## Bulk Growth
## Room Bansal Atrium - Session BG-MoP
## Bulk Growth Poster Session I

**BG-MoP-1 MOCVD Development for Growth of $Ga_2O_3$ Over Large Areas*,* *Muhammad Ali Johar, A. Feldman, G. Provost, K. Vasudevan,* Structured Materials Industries, Inc*; L. Lyle,* Pennsylvania State University*; L. Porter,* Carnegie Mellon University, USA*; A. Popp,* Leibniz-Institut für Kristallzüchtung (IKZ)*; G. Tompa,* Structured Materials Industries, Inc

Growth of high crystal quality gallium oxide ($Ga_2O_3$) at large production scale throughput is not available in the market due to (a) a ready high-volume supply of large wafers for homoepitaxy, (b) MOCVD tools explicitly configured for large area MOCVD growth of $Ga_2O_3$ homo- or heteroepitaxy. One of the primary factors is the unavailability of manufacturing scale substrates and growth tools.

Our company “Structured Materials Industries, Inc, (SMI)” has designed, sold, and fielded several $Ga_2O_3$ MOCVD growth tools as well as adapted several pre-existing (for different materials) tools to grow $Ga_2O_3$ and operated in-house tools for growth on single ($cm^2$ scale, 50mm, 100mm, 150mm, and 200mm) and multiple wafer tools (3×50mm, 19×50mm, and 38×50mm wafer diameters) or multiple 100mm) depositions. These tools have operated using a range of heating systems – induction, radiant filament, and lamps to operate over a range of growth temperatures (~400°C to ~1100°C) using a multiple of binary and alloy growth precursors ($O_2$, $N_2O$, $H_2O$, TEGa, TMGa, TMIn, TMAl, TEAl, $GaCl_3$), process enhancers such as HCl, and dopants ($SiH_4$, TEOS, TMSn) and are compatible with a wide range of other elements.

The results of these works have led SMI to refine a series of reactors for research to production applications – spanning single to multiple wafer tools employing a multiple of growth parameters and heater types. In this presentation, we will review our series of reactor designs and how they are best applied – for example few through multiple small or large wafer epitaxy is best produced using advanced patented (US7573004 B1) high temperature oxide compatible designed filaments in rotating disc reactors supporting growth through 900°C whereas very high temperature growth – through 1100°C is best produced using induction heating when such high temperatures are warranted, whereas enclosed filament geometries are best for large single wafer processing. We note that filament heating is sufficient for most applications and can be used to produce uniform temperature heating. Reactor designs, supported by simulations of a variety of parameters, such as gas mixtures, flows, temperatures, pressures, carrier flows, flow rates of precursors, and the gap between showerhead and susceptor, for example, are reviewed along with areas of demonstrated results. Overall, production tool designs are based upon optimized parameters and they are reviewed.

**BG-MoP-2 Quality Improvement of Sn-doped $\beta$-$Ga_2O_3$ Single Crystal by Optimizing Temperature Gradient Control in Growth Zone*,* *Su-Min Choi, H. Jang, S. Seo, M. Chae, M. Park, Y. Jang,* Department of Advanced Materials Engineering, Dong-Eui University, Republic of Korea*; Y. Moon, Y. Sung, J. Kang,* AXEL, Republic of Korea*; Y. Shin, S. Bae,* Korea Institute of Ceramic Engineering and Technology, Republic of Korea*; W. Lee,* Department of Advanced Materials Engineering, Dong-Eui University, Republic of Korea

As an ultra-wide bandgap semiconductor(UWBG), $\beta$-$Ga_2O_3$ is a very promising materials for various applications such as UV photodetectors, power rectifiers, gas sensors, MOSFETs, SBDs, and LEDs because $\beta$-$Ga_2O_3$ has a wide bandgap of 4.9eV and a high breakdown voltage of 8MV/cm. Furthermore, since $\beta$-$Ga_2O_3$ is capable of melt growth, it is possible to obtain a higher growth rate and a lower manufacturing cost than other WBG semiconductors such as SiC, GaN, and Diamond.[1,2]

