Epitaxial Growth
Room Davis Hall 101 - Session EG+BG+MD-WeM

Epitaxial III

Moderators: Hari Nair, Cornell University, **Uttam Singisetti**, University of Buffalo, SUNY

9:15am **EG+BG+MD-WeM-4 Growth of α-$(Al_xGa_{1-x})_2O_3$ by Suboxide Molecular-Beam Epitaxy**, ***Jacob Steele***, *K. Azizie, N. Pieczulewski, J. McCandless, D. Muller, H. Xing, D. Jena,* Cornell University*; T. Onuma,* Kogakuin University, Japan*; D. Schlom,* Cornell University (USA) and Leibniz-Institut für Kristallzüchtung (Germany)

Ga_2O_3 has attracted significant interest due to its ultra-wide bandgap, high electron mobility, and large breakdown field. These properties exceed the current benchmarks set by materials such as SiC and GaN, making Ga_2O_3 optimal for next-generation power devices. Still, it has been proposed that the properties of Ga_2O_3 can be extended further by alloying with Al to form $(Al_xGa_{1-x})_2O_3$ which can raise the bandgap to 8.6 eV. This goal presents a challenge for the most researched phase, β, as β-Ga_2O_3 thermodynamically prefers a monoclinic structure and α-Al_2O_3 is stable in the corundum structure. This structural mismatch limits the compositional range and the range of attainable bandgaps. In contrast, α-Ga_2O_3 occupies the corundum structure and has been shown to alloy over the full compositional range, enabling bandgaps from 5.3 - 8.6 eV. One method of growing α-$(Al_xGa_{1-x})_2O_3$ is molecular-beam epitaxy (MBE). MBE is a powerful and highly controllable growth technique for α-$(Al_xGa_{1-x})_2O_3$ thin films with drawbacks being slow growth rates of a few hundred nm/h and narrow adsorption-controlled growth windows. One method to improve the growth rate is the technique of suboxide MBE, which allows growth of β-Ga_2O_3 thin films at rates exceeding 1 µm/h with large adsorption-controlled growth regimes.

We show that suboxide MBE can be used for the epitaxial growth of high quality α-$(Al_xGa_{1-x})_2O_3$ thin films on A plane sapphire substrates over the full range of x at greater than 1 µm/h. For our study, gallium suboxide, Ga_2O, and elemental Al are the MBE sources. The oxidant is 80% distilled ozone which is held at constant pressure (5 x 10-6 Torr) while the Ga_2O and Al fluxes are varied to control composition. We measure the composition of our films with XRD and confirm that we cover the full range of $0 < x < 1$ with vacuum ultraviolet transmittance measurements showing that the bandgaps of our films shift from α-Ga_2O_3 to α-Al_2O_3. We show that the film composition can be controlled directly by the relative ratios of the Ga_2O and Al fluxes. Our films have high structural quality as revealed by the full width at half maximum (FWHM) of rocking curves of the α-$(Al_xGa_{1-x})_2O_3$ films ranging from 11 - 15 arcseconds; these FWHMs are identical to the underlying sapphire substrates. The surfaces of the films are also smooth with RMS roughnesses measured by atomic force microscopy ranging from 0.3 - 1.1 nm on α-$(Al_xGa_{1-x})_2O_3$ films with thicknesses in the 17.8 - 47.8 nm range. We also show our progress with growing α-$(Al_xGa_{1-x})_2O_3$ films over 100 nm thick and with doping using a SiO_2 source.

9:30am **EG+BG+MD-WeM-5 Structural, Electrical, and Thermal Characterization of CIS-MOCVD β-Ga_2O_3 Epitaxial Buffer Layers**, ***Hannah Masten***, Naval Research Laboratory*; G. Alvarez,* Cornell University*; C. Halverson,* Washington State University*; M. Liao, J. Lundh,* Naval Research Laboratory*; F. Alema, A. Osinsky,* Agnitron Technology*; A. Jacobs,* Naval Research Laboratory*; M. Weber,* Washington State University*; Z. Tian,* Cornell University*; K. Hobart, M. Tadjer,* Naval Research Laboratory

Epitaxial growth of β-Ga_2O_3 using metalorganic chemical vapor deposition (MOCVD) has seen great advancements demonstrating high-quality films with low point defect concentrations and high mobility with low doping concentrations [1]. Here, we investigate the impact of buffer layer thickness for these MOCVD epitaxial films on electrical characteristics, thermal conductivity, and defect concentrations.

