Epitaxial Growth
Room Davis Hall 101 - Session EG-MoM

Bulk/Epitaxial I
Moderator: Hongping Zhao, Ohio State University

10:45am **EG-MoM-10 Advances in the MOCVD Growth of β-Ga_2O_3 and Related Heterostructures**, ***Andrei Osinsky,*** Agnitron Technonolgy, Inc.*; F. Alema,* Agnitron Technology, Inc. **INVITED**

β-Ga_2O_3 has attracted extensive interest in power electronic applications owing to its large bandgap of ~ 4.9 eV, estimated high breakdown field of ~ 8 MV/cm, and availability of melt grown high quality β-Ga_2O_3 substrates. The growth of high-quality epitaxial films with low dislocation density and background impurity is critical to realize the projected device performances. Available epitaxial methods to grow β-Ga_2O_3 thin films include MBE, HVPE, and MOCVD. But, despite coming late to the field, the MOCVD method has proven to be suitable for producing high-quality epitaxial β-Ga_2O_3 films at a fast growth rate with uniform and controllable doping [1]. The highest purity β-Ga_2O_3 films have been reported from MOCVD with record low-temperature electron mobility exceeding 23,000 cm^2/Vs and low~10^{13} cm^{-3} compensating acceptors [2]. Also, a recent record-breaking result for lateral Ga_2O_3 MESFETs with a lateral figure of merit (LFOM) of 355 MW/cm^2 and a breakdown voltage of ~2.5 kV [3], and a record low specific contact resistance ~10^{-7} Ωcm[24] were reported based on MOCVD grown epitaxial Ga_2O_3 films.

This presentation will discuss recent progress in the growth of high-quality β-Ga_2O_3 thin films and related materials using MOCVD. The use of Ga precursors, including triethylgallium (TEGa) and trimethylgallium (TMGa), for the growth of Ga_2O_3 will be presented. Their advantages and disadvantages in realizing high-purity, carbon-free, epitaxial Ga_2O_3 films will be discussed. Critical process conditions and MOCVD reactor geometries on achieving high purity β-Ga_2O_3 films with high electron mobility and low background carrier concentration, including doping control in this range, will be discussed. This paper will also discuss the MOCVD growth of high Al composition (up to 30%) high quality strained β-$(AlGa)_2O_3/Ga_2O_3$ heterostructures and superlattices on various orientations of β-Ga_2O_3 substrates. The MOCVD growth of heavily doped (>10^{20} 1/cm^3), highly conductive β-Ga_2O_3, andstrained β-$(Al_xGa_{1-x})_2O_3$/β-Ga_2O_3 heterostructures will be presented. We will also present the demonstration of record low resistance Ohmic contacts on heavily Si doped epitaxial β-Ga_2O_3 and strained β-$(Al_xGa_{1-x})_2O_3$ epilayers with varying Al composition. A recent in-situ non-destructive etching of Ga_2O_3 in MOCVD followed by a regrowth process will also be discussed.

[1] F. Alema *et al.*, Journal of Crystal Growth 475 (2017) 77-82.

[2] G. Seryogin *et al.,* Applied Physics Letters 117 (2020) 262101.

[3] A. Bhattacharyya *et al.*, IEEE Electron Device Letters 42 (2021) 1272-1275.

[4] F. Alema *et al.,* EEE Electron Device Letters 43 (2022) 1649-1652.

11:15am **EG-MoM-12 MOVPE of (100) β-Ga2O3 for Vertical Power Devices - Challenges to Epitaxial Growth Process**, ***Andreas Popp,*** *T. Chou, S. Bin Anooz, R. Grüneberg, V. Thuy, J. Rehm, A. Akhtar, Z. Galazka, P. Seyidov, K. Irmscher,* LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e.V, Germany*; M. Albrecht,* LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e., Germany*; A. Fiedler,* LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e.V, Germany

Beta-gallium oxide (β-Ga_2O_3) is a promising ultra-wide bandgap (~ 4.8 eV) semiconductor material for the application field of power electronic converters. The theoretical breakdown field strength of up to 8 MV/cm[1] can be best exploited using a vertical architecture for β-Ga_2O_3 based transistors.A high-quality homoepitaxial drift layer plays a crucial role in such a vertical device structure. Here, we report our developed process via metal-organic vapor phase epitaxy (MOVPE) to overcome the main issue associated with the homoepitaxial drift layer: (a) low doping (10^{16} cm^{-3} range) concentrations, (b) layer thicknesses of several µm while maintaining a low density of structural and point defects and (c) high growth rates (µm/h).

A high growth rate process of up to 1.5 µm/h was achieved for Si-doped (100) β-Ga_2O_3 homoepitaxial films grown via MOVPE on Czochralski-grown semi-insulating[2,3] and conductive[4] (100) 4° off β-Ga_2O_3 substrates while maintaining the step-flow growth mode up to a film thickness of 4 µm[5]. The enhanced diffusion channel due to forming a Ga adlayer was proposed as the possible growth mechanism[6]. Furthermore, we also report the formation of parasitic particles as a killer issue during the growth, which can be suppressed by a close showerhead to substrate gap and a high total gas flow[7]. With our optimized process, Si doping enabled precise control of the n-type conductivity of the layers with free electron concentrations ranging from 5 × 10^{16} $cm^{-3}$to 1.5 × 10^{19} $cm^{-3,}$ and corresponding mobilities from 163 $cm^2 \cdot V^{-1} \cdot s^{-1}$ to 21 $cm^2 \cdot V^{-1} \cdot s^{-1}$ were measured by room temperature Hall measuerements. Temperature-dependent Hall measurements let expect a low background compensating acceptor concentration of 4 x $10^{15}cm^{-3}$, indicating a doping level down to mid $10^{15}cm^{-3}$ is still possible.

[1]M. Higashiwaki et al., *Semicond. Sci. Technol.* **2016**, *31*, 34001.

[2]Z. Galazka et al., *Prog. Cryst. Growth Charact. Mater.* **2021**, *67*, 100511.

[3]P. Seyidov et al., *APL Materials* **2022**, *10, 111109* (2022)

[4]Z. Galazka et al., *Appl. Phys. Lett.* **2022**, *120*, 152101.

[5]T.-S. Chou et al., *Jpn. J. Appl. Phys.* **2023**, *62*, SF1004.

[6]T.-S. Chou et al., *AIP Adv.* **2021**, *11*, 115323.

[7]T.-S. Chou et al., *Appl. Phys. Lett.* **2023**, *122*, 052102.

