Influence of Annealing Atmosphere on the Characteristics of \( \text{Ga}_2\text{O}_3/4\text{H-SiC n-n Heterojunction Diodes} \)

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Abstract: \( \text{Ga}_2\text{O}_3/4\text{H-SiC n-n isotype heterojunction diodes were fabricated by depositing Ga}_2\text{O}_3 \) thin films by RF magnetron sputtering. The influence of annealing atmosphere on the film quality and electrical properties of \( \text{Ga}_2\text{O}_3 \) layers was investigated. X-ray diffraction (XRD) analysis showed a significant increase in the peak intensities of different faces of \( \beta-\text{Ga}_2\text{O}_3 \{−201\}, \{−401\} \) and \( \{002\} \). X-ray photoelectron spectroscopy (XPS) measurement showed that the atomic ratio of oxygen increases under high-temperature annealing. Moreover, an \( \text{N}_2 \)-annealed diode exhibited a greater rectifying ratio and a lower thermal activation energy owing to the decrease in oxygen-related traps and vacancies on the \( \text{Ga}_2\text{O}_3 \) film and \( \text{Ga}_2\text{O}_3 \)–metal interface.

Keywords: gallium oxide; silicon carbide; heterojunction diodes; thermal activation energy

1. Introduction

Wide bandgap (WBG) semiconductors find applications in high-power transistors and light detectors. Among the more promising WBG materials, gallium oxide (\( \text{Ga}_2\text{O}_3 \)) is uniquely transparent to visible and ultraviolet light [1–3]. It has a bandgap ranging from ~4.6 to ~4.9 eV, resulting in a high electric breakdown field strength of ~8 MV/cm. The Baliga’s figure of merit (BFOM) of \( \text{Ga}_2\text{O}_3 \) is 3400, which is roughly four times higher than that of gallium nitride [4,5]. \( \text{Ga}_2\text{O}_3 \) has five crystalline modifications (\( \alpha, \beta, \gamma, \delta, \) and \( \epsilon \)), among which the monoclinic \( \beta \)-phase is most stable. Metastable \( \text{Ga}_2\text{O}_3 \) films can be obtained by thermal annealing and can be subsequently converted into \( \beta-\text{Ga}_2\text{O}_3 \) in a relatively convenient manner. \( \text{Ga}_2\text{O}_3 \) is natively n-doped in the range of \( 10^{16}–10^{18} \) cm\(^{-3} \) due to oxygen vacancies and can be further n-doped to free carrier densities by adding Si, Sn, or Ge [6–11].

Recrystallization through thermal annealing helps reduce oxygen-related charge traps and is generally an effective method for improving the quality of \( \text{Ga}_2\text{O}_3 \) [12,13]. Therefore, investigating the annealing process for \( \text{Ga}_2\text{O}_3 \) is a promising research direction. Polycrystalline \( \text{Ga}_2\text{O}_3 \) films on glass or sapphire substrates have been converted from amorphous phase through high-temperature annealing [14–16]. Hexagonal silicon carbide (4H-SiC; bandgap of ~3.26 eV) can be used as a substrate to grow \( \beta-\text{Ga}_2\text{O}_3 \) layers [17]. Hexagonal silicon carbide (a, b = 3.10 Å and c = 10.12 Å) has a low lattice mismatch of ~2 % with \( \text{Ga}_2\text{O}_3 \) (a = 12.33 Å, b = 3.04 Å, and c = 5.80 Å). It also and exhibits a higher thermal conductivity (~4.5 W/cm·°C) than other WBG materials such as GaN (~1.3 W/cm·°C) and \( \text{Ga}_2\text{O}_3 \) (0.5 W/cm·°C), making it a suitable substrate for high power applications.