In this study, the quality improvement of Sn-doped $\beta$-$Ga_2O_3$ crystal was systematically investigated with controlling temperature gradient in growth cell of edge-defined film-fed growth (EFG) method. Flat-shaped $\beta$-$Ga_2O_3$crystal ribbons were typically grown by the EFG method. The principal surface and the growth direction were set to be (001) and [010], respectively. To prepare Sn-doped n-type bulk crystals, different amounts of Sn powder were added to $Ga_2O_3$ source powder. The temperature gradient in growth cell was controlled by changing structure of surrounding refractory materials and using the after-heater(A/H). The doping characteristics and crystal quality of various $\beta$-$Ga_2O_3$ crystal ribbons grown with changing the temperature gradient were systematically analyzed. A phase analysis of $\beta$-$Ga_2O_3$ bulk crystals was performed by Raman analysis, and the quality of the crystals was analyzed by a high-resolution X-ray diffraction (HRXRD). Electrical properties and impurity concentration of unintentionally doped (UID) $\beta$-$Ga_2O_3$ and Sn-doped $\beta$-$Ga_2O_3$ crystals were analyzed using the Hall effect measurement system and the secondary ion mass spectrometry (SIMS), respectively.

High-quality Sn-doped (001) $\beta$-$Ga_2O_3$ crystal with a full width at half maximum (FWHM) of 38 arcsec and an etch pit density (EPD) of $3700/cm^2$ was obtained with optimizing temperature gradient. Proper temperature gradient resulted in the reduction of $SnO_2$ volatilization during the growth process and the uniform distribution of bluish color throughout the entire ribbon. The carrier concentration of $\geq 10^{18}/cm^3$ was obtained on Hall effect measurement.

Reference

[1] S. Ohira et al., Thin Solid Films 516, 5763-5767 (2008)

[2] Stephan Lany, APL Mater. 6, 046103 (2018)

**BG-MoP-4 Various Crystal Planes and their Characteristics obtained from $\beta$-$Ga_2O_3$ Single Crystal Blocks Grown by the Multi-slit Structure of the EFG Method***, Y. MOON,* AXEL, Republic of Korea*;* ***HUIYEON JANG,*** Dongeui University, Republic of Korea*; Y. SUNG,* AXEL, Republic of Korea*; S. CHOI, M. CHAE, S. SEO, M. PARK, Y. JANG, W. LEE,* Dongeui University, Republic of Korea*; Y. SHIN, S. BAE,* Korea Institute of Ceramic Engineering and Technology, Republic of Korea*; T. LEE, H. KIM,* Korea Institute of Industrial Technology, Republic of Korea*; J. KANG,* AXEL, Republic of Korea

$\beta$-$Ga_2O_3$ 4.9eV 의. It has a wide bandgap and a high breakdown voltage of 8MV/cm, so it is receiving a lot of attention for power device applications. In addition, it has the outstanding advantage of being able to grow a single crystal with a higher growth rate and lower manufacturing cost than other WBG semiconductor materials such as SiC, GaN, and diamond. [1-4] In this study, a $\beta$-$Ga_2O_3$ crystal block with a thickness of 10 mm was grown on an Ir die with a multi-slit structure through an edge-define film-fed growth (EFG) process. The growth direction and main surface of the $\beta$-$Ga_2O_3$ crystal were set to the [010] direction and the (100)/(001) plane, respectively, and the crystal growth rate was set to about 12 mm/h. . The crystalline blocks were annealed in a nitrogen environment to reduce residual stress in the crystals before preparing crystal substrates with different orientations. A chemical mechanical polishing (CMP) process using Hastilite Ditron 3.0 slurry was chosen as the final processing step to create an epi-ready surface of the $\beta$-$Ga_2O_3$ crystal substrate. Raman spectra showed successful growth of $\beta$-$Ga_2O_3$ bulk crystals. Crystal quality was assessed by high-resolution X-ray diffraction (HRXRD). Etch pit density (EPD) and defect types were measured on crystal surfaces etched with $H_3PO_4$ solution.

