

Program Overview

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Material and Device Processing and Fabrication Techniques

Room Jefferson 1 & Atrium - Session MD-TuP

Material and Device Processing and Fabrication Techniques Poster Session

MD-TuP-1 Record Low Specific Resistance Ohmic Contacts to Highly Doped MOVPE-Grown β -Ga₂O₃ and β -(Al_xGa_{1-x})₂O₃ Epitaxial Films, *Carl Peterson*, University of California Santa Barbara; *F. Alema*, Agnitron Technology; *S. Roy*, University of California Santa Barbara; *A. Bhattacharyya*, University of Utah; *A. Osinsky*, Agnitron Technology; *S. Krishnamoorthy*, University of California Santa Barbara

We report on the growth and characterization of low resistance ohmic contacts on highly Silicon-doped beta-gallium oxide (β -Ga₂O₃) and pseudomorphic beta aluminum gallium oxide (β -(Al_xGa_{1-x})₂O₃) epilayers using metalorganic vapor phase epitaxy (MOVPE). The epitaxial structure consists of Si-doped β -Ga₂O₃ and Si-doped β -(Al_xGa_{1-x})₂O₃ epilayers grown on Fe-doped (010) bulk substrates. MOVPE growth was done using Agnitron Technology's Agilis 100 MOVPE reactor with TEGa, O₂, silane (SiH₄), and TEAL as precursors with argon used as the carrier gas. The electrical properties of the Si-doped epilayers are characterized by hall measurements and transfer length measurements (TLM). A 20nm/150nm/50nm Ti/Au/Ni metal stack was deposited via e-beam evaporation and was annealed at 470 °C for 1min. TLM structures were mesa isolated using BCl₃ chemistry-based reactive ion etching process. Four probe current-voltage (I-V) measurements were performed on the TLM structures to obtain the specific contact resistance (ρ_c) of the ohmic contact. Highly linear I-V characteristics were measured on both β -Ga₂O₃ and β -(Al_xGa_{1-x})₂O₃. The lowest specific resistance ohmic contact was measured on heavily doped β -Ga₂O₃ with a hall electron concentration of 3.23×10^{20} cm⁻³. Record low specific contact resistance, as low as 2.3×10^{-7} $\Omega \cdot \text{cm}^2$, was measured. This sample had a low sheet resistance of 29.8 Ω/\square which was measured via TLM. The sheet resistances measured through TLM matched with those obtained by hall measurements. For the heavily Si-doped aluminum gallium oxide samples, the lowest specific resistance ohmic contact was measured on a sample with a hall electron concentration of 1.23×10^{20} cm⁻³ and an aluminum composition of 12.3%. Record low specific contact resistance to β -(Al_xGa_{1-x})₂O₃, as low as 3.9×10^{-6} $\Omega \cdot \text{cm}^2$, was measured on this sample. The low specific resistance also corresponded with the lowest Al composition. Increasing the aluminum composition to 21.6% increased the specific resistance to 4.6×10^{-4} $\Omega \cdot \text{cm}^2$. This 21.6% Al sample had an electron concentration of 1.23×10^{20} cm⁻³. Increasing the aluminum composition in β -(Al_xGa_{1-x})₂O₃ also decreased the effectiveness of annealing the Ti/Au/Ni ohmic contacts. Annealing these high aluminum composition samples did not significantly change specific contact resistance and in some cases increased the value of ρ_c . Successful epitaxial growth of highly Si-doped β -Ga₂O₃ and β -(Al_xGa_{1-x})₂O₃ films with very low specific contact resistance can facilitate high-performance field-effect transistors.

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MD-TuP-3 MOCVD β -Ga₂O₃ Gate-recessed MESFET, *Hannah Masten, J. Lundh, J. Spencer*, US Naval Research Laboratory; *F. Alema, A. Osinsky*, Agnitron Technology; *A. Jacobs, K. Hobart, M. Tadjer*, US Naval Research Laboratory

Metalorganic chemical vapor deposition (MOCVD) has shown great potential in growing high-quality epilayer β -Ga₂O₃ films with high growth rates, which will lead to lower cost power electronics [1], [2]. Here, we demonstrate gate-recessed MOCVD metal-semiconductor field-effect transistors (MESFETs) on a 2-inch domestically grown β -Ga₂O₃ substrate.

