Program Overview

Room /Time	Jefferson 2-3
MoA	MD-MoA: Process & Devices I

Monday Afternoon, August 8, 2022

Material and Device Processing and Fabrication Techniques

Room Jefferson 2-3 - Session MD-MoA

Process & Devices I

Moderator: Man-Hoi Wong, University of Massachusetts Lowell

1:45pm MD-MoA-1 High Aspect Ratio Ga₂O₃-based Homo and HOeterostructures by Plasma-free Metal-assisted Chemical Etching, *Xiuling Li*, University of Texas at Austin; *H. Huang, C. Chan, J. Michaels*, University of Illinois, Urbana-Champaign INVITED

 β -Ga₂O₃, with an ultra-wide bandgap (UWB) of ~ 4.8 eV and bulk substrate availability, has drawn enormous interests in the power electronics and solar-blind optoelectronics community. Fabricating high-aspect-ratio β -Ga₂O₃ 3D nanostructures without surface damage is essential for next-generation high power and high speed devices. However, dry etch typically damages the surface due to the high-energy ions, while most wet etching techniques can only produce very limited aspect ratios.

Metal-assisted chemical etch (MacEtch) is an unorthodox anisotropic chemical etching method, that defies the isotropic nature of chemical etch through metal catalysis effect and enables site-controlled semiconductor nanostructure fabrication with unpresented aspect ratio (e.g. > 200:1 for Si) and versatility. Since it was first developed for open-circuit porous silicon formation, catalyst-site-specific MacEtch has enabled the formation of nanostructures of a broad range of semiconductors, including silicon, germanium, III-As, III-P, III-N, SiC, and oxides, as well as some of their heterostructures. The versatility of MacEtch is also evident in the unique characteristics of different types of MacEtch. Inverse-MacEtch (i-MacEtch) allows the formation of atomically smooth sidewalls; magnetic-field guided MacEtch (h-MacEtch) enables 3D control of the etching trajectory; Self-Anchored Catalyst MacEtch (SAC-MacEtch) promotes the sidewall verticality for large via by using porous catalyst; UV-assisted MacEtch (UV-MacEtch) makes plasma-free wide-bandgap semiconductor etch possible; and the ultimate vapor phase MacEtch (VP-MacEtch), while maintaining the damage-free nature, truly takes the technology towards scalability and manufacturability, including the successful demonstration of CMOS compatible titanium nitride (TiN) catalyzed etch. The simplicity, versatility, manufacturability, and realistic potential of MacEtch make it wellpositioned to enhance or replace dry etch methods for future generation of 3D transistors, through-silicon-vias, trench memory, thermoelectric, detectors, and photovoltaic devices.

In this talk, high aspect ratio β -Ga₂O₃ nanofins and AlGaO/GOX nanostructures fabricated by inverse metal-assisted chemical etching (MacEtch), under UV light irradiation, will be presented. The etched surface and interface properties, including Schottky barrier height and interface trap density, will be characterized for device applications.

2:15pm MD-MoA-3 Blocking Behavior of N and Fe Ion Implanted β-Ga₂O₃, Bennett Cromer, Cornell University; W. Li, University of California at Berkeley; K. Smith, Cornell University; K. Gann, Cornell University, Iceland; K. Nomoto, Cornell University; N. Hendriks, University of California at Santa Barbara; A. Green, K. Chabak, Air Force Research Laboratory; M. Thompson, D. Jena, G. Xing, Cornell University

 β -Ga₂O₃ is an actively studied material for high power devices, largely due to its high breakdown electric field of 8 MV/cm and commercially available substrates. Due to the lack of effective p-type conduction in Ga₂O₃, state of the art devices utilize implantation of deep acceptors and heterojunctions for edge termination and device isolation. Implantation of deep acceptors such nitrogen and magnesium have yielded semi-insulating behavior under quasi-static conditions[1]. Nitrogen, specifically, exhibited long-lasting charge trapping in partially activated devices which suggests some timedependent blocking ability. [2]. This begs the question: Does the semiinsulating behavior of N-implantation change significantly with frequency? Further, despite being ubiquitous in insulating Ga₂O₃ substrates, Iron has yet to be implanted and studied for select-area isolation. Key information such as activation temperature and frequency response is not known. In this work, we utilize N and Fe ion implantation to fabricate metal-"insulator"-semiconductor, field-plated, and Schottky barrier diode structures to evaluate the resistive behavior of N- and Fe-implanted Ga₂O₃.

