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Electrical properties of unintentionally doped β -Ga₂O₃ (010) thin films grown by a low-pressure hot-wall metalorganic chemical vapor deposition

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We investigated the electrical properties of unintentionally doped (UID) Ga₂O₃ (010) layers grown by low-pressure hot-wall metalorganic chemical vapor depositions from device characteristics of Schottky barrier diodes (SBDs) fabricated on them. Highly resistive properties of the UID Ga₂O₃ layers were confirmed from current–voltage characteristics. The specific on-resistance of the SBD with the most resistive UID Ga₂O₃ layer was $2.2 \times 10^7 \Omega \text{cm}^2$. Capacitance–voltage characteristics revealed that most of the SBDs had complete depletion of the UID layers at thermal equilibrium, indicating that their residual effective donor densities were less than $3.0 \times 10^{13} \text{cm}^{-3}$. © 2024 The Japan Society of Applied Physics

β -Ga₂O₃ has been attracting a great deal of interest as a next-generation semiconductor material owing to its superior material properties such as a large bandgap energy of 4.5 eV,¹⁾ a large breakdown electric field of over 7 MV cm⁻¹,^{2–4)} and controllable *n*-type conductivity in a wide range of electron density by intentional donor doping.^{5–7)} The breakdown electric field leads to a large Baliga's figure of merit of β -Ga₂O₃,⁸⁾ which is several times larger than those of SiC and GaN. Another important material feature of β -Ga₂O₃ is that bulk single crystals can be synthesized by various melt growth methods such as floating zone,⁹⁾ Czochralski,^{10,11)} edge-defined film-fed growth,¹²⁾ vertical Bridgman,^{13,14)} and crucible-less bulk melt growth.¹⁵⁾ These physical properties suggest the great potential of β -Ga₂O₃ for various applications to power electronics and harsh environment electronics. Taking advantage of the availability of high-quality β -Ga₂O₃ epitaxial substrates, vertical Ga₂O₃ FETs and diodes with homoepitaxial drift layers have been energetically developed.^{16–24)} From a productivity perspective, there are two representative epitaxial growth techniques suitable for future mass production of Ga₂O₃ epitaxial wafers: halide vapor phase epitaxy (HVPE) and metalorganic chemical vapor deposition (MOCVD). HVPE can provide high-quality Ga₂O₃ thin films at a high growth rate of over 20 $\mu\text{m h}^{-1}$ ^{25,26)} and thus has been most commonly used to grow drift layers of vertical Ga₂O₃ devices. In contrast, MOCVD growth of Ga₂O₃ had been suffering from a severe problem of low growth rate.^{27–31)} Furthermore, residual electron densities of typical cold-wall MOCVD-grown unintentionally doped (UID) Ga₂O₃ films are on the order of 10¹⁵ cm⁻³ or larger^{29,31–36)}; therefore there is still a need for improvement. Recently, Yoshinaga et al. reported high-speed growth of high-purity Ga₂O₃ homoepitaxial films by low-pressure hot-wall MOCVD that they newly developed.³⁷⁾ It has been confirmed that the structural properties of the MOCVD-grown Ga₂O₃ films are as good as those of HVPE-grown ones; however, their electrical properties have never been investigated. In this work, we studied room-temperature electrical properties of UID Ga₂O₃ thin films grown by the MOCVD technique.

After hydrofluoric acid (HF) cleaning of Sn-doped *n*⁺-Ga₂O₃ (010) substrates, 15 μm -thick UID Ga₂O₃ layers were grown on the substrates by the low-pressure hot-wall MOCVD using

trimethylgallium as a Ga precursor. The growth rate was 5 $\mu\text{m h}^{-1}$, and details of the other growth parameters were reported elsewhere.³⁷⁾ In cases of as-grown Sn-doped *n*-Ga₂O₃ bulk single crystals and HVPE-grown Si-doped *n*-Ga₂O₃ films, high-temperature annealing is required to activate doped donors.^{12,38)} Therefore, in order to investigate the annealing effect on the electrical properties of the MOCVD-grown UID Ga₂O₃ layers, we prepared two types of samples with and without high-temperature annealing. All the other device processes were simultaneously conducted under the same conditions.

