GROWTH AND CHARACTERIZATION OF GaN NANOSTRUCTURES AND AlGaN/GaN HETEROSTRUCTURES FOR DEVICE APPLICATIONS

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by

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CERTIFICATE

This is to certify that the thesis entitled "**Growth and Characterization of GaN nanostructures and AlGaN/GaN Heterostructures for Device Applications"** being submitted by **Ms. Mansi Agrawal** to Indian Institute of Technology Delhi, New Delhi, for the award of the degree of **Doctor of Philosophy** is a record of bonafide research work carried out by her. She has worked under our supervision and guidance and has fulfilled the requirements for the submission of this thesis, which in our opinion has reached the requisite standard. The results contained in this thesis have not been submitted, in part or full, to any other University or Institute for the award of any degree/diploma.

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Mansi Agrawal

ABSTRACT

The technological advancement and new device concepts in using semiconducting materials such as III-nitrides with exceptional electronic and optical properties have attracted substantial interest worldwide. One of the semiconducting materials is gallium nitride, belonging to the family of III-nitrides and a wide bandgap (3.42eV) semiconductor. It has a diverse range of applications, including military, high power, automobile and optoelectronic applications. The possibility to grow heterostructures (AlGaN/GaN) is another advantage of GaN. A 2DEG is formed in AlGaN/GaN heterostructures resulting from strong spontaneous and piezoelectric polarisation, make them suitable for the next generation high electron mobility transistors. Their efficient device performance is significantly influenced by the surface of these heterostructures. Another significant problem with GaN is the absence of a native substrate, which leads to threading dislocations (10^8 cm^{-2}-10^{-10} cm^{-2}) with high density. However, GaN nanowires are one of the most probable solutions to the lattice mismatch problem. The present thesis is focussed on the growth and characterisation of gallium nitride nanowires on high index silicon substrates, to investigate how surface treatments affect GaN epitaxial films and AlGaN/GaN heterostructures in order to solve device-related issues caused by surface-related problems, and fabricate AlGaN/GaN circular HEMTs on 200 mm silicon and 3" sapphire wafers for sensing applications as the circular geometry provides a large sensing area.

In the present work, firstly MBE technique was used for the growth of gallium nitride nanowires on high index Si (211) substrates without using any catalyst. The synthesis process started with the nitridation of the Si (211) substrates which was carried out at 1170^0C. The nitridation time of the Si (211) substrates was then optimised for the growth of the nanowires. The stepped nature of Si (211) substrates resulted in preferential orientation of the nanowires. The nanowires were characterised using various structural, morphological and optical techniques. The length of the nanowires is approximately 450 nm. The diameter of gallium nitride nanowires had a range from 20 nm to 80 nm. FESEM results confirmed that GaN nanowires are highly dense and oriented at 70^0 with respect to Si (211) substrate. The UV and NOx gas sensing application of the nanowires were also investigated. The nanowires showed a response of 2.2 % to UV and 24% to NOx gas with a fast recovery and excellent reproducibility.

Further growth and characterisation of heterostructures based upon MoS_2-GaN nanowires on Si (111) substrates were carried out. Their structural and vibrational properties were studied. PAMBE technique was used to grow GaN nanowires on Si (111) substrates followed by the deposition of 2D MoS_2 layers on the nanowires by CVD technique. SEM results confirmed that GaN nanowires are decorated by triangular shaped MoS_2 domains. The numbers of MoS_2 layers were investigated using Raman spectroscopy. The difference between A_{1g} and E_{2g} Raman peaks is 24.8 cm^{-1} which indicates the presence of few monolayers of MoS_2 on GaN nanowires.

Thus, the results obtained indicate that GaN nanowires can be integrated with two dimensional MoS_2 layers.

The effect of chemical surface treatments in GaN epitaxial films and AlGaN/GaN heterostructures is investigated. Hot phosphoric acid treatment was used for polarity determination in gallium nitride films which leads to the formation of hexagonal pits in Ga-polar films and dodecagonal pyramids in N-polar films as studied using FESEM. In addition, it was observed that the hydrogen peroxide treatment in AlGaN/GaN heterostructures results in passivation of AlGaN surface and hence can result in improving the device performance. Hydrogen peroxide treatment leads to the increase in PL intensity and decrease in surface barrier height by 0.19 eV as assessed by XPS. Surface photovoltage spectroscopy, which uses surface photovoltage as a parameter, was also used to investigate the impact of hydrogen peroxide on the AlGaN surface.

