International Workshop on Gallium Oxide and Related Materials (IWGO-6)

Room ESJ 0202 - Session IWGO-TuM2

Substrate Development and Material Quality II

Moderators: Zbigniew Galazka, Leibniz Institute for Crystal Growth, **Kohei Sasaki**, Novel Crystal Growth

11:00am **IWGO-TuM2-37 Defect Complexes and β-to-γ Phase Boundaries in Si-implanted Ga_2O_3, *Naomi Pieczulewski,*** *Katie Gann,* Cornell University*; Thaddeus Asel, Brenton Noesges,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA*; Michael Thompson, David Muller,* Cornell University **INVITED**

Multislice electron ptychography is used to resolve the three-dimensional atomic structure of Si-implanted β-Ga_2O_3, containing both transformed β-phase and residual γ-phase, enabling direct characterization of defect structures and β-γphase boundary. We provide the first three-dimensional experimental evidence of interstitial–vacancy complexes in β-Ga_2O_3. Analysis of the β-to-γ phase transformation reveals a shared oxygen sublattice that facilitates localized transformation by a continuous transformation or a martensitic-like transition. Experimental evidence shows gradual displacement of Ga atoms can evolve into ordered interstitial defect chains forming γ-Ga_2O_3 through a static oxygen sublattice, indicating a continuous transformation. This relationship is described by a coincidence lattice, from which a structural model of the γ-phase is constructed. Additionally, we identify implant-damaged regions where we observe dislocation formation and lattice strain of the oxygen sublattice, indicating a non-continuous mechanism. By overcoming the limitations inherent to conventional scanning transmission electron microscopy, multislice electron ptychography directly resolves the β-γ crystallographic relationship at the nanoscale, providing critical experimental insight into defect-mediated phase evolution in Ga_2O_3.

11:25am **IWGO-TuM2-42 Thulium-doped Gallium Oxide Scintillators: Growth and Characterization*, Marko Tadjer,*** *Lee Mitchell,* Naval Research Laboratory*; Robert Lavelle,* Pennsylvania State University Applied Research Laboratory*; Alex Lebedinsky,* University of Houston*; Darshana Wickramaratne, Evan Glaser,* Naval Research Laboratory*; Katie Gann, Tia Gray,* National Research Council*; Alan Jacobs, James Spencer Lundh, Steven Bennett,* Naval Research Laboratory*; Matthew Krohn,* Pennsylvania State University Applied Research Laboratory*; Bernard Phlips,* Naval Research Laboratory*; Kumar Saurabh, P. Shiv Halasyamani,* University of Houston*; Karl Hobart,* Naval Research Laboratory

Versatility in melt growth capabilities for β-Ga_2O_3 provide not only for excellent material availability but also for a wide range of dopants in these crystals including shallow donors such as Si and Ge, deep donors and acceptors, and other rare earth dopants investigated for applications such as neutron detectors and scintillators [1, 2]. In this work, we report float zone (FZ) growth of rare earth thulium (Tm) doped β-Ga_2O_3. Whereas Tm-doped β-Ga_2O_3 deposition has been reported via several techniques (e.g., PLD), single-crystal growth of Tm-doped β-Ga_2O_3 has not been reported [3, 4].

