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Room Jefferson 2-3 - Session EG-TuA
Bulk & Epitaxy II
Moderator: Xiuling Li, University of Texas Austin

1:45pm **EG-TuA-1 Progress in Beta-Gallium Oxide Materials and Properties*, James Speck,*** University of California Santa Barbara **INVITED**

In this presentation, we present recent work on the development of β-Ga_2O_3 materials and their properties. The talk will include the following topics:

*Coherently strainedβ-$(Al_xGa_{1-x})_2O_3$ thin films on β-Ga_2O_3 Part I: Growth of (001) β-$(Al_xGa_{1-x})_2O_3$ thin films via metal oxide catalyzed epitaxy. In this work, we report on the growth of (001) β-$(Al_xGa_{1-x})_2O_3$ films in molecular beam epitaxy via metal oxide catalyzed epitaxy. Films with Al contents up to 15% were grown and Al content was measured with atom probe tomography. A relationship between Al content and out of plane lattice parameter was determined. Transmission electron microscopy showed no evidence of extended defects in the (001) β-$(Al_xGa_{1-x})_2O_3$ and reciprocal space maps confirmed that the β-$(Al_xGa_{1-x})_2O_3$ films were coherently strained to the (001) β-Ga_2O_3. Sn was also demonstrated to act as a surfactant for (001) β-$(Al_xGa_{1-x})_2O_3$ growth, allowing for high quality, uniform films with smooth morphologies.

*Coherently strainedβ-$(Al_xGa_{1-x})_2O_3$ thin films on β-Ga_2O_3 Part II - composition determination. We derive the relationships between lattice parameters for β-$(Al_xGa_{1-x})_2O_3$ and Al content x assuming the β-$(Al_xGa_{1-x})_2O_3$ is coherently strained to β-Ga_2O_3. The fundamental stiffness tensor of β-$Ga_2O_3$and stress-strain relationships are used to determine out of plane lattice parameters for (010) and (001) β-$(Al_xGa_{1-x})_2O_3$. Additionally, transformation of the stiffness tensor allows for derivation of similar relationships for (100) β-$(Al_xGa_{1-x})_2O_3$. For all three orientations, the relationships between peak spacing for β-$(Al_xGa_{1-x})_2O_3$ and β-Ga_2O_3 peaks in HRXRD and Al content x are calculated.

*We describe two recent ultrafast optical pump probe experiments that have determined the electron-phonon scattering time - 4.5 fs for electron-polar optical phonon scattering. These experiments also determined the energy separation of the CBM to the first side valley: 2.6 eV [Marcinkevicius et al., Appl. Phys. Lett. **118**, 242107 (2021)]. In a separate ultra-fast pump probe spectroscopy study, the time scale for the formation of polarons (from optically generated free holes) was determined: 0.5 to 1.1 ps [[Marcinkevicius et al., Appl. Phys. Lett. **116**, 132101 (2020)].

2:15pm **EG-TuA-3 (110) β-Ga_2O_3 Epitaxial Films Grown by Plasma-Assisted Molecular Beam Epitaxy*, Takeki Itoh,*** *A. Mauze, Y. Zhang, J. Speck,* University of California at Santa Barbara

Epitaxial growth of β-Ga_2O_3 with superior crystal quality has been achieved on different crystal orientations such as (100), (010) and (-201) via plasma-assisted molecular beam epitaxy (PAMBE)[1]. So far, most of the research has been performed on (010) substrates. However, investigation on (010) substrates has shown that (110) facets are revealed the chevron consistent features in RHEED studies, which indicates (110) is a natural plane in β-Ga_2O_3[2]. Figure 1 shows atomic models of (110) and (010) planes projected along [001] direction.