Gateless circular HEMTs on 200 mm silicon substrates and circular HEMTs on 3” sapphire substrates were fabricated respectively for sensing applications. The saturated drain current (I_{Dsat}) of gateless HEMTs was found to be around 383 mA with a knee voltage at around 6.2 V. The gateless circular HEMTs showed a response ($\delta I_{DS}/I_{DS}$) of about 0.694% at source drain voltage of 100 mV along with a slow recovery time of 2394s due to persistent photoconductivity effect to ultraviolet light at room temperature with excellent repeatability. In addition for the AlGaN/GaN circular HEMTs on sapphire wafers with Pd as a gate metal I_{DSS} was found to be 0.95 A/mm with a knee voltage of 5.6 V. The hydrogen and UV sensing applications of

circular HEMTs were then studied. The HEMTs exhibited a sensitivity of 12.67 % to hydrogen gas and 4.58% to ultraviolet light at room temperature respectively.

The results of the present thesis show that GaN nanowires on high index silicon substrates have immense potential for sensing applications and circular HEMTs are desirable candidates for sensing applications as they provide a large surface area in comparison to conventional HEMTs.

सार

वैश्विक तकनीकी प्रगति ने दुनिया भर में असाधारण इलेक्ट्रॉनिक और ऑप्टिकल गुणों के साथ III-नाइट्राइड जैसे सेमीकंडक्टिंग सामग्रियों पर आधारित उपकरणों की महत्वपूर्ण रुचि को आकर्षित किया है। सेमीकंडक्टिंग सामग्रियों में से एक गैलियम नाइट्राइड है, जो III-नाइट्राइड्स के परिवार से संबंधित है और एक विस्तृत बैंडगैप (3.42eV) सेमीकंडक्टर है। इसमें सैन्य, उच्च शक्ति, ऑटोमोबाइल और ऑप्टोइलेक्ट्रॉनिक अनुप्रयोगों सहित विविध प्रकार के अनुप्रयोगों की विस्तृत श्रृंखला है। हेटरोस्ट्रक्चर (AlGaN/GaN) के बनाने की संभावना GaN का एक और फायदा है। AlGaN/GaN हेटरोस्ट्रक्चर में 2DEG बनता है, जो मजबूत सहज और पीजोइलेक्ट्रिक ध्रुवीकरण से उत्पन्न होता है, जो उन्हें अगली पीढ़ी के उच्च इलेक्ट्रॉन गतिशीलता ट्रांजिस्टर के लिए उपयुक्त बनाता है। उनका कुशल उपकरण प्रदर्शन इन हेटरोस्ट्रक्चर की सतह से काफी प्रभावित होता है। GaN के साथ एक अन्य महत्वपूर्ण समस्या एक देशी सब्सट्रेट की अनुपस्थिति है, जो उच्च घनत्व के साथ अव्यवस्थाओं (10^8 cm^{-2}-$10^{-10}cm^{-2}$) को फैलाने की ओर ले जाती है। हालांकि, GaN नैनोवायर इस समस्या के सबसे संभावित समाधानों में से एक हैं। प्रस्तुत शोध कार्य उच्च सूचकांक सिलिकॉन सबस्ट्रेट्स पर गैलियम नाइट्राइड नैनोवायरों के विकास और लक्षण वर्णन पर केंद्रित है, यह जांच करने के लिए कि सतह से संबंधित समस्याओं के कारण डिवाइस से संबंधित मुद्दों को हल करने के लिए सतह के उपचार GaN एपिटैक्सियल फिल्मों और AlGaN/GaN हेट्रोस्ट्रक्चर को कैसे प्रभावित करते हैं, और गढ़ते हैं। और संवेदन अनुप्रयोगों के लिए 200 मिमी सिलिकॉन और 3 "नीलम वेफर्स पर AlGaN / GAN परिपत्र HEMT का निर्माण किया गया क्योंकि परिपत्र ज्यामिति एक बड़ा संवेदन क्षेत्र प्रदान करती है ।

