

# A Systematic Study of How Different Phases of Niobium Nitride ( $\text{Nb}_x\text{N}$ ) React to Xenon Difluoride ( $\text{XeF}_2$ ) Undercut Etch

**2023 CNF REU Intern: Daniel Joel Harrison**

**Intern Affiliation: Electrical and Computer Engineering, Morgan State University**

2023 CNF REU Principal Investigator: Professor Debdeep Jena, Electrical and Computer Engineering, Cornell

2023 CNF REU Mentor: Wenwen Zhao, Applied and Engineering Physics, Cornell University

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Contact: dahar73@morgan.edu, djena@cornell.edu, wz344@cornell.edu

Website(s): <https://cnf.cornell.edu/education/reu/2023>

Primary CNF Tools Used: ABM Contact Aligner, Glen 1000 Resist Strip, SC4500 Electron Beam Evaporator, Xactix Xenon Difluoride Etcher, Bruker Energy-dispersive X-ray Spectrometer (EDS), Zygo 3D Optical Profilometer

## Abstract:

The superconducting niobium nitride ( $\text{NbN}$ ) was successfully integrated epitaxially with the III-nitride heterostructures ( $\text{AlN}$ ,  $\text{GaN}$ , etc.) recently [1]. This new technology opens the possibilities for epitaxial metal/semiconductor Schottky diodes, epitaxial gate junctions for III-nitride transistors as well as all-epitaxial bulk acoustic wave resonators. The metallic epitaxial  $\text{NbN}$  also offers a way to be selectively etched chemically, which allows the lift-off of the epilayers or devices.

The  $\text{NbN}$  system is complex and presents various phases (i.e., beta, delta, epsilon, and gamma) [2]. Here in this work, by taking advantage of the high crystalline quality niobium nitride ( $\text{Nb}_x\text{N}$ ) films grown by molecular-beam epitaxy (MBE), we propose to do a conclusive study to understand the xenon difluoride ( $\text{XeF}_2$ ) undercut etch characteristics of different phases of  $\text{Nb}_x\text{N}$ . This study identifies the prerequisite conditions for the epilayer lift-off with a sacrificial layer of  $\text{Nb}_x\text{N}$ .

## Summary of Research:

The  $\text{Nb}_x\text{N}$  films were epitaxially grown on 2-inch sapphire wafers. The  $\text{Nb}_x\text{N}$  phases used in this study are Delta phase grown at  $600^\circ\text{C}$ , Epsilon phase grown at  $700^\circ\text{C}$ , Gamma phase grown at  $800^\circ\text{C}$ , and Beta phase grown at  $1000^\circ\text{C}$ .

The  $\text{Nb}_x\text{N}$  samples were cut into  $1\text{ cm} \times 1\text{ cm}$  pieces. A total number of 12 samples were used for this research. Photolithography was done to develop a pattern of different sized circular pads ranging from  $12.5\ \mu\text{m}$  to  $200\ \mu\text{m}$  in diameter. To have a better visualization of the etch process, a transparent silicon dioxide ( $\text{SiO}_2$ ) mask was deposited on the samples using an electron beam evaporator.

A  $\text{XeF}_2$  etcher was used for the undercut etch process. Reference [3] indicates that the  $\text{XeF}_2$  undercut etch rate

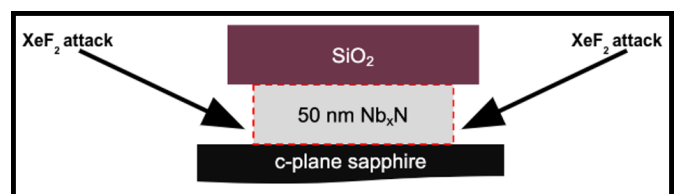


Figure 1: Cross-sectional representation of undercut process.

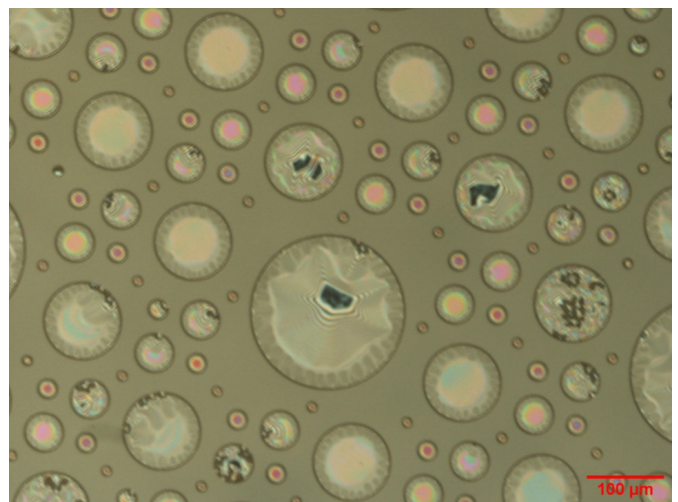


Figure 2: Delta phase niobium nitride after full undercutting at 4 Torr  $\text{XeF}_2$  pressure.

increases with the increase of chamber temperature. The chamber temperature in this work was accordingly set at  $100^\circ\text{C}$ , which is the highest temperature the current etcher can achieve. Each sample for each phase was etched at a different  $\text{XeF}_2$  pressure (4 Torr, 3 Torr and 2 Torr) for understanding the impact of the  $\text{XeF}_2$  partial gas pressure on the undercut etch rate. It was observed that the vertical etching of  $\text{Nb}_x\text{N}$  by  $\text{XeF}_2$  was done effortlessly, i.e., the  $\text{Nb}_x\text{N}$  not covered by  $\text{SiO}_2$  would be etched away after the first etch cycle. The difficulty then was for the  $\text{XeF}_2$  to go under the  $\text{SiO}_2$  membranes

