Materials Characterization: SEM/CL & XPS

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MURI Review Meeting
May 2010
SEM & CL

Scanning Electron Microscope (SEM):
• Incident electron beam bombards sample
• Various electron and photon signals are generated
• Amount and type of signal depends on beam energy and excitation volume

Cathodoluminescence (CL):
• Electron-hole pair recombination can result in both phonons and photons
• The photons generated are the basis of CL
SEM/CL Equipment

FEI NanoSEM 430 with a Gatan MonoCL4 attachment
Microscope Set-up & Beam Path

• Path A: Light bypasses the dispersive system and is directly coupled to the PMT
• Path B: Light is coupled through the dispersive system such that a chosen wavelength reaches the PMT
HEMT Device Structure

- Unstressed device
- Area analyzed includes T-gate and SiN\textsubscript{x} passivation layer
"Non-destructive" analysis is done before and after failure
- Area of interest is in the channel/gate region
- Results shown are of depth dependent CL spectra
- Accelerating voltages were varied from 1 kV to 30 kV
• Several devices stressed and failed in different ways all show the same defect structures (i.e. threading dislocations) due to similar contrast
• Accelerating voltages were too high and images are mainly representative of deep buffer layer
Unstressed device was analyzed to form a baseline for microstructural defects for this study.

15kV 1000x SE and CL images
The penetration depth of the incident beam varies according to the following equation:

\[ R(\mu m) = (0.052 / \rho) \times E^{1.75} \]

With: \( \rho(GaN) = 6.1 \text{g/cm}^3 \)
- 2 kV \( \sim \) 30 nm
- 5 kV \( \sim \) 200 nm
- 30 kV \( \sim \) 3200 nm

The thickness of the passivation layer is \( \sim \) 200 nm
Depth Dependence of CL Signal

- SE image 10000x
- 30 kV CL image
- 20 kV CL Image
- 15 kV CL image
- 10 kV CL Image
- 5 kV CL image
- 3 kV CL image
- 2 kV CL image
- 1 kV CL image
Substrate Defects

20 kV 10000x SE and CL images

- SiN$_x$ passivation
- Mesa
Monochromatic Spectroscopy

- Accelerating voltages:
  - Varied from 1 kV to 30 kV
  - Penetration depths from 8 nm to >3 mm
- Wavelengths scanned:
  - Spectrum from 200 to 700 nm

GaN band edge shows a maximum signal at 10 kV or ~366 nm
Defect Peaks

• Visible defect peaks:
  • Blue luminescence (BL) at ~440 nm
  • Yellow luminescence (YL) at ~556 nm
• The BL signal is stationary until we drop to 5 kV while the YL signal increases steadily with voltage
BL & YL Peaks

BL:
- Disappears at 5 kV due to depth profiling within the top AlGaN/GaN layers
- The two most likely causes of the BL signal are:
  - $O_{Ga}$-related defect
  - Fe from the doped GaN buffer layer

YL:
- Begins to appear at 5 kV and increases with depth (voltage) due to more defects from thicker buffer layer
- Typically associated with $V_{Ga}$-related defects
SEM/CL Conclusions & Future Work

Conclusions:
• Threading dislocations are present at all visible accelerating voltages and are unlikely to be sole contributor to YL and/or BL
• BL and YL are depth dependent
• Depth dependence and passivation layer thickness have significant effect on signal strength and wavelengths detected

Future Work:
• Sample population of unstressed devices to be stressed will include stripping off passivation layer and gate to view the entire channel clearly through CL to determine cause of failure
• High resolution depth profiling
X-ray Photoelectron Spectroscopy (XPS)

- AlGaN surface passivation:
  - Effects of UV ozone exposure time and XPS take-off angle
- Chemical changes of gate metal contact on GaN under device operation/stressing conditions
  - 300°C anneal in UHV to analyze material behavior under typical device operation conditions
  - 10 Å Ni deposited on GaN (Ga peak still visible)
  - 10 Å Pt deposited on GaN (Ga peak still visible)
Previous Surface Passivation Studies