The crystal growth was performed in an optical FZ furnace (Crystal Systems Co.). O_2 was flowed through the chamber at a rate of 50 mL/min at a pressure of 3.5 bar. The feed and seed rods were rotated in opposite directions at a rate of 15 rpm, and the growth rate was 5.0 mm/h. The entire crystal growth process took approximately 11 hours. FZ β-Ga_2O_3 crystal dimensions were about 11.6 mm in diameter and 57.6 mm in length, reproduced over several growths to-date with good repeatability. This method was used to grow crystals of Tm-doped Ga_2O_3 with a Tm concentration of up to 1%. Here, we show a 0.01% Tm doped crystal (0.0206g of Tm_2O_3 in 100g of Ga_2O_3). The Tm concentration of 10^{19} cm^{-3} was measured via X-ray fluorescence and further quantified via SIMS. Prior to slicing, the Tm-doped β-Ga_2O_3 boule was mounted on a three-axis goniometer and oriented for slicing into (010) samples. The orientation of the boule was corrected by modifying the goniometer axes to align with the (010) Laue reference. After final chemical-mechanical polishing (CMP), the surface topography of the substrates was evaluated via optical profilometry. The substrates were also characterized by high-resolution XRD by collecting (020) X-ray rocking curves. First-principles calculations were also performed, confirming Tm acts as a deep donor in β-Ga_2O_3. EPR spectroscopy confirmed these samples were conductive via the unintentional incorporation of Si shallow donors. The double numerical integration of the Lorentzian EPR line shapes yields a concentration of uncompensated shallow donors (i.e., N_D-N_A) of 8×10^{16} cm^{-3}. Scintillation counts were obtained from one sample using various radioactive sources. Characteristic gamma rays from low energy isotopes such as Am-241 (59.5 keV), Cd-109 (88 keV), and Co-57 (122 keV) were easily observed. Further details will be presented at the workshop.

[1] C. Prasad et al., Mater. Today Phys. **35**,101095 (2023).

[2] D. Valdes et al., APL Mater. **13**, 041116 (2025).

[3] Q. Guo et al., Thin Solid Films **639**, 123 (2017).

[4] Z. Chen et al., Appl. Phys. Expr. **14,** 081002 (2021).

11:40am **IWGO-TuM2-45 Growth of β-Ga_2O_3 Crystals by Plling-Down EFG Method with a Raw Material Supply System*, Yuki Ueda,*** Novel Crystal Technology Inc., Japan*; Kimiyoshi Koshi, Sho Hasegawa, Kohei Sasaki, Akito Kuramata,* Novel Crystal Technology, Inc., Japan

We have successfully commercialized 4-inch β-Ga_2O_3 single-crystal substrates and demonstrated the feasibility of 6-inch substrates using the edge-defined film-fed growth (EFG) method. However, increases in crystal diameter and length require larger and more expensive iridium (Ir) crucibles to accommodate the greater volume of raw materials, posing a significant cost challenge. Therefore, to reduce the total Ir required for crystal growth, we have developed a novel EFG method based on a pulling-down growth technique with continuous feeding of raw materials. In this study, we attempted to use the developed method to grow approximately 2-inch β-Ga_2O_3 crystals.

A droplet of Ga_2O_3 melt supplied onto the die passed through a slit formed in the die and subsequently reached the seed crystal.Crystal was grown at a pulling-down rate of approximately 10 mm/h in a mixed N_2/O_2 atmosphere (2 vol% O_2) with continuous feeding of raw materials. As a result, an approximately 2-inch β-Ga_2O_3 crystal was successfully obtained. The full width at half maximum (FWHM) of the x-ray rocking curve (XRC) *002* peak for a {001} sample prepared from the crystal center was 27 arcsec, comparable to that of conventional EFG crystals.On the other hand, x-ray diffraction (XRD) measurements of the {010} samples revealed that the crystallinity deteriorates as growth proceeds.This may be attributed to the temperature gradient increasing inside the furnace during growth.

11:55am **IWGO-TuM2-48 Redefining β-Ga_2O_3 Smart Cut™ Through Optimized Ion Implantation*, Adrien Roth,*** *Guillaume Gelineau, Lucas Colonel, Frédéric Mazen, Frédéric Milési, Florence Madeira, Nicolas Troutot,* CEA-Leti, France*; David Eon,* CNRS, France*; Julie Widiez,* CEA-Leti, France

Wide bandgap semiconductors are widely used for high-efficiency power electronics, but their bulk crystal growth remains costly and energy-intensive [1]. In this context, β-Ga_2O_3 has emerged as a promising alternative thanks to its ability to be grown from a liquid phase [2]. However, its very low thermal conductivity (0.15 $W.cm^{-1}.K^{-1}$) strongly limits heat dissipation, hindering the widespread adoption of Ga_2O_3-based power devices in industry [3]. To address this limitation, we investigate the transfer of (001)-oriented β-Ga_2O_3 layers, leveraging the availability of large-area (4-inch) substrates for process development, whereas prior studies mainly focused on the 2-inch (-201) orientation [4-7]. The process based on the Smart Cut™ technology, consists of implanting the bulk substrate with light ions, bonding it to the carrier substrate, and performing a fracture annealing that leads to the transfer [8].

