Oxygen annealing induced changes in defects within $\beta$-Ga$_2$O$_3$ epitaxial films measured using photoluminescence

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Abstract

In this work, we use photoluminescence spectroscopy (PL) to monitor changes in the UV, blue, and green emission bands from n-type (010) Ga$_2$O$_3$ films grown by metalorganic vapor phase epitaxy induced by annealing at different temperatures under O$_2$ ambient. Annealing at successively higher temperatures decreases the overall PL yield and UV intensity at nearly the same rates, indicating the increase in the formation of at least one non-radiative defect type. Simultaneously, the PL yield ratios of blue/UV and green/UV increase, suggesting that defects associated with these emissions increase in concentration with O$_2$ annealing. Utilizing the different absorption coefficients of 240 and 266 nm polarization-dependent excitation, we find activation energy for the generation of non-radiative defects of 1.34 eV in the bulk but 2.53 eV near the surface. We also deduce activation energies for the green emission-related defects of 1.20 eV near the surface and 0.74 eV at high temperatures through the films, whereas the blue-related defects have activation energy in the range 0.72–0.77 eV for all depths. Lastly, we observe hillock surface morphologies and Cr diffusion from the substrate into the film for temperatures above 1050 °C. These observations are consistent with the formation and diffusion of V$_{Ga}$ and its complexes as a dominant process during O$_2$ annealing, but further work will be necessary to determine which defects and complexes provide radiative and non-radiative recombination channels and the detailed kinetic processes occurring at surfaces and in bulk amongst defect populations.

Keywords: Ga$_2$O$_3$, O$_2$ annealing, photoluminescence, intrinsic defect, Ga vacancy

(Some figures may appear in colour only in the online journal)

1. Introduction

Ultra-wide bandgap $\beta$-Ga$_2$O$_3$ is promising in applications such as power electronic devices, solar-blind UV photodetectors, solar cells, and gas sensors [1]. Melt growth techniques such as edge-defined film fed growth [2] and Czochralski method [3] have been used to grow large-size single crystals, which sets $\beta$-Ga$_2$O$_3$ apart from other ultrawide gap materials like SiC, AlN, and diamond. The identification and manipulation of defects in Ga$_2$O$_3$ are important for its future applications. Thermal annealing under oxidizing atmospheres [2, 4–6] (oxygen partial pressure $p_{O_2}$ higher than the equilibrium vapor pressure of O$_2$ over Ga$_2$O$_3$ [7]) is found to increase the resistivity of Ga$_2$O$_3$ crystals. The few 100 nm region near surface is more resistive than bulk after O$_2$ annealing [4, 5] and its thickness can be suppressed by a capping SiO$_2$ layer [4]. The relation between resistivity and $p_{O_2}$ also applies in single crystal growth [8] and thin film deposition [9].
Furthermore, under air annealing at 1450 °C for 20 h, O content of unintentionally-doped (UID) crystals increases while Ga content decreases as measured by energy-dispersive x-ray spectroscopy and x-ray photoelectron spectroscopy [6], indicating detectable mass change/loss [10].

For many decades, the reduction in electron density upon annealing in oxidizing atmospheres is attributed to a reduction in oxygen vacancy \((V_O)\) concentration [8], which was assumed to be a shallow donor without direct evidence. However, density functional theory calculations using hybrid functions, which in recent years have significantly increased the accuracy of calculations of defect charge transition levels and other energies, indicate that \(V_O\) should be a deep donor [11]. Thus, \(V_O\) should not be the primary defect responsible for the resistivity increase under O2 annealing. Note that Si and H impurities are leading candidates for the background n-type conductivity with theoretical [11] and experimental [12–14] evidence. Although direct experimental evidence of the microscopic changes in defect concentrations is sparse, positron annihilation spectroscopy (PAS) studies demonstrate that \(V_{Ga}\) is efficiently formed during epitaxial growth of UID and Si-doped Ga2O3 films and under O2 annealing [15, 16]. Electron paramagnetic resonance (EPR) spectra show O2 annealing induces an IR1 center which is assigned to the double negative charge state of either isolated \(V_{Ga}\) at the tetrahedral site or its relaxed configuration sometimes referred to as a \(V_{Ga0}−G_{Ga0}−V_{Ga0}\) split vacancy complex [17]. Despite these basic insights, distinguishing main defects accounting for resistivity increases and fully investigating the formation, diffusion, and complexing mechanisms involved remain to be completed.