Ni- β -Ga₂O₃ diodes were fabricated on (001) 10 μ m HVPE epitaxial substrates. Prior to implantation, photolithography was used to create areas under the anode that were blanket implanted (MIS), partially implanted (field-plated), and un-implanted (SBD). The N- and Fe-implanted samples underwent recovery annealing prior to cathode Ti/Au and anode

Ni/Au contacts deposited by e-beam evaporation, aligned to the implanted regions.

We remark that N-implanted MIS structures exhibit reduced forward current by a factor of 10^3 and no significant enhancement of the breakdown voltage compared to comparative un-implanted SBDs. Pulsed I-V reveals that below pulse-widths of 5 us, corresponding to a frequency of 200 kHz, N-implanted regions behave similarly to bulk Ga₂O₃ and does not act as blocking. Fe-implanted MIS structures, by comparison, exhibit reduced forward current by 10^8 , enhanced breakdown voltage, and minimal dependence on frequency.

Acknowledgements: We acknowledge support from the AFOSR Center of Excellence Program FA9550-18-1-0529. This work was performed in part at the Cornell Nanoscale Facility, a NNCI member supported by NSF grant NNCI-2025233.

[1] M. H. Wong et al., "Acceptor doping of β -Ga2O3 by Mg and N ion implantations," Appl. Phys. Lett., Sep. 2018.

[2] M. Fregolent *et al.*, "Impact of thermal annealing on deep levels in nitrogen-implanted β -Ga2O3 Schottky barrier diodes," *J. Appl. Phys.* Dec. 2021.

2:30pm MD-MoA-4 Evolution and Recovery of Ion Implantation-Induced Damage Zone in β -Ga2O3, *Elaf Anber*, *D. Foley*, *J. Nathaniel*, Johns Hopkins University; *A. Lang*, American Society for Engineering Education; *J. Hart*, Johns Hopkins University; *M. Tadjer*, *K. Hobart*, US Naval Research Laboratory; *S. Pearton*, University of Florida, Gainesville; *M. Taheri*, Johns Hopkins University

β-Ga2O3 has drawn substantial attention due to its large band gap, high electric breakdown field, and high thermal stability [1-4]. These properties make β-Ga2O3 a promising material for application in harsh environments including high temperature and high radiation dose applications [3].However, the structural complexity of β -Ga2O3, including the two different crystallographic positions of Ga, and numerous crystallographic polymorphs, lead to a large number of complex defects which can form due to radiation exposure [4]. While radiation damage of $\beta\text{-}Ga2O3$ has been studied recently, to date, no study has focused on the structural defects arising from ion implantation. Therefore, in this study we examine B-Ga2O3 in three conditions:as-received, as implanted with Ge with a concentration of ~1020 cm-3, and implanted-annealed at 1150°C for 60 seconds [5]. We analyze these samples using analytical electron microscopy via scanning/transmission electron microscopy (S/TEM), and electron energy loss spectroscopy(EELS). Additionally, precession electron diffraction was utilized to measure strain induced from complex defects formed upon radiation. Electron microscopy imaging revealed an isolated band of structural damage after Ge implantation, which extended ~130 nm from the sample surface and corresponds to the projected range of the ions. Electron diffraction demonstrates that the entirety of the damage band is the κ phase, indicating an implantation-induced phase transition from β to $\kappa\text{-}Ga_2O_3.$ Post-implantation annealing at 1150 °C for 60 s under an O_2 atmosphere a return from κ to $\beta;$ however, an ${\sim}17\,\text{nm}$ damage zone remained at the sample surface. These data indicate differences in the electronic/chemical structure beyond the implantation zone (~130 nm) due to the diffusion of Ge into the bulk material, which, in turn, causes a change in material properties[5]. Outstanding questions remain regarding the ability to have a full reversal to the original crystal structure, and the ability to do so represents a major milestone for use of these materials in next generation electronics.

To answer these outstanding questions, in-situ transmission electron microscopy annealing will be performed to study the role of Ge. Implanted Ga2O3 will be analyzed using high resolution electron microscopy imaging for structural analysis, coupled with electron energy loss spectroscopy for chemical analysis. Electronic structure and atomic bonding of Ga-O will be monitored throughout various stages of the annealing treatments.