SiN\textsubscript{x} on u-GaN:
- Shift in valence band (VB) of -0.4 eV and Ga 3d peak lost Ga-O portion after vacuum anneal at 200°C
- VB offset recovers to initial value after anneal in air at 200°C and Ga 3d peak recovered Ga-O portion
- Peak positions and relative intensities are nearly identical to pre-vacuum anneal values

SiN\textsubscript{x} on HEMT:
- Shift in VBM of -0.9eV after vacuum anneal and no determined Ga-O bonding in the Ga 3d peak
- VB offset recovers to initial value after air anneal, similar to SiN\textsubscript{x}/u-GaN sample

u-GaN:
- Shift in core level to VB only at anneal temp.
- Returns to pre-annal value upon cool down
- Ga 3d peak shape consistent throughout anneal, no loss of Ga-O portion

Different mechanism for the different samples due to the significant difference in VB shift.
AlGaN Surface Passivation

Ozone effects on AlGaN:
• AlGaN samples placed in UV ozone oven, max. temp. from lamps ~50°C
• O/N ratio is surface dependent → creation of oxynitride layer
• Future studies of SiN\textsubscript{x} passivation layer after ozone exposure

Take-off angle (TOA): angle between energy analyzer and sample surface

TOA = 90°

TOA = 15°
GaN Surface Treatment

Sample Preparation:
- Substrate: 3 µm GaN on sapphire, 2" wafer
- Pretreat:
  - 3 min. 1:1 HCl:DI H₂O, DI H₂O rinse, N₂ dry
  - 25 min. UV ozone exposure
  - 5 min. BOE, DI H₂O rinse, N₂ dry
- MBE anneal: 700°C for 30 min. in UHV
- Wafer cleaved into quarters:
  - One quarter for standard
  - One quarter 10 Å Ni metal deposition
  - One quarter 10 Å Pt metal deposition
  - One quarter set aside for future studies
Ni on GaN Anneal Study

**Pre-vacuum anneal**

Ni 2p$_{3/2}$ Peak Curves:
1. Ni-O
2. Ni satellite
3. Ni metal

**Post-vacuum anneal**

Vacuum anneal 300°C

Curves:
1. Ni-O
2. Ni-O satellite
3. Ni metal
Ni on GaN Anneal Study

Pre-vacuum anneal

Vacuum anneal 300°C

Binding Energy (eV)

Curve Fit Summary
Goodness of Fit: 4.0
# Position Intens
1  530.7317701
2  528.6610116

O 1s Peak Curves:
1. Ga-O
2. Ni-O

Post-vacuum anneal

Curves:
1. Ni-O
2. Ga-O

Curve Fit Summary
Goodness of Fit: 8.
# Position Intens
1  528.771868
2  529.871491

Min: 0Max: 2717
Min: 0Max: 6065
Pt on GaN Anneal Study

Pre-vacuum anneal

Pt 4f Peaks Curves:
1. Pt 4f_{7/2}
2. Pt 4f_{5/2}

Post-vacuum anneal

Vacuum anneal 300°C
XPS Conclusions & Future Work

Conclusions:
• Oxynitride layer on AlGaN possible barrier for SiN$_x$/AlGaN reaction
• Gate metal/GaN chemical changes due to 300°C anneal in UHV:
  • Ni reacts with O from native oxide on GaN $\rightarrow$ material properties of NiO detrimental to Ni gate contact operation
  • Pt does not readily react with O from native oxide on GaN $\rightarrow$ better suited for gate contact operation

Future Work:
• AlGaN UV ozone exposure followed by SiN$_x$ passivation $\rightarrow$ chemical changes due to vacuum anneal
• Introduction of oxynitride layer on GaN before gate metal deposition $\rightarrow$ chemical changes due to vacuum anneal