Photoluminescence spectroscopy is widely used to characterize defects. Four characteristic PL bands are observed for a majority of \(\beta\)-Ga2O3 samples upon super-gap excitation, namely, UV (centered at 3.4 eV), UV’ (centered 3.0–3.2 eV), blue (centered 2.7–2.8 eV), and green (centered near 2.4 eV) [18]. Recent computations have shown that many different point defects and complexes offer interstitial transitions that should fall into the observed green-blue-UV’ bandwidths and be vibronically broadened [19]. This may ultimately prevent unambiguous attribution of any of these peaks to one particular defect or complex. Besides, both wide and sharp red luminescence features are observed in some Ga2O3 crystals. The very sharp \(R1\) and \(R2\) lines very similar to ruby emissions as well as a broadened structure are caused by Cr [20, 21] (not Fe) while some evidence exists for some different broad red features being associated with defects like N [22] and \(V_{Ga}−H\) acceptor [23].

The UV band’s behavior is consistent with recombination of conduction electrons with self-trapped holes (STH) [24, 25], while the blue and green bands are generally ascribed to donor-acceptor pair recombination [26]. The ratios of blue and green (3.0 eV and 2.5 eV) peak yields to the UV yield are enhanced greatly after neutron irradiation [27], suggesting they arise from native defects. Galazka et al observed that the blue emission in Sn-doped Ga2O3 crystal is greatly enhanced after O2 annealing [10], but offered no further explanations. The green band is observed to be enhanced in the presence of specific impurities such as Be, Li, Ge, and Sn [28], and with higher p02 during crystal growth or after O2 annealing in UID crystals [29].

In this work, we use photoluminescence spectroscopy from MOVPE-grown homoepitaxial (010) \(\beta\)-Ga2O3 films subjected to O2 annealing to investigate the defect-related processes during annealing. The ratios of blue and green peaks to the UV systematically increase with annealing temperature suggesting increases in the associated defect concentrations. Relying on PL interpretation along with prior positron annihilation, EPR, and computational results, we associate the increased blue emission ratio to increased concentrations of compensating \(V_{Ga}\) and its acceptor-like complexes. We derive activation energies for the process(es) introducing \(V_{Ga}\)-related defects into the films, which may include surface and bulk formation as well as bulk diffusion energies. Lastly, we observed isolated hillock surface morphologies and Cr diffusion from the substrate into the depth sampled by PL at sufficiently high temperatures, again supporting the hypothesis that the formation of \(V_{Ga}\) and related complexes is the major process during annealing in high p02 atmospheres.

2. Experimental

About 800 nm thick Si-doped \(\beta\)-Ga2O3 films were grown by MOVPE [30] (Agnitron Agilis) on (010)-oriented NCT Fe-doped Ga2O3 substrates using triethyl gallium (TEGa), diluted silane, and oxygen precursors. Annealing was conducted at 1 atm in a quartz tube furnace in which the heating rate and O2 flow rate were 10 °C min\(^{-1}\) and 20 sccm respectively. Temperatures of 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C, and 1100 °C were selected with a holding time of 2 h. To control for any sample-to-sample variations, the same sample was measured in PL at 25 °C and then annealed at the next increased temperature. Hall effect measurements (Ecopia HMS 7000) were used to characterize carrier density and mobility of as-grown film (then the contacts were etched off) and after annealing at 1100 °C to avoid any contamination or alteration of the sample arising from adding and removing contacts between the intermediate annealing steps. Photoluminescence measurements were performed using both 266 nm and 240 nm excitations which provide depth selectivity. The polarization direction of the incident pulsed laser was rotated by a half-wave plate. The laser power was actively monitored (∼25 mW, 0.07 cm\(^2\)). Emission spectra were collected in reflection mode using an integrating sphere with a fiber connected to an Avantes spectrometer. For the analysis of PL spectra, the UV, green and blue bands were fit using fixed peak energies of 3.4 eV, 3.05 eV, and 2.6 eV all having fixed full-width at half-maximum of 0.4 eV.

3. Results and discussion

The Si-doped as-grown film exhibits a carrier density of \(3 × 10^{17} \, \text{cm}^{-3}\) and mobility of 90 cm\(^2\) (V s\(^{-1}\)) at room temperature, measured by the Hall effect. After O2 annealing at 1100 °C, the resistivity exceeds the measurement limit of 10\(^6\) Ω cm, agreeing with the
reported resistivity increase after O$_2$ annealing. All samples exhibited UV, blue, and green emission (Figure 1). The shape and intensity of PL spectra exhibit similar features under 240 nm excitation for annealing at 850 °C and 900 °C. As the annealing temperature increases, the total emission yield decreases continuously which we take as an indication of increasing non-radiative processes. Thus, the formation of at least one non-radiative defect center, whether in the bulk or at the surface, is the first defect-related process induced by annealing in O$_2$.

