A Systematic Study of How Different Phases of Niobium Nitride (Nb\(_x\)N) 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\(_x\)N) 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\(_x\)N. This study identifies the prerequisite conditions for the epilayer lift-off with a sacrificial layer of Nb\(_x\)N.

Summary of Research:

The Nb\(_x\)N films were epitaxially grown on 2-inch sapphire wafers. The Nb\(_x\)N 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\(_x\)N samples were cut into 1 cm × 1 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 µm to 200 µm 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 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\(_2\) partial gas pressure 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\(_x\)N by XeF\(_2\) was done effortlessly, i.e., the Nb\(_x\)N 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 1: Cross-sectional representation of undercut process.

Figure 2: Delta phase niobium nitride after full undercutting at 4 Torr XeF\(_2\) pressure.
as seen in Figure 1 to attack the Nb\textsubscript{2}N. This undercut etch will result in the release of the SiO\textsubscript{2} membranes. All the samples in this study experienced vertical etching of Nb\textsubscript{2}N, but not all experienced a complete undercut of Nb\textsubscript{2}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 XeF\textsubscript{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\textsubscript{2} pressure observed for Epsilon phase, but it was noticed that the average etch rates increased overtime.

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

**Conclusions and Future Steps:**

Results from the research show that Delta and Epsilon phase Nb\textsubscript{N} can be fully undercut by XeF\textsubscript{2}; Gamma phase Nb\textsubscript{N} does exhibit some XeF\textsubscript{2} undercut capabilities, but the reaction is unstable and unpredictable; Beta phase Nb\textsubscript{N} cannot be fully undercut by XeF\textsubscript{2}. In general, Nb\textsubscript{N} phases grown at higher MBE temperatures are more difficult to be fully undercut by XeF\textsubscript{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\textsubscript{N} as a sacrificial layer.

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**References:**