Unintentionally doped (UID) β-Ga_2O_3 epitaxial films were grown on (110) substrates by PAMBE while (010) substrates were co-loaded as growth reference. The temperatures of the substrates were kept at 600 ℃ and 700 ℃. To optimize the growth condition, the Ga fluxes were changed from 3.0×10^{-8} Torr to 2.5×10^{-7} Torr which were measured by beam equivalent pressure (BEP). Prior to the growth, oxygen polishing and Ga polishing were performed to remove the residual impurities from the surfaces. The film thickness was determined by measuring high-resolution X-ray diffraction (HRXRD). The surface morphology of the epitaxial films was measured by atomic force microscopy (AFM). Figure 2 shows the RHEED pattern of (110) and (010) substrates after Ga polishing. Streaky patterns were observed from the surface of (110) substrates, which indicates atomically flat surface. Conversely, crossed lines (red guideline) corresponding to (110) facets were observed from [001] azimuth on (010) substrate. Figure 3 shows the HRXRD result of the (110) β-Ga_2O_3 epitaxial film. Clear thickness fringes indicate abrupt interface between β-Ga_2O_3 and β-$(Al_{1-x}Ga_x)_2O_3$ spacer layers. Figure 4 shows the growth rate dependence on Ga flux of (010) and (110) substrates at 600 ℃ and 700 ℃. This result suggests that the growth rate is not reduced on the (110) plane compared to (010)[3]. In the oxygen rich regime, the growth rate increases linearly with Ga flux. In the plateau regime, there was still too low excess Ga flux to have a reduced growth rate. We expect higher Ga flux to yield reduced growth rates. Figure 5 shows the surface morphology of β-Ga_2O_3 films grown at 700 ℃ on (110) and (010) substrates. The RMS values indicate smooth surface morphology was obtained by growing on (110) substrates. Despite the appearance of (110) facets in the growth of (010) β-Ga_2O_3, the (110) plane does not have the tendency to show a well-defined step-terrace structure.

[1] A. Mauze *et al.,* APL Mater. **8**, 021104 (2020). [2] P. Mazzolini *et al.,* APL Mater. **7**, 022511 (2019). [3] T. Itoh *et al.,* Appl. Phys. Lett. **117**, 152105 (2020).

2:30pm **EG-TuA-4 Si-doped β-Ga2O3 Films Grown at 1 µm/hr by Suboxide MBE*, Kathy Azizie,*** *P. Vogt, F. Hensling, D. Schlom, J. McCandless, H. Xing, D. Jena,* Cornell University*; D. Dryden, A. Neal, S. Mou, T. Asel, A. Islam, A. Green, K. Chabak,* Air Force Research Laboratory

In this work we further develop suboxide molecular-beam epitaxy (S-MBE) to establish a means to Si-dope β-Ga2O3 grown by S-MBE and investigate its electrical properties. S-MBE was recently shown to enable the growth of β-Ga2O3 at growth rates exceeding 1 µm/hr with excellent crystallinity, surface smoothness, and at a low growth temperature. The key concept of S-MBE is to eliminate the first step of the two-step reaction mechanism involved in the growth of β-Ga2O3 by conventional MBE. In S-MBE, pre-oxidized gallium in the form of a molecular beam that is 99.98% Ga2O, i.e., gallium suboxide, is supplied. By eliminating the rate limiting step of conventional MBE—the oxidation of gallium to its suboxide—we achieve higher growth rates and avoid the etching that occurs in the conventional MBE growth of Ga2O3 at high fluxes of metallic gallium. Building upon S-MBE, we have studied Si-doped β-Ga2O3 films while maintaining a 1 µm/hr growth rate and high quality crystallinity, as confirmed by x-ray diffraction (XRD), atomic force microscopy (AFM), and reflection high-energy electron diffraction (RHEED). We investigate the incorporation and electrical properties of Si-doped β-Ga2O3 films using a variety of Si-based sources, including suboxide sources, with the goal of achieving replicable and controllable Si-doped β-Ga2O3 in the 1016 to 1018 cm-3 regime. The concentration of silicon incorporated as well as impurities present in the films are measured by secondary ion mass spectroscopy (SIMS). The electrical mobility and mobile carrier concentration is assessed by the Hall effect, including temperature-dependent Hall measurements. We have also fabricated and tested MESFETs from Si-doped β-Ga2O3 films grown by S-MBE at growth rates of 1 µm/hr.