2:45pm MD-MoA-5 Heterogeneous Integration of Single-Crystal β -Ga₂O₃ and N-Polar GaN Substrates With ZnO Interlayer Deposited by Atomic Layer Deposition, *Zhe (Ashley) Jian*, University of Michigan, Ann Arbor; *C. Clymore*, University of California, Santa Barbara; *D. Agapiou*, University of Michigan, Ann Arbor; *U. Mishra*, University of California, Santa Barbara; *E. Ahmadi*, University of Michigan, Ann Arbor

Recently, β -Ga₂O₃ has attracted great attention as a promising candidate for high power switching applications. However, two main challenges of β -Ga₂O₃ are its relatively low electron mobility (180 cm²V⁻¹s⁻¹) and low thermal conductivity (10-30 W/m·K) [1], [2]. Additionally, p-type doping does not seem feasible currently for this material system. On the other

Monday Afternoon, August 8, 2022

hand, GaN, a well-known wide bandgap semiconductor, has a high electron mobility (2050 cm²V⁻¹s⁻¹), high 2D charge (2DEG) density, moderate thermal conductivity (253 W/m·K), and the availability of p-type doping [3]. Therefore, the integration of β -Ga₂O₃ with GaN can potentially enable the fabrication of novel GaN/Ga₂O₃ high-frequency and high-power devices combining the merits of both GaN and Ga₂O₃ in addition to novel optoelectronic devices.

This work integrated single-crystal (0001) N-polar GaN and (-201) UID β -Ga₂O₃ substrates via wafer bonding using ZnO interlayer deposited by atomic layer deposition (ALD). Both 5×5 mm² GaN and 10×10 mm² Ga₂O₃ substrates were soaked in buffered hydrofluoric acid (BHF) for 30 seconds prior to the deposition of 10 nm-thick ZnO via thermal-ALD at 200 °C. Next, the Ga₂O₃ and GaN samples were placed into contact with each other. The bonding was conducted at 400 °C under the pressure of 4 MPa. The surfaces were 100% fully bonded. After bonding, both the front and back sides of the sample were evaporated by Ti/Au. To investigate the impact of post-annealing temperature on the bonding interfaces, the N-polar GaN/ZnO/Ga₂O₃ test structure was annealed in N₂ for 30 minutes at temperatures of 600 °C and 900 °C.

Temperature dependent I-V measurements were performed on the GaN/ZnO/Ga₂O₃ test structure at temperatures from 300 K to 650 K with step increment of 25 K. As-bonded sample without annealing demonstrated Schottky behavior. The Schottky barrier of around 0.56 eV was extracted from a linear fit to the Richardson plot. After annealing at 600 °C, the I-V curves showed almost linear behaviors, indicating that the test structure could be modeled by a series resistor. The maximum current density of 0.02 A/cm² at V_{bias} = 6 V and the resistance of 1714 Ω were measured at room temperature. Additionally, increasing the annealing temperature to 900 °C caused a substantial change in the I-V-T characteristics.

[1] Z. Guo et al., Appl. Phys. Lett., vol. 106, no. 11, p. 111909, Mar. 2015.

[2] Z. Feng et al., Appl. Phys. Lett., vol. 114, no. 25, p. 250601, Jun. 2019.

[3] H. Shibata et al., Mater. Trans., vol. 48, no. 10, pp. 2782–2786, Oct. 2007.

3:00pm MD-MoA-6 Structural Transformation of β-Ga₂O₃ through Siimplantation, Snorre Braathen Kjeldby, A. Azarov, P. Nguyen, Centre for Materials Science and Nanotechnology, University of Oslo, Norway; V. Venkatachalapathy, Centre for Materials Science and Nanotechnology, University of Oslo and Department of Materials Science, National Research Nuclear University, "MEPhl", Norway; R. Mikšová, Nuclear Physics Institute of the Czech Academy of Sciences, Czechia; A. Macková, Nuclear Physics Institute of the Czech Academy of Sciences and Department of Physics, Faculty of Science, J.E. Purkyně University, Czechia; J. García-Fernández, A. Kuznetsov, Ø. Prytz, L. Vines, Centre for Materials Science and Nanotechnology, University of Oslo, Norway

Implantation doping is important in device fabrication, but the understanding of the process in β -Ga₂O₃ remains incomplete. In particular, recent works have shown that polymorph transformations can occur in ion-implanted β -Ga₂O₃^{1,2}. If such a transformation also occurs for Si- and Sn-implantation, it could potentially impact donor implantation and detrimentally affect device fabrication. On the other hand, the transformation may open new fabrication routes, e.g. templated growth of other Ga₂O₃ polymorphs. In the present work, we undertook a systematic investigation of Si-implantation in Ga₂O₃, focusing on the effects of implantation fluence and post-implantation annealing temperature on the crystal structure of the sample³.