11:30am **EG-MoM-13 MOCVD Epitaxy of (010) β-Ga_2O_3 with Fast Growth Rate and the Role of Carbon in Charge Compensation**, ***Lingyu Meng,*** *A. Bhuiyan, D. Yu, H. Zhao,* The Ohio State University

β-Ga_2O_3 has emerged as a promising semiconductor candidate for future power electronic and radio frequency device applications, because of its ultra-wide bandgap (4.8 eV) and high critical field strength (8 MV/cm). Previously, metalorganic chemical vapor deposition (MOCVD) of β-Ga_2O_3 using triethylgallium (TEGa) as the Ga precursor has been demonstrated with record electron mobilities approaching the theoretical values. The use of trimethylgallium (TMGa) as the Ga precursor with higher vapor pressure enables the MOCVD growth of β-Ga_2O_3 with much faster growth rates (> 3µm/hr) for thick film growth.

In this work, a systematic study of the MOCVD growth of (010) β-Ga_2O_3 using TMGa was performed. Both TMGa molar flow rate and growth temperature play an important role on the β-Ga_2O_3 MOCVD growth rate. With a TMGa molar flow rate of 116 µmol/hr, β-Ga_2O_3 fast growth rate of 6.7 µm/hr was achieved at 950°C. The surface morphologies of the MOCVD β-Ga_2O_3 films were highly dependent on the growth conditions, particularly chamber pressure. For films grown at a growth rate of ~3 µm/hr, record room temperature electron Hall mobilities ranging between 190 cm^2/Vs and 93 cm^2/Vs were achieved with carrier concentrations between 1.6×10^{16} and 3.8×10^{19} cm^{-3}. Temperature-dependent charge transport characteristics revealed a low charge compensation level (~1.5×10^{16} cm^{-3}) and a decent low-temperature peak electron mobility (3425 cm^2/Vs at 53K).

The impurities incorporation in the MOCVD grown (010) β-Ga_2O_3 thin films with different growth rates was probed by the quantitative secondary-ion mass spectroscopy (SIMS). SIMS results revealed that both [C] and [H] increase as the TMGa molar flow rate/growth rate increases. However, [C] increases at a much faster rate as compared to [H]. By comparing the electron Hall measurement results and the quantitative SIMS characterization, the net impurity concentration ([C]-[H]) matches well with the compensation level in the MOCVD β-Ga_2O_3 grown at the different growth rates. Therefore, the incorporated H forms neutral C-H complexes and thus passivates the compensation effect from pure C. This mechanism has been proposed from theoretical study based on the DFT calculations.

In summary, MOCVD growth of (010) β-Ga_2O_3 using TMGa as the Ga precursor was systematically studied. The role of C compensation and passivation from C-H complexes in the MOCVD grown films were proposed based on the experimental evidence.

Acknowledgment: The authors acknowledge the funding support from AFOSR (FA9550-18-1-0479) and the NSF (No. 2231026, No. 2019753).

11:45am **EG-MoM-14 Controllable Deep Acceptor Doping in MOCVD β-Ga_2O_3 to Compensate Parasitic Interface Charges**, ***Fikadu Alema,*** Agnitron Technology*; T. Itoh,* Materials Department, University of California, Santa Barbara*; W. Brand, A. Osinsky,* Agnitron Technology*; J. Speck,* Materials Department, University of California, Santa Barbara

One of the challenges in developing β-Ga_2O_3 lateral devices is the presence of parasitic charges at the epilayer-substrate interface due to the Si impurity that accumulates at the interface. One method that has been proposed recently to manage the interface Si is to etch the β-Ga_2O_3 substrate in hydrofluoric acid (HF) for an extended time before the growth of the films [1]. This method does not eliminate the Si at the interface but

reduces its concentration just below the concentration of Fe in the substrate, leading to partial compensation. The other method to manage the interface Si is to compensate with deep acceptor dopants, such Mg, Fe, and N, among which N is less affected by thermal diffusion, making it a dopant of choice [2].

In this work, we report on controllable doping of N into Ga_2O_3 using nitrous oxide (N_2O) and ammonia diluted in nitrogen (NH_3/N_2) as sources for N. The incorporation efficiency, reproducibility, and controllability of N doping into Ga_2O_3 films will be discussed as a function of process conditions for both sources. Incorporating N into Ga_2O_3 is found to be sensitive to process conditions when N_2O is used as a source. A maximum N concentration of ~2×1019 cm-3 was achieved by growing the layer at a low substrate temperature. However, using NH_3/N_2, the doping of N into Ga_2O_3 is controllable through the molar flow rate. With the increase in the NH_3/N_2 molar flow rate from ~1.8×10^{-8} mol/min to 1.45×10^{-6} mol/min, the N impurities incorporated into the β-Ga_2O_3 layers increased from ~1×10^{18} cm^{-3} to ~2×10^{20} cm^{-3}. For both sources, hydrogen was found to incorporate into the films along with N, but annealing at reduced pressure helps to drive out the hydrogen from the film. For the NH_3/N_2 source, growing the layers at elevated temperatures (>900 C) also effectively reduced H incorporation into the film by as much as ~10× with no effect on the incorporation of N. This paper will also discuss the impact of exposing the surface of Ga_2O_3 substrate to NH_3/N_2 and halide based precursors to manage the interface Si.

[1] A. Bhattacharyya etal. C APL Materials 11, 021110 (2023).

[2] M.H. Wong et.al. Applied Physics Letters 113 , 102103 (2018).

12:00pm **EG-MoM-15 Si Accumulation on Ga_2O_3 Surfaces**, ***Jon McCandless***, *C. Gorsak, V. Protasenko, D. Schlom, M. Thompson, H. Xing, H. Nair, D. Jena,* Cornell University

In 2022, we demonstrated a high degree of doping control ($1x10^{17}$ to $1x10^{20}/cm^3$) in *β*-Ga_2O_3 films grown by molecular beam epitaxy with mobilities of ~130 cm^2/Vs in the lowest doped samples.[1] However, we discovered some samples where, despite being grown under the same conditions, the mobility would be ~1 cm^2/Vs. To understand a potential impurity related origin of these low mobilities, secondary ion mass spectrometry (SIMS) was performed. Over 8 samples, we observed significant Fe variation within the substrate and significant Si variation at the surface. Depending on the substrate, the surface Si may or may not have been fully compensated by the Fe. In the worst case, a high-density of uncompensated free carriers exist at the interface with a low mobility which in turn affects the Hall effect measurement. Moreover, this same uncompensated charge can prevent confinement, or create a parallel conducting path for 2D electron gasses, thereby hindering the performance of high electron mobility transistors.[2]

The Si contamination is thought to arise from the polishing process and/or from siloxanes adsorbed from the air, and which are particularly difficult to remove.[3]

To quantify and study the Si accumulation and possible removal strategies, we investigated how Si accumulates on the film surface when exposed to air. We grew UID layers by molecular beam epitaxy, removed the sample and exposed it to air for different amounts of time. After less than 20 minutes of exposure to air, the accumulated Si on a clean surface had a sheet density (n_s) of ~$2x10^{12}/cm^2$. The n_s continued to increase with longer exposure times in air, saturating at ~ $7x10^{12}/cm^2$ after an 8-hour exposure. Next, etching studies were performed to investigate possible removal of the SiO_x on the surface. *β*-Ga_2O_3 films were grown by metal-organic chemical vapor deposition and the surface was exposed to air for 2 hours to allow for the accumulation of Si. The surface was then etched in HF (49%) for varying times to remove the accumulated Si. After etching for 15 minutes, the Si sheet charge was reduced by ~1 order of magnitude to~ $3x10^{11}/cm^2$.