2:45pm **EG-TuA-5 MOCVD Growth of Ga_2O_3 and $(Al_xGa_{1-x})_2O_3$*, Hongping Zhao,*** The Ohio State University **INVITED**

Ultrawide bandgap (UWBG) gallium oxide (Ga_2O_3) represents a promising semiconductor material with excellent chemical and thermal stability. Its wide energy bandgap (4.5-4.9 eV) predicts a breakdown field of 6-8 MV/cm, which is much larger than that of the 4H-SiC or GaN. The key advantages from this material system arise from the availability of high quality scalable bulk substrate and the capability of a wide range of doping.

Metalorganic chemical vapor deposition (MOCVD) growth technique has been demonstrated to produce high quality β-Ga_2O_3 thin films and its ternary $(Al_xGa_{1-x})_2O_3$ alloys. Record charge carrier mobilities approaching theoretical limit were reported from MOCVD grown materials. In this talk, I will discuss the control of background and n-type doping in MOCVD β-Ga_2O_3, and the impact of metalorganic precursor on Ga_2O_3 growth rate and material quality.

Growth and fundamental understanding of $(Al_xGa_{1-x})_2O_3$ with different phases are still limited. The limit of Al incorporation in beta-phase Ga_2O_3 has not been well understood or experimentally verified, although it was predicted up to 60% of Al composition could be incorporated into β-Ga_2O_3. In this talk, MOCVD growth of β-AlGaO with targeted Al composition of > 40%, n-type doping capability as a function of Al composition in $(Al_xGa_{1-x})_2O_3$, and MOCVD growth of different phase AlGaO will be discussed.

Acknowledgement: The authors acknowledge the funding support from the Air Force Office of Scientific Research FA9550-18-1-0479 (AFOSR, Dr. Ali Sayir) and the National Science Foundation (1810041, 2019753, 1755479).

Epitaxial Growth
Room Jefferson 1 & Atrium - Session EG-TuP
Epitaxial Growth Poster Session

EG-TuP-1 α-phase Gallium Oxide Thin Films Stabilized on a-, r- and m-plane Sapphire Substrates via Reactive Magnetron Sputtering and Pulsed Laser Deposition*, Edgars Butanovs,* Institute of Solid State Physics University of Latvia

Gallium oxide Ga_2O_3 has recently attracted a lot of scientific attention as a prospective ultra-wide bandgap semiconductor. It has five different polytypes α, β, δ, γ and ε among which the most studied and thermodynamically stable phase is β-Ga_2O_3. However, corundum-structured α-Ga_2O_3 with 5.2 eV bandgap is a better alternative for power electronics and ultraviolet optoelectronics applications. α-Ga_2O_3 is a metastable phase and it cannot be obtained as bulk crystals used for homoepitaxial growth. On the other hand, sapphire (α-Al_2O_3) is a convenient substrate for heteroepitaxy since some of its crystalline planes have a small lattice mismatch with α-Ga_2O_3, but there are only few and recent reports on the use of other orientation sapphire substrates than c-plane. In this work, we demonstrate growth of α-Ga_2O_3 thin films on a-, r- and m-plane sapphire wafers at various substrate temperatures via two different methods – reactive magnetron sputtering and pulsed laser deposition. Crystalline structure, elemental composition, surface morphology and optical properties were characterized by X-ray diffraction, X-ray photoelectron spectroscopy, scanning and transmission electron microscopy, atomic force microscopy and UV-VIS measurements. α-phase stability dependence on film thickness was also investigated. Such epitaxial stabilization of high-quality thin films with commonly used deposition methods is a perspective way how to integrate α-Ga_2O_3 on available substrates.

The financial support of Latvian Council of Science FLPP project LZP-2020/1-0345 is greatly acknowledged.