वर्तमान कार्य में, किसी उत्प्रेरक का उपयोग किए बिना उच्च सूचकांक Si (211) सबस्ट्रेट्स पर गैलियम नाइट्राइड नैनोवायर्स के विकास के लिए सबसे पहले एमबीई तकनीक का उपयोग किया गया था। संश्लेषण प्रक्रिया Si (211) सबस्ट्रेट्स के नाइट्रिडेशन के साथ शुरू हुई जो 1170^0 C पर किया गया था। Si (211) सबस्ट्रेट्स का नाइट्रिडेशन समय नैनोवायरों के विकास के लिए अनुकूलित किया गया था। Si (211) सबस्ट्रेट्स की चरणबद्ध प्रकृति के परिणामस्वरूप नैनोवायरों का अधिमान्य अभिविन्यास हुआ। नैनोवायरों को विभिन्न

संरचनात्मक, रूपात्मक और ऑप्टिकल तकनीकों का उपयोग करके चित्रित किया गया था। नैनोवायरों की लंबाई लगभग 450 nm है। गैलियम नाइट्राइड नैनोवायरों के व्यास की सीमा 20 nm से 80 nm तक थी। FESEM के परिणामों ने पुष्टि की कि GaN नैनोवायर अत्यधिक सघन हैं और Si (211) सब्सट्रेट के संबंध में 70^0 पर उन्मुख हैं। नैनोवायरों के यूवी और एनओएक्स गैस सेंसिंग एप्लिकेशन की भी जांच की गई। नैनोवायर्स ने यूवी के लिए 2.2% और एनओएक्स गैस के लिए 24% की तेजी से रिकवरी और उत्कृष्ट प्रजनन क्षमता के साथ प्रतिक्रिया दी।

Si (111) सबस्ट्रेट्स पर MoS_2-GaN नैनोवायर्स पर आधारित हेटरोस्ट्रक्चर का आगे विकसित और निरूपित किया गया और उनके संरचनात्मक और कंपन गुणों का अध्ययन किया गया। PAMBE तकनीक का उपयोग Si (111) सबस्ट्रेट्स पर GaN नैनोवायरों को विकसित करने के लिए किया गया था, जिसके बाद CVD तकनीक द्वारा नैनोवायर्स पर 2D MoS2 परतों का निक्षेपण किया गया। SEM परिणामों ने पुष्टि की कि GaN नैनोवायरों को त्रिकोणीय आकार के MoS2 डोमेन से सजाया गया है। रमन स्पेक्ट्रोस्कोपी का उपयोग करके MoS2 परतों की संख्या की जांच की गई। A1g और E2g रमन चोटियों के बीच का अंतर 24.8 cm^{-1} है जो GaN नैनोवायरों पर MoS_2 के कुछ मोनोलेयर्स की उपस्थिति को इंगित करता है। इस प्रकार, प्राप्त परिणाम बताते हैं कि GaN नैनोवायरों को दो आयामी MoS_2 परतों के साथ एकीकृत किया जा सकता है।

अध्ययन के अगले चरण में GaN एपीटैक्सियल फिल्मों और AlGaN/GaN हेट्रोस्ट्रक्चर में रासायनिक सतह के उपचार के प्रभाव का अन्वेषण किया गया है। गर्म फॉस्फोरिक एसिड उपचार का उपयोग ध्रुवता निर्धारण के लिए किया गया था, जो गैलियम नाइट्राइड फिल्मों का निर्धारण करता था, जो Ga-ध्रुवीय फिल्मों में हेक्सागोनल गड्ढों के निर्माण की ओर जाता है और N-ध्रुवीय फिल्मों में डोडेकागोनल पिरामिड के रूप में FESEM का उपयोग करके अध्ययन किया जाता है। इसके अलावा, यह देखा गया कि AlGaN/GaN हेटरोस्ट्रक्चर में हाइड्रोजन पेरोक्साइड उपचार के परिणामस्वरूप AlGaN सतह निष्क्रिय हो जाती है और इसलिए डिवाइस के प्रदर्शन में सुधार हो सकता है। हाइड्रोजन पेरोक्साइड उपचार से पीएल तीव्रता में वृद्धि होती है और एक्सपीएस द्वारा मूल्यांकन के अनुसार सतह बाधा ऊंचाई में 0.19 eV की कमी होती है। भूतल