Lastly, simulations were carried out to investigate compensation doping. Under the worst case, (i.e. low Fe density within the substrate and high Si density at the interface), the required compensation would have to be > $10^{20}/cm^3$, but at this Fe density the film quality degrades. Therefore, we believe the optimal solution requires HF etching along with compensation doping.

1. APL, 121, 072108 (2022); 2. APEX, 10, 071101 (2017); 3. Chemosphere,92, 905-910 (2013)

Epitaxial Growth
Room Bansal Atrium - Session EG-MoP
Epitaxial Growth Poster Session I

EG-MoP-1 A Study of the Critical Thickness for Phase Transition of α-Gallium Oxide Grown on Sapphire Substrates by MOCVD, ***Cheng-Han Lee,*** *C. Gorsak, H. Nair,* Department of Materials Science and Engineering, Cornell University

The ultra-wide bandgap semiconductor material α-Ga_2O_3 exhibits promising potential for applications in next-generation high-power electronics and deep-ultraviolet photodetectors. Its large bandgap of 5.3 eV enables a high breakdown voltage and a potentially high Baliga's figure of merit while its rhombohedral corundum structure allows heteroepitaxy of α-Ga_2O_3 on inexpensive isostructural sapphire substrate. One of the major challenges is achieving thick, phase-pure α-Ga_2O_3 films using metal-organic chemical vapor deposition (MOCVD) due to the metastable nature of α-Ga_2O_3. Metastable α-Ga_2O_3 has a strong tendency to revert back to the thermodynamically stable monoclinic β-Ga_2O_3 phase. This gives rise to the natural question: Can epitaxial growth of single-phase α-Ga_2O_3 with high crystalline quality be achieved via MOCVD and how does the critical thickness of α-Ga_2O_3 films depend on MOCVD growth parameters and different surface orientations of sapphire substrates, and what is the mechanism for phase transformation to β-Ga_2O_3?

In this study, we investigate the critical thickness for structural phase transition of MOCVD-grown α-Ga_2O_3 epitaxial thin films grown on c-, m-, r-, and a-plane sapphire substrates. To ensure high crystallinity and to suppress defect generation, we develop a two-step growth method in which a 10-nm-thick α-Ga_2O_3 low-temperature nucleation layer was deposited at 600°C at a low growth rate of 0.4 μm/hr, followed by a high temperature growth of Ga_2O_3 epilayer with thickness ranging from 100 nm to 1600 nm at 600~800°C with a higher growth rate of 0.7~1.0 μm/hr. X-Ray diffraction (XRD) was utilized to confirm the phase purity of α-Ga_2O_3 films and to evaluate crystalline quality based on rocking curve Full-Width-at Half Maximum (FWHM). We successfully demonstrated that single-phase, highly-crystalline α-Ga_2O_3 films with thickness ~400 nm can be achieved on m-plane sapphire substrate through a low-pressure, low-temperature, and high growth rate heteroepitaxial growth, resulting in one of the highest α-Ga_2O_3 critical thickness ever reported on m-plane α-Al_2O_3 via MOCVD.

This work highlights the importance of selecting suitable differently oriented sapphire substrates for the growth of phase-pure α-Ga_2O_3 epitaxial thin films using MOCVD. We pursue to further increase the critical thickness and aim to delineate the mechanism for phase transformation of α-Ga_2O_3. Thicker films and controllable doping of α-Ga_2O_3 thin films can potentially pave the way for the fabrication of α-Ga_2O_3 based high-power electronic devices.

EG-MoP-2 Epitaxial Growth of β-Ga_2O_3 Films on Mgo Substrate via Mist Chemical Vapor Deposition Method, ***Takumi Ikenoue,*** Kyoto University, Cronell University, Japan*; Y. Cho, V. Protasenko, C. Savant, B. Cromer,* Cornell University*; M. Miyake, T. Hirato,* Kyoto University, Japan*; M. Thompson, D. Jena, H. Xing,* Cornell University

Gallium oxide (Ga_2O_3) is expected to be a promising material for power devices due to its wide bandgap and high Baliga figure of merit. Various methods of epitaxial growth have been explored for β-Ga_2O_3, including growth on MgO substrates which possess a larger bandgap than β-Ga_2O_3 and sufficient insulation. MgO has the same crystal structure as NiO, which is expected to be a p-type wide-gap oxide semiconductor, and has a small lattice mismatch, making it the most promising candidate for the substrate for epitaxial growth of NiO. Consequently, the growth of β-Ga_2O_3 on MgO is important, particularly for the development of devices that combine NiO and β-Ga_2O_3. We previously conducted a study on the growth of NiO on MgO substrates using mist chemical vapor deposition (CVD). This oxide film growth technique utilizes a solution as a source under atmospheric pressure and is known for its high quality and productivity. By using this method, we showed the ability to not only produce high-quality NiO(001) single crystals on MgO(001), but also achieve conductivity control by using Li as a dopant. The growth of β-Ga_2O_3 on MgO would lead directly to heterojunction formation with NiO, which has almost the same lattice constant as MgO. Therefore, in this study, we report the growth of β-Ga_2O_3 on MgO(001) substrates using the mist CVD method.

Prior to growth, MgO substrates were ultrasonically cleaned with acetone and methanol. The precursor used for β-Ga_2O_3 growth was gallium acetylacetonate, which was diluted in deionized water at a concentration of 0.020 mol/L. Hydrochloric acid was added for complete dissolution. Growth temperature and time were set at 600 ˚C and 6 minutes respectively.

