Etching of Nitrides-NbN Heterostructures

Authors: Nicolas Delpuech (III-V Lab), Nicolas Michel (III-V Lab)

Version: 1.0

Date: 7 September 2022

Project: Niobium

Funding Agency: Agence Nationale de la Recherche (France)

Grant Number: ANR-21-CE08-0037

Data Repository: https://zenodo.org

DOI:

Abstract

Niobium Nitride (NbN) is metallic at room temperature. NbN / III-N heterostructures are of interest for new devices applications such as the Metal Base Transistor (MBT, Patented by III-V Lab) [Delage]. Niobium Nitride etching in fluorine plasma is documented by Reactive Ion Etching (RIE) in [Meng] and also [Guo]. In this document we present the etching of a III-Nitride NbN heterostructure with fluorine chemistry Inductively Coupled Plasma (ICP).

Heterostructure

The bottom layer is 95 nm undoped aluminium nitride (AlN), the middle layer is 10 nm Niobium Nitride (NbN) and the top layer is 10 nm of aluminium gallium nitride (AlGaN).

Equipment

Etches were performed at III-V Lab in a Sentech SI 500 fluorine ICP-RIE system equipped with a Sentech SLI laser interferometer at 670 nm.

Plasma recipe

The fluorine plasma is not the most usual for etching III-N materials because of low rates [Sreenhidi]. In this case, it is chosen for an accurate control of the etch depth, indeed, the aim is to be able to stop inside the NbN layer. In order to decrease, the etch rate in NbN was lowered as much as possible. The optimized plasma parameters are listed in Table 1.

Table 1: Summary of fluorine ICP etching parameters

Parameter	Value	Unit
Chuck Temperature	20	°C
Pressure	8	mT
ICP Power	100	Watt
RIE Power	15 to 130	Watt
CF_4 flow	10	sccm
O_2 flow	0 to 20	sccm
Ar flow	50	sccm

Endpoint Detection and Simulation

The actual EPD signal was recorded on the Sentech SLI interferometer when etching the whole structure (Figure 1). The structure was etched without oxygen with 80 Watt RIE power before 280 seconds etch time and then (after 280 seconds up to 600 seconds) with 130 W to increase the AlN etch rate. Etching of the NbN layer is clearly seen as a drop of the EPD signal at time between 167 to 185 seconds. The metallic nature allows a strong optical contrast in the EPD signal between NbN and the III-Nitrides which are transparent at 670 nm.

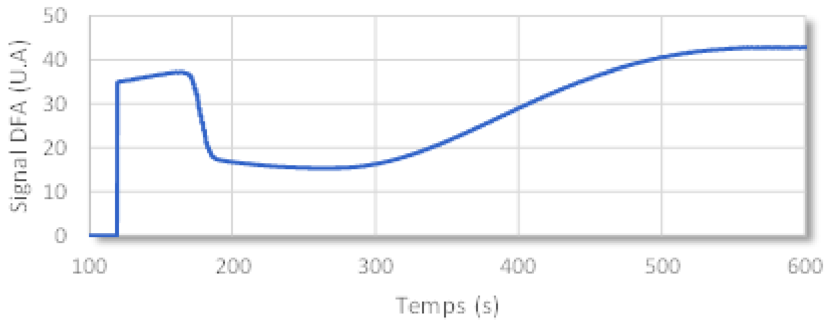

Figure 1: Experimental End Point Detection Signal (EPD). All structures etched in 600 seconds

The EPD signal was simulated using the EMPy simulation code in Python from the University of California Santa Barbara website [EMPy] (Figure 2). The optical index and absorption coefficient of NbN relevant for simulation may be found for instance in [Du]. The curve is reproduced by simulation through the NbN layer between 10 nm and 20 nm depth (Figure 2).

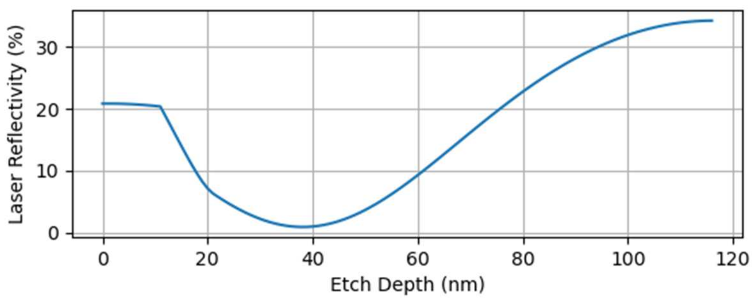

Figure 2: Simulated End Point Detection Signal (EPD)

Based on the same material, it was possible to vary the RIE power and associated bias in order to measure the NbN etch rate at different plasma powers. The etch rate is measured from the EPD signal assuming a 10 nm NbN thickness (Figure 3). By adjusting the bias it was possible to scale the etch rate below 5 nm/min, which allows for a precise control of the etch depth inside the NbN layer.