फोटोवोल्टेज स्पेक्ट्रोस्कोपी, जो एक पैरामीटर के रूप में सतह फोटोवोल्टेज का उपयोग करता है, का उपयोग AlGaN सतह पर हाइड्रोजन पेरोक्साइड के प्रभाव की जांच के लिए भी किया गया था।

इसके अलावा, 200 मिमी सिलिकॉन सबस्ट्रेट्स पर गेटलेस सर्कुलर एचईएमटी और 3" नीलम सबस्ट्रेट्स पर सर्कुलर एचईएमटी क्रमशः सेंसिंग अनुप्रयोगों के लिए तैयार किए गए थे। गेटलेस एचईएमटी का संतृप्त ड्रेन करंट (आईडीसैट) लगभग 6.2 V पर नी वोल्टेज के साथ लगभग 383 mA पाया गया था। गेटलेस सर्कुलर एचईएमटी ने उत्कृष्ट पुनरावृत्ति के साथ कमरे के तापमान पर पराबैंगनी प्रकाश के लिए लगातार फोटोकंडक्टिविटी प्रभाव के कारण 2394s के धीमे रिकवरी समय के साथ 100 mV के स्रोत ड्रेन वोल्टेज पर लगभग 0.694% की प्रतिक्रिया (δIDS/IDS) दिखाई। इस के अलावा गेट मेटल के रूप में पीडी के साथ नीलम वेफर्स पर AlGaN/GaN सर्कुलर एचईएमटी में 5.6 V के नी वोल्टेज के साथ आईडीएसएस 0.95 A/mm पाया गया। इसके बाद सर्कुलर एचईएमटी के हाइड्रोजन और यूवी सेंसिंग अनुप्रयोगों का अध्ययन किया गया। एचईएमटी ने क्रमशः कमरे के तापमान पर हाइड्रोजन गैस के लिए 12.67% और पराबैंगनी प्रकाश के लिए 4.58% की संवेदनशीलता प्रदर्शित की।

इस प्रकार, शोध अध्ययन के परिणाम बताते हैं कि उच्च सूचकांक सिलिकॉन सबस्ट्रेट्स पर विकसित किये गए GaN नैनोवायरों में संवेदन अनुप्रयोगों के लिए अपार संभावनाएं हैं और संवेदन अनुप्रयोगों के लिए सर्कुलर एचईएमटी में पारंपरिक एचईएमटी की तुलना में बड़े सतह क्षेत्र के कारण सेंसिंग एप्लिकेशन के लिए संभावनाएं हैं ।

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Symbols

S	Source
D	Drain
G	Gate
ω	Omega
φ	Azimuth
χ	Tilt
R_{sh}	Sheet resistance
μ	Mobility
n_s	2DEG sheet carrier concentration
R_H	Hall co-efficient
q	Electronic charge
E_s	Energy of the scattered photon
E_i	Energy of the incident photon
$E_{K.E.}$	Core-shell electron Kinetic energy
E_{Bin}	Core-shell binding energy
ϕ	Work function
$h\nu$	Energy of X-rays
s	Streak spacing
L	Distance between the screen and the sample
a	Intra-row spacing of the atoms
λ	Wavelength
n	Diffraction order
d_{hkl}	Inter-planar distance
Θ	Angle between the incident X-rays and the sample
2Θ	Angle between the incident X-ray beam and the diffracted beam
NH_3	Ammonia
NO_x	Nitrogen oxide
R_{air}	Resistance of air
R_{gas}	Resistance upon exposure to NO_x gas
R_{UV}	Resistance upon exposure to UV light
A_{1g}	Out-of-plane Raman mode
E_{2g}	In-plane Raman mode
σ	Biaxial stress
$\omega - \omega_0$	Frequency difference in the E_2 high peak positions of GaN nanowires and stress free GaN
k_s	Proportionality constant
N_{screw}	Screw dislocations density
N_{edge}	Edge dislocations density
β_{002}	Tilt
β_{102}	Twist
b_{screw}	Length of Burger vector of screw dislocations

