

# Atomic Layer Etching of III-Nitride Semiconductors

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*Summer Program Website: <https://cnf.cornell.edu/education/reu/2024>*

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*Primary CNF Tools Used: Veeco Icon AFM, ABM Contact Aligner, Zeiss SEMS, Plasma-Therm Takachi ALE, Oxford 81 RIE etcher, Oxford 100 PECVD, P-7 Profilometer, Woolam RC-2 Ellipsometer*

## Abstract:

III-nitride semiconductors offer unique advantages in the manufacturing of high-voltage field effect transistors (FETS). However, production of nanoscale III-nitride FETs requires precise etching of III-nitride semiconductor films. We identified atomic layer etching (ALE) as an ideal technique for this application. ALE weakens the bond between a film and its surface layer via a chemical reaction with a reagent gas or plasma. The reagent is then purged, and the surface is bombarded with non-reactive ions. These ions impart enough energy to sputter away the reacted surface layer, but not enough to remove unreacted material underneath. These self-limiting characteristics allow for consistent removal of a few atomic layers at a time [1].

To adapt and characterize ALE for the processing of III-nitride films, samples with gallium nitride (GaN), aluminum nitride (AlN), and aluminum-gallium nitride (AlGaN) films were etched in the Cornell NanoScale Facility's (CNF) Plasma-Therm Takachi ALE tool. All ALE recipes tested used a  $\text{Cl}_2$  and  $\text{BCl}_3$  chemistry. However, key parameters such as gas flow rate, bias power, inductively coupled plasma (ICP) power, and step times were varied. Critical etch metrics such as surface roughness, film thickness, etch rate, and etch selectivity were recorded for each trial. By identifying recipe elements that yielded favorable etch results, this report gives preliminary guidance for the ALE of III-Nitride semiconductors.

## Summary of Research:

The first round of ALE processing was performed with one of each sample type (GaN, AlN, AlGaN) and a photoresist mask process. Nlof-2020 negative photoresist was chosen due to its popularity in device fabrication. Test features were patterned onto the samples to provide convenient etch characterization sites. The recipe flowed 30 sccm each of  $\text{Cl}_2$  and  $\text{BCl}_3$  for

surface modification and applied 10 W of bias power for ion bombardment. All three samples were processed at the same time, for 20 ALE cycles.

Following ALE, profilometry was performed on the AlGaN sample, producing the reading shown in Figure 1. Notably, this reading indicated that the "etched" areas were  $\sim 250$  nm higher than the unetched areas. This meant that something must have deposited during the etching process. This substance can be seen in SEM imagery of the sample (Figure 2), where the raised squares should be etched into the surface. Both the GaN and AlN samples exhibited deposition as well.

To identify the substance, energy-dispersive x-ray spectroscopy (EDS) was employed to map elements on the sample's surface. Figure 3 shows an SEM image of the AlGaN sample with a corresponding boron EDS map. Boron correlates with deposits along the edge of the etch pad, suggesting that it played a role in deposition. However, EDS is not sensitive enough to lighter elements to positively identify the material.

While inconclusive, the EDS results hinted that photoresist might play a role in the deposition. Consequently, our process flow was adapted to use an  $\text{SiO}_2$  hard mask for the next trial. Only AlN was tested, due to limited sample availability. The ALE recipe was altered to flow 30 sccm of  $\text{Cl}_2$  and 3 sccm of  $\text{BCl}_3$ , while the bias power was reduced to 8 W. These changes limited boron concentration, reducing the likelihood of adverse reactions. The sample was processed for 100 total ALE cycles, with measurements taken after 50 cycles.

Initial results from the hard-mask sample were promising, with ellipsometry indicating an etch depth of 20 nm after 50 cycles. The etched surface was also significantly smoothed, with a pre-etch roughness of 2.66 nm RMS and a post-etch roughness of 1.70 nm RMS. Unfortunately, the sample also exhibited crystalline defects across all exposed AlN surfaces.

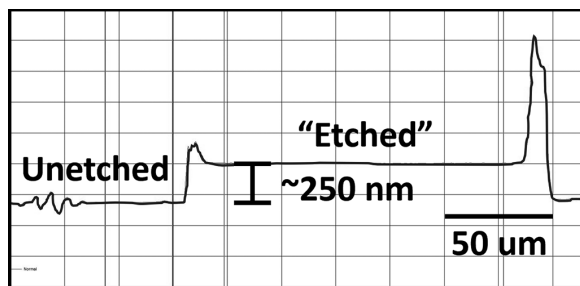


Figure 1: Profilometry reading of “etched” feature.