In this work, heterojunction diodes were fabricated by depositing \( \text{Ga}_2\text{O}_3 \) on a 4H-SiC substrate and annealing the diodes under different annealing gases. The effects of the applied annealing gas on the material properties of the resulting \( \text{Ga}_2\text{O}_3 \) thin films and the electrical performance of the diodes manufactured from this material are investigated.
2. Materials and Methods

As a substrate for the gallium oxide film, we used a n-type 4H-SiC wafer (doping concentration: $5 \times 10^{16}$ cm$^{-3}$), with a layer of epitaxially grown 4H-SiC (n-type; $1.0 \times 10^{19}$ cm$^{-3}$), as shown in Figure 1. After cleaning the SiC wafer with SPM solution ($\text{H}_2\text{SO}_4$:$\text{H}_2\text{O}_2 = 4:1$), we stripped the native silicon dioxide ($\text{SiO}_2$) layer using a buffered oxide etch (BOE). A 200-nm-thick nickel film cathode was formed on the bottom side of the SiC wafer by E-beam evaporation. After Ni deposition, the samples were annealed at 950 °C in N$_2$ for 10 min by rapid thermal annealing (RTA) for forming ohmic contacts. Gallium oxide thin films were then deposited by radio frequency (RF) sputtering of a Ga$_2$O$_3$ (99.99% purity) target. Before deposition, the chamber was evacuated to $2.0 \times 10^{-6}$ Torr. The films were grown on the epitaxial 4H-SiC layer under 35 mTorr at a pure argon mass flow rate of 4.6 sccm. The RF power was 140 W, and the films were deposited on room temperature. The thickness of the deposited films ranged from 100 to 250 nm. The SiC wafers, with the deposited Ga$_2$O$_3$ films, were annealed at 800 °C for 40 min under different atmospheres (pure oxygen and nitrogen gas). An electrode was formed by deposition of 120 nm of nickel on the Ga$_2$O$_3$ layer.

![Figure 1. Structure of Ga$_2$O$_3$/4H-SiC heterojunction diode and fabrication flow.](image)

3. Results

3.1. Material Properties

To compare the influence of different annealing atmospheres on the crystallinity of Ga$_2$O$_3$ deposited on the 4H-SiC substrates, X-ray diffraction (XRD) $\theta$–$2\theta$ scans were performed on the as-grown, O$_2$ and N$_2$–annealed samples. As shown in Figure 2, all the sample sets show reflections corresponding to polycrystalline Ga$_2$O$_3$ with a monoclinic structure from Rietveld refinement by using General Structure Analysis System (GSAS) [18,19]. All the manufactured samples give $\beta$-Ga$_2$O$_3$ diffraction peaks corresponding to ($-201$), ($-401$), and (002) faces. The crystal structures remained stable. In fact, the peak intensities were further enhanced after annealing. In particular, the peak intensities corresponding to the ($-201$) and ($-401$) faces significantly increased after N$_2$ annealing. As explained in the literature, Ga and O atoms migrate under high-temperature annealing and thus help improve the crystallinity of Ga$_2$O$_3$. Furthermore, dangling bonds related to oxygen defects at grain boundaries can be passivated by N$_2$ annealing by incorporating nitrogen atoms at gallium or oxygen lattice sites [20,21]. Consequently, the diffraction peak intensities of the N$_2$-annealed samples are higher than those of the other samples, as nitrogen appears to improve the crystal quality of the Ga$_2$O$_3$ [22,23].
where \( h \) is the photon energy.

The bandgap of the \( \beta \)-phase \( \text{Ga}_2\text{O}_3 \) film is found to be \( \approx \) 5.01 eV. The bandgaps of the samples annealed under \( \text{O}_2 \) and \( \text{N}_2 \) atmosphere are \( \approx \) 4.91 and \( \approx \) 4.89 eV, respectively. The bandgap of the \( \text{N}_2 \)-annealed sample is close to the typically reported bandgap value of \( \approx \) 4.9 eV for \( \beta \)-phase \( \text{Ga}_2\text{O}_3 \) [26].

![XRD spectra of samples and refinement results of \( \text{Ga}_2\text{O}_3 \) annealed under different atmospheres.](image)

**Figure 2.** XRD spectra of samples and refinement results of \( \text{Ga}_2\text{O}_3 \) annealed under different atmosphere.

