A two-step growth of β-Ga_2O_3 films on (100) diamond via low pressure chemical vapor deposition

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Abstract

One of the major challenges in β-Ga_2O_3-based high power and high frequency devices is anticipated to be related to the low thermal conductivity of the material which is on the order of 10 – 30 W/m·K. The use of diamond (thermal conductivity ~ 2000 W/m·K) as substrate can be one effective approach for achieving better thermal management in β-Ga_2O_3-based devices. In this work, low pressure chemical vapor deposition was used to grow β-Ga_2O_3 films on (100) oriented, single crystalline diamond substrates. A two-step growth technique was employed to avoid the oxidation of diamond surface at high temperature. From X-ray diffraction measurements, the β-Ga_2O_3 films grew along the <-201> crystalline axis with the β-Ga_2O_3 (002) planes rotated by ±24.3-27° with respect to the diamond (111) planes. High magnification scanning transmission electron microscopy imaging revealed abrupt β-Ga_2O_3/diamond interface without any voids which is essential for high rate of heat transfer across the interface. N-type electrical conductivity was measured in a Si-doped β-Ga_2O_3 film with 1.4×10^{19} cm^{-3} electron concentration and ~3 $cm^2/V\cdot s$ electron mobility. This work demonstrates the feasibility of heteroepitaxy of β-Ga_2O_3 films on diamond substrates for potential device design and device fabrication with efficient thermal management.

Keywords: β-Ga_2O_3, low pressure chemical vapor deposition (LPCVD), single crystalline diamond substrate, twin domains

1. Introduction

β-Ga_2O_3, an ultrawide bandgap ($E_g \sim 4.5$-4.9 eV) material with a two-three fold higher estimated critical electric field and a three-five fold higher Baliga figure of merit than GaN and 4H-SiC [1], possesses great potential for power electronics [2] and ultraviolet optoelectronic [3] device applications. However, the thermal conductivity (Κ = 10-30 W/m·K) [4] of β-Ga_2O_3 is low, by about one order of magnitude as compared to GaN and 4H-SiC, which is unfavorable for fast heat dissipation from high power-density devices. A poor heat dissipation rate can cause localized Joule-heating leading to deteriorated transport properties and in turn, to unreliable device performance and faster device degradation [5].

Fabrication of β-Ga_2O_3-based devices on single-crystalline diamond (Κ > 2000 W/m·K) substrates could potentially circumvent the issues associated with the low thermal conductivity of the material. The use of diamond as the substrate for improved thermal management in AlGaN/GaN high electron mobility transistors has been investigated extensively [5-8]. Recently, β-Ga_2O_3 based field effect transistors fabricated on diamond substrates has been demonstrated via mechanical exfoliation [9-11]. However, the weak nature of the van der Waals bonding between the exfoliated β-Ga_2O_3 and diamond substrate can limit the heat transfer rate across the heterointerface [12] which can be improved by depositing β-Ga_2O_3 directly on diamond [13].

Low pressure chemical vapor deposition (LPCVD) is a scalable, cost-effective epitaxial technique for high-quality homoepitaxy and heteroepitaxy of β-Ga_2O_3 with a relatively fast growth rate. So far, homoepitaxy β-Ga_2O_3 has been demonstrated on (010) and (001)-orientated native substrates [14, 15]. On c-plane (0001) sapphire, phase pure β-Ga_2O_3 can be epitaxially grown with smooth surface morphologies [16, 17]. LPCVD β-Ga_2O_3 is one of the few heteroepitaxial thin film growth techniques [18, 19] which has demonstrated effective n-type conductivity by Si doping with decent Hall mobility measured (up to 126 $cm_2/V\cdot s$) at room temperature [17, 20, 22]. In-plane rotational domains with six-fold symmetry were observed in our LPCVD β-Ga_2O_3 on c-plane sapphire. By using off-axis sapphire substrates, the rotated domains can be gradually suppressed as the off-cut angle increases up to Δ_a=6° [20]. Later, a systematic study was performed to understand the dependence of key growth parameters on LPCVD Ga_2O_3 and demonstrated a wide tunable range of growth rate between <1 ~ 10 μm/hr [21]. In our most recent study, we explored the higher temperature growth (HT-LPCVD, up to 1050 °C) on both (010) native substrates and off-axis c-plane sapphire [16]. Decent room temperature transport characteristics were demonstrated with μ=156 $cm^2/V\cdot s$ on (010) native substrate and μ~120 $cm^2/V\cdot s$ on off-axis c-plane sapphire [22].