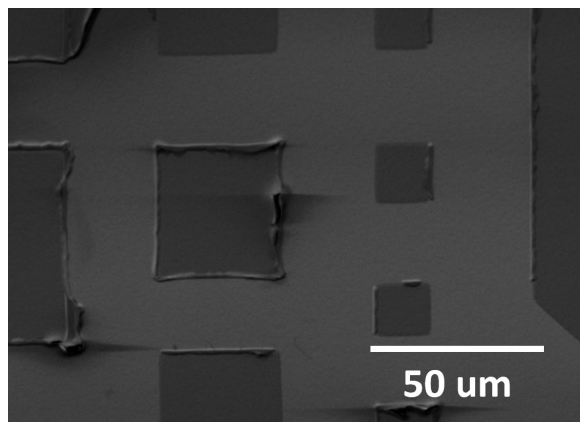


Figure 2: SEM image of “etched” squares.

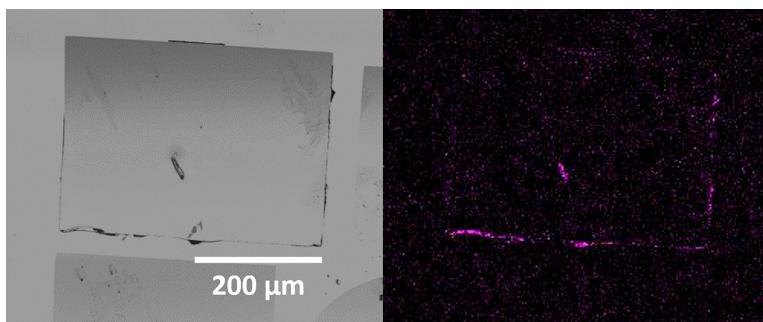


Figure 3: SEM and boron EDS imagery of photoresist-masked sample.

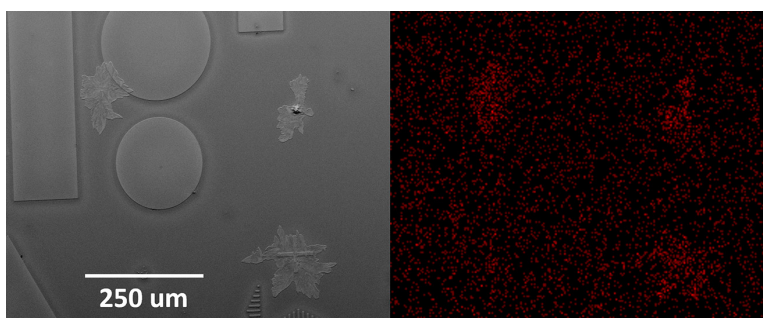


Figure 4: SEM and chlorine EDS image of hard-mask sample, including crystalline defects.

These defects are visible in the SEM image of Figure 4, where they appear to have grown across the patterned surface.

EDS was once again used to analyze the defects, with the EDS image Figure 4 showing a chlorine map of the sample. Chlorine hotspots strongly correlate with the defects, indicating that they contain chlorine. However, no other elements demonstrated a similar correlation. This poses an issue, as pure chlorine is a gas at room temperature. Logically, there must be other elements in the defects that we were unable to positively identify.

### Conclusions and Future Steps:

While ALE processing was not wholly successful, these trials highlight “best practices” that will hopefully contribute to a mature process flow in the future. The use of a hard mask is the most apparent, as it likely contributed to the significantly cleaner result of the second trial. Hard masks, especially  $\text{SiO}_2$ , are known for their resistance to unwanted chemical interaction. This is especially valuable for a high precision process like ALE, where even minimal unexpected reactions can disrupt the etching cycle.

Looking to the future, there are several promising approaches to obtain more favorable results. First, it would be useful to analyze the deposits with wavelength-dispersive x-ray spectroscopy (WDS). This technique is similar to EDS, but

it offers increased sensitivity to lighter elements. This may allow for positive identification of the deposited materials, and subsequent process alterations to prevent their formation. Aside from WDS, an ALE recipe without  $\text{BCl}_3$  would entirely remove a potential source of unwanted chemical reactions. We believe that these techniques and alterations will bring us one step closer to the successful atomic layer etching of III-nitrides.

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### References:

- [1] Keren J. Kanarik, Samantha Tan, and Richard A. Gottscho. Atomic Layer Etching: Rethinking the Art of Etch; The Journal of Physical Chemistry Letters 2018 9 (16), 4814-4821. DOI: 10.1021/acs.jpcclett.8b00997 Copyright 2018 by American Chemical Society.