

## Epitaxial Growth

Room Davis Hall 101 - Session EG-MoM

### Bulk/Epitaxial I

Moderator: Hongping Zhao, Ohio State University

10:45am **EG-MoM-10 Advances in the MOCVD Growth of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and Related Heterostructures**, *Andrei Osinsky*, Agnitron Technology, Inc.; *F. Alema*, Agnitron Technology, Inc. **INVITED**

$\beta$ -Ga<sub>2</sub>O<sub>3</sub> has attracted extensive interest in power electronic applications owing to its large bandgap of  $\sim 4.9$  eV, estimated high breakdown field of  $\sim 8$  MV/cm, and availability of melt grown high quality  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates. The growth of high-quality epitaxial films with low dislocation density and background impurity is critical to realize the projected device performances. Available epitaxial methods to grow  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films include MBE, HVPE, and MOCVD. But, despite coming late to the field, the MOCVD method has proven to be suitable for producing high-quality epitaxial  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films at a fast growth rate with uniform and controllable doping<sup>1</sup>. The highest purity  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films have been reported from MOCVD with record low-temperature electron mobility exceeding 23,000 cm<sup>2</sup>/Vs and low  $\sim 10^{13}$  cm<sup>-3</sup> compensating acceptors<sup>2</sup>. Also, a recent record-breaking result for lateral Ga<sub>2</sub>O<sub>3</sub> MESFETs with a lateral figure of merit (LFOM) of 355 MW/cm<sup>2</sup> and a breakdown voltage of  $\sim 2.5$  kV<sup>3</sup>, and a record low specific contact resistance  $\sim 10^{-7}$   $\Omega$ cm<sup>24</sup> were reported based on MOCVD grown epitaxial Ga<sub>2</sub>O<sub>3</sub> films.

This presentation will discuss recent progress in the growth of high-quality  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films and related materials using MOCVD. The use of Ga precursors, including triethylgallium (TEGa) and trimethylgallium (TMGa), for the growth of Ga<sub>2</sub>O<sub>3</sub> will be presented. Their advantages and disadvantages in realizing high-purity, carbon-free, epitaxial Ga<sub>2</sub>O<sub>3</sub> films will be discussed. Critical process conditions and MOCVD reactor geometries on achieving high purity  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films with high electron mobility and low background carrier concentration, including doping control in this range, will be discussed. This paper will also discuss the MOCVD growth of high Al composition (up to 30%) high quality strained  $\beta$ -(AlGa)<sub>2</sub>O<sub>3</sub>/Ga<sub>2</sub>O<sub>3</sub> heterostructures and superlattices on various orientations of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates. The MOCVD growth of heavily doped ( $>10^{20}$  1/cm<sup>3</sup>), highly conductive  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, and strained  $\beta$ -(Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub>/ $\beta$ -Ga<sub>2</sub>O<sub>3</sub> heterostructures will be presented. We will also present the demonstration of record low resistance Ohmic contacts on heavily Si doped epitaxial  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and strained  $\beta$ -(Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> epilayers with varying Al composition. A recent in-situ non-destructive etching of Ga<sub>2</sub>O<sub>3</sub> in MOCVD followed by a regrowth process will also be discussed.

[1] F. Alema *et al.*, Journal of Crystal Growth 475 (2017) 77-82.

[2] G. Seryogin *et al.*, Applied Physics Letters 117 (2020) 262101.

[3] A. Bhattacharyya *et al.*, IEEE Electron Device Letters 42 (2021) 1272-1275.

[4] F. Alema *et al.*, IEEE Electron Device Letters 43 (2022) 1649-1652.

11:15am **EG-MoM-12 MOVPE of (100)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> for Vertical Power Devices - Challenges to Epitaxial Growth Process**, *Andreas Popp*, *T. Chou*, *S. Bin Anooz*, *R. Grüneberg*, *V. Thuy*, *J. Rehm*, *A. Akhtar*, *Z. Galazka*, *P. Seyidov*, *K. Irmischer*, LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e.V, Germany; *M. Albrecht*, LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e., Germany; *A. Fiedler*, LEIBNIZ-INSTITUT FÜR KRISTALLZÜCHTUNG im Forschungsverbund Berlin e.V, Germany

Beta-gallium oxide ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) is a promising ultra-wide bandgap ( $\sim 4.8$  eV) semiconductor material for the application field of power electronic converters. The theoretical breakdown field strength of up to 8 MV/cm<sup>[1]</sup> can be best exploited using a vertical architecture for  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> based transistors. A high-quality homoepitaxial drift layer plays a crucial role in such a vertical device structure. Here, we report our developed process via metal-organic vapor phase epitaxy (MOVPE) to overcome the main issue associated with the homoepitaxial drift layer: (a) low doping ( $10^{16}$  cm<sup>-3</sup> range) concentrations, (b) layer thicknesses of several  $\mu$ m while maintaining a low density of structural and point defects and (c) high growth rates ( $\mu$ m/h).