Figures 1(a) and 1(b) show a schematic cross-section of Ga₂O₃ Schottky barrier diodes (SBDs) fabricated in this work and a photo of the substrate on which the SBDs were fabricated, respectively. Blue circles in Fig. 1(b) indicate the devices used for current density–voltage (*J*–*V*) measurements. For one of the samples, high-temperature annealing was performed at 1150 °C in a purified N₂ atmosphere for 60 min prior to the device processing, and the other was as-grown. The SBD fabrication process began with lapping and chemical mechanical polishing (CMP) of the substrate back-sides. After the cleaning of the substrates with organic solvent, buffered HF, and piranha solution, Si-ion implantation doping was performed for the backsides to form a 150 nm-deep box profile with a Si density of $5 \times 10^{19} \text{cm}^{-3}$, followed by activation annealing at 925 °C in a N₂ atmosphere for 20 min.^{39,40)} Note that both the CMP process to remove a damaged region and the high-density Si doping are necessary to form low-resistance ohmic contacts on the Ga₂O₃ (010) surface. Subsequently, another CMP was performed for the epitaxial layers to ensure surface flatness and remove a surface depletion region formed during the high-temperature annealing.¹⁹⁾ The epitaxial layer thickness after the CMP was 8–9 μm . After 60 nm-deep BCl₃ reactive ion etching (RIE) of the substrate back surfaces, a Ti(20 nm)/Au(200 nm) metal stack was evaporated on them for ohmic cathode electrodes, followed by rapid thermal annealing at 470 °C in a N₂ atmosphere for 1 min.⁴¹⁾ Finally, circular Schottky anode electrodes composed of Pt(15 nm)/Ti(10 nm)/Au(200 nm) layers were formed on top of the epitaxial layers by liftoff. Anode electrodes with diameters of 200 and 400 μm were used for *J*–*V* and capacitance–voltage (*C*–*V*) measurements, respectively.

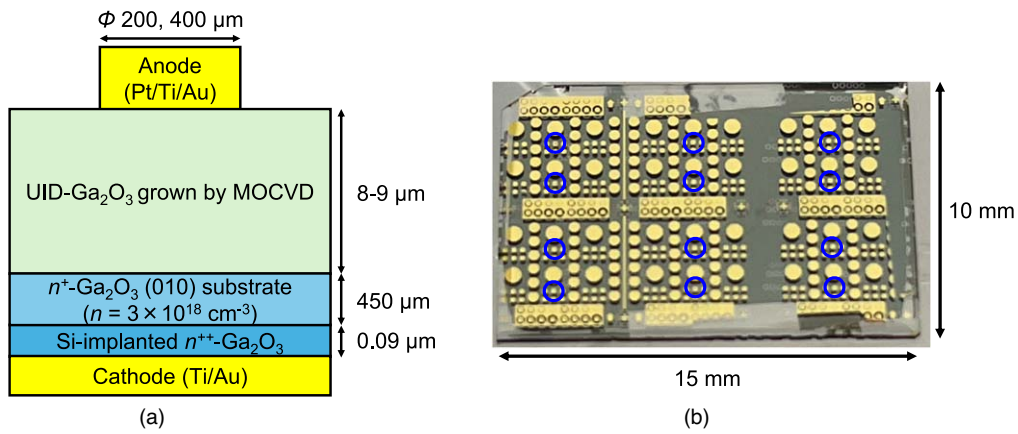


Fig. 1. (a) Cross-sectional schematic of Ga₂O₃ SBD structure and (b) photo of epitaxial substrate with fabricated SBDs.

Figures 2(a) and 2(b) show the J - V characteristics of the SBDs fabricated on the annealed and non-annealed Ga₂O₃ (010) substrates, respectively. Each figure includes typical characteristics of the SBDs fabricated at a center and an edge part on the substrate. Here, “edge” refers to the area within approximately 3 mm from both the left and right ends of the substrate. All the SBDs exhibited n -type diode behavior. When comparing the center- and edge-positioned devices, a significant difference in forward J was observed for both annealed and non-annealed substrates. High resistivities of the MOCVD-grown Ga₂O₃ thin films were confirmed from large specific on-resistances ($R_{\text{on,sp}}$) of the SBDs fabricated at the center parts; the largest $R_{\text{on,sp}}$ were 2.2×10^7 and $4.2 \times 10^6 \Omega\text{cm}^2$ on the annealed and non-annealed substrates, respectively. On the other hand, the SBDs at the edge parts had relatively low $R_{\text{on,sp}}$ of 89 and $2.8 \times 10^{-2} \Omega\text{cm}^2$ on the respective substrates. The in-plane distributions of J at $V = +3 \text{ V}$ for the SBDs on the annealed and non-annealed substrates are summarized in Figs. 3(a) and 3(b), respectively, to see resistivity distributions in more detail. With or without the annealing process, the SBDs fabricated at the center part showed extremely low forward J of less than $1 \times 10^{-6} \text{ A cm}^{-2}$, which were at least more than four orders of magnitude lower than those of the SBDs at the edge part. This large difference in forward J , i.e., the $R_{\text{on,sp}}$ between the center and edge parts was probably due to differences in MOCVD growth parameters such as the substrate temperature and the gas flow rate, which could be caused by fixtures holding four corners of a substrate. It

seems that the resistivity was a little increased by the annealing process; however, the causal relationship is still unclear.