XRR measurements determined the film thickness to be 80.6 nm with a growth rate of 0.806 μm/h, which is comparable to HVPE. Peaks from β-Ga_2O_3 (400) and β-Ga_2O_3 (600) were observed by the XRD 2θ scan, indicating that (100)-oriented β-Ga_2O_3 was grown. It should be noted that the peak of γ-Ga_2O_3 (400) (at 2θ=43.93°), which was observed in the growth of β-Ga_2O_3 on MgO using PLD and MOCVD, was not observed. With no γ-Ga_2O_3 phase at the interface between MgO and β-Ga_2O_3, it may be possible to fabricate a sharp interface in β-Ga_2O_3 growth on MgO and p-NiMgO. The full width at half maximum of the ωrocking curve from β-Ga_2O_3 (400) was 145 arcsec, indicating high-quality crystal growth. From the ϕ scan, the in-plane orientation was determined to be β-Ga_2O_3 (100)[001] ‖ MgO (001)[110]. In addition, the RSM roughness of the 5x5 μm^2 AFM image was 0.74 nm.

EG-MoP-3 Fluid Analysis of MIST-CVD Chamber for Uniformity Improvement in Gallium Oxide Epitaxial Growth, ***Jungyeop Hong,*** *Y. Jung, D. Chun, J. Park, N. Joo, T. Kim,* Hyundai Motor Company, Republic of Korea

Gallium oxide has emerged as a promising material for high-performance electric vehicle and UV application applications. Among the various crystal phases of gallium oxide, the alpha phase has the widest band gap, making it suitable for manufacturing high voltage semiconductor devices. However, the alpha phase cannot be grown by homo-epitaxy, and the method of growing on a foreign substrate is mainly employed.

This paper focuses on the deposition of gallium oxide on wafers and highlights the limitation of the current maximum wafer size to 2 inches. To enable mass productivity, there is a need for equipment and process technologies that can accommodate larger wafer sizes for deposition. The research presented in this study aims to address this challenge by proposing solutions that can potentially contribute to the development of more efficient and cost-effective production processes for gallium oxide-based devices.

The study investigated the challenge of achieving stable uniformity in Ga2O3 epi grown on a 4-inch substrate compared to a 2-inch substrate. To overcome this issue, the authors proposed a fluid analysis of the MIST-CVD chamber to identify potential improvements for achieving better uniformity. This research can contribute to the development of Ga2O3 epi with higher uniformity and quality on larger substrates, which is crucial for its practical applications.

We conducted a study on growing Ga2O3 epi layers on a 4-inch substrate using MIST-CVD equipment. To achieve uniform growth of the epi on the 4-inch substrate, they evaluated the distribution level of the mist using fluid analysis methods. The chamber was modeled, and the mist distribution level was numerically interpreted under various conditions using fluid analysis tools. The optimal epi growth conditions with high mist distribution level and uniformity were derived based on the results. Growth technique was developed to obtain process reproducibility and excellent epitaxy quality even on a 4-inch wafer.

The research demonstrated the up-scale growth of high-quality Ga2O3 epi-layers on sapphire substrates, which could have potential applications in high-power and high-frequency electronic devices. To improve the uniformity of the large substrate, Our group modeled the sample position and gas flow in the Chamber and optimized the process conditions to achieve excellent uniformity. To verify the improvement in the uniformity of the epitaxial deposition, measurements using an Ellipsometer and XRD were performed.

EG-MoP-6 The Effect of Excess Ga on Electron Transport in *β*-Ga_2O_3 Grown via Plasma Assisted Molecular Beam Epitaxy, ***Thaddeus Asel,*** *B. Noesges, Y. Kim, A. Neal, S. Mou,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

Since the first demonstration of *β*-Ga_2O_3 based devices, there has been significant work done developing epitaxial growth methods for high quality *β*-Ga_2O_3 films. Improvement in the mobility of epitaxial films has been demonstrated in recent years. Additionally, the quantification of defect states in thin films has been performed for several growth techniques, including molecular beam epitaxy (MBE). However, there has yet to be a systematic study of the relationship between the *β*-Ga_2O_3 growth parameters, the electronic transport properties, and the defects present in *β*-Ga_2O_3 films grown via MBE. In this work we investigate the effect of excess Ga beam flux on the electronic properties of *β*-Ga_2O_3 films grown via MBE. Utilizing temperature dependent Hall Effect measurements and a self-

consistent fitting of the temperature dependent carrier density and mobility data, we are able to quantify the concentration defect states, including compensating acceptors, deep level impurities, and unintentional donors, present in the epitaxial films. The Ga beam flux can control the oxygen to Ga ratio present in the chamber as our oxygen is held constant for each growth run. This allows for growths that occur in the "oxygen rich" regime where the amount of Ga supplied limits the growth rate and the "gallium rich" regime where the amount of O supplied limits the growth rate. In both regimes the formation of the volatile suboxide Ga_2O occurs, but in the gallium rich regime, the desorption of Ga_2O causes a lower growth rate than that seen in the oxygen rich regime, due to the excess of Ga presence preventing the second reaction step in Ga_2O_3 growth. Two preliminary samples were grown in the gallium rich (Ga Beam Flux = 1×10^{-7} Torr) and oxygen rich (Ga Beam Flux = 6×10^{-8} Torr) regimes. The samples had different doping densities of 4.25×10^{17} cm^{-3} in the gallium rich sample and 2.60×10^{17} cm^{-3} in the oxygen rich sample, this discrepancy is due to an inaccuracy in the Si doping source. The acceptor concentration in the gallium rich sample was calculated to be 7.26×10^{16} cm^{-3} and 4.50×10^{15} cm^{-3} in the oxygen rich sample, a factor of 16 different based on the Ga overpressure. This is likely due to Ga vacancies that can form during the desorption of Ga_2Oduring growth. These results indicate that there is significant impact on the electron transport properties of β-$Ga_{2}O_3$ when there is excess Ga present during the growth, and that optimization of growth parameters is needed to optimize the electronic properties of MBE grown β-$Ga_{2}O_3$.

EG-MoP-7 Low-Pressure Chemical Vapor Deposition of Ultrawide Bandgap $LiGa_5O_8$ Thin Films, ***Kaitian Zhang***, *L. Meng, H. Huang,* The Ohio State University*; J. Sarker,* University of Buffalo, SUNY*; A. Bhuiyan,* The Ohio State University*; B. Mazumder,* University of Buffalo, SUNY*; J. Hwang, H. Zhao,* The Ohio State University

$LiGaO_2$ represents an ultrawide band gap semiconductor with an energy gap of 5.8 eV. It is considered as a group I-III-VI_2 counterpart of the group II-VI ZnO, and has an orthorhombic crystal structure. The ground state β-$LiGaO_2$ has a $Pna2_1$ space group. It has recently been predicted that $LiGaO_2$ is n-type dopable with Si or Ge and the possible p-type doping is under investigation. $LiGaO_2$ has been considered for piezoelectric and nonlinear optical applications in the past. Bulk growth of single-crystal $LiGaO_2$ has been studied as closely lattice-matched substrate (a = 5.407 Å, b = 6.405 Å and c = 5.021 Å) for GaN epitaxy. However, thin film growth of $LiGaO_2$ is still at an early stage with only PLD growth of $LiGaO_2$ on ZnO template was reported.

