HVPE-Based Gallium Oxide Epiwafer Development

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Abstract

The emergence of large-area high quality β-Ga_2O_3 substrates in recent years has resulted in a plethora of interest and research in this emerging new ultra-wide bandgap semiconductor. From the perspective of the size of available wafers and the ability to produce high quality epitaxy, the (001) orientation is becoming the preferred orientation for vertical architecture power devices. Halide vapor phase epitaxy (HVPE) is a proven technique to produce homoepitaxial films of (001) β-Ga_2O_3, but the manufacturability of thick epilayers at low cost remains challenging due to the growth rates for the highest quality epilayers remaining <10µm/hr. In this work, we demonstrate thick, high quality, uniform epilayers grown by HVPE with growth rates >14µm/hr. The vapor phase pre-reactions were eliminated through careful control of the growth conditions, and high growth rates were demonstrated which could be applied to high volumes of (001) β-Ga_2O_3 films suitable for high voltage power electronics applications.

INTRODUCTION

Monoclinic β-Ga_2O_3 has recently emerged as a promising material for "ultrawide" bandgap electronics for next-generation high voltage lateral and vertical power switching devices, thanks to its large bandgap (4.9eV), high expected critical breakdown field (8MV/cm), and its accordingly large high voltage and high frequency Baliga figures of merit [1]. In a matter of only a few years, many reports of high-performance unipolar devices have been made, including high voltage Schottky barrier diodes, MESFETs, MOSFETs and HEMTs [2].

Development of epitaxial growth techniques which support the production of high quality and thick homoepitaxial films of Ga_2O_3 for power devices with vertical architectures are still emerging, but the HVPE technique, which is a chemically pure and high growth rate technology has been identified as a potentially suitable candidate [2,3], and small volumes of commercially available HVPE epilayers are now available.

In vertical devices, killer defects which give rise to unacceptably high leakage currents in e.g. Schottky barrier diodes have been identified for Ga_2O_3 devices on (010) [4], (-201) [5], and (001) [6] orientations, all of which are thought to arise from the same type of substrate-associated nano-void defect. For substrates grown by the edge-defined film-fed growth (EFG) technique, these defects have densities on the order of 10^3-10^4 cm^{-2}.

Additionally, microparticles which can form in the vapor phase under non-optimized growth conditions have been shown to promote sequences of stacking faults to form in epitaxial layers which provide an additional leakage pathway in SBDs grown on (001) substrates [7]. These defects can be highlighted using various alkaline treatments after epitaxy is complete, but are not generally observable in traditional X-ray rocking curve analyses. Nevertheless, the mitigation of both types of defects will be required to achieve the ultimate performance in Ga_2O_3 power devices. The formation of these defects is a particular challenge for HVPE films which are grown at high growth rates, which somewhat weakens the economic arguments to utilize HVPE over e.g. MBE or MOCVD for thick film growth in the first place. In this work, we demonstrate an optimized HVPE growth scheme which both eliminates the production of microparticles while simultaneously allowing growth rates of ~14µm/hr.

EXPERIMENT

Homoepitaxial β-Ga2O3 films were grown on commercially available (001) substrates in a 4"-capable, custom-designed Kyma hydride vapor phase epitaxy (HVPE) deposition system. GaCl was generated using a combination of Cl_2/H_2 with liquid gallium and dry air was used as the O_2 source. N_2 was used as the carrier gas, the temperature of the growth zone was 950°C, and the growth pressures were varied from 50 to 100torr. After growth, thicknesses were estimated both by measuring the change in weight of the wafers, as well as by Fourier-transform infrared spectroscopy (FTIR). Structural quality was assessed by evaluating the full-width at half-maximum of the (004) reflection using a high-resolution X-Ray diffractometer (X-Pert Pro). Evaluation of the density of stacking faults was evaluated by etching the epitaxial layers in a KOH batch (11.7M) for 30minutes at 100°C and evaluating the etched surface using an optical microscope.