Figures 4(a) and 4(b) show the C - V characteristics of the SBDs fabricated at the center parts on the annealed and non-annealed substrates, respectively. Both SBDs had almost constant C regardless of V , indicating that the MOCVD-grown UID Ga₂O₃ layers were completely depleted over the whole V range. From calculations using the mean value of C and the relative permittivity of 10.87 in the [010] orientation,⁴²⁾ residual effective donor densities ($N_{\text{d}}-N_{\text{a}}$) of the UID Ga₂O₃ layers on both of the annealed and non-annealed substrates were estimated to be less than $3.0 \times 10^{13} \text{ cm}^{-3}$. Note that the $N_{\text{d}}-N_{\text{a}}$ were the lowest for MOCVD-grown homoepitaxial Ga₂O₃ films,^{29,31-36)} and that even the relatively large $N_{\text{d}}-N_{\text{a}}$ for the edge devices were still at the level of low 10^{15} cm^{-3} . These results are qualitatively consistent with the fact that UID Si and C densities in Ga₂O₃ layers grown at similar conditions by the same MOCVD reactor were less than the detection limits of the secondary ion mass spectrometry measurement.³⁷⁾

Reverse J - V characteristics of the Ga₂O₃ SBDs fabricated on the annealed and non-annealed substrates are shown in Figs. 5(a) and 5(b), respectively. The breakdown measurements were executed using Fluorinert™ to avoid discharging. Both SBDs on the annealed and non-annealed substrates revealed high destructive breakdown voltages of 1.63 and 1.75 kV, respectively. Using thicknesses of the Ga₂O₃ epitaxial layers extracted from the C - V characteristics, the average electric fields in the

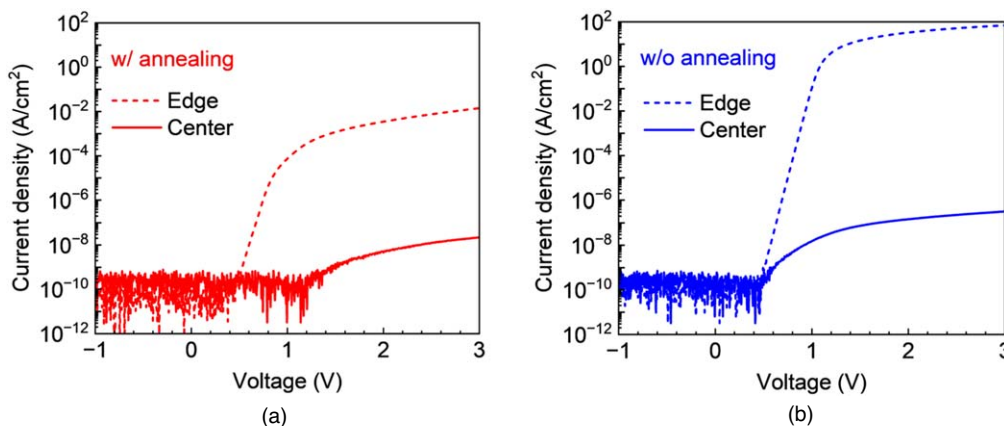


Fig. 2. J - V characteristics of UID Ga₂O₃ SBDs fabricated at center and edge parts on (a) annealed and (b) non-annealed epitaxial substrates.

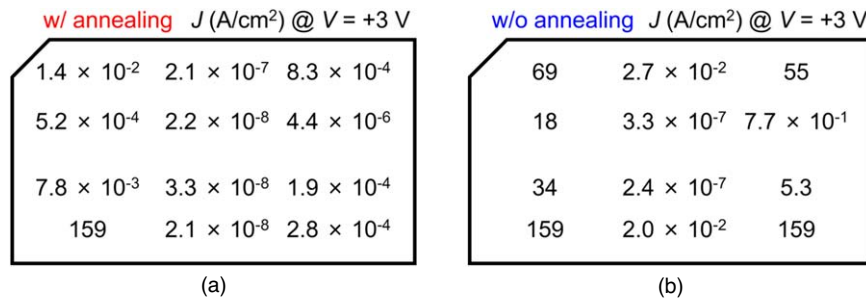


Fig. 3. In-plane distribution of J at $V = +3$ V for SBDs fabricated on UID Ga₂O₃ layers (a) with and (b) without the annealing process.