In this work, low pressure chemical deposition (LPCVD) of $LiGaO_2$ on various substrates were studied. C-plane sapphire, (010) and (001) $LiGaO_2$ substrates were used in this study. High purity Ga pallets, LiCl powder and O_2 were used as the precursors whereas Ar was used as the carrier gas. The growth temperature was kept at 900 °C. From a series of material characterization, the film grown on top of the (001) $LiGaO_2$ substrate is stabilized with a spinel-cubic structure $LiGa_5O_8$ with a lattice constant of 8.203 Å and space group P4_33-2 (Ga in both tetrahedral and octahedral sites). The spinel-cubic crystal structure of the $LiGa_5O_8$ film was confirmed from the atomic resolution HAADF STEM image and selected area electron diffraction (SAED) pattern. Additionally, the atom probe tomography (APT) measurement on the same sample confirmed a uniform Li distribution and a near-stoichiometric ratio of ~1:5:8 for Li/Ga/O.

$LiGa_5O_8$ has been studied as a phosphor host material when doped with transition metal and rare earth elements. To the best of our knowledge, this work represents the first experimental demonstration of thin film growth of pure $LiGa_5O_8$. Recent theoretical calculations suggest that $LiGa_5O_8$ has an indirect bandgap of approximately 5.7 eV with the direct gap about 0.1 eV higher, which renders $LiGa_5O_8$ a potential ultrawide bandgap semiconductor for power electronics applications.

In conclusion, this investigation explored the LPCVD growth of $LiGaO_2$ thin films on c-sapphire and $LiGaO_2$substrates. The results revealed a successful growth of $LiGa_5O_8$ film on (001) $LiGaO_2$ substrate. Comprehensive growth conditions still need to be mapped to determine the correlation between the growth condition, substrate selection and stabilization of crystal structure of $LiGa_xO_y$.

Acknowledgment: The authors acknowledge the funding support from Air Force Office of Scientific Research (AFOSR, FA9550-23-1-0142).

EG-MoP-8 Controlling Si Dopant Profiles in n-type β-Gallium Oxide, ***Brenton Noesges***, *Y. Kim, A. Neal, S. Mou, T. Asel,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

Oxidation of dopant sources affects dopant vapor pressures which can impact dopant profile uniformity in β-Ga_2O_3 grown via plasma-assisted molecular beam epitaxy (PAMBE).[1,2] In this work, we will focus on optimizing uniform dopant profiles in the low Si doping regime (<10^{18} cm^{-3}) of β-Ga_2O_3 films since Si doping concentrations show a gradient, increasing toward the surface of β-Ga_2O_3 thin films. Si concentrations of ~$3x10^{17}$ cm^{-3} are achievable as demonstrated by secondary ion mass spectroscopy (SIMS). Capacitance-voltage (CV) measurements to extract carrier concentrations agree well with Si concentrations observed in SIMS. We will also look at another aspect of Si incorporation in β-Ga_2O_3 that needs consideration when using traditional effusion cells as a Si dopant source. Previous work in PAMBE β-Ga_2O_3 growth demonstrated the importance of oxygen plasma power on controlling the background amount of Si present in β-Ga_2O_3 films.[3] In this work, we continued to explore sources of unintentional Si accumulation during the PAMBE growth process. The presence of Si at the interface between β-Ga_2O_3 substrate and film provides a parasitic conduction channel which is problematic for device performance. Attempts have been made to remove this Si at β-Ga_2O_3 interfaces via etching. Our results indicate that removing interfacial Si may not be as simple since Si can re-accumulate during PAMBE processes, thus limiting the effectiveness of pre-growth surface treatments. We created a test sample for SIMS simulating the effects of exposing clean β-Ga_2O_3 surfaces to potential sources of Si in a PAMBE system including quartz plasma bulb and dopant Si effusion cells. Exposure to only quartz plasma bulb did not produce Si accumulation at the surface. On the other hand, a growth interrupt that exposed a fresh β-Ga_2O_3 to the hot Si source for several minutes produced Si accumulation equal to that of the interfacial Si between substrate and film. These results demonstrate that removing Si prior to loading into PAMBE may be inadequate to remove interfacial Si since Si can be re-introduced from the Si dopant cell during pre-deposition stages like plasma stabilization. This work and results point toward important challenges and potential solutions when creating Si-doped β-Ga_2O_3 thin films.

[1] Kalarickal, N.K., et al., *Appl. Phys. Lett.***115**, 152106 (2019).

[2] McCandless, J.P., et al. *Appl. Phys. Lett.***121**, 072108 (2022).

[3] Asel, T. J., et al., *J. Vac. Sci. Technol*. A **38**, 043403 (2020).

EG-MoP-9 Silicon-doped β-Ga2O3 Films Grown at 1 µm/h by Suboxide Molecular-Beam Epitaxy, ***Kathy Azizie***, *F. Hensling, C. Gorsak,* Cornell University*; Y. Kim,* Air Force Research Laboratory*; N. Pieczulewski,* Cornell University*; D. Dryden,* Air Force Research Laboratory*; M. Senevirathna, S. Coye,* Clark Atlanta University*; S. Shang,* Penn State University*; J. Steele, P. Vogt, N. Parker, Y. Birkhölzer, J. McCandless, D. Jena, H. Xing,* Cornell University*; Z. Liu,* Penn State University*; M. Williams,* Clark Atlanta University*; A. Green,* Air Force Research Laboratory*; D. Schlom,* Cornell University

We report the use of suboxide molecular-beam epitaxy (*S*-MBE) to grow β-Ga_2O_3 at a growth rate of ~1 µm/h with control of the silicon doping concentration from $5*10^{16}$ to 10^{19} cm^{-3}. In *S*-MBE, pre-oxidized gallium in the form of a molecular beam that is 99.98% Ga_2O, i.e., gallium suboxide, is supplied. Directly supplying Ga_2O to the growth surface bypasses the rate-limiting first step of the two-step reaction mechanism involved in the growth of β-Ga_2O_3 by conventional MBE. As a result, a growth rate of ~1 µm/h is readily achieved at a relatively low growth temperature (T_{sub} ≈ 525 °C), resulting in films with high structural perfection and smooth surfaces (rms roughness of < 2 nm on ~1 µm thick films). Silicon-containing oxide sources (SiO and SiO_2) producing an SiO suboxide molecular beam are used to dope the β–Ga_2O_3 layers. Temperature-dependent Hall effect measurements on a 1 µm thick film with a mobile carrier concentration of $2.7*10^{17}$ cm^{-3} reveal a room-temperature mobility of 124 cm^2 V^{-1} s^{-1} that increases to 627 cm^2 V^{-1} s^{-1} at 76 K; the silicon dopants are found to exhibit an activation energy of 27 meV. We also demonstrate working MESFETs made from these silicon-doped β-Ga_2O_3 films grown by *S*-MBE at growth rates of ~1 µm/h.