Etch pit density (EPD) and defect type were measured on the liquid etched crystal surfaces. A typical Sn-doped Ga 2 O 3 crystal block grown by the EFG method was bluish. The X-ray rocking curve values of the $Ga_2O_3$ single crystal ribbon depend on the Sn doping concentration and show slightly different values depending on the measurement position in the $Ga_2O_3$ crystal ribbon, indicating the spatial change of the $Ga_2O_3$ crystal quality. decision. The crystal quality and surface properties of various β-Ga2O3 crystal substrates with (100), (001), (-201), and (hkl) orientations after CMP process were systematically investigated.

Reference:

[1] JY Tsao 등, *Adv. 전자. 엄마.* 4, 1600501 (2018)

[2] M. Higashiwaki, GH Jessen, *Appl. Physics Rhett. Man* 112, 060401 (2018)

[3] Kun Zhang 외, *J. Alloys and Compounds,*881,160665 (2021)

[4] Shengnan Zhang 외, *J. Semicond.* 39, 083003 (2018)

**BG-MoP-5 Investigation of Defects in(100) and (001) β-$Ga_2O_3$Single Crystal GrownbyEFG Method***, M. Choi,* Korea Institute of Ceramic Engineering and Technology/Pusan National University, Republic of Korea*; **Yun-Ji Shin,*** Korea Institute of Ceramic Engineering and Technology, Republic of Korea*; W. Jeong, T. Gu, A. Shin, S. Cho,* Korea Institute of Ceramic Engineering and Technology/Pusan National University, Republic of Korea*; Y. Moon, J. Kang,* AXEL, Republic of Korea*; W. Lee,* Dong-Eui University, Republic of Korea*; S. Jeong,* Korea Institute of Ceramic Engineering and Technology, Republic of Korea*; S. Harada,* Nagoya University, Japan*; K. Ishiji,* Kyushu Synchrotron Light Research Center, Japan*; H. Lee,* Pusan National University, Republic of Korea*; S. Bae,* Korea Institute of Ceramic Engineering and Technology, Réunion

Gallium oxide ($Ga_2O_3$) has been highlighted as an emerging material for power semiconductor applications [1]. Recent achievements in bulk substrates and epitaxy pave a way for demonstrating several kV level power devices. However, various defects in β-$Ga_2O_3$ such as dislocations, stacking faults, and twin defects have been still issued, requiring further crystal quality with defect analysis.X-ray topography is one of powerful techniques to observe defect distribution in a non-destructive way [2].The etch pit technique destructively visualize the defect distribution of the substrate as it chemically etches down the defect regions with an acid (or base). In this study, we compare the defect characteristics of (100) and (001) β-$Ga_2O_3$ single crystals grown by edge-defined film-fed growth (EFG).

In experiment, the etch pits of (100) and (001) β-$Ga_2O_3$ single crystals were formed by wet etching with 85 wt% phosphoric acid ($H_3PO_4$) solution at 140 °C for 120 min. The profile of etched surfaces was observed using atomic force microscope. In addition, (001)-oriented β-$Ga_2O_3$ single crystal was analyzed using an X-ray topography (Beamline 09, Synchrotron Radiation Facility SAGA-LS).

Figure 1 shows the AFM images of the etched surface for (100) and (001) surfaces. The etch pit of (100) plane are shallower and narrower compared to (001) plane as the (100) plane is parallel to the dislocation direction and has a cleavage property [3]. The density of the etch pits in the (100) and (001) planes were counted to be ~$3.8\times10^5$ $cm^{-2}$ and ~$6.0\times10^4$ $cm^{-2}$, respectively. Figure 2 shows the XRT image of (001) β-$Ga_2O_3$ with a diffraction vector, ***g***=605. It was found that the dominant features were dot, line along [010] direction, and curved line contrasts. The line contrasts are mostly associated with b-axis screw dislocations with Burgers vectors (b) parallel to [010], and the dot contrasts are related to edge dislocations with <001> burgers vectors [4]. In addition, many wandering dislocations are observed on the (001) surface, which show curved contrasts. This defect observation might assist to improve the crystal quality of $Ga_2O_3$ substrates as we perform further in-depth characterization in the future.