RESULTS

Initial growth development focused on demonstrations of growing ~5µm thick films of Ga_2O_3 on (001) substrates without appreciably increasing the XRD linewidths of the subsequent epilayers' (004) reflections. This primarily

consisted of changing the molar fractions of GaCl and O_2 through changes of the Cl, H_2, and O_2 flows into the reactor. Films with thicknesses of ~5µm exhibited XRD linewidths <30 arcsec (equivalent to the starting substrates) over a range of the GaCl:O_2 flows (having ratios of ~2.5 to 5) and temperatures (from 950°C to 1050°C), while thicker films (~15µm and above) required the O_2 flow to be reduced. Thus, thick films apparently have a narrower the growth window for realizing high structural quality films.

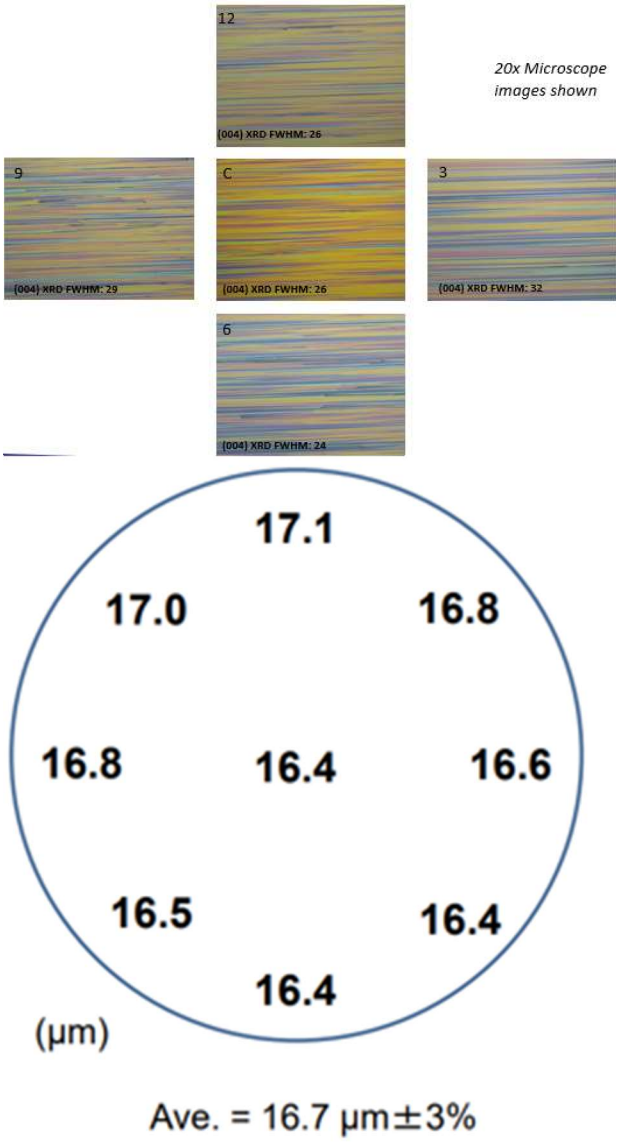

Fig. 1. (Above) Surface morphologies in several regions along with XRD linewidths (insets) over a thick Ga_2O_3 layer grown on a 2" (001) wafer. (Below) Thickness mapping over the same 2" epiwafer.