A high growth rate process of up to 1.5  $\mu$ m/h was achieved for Si-doped (100)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> homoepitaxial films grown via MOVPE on Czochralski-grown semi-insulating<sup>[2,3]</sup> and conductive<sup>[4]</sup> (100) 4° off  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrates while maintaining the step-flow growth mode up to a film thickness of 4  $\mu$ m<sup>[5]</sup>.

The enhanced diffusion channel due to forming a Ga adlayer was proposed as the possible growth mechanism<sup>[6]</sup>. Furthermore, we also report the formation of parasitic particles as a killer issue during the growth, which can be suppressed by a close showerhead to substrate gap and a high total gas flow<sup>[7]</sup>. With our optimized process, Si doping enabled precise control of the n-type conductivity of the layers with free electron concentrations ranging from  $5 \times 10^{16}$  cm<sup>-3</sup> to  $1.5 \times 10^{19}$  cm<sup>-3</sup> and corresponding mobilities from 163 cm<sup>2</sup>·V<sup>-1</sup>·s<sup>-1</sup> to 21 cm<sup>2</sup>·V<sup>-1</sup>·s<sup>-1</sup> were measured by room temperature Hall measurements. Temperature-dependent Hall measurements let expect a low background compensating acceptor concentration of  $4 \times 10^{15}$  cm<sup>-3</sup>, indicating a doping level down to mid  $10^{15}$  cm<sup>-3</sup> is still possible.

[1] M. Higashiwaki *et al.*, *Semicond. Sci. Technol.* **2016**, *31*, 34001.

[2] Z. Galazka *et al.*, *Prog. Cryst. Growth Charact. Mater.* **2021**, *67*, 100511.

[3] P. Seyidov *et al.*, *APL Materials* **2022**, *10*, 111109 (2022)

[4] Z. Galazka *et al.*, *Appl. Phys. Lett.* **2022**, *120*, 152101.

[5] T.-S. Chou *et al.*, *Jpn. J. Appl. Phys.* **2023**, *62*, SF1004.

[6] T.-S. Chou *et al.*, *AIP Adv.* **2021**, *11*, 115323.

[7] T.-S. Chou *et al.*, *Appl. Phys. Lett.* **2023**, *122*, 052102.

11:30am **EG-MoM-13 MOCVD Epitaxy of (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> with Fast Growth Rate and the Role of Carbon in Charge Compensation**, *Lingyu Meng*, *A. Bhuiyan*, *D. Yu*, *H. Zhao*, The Ohio State University

$\beta$ -Ga<sub>2</sub>O<sub>3</sub> has emerged as a promising semiconductor candidate for future power electronic and radio frequency device applications, because of its ultra-wide bandgap (4.8 eV) and high critical field strength (8 MV/cm). Previously, metalorganic chemical vapor deposition (MOCVD) of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> using triethylgallium (TEGa) as the Ga precursor has been demonstrated with record electron mobilities approaching the theoretical values. The use of trimethylgallium (TMGa) as the Ga precursor with higher vapor pressure enables the MOCVD growth of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> with much faster growth rates ( $> 3 \mu$ m/hr) for thick film growth.

In this work, a systematic study of the MOCVD growth of (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> using TMGa was performed. Both TMGa molar flow rate and growth temperature play an important role on the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> MOCVD growth rate. With a TMGa molar flow rate of 116  $\mu$ mol/hr,  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> fast growth rate of 6.7  $\mu$ m/hr was achieved at 950°C. The surface morphologies of the MOCVD  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films were highly dependent on the growth conditions, particularly chamber pressure. For films grown at a growth rate of  $\sim 3 \mu$ m/hr, record room temperature electron Hall mobilities ranging between 190 cm<sup>2</sup>/Vs and 93 cm<sup>2</sup>/Vs were achieved with carrier concentrations between  $1.6 \times 10^{16}$  and  $3.8 \times 10^{19}$  cm<sup>-3</sup>. Temperature-dependent charge transport characteristics revealed a low charge compensation level ( $\sim 1.5 \times 10^{16}$  cm<sup>-3</sup>) and a decent low-temperature peak electron mobility (3425 cm<sup>2</sup>/Vs at 53K).