References

[1] J. Y. Tsao, et al., *Adv. Electron. Mater.* 4, 1600501 (2018)

[2] M. Higashiwaki, et al., *Appl. Phys. Lett. 100, 013504 (2012).*

[3] Kun Zhang, et al., *J. Alloys and Compounds,* 881,160665 (2021)

[4] Shengnan Zhang, et al., *J. Semicond.* 39, 083003 (2018)

# Tuesday Afternoon, August 15, 2023

## 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.

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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

## Bulk Growth
## Room Bansal Atrium - Session BG-TuP
## Bulk Growth Poster Session II

**BG-TuP-5 β-$Ga_2O_3$ Single Crystal Growth by EFG Method using Die with Multi-Slit Structure***, **Yeon-Geun Seong**, Y. Moon,* Axel, Republic of Korea*; H. Jang, S. Choi, C. Min-Ji, S. Seo, M. Park, Y. Jang, W. Lee,* Dongeui University, Republic of Korea*; J. Kang,* Axel, Republic of Korea

β-$Ga_2O_3$is attracting attention as a next-generation power semiconductor. β-$Ga_2O_3$ has a high bandgap of 4.9eV and a high breakdown voltage of 8MV/cm. In addition, β-$Ga_2O_3$ grown by the EFG (Edge Defined Film-Fed Growth) method is superior to other power semiconductor materials such as SiC and GaN due to its fast growth rate and low manufacturing cost. However, since the β-Ga2O3 crystal grown by the EFG method grows in a ribbon morphology, the number of wafers that can be extracted from one ingot is small. [1-4]

In this study, the thickness of the ingot was increased through a die with multi-slit structure. Crystal growth from multi-slit structure is divided into 'diameter direction', which determines the size of the wafer, and 'thickness direction', which determines the extraction numbers of wafer. As a result of β-Ga2O3 growth experiments using Muliti-Slit Die, we found that thick crystal growth is difficult if the growth rate in the diameter direction is too fast, and polycrystals are easily to occur if the growth rate in the thickness direction is too fast. Therefore, in order to overcome these problems, the two-dimensional temperature distribution and the temperature gradient in the vertical direction were adjusted to secure reproducibility to stably grow thick crystal with high crystallinity.

As a result of the experiment, various process conditions, such as the type and structure of insulation, three-dimensional temperature gradient, and pulling speed, had a more sensitive effect on the growth of thickness direction in multi-slit die compared with single-slit die. By adjusting the thermal balance of upper and lower parts of crucible and the temperature gradient of die in diameter direction and thickness direction, the growth of thick β-Ga2O3 single crystals was successfully achieved. This result can contribute to lower the manufacturing cost of Ga2O3 crystals as a substrate for power semiconductor fabrication.

Reference

[1] J. Y. Tsao et al, Adv. Electron. Mater. 4, 1600501 (2018)

[2] M. Higashiwaki, G.H. Jessen, Appl. Phys. Lett. 112, 060401 (2018)

[3] Kun Zhang et al, J. Alloys and Compounds, 881,160665 (2021)

[4] Shengnan Zhang et al, J. Semicond. 39, 083003 (2018)

## 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 $2.25 \times 10^{-6}$Ω·cm$^2$ and sample A had the highest of $1.99 \times 10^{-4}$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 $3.8\times10^{-9}$ to $2.0\times10^{-8}$ 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-$(Al_xGa_{1-x})_2O_3$ ($x \leq 15\%$) 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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