Fig. 1 shows surface morphologies after growth in five locations across a 2" wafer, along with the XRD linewidths for the (004) reflections in those same locations, and a thickness map by FTIR of the same film grown under a GaCl:O_2 ratio of 2.66. The growth rate was 4.8µm/hr for this film. The surface morphology over this whole 2" wafer appears to be uniform and smooth, and the thickness uniformity is better than 5% over the whole wafer. Evaluation of the density of killer stacking fault defects was performed on another wafer which was grown under identical conditions, but with a thickness of only ~5µm. EPD was qualitatively assessed by evaluating the density of pit features on the wafer surface after subjecting the film to 30 minutes of hot (100°C) KOH. Results are shown in Fig. 2 (top row). The EPD appears to be significantly higher than that of the estimated density of defects in the starting substrate (expected to be in the high 10^3 cm^{-2} range). This suggests that the defects' origin may be from the vapor phase pre-reactions as discussed previously. In order to reduce the propensity for the pre-reaction to occur, the gas velocity in the reactor was increased by reducing the growth pressure from 100 torr to 80 torr and to 65 torr. This reduction in growth pressure also reduced the growth rates from 4.7µm/hr (100 torr), to 4.0µm/hr (80 torr), to 3.1µm/hr (65 torr). Films were grown to thicknesses of ~5µm under each of these conditions on small wafer piece, and images of the as-grown surfaces as well as the surfaces after KOH etching are given in Fig. 2. The EPD reduced to densitie near the starting substrate through this reduction in pressure.

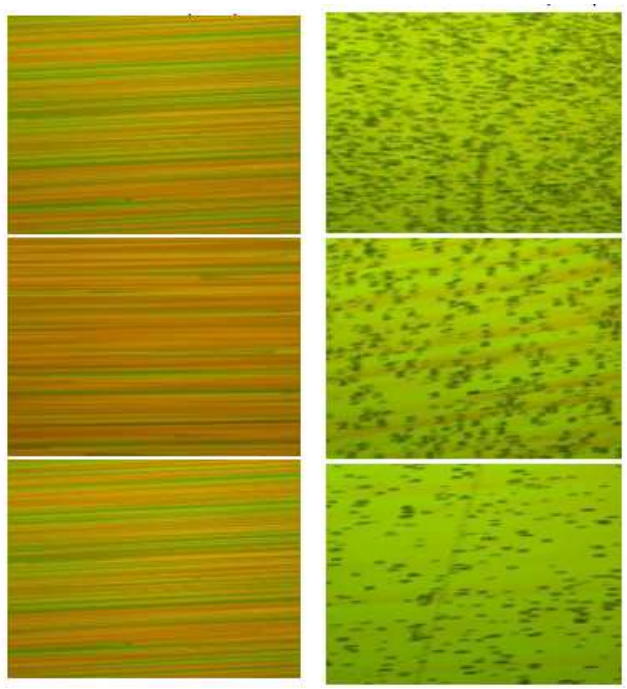

Fig. 2. (Left) 20x Nomarski microscope images after growth of ~5µm thick epilayers grown at 100torr, 80torr, and 65torr (top to bottom). (Right) 10x images after KOH etching of the same three layers.

Next, this growth condition was applied for a longer growth time on a 2" (001) in order to assess the growth uniformity and evaluate the EPD over an entire 2" wafer area for a ~15µm thick film. Results are given in Fig. 3.