The impurities incorporation in the MOCVD grown (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films with different growth rates was probed by the quantitative secondary-ion mass spectroscopy (SIMS). SIMS results revealed that both [C] and [H] increase as the TMGa molar flow rate/growth rate increases. However, [C] increases at a much faster rate as compared to [H]. By comparing the electron Hall measurement results and the quantitative SIMS characterization, the net impurity concentration ([C]-[H]) matches well with the compensation level in the MOCVD  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> grown at the different growth rates. Therefore, the incorporated H forms neutral C-H complexes and thus passivates the compensation effect from pure C. This mechanism has been proposed from theoretical study based on the DFT calculations.

In summary, MOCVD growth of (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> using TMGa as the Ga precursor was systematically studied. The role of C compensation and passivation from C-H complexes in the MOCVD grown films were proposed based on the experimental evidence.

**Acknowledgment:** The authors acknowledge the funding support from AFOSR (FA9550-18-1-0479) and the NSF (No. 2231026, No. 2019753).

11:45am **EG-MoM-14 Controllable Deep Acceptor Doping in MOCVD  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> to Compensate Parasitic Interface Charges**, *Fikadu Alema*, Agnitron Technology; *T. Itoh*, Materials Department, University of California, Santa Barbara; *W. Brand*, *A. Osinsky*, Agnitron Technology; *J. Speck*, Materials Department, University of California, Santa Barbara

One of the challenges in developing  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> lateral devices is the presence of parasitic charges at the epilayer-substrate interface due to the Si impurity that accumulates at the interface. One method that has been proposed recently to manage the interface Si is to etch the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrate in hydrofluoric acid (HF) for an extended time before the growth of the films [1]. This method does not eliminate the Si at the interface but

# Monday Morning, August 14, 2023

reduces its concentration just below the concentration of Fe in the substrate, leading to partial compensation. The other method to manage the interface Si is to compensate with deep acceptor dopants, such Mg, Fe, and N, among which N is less affected by thermal diffusion, making it a dopant of choice [2].

In this work, we report on controllable doping of N into Ga<sub>2</sub>O<sub>3</sub> using nitrous oxide (N<sub>2</sub>O) and ammonia diluted in nitrogen (NH<sub>3</sub>/N<sub>2</sub>) as sources for N. The incorporation efficiency, reproducibility, and controllability of N doping into Ga<sub>2</sub>O<sub>3</sub> films will be discussed as a function of process conditions for both sources. Incorporating N into Ga<sub>2</sub>O<sub>3</sub> is found to be sensitive to process conditions when N<sub>2</sub>O is used as a source. A maximum N concentration of  $\sim 2 \times 10^{19}$  cm<sup>-3</sup> was achieved by growing the layer at a low substrate temperature. However, using NH<sub>3</sub>/N<sub>2</sub>, the doping of N into Ga<sub>2</sub>O<sub>3</sub> is controllable through the molar flow rate. With the increase in the NH<sub>3</sub>/N<sub>2</sub> molar flow rate from  $\sim 1.8 \times 10^{-8}$  mol/min to  $1.45 \times 10^{-6}$  mol/min, the N impurities incorporated into the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> layers increased from  $\sim 1 \times 10^{18}$  cm<sup>-3</sup> to  $\sim 2 \times 10^{20}$  cm<sup>-3</sup>. For both sources, hydrogen was found to incorporate into the films along with N, but annealing at reduced pressure helps to drive out the hydrogen from the film. For the NH<sub>3</sub>/N<sub>2</sub> source, growing the layers at elevated temperatures (>900 C) also effectively reduced H incorporation into the film by as much as  $\sim 10\times$  with no effect on the incorporation of N. This paper will also discuss the impact of exposing the surface of Ga<sub>2</sub>O<sub>3</sub> substrate to NH<sub>3</sub>/N<sub>2</sub> and halide based precursors to manage the interface Si.

[1] A. Bhattacharyya et al. C APL Materials 11, 021110 (2023).

[2] M.H. Wong et al. Applied Physics Letters 113, 102103 (2018).

