MBE
Room Silver Creek - Session MBE-1MoM
Oxides and Nitrides

Moderator: Jason Kawasaki, University of Wisconsin Madison

8:00am **MBE-1MoM1 Oxide MBE Rocks!**, ***Darrell Schlom,*** Cornell University **INVITED**

MBE is renowned for preparing semiconductor heterostructures with high purity, high mobility, and exquisite control of layer thickness at the atomic-layer level. In recent decades it has become the definitive method for the preparation of oxide quantum materials as well, particularly when it is desired to perturb or exploit their properties. In this talk I will show examples of how the unparalleled synthesis precision of MBE can be used to tailor oxide quantum materials through the various approaches shown in Fig. 1 to expose hidden ground states [1]. The band structure is revealed by highresolution angle-resolved photoemission (ARPES) on pristine surfaces of these oxide heterostructures made possible by a direct ultra-high vacuum connection between MBE and ARPES [2].

[1] R. Ramesh and D.G. Schlom, "Creating Emergent Phenomena in Oxide Superlattices," *Nature Reviews Materials* **4** (2019) 257–268.

[2] D.G. Schlom and K.M. Shen, "Oxide MBE and the Path to Creating and Comprehending Artificial Quantum Materials," in M. Coll *et al.*, "Towards Oxide Electronics: a Roadmap," *Applied Surface Science* **482** (2019) 1–93.

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8:45am **MBE-1MoM4 RF-plasma MBE Growth and Characterization of β-Ga_2O_3/NbN_x Heterostructures on SiC**, ***Neeraj Nepal,*** *D.S. Katzer, B. Downey, V.D. Wheeler,* U.S. Naval Research Laboratory*; L. Nyakiti,* Texas A&M*; E. Jin, V. Gokhale, M. Hardy, D. Storm, D. Meyer,* U.S. Naval Research Laboratory

Gallium oxide (Ga_2O_3) is an ultra-wide bandgap (UWBG) semiconductor with a bandgap of 4.5-4.9 eV and has higher figure of merit values than GaN and SiC for power and rf devices making it a candidate for next generation high-power/temperature electronics [1-3]. One disadvantage of β- Ga_2O_3 fordevice application is its low thermal conductivity [4]. Approaches to improve thermal performance are through heteroepitaxy directly on high thermal conductivity substrates (for instance β- Ga_2O_3 on SiC [5]), heat extraction from top side, or by transferring β- Ga_2O_3 to a high thermal conductivity substrate. While β- Ga_2O_3 nano-membranes have been transferred recently from a bulk substrate [6], it would be preferable to perform device processing and yield screening prior to transferring to a target substrate. To accommodate this approach, we present here our work investigating heteroepitaxial growth of Ga_2O_3 films on NbN_x, subsequent epitaxial lift-off, and transfer printing of Ga_2O_3 epilayers and devices. This work leverages our previous experience with demonstrated transfer printing of III-Nitride on NbN_x devices [7].

Epitaxial NbN_x films were grown on 6H-SiC in the PRO-75 rf-plasma molecular beam epitaxy (MBE) chamber, characterized *ex situ* and transferred to V80H oxide rf-MBE to grow β-Ga_2O_3. β- Ga_2O_3 layers were grown at 650 ° C on these NbN_x/6H-SiC templates. The baseline growth parameters were optimized with deposition of β- Ga_2O_3 films on c-plane sapphire substrates. Optimized MBE growth conditions were then used to grow 53 nm thick film on NbN_x, which has only (-201), (-402), (-603), and (-804) β- Ga_2O_3 peaks in the 2θ X-ray diffraction range of 10 - 90°. Transmission electron microscopy (TEM) shows that the Ga_2O_3 layer is crystalline and confirmed the XRD orientation relationship of $(-201)_{Ga2O3}||(0001)_{NbNx}||(0001)_{SiC}$. A high vertical breakdown field of ~2 to 3 MV/cm was found for MBE grown Ga_2O_3 on NbN_x. Finally, lateral XeF_2 etching of the buried NbN_x layer to demonstrate lift-off capabilities and study transferability to foreign substrates.

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1. H.H. Tippins, Physical Review **140**, A316 (1965).