EG-MoP-10 Epitaxial Growth of Metastable Ga_2O_3 Polymorphs Using MOCVD and HVPE, ***Jingyu Tang***, *M. Moneck, M. Weiler, K. Jiang, R. Davis, L. Porter,* Carnegie Mellon University

In recent years, there has been an increasing interest in the growth and electronic properties of metastable Ga_2O_3 polymorphs, e.g., κ-Ga_2O_3 with its high predicted spontaneous polarization as a potential ferroelectric material and α-Ga_2O_3 with its wider bandgap and ability to alloy with α-Al_2O_3. The metastable κ-Ga_2O_3 phase can be grown in epitaxial film form on substrates such as 4H/6H-SiC, Al_2O_3, AlN, GaN using vapor phase techniques. The κ-Ga_2O_3 films reported in the literature have varying degrees of phase purity and commonly contain three in-plane rotational domains, although there are some reports of suppressed rotational domains or increased domain sizes when grown on lattice-matched $GaFeO_3$ substrates[1], patterned sapphire[2] and SiH_4 gas introduction[3]. α-Ga_2O_3 films with high dislocation densities have been commonly grown on c-plane sapphire as indicated by the broader full-width-at-half-maxima (FWHM) of the X-ray rocking curves (XRC) of the tilted planes. Film growth techniques such as epitaxial lateral overgrowth4, depositing buffer layers[5] have been implemented to reduce the dislocation density. In this study, nominally phase-pure α-Ga_2O_3 and κ-Ga_2O_3 epitaxial films were grown on vicinal a-plane and c-plane sapphire substrates, respectively, at Carnegie Mellon University using halide vapor phase epitaxy (HVPE) and/or metalorganic chemical vapor deposition (MOCVD). Phase and microstructural characterization studies were conducted using high-resolution X-ray diffraction (HR-XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The growth of κ-Ga_2O_3 using MOCVD[6] revealed the presence of a 20-60 nm thick transition layer containing a mixture of β- and γ-Ga_2O_3 near the interface. In contrast, there was no detectable β peak or even a β shoulder in the κ-Ga_2O_3 films grown by HVPE. The FWHM of the (004) κ-Ga_2O_3 XRC for the HVPE film was 5x more narrow than that of the MOCVD film with respective values of 0.11° and 0.49°. Comparable FWHM values in the literature for κ-Ga_2O_3 are 0.16° to 0.82°3,7,8. Whereas. α-Ga_2O_3 has been difficult to produce using MOCVD, nominally phase-pure (11-20) α-Ga_2O_3 films with exceptionally low FWHM for (11-20) = 0.034° are readily produced at 650°C using HVPE. In summary, our findings indicate that HVPE growth leads to nominally phase-pure α-Ga_2O_3 and κ-Ga_2O_3 epitaxial films with narrow XRCs, and offers promising avenues for further investigation of the properties of metastable Ga_2O_3 phases.

EG-MoP-11 Pulsed Laser Deposition of α-Ga_2O_3 on M-Plane Al_2O_3: Growth Regime, Growth Process and Structural Properties, ***Clemens Petersen***, University Leipzig, Felix Bloch Institute for Solid State Physics, Semiconductor Physics Group, Leipzig, Germany*; S. Vogt, H. von Wenckstern, M. Grundmann,* University Leipzig, Felix Bloch Institute for Solid State Physics, Semiconductor Physics Group, Germany

Due to its wide band gap of 4.6 eV - 5.6 eV and large predicted breakdown field of 8 MV cm^{-1} [1], much attention is drawn to the ultra-wide bandgap semiconductor Ga_2O_3 for applications in high-power devices. However, besides the well-studied thermodynamically stable monoclinic β-phase of Ga_2O_3, the metastable α-polymorph with corundum structure is rapidly gaining interest in the scientific community. Since it is isostructural to α-Al_2O_3, growth of layers with high structural quality on cost-efficient sapphire substrates is feasible. In particular, m-plane sapphire facilitates the growth of the corundum phase by suppressing c-plane facets on which the β-phase favorably crystallizes [2]. Further, Sn-doped α-Ga_2O_3 thin films grown on m-plane sapphire show electron mobilities of up to 65 $cm^2 V^{-1} s^{-1}$, which is three times higher than what can be achieved on c-plane cut substrates as reported by Akaiwa *et al*. [3]. To our knowledge, a systematic study of the growth of Ga_2O_3 on m-plane sapphire by pulsed laser deposition (PLD) has not been published to date.

We present phase-pure α-Ga_2O_3 thin films with high surface quality and crystallinity grown on m-plane sapphire using PLD [4]. Therefore, the influence of growth temperature, oxygen background pressure, and film thickness on structural properties is investigated to determine the growth window for phase-pure α-Ga_2O_3. Samples were analyzed using X-ray diffraction, atomic force microscopy, and spectroscopic ellipsometry measurements. A distinct growth window for phase-pure (10.0)-oriented α-Ga_2O_3 at growth temperatures above 450°C and low oxygen partial pressures p(O2) of 3×10^{-4} mbar is identified. It was found that for thicker layers (> 200 nm) the growth of monoclinic β-Ga_2O_3 is promoted. This is likely induced by the c-facets of the α-Ga_2O_3 grains, leading to a (010) orientated island growth corroborated by stripe-like features in atomic force microscopy scans and a corresponding in-plane orientation confirmed by x-ray diffraction ϕ-scans. For oxygen partial pressures above 3×10^{-4} mbar and growth temperatures below 530°C, the formation of mixed (10.0) α-Ga_2O_3 and spinel-defective (110) γ-Ga_2O_3, manifesting as inclusions, was observed independently of the layer thickness. A corresponding phase diagram for Ga_2O_3 growth on m-plane sapphire by PLD is presented.