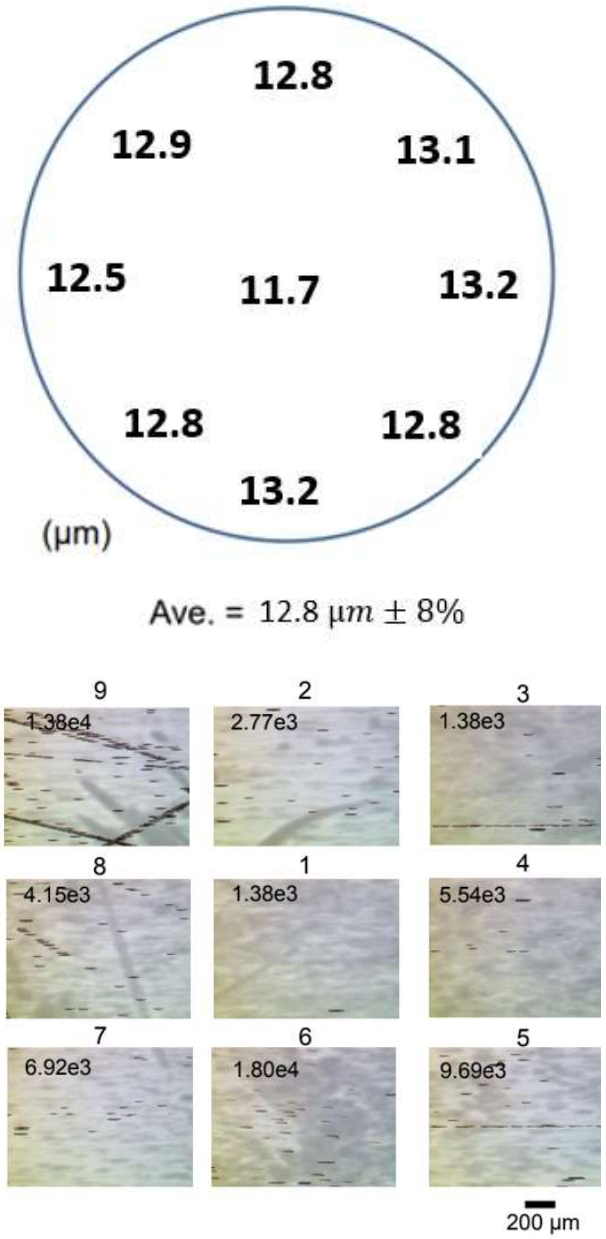

Fig. 3. (Above) Thickness mapping using the optimized growth recipe over a 2" wafer at a target growth thickness of ~15µm. (Below) Etch pit analysis showing that the EPD after KOH etching for this epilayer is similar to that of the starting substrate.

FTIR analysis of the epilayer thickness suggested that the growth rate for this film was only 1.8µm/hr. The lower-than-anticipated growth rate was likely either due to discrepancies between FTIR and weight-based thickness analyses (the test runs described in Fig. 2 were evaluated by weight-based thickness) or differences stemming from loading effects in the reactor when using a whole 2" wafer vs. a small test piece. In the images shown after etching (bottom of Fig. 3), position '1' is the wafer center, '2' is the 12 o'clock position, and subsequent images (3, 4, 5..) are clockwise from12 o-clock. From these images, the average EPD was 7.1 x 10^3 cm^{-3} which is in line with the estimated defect density associated with the substrate itself.

Finally, in an effort to increase the growth rates under conditions which maintain low defect densities and associated low EPDs, the growth conditions were optimized using a combination of pressure and variations to the Cl_2, H_2, and O_2 flows to increase the growth rates to values more in-line with those required for a production deposition system. EPDs were evaluated in all layers and Nomarski microscope images of the final result of the optimization process are given in Fig. 4. The final growth rate realized was 14.1µm/hr, which exhibited an XRD linewidth of the (004) reflection of 26 arcsec, a very smooth surface morphology, and exhibited very few discernable etch pits after being subjected to the 30min KOH etch process. This represents an optimal growth condition to realize rapid production of thick epilayers on (001) substrates. Further work to evaluate the ability to controllably dope such layers, as well as demonstrations of the optimized growth process on 100mm substrates are currently underway and will be reported elsewhere.

Fig. 4. (Left) 20x Nomarski microscope images after growth of ~15µm thick epilayer under optimized conditions. (Right) 10x images after KOH etching of the same layers.

CONCLUSIONS

In this work, we demonstrated the ability to grow high quality ~15µm thick Ga_2O_3 epilayers on 2" (001) substrates using a high growth rate HVPE process with a minimal density of defects associated with vapor-phase pre-reactions. Such a demonstration is crucial to the eventual realization of high volume Ga_2O_3 epiwafer manufacturing.

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ACRONYMS

EPD: Etch Pit Density
FTIR: Fourier-Transform Infrared Spectroscopy
HVPE: Halide Vapor Phase Epitaxy
KOH: Potassium Hydroxide
XRD: X-ray Diffraction