2. K. Akito et al., Jpn. J. Appl. Phys. **55**, 1202A2 (2016).

3. J.Y. Tsao, Adv. Electron. Mater. **4**, 1600501 (2018).

4. M. Santia et. al., Appl. Phys. Lett. **107**, 041907 (2015).

5. N. Nepal et al., 34th NAMBE Conference, MBE-MoA1, Banff, Canada October 1st, 2018.

6. H. Zhou et al., IEEE Electon Device Lett. **38**, 103 (2017).

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9:00am **MBE-1MoM5 Development of a High-Purity, High-Concentration Ozone Delivery System for MBE and Growth of β-Ga_2O_3**, ***Mark O'Steen,*** *T. Campbell, S. Farrell, E. Tucker, D. Hanser,* Veeco Instruments Inc.

Each year, research in oxide materials and devices increases and molecular beam epitaxy (MBE) serves as a critically important tool for the synthesis of these materials. The ever-increasing interest in oxide materials is attributable to the wide range of potential uses of these materials in applications including power electronics, structural buffer layers, gate dielectrics, superconductors, 2D electron gases, ferroelectrics, ferromagnetics, electrical isolators, and optically transparent conductive layers. For many of these applications, a key challenge for MBE growth is adequately oxidizing compounds to achieve desirable properties.

Molecular oxygen and oxygen plasma have been used extensively in the growth of oxide materials but have limitations including reactivity and growth rate. An alternative to these approaches is to use a stronger oxidizer such as ozone. However, using ozone as a source gas in MBE presents challenges related to generating, storing, and injecting ozone that is of both high purity and high concentration. Additionally, ozone has inherent challenges with toxicity and chemical instability.

To address these practical issues in safely implementing ozone in MBE, a distillation system has been integrated to a novel delivery system with key features including: delivery of high-purity, high-concentration ozone; wide-ranging, precise pressure control for the injected ozone gas; and comprehensive safety controls. Growth pressures have been actively controlled from $5x10^{-9}$ to $5x10^{-5}$ Torr with high precision and highly-linear ramp rate control. Additionally, to maximize process flexibility, this system provides for automated, rapid switching between injecting molecular oxygen, low-concentration ozone (15-25 wt%), and high-concentration ozone (~90 wt%).

Finally, this ozone system has been used in the growth of β-Ga_2O_3 epitaxial films. Details of the growth interactions will be discussed. The resulting epitaxial films were measured using techniques including RHEED, AFM, high-resolution XRD, and ellipsometry.

9:15am **MBE-1MoM6 Advancements in High Indium Content AlInN Grown Via Metal Modulated Epitaxy and Application Towards Polar/Non-Polar Optical Devices**, ***Zachary Engel,*** *E. Clinton, W.A. Doolittle,* Georgia Institute of Technology

AlInN has been a topic of recent study due to its unique property among III-nitrides of lattice matching to GaN at a composition of 18% indium, making it a strong candidate for power electronic and optoelectronic applications. A widely unexplored area is the application of AlInN towards visible/Near-IR optical devices. AlInN has a tunable bandgap, spanning the infrared to ultraviolet range, of 0.7 to 6.1eV. At a composition of about 70% indium AlInN has a perfect bandgap, 1.7eV, for tandem solar cells with silicon and for various optical communications applications. Challenges exist with the growth of AlInN as a result of the large lattice parameter mismatch and differences in growth regimes between the two binary components. At the low temperatures required for the growth of AlInN the aluminum adatoms have a low mobility, often leading to lateral phase separation in the film. Metal Modulated Epitaxy (MME) offers a good solution to the growth issues of AlInN. This flux modulated technique allows for growth under metal rich conditions, increasing surface diffusion lengths of the Aluminum while limiting droplet formation and terminating in a dry surface suitable for devices.

For comparison ~100 nm thick high indium content AlInN samples were grown using nitrogen rich (0.8 III/V ratio) MBE and MME using a III/V ratio of 1.3 with a dose designed to prevent surface segregation (details supplied at the conference). Both films were grown cold at 375 degrees C to limit phase separation The MME sample showed improvement over the MBE sample in crystal quality and surface roughness, with the XRD (0002) reflection FWHM improving from 783 arcsec to 184 arcsec, the XRD (10-15) reflection FWHM improving from 2456 arcsec to 1421 arcsec, and the AFM surface roughness improving from 1.52nm rms to 0.882nm rms between the MBE and MME samples respectively. The carrier concentrations of both samples were found to be about 1E19 cm-3 via hall measurement implying that like InN, In-rich AlInN may suffer from excessive residual electron concentrations and/or surface fermi-level pinning. Optical measurements will be presented at the conference.