Given with the successful demonstration of heteroepitaxial β-Ga_2O_3 on sapphire, LPCVD Ga_2O_3 on diamond can be promising as long as the oxidation of the diamond surface is suppressed effectively. The oxidation rate of (100) diamond surface demonstrates an Arrhenius behavior [23] which requires a low growth temperature for reducing the risk of thermal degradation of the diamond substrate in the O_2 environment. However, the crystalline as well as carrier transport properties of LPCVD grown β-Ga_2O_3 improves with growth temperature [14] in the range from 780 °C to 950 °C. In this letter, we report on LPCVD of β-Ga_2O_3 films on (100) oriented, single crystalline diamond substrates using a two-step growth technique. This technique consists of (i) a low temperature capping step – a thin layer of Ga_2O_3 layer was grown at relatively low temperature (750 °C) to cap the diamond surface with the purpose to protecting it from thermal degradation and (ii) a high temperature growth step. Similar two-temperature technique has been previously used for metalorganic chemical vapor deposition of ZnO films on diamond substrates [24]. The out of plane orientation of the β-Ga_2O_3 films grown in this work was determined to be (-201) from X-ray diffraction (XRD) measurements and nano-diffraction patterns. XRD Φ scan profile indicated a ±24.3-27° rotation of β-Ga_2O_3 (002) planes with respect to the diamond (111) planes. Room temperature electron mobility of ~3 $cm^2/V\cdot s$ was measured in a Si doped sample with carrier concentration of 1.4×10^{19} cm^{-3}.

2. Experimental Section

β-Ga_2O_3 films were grown on (100) oriented, single crystalline diamond substrates using low pressure chemical vapor deposition. The growths were carried out in a custom-designed horizontal flow tube furnace equipped with programmable temperature and pressure controllers. High purity gallium (Ga) pallets (Alfa Aesar, 99.99999%) and oxygen (O_2) gas (99.999%) were used as the precursors whereas high purity argon (Ar) was used as the carrier gas. High purity silicon tetrachloride ($SiCl_4$) balanced by Ar was used as the n-type doping source. The metallic Ga was placed in a quartz crucible in such a way that the substrates were at a horizontally downstream location with respect to it. Once the Ga containing crucible and the substrates were loaded, the growth chamber was pumped for one hour and then purged under Ar flow for one hour at room temperature. The positions of the Ga containing crucible and the substrates were fixed during the growth. To prevent oxidation of the diamond surface during the ramping step, O_2 gas manifold was pumped beforehand to remove any residual gas. The O_2 precursor was not supplied until the reactor reached the desired growth temperature.

Three controlled samples were grown which are, hereafter, referred to as Sample A, Sample B, and Sample C, respectively. Sample A was grown at 750 ℃ with an O_2-to-Ar flow rate ratio ($R_{O2/Ar}$) of 1.7%. For sample B and C, a two-step growth technique was used which consists of (i) a low temperature step – 3 minutes of growth at 750 ℃ with $R_{O2/Ar}$ = 1.7% and (ii) a high temperature step – 20 minutes of growth at 900 ℃ with $R_{O2/Ar}$ = 3.3% (Sample B) or at 920 ℃ with $R_{O2/Ar}$ = 5% (Sample C). The $R_{O2/Ar}$ values for Sample B and Sample C were selected based on our previous growth optimization [21, 22]. No O_2 was flown during the temperature ramp between step (i) and (ii). Ar flow rates during the temperature ramp step were 100 sccm and 300 sccm in case of Sample B and Sample C, respectively. Therefore, the differences between the growth conditions of Sample B and Sample C are (i) the Ar flow rates during the temperature ramp up step, (ii) the temperature of the second growth step and (iii) the O_2 flow rates during the high temperature growth. For Sample C, 1.2 sccm of $SiCl_4$ was flown only during the high temperature to probe the doping properties in β-Ga_2O_3 grown on diamond.