EG-TuP-2 Epitaxial Growth of $(Al_xGa_{1-x})_2O_3$ by Suboxide MBE*, Jacob Steele, K. Azizie, J. McCandless,* Cornell University*; T. Asel,* Air Force Research Lab*; H. Xing, D. Jena, D. Schlom,* Cornell University

Ga_2O_3 has garnered significant interest due in part to its ultra wide bandgap (~4.7ev) and large breakdown field which make it optimal for next-generation power devices. This already exceeds benchmark materials such as SiC and GaN but it is possible to alloy with Al to form $(Al_xGa_{1-x})_2O_3$ and further raise the bandgap up to 8.3 eV, higher than diamond. This can be desirable but presents a challenge for the most commonly researched phase, β-Ga_2O_3, as it is structurally unstable at higher Al concentrations, limiting the range of possible alloying. In contrast, α-phase Ga_2O_3 has been shown to alloy over the full range of x with one technique for growing α-$(Al_xGa_{1-x})_2O_3$ being molecular beam epitaxy (MBE). MBE is a powerful technique with one drawback being its relatively slow growth rate, which has a maximum of roughly 0.2 µm/hour. Fortuitously, the recent development of suboxide MBE, has enabled the epitaxial growth of β-Ga_2O_3 with growth rates exceeding 1 µm/hr without compromising film quality.

This work investigates the application of suboxide MBE to the growth α-$(Al_xGa_{1-x})_2O_3$ thin films. The goal of this project is to determine whether suboxide MBE can grow high quality epitaxial α-$(Al_xGa_{1-x})_2O_3$ over the full range of x with growth rates exceeding 1 µm/hr. For this suboxide MBE study, gallium suboxide and elemental aluminum sources were used and the ozone pressure, growth temperature, Al_2O_3 substrate orientation, gallium suboxide flux, and elemental aluminum flux were varied to map out promising growth conditions. Our experiments reveal that a-plane sapphire substrates consistently enable the epitaxial growth of α-$(Al_xGa_{1-x})_2O_3$ over a broad range of growth conditions. We also show that the growth rates of α-$(Al_xGa_{1-x})_2O_3$ using suboxide MBE approach 1 µm/hr with high surface quality by rocking curves and atomic force microscopy measurements. Lastly, we demonstrate that the aluminum content of the α-$(Al_xGa_{1-x})_2O_3$ films covers a large range of x including the high x values where β-$Ga_2O_3$is unstable.

EG-TuP-5 Free Carrier Control in Homoepitaxial β-Ga_2O_3 Thin Films by Tin Impurity Doping*, Neeraj Nepal, B. Downey, V. Wheeler, D. Katzer, E. Jin, M. Hardy, V. Gokhale, T. Growden,* US Naval Research Laboratory*; K. Chabak,* Air Force Research Laboratory*; D. Meyer,* US Naval Research Laboratory

Ultra-wide bandgap (UWBG) semiconductors such as c-BN, AlN, high Al content AlGaN, β-Ga_2O_3, anddiamond, with a bandgap greater than 3.4 eV, have higher figures of merit values than GaN and SiC for power and rf devices making them candidates for next generation high-power/temperature electronic materials [1-3]. The availability of inexpensive large-area bulk substrates synthesized by melt growth techniques at atmospheric pressure provides a scaling advantage for β-Ga_2O_3 over other UWBG semiconductors [2]. In addition, homoepitaxial growth on bulk substrates offers the potential of low defect density films for vertical power devices. Further, controlled n-type doping with a shallow donor level (15-50meV [4]) is another advantage of β-Ga_2O_3 compared to AlN and high Al-content AlGaN. For these reasons, homoepitaxial growth of unintentionally- and impurity-doped Ga_2O_3 films and their electrical and structural properties are of great interest.