A 2-inch (010) β -Ga₂O₃ substrate (NG Synoptics) was used for epitaxial growth. The following homoepitaxial structure was grown via Agilis 500 close-injection showerhead MOCVD (CIS-MOCVD) process at Agnitron Technology: about 1000 nm thick unintentionally doped Ga₂O₃ buffer layer, followed by a 30 nm thick n-type Ga₂O₃ layer ($\sim 10^{18}$ cm⁻³), and a 10 nm

thick n⁺ Ga₂O₃ layer for improved channel conductivity and reduced contact resistance. Room temperature Hall measurements indicated a sheet resistance of $R_{SH} = 7385$ $\Omega/\text{sq.}$, sheet carrier concentration n_s of 8.68×10^{12} cm⁻², with mobility of 97 cm²/V·s. Mesa isolation was performed via BCl₃ plasma dry etch process (800 W ICP, 60 W RIE, 5 mT, ~ 30 nm/min). Ohmic contacts were formed via Si ion implantation (3×10^{19} cm⁻³ dose, 100 nm box profile) with an activation anneal of 925 °C for 30 minutes in N₂ atmosphere, followed by lift-off of a 20/200 nm thick Ti/Au metal stack annealed at 475 °C for 1 minute in N₂. This process resulted in a specific contact resistivity of 1.6×10^{-4} Ω/cm^2 . An ~ 15 nm gate recessed was formed via Cl₂ plasma dry etch process (100 W ICP, 150 W RIE, 5 mT, ~ 15 nm/min). Gate contacts were then formed by lift-off of a 20/200 nm thick Pt/Au metal stack. DC I-V measurements were performed on the MESFETs with and without the gate recess etch. Without the gate recess, the MESFETs required a large negative gate bias ($V_{GS} = -40$ V) to turn off the device due to the highly conductive 10 nm channel. With the 15 nm gate recess, the MESFET is successfully able to turn off at V_{GS} of -10 V. Optimization of the gate recess etch and plasma damage recovery can lead to high-performing, enhancement-mode β -Ga₂O₃ power devices.

[1] S. J. Pearton et al., Appl. Phys. Rev., vol. 5, no. 1, p. 011301, Mar. 2018, doi: 10.1063/1.5006941. [2] M. J. Tadjer et al., J. Phys. D: Appl. Phys., vol. 54, no. 3, p. 034005, Oct. 2020, doi: 10.1088/1361-6463/abbc96.

MD-TuP-4 Subsurface Damage Analysis of Chemical Mechanical Polished (010) β -Ga₂O₃ Substrates, *Michael Liao, K. Huynh, L. Matto, D. Luccioni, M. Goorsky*, UCLA

The impact of pressure and abrasives on the subsurface damage for chemical mechanical polished (010) β -Ga₂O₃ substrates was assessed. A combination of 1 kPa of applied pressure and colloidal silica was found to be necessary for achieving both smooth surfaces (< 0.5 nm rms) and subsurface-damage-free material. The as-received grounded surfaces of (010) Tamura substrates were lapped and chemical mechanical polished to a damage-free state. Symmetric (020) triple-axis X-ray diffraction rocking curves were employed to assess the subsurface lattice damage by measuring diffuse scatter intensity (i.e. lattice damage from lapping/polishing) quantified by rocking curve widths below the full width at half maximum (FWHM where $X < 0.5$) [1,2]. The rocking curve FWHM and FW(0.001) θ for the as-received ground surface were $\sim 180''$ and $\sim 8300''$, respectively. These broad widths correspond to cracks, voids, and dislocations induced by the grinding process. The substrates were lapped with 5 μm alumina particles, then lapped with 0.3 μm alumina particles in water. Colloidal alumina in NaOCl was then used to polish, which was followed by colloidal silica in NaOH. A final cleaning step used abrasive-free diluted bleach and citric acid to remove residual silica on from the surface [3]. Compared to our previous work that used diluted bleach and citric acid to polish various III-V materials [1,2], we show this chemistry combination is inert for β -Ga₂O₃ but is effective in cleaning off colloidal silica particles and other forms of silicon on the substrate surface. The final rocking curve FWHM and FW(0.001) θ were $\sim 16''$ and $\sim 150''$, respectively after using the colloidal silica, which matches the widths of the as-polished commercial wafers. Material removal rates were measured for each lapping and polishing steps: 5 μm alumina particles yielded a removal rate of ~ 20 $\mu\text{m/hr}$, and colloidal silica yielded the slowest ~ 0.4 $\mu\text{m/hr}$. The depths of the subsurface damage induced by: lapping with 5 μm alumina particles was ~ 20 μm and lapping with 0.3 μm alumina was ~ 6 μm . Determining both the removal rates and depth of subsurface damage is crucial in optimizing lapping and polishing recipes.