EFG-grown bulk (-201)- β -Ga₂O₃ samples were implanted with 300 keV ²⁸Si⁺ions at room temperature to fluences in the 1 × 10¹⁴-2 × 10¹⁶ Si/cm² range. We then exposed the samples to annealing in air between 300 and 1300 °C. Rutherford backscattering spectrometry in channeling mode (RBS/c) and X-ray diffraction (XRD) were used for characterization after every step. Samples implanted to fluence 2 × 10¹⁶ Si/cm² (as-implanted and annealed at 1100 °C) were characterized with (scanning) transmission electron microscopy [(S)TEM].

For fluences of 1×10^{15} Si/cm² and above, XRD revealed emergence of additional diffraction peaks in the as-implanted samples (Figure 1). We attributed these diffraction peaks to structural transformation in the implanted layer. After annealing at 700 °C, these features disappeared, and the XRD results were consistent with a return to the β -phase, further supported by the RBS/c results.

In (S)TEM, selected area electron diffraction patterns from the implanted layer in the as-implanted sample had hexagonal symmetry, consistent with previous reports for implantation of other species, where it was interpreted as a β -to- κ polymorph transformation^{1,2}. Our data also show a crystalline-to-crystalline phase transformation, although the data were not fully consistent with identification of the transformed structure as κ -Ga₂O₃³. For the annealed sample, (S)TEM revealed that the implanted layer consisted of defective β -Ga₂O₃. Finally, electron energy loss spectroscopy demonstrated accumulation of Si into SiO₂ nanoparticles in the implanted layer after annealing, which could reduce the doping efficiency for high implantation fluences.

1 Azarov, A. et al., Phys. Rev. Lett. 128, 015704 (2022).

2 Anber, E. A. et al., Appl. Phys. Lett. 117, 152101 (2020).

3 Kjeldby, S. B. et al., J. Appl. Phys. 131, 125701 (2022).

3:15pm MD-MoA-7 Electrical Characteristics of *in Situ* Mg-Doped Ga₂O₃ Current-Blocking Layer for Vertical Devices, *Sudipto Saha*, University at Buffalo-SUNY; *L. Meng, A. Bhuiyan, Z. Feng, H. Zhao*, Ohio State University; *U. Singisetti*, University at Buffalo-SUNY

Monoclinic beta-gallium oxide (Ga₂O₃) has recently attracted tremendous interest in power electronics and RF switching applications due to its ultrawide bandgap. Vertical devices are generally preferred over lateral geometries for power electronics applications due to the absence of surface effects. Vertical Ga₂O₃ transistors with a current blocking layer (CBL) could potentially achieve kilo-volt ratings. Due to the deep acceptor nature of Mg in Ga₂O₃, Mg-doped Ga₂O₃ layers can potentially form CBLs, critical for high voltage power devices.

In this work, we demonstrate in-situ Mg doping in MOCVD-grown films. Two vertical n-CBL-n structures, n-Ga₂O₃/Ga₂O₃:Mg/n-Ga₂O₃, and n-Ga₂O₃/ Ga₂O₃:Mg/UID-Ga₂O₃/n-Ga₂O₃, labeled as S1-FD and S2-PD, were grown to study and assess the blocking capability of Mg-doped Ga₂O₃. The thickness of the Mg-doped CBL for S1-FD and S2-PD are 500 nm and 250 nm, respectively. The Mg target doping density is 1x10¹⁹ cm⁻³. Two-terminal structures were fabricated, and systematic electrical diode characterizations were performed to compare the electrical properties of the two structures. While sweeping voltage from -10 V to 10 V, S1-FD showed better current blocking capability compared to S2-PD, indicating the dependence of Mg-doped layer thickness on the current blocking capability. With the increase of temperature up to 300 °C, the forward blocking voltage (V_{fb}) decreased from 10 V to 5.53 V for S1-FD, whereas V_{fb} remains pretty unchanged (~6 V) for S2-PD with the rise of temperature. S1-FD structure showed a higher reverse leakage current (0.14 nA/ μ m² at -10 V) compared to S2-PD (1.5 x 10^{-4} nA/ μ m² at -10 V). S2-PD gave a destructive reverse breakdown voltage of 35 V with ~ 1.6 MV/cm average field strength.

From the analysis of the device *J*-*V*s of the two structures, it's hypothesized that with the increased Mg-doped CBL thickness, the forward blocking capability increases but the reverse leakage current increases as well. Further investigation of the interplay between leakage and blocking voltage due to Mg dopants is needed. TCAD simulation of the current-voltage characteristics shows that the effective acceptor doping is $1x10^{17}$ cm⁻³. Optimization of the growth conditions can increase the accepter activation efficiency. The development of an *in situ* acceptor doping technology for Ga₂O₃ creates unique opportunities for designing and engineering a variety of high-voltage Ga₂O₃ devices.

Author Index