1. Higashiwaki *et al.*, Appl. Phys. Lett., Vol. 100, 013504 (2012)

2. Jinno *et al.*, Sc. Advances, Vol. 7, No. 2, eabd5891 (2021)

3. Akaiwa *et al.*, pss (a), Vol. 217, No. 3, 1900632 (2020)

4. Petersen *et al.,* APL Materials, accepted manuscript, (2023)

EG-MoP-14 High-Quality Power Device Grade β-Ga_2O_3 on 4H-SiC via Metal Organic Chemical Vapor Deposition, *I. Sanyal, A. Nandi,* ***Martin Kuball***, University of Bristol, UK

Ga_2O_3 suffers from low thermal conductivity which would translate into excessive device temperatures. Integration with high thermal conductivity materials is, therefore, essential to explore. We report epitaxial growth of high material quality β-Ga_2O_3 on 4H-SiC (0001) substrates using MOCVD. Recent reports indicated β-Ga_2O_3 growth on 4H-SiC at a deposition temperature of 650 °C using LPCVD, resulting in rms surface roughness of 2.25 nm and FWHM of 1.3°, and at 700 °C using liquid-injection MOCVD with rms surface roughness of 8 nm. However, we observed formation of incomplete misoriented particles when the layer was grown at a temperature between 650 °C to 750 °C as shown in fig 1. Two steps modified growth method was then adopted where the nucleation layer was grown at 750 °C followed by a buffer layer grown at various temperatures from 750 to 950 °C. Three samples namely S1, S2 and S3 were compared to understand the growth mechanism. S1 and S2 are identical except the bulk layer where the growth temperature was 920 °C and 950 °C respectively. S1 shows partly coalesced surface with misoriented grains or nano-crystallites as shown in fig.2. Whereas, S2 forms fully coalesced surface without any pseudohexagonal domains as shown in fig. 3. XRD 2θ-ω scan confirms β-polymorph of Ga_2O_3 with dominant peak in (-201) direction and FWHM of 0.9°. One of the challenges in growing β-Ga_2O_3 on SiC substrate with O_2 source at high temperature is the formation of amorphous SiO_2 layer on the growth surface that results in poor crystal quality. Si-terminated 4H-SiC/ β-Ga_2O_3interface is highly sensitive to O_2 due to its lowest migration energy. O-terminated β-Ga_2O_3 in (-201) direction and Si-terminated 4H-SiC in (0001) direction offer lowest relaxation energy and thus highest stability by forming covalent bond between Si-O at the interface. Furthermore, the nucleation layer grown at lower temperatures is polycrystalline in nature with a high density of defects that propagate into the top layer. Instead of growing a thin nucleation layer at a fixed lower temperature, we implemented a new 'ramp-growth' technique in S3 where the growth of the nucleation layer starts at 780 °C and the temperature linearly increases to 950 °C within three minutes followed by the growth of the buffer layer at 950 °C. As can be seen in fig. 4, the surface roughness reduced to 3 nm with FWHM as low as 0.79°, comparable to the most mature β-Ga_2O_3 heteroepitaxy on sapphire using MOCVD, greatly advanced to currently available Ga_2O_3 on SiC, paving the way for the development of high voltage device fabrication with improved thermal property.

Epitaxial Growth
Room Davis Hall 101 - Session EG+BG-TuA

Bulk/Epitaxy II

Moderator: Sriram Krishnamoorthy, University of California Santa Barbara

1:45pm **EG+BG-TuA-1 Suitable Orientation for Homoepitaxial Growth of Gallium Oxide**, ***Kohei Sasaki,*** *A. Kuramata,* Novel Crystal Technology, Inc., Japan **INVITED**

The surface orientation is an important condition in homoepitaxial growth. β-Ga_2O_3 has an unusual crystal structure, named β-Gallia, so we cannot use the knowledge of the usual crystal structures, such as diamond, zinc blende or wurtzite, when the selecting the surface orientation. Here, we investigated suitable orientations for homoepitaxial growth of gallium oxide by growing films on gallium oxide substrates with various orientations.

The β-Gallia structure is monoclinic, and its low index planes are (100), (010), and (001). We made gallium oxide substrate with surface orientations from the (100) to (010) plane or from the (100) to (001) plane and investigated the crystal quality, surface roughness, and growth rate of the films grown by molecular beam epitaxy. We sliced the surface in ten degrees steps from the (100) plane rather than adjusting the specific orientation. Growth temperature was fixed at 700 degrees Celsius. Ozone gas was used as the oxygen source.

Of the planes between the (100) and (010) plane, only the (100) plane showed a peculiarly low growth rate. On the other hand, there were no unusual features on the planes except the (100) plane; the growth rate was about 700 nm/h, and surface roughness (RMS) was about 1-2 nm.

On the other hand, the planes between the (100) and (001) plane showed severe surface roughness especially around the (101) plane and (-201) plane. The surface roughness on the (101) plane was due to crystal defects in which (-201) crystal grew on the (101) plane, whereas on the (-201) plane it was due to (-201) twin defects. We obtained very smooth surfaces with an RMS of 1 nm or less by using the (001), (-102), (401), (-401) planes.

It is known that the surface orientation of gallium oxide homoepitaxial growth depends on the growth method. The surfaces of films grown by MBE and metalorganic chemical vapor deposition (MOCVD) show similar morphologies. Thus, the knowledge gained in this research may be applicable to MOCVD.

2:15pm **EG+BG-TuA-3 Pushing the Al composition limit up to 99% in MOCVD β-$(Al_xGa_{1-x})_2O_3$ films using TMGa as Ga precursor**, ***A F M Anhar Uddin Bhuiyan,*** *L. Meng, H. Huang, J. Hwang, H. Zhao,* The Ohio State University

Recent research progresses have highlighted the promising potential of the MOCVD growth method in developing β-$(Al_xGa_{1-x})_2O_3$ alloys along different crystal orientations with high Al composition and controllable n-type doping. The coexistence of β and γ phases in (010) β-AlGaO films with Al>27% indicates challenges for incorporating higher Al compositions. Using alternative crystal planes of β-Ga_2O_3 substrates, such as (100) and (-201), has yielded single phase β-AlGaO films, with over 50% of Al incorporation. These prior efforts on MOCVD growth of β-AlGaO alloys using TEGa as the Ga precursor limit the film growth rate to below ~0.7 μm/hr.