12:00pm **EG-MoM-15 Si Accumulation on Ga<sub>2</sub>O<sub>3</sub> Surfaces, Jon McCandless, C. Gorsak, V. Protasenko, D. Schlom, M. Thompson, H. Xing, H. Nair, D. Jena, Cornell University**

In 2022, we demonstrated a high degree of doping control ( $1 \times 10^{17}$  to  $1 \times 10^{20}$ /cm<sup>3</sup>) in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films grown by molecular beam epitaxy with mobilities of  $\sim 130$  cm<sup>2</sup>/Vs in the lowest doped samples.<sup>1</sup> However, we discovered some samples where, despite being grown under the same conditions, the mobility would be  $\sim 1$  cm<sup>2</sup>/Vs. To understand a potential impurity related origin of these low mobilities, secondary ion mass spectrometry (SIMS) was performed. Over 8 samples, we observed significant Fe variation within the substrate and significant Si variation at the surface. Depending on the substrate, the surface Si may or may not have been fully compensated by the Fe. In the worst case, a high-density of uncompensated free carriers exist at the interface with a low mobility which in turn affects the Hall effect measurement. Moreover, this same uncompensated charge can prevent confinement, or create a parallel conducting path for 2D electron gasses, thereby hindering the performance of high electron mobility transistors.<sup>2</sup>

The Si contamination is thought to arise from the polishing process and/or from siloxanes adsorbed from the air, and which are particularly difficult to remove.<sup>3</sup>

To quantify and study the Si accumulation and possible removal strategies, we investigated how Si accumulates on the film surface when exposed to air. We grew UID layers by molecular beam epitaxy, removed the sample and exposed it to air for different amounts of time. After less than 20 minutes of exposure to air, the accumulated Si on a clean surface had a sheet density ( $n_s$ ) of  $\sim 2 \times 10^{12}$ /cm<sup>2</sup>. The  $n_s$  continued to increase with longer exposure times in air, saturating at  $\sim 7 \times 10^{12}$ /cm<sup>2</sup> after an 8-hour exposure. Next, etching studies were performed to investigate possible removal of the SiO<sub>x</sub> on the surface.  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films were grown by metal-organic chemical vapor deposition and the surface was exposed to air for 2 hours to allow for the accumulation of Si. The surface was then etched in HF (49%) for varying times to remove the accumulated Si. After etching for 15 minutes, the Si sheet charge was reduced by  $\sim 1$  order of magnitude to  $\sim 3 \times 10^{11}$ /cm<sup>2</sup>.

Lastly, simulations were carried out to investigate compensation doping. Under the worst case, (i.e. low Fe density within the substrate and high Si density at the interface), the required compensation would have to be  $> 10^{20}$ /cm<sup>3</sup>, but at this Fe density the film quality degrades. Therefore, we believe the optimal solution requires HF etching along with compensation doping.

1. APL, 121, 072108 (2022); 2. APEX, 10, 071101 (2017); 3. Chemosphere, 92, 905-910 (2013)

## Author Index

### Bold page numbers indicate presenter

— A —

Akhtar, A.: EG-MoM-12, 1

Albrecht, M.: EG-MoM-12, 1

Alema, F.: EG-MoM-10, 1; EG-MoM-14, 1

— B —

Bhuiyan, A.: EG-MoM-13, 1

Bin Anooz, S.: EG-MoM-12, 1

Brand, W.: EG-MoM-14, 1

— C —

Chou, T.: EG-MoM-12, 1

— F —

Fiedler, A.: EG-MoM-12, 1

— G —

Galazka, Z.: EG-MoM-12, 1

Gorsak, C.: EG-MoM-15, 2

Grüneberg, R.: EG-MoM-12, 1

— I —

Irmscher, K.: EG-MoM-12, 1

Itoh, T.: EG-MoM-14, 1

— J —

Jena, D.: EG-MoM-15, 2

— M —

McCandless, J.: EG-MoM-15, 2

Meng, L.: EG-MoM-13, 1

— N —

Nair, H.: EG-MoM-15, 2

— O —

Osinsky, A.: EG-MoM-10, 1; EG-MoM-14, 1

— P —

Popp, A.: EG-MoM-12, 1

Protasenko, V.: EG-MoM-15, 2

— R —

Rehm, J.: EG-MoM-12, 1

— S —

Schlom, D.: EG-MoM-15, 2

Seyidov, P.: EG-MoM-12, 1

Speck, J.: EG-MoM-14, 1

— T —

Thompson, M.: EG-MoM-15, 2

Thuy, V.: EG-MoM-12, 1

— X —

Xing, H.: EG-MoM-15, 2

— Y —

Yu, D.: EG-MoM-13, 1

— Z —

Zhao, H.: EG-MoM-13, 1