
Oxidation Behavior of OVPE-GaN Substrates for Slurry-less Photoelectrochemical Mechanical Polishing

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Abstract

Gallium nitride (GaN), characterized by a wide bandgap, high breakdown field, and excellent electron mobility, is a leading candidate for next-generation power semiconductors. Low-resistivity GaN substrates grown by oxide vapor phase epitaxy (OVPE) are well suited to vertical device architectures, enabling high current density and reduced on-resistance. However, spatial non-uniformity of carrier concentration inherent to OVPE growth alters surface chemical reactivity and impedes attainment of atomically flat, sub-nanometer surfaces. Moreover, it is particularly challenging to process GaN because of its chemical inertness, high hardness, and brittleness. Conventional chemical-mechanical polishing (CMP) exhibits low removal rates, high slurry cost, and post-polish topography correlated with local carrier density; UV- or electrochemical-assisted variants improve removal via surface modification yet still imprint carrier-dependent roughness. In this study, slurry-less photoelectrochemical mechanical polishing (PECMP) was proposed. UV irradiation combined with an electrochemical field induced a controllable modified layer on GaN, which is subsequently removed using fixed-abrasive instead of slurry to achieve high-efficiency, high-quality polishing. This method selectively removes only the oxidized layer, making the oxidation step central to both material removal and final surface quality. Accordingly, the modification mechanism was investigated, and the distinct carrier-concentration dependences of the UV-induced and electrochemical oxidation processes was clarified. By tuning the relative contributions of the two processes, modification is equalized across regions with differing carrier densities, yielding a spatially uniform modified layer.

Keywords: Electro chemical machining (ECM), Nano manufacturing, Oxidation, Photochemical machining

1. Introduction

Gallium nitride (GaN) has attracted widespread attention as a next-generation power device material owing to its superior physical properties. In particular, vertical GaN power devices require substrates with both low electrical resistivity and high crystalline quality, since large currents flow through the substrate thickness. Among the growth techniques for GaN single-crystal substrates that satisfy these requirements, oxide vapor phase epitaxy (OVPE) has recently gained significant interest. OVPE enables the growth of GaN crystals with high carrier concentrations and low threading dislocation densities, making it a promising approach for realizing vertical GaN power devices with low on-resistance [1]. To fully exploit these advantages, OVPE-GaN substrates with highly smooth surfaces are required.

Conventionally, chemical mechanical polishing (CMP) has been employed for the planarization of semiconductor substrates. In CMP, a slurry containing oxidizing agents and abrasive particles is used. Substrates surface is lightly modified with the oxidizing agents and surface planarization is achieved by mechanically removing the oxidized and softened surface layer. However, OVPE-GaN substrates inherently exhibit in-plane non-uniformity in dopant concentration originating from the crystal growth process. When CMP is applied to such substrates, spatial variations in chemical reactivity translate into local differences in material removal rate (MRR), which degrade surface roughness and can leave characteristic topographic non-uniformities after polishing. In addition, GaN is chemically inert, and the oxidation

reaction that initiates CMP proceeds slowly, limiting the MRR to below ~100 nm/h. The resulting long processing times and high slurry consumption increase manufacturing costs and impose an environmental burdens associated with slurry handling and disposal. Consequently, the CMP process has become a bottleneck in the mass production of GaN substrates.

To overcome these challenges, it is necessary to control the oxidation reactivity on OVPE-GaN in a manner that is insensitive to dopant non-uniformity, and selectively remove the modified layer under damage-free mechanical conditions using soft fixed abrasives, without slurry. Since the oxidation reaction of GaN is strongly dependent on hole supply processes and interfacial reaction kinetics, reaction fields with different carrier generation mechanisms and driving forces are expected to exhibit distinct oxidation responses to dopant concentration.

In this study, photochemical and electrochemical reaction fields were considered and the oxidation behavior under individual and combined fields were systematically investigated. By examining the oxidation reactivity as a function of dopant concentration, oxidation conditions capable of mitigating in-plane non-uniformity and enabling the formation of a uniform surface modified layer were discussed.

2. Experimental setup

An n-type OVPE-GaN (0001) substrate provided by Panasonic Holdings Corporation was used in this study. Prior to the oxidation experiments, the substrates were pre-processed by diamond lapping, resulting in a

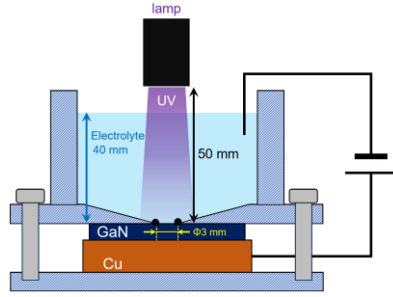


Figure 1. Schematic of the experimental setup.

Table 1. PEC oxidation conditions

Electrolyte	Phosphoric Buffered Solution (pH6.86)
Applied Bias	3.0 / 4.0 / 10.0 V
UV irradiation intensity	250 [mW/cm ²] @365 nm
Oxidation Time	60 / 30 / 1 s

surface roughness of $S_a = 0.8$ nm. The GaN substrate was fixed onto a copper plate and used as the anode, while a platinum mesh served as the cathode. Both electrodes were immersed in the electrolyte.

