

A Systematic Study of How Different Phases of Niobium Nitride (Nb_xN) React to Xenon Difluoride (XeF_2) Undercut Etch

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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 (NbN) was successfully integrated epitaxially with the III-nitride heterostructures (AlN , 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 NbN also offers a way to be selectively etched chemically, which allows the lift-off of the epilayers or devices.

The 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 (Nb_xN) films grown by molecular-beam epitaxy (MBE), we propose to do a conclusive study to understand the xenon difluoride (XeF_2) undercut etch characteristics of different phases of Nb_xN . This study identifies the prerequisite conditions for the epilayer lift-off with a sacrificial layer of Nb_xN .

Summary of Research:

The Nb_xN films were epitaxially grown on 2-inch sapphire wafers. The Nb_xN phases used in this study are Delta phase grown at 600°C , Epsilon phase grown at 700°C , Gamma phase grown at 800°C , and Beta phase grown at 1000°C .

The Nb_xN 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 (SiO_2) mask was deposited on the samples using an electron beam evaporator.

A XeF_2 etcher was used for the undercut etch process. Reference [3] indicates that the XeF_2 undercut etch rate

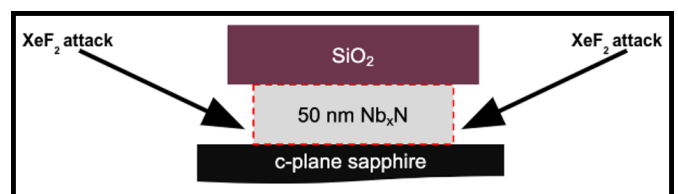


Figure 1: Cross-sectional representation of undercut process.

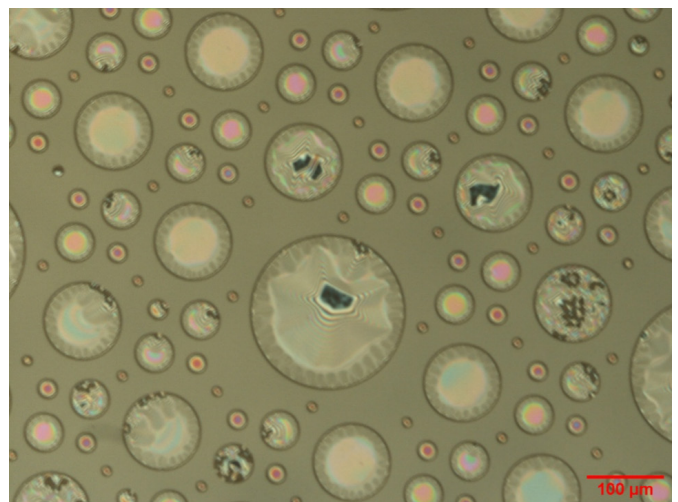


Figure 2: Delta phase niobium nitride after full undercutting at 4 Torr XeF_2 pressure.

increases with the increase of chamber temperature. The chamber temperature in this work was accordingly set at 100°C , which is the highest temperature the current etcher can achieve. Each sample for each phase was etched at a different XeF_2 pressure (4 Torr, 3 Torr and 2 Torr) for understanding the impact of the XeF_2 partial gas pressure on the undercut etch rate. It was observed that the vertical etching of Nb_xN by XeF_2 was done effortlessly, i.e., the Nb_xN not covered by SiO_2 would be etched away after the first etch cycle. The difficulty then was for the XeF_2 to go under the SiO_2 membranes

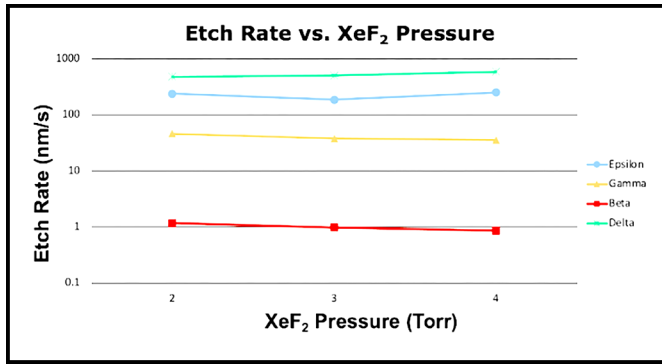


Figure 3: Average etch rates as a function of XeF_2 pressure for all four phases of Nb_xN .

as seen in Figure 1 to attack the Nb_xN . This undercut etch will result in the release of the SiO_2 membranes. All the samples in this study experienced vertical etching of Nb_xN , but not all experienced a complete undercut of Nb_xN .

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 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 XeF_2 pressure observed for Epsilon phase, but it was noticed that the average etch rates increased overtime.

The undercut etch of Gamma phase Nb_4N_3 at 4 Torr and 3 Torr XeF_2 pressure were not completely released after 12 minutes. Differently, a duality was seen at 2 Torr 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 XeF_2 and Gamma phase Nb_xN is unstable and unpredictable. The initial average etch rates for Gamma phase Nb_4N_3 decreased as the 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 Nb_2N was seen even after 14 minutes. Similar to Gamma phase, the initial average etch rates for Beta phase Nb_2N decreased as the 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 XeF_2 pressure increases. Figure 4 shows how the average etch rates changes as we move through the different Nb_xN phases. It was noticed that Nb_xN films grown at higher MBE temperatures are more difficult to be fully undercut by XeF_2 .

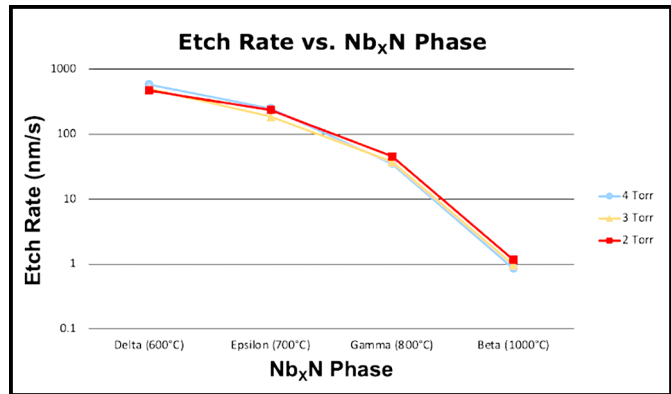


Figure 4: Average etch rates as a function of Nb_xN phase.

Conclusions and Future Steps:

Results from the research show that Delta and Epsilon phase Nb_xN can be fully undercut by XeF_2 ; Gamma phase Nb_xN does exhibit some XeF_2 undercut capabilities, but the reaction is unstable and unpredictable; Beta phase Nb_xN cannot be fully undercut by XeF_2 . In general, Nb_xN phases grown at higher MBE temperatures are more difficult to be fully undercut by 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 Nb_xN as a sacrificial layer.

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