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References

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MD-TuP-5 Diffusion of Zn in β -Ga₂O₃, *Ylva Knausgård Hommedal, Y. Frodason, L. Vines, K. Johansen*, Centre for Materials Science and Nanotechnology/Dep. of Physics, University of Oslo, Norway

The formation of insulating layers by acceptor dopants is important for β -Ga₂O₃ device fabrication. Commonly used acceptors in Ga₂O₃ are Mg, Cr, Zn and Cu. Typically the acceptors are introduced during growth, or by ion implantation. Acceptor incorporation by indiffusion is also viable but

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controlling the depth and concentration can be challenging. In this study, we utilize a combination of experimental techniques and first-principles calculations to identify the mechanism for Zn indiffusion, and to assess the prevailing defect configurations and their thermal stability.

Zn has been introduced into (001) and (-201) oriented β -Ga₂O₃ through vapor phase in sealed evacuated quartz ampules heated to temperatures in the range 900-1100°C, for 1 h. The concentration of Zn as a function of depth was measured using Secondary Ion Mass Spectrometry. The Zn concentration was found to reach a value of approximately 10^{20} cm⁻³, before it drops off abruptly, i.e., the concentration versus depth profile resembles that of a box profile. The profiles were successfully fitted by a trap-limited diffusion model, assuming that interstitial Zn (Zn_i) is the mobile specie, which then is trapped and released through dissociation from a hereto unknown defect. From this model, Zn_i migration barriers of 2.0±0.1 eV and 2.2±0.1 eV were extracted for the (001) and (-201) orientations, respectively, with corresponding dissociation energies of 3.2±0.8 eV and 3.0±0.5 eV.

Through first-principles calculations it was found that the Zn_i donor favors a split-interstitial configuration in which it shares a tetrahedral lattice site with Ga. The calculated migration barrier was found to be 2.3 eV in both [001] and [-201] directions, which is in line with the extracted values from the trap-limited model. Ga vacancies are known to trap donor impurities in β -Ga₂O₃ and is a candidate for the trap. However, the Zn_{Ga} acceptor is predicted to have a dissociation energy of 7.0 eV, making it highly stable and unlikely to dissociate once formed. Interestingly, the Zn_{Ga} acceptor can trap a second Zn_i on a Ga site, forming the donor complex Zn_iZn_{Ga}. The dissociation energy of this complex was found to be 3.7 eV. After Zn indiffusion, the samples were highly conductive, suggesting that the dominating defect is a donor, e.g., Zn_iZn_{Ga}. After a second heat treatment at 1000°C under O₂ flow, the samples turned insulating. This is consistent with Zn_iZn_{Ga} donors being the dominant defect after indiffusion, as such complexes would be expected to dissociate during the post-diffusion anneal, leaving behind compensating Zn_{Ga} acceptors.

MD-TuP-6 Initial Nucleation of Metastable γ -Ga₂O₃ During sub-Millisecond Thermal Anneals of Amorphous Ga₂O₃, *Katie Gann, C. Chang, M. Chang, D. Sutherland, A. Connolly, D. Muller, R. van Dover, M. Thompson*, Cornell University

Beta-phase gallium oxide (β -Ga₂O₃) is a promising semiconductor for high frequency, high temperature, and high voltage applications. But in addition to the thermodynamically stable β -phase, numerous other polymorphs exist and competition between various phases must be understood to control defects, especially during growth. The phase formation sequence of Ga₂O₃ from amorphous thin films deposited on non-crystalline neutral substrates was determined using lateral-gradient laser spike annealing to temperatures between 500 and 1400 °C on 400 μ s to 10 ms timescales (dwell). The resulting thermal anneal and quench ($10^4 - 10^6$ K/s) induced phase transformations were characterized with optical microscopy, X-ray diffraction, and transmission electron microscopy (TEM) to develop a processing phase diagram. X-ray characterization indicates that the γ -phase, a defect-spinel structure, first nucleates under all annealing time scales for peak temperatures between 650 °C and 800 °C. At peak anneal temperatures above 850 °C, the thermodynamically stable β -phase is observed after quenching to room temperature, with a small two-phase region near the boundary. Cross-sectional TEM at the onset of γ -phase formation shows nucleation near the center of the film with no heterogeneous nucleation at interfaces. For short duration anneals, the β -phase exhibits large grains which become finer and equiaxed for mid-duration anneals, and finally becoming textured for the highest temperatures at the longest dwells. The formation of the γ -phase prior to all β -phase formation, and the time-dependence of the grain structure, indicates that the γ -phase is initially the kinetically preferred phase, and that the subsequent β -phase formation occurs by heterogeneous nucleation at higher temperatures off of existing γ -phase grains. The low surface energy of the γ -phase implied by these results suggests a reason for widely observed γ -phase inclusions in β -phase Ga₂O₃ films grown by a variety of synthesis methods.