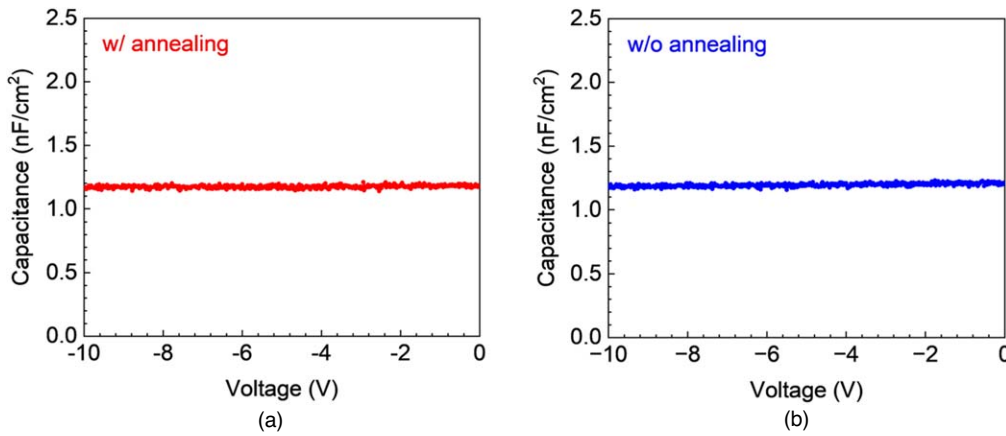


Fig. 4. C - V characteristics of UID Ga₂O₃ SBDs fabricated at center parts on (a) annealed and (b) non-annealed substrates.

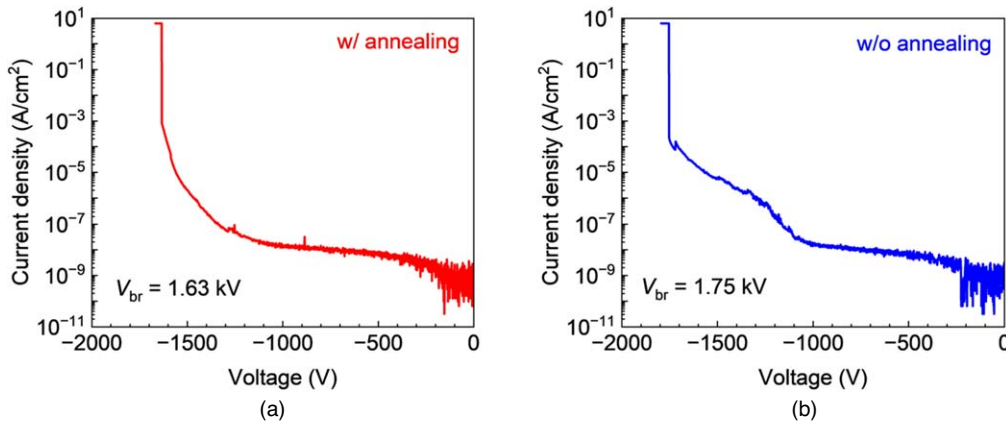


Fig. 5. Reverse J - V characteristics of UID Ga₂O₃ SBDs fabricated at center parts on (a) annealed and (b) non-annealed substrates.

UID Ga₂O₃ layers at the breakdown events were estimated to be 2.0 and 2.2 MV cm⁻¹ for the annealed and non-annealed epitaxial layers, respectively. These excellent breakdown characteristics of the SBDs without any edge termination can be attributed to the high crystal quality of the MOCVD-grown UID Ga₂O₃ layers.

We investigated the electrical properties of UID Ga₂O₃ thin films grown by low-pressure hot-wall MOCVD. The UID layers had extremely high resistivities, which were typified by large $R_{on,sp}$ on the order of 10^7 Ωcm² or higher for SBDs with them as drift layers. Complete depletion of the UID layers was also confirmed at thermal equilibrium, which corresponded to a $N_d - N_a$ of less than 3.0×10^{13} cm⁻³. The average electric fields in the UID layers when the destructive breakdown happened were estimated to be ~ 2 MV cm⁻¹. These results indicate that the low-pressure hot-wall MOCVD-grown Ga₂O₃

films had highly resistive properties and should be suitable for future device developments.

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