Program Overview

Room /Time	Jefferson 2-3
MoM	BG-MoM: Bulk & Epitaxy I

Bulk Growth
Room Jefferson 2-3 - Session BG-MoM
Bulk & Epitaxy I

Moderator: John Blevins, Air Force Research Laboratory

10:45am **BG-MoM-10 b-Ga_2O_3 Growth and Wafer Fabrication**, *A. Brady, G. Foundos,* ***Chase Scott***, Northrop Grumman SYNOPTICS*; V. Gambin,* Northrop Grumman Corporation*; K. Stevens,* Northrop Grumman SYNOPTICS*; J. Blevins,* Air Force Research Laboratory, Afghanistan **INVITED**

SYNOPTICS began growing b-Ga_2O_3 under an AFRL contract FA8650-15C-1796 in 2015. Currently, 50mm diameter boules are consistently grown to lengths up to 35mm from seeds oriented along the [010] direction. SYNOPTICS has grown boules as both unintentionally doped (UID) and doped with Mg and Fe using high purity (99.99+%) oxides to obtain semi- and highly insulating substrates. Doping levels for Mg were 0.10mol% and for Fe ranged from 0.0025mol% to 0.0100mol%.

Crystal growth is performed at the melting point of 1820°C via the Czochralski method using iridium crucibles in an induction-heated furnace using high purity Ga_2O_3 powder (99.999%). Ga_2O_3 is known to dissociate at high temperatures into sub-oxides including GaO and Ga_2O with O_2 evolution in oxygen-deficient atmospheres [1]. Growth in an iridium crucible, however, requires a low-oxygen environment to reduce oxidation of Ir to IrO_2 which leads to excessive crucible degradation and Ir-related defects in the crystal. SYNOPTICS growth chambers use free-flowing gas at ambient pressure to control the atmosphere. In order to suppress the dissociation of Ga_2O_3 and minimize excess oxidation of Ir, a mixed CO_2 and O_2 growth atmosphere is supplied to the melt surface. The crystals are rotated between 2 and 10 RPM and pulled at rates ranging between 0.5mm/hr and 2mm/hr during growth. Boules grown are largely free of twins and will occasionally have cleavage cracking on the (100) and (001) planes, the former being predominant.

The as-grown boules are oriented to within ~1° of the [010] growth axis and prepared for coring or shaping. 25mm diameter cores are drilled. 50mm diameter cores are obtained by turning down the entire boule on a lathe with a diamond tool. These cores are then encased in epoxy and sliced to ~700mm wafers using a multi-wire saw with a 150mm wire and B_4C loose abrasive slurry. The easily-cleaved (100) plane is used as the reference flat for wafers. The wafers are chemical mechanical polished to average surface roughness of <2Å via AFM scans of 20mm x 20mm. High resolution x-ray rocking curve measurements of the (020) show good crystal quality with FWHM of 63 arc-sec averaging parallel and perpendicular to the (100) reference flat respectively. Total thickness variation of less than 20 microns has been achieved on 50 mm epi-ready wafers.

References:

1. Z. Galazka et al., Cryst. Res. Technol. 45, No. 12, 1229 – 1236 (2010)

11:15am **BG-MoM-12 Increasing the Bandgap of β-Ga_2O_3 via Alloying with Al_2O_3 or Sc_2O_3 in Czochralski-grown Crystals**, ***Benjamin Dutton***, *J. Jesenovec, B. Downing, J. McCloy,* Washington State University