Figure 3a shows the optical transmittance spectra of the samples for wavelengths between 200 and 400 nm. All the samples exhibit a high transmittance (over ~80 %) at wavelengths longer than 300 nm. The oxygen concentration in the \( \text{Ga}_2\text{O}_3 \) crystals will affect the charge states, which in turn will influence such electrical parameters as bandgap and, consequently, the transmittance [24,25]. The optical bandgap is extracted from the linear part of the graph, shown in Figure 3b, for (\( \alpha h\nu \)) \( ^2 = 0 \), where \( h\nu \) is the photon energy, and \( \alpha \) is the coefficient of absorption. \( \alpha = \ln(100/T)/d \), where \( T \) and \( d \) is the transmittance and thickness (120 nm) of the \( \text{Ga}_2\text{O}_3 \) films, respectively. For the as-grown samples, the bandgap of the \( \text{Ga}_2\text{O}_3 \) film is found to be \( \approx \) 5.01 eV. The bandgaps of the samples annealed under \( \text{O}_2 \) and \( \text{N}_2 \) atmosphere are \( \approx \) 4.91 and \( \approx \) 4.89 eV, respectively. The bandgap of the \( \text{N}_2 \)-annealed sample is close to the typically reported bandgap value of \( \approx \) 4.9 eV for \( \beta \)-phase \( \text{Ga}_2\text{O}_3 \) [26].

![Transmittance spectra and \((\alpha h\nu)^2\)–\(h\nu\) plots of \( \text{Ga}_2\text{O}_3 \) film samples annealed under different atmospheres.](image)

**Figure 3.** (a) Transmittance spectra and (b) (\( \alpha h\nu \)) \( ^2 \)–\( h\nu \) plots of \( \text{Ga}_2\text{O}_3 \) film samples annealed under different atmospheres.

Figure 4 shows the XPS spectra of the O 1s peaks of the three different sample sets. The peaks were calibrated using C 1s at 284.6 eV, in which the O 1s peaks were fitted using two Gaussian peaks, corresponding to \( \text{Ga}_2\text{O}_3 \) and \( \text{GaO}_x \) phases, respectively. After annealing in \( \text{O}_2 \) and \( \text{N}_2 \) atmosphere, the peak intensity of the \( \text{GaO}_x \) phase decreases, whereas that of the \( \text{Ga}_2\text{O}_3 \) phase increases. The \( \text{GaO}_x \) phases.
peak is reported to have a connection with oxygen vacancies [26]. The magnitude of the peak intensity corresponding to the GaO2 phase was reduced, from 37.5% for the as-grown sample to 20.3% and 13.6% for the O2 and N2-annealed samples, respectively. This is considered to indicate a decrease in the number of defects, such as oxygen vacancies and oxygen sites. The atomic ratios of O to Ga in the samples are 1.40, 1.43 and 1.42 for as-grown, O2 and N2 annealed Ga2O3. These different stoichiometric ratios indicate that an increased concentration of O2 in the annealing gas can somewhat raise the number of oxygen atoms in the films.

![Graph showing XPS spectra of Ga2O3 films annealed under different atmospheres.](image)

**Figure 4.** O 1s XPS spectra of the Ga2O3 films annealed under different atmospheres.

### 3.2. Electrical Properties

Figure 5 shows the graph of 1/C2 as a function of the reverse voltage bias applied to the Ga2O3/4H-SiC diodes. The built-in voltage (Vbi) and doping concentration can be extracted from the extrapolated graph of 1/C2 versus the voltage. Vbi is calculated from the V-axis intercept of the fitted graph. The doping concentration is derived from the slope of 1/C2−V using Equation (1).

\[
\frac{1}{C^2} = \frac{2}{qA^2} \left( \frac{\varepsilon_{Ga2O3} N_{Ga2O3} + \varepsilon_{SiC} N_{SiC}}{N_{Ga2O3} N_{SiC} \varepsilon_{Ga2O3} \varepsilon_{SiC}} \right) (V_{bi} - V)
\]  

(1)

The extracted values of Vbi and doping concentration of the Ga2O3 thin films are 0.47, 0.86, and 1.01 V and 9.59 \ times 10^{15}, 1.62 \ times 10^{16}, and 2.01 \ times 10^{16} cm^{-3} for the as-grown, O2-annealed, and N2-annealed samples, respectively. The Vbi and doping concentration values increased after annealing, because of the decrease in oxygen-related traps. The increase in the built-in voltage can be attributed to the changes in the dopant concentration and the concentration of interface states of Ga2O3.

