A. Armstrong, A.R. Arehart, D. Green, U.K. Mishra, J.S. Speck, S.A. Ringel, *Journal of Applied Physics* 98/5 (2005) 53704.
- [235] F. Demangeot, J. Frandon, M.A. Renucci, O. Briot, B. Gil, R.L. Aulombard, *Solid State Communications* 100/4 (1996) 207.
- [236] S. Choi, E. Heller, D. Dorsey, R. Vetury, S. Graham, *Journal of Applied Physics* 113/9 (2013) 164501.
- [237] J.M. Wagner, F. Bechstedt, *Physical Review B* 66/11 (2002) 115202.
- [238] F. Demangeot, J. Frandon, P. Baules, F. Natali, F. Semond, J. Massies, *Physical Review B (Condensed Matter and Materials Physics)* 69/15 (2004) 155215.
- [239] R.X. Wang, S.J. Xu, S. Fung, C.D. Beling, K. Wang, S. Li, Z.F. Wei, T.J. Zhou, J.D. Zhang, H. Ying, M. Gong, *Applied Physics Letters* 87/3 (2005) 31906.
- [240] W. Götz, N.M. Johnson, H. Amano, I. Akasaki, *Applied Physics Letters* 65/4 (1994) 463.
- [241] H. Harima, S.i. Nakashima, T. Uemura, *Journal of Applied Physics* 78/3 (1995) 1996.
- [242] A.S. Barker, Jr., M. Ilegems, *Physical Review B* 7/2 (1973) 743.
- [243] C.Q. Chen, J.P. Zhang, V. Adivarahan, A. Koudymov, H. Fatima, G. Simin, J. Yang, M. Asif Khan, *Applied Physics Letters* 82/25 (2003) 4593.
- [244] Y. Cordier, F. Semond, M. Hugues, F. Natali, P. Lorenzini, H. Haas, S. Chenot, M. Laügt, O. Tottereau, P. Vennegues, J. Massies, *Journal of Crystal Growth* 278/1–4 (2005) 393.
- [245] I.H. Tan, G.L. Snider, L.D. Chang, E.L. Hu, *Journal of Applied Physics* 68/8 (1990) 4071.
- [246] O. Ambacher, *Journal of Physics D (Applied Physics)* 31/20 (1998) 2653.
- [247] J. Kuzmik, *IEEE Electron Device Letters* 22/11 (2001) 510.
- [248] D.S. Katzer, D.F. Storm, S.C. Binari, B.V. Shanabrook, A. Torabi, L. Zhou, D.J. Smith, *Journal of Vacuum Science & Technology B* 23/3 (2005) 1204.

- [249] S. Fernández-Garrido, Ž. Gačević, E. Calleja, *Applied Physics Letters* 93/19 (2008) 191907.

11. Author's Publications

11.1 Journal Publications

1. **Strain states of AlN/GaN-stress mitigating layer and their effect on GaN buffer layer grown by ammonia molecular beam epitaxy on 100-mm Si(111)**
L. Ravikiran, K. Radhakrishnan, N. Dharmarasu, M. Agrawal and S. Munwar Basha, Journal of Applied Physics **114**, 123503 (2013).
2. **Effect of stress mitigating layers on the structural properties of GaN grown by ammonia MBE on 100 mm Si (111)**
L. Ravikiran, M. Agrawal, N. Dharmarasu, and K. Radhakrishnan, Japanese Journal of Applied Physics **52**, 08JE05 (2013).
3. **Demonstration of AlGaIn/GaN high electron mobility transistors on 100-mm-Diameter Si (111) by ammonia molecular Beam epitaxy.**
N. Dharmarasu, K. Radhakrishnan, M. Agrawal, L. Ravikiran, S. Arulkumaran, K. E. Lee, and N. G. Ing, Applied Physics Express **5**, 091003 (2012).
4. **Structural properties of GaN grown on AlGaIn/AlN stress mitigating layers on 100-mm Si (111) by ammonia molecular beam epitaxy.**
M. Agrawal, N. Dharmarasu, K. Radhakrishnan, and L. Ravikiran, Thin Solid Films **520**, 7109 (2012).
5. **Origin of tensile strain in GaN grown on AlGaIn/AlN stress mitigating layers on 100-mm Si (111) by ammonia molecular beam epitaxy.**
M. Agrawal, N. Dharmarasu, K. Radhakrishnan, and L. Ravikiran, Journal of Crystal Growth **378**, 283 (2012).

11.2 Conference Presentations

1. **Effect of stress mitigating layers on the structural properties of GaN grown by ammonia MBE on 100 mm Si (111).**
L. Ravikiran, M. Agrawal, N. Dharmarasu, K. Radhakrishnan, International Workshop on Nitrides Semiconductors, Oct 14-19, 2012, Sapporo, Japan.
2. **Carbon doped GaN buffer for AlGaIn/GaN HEMT heterostructure using ammonia-MBE.**
L. Ravikiran, K. Radhakrishnan, Munawar Basha, N. Dharmarasu, M. Agrawal, C. M. Manoj Kumar and S. Arulkumaran, International Workshop on Nitrides Semiconductors, Aug 24-29, 2014, Wroclaw, Poland
3. **Growth of AlGaIn/GaN HEMT on 100 mm Si (111) by Ammonia-MBE.**
N. Dharmarasu, K. Radhakrishnan, M. Agrawal, L. Ravikiran, S. Arulkumaran, K.E. Lee, N.G. Ing, International Workshop on Nitrides Semiconductors, Oct 14-19, 2012, Sapporo, Japan.