In this paper, we report MBE growth and Sn impurity doping of β-Ga_2O_3 thin films on (010) β-Ga_2O_3 substrates from Novel Crystal Technology and Northrop Grumman SYNOPTICS vendors. Maintaining smooth surface in in-situ RHEED, the growth rate was increased approximately from 1 to 3nm/min by optimizing the growth conditions such as Ga flux, plasma conditions and growth temperature (T_g). At optimal conditions with a T_g of 725 ^oC, surface roughness, and X-ray rocking curve full-width at half maximum were about 0.36 nm, and 20 arc-sec for ~390 nm thick films, respectively. Optimal growth conditions that resulted in high structural and surface quality were used to explore doping parameter space using Sn impurity. The Sn cell temperature was varied from 570 to 630 ^oC to control incorporated Sn concentration and hence free carrier density. Hall effect measurements were carried out on Sn-doped layers using In-dots contacts, and selected samples were processed into van der Pauw Hall measurement structures to verify the In-dot-based Hall effect values. Hall effect measurements demonstrate that free carrier density can be controlled in the range $1x10^{16}$ to $3x10^{19}$ cm^{-3}. A mobility of 49 cm^2/V-s with free carrier density of $3x10^{19}$ cm^{-3} was measured which is comparable to the previously reported values for Sn-doped β-Ga_2O_3 [5].

This work was funded by the ONR and OSD.

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EG-TuP-6 MBE Growth of Doped and Insulating Homoepitaxial β-Ga_2O_3*, Jon McCandless, V. Protasenko, B. Morell,* Cornell University*; E. Steinbrunner, A. Neal,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA*; Y. Cho, N. Tanen, H. Xing, D. Jena,* Cornell University

The group IV atoms Si, Ge, and Sn have been investigated as *in-situ* n-type dopants in the molecular beam epitaxy (MBE) growth of β-Ga_2O_3. However, it has remained challenging to achieve controllable and intentional n-type doping of low doping densities in MBE due to the tendency of the dopants to oxidize during the growth. As a result, doping is restricted to a small window of growth conditions. Recently, we have overcome this doping challenge by modifying the effusion source design. This modification allowed us to achieve room temperature mobilities of ~130 cm^2/Vs at $1\times10^{17}/cm^3$ with Si doping.

Variations in the transport measurements between samples doped under similar conditions was observed. For example, some unintentionally doped samples (UID) exhibit electrically insulating behavior in Hall effect measurements, while others exhibit measurable conductivity. Secondary ion mass spectrometry (SIMS) measurements revealed that Si, which is universally observed at the substrate-film interface, and Fe (the compensating acceptors included in the substrate to make it semi-insulating) varied from sample to sample. Depending on the chosen substrate, this resulted in the variability of the nominal conductivity of UID samples.

To address this it is necessary to grow an additional layer, doped with deep, compensating acceptors, on top of the substrate to compensate the interface charge. Here we will present our latest research on controllable doping, design of a compensating layer to achieve insulating films, and the impact these have on the growth of field-effect transistors.

EG-TuP-7 High Conductivity Homoepitaxial β-Ga_2O_3 Regrowth Layers by Pulsed Laser Deposition*, **Hyung Min Jeon,*** KBR*; K. Leedy,* Air Force Research Laboratory