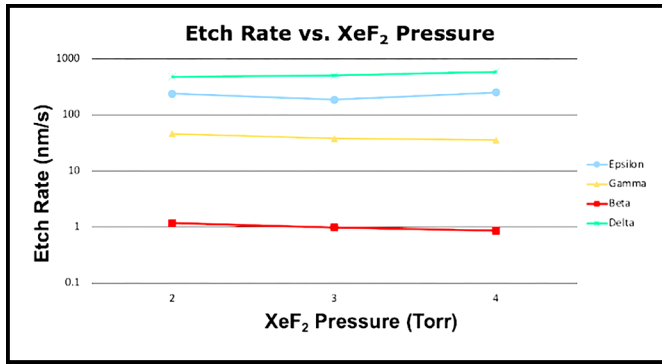


Figure 3: Average etch rates as a function of  $\text{XeF}_2$  pressure for all four phases of  $\text{Nb}_x\text{N}$ .

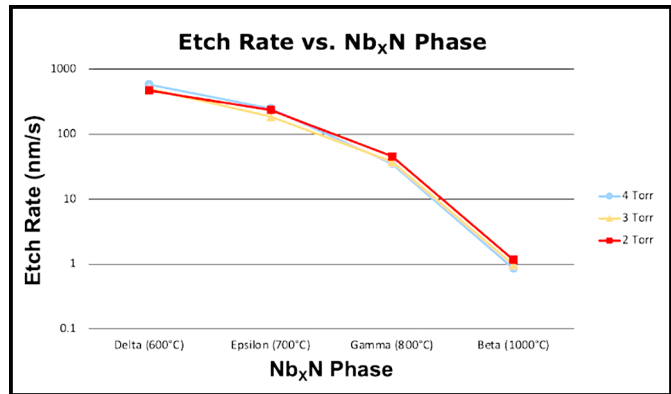


Figure 4: Average etch rates as a function of  $\text{Nb}_x\text{N}$  phase.

as seen in Figure 1 to attack the  $\text{Nb}_x\text{N}$ . This undercut etch will result in the release of the  $\text{SiO}_2$  membranes. All the samples in this study experienced vertical etching of  $\text{Nb}_x\text{N}$ , but not all experienced a complete undercut of  $\text{Nb}_x\text{N}$ .

The samples of Delta phase were fully undercut after 70 seconds as seen in Figure 2. A change in the average etch rate was observed, which was proportional to the change of the  $\text{XeF}_2$  pressure. Similarly, the samples of Epsilon phase were fully undercut after 3-4 minutes. There was no direct correlation of etch rates and  $\text{XeF}_2$  pressure observed for Epsilon phase, but it was noticed that the average etch rates increased overtime.

The undercut etch of Gamma phase  $\text{Nb}_4\text{N}_3$  at 4 Torr and 3 Torr  $\text{XeF}_2$  pressure were not completely released after 12 minutes. Differently, a duality was seen at 2 Torr  $\text{XeF}_2$  pressure, in which case some membranes became close to being fully undercut after 12 minutes. Unfortunately, most membranes became darker over time, implying that the reaction between  $\text{XeF}_2$  and Gamma phase  $\text{Nb}_x\text{N}$  is unstable and unpredictable. The initial average etch rates for Gamma phase  $\text{Nb}_4\text{N}_3$  decreased as the  $\text{XeF}_2$  pressure increase, but overtime the etch rates went to zero, as none of the samples were fully undercut.

No full undercut for any of the samples for Beta phase  $\text{Nb}_2\text{N}$  was seen even after 14 minutes. Similar to Gamma phase, the initial average etch rates for Beta phase  $\text{Nb}_2\text{N}$  decreased as the  $\text{XeF}_2$  pressure increased, but overtime the etch rates went to zero, as none of the samples were fully undercut.

Figure 3 shows how the average etch rate for each phase changes as the  $\text{XeF}_2$  pressure increases. Figure 4 shows how the average etch rates changes as we move through the different  $\text{Nb}_x\text{N}$  phases. It was noticed that  $\text{Nb}_x\text{N}$  films grown at higher MBE temperatures are more difficult to be fully undercut by  $\text{XeF}_2$ .

## Conclusions and Future Steps:

Results from the research show that Delta and Epsilon phase  $\text{Nb}_x\text{N}$  can be fully undercut by  $\text{XeF}_2$ ; Gamma phase  $\text{Nb}_x\text{N}$  does exhibit some  $\text{XeF}_2$  undercut capabilities, but the reaction is unstable and unpredictable; Beta phase  $\text{Nb}_x\text{N}$  cannot be fully undercut by  $\text{XeF}_2$ . In general,  $\text{Nb}_x\text{N}$  phases grown at higher MBE temperatures are more difficult to be fully undercut by  $\text{XeF}_2$ .

This research is a preliminary step to help us understand the required conditions for doing an epitaxial lift-off of high-quality aluminum nitride (AlN) membranes. The next step is a research project titled: "Monolithic integration of acoustic resonators and high electron mobility transistors (HEMTs) utilizing aluminum nitride platform." For this project, we will fabricate acoustic resonators from AlN using Delta or Epsilon phase  $\text{Nb}_x\text{N}$  as a sacrificial layer.

## Acknowledgements:

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## References:

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