MOCVD films were grown on Novel Crystal Technology's Fe-doped (010) β-Ga_2O_3 substrates using Agnitron Technology's Agilis close-injection showerhead MOCVD (CIS-MOCVD). The unintentionally doped (UID) buffer layer thickness was varied on the 3 samples: A-300, B-500, and C-1000 nm. The UID layers were followed by a 10 nm thick n^+ (~10^{19} cm^{-3}) Ga_2O_3 layer for improved channel conductivity. A 100 nm highly n^+ layer was selectively regrown following ref. [2]. Ohmic contacts were formed in the regrown areas with an annealed 20/200 nm Ti/Au metal stack (470 °C, 1 min., N_2). Mesa isolation was formed with an etch of ~170 nm. Transmission line measurements (TLM) showed sample C had the lowest specific contact resistance of 2.25×10^{-6} Ω·cm^2 and sample A had the highest of 1.99×10^{-4} Ω·cm^2. Room temperature Hall effect measurement showed similar mobility for B and C of 115-116 cm^2/V·s, while sample A showed a much lower mobility of 71 cm^2/V·s. Samples B and C, both showed high open-gated source-drain current (I_D) (>0.05 A/mm at V_{DS}= 5 V) and low isolation (mesa-mesa) current (I_{iso}) of < 0.1 µA/mm at V_{DS}= 10 V. Sample A (300 nm thick buffer layer), showed 10X lower open-gated I_D and a high I_{iso} of ~3 mA/mm at V_{DS}= 10 V. Higher I_{iso} for samples with thin buffer layers, such as sample A, have been frequently attributed to a peak in Si concentration at the epilayer/substrate interface observed in secondary-ion mass spectroscopy [1]. Here, we offer further insight on this effect via frequency-domain thermoreflectance (FDTR) and positron annihilation spectroscopy (PAS). Preliminary FDTR data showed decreasing thermal conductivity for thicker epilayers. PAS data fitted with a 3-layer model consistently showed higher density of Ga-related vacancies in the epilayers compared to each substrate. More detailed measurements, including XRD and device-level FDTR, will be performed. This preliminary data suggested that MOCVD Ga_2O_3 was affected by both unintentional impurities and point defects in addition to the known issue of interfacial Si accumulation. [1] A. Waseem, *et al., Physica Status Solidi (*A), p. 2200616, 2022. [2] Z. Xia, *et al.,IEEE EDL*, 39(4), 568–571, 2018.

9:45am **EG+BG+MD-WeM-6 Electrical and Optical Properties of Melt-Grown Mn Doped β-Ga_2O_3**, ***Benjamin Dutton***, *C. Remple, J. Jesenovec,* Washington State University*; J. Varley, L. Voss,* Lawrence Livermore National Laboratory*; M. McCluskey, J. McCloy,* Washington State University

Several acceptor dopants have been explored in β-Ga_2O_3 to produce semi-insulating substrates and epitaxial films. Fe and Mg make up the majority of research thus far, however, other transition metals provide potential alternatives for optimized performance. β-Ga_2O_3 bulk single crystals were grown by the Czochralski and vertical gradient freeze methods with a nominal dopant concentration of 0.25 at.% Mn. Ultraviolet-visible-near infrared spectroscopy and photoluminescence revealed polarization and orientation dependent optical absorptions and a unique orange luminescence. All samples were electrically insulating, indicative of acceptor doping on the order of 10^9 – 10^{11} ohm·cm at room temperature. Actual dopant concentrations of the intentionally doped transition metal and background impurities were determined via glow discharge mass spectrometry, indicating the macro-scale segregation behavior. Laser-ablation inductively-coupled plasma mass spectrometry along with photoluminescence mapping revealed micro-scale segregation of impurity ions. Density functional theory calculations were carried out to elucidate likely site-occupancy and the acceptor level of Mn in the band gap.

10:00am **EG+BG+MD-WeM-7 Mg and Zn Counter doping of Homoepitaxial β-Ga_2O_3 Grown by Molecular Beam Epitaxy**, ***Stephen Schaefer***, *K. Egbo, S. Harvey, A. Zakutayev, B. Tellekamp,* National Renewable Energy Laboratory

Gallium oxide has attracted attention as a candidate material for high-power diodes and transistors owing to its wide bandgap and high breakdown voltage. Homoepitaxial β-Ga_2O_3 has been successfully grown by plasma-assisted molecular beam epitaxy, however it is well-documented that unintentional Si donors at the epitaxial interface lead to the formation of an undesirable parasitic conducting channel. Mg and Zn are deep acceptor levels in β-Ga_2O_3 and Mg counterdoping by MBE has been shown to compensate unintentional donor impurities. However counterdoping with other elements such as Zn remains sparsely investigated.

We report on Mg and Zn counterdoping in homoepitaxial β-Ga_2O_3 grown by MBE on (010) Fe-doped (semi-insulating) and (001) Sn-doped (n-type) wafers. A valved cracker source is used for Mg while Zn is evaporated from a conventional effusion cell. Mg- and Zn-doped stacks are measured by secondary ion mass spectroscopy to calibrate the cell temperatures and valve positions to the dopant incorporation. A typical Ga_2O_3 growth temperature is 600 °C and growth rates are 0.47 – 0.70 Å/s. β-Ga_2O_3 samples composed of a ~2 nm Mg- or Zn-doped layer and a 300 nm unintentionally doped layer are grown with dopant fluxes ranging from 3.8×10^{-9} to 2.0×10^{-8} torr. Counterdoped samples grown on (001) Sn-doped and (010) Fe-doped wafers are processed into vertical and lateral Schottky devices, respectively. In both devices the Ohmic contact is formed by stable 5 nm Ti / 100 nm Au annealed under N_2 at 550 °C while the Schottky contact is formed by 30 nm Ni / 100 nm Au. The Schottky devices are characterized by capacitance-voltage (C-V) measurements at 20 kHz.

