

# Tuesday Morning, August 15, 2023

## Advanced Characterization Techniques

Room Davis Hall 101 - Session AC+MD-TuM

### Characterization/Modeling IV

Moderator: Baishakhi Mazumder, University of Buffalo, SUNY

10:45am **AC+MD-TuM-10 Defects in Ga<sub>2</sub>O<sub>3</sub>: An Ultra-high Resolution Electron Microscopy Study**, *Nasim Alem*, The Pennsylvania State University; *A. Chmielewski*, CEMES-CNRS, France

INVITED

Interest in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> has dramatically increased in recent years due to the material's potential promise for use in power electronics and extreme environments. Its combination of a monoclinic structure (C2/m space group), two inequivalent tetrahedral and octahedral gallium sites and three inequivalent oxygen sites, and a bandgap of 4.8 eV, 1.4 eV above that of gallium nitride, creates a semiconductor material with a unique set of properties. This is further aided by  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>'s uncommon capability among the ultra-wide bandgap oxides to be grown into high quality single crystal substrates using both melt-based bulk and thin film growth and deposition methods. Defects and their stability and dynamics under static and extreme environments can limit the incorporation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> into new applications. Therefore, a direct visualization and in-depth understanding of the defects and their interplay with the environment is vital for understanding the materials properties and the device breakdown under extreme conditions. In this presentation we will discuss the atomic, electronic, and chemical structure of the defects in doped and UID  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> using scanning transmission electron microscopy (S/TEM) imaging and electron energy loss spectroscopy (EELS). In addition, we will discuss the electronic structure and the local properties in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> under extreme conditions using STEM-EELS. This fundamental understanding is important to uncover the breakdown behavior in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and the impact of defects on its device performance.

11:15am **AC+MD-TuM-12 Sub-oxide Ga to Enhance Growth Rate of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> by Plasma-assisted Molecular Beam Epitaxy**, *Zhuoqun Wen*, *K. Khan*, *E. Ahmadi*, University of Michigan, Ann Arbor

In recent years, there has been significant interest in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> as a potential candidate for the next generation of power electronics, solar-blind ultraviolet (UV) detectors, and as a substrate for UV light emitting diodes (LEDs). This interest stems from its ultra-wide bandgap of 4.8eV. Thin film growth and n-type doping (Si, Sn, Ge) of Ga<sub>2</sub>O<sub>3</sub> have been achieved through various methods such as metal-organic chemical vapor deposition (MOCVD), pulsed laser deposition (PLD), and molecular beam epitaxy (MBE). However, MBE has limitations in terms of the growth rate of Ga<sub>2</sub>O<sub>3</sub> due to the desorption of volatile Ga<sub>2</sub>O, which is formed from the reaction between Ga and Ga<sub>2</sub>O<sub>3</sub>. Using gallium sub-oxide (Ga<sub>2</sub>O) instead of elemental gallium has been previously employed [1] as a technique to enhance the growth rate of Ga<sub>2</sub>O<sub>3</sub> by Ozone-MBE. However, this technique has not yet been investigated in plasma-assisted MBE. In my talk, I will present the results of our recent studies on using Ga<sub>2</sub>O as Ga source in PAMBE. Using the same plasma conditions, we show that using Ga<sub>2</sub>O instead of Ga can at least double the growth rate of Ga<sub>2</sub>O<sub>3</sub>.

Previously, we have demonstrated uniform and controllable silicon doping of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> by utilizing disilane (Si<sub>2</sub>H<sub>6</sub>) as the Si source. [2] In my talk, I will show that this technique is also compatible with utilizing Ga<sub>2</sub>O as Ga source. The silicon doping can be tuned from  $3 \times 10^{16} \text{ cm}^{-3}$  to  $1 \times 10^{19} \text{ cm}^{-3}$  using the diluted disilane source.

References:

1. Vogt, P., Hensling, F. V., Azizie, K., Chang, C. S., Turner, D., Park, J., ... & Schlom, D. G. (2021). Adsorption-controlled growth of Ga<sub>2</sub>O<sub>3</sub> by suboxide molecular-beam epitaxy. *Apl Materials*, 9(3), 031101.
2. Wen, Z., Khan, K., Zhai, X., & Ahmadi, E. (2023). Si doping of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> by disilane via hybrid plasma-assisted molecular beam epitaxy. *Applied Physics Letters*, 122(8)