The same MME growth conditions were used to grow 100nm of indium rich AlInN directly on a (111) p-silicon substrate. Simple indium contacts were used to contact the Si and AlInN. Vertical conduction and rectification between the silicon and AlInN layers were observed in this large area (~0.25x0.25 cm2) device. Understanding of the limitations of and physics governing this promising vertical polar/non-polar heterojunction will be given at the conference. Additionally, results from various growth conditions will be detailed.

9:30am **MBE-1MoM7 Structural and Electronic Properties of NbN and III-N/NbN Heterostructure Grown by Molecular Beam Epitaxy***, **John Wright**, G. Khalsa, H.G. Xing, D. Jena,* Cornell University

We have investigated the growth of NbN thin films and the incorporation of NbN into III-N semiconductor heterostructures using nitrogen plasma-assisted molecular beam epitaxy. We demonstrate how the structural and electronic properties of NbN vary as a function of the growth parameters including the substrate material, the substrate temperature, the nitrogen plasma conditions, and the Nb flux. NbN thin films possess a metallic state resistivity of around 100µΩ-cm and transition to the superconducting state below 17K. We demonstrate that atomically smooth epitaxial NbN thin films can be grown on both 6H-SiC and GaN. We have also investigated the epitaxial growth of GaN and AlN on NbN films with the goal of integrating NbN into heteroepitaxial III-N devices, creating new opportunities to utilize both the metallic and superconducting properties of NbN in III-N electronic devices.

We observe 2D layer-by-layer growth of NbN for over 100 monolayers on 6H-SiC substrate as evidenced by RHEED intensity oscillations during growth, yielding atomically smooth epitaxial NbN films with monolayer step heights. We show that when grown on GaN, NbN will grow in a layer-by-layer mode only below a critical thickness of between 6nm and 9nm, at which point the growth transitions to a 3D growth mode. When GaN films are grown on NbN using the metal rich growth conditions typically used in MBE growth of GaN it is observed that GaN will not form a continuous film but rather segregates into islands on the NbN surface. Our effort to optimize the growth of III-N thin films on NbN is ongoing.

We use X-ray characterization to assess the structure of NbN films and NbN/III-N heterostructures. We characterize the crystalline quality of the films using the rocking curve measurement technique, finding full width at half maximum of NbN on 6H-SiC of 0.05°. We also investigate variation in the measured out of plane lattice parameter of NbN over a large range, from 2.50Å to 2.56Å, a difference of 2.37%, depending on the MBE growth process.

9:45am **MBE-1MoM8 Optically-induced 2DEGs in GaN/AlGaN Heterostructures***, **Stefan Schmult,*** TU Dresden, Germany*; S. Wirth,* Max-Planck-Institute for Chemical Physics of Solids, Germany*; V. Solovyev,* Institute of Solid State Physics RAS, Russia*; R. Hentschel, A. Wachowiak,* NaMLab gGmbH*; T. Scheinert,* TU Dresden*; A. Grosser,* NaMLab gGmbH, Germany*; I. Kukushkin,* Institute of Solid State Physics RAS, Russia*; T. Mikolajick,* TU Dresden & NaMLab gGmbH, Germany

In our MBE-grown ultra-pure GaN/$Al_{0.06}Ga_{0.94}N$ heterostructures with barrier thickness of 16 nm, a 2-dimensional electron gas (2DEG) is absent in dark environment and at room temperature. However, illumination with ultra-violet light (UV, photon energies larger than the GaN band-gap) generates a conductive 2D channel at the GaN/AlGaN interface. An immediate consequence for lateral field-effect transistors (FETs) is their normally-off switching characteristics in the dark.

Upon UV illumination below 100 K the 2DEG persists after switching off the illumination, with a charge carrier density depending only weakly on the excitation power. Shubnikov-de Haas-oscillations and Zeroes in the longitudinal resistance recorded from Hall bars at T = 0.5 K clearly point at the 2D channel character with no parasitic current paths. The respective electron densities are extracted from Landau level filling factors.

Band diagram simulations point at the significance of the GaN surface potential value for the existence of a 2DEG in the dark. Two factors, namely the residual impurity background in the GaN/AlGaN layer stack as well as impinging UV radiation, appear to influence the surface potential, resulting in the difference between normally-on or -off FET operation. The reported characteristics demonstrate the technical feasibility of next-generation normally-off as well as light-sensitive GaN-based device concepts.

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