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Widening the bandgap of β-Ga_2O_3 is desirable for improving the critical breakdown field and enabling transmission further into the ultraviolet. In this work, alloyed β-Ga_2O_3 bulk single crystals were grown by the Czochralski and vertical gradient freeze methods with batched alloy compositions of β-$(Sc_{0.1}Ga_{0.9})_2O_3$ (10 mol.% Sc_2O_3, SGO) and β-$(Al_{0.1}Ga_{0.9})_2O_3$ (10 mol.% Al_2O_3, AGO). Dopant incorporation and site occupancy were analyzed by X-ray fluorescence and nuclear magnetic resonance, respectively. Al incorporated more readily into the Czochralski pulled crystal with approximately 11.7 mol.% Al_2O_3 present, while only 6.2 mol.% Sc_2O_3 incorporated into the Czochralski pulled SGO crystal.Lattice changes were characterized by powder X-ray diffraction, X-ray rocking curve, and Raman spectroscopy, with AGO exhibiting higher crystal quality than SGO. Optical transmission measurements (200 nm – 20 µm) indicated a shift to a larger bandgap in both AGO and SGO compared to undoped β-Ga_2O_3. AGO samples were highly insulating, with resistivities on the order of 10^{11} Ω·cm, while SGO samples exhibited resistivities in the range of 10^1 - 10^2 Ω·cm. SGO exhibited luminescence characteristic of rare earth element impurities, originating from the Sc_2O_3 precursor powder. AGO in general appears to be a better candidate for producing epi-ready substrates compared to SGO, due to better lattice match and improved incorporation into the growing crystal without precipitation of secondary phases.

11:30am **BG-MoM-13 Chemi-Mechanical Polishing and Subsurface Damage Characterization of 2-inch (010) Semi-Insulating β-Ga_2O_3 Substrates**, ***David Snyder***, Penn State Applied Research Laboratory

Results will be presented for the development of a high removal rate 2-step chemi-mechanical polishing (CMP) process for epi-ready 2-inch semi-insulating (010) β-Ga_2O_3 substrates grown by the Czochralski process at SYNOPTICS.First, the depth of subsurface damage was characterized to determine how much material needed to be removed from the surface. Using a combination of x-ray rocking curves (XRRC's) and cross-sectional SEM imaging the maximum depth of damage was shown to be on the order of 75 µm after multi-wire sawing with 150 µm wire and B4C loose abrasive slurry.This was significantly deeper than predicted from abrasive size correlations and surface roughness measurements of the as-sawn wafers.

To minimize processing time, we developed a two-step polishing process with the target removal of 75 µm using an intermediate process and 25 µm using a final CMP step. For the initial step, several options for polishing slurries, including colloidal alumina and diamond, were evaluated. To further understand and quantify subsurface damage we used in-line x-ray diffraction (XRD). Unmounted wafers were characterized, and we also developed custom polishing plates that could be mounted within a high-resolution XRD system so that rocking curves could be collected at intermediate points in the process without removing wafers from the polishing plate.These scans were collected as a function of removal amount and were found to be surprisingly sensitive to subsurface damage.Excellent results were achieved with 1-3 µm diamond with very high removal rates on the order of 25 µm/hr resulting in a process time of only 3 hrs for the initial 75 µm removal

Subsequently, we studied alternative final CMP steps and identified two options as promising final steps.We studied pH modified colloidal silica over the pH range of~3 to 9.At a pH of 4, we were able to get extremely low surface roughness values (<2 Å) and excellent XRRC FWHM values (40 arcsec) while increasing removal rate by ~2.5X to nearly 1.0 µm/hr.Similar results were obtained using pH modified nano-diamond providing a potential route to a non-silica based polishing process.

11:45am **BG-MoM-14 Ge-Delta Doped *β*-Ga_2O_3 Grown Via Plasma Assisted Molecular Beam Epitaxy**, ***Thaddeus Asel***, Air Force Research Laboratory, Materials and Manufacturing Directorate, USA*; E. Steinbrunner,* Wright State University, Department of Electrical Engineering*; J. Hendrick,* Air Force Institute of Technology, Department of Engineering Physics*; A. Neal, S. Mou,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA

We utilized secondary ion mass spectroscopy (SIMS) and capacitance voltage measurements to develop the capability to delta dope *β*-Ga_2O_3 thin films with Germanium grown via plasma assisted molecular beam epitaxy (PAMBE). Doping PAMBE grown *β*-Ga_2O_3 has proven to be a challenge in the community. Early Si delta doping studies showed promise with a Si background of 1×10^{17} cm^{-2} with good sheet carrier concentrations of 1.2×10^{13} cm^{-2}.[1] However, the quartz plasma bulb has been demonstrated to be a source of unintentional Si doping. Additionally, a background can be observed from the Si cell itself when it is left hot in the chamber with the shutter closed during growth.[2] This effect appears to be based on chamber geometry as it is not consistent from system to system, but does prevent Si from being consistently used for delta doping studies. Ge is another n-type dopant that could allow for delta doped structures to be grown via PAMBE. There are difficulties tuning Ge doping in *β*-Ga_2O_3, such as the sensitivity of substrate temperature where the Ge doping can vary over two orders of magnitude over a 100 °C range. Site competition occurs as well, as the Ga beam flux is increased during growth, the Ge concentration will drop as well. Uniform doping with Ge seems unlikely as the Ge incorporation decreases as a function of time, indicating that the ge source material is oxidizing during the growth. However, the background doping caused by Ge is in the range of $1\text{-}7 \times 10^{15}$ cm^{-3} as seen in SIMS as opposed to 1×10^{18} cm^{-3} for Si in our chamber. Doping with Ge up to 5 ×

10^{19} cm^{-3} has been achieved as well. This suggests Ge is a good candidate for delta doping applications. Using a Ge cell temperature of 700 °C, a substrate temperature of 550 °C, a delta doped structure that was capped with 50 nm of unintentionally doped *б*-Ga_2O_3 was grown and then measured by CV. The CV profile revealed a sharp peak at 58 nm into the sample with an integrated sheet concentration of 1×10^{13} cm^{-2} and a peak width of 12 nm at 10% of the maximum volume concentration. In summary, this demonstrates that Ge is a good dopant choice for delta doping in PAMBE, providing a lower background density than that of Si doping.

[1] Z. Xia, C. Joishi, S. Krishnamoorthy, S. Bajaj, Y. Zhang, M. Brenner, S. Lodha, and S. Rajan, IEEE Electron Device Letters **39**, (4) 568-571 (2018).

[2] T.J. Asel, E. Stein Brunner, J. Hendricks, A.T. Neal, and S. Mou,J. Vac. Sci. Technol. A **38**, 043403 (2020).

12:00pm **BG-MoM-15 High Purity n-type β-Ga_2O_3 Films with 10^{13} cm^{-3} Residual Accepter Concentration by MOCVD**, ***Andrei Osinsky***, *F. Alema,* Agnitron Technology

MOCVD growth of epitaxial Ga_2O_3 films using TMGa precursor has not been widely investigated due to its challenges with carbon incorporation and difficulty in realizing device quality films. However, given its high vapor pressure and shorter reaction kinetics, it offers a fast growth rate,which is vital for Ga_2O_3 based high voltage power devices that require tens of microns of layer thickness. With optimal process conditions and a suitable MOCVD reactor, the TMGa precursor has the potential to produce films with comparable or even better material quality to those grown using the more popular TEGa precursor. In this work, we will present the growth of high purity homoepitaxial β-Ga_2O_3 thin films by using a TMGa precursor with LT electron mobility exceeding 23,000 cm^2/Vs and acceptor concentration of $2x10^{13}$ cm^{-3}[1]. A wide range of controllable doping of TMGa grown Ga_2O_3 films with Ge and Si dopants will be discussed. Smooth films with RT electron mobility of >130 cm^2/Vs for n ~$3x10^{17}$ $1/cm^3$ have been demonstrated. Temperature-dependent Hall measurement will be used to identify the nature of impurities in the TMGa grown films lightly doped with Ge and Si and the results will be compared with TEGa grown films doped in similar conditions. The obtained results confirm the suitability of TMGa precursor for the growth of high purity β-Ga_2O_3 films at a fast growth rate, meeting the demands for commercializing Ga_2O_3 based high voltage power devices by MOCVD. In this presentation, we will also report on the intentional doping of Ga_2O_3 films with nitrogen and carbon to grow semi-insulating films.

[1] G. Seryogin, et al. Appl. Phys. Lett. **117** 262101 (2020).

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