Surface morphology of the films were investigated by field emission scanning electron microscopy (FESEM) using an FEI Apreo LoVac Analytical SEM. X-ray diffraction measurements were carried out using a Bruker Discover D8 XRD. Scanning transmission electron microscopy (STEM) was performed using a Thermofisher probe-corrected Titan STEM operated at 300 kV. Room temperature cathodoluminescence (CL) measurements were carried out using a Horiba H-Clue Cathodoluminescence detector integrated with a Thermo Scientific Quattro Environmental Scanning Electron Microscope (ESEM). The electron beam acceleration voltage was set at 15 kV. Room temperature Van der Pauw Hall measurement was performed using an Ecopia HMS-3000 Hall effect measurement system. The strength of the magnetic field was 1T.

3. Results and Discussion

Figure 1(a-c) present the XRD 2θ-ω scan profile obtained from Sample A, Sample B, and Sample C, respectively. For all three samples, three peaks were observed at 2θ = 18.98°, 38.49°, and 59.19°, identified as β-Ga_2O_3 (-201), (-402), and (-603) planes, respectively [25]. The peak at 2θ = 43.12° in the 2θ-ω scan profile of Sample C (Fig. 1(c)) corresponds to the Au (200) plane from a 200 nm Au layer which was deposited during TEM foil preparation [13]. Therefore, the Ga_2O_3 films in this work have monoclinic crystal structures regardless of the growth temperature. In addition, the dominant growth direction of β-Ga_2O_3 on (100) oriented, single crystalline diamond is along <-201> crystalline axis. The ω-rocking curves around the β-Ga_2O_3 (-201) peak obtained from Samples A-C are shown in Fig. 1(b). The additional peak observed in case of Sample C is probably due to an asymmetric plane which is slightly tilted with respect to the surface [20]. The *full width at half maxima* (FWHM) values are 2.1°, 2.7° and 1.1° for Samples A-C, respectively. For Sample A, a relatively low growth temperature (T = 750 °C) as compared to those used in previous works (900 °C - 1050 °C) [14-17, 20-22] was selected to prevent the oxidation-induced damage of the diamond surface [24]. A low substrate temperature results in low adatom mobilities on the growth surface [22, 26] which adversely affect the crystalline quality. In case of Sample C, the growth temperature was increased to T_H = 920 °C after initializing the growth at T_L = 750 °C. The increased adatom mobility at 920 °C improved the crystalline quality of the grown film resulting in a narrower (-201) ω-rocking curve. As mentioned in the experimental details section, the growth temperature and precursor flow rates used in this work were selected based on our previously optimized growth condition for β-Ga_2O_3 films grown on native as well as sapphire substrates. Additional investigation of β-Ga_2O_3 growth on diamond substrate including growth at higher temperatures is a part of the future work.

A large mis-oriented crystallite formed during the low temperature growth and/or the temperature ramp up step can work as a physical barrier for the adatoms and limit their mobility even at higher temperature [26]. Besides, such mis-oriented crystals can continue to grow during the higher temperature step further deteriorating the crystalline quality of the film. This is a possible reason for the deterioration of the crystalline quality of Sample B as compared to Sample A indicated by the increased FWHM of the (-201) ω-rocking curve in Fig. 1(b). This hypothesis is further supported by the FESEM images. Figures 2(a-c) depict the plan-view FESEM images of Samples A-C, respectively. The surface of Sample A, as shown in Fig. 2(a), can be characterized by the presence of small incompletely formed pseudo-hexagonal domains. In addition, few small mis-oriented crystallites protruding from the film are also visible. For Sample B, the FESEM image in Fig 2(b) shows an increase in the size of both the pseudo-hexagonal domains and the mis-oriented crystallites. On the other hand, the FESEM image in Fig 2(c) shows the completely coalesced surface of Sample C which has similar morphology as the β-Ga_2O_3 films grown on c-sapphire [14, 20]. The difference in the Ar flow rates during the temperature ramp up steps may have partly contributed to the observed difference between Sample B (100 sccm) and Sample C (300 sccm). Since the Ga containing crucible was kept inside the chamber, the supply of Ga adatoms to the sample surface continued during the ramp up step. An excess number of Ga adatoms at the growth surface can etch the already grown Ga_2O_3 by forming sub-oxides, for example, Ga_2O [27]. The higher concentration of Ga adatoms carried by the higher Ar flow rates in the case of Sample C can help reducing the size of the misoriented crystals resulting in the improved morphology as compared to Sample B.