11:30am **AC+MD-TuM-13 Microscopic-Scale Defect Analysis on Ga<sub>2</sub>O<sub>3</sub> through Microscopy**, *M. Kim*, NIST-Gaithersburg, Republic of Korea; *A. Winchester*, *O. Maimon*, NIST-Gaithersburg; *S. Koo*, KwangWoon University, Korea; *Q. Li*, George Mason University; *Sujitra Pookpanratana*, NIST-Gaithersburg

Crystalline defects of technologically mature materials have been identified and classified by the semiconductor industry [1,2], since it is economically beneficial to isolate failure mechanisms at the source rather than relying on

backend testing. This has significantly improved device reliability. The various defects could be categorized into killer or non-killer defects, where killer defects can hinder the operation of high-performance devices by trapping charge carriers or causing increased leakage current. Although  $\beta$ -gallium oxide ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) is expected to surpass silicon carbide (SiC), defects in Ga<sub>2</sub>O<sub>3</sub> are prevalent and largely unclassified. Therefore, screening out defects that cause electrical device degradation must be solved for widespread adoption of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>.

In this work, photoemission electron microscopy (PEEM) is used to visualize micrometer-scale defects and determine their electronic impact. PEEM is based on the photoelectric effect and is a non-destructive analysis method where light is used to excite and eject electrons from the sample surface and these electrons are analyzed. We investigated the defects on commercially-available epitaxially-grown  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> on (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates. The epitaxy was formed by hydride vapor phase epitaxy (HVPE) with a target doping of  $1 \times 10^{18} \text{ cm}^{-3}$  on the (010) semi-insulating  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> wafer. We identified elongated structures on the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> epi-layer as shown in Figure 1a, and they appear in multiple instances of the sample surface and in a parallel configuration. These features resemble the "carrot" defect observed in SiC epitaxy [3]. From the imaging spectroscopy mode of the PEEM (Figure 1b), the base and tip of the carrot were found to have similar valence band maxima but dissimilar work functions. The spectra from the tip of the carrot resembles that of the surrounding  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> epi-layer. We are performing ongoing work to identify this feature as a microscopic defect. For understanding the electrical influence of these elongated features on HVPE epi-layer, we will perform tunneling atomic force microscopy (TUNA) to measure the electrical properties on and off the defect surface. Together, we will present a discussion on the nature of these distinct features and their implication on device performance.

11:45am **AC+MD-TuM-14 Characterization and Processing Improvements for Fabricating and Polishing  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Substrates**, *Robert Lavelle*, *D. Snyder*, *W. Everson*, *D. Erdely*, *L. Lyle*, *N. Alem*, *A. Balog*, Penn State University; *N. Mahadik*, *M. Liao*, Naval Research Laboratory

As progress continues to be made in fabricating and polishing uniform, high-quality  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates, it is increasingly important to link commercial suppliers and research groups with expertise in crystal growth, substrate processing, epi growth/synthesis, characterization, and devices. This creates a vertically integrated feedback loop that drives answering fundamental research questions and increasing the manufacturability of the substrates. We will review our latest results in optimizing the chemical-mechanical polishing (CMP) methods and related processing steps for  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates and materials characterization. This includes quantifying and minimizing subsurface damage related to processing, investigating the propagation of defects such as nanopipes, fabricating off-cut/off-axis substrates, and extending the fabrication/polishing methods to different alloy compositions.

Previous results showed that an excellent surface finish (Ra < 2 Å over a >0.175 mm<sup>2</sup> area) could be achieved for Czochralski (Cz) grown  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates using a two-step CMP process with a nearly 10X reduction in polishing cycle time. After continuing to develop this process, we observed that a similar surface finish could be achieved by optimizing the pH of the colloidal silica slurry while realizing a further 3-4X reduction in cycle time. This establishes a path toward a milestone 1-day polishing process for  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates. While the surface finish is similar, further reduction in the FWHM of the x-ray rocking curves (XRRCs) was also obtained by reducing the force and optimizing the other polishing parameters during the final CMP step. These processing changes suggest improvement in polishing related subsurface damage, which we assessed using high-resolution x-ray diffraction (HRXRD) by varying the x-ray penetration depth and advanced microscopy techniques.

Uniformity continues to be an important consideration as commercial 2"+ substrates become increasingly available. We continue to map and collect characterization data from across substrates grown by Cz and edge-defined film-fed growth (EFG) and will share our observations. This includes site-specific XRRC measurements as well as etch pit density (EPD) mapping and defect analysis for full substrates. In this discussion, we will also integrate feedback from epi growers for different types of substrates. Finally, we will discuss our methodology for processing off-cut/off-axis as well as alloyed substrates and latest characterization results.

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