b_{edge}	Length of Burger vector of edge dislocations
E_C	Conduction band minimum
E_F	Fermi level
S_V	Surface recombination velocity
D_p	Diffusion co-efficient of the minority carriers
L_p	Diffusion length of the minority carriers
α	Absorption co-efficient
γ	Ratio of PL intensities of H_2O_2 treated sample and as grown sample
k_B	Boltzmann constant
T	Temperature
I_{ph}	Photo generated current
V_{OC}	Open circuit voltage
I_0	Reverse saturation current
A^*	Richardson's constant
$\emptyset$	Surface barrier height
σ_p	Net polarisation charge
d	Thickness of AlGaN layer
$\in_0$	Permittivity of free space
$\in_{AlGaN}$	Permittivity of AlGaN
ΔE_c	Conduction band offset
I_{Dsat}	Saturated drain current
I_D	Drain current
V_{DS}	Drain source voltage
I_0	Photocurrent after illumination is switched off
I_D	Dark current
β	Decay constant
τ	PPC decay time constant
V_{GS}	Gate source voltage
I_{PPC}	Photocurrent
I_{DSS}	Maximum drain current at gate source voltage of 0 V
gm	Transconductance
I_G	Gate current
η	Ideality factor
ϕ_B	Schottky barrier height
A	Area of the device
m^*	Effective mass of charge carriers
h	Planck’s constant
$N_{D_{C-V}}$	Doping concentration
w	Depth
C	Capacitance
$\in_s$	Dielectric constant
ΔI	Change in drain current
r_1, r_2	Rate constants

Abbreviations

2D	Two- dimensional
2DEG	Two-dimensional electron gas
AP	Atmospheric pressure
AST	Advanced System Technology
BSFs	Basal-stacking faults
CL	Cathodoluminescence
C-V	Capacitance-Voltage
CVD	Chemical vapor deposition
ESCA	Electron spectroscopy for chemical analysis
FE	Field Emission
FWHM	Full width half maximum
GaCl	Gallium monochloride
GaN	Gallium Nitride
GI	Grazing Incidence
H_3PO_4	Phosphoric acid
HEMTs	High electron mobility transistors
HMDS	Hexamethyldisilazane
HR	High resolution
HVPE	Hydride vapour phase epitaxy
ITO	Indium tin oxide
I-V	Current-Voltage
JCPDS	Joint Committee on Powder Diffraction Standards
KPFM	Kelvin probe force microscopy
LA (M)	longitudinal acoustic mode
LEDs	Light emitting diodes
LP	Low pressure
MBE	Molecular beam epitaxy
MIS	Metal insulator semiconductor
MOCVD	Metal organic chemical vapour deposition
MODFETs	Modulation-doped FET
MoS_2	Molybdenum disulphide
MOVPE	Metal organic vapour phase epitaxy
NBE	Near band edge
NWs	Nanowires
PA	Plasma assisted
PBN	Pyrolitic boron nitride
PE	Plasma enhanced
PIPS	Precision Ion Polishing System
PL	Photoluminescence
PMGI	Polymethyl glutarimide
PPC	Persistent photoconductivity effect
QCSE	Quantum confined stark effect

RF	Radio Frequency
RHEED	Reflection high energy electron diffraction
SADs	Selected area diffraction patterns
SBH	Surface barrier height
SEM	Scanning electron microscopy
SPS	Surface photovoltage spectroscopy
SPV	Surface photovoltage
TEGFETs	Two-dimensional electron gas field-effect transistors
TEM	Transmission electron microscopy
UV	Ultraviolet
VBM	Valence band maximum
WBDF	Weak- beam dark field
XPS	X-ray photoelectron Spectroscopy
XRD	X-ray diffraction
2D	Two- dimensional
2DEG	Two-dimensional electron gas
C-V	Capacitance-Voltage
CVD	Chemical vapour deposition
FE	Field Emission
GaCl	Gallium monochloride
GaN	Gallium Nitride
HEMTs	High electron mobility transistors
HVPE	Hydride vapour phase epitaxy
MBE	Molecular beam epitaxy
MOCVD	Metal organic chemical vapour deposition
MODFETs	Modulation-doped FET
MOVPE	Metal organic vapour phase epitaxy
NWs	Nanowires
QCSE	Quantum confined stark effect
SEM	Scanning electron microscopy
TEGFETs	Two-dimensional electron gas field-effect transistors