With a high critical field strength, wide bandgap, and transparence, β-Ga_2O_3 is a unique material for high power switching and amplifier applications, as well as optoelectronic applications as it is a transparent conductive oxide. In semiconductor devices, doping of the semiconductor is necessary to modulate a semiconductor's electrical properties. Degenerately doped semiconductors have particular use as an ohmic contact regrowth layer. Highly conductive, measured 2323 S/cm (4.3 x10^{-4} Ω-cm resistivity), homoepitaxial Si-doped epitaxial β-Ga_2O_3 films by pulsed laser deposition has been fabricated in this work. A commercial pulsed laser deposition system with a KrF excimer laser was used for deposition with a Ga_2O_3 - 1 wt. % SiO_2 target. The base pressure of PLD chamber was 2.66 × 10^{-6} Pa with 55 sccm Ar gas introduced during deposition. The substrate temperature, rotating at 30 º/sec, was 590 ºC. The exceptional electrical properties of the Si-doped epitaxial β-Ga_2O_3 films are 2.24 × 10^{20} cm^{-3} carrier concentration and 64.5 cm^2/Vs Hall mobility. The calculated electrical activation efficiency is 77 % calculated using the Si content from secondary ion mass spectrometry depth profile. For practical use of this highly conductive layer in a semiconductor device, large area deposition uniformly is essential. However, PLD suffers from limitation of scale up area due to the point laser energy source. We found that locally non-uniformed and less-conductive layers cause degradation of the electrical properties in large area film. We examined the growth conditions that address this local conductivity non-uniformity and present solutions using improved deposition parameters.A uniform, low resistivity β-Ga_2O_3 layer is anticipated to enable increased tunneling current between metal contacts and β-Ga_2O_3 transistor channel layers reducing power dissipation and improving overall transistor performance. Moreover, wide bandgap β-Ga_2O_3 with unusually high conductivity can be a suitable candidate for future optoelectronic applications.

EG-TuP-9 Highly conductive β-Ga_2O_3 and $(Al_xGa_{1-x})_2O_3$ epitaxial films by MOCVD*, **Fikadu Alema,*** Agnitron Technology*; T. Itoh, J. Speck,* Materials Department, University of California, Santa Barbara*; A. Osinsky,* Agnitron Technology

We report on the growth of highly conductive (n>1020 cm-3) Si or Ge doped β-Ga2O3 and β-$(Al_xGa_{1-x})_2O_3$ epitaxial films grown on (010) β-Ga_2O_3 substrates by MOCVD. Triethylgallium (TEGa), triethylaluminum (TEAl), and pure oxygen were used as Ga, Al, and O_2 sources. Silane (SiH_4) and germane (GeH_4) diluted in nitrogen were used as sources for Si and Ge. The layers were grown at a lower substrate temperature (~500-600 ºC); the effects of O_2 flow, film growth, pressure, and dopant flow rates on the incorporation and activation efficiency of the dopants were studied. By carefully adjusting these process conditions, doping limitations for epitaxial β-Ga_2O_3 and AlGaO alloys with various Al composition were defined. With Si dopant, films with the conductivity of ~2515 S/cm (μ=50.7 cm^2/Vs, n =3.1x10^{20} cm^{-3}) were achieved as determined by Hall effect measurements. This result sets a record compared to the best conductivity value reported in the literature, 2323 S/cm in a layer grown by PLD [1]. Similarly, despite the challenges with Ge doping due to its severe process dependence, highly conductive Ge-doped layers with the conductivity of ~1580 S/cm (μ=38 cm^2/Vs, n = 2.6x10^{20} cm^{-3}) were realized. Temperature-dependent Hall measurement showed no charge density dependence on temperature both for heavily Ge and Si-doped films, indicating degenerate doping. The heavy Si doping process developed for β-Ga_2O_3 has also been extended to β-$(Al_xGa_{1-x})_2O_3$ with varying Al content. The AlGaO layer thickness and Al content in the films were estimated by HRXRD measurement, while Hall effect measurements were used to study the electrical characteristics of the layers. For coherently strained, ~70-75 nm thick, AlGaO layers with Al content of 12.3%, films with a conductivity of 612 S/cm (μ=31.1 cm^2/Vs, n =1.23x10^{20} cm^{-3}) were achieved. However, this value was reduced to ~220 S/cm (μ=25 cm^2/Vs, n =5.5x10^{19} cm^{3}) when the Al content increased to 22%. The effects of AlGaO layer thickness and Al content on Si incorporation, surface roughness, free carrier concentration, and electron mobility in AlGaO were studied and will be discussed. The high free carrier concentration in Ga2O3 and AlGaO thin films achieved by MOCVD at low epitaxial growth temperature enables low resistance ohmic contact layers to realize high-performance β-Ga_2O_3 and β-$(Al_xGa_{1-x})_2O_3$/β-Ga_2O_3 heterostructure devices [2].

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