In this study, we employed TMGa as the Ga precursor, which not only elevates the growth rates of β-AlGaO films up to 2 μm/hr, but also enhances the Al compositions up to a record high value of ~99%. The systematic investigation of MOCVD growth of β-AlGaO films and β-AlGaO/Ga_2O_3 superlattices on different crystal planes revealed a strong impact of substrate orientation on the solubility limit of β-AlGaO grown at relatively high growth rates. The crystalline structure, strain, morphology, stoichiometry, and bandgap of β-AlGaO films are investigated as a function of the Al composition and crystal orientations. β-AlGaO films with Al compositions up to 99%, 29%, 16% are achieved on (100), (010) and (-201) β-Ga_2O_3 substrates, respectively, as determined by XRD, XPS and STEM EDX. Beyond 29% of Al incorporation, the (010) β-AlGaO films exhibit β to γ phase transformation. Owing to its highly anisotropic characteristics, (-201) β-AlGaO films show local segregation of Al. Fully strained coherent β-AlGaO films are obtained for thicknesses of 350 nm (010, Al=15%), 120 nm (100, Al=16%) and 205 nm (-201, Al=13%). The crystalline structure of 20 nm thick β-$(Al_{0.99}Ga_{0.01})_2O_3$ film was accessed by atomic resolution STEM imaging, showing sharp interface and alloy homogeneity. The electron nano-diffraction pattern and quantitative STEM-EDX elemental mapping confirm the β-phase growth with Al composition of 99%, which agrees well with XRD and XPS measurement results. A record high bandgap energy of 7.26 eV is achieved from β-$(Al_{0.99}Ga_{0.01})_2O_3$ film using XPS, revealing great promises of developing β-AlGaO/Ga_2O_3 interfaces with high band offsets. The findings of this study offer valuable insights on the MOCVD epitaxy and properties of high Al composition β-AlGaO films and β-AlGaO/Ga_2O_3 heterostructures for device applications.

Acknowledgment: AFOSR (FA9550-18-1-0479) and NSF (Grant No. 2231026, and 2019753).

2:30pm **EG+BG-TuA-4 Fast Growth and Characterization of Undoped β-Ga_2O_3 on 2-Inch Substrates Using a Horizontal Hot-Wall MOVPE System**, ***Kazutada Ikenaga,*** Tokyo University of Agriculture and Technology / TAIYO NIPPON SANSO CORPORATION, Japan*; J. Yoshinaga, P. Guanxi,* TAIYO NIPPON SANSO CORPORATION, Japan*; H. Tozato, T. Okuyama, K. Goto, Y. Kumagai,* Tokyo University of Agriculture and Technology, Japan

Metalorganic vapor phase epitaxy (MOVPE) is one of the attractive methods for the epitaxial growth of β-Ga_2O_3. However, it requires control of the hazardous reactions between organometallics and oxygen (O_2) while suppressing the incorporation of carbon (C) and hydrogen (H) impurities derived from the organometallics. Our research group has clarified the key conditions that enable the growth of high-purity β-Ga_2O_3 layers with suppressed C and H incorporation by thermodynamic analysis and in situ mass spectrometry of gaseous species in the reactor [1-3]. In this work, we report on the uniform and fast growth of β-Ga_2O_3 on 2-inch substrates.

A horizontal low-pressure hot-wall MOVPE system (TAIYO NIPPON SANSO CORPORATION, FR2000-OX) with a facedown holder capable of placing 2-inch diameter substrates was used. One 2-inch diameter c-plane sapphire wafer or three 10 mm × 15 mm sized β-Ga_2O_3(010) substrates were set for each growth. Epitaxial layers were grown in a temperature range of 900–1050 °C using trimethylgallium (TMGa) and O_2 as precursors, and Ar as a carrier gas, respectively. Under a constant O_2 supply, TMGa was supplied in the range of 111 – 546 μmol/min (corresponding to the input VI/III ratio from 1609 to 327).

The growth rate was found to be constant regardless of the growth temperature. At a growth temperature of 1000°C, the growth rate increased linearly up to about 15 μm/h with increasing TMGa supply rate, while the C impurity concentration increased. Since an increase in H and C impurity concentrations was observed with decreasing growth temperature, it is likely that the increase in these impurities is due to the increase in TMGa-derived hydrocarbons and their insufficient combustion. It was also found that there is no difference in growth rate between heteroepitaxial growth and homoepitaxial growth under the same conditions. In this presentation, the uniformity of the grown layer is also reported.

This work was supported by Ministry of Internal Affairs and Communications (MIC) research and development (JPMI00316).

[1] K. Goto et al., Jpn. J. Appl. Phys. **60**, 045505 (2021).

[2] K. Ikenaga et al., J. Cryst. Growth **582**, 126520 (2022).

[3] K. Ikenaga et al., Jpn. J. Appl. Phys., in press.

2:45pm **EG+BG-TuA-5 MBE Growth and Properties of Ultra-wide Bandgap Oxide Layers Spanning 5.0 - 9.0 eV Energy Gaps**, ***Debdeep Jena,*** Cornell University **INVITED**

3:15pm **EG+BG-TuA-7 Structural Defect Formation and Propagation in Fe-doped Czochralski-grown b-Ga_2O_3 Boules**, ***Luke Lyle,*** Pennsylvania State University - Applied Research Lab*; R. Lavelle,* Penn State University - Applied Research Lab*; D. Erdely,* Pennsylvania State University - Applied Research Lab*; W. Everson,* Penn State University - Applied Research Lab*; A. Balog, N. Alem,* Pennsylvania State University*; D. Snyder,* Pennsylvania State University - Applied Research Lab

Over the last decade, β-Ga_2O_3 has garnered increased attention due to its ultrawide bandgap of 4.7-4.9 eV, controllable range of shallow, n-type dopants (Sn, Si, Ge), and easily scalable and economic melt growth processes. Popular melt-growth processes that have been developed for β-Ga_2O_3 include the edge-defined film fed method, vertical bridgeman, and the Czochralski process. Although different types of structural defects in these melt-grown crystals have been identified, how they form and propagate throughout the growth process remains elusive. Specifically, it has been found that the density of structural defects can vary across wafers in the same boule and even across a single wafer.

We etch and analyze double side, chemi-mechanically polished 2” diameter wafers and 1” diameter wafers taken from ‘cores’ from 2” diameter boules at the tip, center, and tail of Fe-doped (010) Czochralski-grown boules. The etch pits were formed using an optimized H_2PO_4 etch process and are

mapped using automated optical microscopy, statistical analysis software and scanning electron microscopy and are organized by defect type and density across each wafer analyzed in the boule. Wafers from the length of the boule were used to assess seeding and growth initiation related defect structures and long-range propagation and results from adjacent wafers at various locations were studied to understand short range defect formation and propagation. Trends regarding the presence of dislocations/nanopipes and their formation throughout the boule are discussed along with differentiation between process- and growth-related defects. Particular attention in this talk is paid to the formation and propagation of so-called “nanopipe” defects, as they are poised to act as killer-defects for high-voltage devices

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.

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