MD-TuP-7 Heavily Doped β -Ga₂O₃ Deposited by Magnetron Sputtering, *Adetayo Adedeji*, Elizabeth City State University; *J. Lawson, C. Ebbing*, University of Dayton Research Institute; *J. Merrett*, Air Force Research Laboratory

Epilayer of doped β -Ga₂O₃ films were deposited on 5 mm x 5 mm semi-insulating Fe-doped (010) β -Ga₂O₃ substrates and conducting Sn-doped (-201) β -Ga₂O₃ by magnetron sputtering. Doping of the epilayers was

achieved by co-sputtering pure Ga₂O₃ target with Si target or Fe target. Conducting Si-doped and insulating Fe-doped epilayers were achieved at substrate temperature of 570°C during a 2-hour deposition in Ar/O₂ gas mixtures (5% O₂ by flow rate). SIMS analysis indicated that the concentration of dopant can be controlled with the RF power on the targets. Si concentration $> 10^{20}$ cm⁻³ in the epilayer was achieved. Electrical transport measurements showed that the Fe-doped epilayer is insulating with expected high breakdown field strength. At the corners of Si-doped epilayer, Ti/Au contact metals were deposited by magnetron sputtering as well. Hall-Effect measurements at elevated temperatures indicated donor concentration ($> 5 \times 10^{18}$ cm⁻³) that increases slightly with temperature. Carrier mobility between 40 - 50 cm²/V·s was measured, and the value decreases slightly with temperature while the resistivity of the films did not change significantly with temperature. However, contact issues were observed at lower temperatures. High resolution x-ray diffraction (XRD) 2 θ -w, 2 θ and rocking curve measurements have shown that the epilayers are single crystalline.

MD-TuP-8 Point Defect Distributions in Ultrafast Laser Induced Periodic Surface Structures on β -Ga₂O₃, *D. Ramdin, E. DeAngelis, M. Noor, M. Haseman, E. Chowdhury, Leonard Brillson*, Ohio State University

β -Ga₂O₃ has received widespread attention due to its ultrawide bandgap, which can enable applications under extreme conditions. Ultrafast laser irradiation of β -Ga₂O₃ provides a means to explore the response of the material under these extreme conditions, which can generate point defects as well as modify structural features with electronic properties that differ from the pristine surface. However, an understanding of defects generated by femtosecond laser irradiation in the vicinity of laser induced periodic surface structures (LIPSS) remains to be explored. We correlate topographic features with the presence of defects and relative crystallinity using depth- and spatially resolved cathodoluminescence spectroscopy (DRCLS). We also explore how these defects change work function and associated band bending using Kelvin Probe Force Microscopy (KPFM). Defects are found to correlate with crystalline order and near-surface morphology, even in morphologically flat areas, thus a factor in building many applications such as ultrashallow ohmic contacts and quantum dot patterning.

Scanning electron microscopy, AFM, KPFM and DRCLS provide near-nanometer scale depth and spatially resolved information that reveals the interplay between topography, lattice disorder, and defect formation in the vicinity of LIPSS formed by femtosecond laser irradiation. Reduction in crystalline order can be correlated with the concentration of a defect with an ~ 2.4 eV optical emission but is also suggested to be affected by defects not observed by DRCLS, including a continuum of states increasing towards the band edges, typical of amorphous semiconductors. Thermodynamic calculations currently available suggest oxygen interstitials and divacancy complexes as likely candidates for the ~ 2.4 eV emission. Furthermore, laterally resolved CLS reveals that defects can be generated even in the region surrounding the LIPSS. KPFM measurements along with spatially resolved CLS reveal differences in the work function and crystalline order between crests and troughs, lending support to the hypothesis that the LIPSS are formed via the diffusion of point defects because of a relaxation of the surface instability caused by ultrafast laser irradiation. These results suggest that defect formation is an intrinsic feature of ultrafast laser irradiation and LIPSS formation in Ga₂O₃, which has extensive implications for both exploring the robustness of this material under extreme conditions as well as for device fabrication involving LIPSS. This work supported by AFOSR grants FA9550-18-1-0066, FA9550-20-1-0278, and FA9550-16-1-0069 and NSF grant DMR-18-00130.

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