![Graph showing 1/C2−reverse voltage plots for different diodes measured at room temperature.](image)

**Figure 5.** 1/C2−reverse voltage plots for different diodes measured at room temperature.
Figure 6 shows the typical I–V characteristics of the fabricated Ga$_2$O$_3$/4H-SiC n-n diodes both in the logarithmic and linear scales. As shown in the figure, the as-grown diode has a high leakage current ($\sim 1.60 \times 10^{-5}$ A) and a low rectifying ratio ($\sim 3.0 \times 10^3$) measured at forward (3 V) and reverse biases (−3 V). The rectifying behavior of the O$_2$ and N$_2$-annealed diodes is improved. The different samples exhibit a similar leakage current value of approximately $8.1 \times 10^{-11}$ A. The N$_2$-annealed diode exhibits a higher on-current when a forward voltage is applied, with a rectifying ratio of $\sim 5.0 \times 10^7$, which may be related to the reduced oxygen trap concentrations after annealing. The threshold voltages of the diodes are $\sim 1.55$, $\sim 1.47$, and $\sim 1.27$ V for the as-grown, O$_2$ and N$_2$-annealed samples, respectively. The ideality factor at room temperature can be extracted from Equation (2).

$$I = I_0 [e^{\frac{V}{kT}} - 1]$$

Here, $I$ and $V$ are the forward current and voltage, respectively, $I_0$ is the saturation current, $k_B$ is Boltzmann’s constant, $T$ is the absolute temperature, and $\eta$ is the ideality factor. The ideality factor is significantly reduced after the annealing process; the ideality factor of the N$_2$-annealed diode is 2.8, which is half that of the O$_2$-annealed diode. The lower ideality factor and the higher built-in voltage of the annealed diodes are attributed to the improved crystallinity and interface properties.

![Figure 6. I–V characteristics of diodes at room temperature: (a) log-scale and (b) linear curves.](image)

The thermal activation energy ($E_A$) is obtained from the ln ($I_0$)−1/kT plot shown in Figure 7. The graph was plotted in the temperature range of 298–523 K with a temperature step of 25 K, where $I_0$ is the reverse saturation current at $-3\, V$, and $T$ and $k$ are the absolute temperature and Boltzmann’s constant, respectively. The extracted activation energy from the experimental measurements are related to trap states at the metal–Ga$_2$O$_3$ interfaces and the barrier heights. Low activation energy values suggest a high concentration of the trap states at the interface, which results in increased trap-assisted tunneling or thermionic emission probabilities across the barrier. As shown in Figure 6, the extracted activation energy of the devices increases after annealing. In particular, the activation energy of the N$_2$-annealed sample (0.504 eV) is twice that of the as-grown sample. The improved rectifying ratio of the N$_2$-annealed diode is also attributed to the increased activation energy.
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**Figure 7.** ln(Iₒ)–1/kT curve for activation energy values derived from the increasing temperature.

4. Conclusions

We fabricated polycrystalline β-Ga₂O₃/4H-SiC heterojunction diodes annealed under different gas atmospheres (O₂ and N₂). The material and electrical properties of the diodes were investigated to understand the effects of the different annealing gases on the device characteristics. X-ray diffraction peaks corresponding to the different faces of β-Ga₂O₃ (−201), (−402), and (002)) were observed to significantly increase, while the bandgap somewhat decreased to ~4.9 eV after annealing. The post-annealing decrease in the GaO₆ peak intensity indicates a decrease in the number of oxygen vacancies. With regard to the electrical properties, the leakage current decreased nearly 1000 times after annealing. To summarize, the N₂-annealed sample exhibited higher rectifying ratio and built-in voltage, decreased threshold voltage, lower ideality factor, and higher activation energy than the as-grown and O₂-annealed samples. Therefore, we conclude that the performance of N₂-annealed diodes at high temperatures is more stable due to higher activation energy compare with built-in voltage due to a lower concentration of trap states.
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