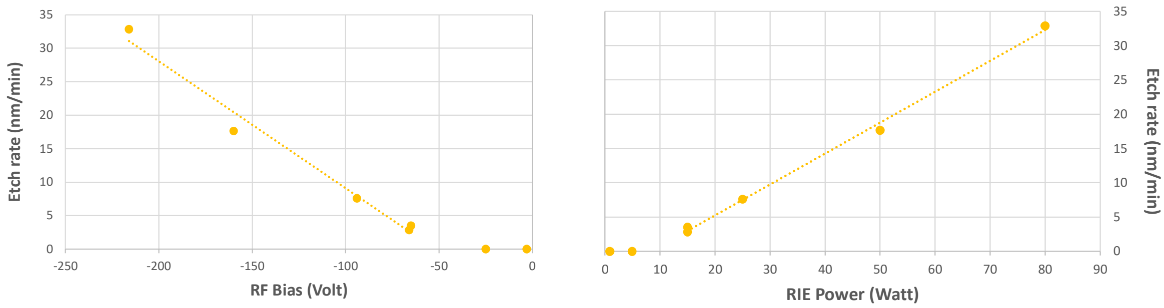

Figure 3: NbN etch rate as a function of RF bias (left) and RF power (right).
CF4/Ar Plasma. Other Parameters in Table 1.

Adding O_2 to the plasma

Adding 20 sccm O_2 to the plasma does not change the above results significantly. However, addition of O_2 advantageously avoids the photoresist etch mask to be burnt by the CF4 and Ar gases during the process and this is important for the subsequent removal of resist mask.

Stopping the etching inside a 10 nm-thick NbN layer

The etch rate can now be controlled when entering the NbN layer and reduced to below 5 nm/min by moving the RIE power from 80 Watt to 15 Watt once the etch penetrates the NbN layer. Using this technique, it is now possible to stop the etching inside the NbN layer, which may be desirable for device applications.

We performed an etch stop inside the NbN layer on the same material. The correct etch stop was validated by electrical short-circuit on the NbN layer exposed to air after etching (Figure 4).

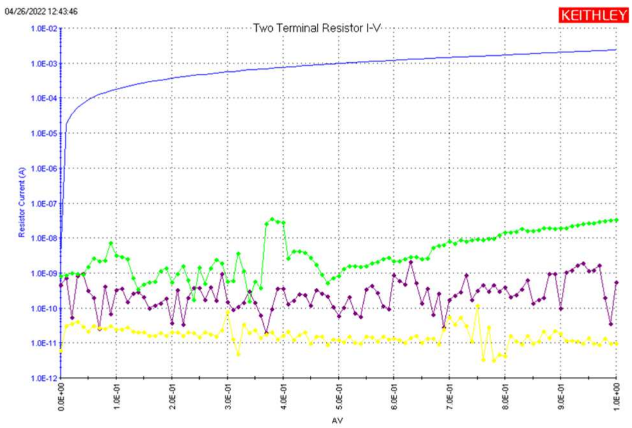

Figure 4: Two-probe electrical measurements on same sample. Green: unetched / Purple: etched until the HR Si substrate / Yellow: probes disconnected / Blue: Etch stop in NbN

Conclusions

In this data repository, we present an NbN and III-Nitride etching technology based on CF_4/Ar and CF_4/Ar/O_2 ICP-RIE plasmas, which is intended for NbN-III-N device manufacturing. The etch depth is controlled by laser interferometry. The strong optical contrast of the metal NbN versus the III-V transparent layers allows the detection of the NbN/nitride interfaces. Adjusting the RF bias allows to slow down the etch rate and therefore controlling of the etch depth. Oxygen incorporation does not significantly impact the results, however it avoids burning of the resins which helps to an easy removal of the masks.

References

[Delage] S. Delage, WO2019020737 – Metal-based transistor comprising materials comprising at least one group III element and one group V element, international filing date: 26.07.2018

[Meng] X.F. Meng,R.S. Amos, A.W. Lichtenberger, R.J. Mattauch, and M.J. Feldnian ; NbN Edge Junction Fabrication : Edge Profile Control by Reactive Ion Etching ; IEEE Transactions on Magnetics, Vol. 25 n°2, pp. 1239-1242, March 1989 ;

[Guo] S. Guo, Q. Chen, D. Pan, Y. Wu, X. Tu, G. He, H. Han, F. Li, X. Jia, Q. Zhao, H. Zhang, X. Bei, J. Xie, L. Zhang, J. Chen, L. Kang, P. Wu ; Fabrication of superconducting niobium nitride nanowire with high aspect ratio for X-ray photon detection ; Nature (2020) 10:9057 | https://doi.org/10.1038/s41598-020-65901-5

[Sreennidhi] T. Sreenidhi, K. Baskar, A. DasGupta, N. DasGupta ; Reactive ion etching of GaN in SF6 + Ar and SF6 + N2 plasma ; Semicond. Sci. Technol. 23 (2008) 125019 (7pp) ; doi:10.1088/0268-1242/23/12/125019

[EMPy] https://wiki.nanotech.ucsb.edu/wiki/Laser_Etch_Monitor_Simulation_in_Python

[Du] X.K. Du, T.M. Wang, C. Wang, B.L. Chen, L. Zhou ; Microstructure and Optical Characterization of Magnetron Sputtered NbN Thin Films ; Chinese Journal of Aeronautics 20(2007) 140-144 ; https://doi.org/10.1016/S1000-9361(07)60021-1

Appendix A

EPD 2767_N1.csv
Supporting Data of Figure 1. Endpoint Detection and Plasma Data. SENTECH Instruments SI Systems Logfile System: SI 500 Lab France [500 182] Reactor: 1 Recipe: NbN_RIE-ICP_CF4_Ar_modifie,rcp Starting time: 26/04/2022 10:28:37

res2t#1@3_Data.csv
res2t#1@3_Data_Append_1.csv
res2t#1@3_Data_Append_2.csv
res2t#1@3_Data_Append_3.csv
res2t#1@3_Data_Settings.csv
Supporting Data of Figure 4. Electrical Measurements after Etch Stop in NbN layer