Before analyzing the PL spectra, normalizing the emission spectra to the UV emission is justified below. This should remove the influence of the non-radiative channels on excess carrier populations and thus we can consider each radiative channel. The absence of band-edge emission [31, 32] indicates free holes are not present in the valence band. Experiments probing the ultrafast dynamics of photo-generated carriers show several ps and hundreds of ps timescales for STHs on oxygen sites [31] and holes trapped by defects [32], respectively. According to the phenomenological rate equation of holes after photo-excitation, holes will be captured by O sites to form STHs, be captured by acceptors, and recombine at non-radiative centers. Thus, the relative emission intensity is proportional to the relative defect density responsible for it since the capture rate constant of defects should not change. Note that the detrapping energy for electrons and holes for the blue emission are measured to be 0.04 and 0.42 eV, respectively [26]. Another work obtained a similar 18 meV for electrons detrapping in blue emission [33]. Thus, the involved donor should be shallow like Si$_{Ga}$ with ionization energy in tens of meV, rather than a deep donor like V$_{O}$. We thus propose that the blue emission is related to transitions between shallow donors and deep acceptors, and similarly for the green emission. Due to the constant concentration of O sites, and thus intensity of the UV emission coming from recombination of conduction electrons with STHs on O sites, we can normalize the other emission features to the UV emission. By normalizing out the dependence on STH lifetime, we can deduce changes arising from changing defect concentrations. We recognize that understanding all subtleties in the PL spectra will require further consideration of the charge states of defects and carrier transport and dynamics which is out of the scope of this study.

The UV intensity changes in nearly the same way as the total PL yield (Figure 2(a)), indicating the dominant process to form STH. The decrease of carrier concentration (increased resistivity) upon annealing suggests a decrease of shallow donors or an increase in compensating acceptors. Si dopant will only diffuse instead of being deactivated under O$_2$ annealing above 1100 °C. A small amount of hydrogen, which could act as a donor as well as passivating acceptors, is possibly removed during oxygen annealing [34]. We have established via Hall, CV, and SIMS that Si is the dominant dopant in our films doped with Si, thus the hydrogen loss mechanism should not be dominant in our experiments [35]. Thus, more likely cause is the increase in compensating acceptors with annealing. The dependence of electron density on O$_2$ annealing arises from the acceptor density, and its related PL intensity should go up with high acceptor concentrations. The relative intensities of the blue and green emissions do change as a function of annealing temperature. V$_{Ga}$ and its complexes are most probable given the existing literature and our observations. In computations for defect formation in the bulk, V$_{Ga}$ exhibits the lowest formation energy amongst native acceptors under oxygen-rich condition [36, 37], especially for n-type doped films in which the Fermi energy approaches the conduction band. Combining with the facts of the reported PAS results [15, 16] and the indicated intrinsic defect nature by neutron irradiation [27], ascribing the increased blue emission to increases in the concentrations of V$_{Ga}$ and related complexes is consistent with the mentioned findings.

Enhanced green peak might also come from intrinsic defects, as revealed by the growth of crystals in high O$_2$ partial pressure [29] and this work under O$_2$ annealing. Compared to blue emission energy, the lower emission energy indicates defects involved could be deeper acceptor (cannot rule out deeper donor). More work is needed to demonstrate the correlation between green emission and related defect, and its correlation to the electrical properties of Ga$_2$O$_3$.

In the absence of mass transport to or from the surface, smoothening to reduce surface area rather than increasing it would be expected. In Figure 3, changes in the surface...
morphology from linear features to isolated hillock features after annealing at 1100 °C do hint at a significant mass transport processes involving the surface. No etching or decomposition and desorption of suboxides is expected at these temperatures, pressures, and $P_{O_2}$ according to thermodynamics [7]. The 1 atm of pure $O_2$ overpressure implies $\sim 10^9 O_2$ atoms s$^{-1}$ would bombard each surface site. Such oxygen flux would be sufficient to grow a similar number of monolayers of Ga$_2$O$_3$ per second were sufficient Ga supplied. Given these considerations, we propose that this change in morphology results from gallium atoms diffusing to the surface and reacting with little adatom diffusion with the copious impinging $O_2$ to form new unit cells of Ga$_2$O$_3$ in hillocks as opposed to smooth layer by layer growth. If the out-diffusion mechanism for Ga is via substitutional Ga or (2$^V_{Ga}$ + $Ga_i$) complexes [38], it could equivalently be viewed as the formation of $V_{Ga}$ or the complex at the surface having one activation energy followed by its in-diffusion under another activation energy.