Additional investigation on the crystal orientation of β-Ga_2O_3 films grown on (100) diamond was carried out by obtaining XRD Φ-scan profiles (Fig. 3) of β-Ga_2O_3 (002) plane and diamond (111) plane from Sample C. The angle between β-Ga_2O_3 (-201) and (002) planes is 50° whereas the angle between diamond (100) and (111) planes is 54.75°. 2θ and tilt angle were set as 31.7° and 50°, respectively, for β-Ga_2O_3 (002) planes. For diamond (111) planes, these values were set as 43.84° and 55°, respectively. Both Φ-scan profiles were obtained with the same mounting of the sample. Three diamond (111) peaks were observed within 270° scan range which is consistent with four-fold rotational symmetry of the diamond (111) plane around the <100> axis. In Fig. 3, each of the diamond (111) peak is surrounded by two β-Ga_2O_3 (002) peaks; the separation (ΔΦ) between the diamond (111) and surrounding β-Ga_2O_3 (002) peaks is ±(24.3°-27°). This indicates the formation of the twin domains which is a consequence of misalignment between the symmetry directions between the substrate and the epilayer [28]. The formation of twin domains due to the misalignment of symmetry direction between the substrate and the epilayer has previously been reported in case of (0001) GaN films grown on (111) Ge [29].

To further investigate the crystalline quality of the β-Ga_2O_3 films grown on diamond substrates, STEM imaging was performed. Figure 4(a) is a low-magnification cross-sectional STEM image obtained from Sample C which shows a sharp contrast between the grown film and the substrate. The thickness of the films was measured to be 1.7 μm indicating a growth rate of ~5.1 μm/hr. The near-interface region of the film has high density of extended defects which deceased near the surface. The high-magnification STEM image in Fig. 4(b) shows abrupt interface between β-Ga_2O_3 and diamond. No voids or exfoliation were observed indicating very high-quality interface which is essential for high thermal boundary conductance [13]. Figure 4(c) shows the nano-diffraction patterns obtained from the Sample C with [010] zone axis which confirms the growth of (-201) oriented β-Ga_2O_3 on (100) diamond substrate.

Room temperature cathodoluminescence measurements were performed to investigate the optical properties of the grown β-Ga_2O_3 films on diamond. Figure 5 presents the CL spectra, measured with a beam acceleration voltage of 15 kV, obtained from an β-Ga_2O_3 sample grown using similar conditions as Sample C. The measured CL spectra shown by the solid black curve in Fig. 5 appears to consist of three major features at 2.8 eV, 3.2 eV, and 3.6 eV (shown by the dotted lines) which are related to deep level defects in β-Ga_2O_3 [30]. Deep level defects at 2.5 eV, 3.0 eV and 3.5 eV in LPCVD grown Ga_2O_3 have previously been identified using depth resolved CL spectroscopy and surface photovoltage spectroscopy which showed spectral correlation with Ga vacancy (2.5 eV and 3.0 eV features) or O vacancy (3.5 eV feature) [30].

