

## Bulk Growth

### Room Bansal Atrium - Session BG-MoP

#### Bulk Growth Poster Session I

**BG-MoP-1 MOCVD Development for Growth of Ga<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub>) 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<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub> MOCVD growth tools as well as adapted several pre-existing (for different materials) tools to grow Ga<sub>2</sub>O<sub>3</sub> and operated in-house tools for growth on single (cm<sup>2</sup> 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<sub>2</sub>, N<sub>2</sub>O, H<sub>2</sub>O, TEGa, TMGa, TMIIn, TMAI, TEAL, GaCl<sub>3</sub>), process enhancers such as HCl, and dopants (SiH<sub>4</sub>, 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 β-Ga<sub>2</sub>O<sub>3</sub> 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), β-Ga<sub>2</sub>O<sub>3</sub> is a very promising materials for various applications such as UV photodetectors, power rectifiers, gas sensors, MOSFETs, SBDs, and LEDs because β-Ga<sub>2</sub>O<sub>3</sub> has a wide bandgap of 4.9eV and a high breakdown voltage of 8MV/cm. Furthermore, since β-Ga<sub>2</sub>O<sub>3</sub> 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 β-Ga<sub>2</sub>O<sub>3</sub> crystal was systematically investigated with controlling temperature gradient in growth cell of edge-defined film-fed growth (EFG) method. Flat-shaped β-Ga<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub> 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 β-Ga<sub>2</sub>O<sub>3</sub> crystal ribbons grown with changing the temperature gradient were systematically analyzed. A

phase analysis of β-Ga<sub>2</sub>O<sub>3</sub> 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) β-Ga<sub>2</sub>O<sub>3</sub> and Sn-doped β-Ga<sub>2</sub>O<sub>3</sub> crystals were analyzed using the Hall effect measurement system and the secondary ion mass spectrometry (SIMS), respectively.

High-quality Sn-doped (001) β-Ga<sub>2</sub>O<sub>3</sub> crystal with a full width at half maximum (FWHM) of 38 arcsec and an etch pit density (EPD) of 3700/cm<sup>2</sup> was obtained with optimizing temperature gradient. Proper temperature gradient resulted in the reduction of SnO<sub>2</sub> volatilization during the growth process and the uniform distribution of bluish color throughout the entire ribbon. The carrier concentration of ≥10<sup>18</sup>/cm<sup>3</sup> 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 β-Ga<sub>2</sub>O<sub>3</sub> 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**

β-Ga<sub>2</sub>O<sub>3</sub> 4.9eV [1]. 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 β-Ga<sub>2</sub>O<sub>3</sub> 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 β-Ga<sub>2</sub>O<sub>3</sub> 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 β-Ga<sub>2</sub>O<sub>3</sub> crystal substrate. Raman spectra showed successful growth of β-Ga<sub>2</sub>O<sub>3</sub> 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<sub>3</sub>PO<sub>4</sub> solution.

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

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- [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)

# Monday Evening, August 14, 2023

**BG-MoP-5 Investigation of Defects in(100) and (001)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>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<sub>2</sub>O<sub>3</sub>) 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  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> 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)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> single crystals grown by edge-defined film-fed growth (EFG).

In experiment, the etch pits of (100) and (001)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> single crystals were formed by wet etching with 85 wt% phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) solution at 140 °C for 120 min. The profile of etched surfaces was observed using atomic force microscope. In addition, (001)-oriented  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> 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  $\sim 3.8 \times 10^5 \text{ cm}^{-2}$  and  $\sim 6.0 \times 10^4 \text{ cm}^{-2}$ , respectively. Figure 2 shows the XRT image of (001)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> with a diffraction vector,  $\mathbf{g}=605$ . 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  $\langle 001 \rangle$  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<sub>2</sub>O<sub>3</sub> substrates as we perform further in-depth characterization in the future.

## References

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