As shown schematically in Fig. 1, the electrolyte was filled into a tank, and only a circular region with a diameter of 3 mm on the GaN substrate was exposed to the electrolyte by sealing the surrounding area with fluororubber. The anode and cathode were connected to a potentiostat (Ivium Technologies, IviumStatXRe) and operated under voltage-controlled conditions. Ultraviolet (UV) irradiation was provided by a UV lamp (USIO Electric Co., SP9-250DB). A neutral phosphate buffer solution (pH 6.86) was used as the electrolyte in order to suppress isotropic etching of gallium oxide. The oxidation conditions are summarized in Table 1. The UV irradiation intensity was 250 mW/cm² at 365 nm, while the applied voltage was systematically varied. The oxidation time was adjusted so that the total reaction charge was maintained at a comparable level among different conditions. After oxidation, the oxide layer was removed by HF cleaning, and the modified interface was exposed and observed using a scanning white light interferometer (SWLI).

3. Results and discussion

Figure 2(a) shows the surface morphology before oxidation under an applied voltage of 3.0 V, while Fig. 2(b) shows the surface after oxidation followed by HF cleaning to remove the oxide layer. As observed in Fig. 2(b), regions where oxidation proceeded more deeply were distributed in a floral-like pattern. This indicated that the local oxidation rate in the floral-like regions was relatively higher than that in the surrounding areas. These floral-shaped regions have been confirmed to correspond to areas with lower carrier concentration [2]. Therefore, in this condition, the oxidation behavior was attributed primarily to UV-assisted oxidation. Under UV irradiation, photogenerated holes were more likely to recombine in regions with higher carrier concentration, resulting in the suppression of the oxidation reaction in those regions [3].

In contrast, Fig. 3(a) shows the surface morphology before oxidation under an applied voltage of 10.0 V, and Fig. 3(b) shows the surface after oxidation followed by HF cleaning. Unlike the results obtained at 3.0 V, the oxidation rate in the floral-shaped low-carrier-concentration regions was relatively lower than that in the surrounding regions. This behavior suggested that anodic oxidation became dominant at 10.0 V. In high-carrier-concentration regions, charge transport was facilitated, which enhanced the oxidation reaction. These results demonstrated that UV-assisted oxidation and anodic oxidation exhibited

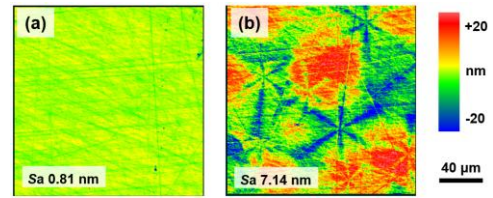


Figure 2. SWLI images of the GaN surface (a) before and (b) after photoelectrochemical oxidation under UV irradiation with an applied bias of 3.0 V.

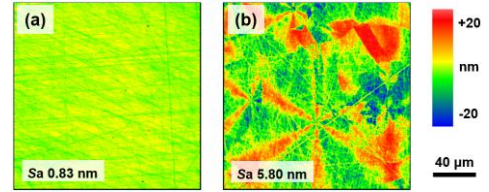


Figure 3. SWLI images of the GaN surface (a) before and (b) after photoelectrochemical oxidation under UV irradiation with an applied bias of 10.0 V.

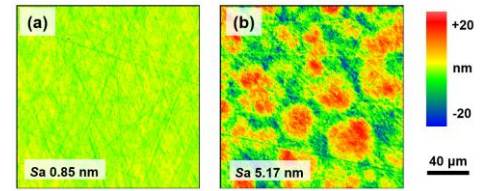


Figure 4. SWLI images of the GaN surface (a) before and (b) after photoelectrochemical oxidation under UV irradiation with an applied bias of 4.0 V.

opposite responses to in-plane carrier concentration non-uniformity during the oxidation of OVPE-GaN substrates.

Based on this finding, oxidation was carried out at an applied voltage of 4.0 V in order to adjust the relative contributions of the photochemical and electrochemical processes. Figure 4(a) shows the surface morphology before oxidation, and Fig. 4(b) shows the surface after oxidation followed by HF cleaning.

At 4.0 V, oxidation suppression caused by hole recombination during UV-assisted oxidation in high carrier concentration regions was counterbalanced by oxidation enhancement associated with improved charge transport under anodic oxidation. Consequently, in-plane uniform oxidation independent of floral-like pattern carrier concentration non-uniformity was achieved.

4. Conclusions

In this study, it was clarified that anodic oxidation and UV-assisted oxidation exhibit opposite dependencies on carrier concentration under fixed UV irradiation intensity. In high-carrier-concentration regions, oxidation was promoted by the electric field, whereas oxidation tended to be suppressed under UV irradiation. By controlling the applied voltage to adjust the relative contributions of these competing reactions, oxidation rate differences originating from in-plane carrier concentration non-uniformity were effectively compensated, enabling uniform surface modification. In future work, a high-efficiency slurry-less polishing process will be developed by combining the uniform oxidation technique demonstrated in this study with polishing using soft fixed abrasives.

References

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