We find that the C-V characteristics of the vertical Schottky devices grown on (001) Sn-doped Ga_2O_3 show a reduction in residual capacitance and corresponding increase in depletion width at high reverse bias voltage for the Mg-counterdoped sample compared to an undoped control sample grown under identical conditions. Additionally, the I-V characteristic of the Mg doped device exhibits lower reverse leakage current. These findings are

mirrored in lateral Schottky devices grown on (010) Fe-doped Ga_2O_3 where counterdoping with 1.0×10^{-8} torr Zn flux results in approximately ~2× reduction of capacitance and effective carrier concentration while counterdoping with the same Mg flux results in ~5× reduction. The C-V results suggest that Mg and Zn effectively compensate unintentional donors in Ga_2O_3. Experiments including an annealing study of Mg and Zn diffusion in β-Ga_2O_3 are expected to yield insight to the controllability of counterdoping in Ga_2O_3.

10:15am **EG+BG+MD-WeM-8 Optimizing Si Implantation and Annealing in β-Ga_2O_3, *Katie Gann,*** *N. Pieczulewski,* Cornell University*; T. Asel,* Air Force Research Laboratory*; C. Gorsak,* Cornell University*; K. Heinselman,* national renewable Energy Laboratory*; K. Smith, J. McCandless,* Cornell University*; B. Noesges,* Air Force Research Lab*; G. Xing, D. Jena, H. Nair, D. Muller, M. Thompson,* Cornell University

Optimizing the thermal anneal of Si implanted β-Ga_2O_3 is critical for low resistance contacts and selective area doping in advanced device structures. We report the impact of annealing time, temperature, and ambient on the activation of ion-implanted Si in β-Ga_2O_3 at concentrations from 5×10^{18} to 1×10^{20} cm^{-3}, and in β-$(Al_xGa_{1-x})_2O_3$ (x≤15%) at 5×10^{19} cm^{-3}. Nearly full activation (>90%) and high mobilities (>70 cm^2/V-s) are achieved in β-Ga_2O_3 with contact resistances below 0.16 Ω-mm. In β-$(Al_xGa_{1-x})_2O_3$, initial results are promising with moderate activation (50%) and high mobility (60 cm^2/V-s).

UID β-Ga_2O_3 films were grown by plasma assisted MBE on Fe-doped (010) β-Ga_2O_3 substrates; comparable β-$(Al_xGa_{1-x})_2O_3$ films were grown by MOCVD. Si was implanted at multiple energies to yield 65 or 100 nm box profiles with concentrations of 5×10^{18}, 5×10^{19}, or 1×10^{20} cm^{-3}. To understand damage accumulation, low and high temperature implants were also studied. Anneals were performed in a UHV-compatible quartz furnace at 1 bar with well-controlled gas ambients.

To maintain β-Ga_2O_3 stability, P_{O2} must be greater than 10^{-9} bar (based on annealing in vacuum or forming gas). For 5×10^{19} cm^{-3} Si, full activation is achieved for $P_{O2}<10^{-4}$ bar while 5×10^{18} cm^{-3} tolerates ~10^{-2} bar. Water vapor is critical even at 1 ppm; at 25 ppm active carriers are reduced by 10x. Optimal results were obtained with H_2O below 10 ppb. Based on recovery with subsequent “dry” anneals, we propose an OH-mediated defect compensating Si dopants.

Lattice recovery (mobility) occurs for T > 900 °C, with carriers and mobility increasing with temperature to 1050 °C. However, SIMS shows substantial Si diffusion above 1000 °C with 950 °C the optimal anneal temperature. Activation at 950 °C is maximized between 5 and 20 minutes with shorter times exhibiting slightly lower mobilities while longer times result in carrier deactivation; this “over-annealing” behavior occurs at all temperatures and becomes more significant at high concentrations. Room temperature implants to 1×10^{20} cm^{-3} are shown to fully activate under these optimal conditions.

To understand lattice damage recovery, implants at varying temperatures were characterized by XRD, Rutherford Backscattering Channeling (RBS/C), and STEM. XRD showed no second phases under any conditions. RBS/C and STEM showed only partial amorphization with remnant aligned β-Ga_2O_3. We propose a model to explain the efficient activation based on 3D lattice recovery in the absence of full amorphization.

Author Index

Bold page numbers indicate presenter