Room temperature Hall measurements were carried out on the samples to investigate the transport properties of the LPCVD grown β-Ga_2O_3 on diamond substrate. Sample A and Sample B, which are undoped, were found to be insulating whereas Sample C, which is Si-doped, showed n-type conductivity. The measured electron concentration and mobility values were 1.4×10^{19} cm^3 and ~3 $cm^2/V\cdot s$. The low electron mobility, partially suppressed by increased scattering rate due to the high carrier concentration, was probably additionally reduced by the high density of extended defects as observed in the cross-sectional STEM image in Fig. 4(a). Nevertheless, the results presented in this letter demonstrate thick, phase-pure β-Ga_2O_3 onto single-crystalline diamond despite the high lattice and thermal mismatch of these materials and the risk of oxidizing the diamond surface. These results thus substantiate the potential of single crystalline diamond to be used as a substrate for hetero-epitaxial β-Ga_2O_3 based devices.

4. Conclusion

In summary, a two-step growth technique was used for growth of β-Ga_2O_3 on (100) oriented, single crystalline diamond substrate using LPCVD. Dominant growth direction of β-Ga_2O_3 films was along the <-201> direction. XRD Φ-scans indicated the formation of twin domains with the β-Ga_2O_3 (002) planes rotated by 24.3-27° with respect to the diamond (111) planes. STEM images show abrupt β-Ga_2O_3/diamond interface without any void which is essential for high thermal boundary conductance. Room temperature electron mobility of ~3 $cm^2/V\cdot s$ was measured from a Si doped β-Ga_2O_3-on-diamond films with carrier concentrations of ~1.4×10^{19} cm^{-3}. This work demonstrates the feasibility of β-Ga_2O_3 film growth on single crystalline diamond substrates for device design and device fabrication with improved thermal management.

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Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Figure Captions

Figure 1. (a-c) X-ray diffraction (XRD) 2θ-ω scan profiles obtained from Sample A, Sample B, and Sample C, respectively. For all three samples, the peaks at 2θ = 18.98°, 38.49°, and 59.19° correspond to β-Ga_2O_3 (-201), (-402) and (-603) planes, respectively. In c, the peak at 2θ = 43.12° corresponds to Au (200) plane from a 200 nm Au layer deposited on Sample C during TEM foil preparation. The dominant growth direction of LPCVD grown β-Ga_2O_3 on (100) diamond is along the <-201> direction. (d) ω-rocking curve around the β-Ga_2O_3 (-201) peak obtained from Samples A-C.

Figure 2. Plan-view FESEM images of (a) Sample A, (b) Sample B, and (c) Sample C. Sample A was grown using one-step process with T = 750 °C. Sample B and Sample C were grown using two-step process with a low temperature step at T = 750 °C and a high temperature step at T = 900 °C and 920 °C, respectively. The Ar flow during the ramp up step from low temperature to the high temperature were 100 sccm and 300 sccm, respectively, for Sample B and Sample C.

Figure 3. XRD Φ-scan profile of the β-Ga_2O_3 (002) plane and diamond (111) plane obtained from Sample C. 2θ position and tilt-angle were 31.7° and 50°, respectively, for β-Ga_2O_3 (002) peaks whereas 43.84° and 55°, respectively, for diamond (111) peak. Both Φ-scan profiles were captured with the same mounting of the sample. A part of the sample was used to prepare TEM foils before the measurements were conducted. That is why the Φ-scan range was set to be 270°.

Figure 4. (a) Low magnification STEM image showing the cross-sectional morphology of Sample C. (b) High magnification STEM image showing the abrupt interface between β-Ga_2O_3 films and diamond substrate. (c) Nano-diffraction pattern obtained from the β-Ga_2O_3 region in (a) with <010> view direction confirming the monoclinic structure and (-201) out of plane orientation of the grown

films. The inset shows simulated TEM diffraction pattern of β-Ga_2O_3 from the <010> view direction.

Figure 5. Room temperature cathodoluminescence spectra (black curve) obtained from a β-Ga_2O_3 film grown on diamond substrate. The deconvolved peaks at 2.8 eV, 3.2 eV, and 3.6 eV positions are shown by dotted curves. The yellow dashed curve is the fitted curve.