First, we investigate the blistering process in β-Ga_2O_3, a necessary preliminary step to confirm the possibility of achieving Smart Cut™. Then, we perform fracture tests on the samples, replacing the carrier substrate with a 4µm SiO_2 deposited layer, which acts as a stiffener for the layer transfer. In this study, we compare three ion implantation conditions: He^+, H^+ and an optimized H^+ implantation process. All three types of implantations resulted in blistering of β-Ga_2O_3. However, the optimized H^+ implantation enables blistering at a lower implantation dose and annealing temperature than conventional H^+ implantation, while maintaining a higher crystalline quality than for He^+ implantation. In terms of blister morphology, H^+ implantation produces a high density of small-diameter blisters with few exfoliations and no coalescence. The other two types of implantations produce larger-diameter blisters with coalescence and exfoliations. These two ion implantation processes also allowed the fracture of a thin layer of β-Ga_2O_3 with transfer yields of 79 % of the sample surface for He^+ implantation and 95 % for optimized H^+ implantation.

This optimized condition, used in the Smart Cut™ process, enable the fabrication of the first
β-Ga_2O_3/β-Ga_2O_3 homostructure on 4-inch (001) wafers, with a 97%

transfer yield and 730nm transferred layer thickness. The presence of surface microdefects is under investigation to improve the subsequent heterostructure fabrication.

[1]Yeboah et al., Intell. Sustain. Manuf.,2025
[2]Huang et al., Eur. Phys. J. Spec. Top.,2025
[3]Kohei Sasaki, APEX 17,2024
[4]Xu et al., ACS Appl. Electron. Mater.,2022
[5]Cheng et al.,2020
[6]Shen et al., Sci. China Mater.,2023
[7]Liao et al., ECS Trans., Vol. 112,2023
[8]Widiez et al., Vol. 223,2025

12:10pm **IWGO-TuM2-51 HCl-based Halide Vapor Phase Epitaxy and HCl Gas Etching on (−112) β-Ga_2O_3 Substrates***, **Takayoshi Oshima**, Yuichi Oshima,* NIMS, Japan

We investigated the (−112) orientation—which is perpendicular and close to the (100) and (011) planes, respectively and corresponds to the fundamental {100} planes of the fcc oxygen sublattice in β-Ga_2O_3 [1]—as a novel platform for homoepitaxial growth and plasma-free gas etching.

Homoepitaxy was performed using HCl-based halide vapor phase epitaxy (HVPE). The resulting epilayer was single crystalline with tilt and twist spreads comparable to those of the substrate. Although slit-like pits sandwiched by vertical (100) facets—similar to those previously reported for epilayers grown on (011) substrates [2]—were observed on the surface, the pit-free regions exhibited a step-and-terrace morphology with a root-mean-square (RMS) roughness as small as 0.10 nm. Notably, the unintentional Cl impurity concentration in the epilayer was as low as 2×10^{15} cm^{-3}, substantially lower than 1×10^{16} cm^{-3} observed in the layer simultaneously grown on a (001) substrate. Such atomically flat epitaxial surface and low Cl impurity incorporation are highly advantageous for device applications.

Selective-area HCl gas etching was conducted using a SiO_2 mask in the same HVPE system. The resulting etched structures reflected crystal anisotropy. The side etching was minimized when the mask windows were aligned along the [02−1] direction due to the formation of (100) facets with the lowest surface energy density. Along the trench direction, the (100) sidewalls were exceptionally flat and perfectly vertical. The vertical etch rate of the (−112) plane was approximately 50 times higher than the lateral etch rate of the (100) plane, enabling high-precision patterning of fin and trench structures.

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[1] T. Oshima, Jpn. J. Appl. Phys. **65**, 038003 (2026).

[2] Y. Oshima and T. Oshima, Sci. Technol. Adv. Mater. **26**, 2585551 (2025).

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