Interestingly, the red luminescence originating from Cr [21] is observed from within the penetration depth of the laser from the top surface of the epilayer after annealing at 1050 °C and 1100 °C (figure 3(c)), but not at lower temperatures. We suspect that this arises via the diffusion of Cr from the Fe-doped substrate into the film. Note that all of the ion-implanted Si, Sn, and Ge exhibit increased concentration near the surface (<20 nm) upon $O_2$ annealing [39–41], indicating that the formation energy of $V_{Ga}$ at the surface may be smaller than that in bulk. The situation could be that the $V_{Ga}$ forms at the surface and diffuses into bulk, while the Cr impurities diffuse into films by exchanging position with $V_{Ga}$, thus moving towards the surface.

Table 1 shows the extracted activation energy ($E_a$) for the UV intensity and the blue/UV and green/UV intensity ratios vs. $1/T_{anneal}$. The $E_a$ of UV absolute intensity is 2.53 eV near the surface and 1.34 eV in deep film, which represents the quenching of the UV peak by increased non-radiative recombination accompanying higher-temperature annealing. The $E_a$ extracted from green emission is higher than that of blue

Table 1. The activation energies fitted by $N_0 \exp \left(-E_a/k_BT\right)$ from 950 °C. Penetration depth is calculated using the absorption coefficients of (010) crystal [43].

| Excitation | Penetration depth (nm) | UV blue/UV | green/UV |
|------------|-----------------------|------------|----------|
| 240 nm $E//c$ | 30                    | 2.53       | 0.72     | 1.20     |
| 266 nm $E//c$ | 500                   | 1.34       | 0.77     | 2.21 at low $T$ | 0.74 at high $T$ |

[11] including Cr [21, 42]. On the other hand, the diffusions of Si, Sn, Ge during $O_2$ annealing are faster than that in $N_2$ annealing [40]. This can be explained by the higher concentration of $V_{Ga}$ in O-rich condition compared to O-poor one, consistent with our results and theoretical indication [37]. The ion-implanted Si, Sn, and Ge exhibit increased concentration near the surface (<20 nm) upon $O_2$ annealing [39–41], indicating that the formation energy of $V_{Ga}$ at the surface may be smaller than that in bulk. The situation could be that the $V_{Ga}$ forms at the surface and diffuses into bulk, while the Cr impurities diffuse into films by exchanging position with $V_{Ga}$, thus moving towards the surface.

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emission, suggesting different defect origins or mechanisms between them. Considering the as-mentioned experimental phenomena, we try to find evidence that the surface formation energy is relatively small and the bulk diffusion process follows a higher barrier value, in the depth (b axis) direction. The blue peak seems to exhibit only a slight change of $E_a$ while the green has a stronger surface vs bulk contrast. Further work will be necessary to determine more accurate activation energies for their formation, reactions, and transport at the surface and within the bulk.

Since there is no reported value on $V_{Ga}$ formation and diffusion energy, we compare it with other processes. Mg starts diffusing already through interstitial Ga [44] at 800 °C [45], indicating a smaller diffusion barrier. The predicted $E_a$ for Mg diffusion (slightly greater than 2 eV) [44] is higher than our extracted $E_a$ related to $V_{Ga}$. This is likely due to an offset between computational and experimental values. Our extracted values are also smaller than the 4.1 eV extracted using the indirect method of temperature and time using an equivalent circuit model of CV measurements [4]. It is reasonable that the extracted $E_a$ differs due to the different physical processes involved.

4. Conclusions

We investigated the photoluminescence of Si-doped (010) β-Ga$_2$O$_3$ MOVPE films as they become semi-insulating under O$_2$ annealing. The overall PL yield and the UV yield decrease in the same ways upon annealing. The blue and green to UV intensity ratios exponentially increase with annealing temperature. We extract activation energy of 0.72–0.77 eV for blue emission for all depths in the film suggesting a bulk process, while the green emission data hints at differentiation between surface and bulk. Cr impurities are observed to diffuse into the epitaxial film at 1050 °C–1100 °C, possibly by substitutional diffusion mediated by $V_{Ga}$. Our findings, including the morphology change of the surface seen in AFM, are all consistent with the formation of $V_{Ga}$ and its complexes as a major defect process during O$_2$ annealing. Note that our results cannot prove that they alone are the cause of the decreased electron concentration, lower overall PL yields, and changes in blue and green emissions.

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