

Growth and Characterization of NbN/III-N Heterostructures by Molecular Beam Epitaxy

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Primary CNF Tools Used: Veeco Icon AFM, Zeiss SEM, e-beam evaporators, AJA sputter deposition, Oxford 81 etcher, PT770 etcher, JEOL 9500, ABM contact aligner, Autostep i-line stepper

Abstract:

Films of the metallic superconductor niobium nitride (NbN) have been grown on 6H-SiC substrate and GaN substrate. The surface morphology, grain structure, and electronic transport properties of the films are characterized and used to optimize the growth conditions with the goal of fabricating epitaxial heterostructures incorporating NbN and the III-N family of semiconductors.

Summary of Research:

NbN is a metallic type-II superconductor that can crystallize in both cubic and hexagonal crystal structures [1]. It has been demonstrated [2] that NbN can be grown epitaxially on silicon carbide (SiC) and gallium nitride (GaN) substrates by molecular beam epitaxy (MBE). Multilayer structures incorporating NbN, GaN, and AlN have also been produced and used to demonstrate the possibility of semiconductor devices integrating epitaxial superconducting thin films [3].

In this work we have characterized NbN thin films and NbN/GaN heterostructures grown by MBE using atomic force microscopy (AFM), x-ray diffraction (XRD), electronic transport measurements, and electron backscatter diffraction (EBSD) techniques. We demonstrate that by altering growth conditions such as Nb/N ratio, growth rate, and substrate temperature, the surface morphology, lattice parameters, stoichiometry, and superconducting properties of NbN thin films can be controlled. The goal of this project is to enable the controlled growth of NbN/III-N heterostructures. To enable the growth of III-N semiconductor films with sufficiently low defect density that electronic properties are not significantly degraded the growth of NbN films must be optimized to achieve smooth, continuous, and highly crystalline and epitaxial films.

Our MBE system utilizes an electron beam evaporator to provide Nb flux, while standard effusion cells can be used to provide Ga, Al, In, and various dopant materials. The nitrogen is provided by an RF plasma source.

The CNF Veeco Icon AFM has been used to study the surface morphology. With the goal of producing epitaxial heterojunctions with atomically sharp interfaces the surface morphology of the NbN thin films as measured by AFM is considered an important material parameter to optimize. By controlling deposition temperature, growth rate, and Nb/N ratio we have produced NbN films on 6H-SiC and measured RMS roughness below 0.5 nm by AFM over a scan area of $10\ \mu\text{m} \times 10\ \mu\text{m}$, as shown in Figure 1.

NbN crystallizes in many polymorphs. Asymmetric XRD diffraction analysis indicates that the NbN films primarily possess cubic crystal symmetry oriented with the $\langle 111 \rangle$ crystallographic axis aligned to the growth axis. GaN possesses the wurtzite hexagonal structure, and 6H-SiC possesses a similar hexagonal structure, with the $\langle 0001 \rangle$ crystallographic axis aligned to the growth direction in both cases. This means that for both growth on GaN and 6H-SiC, the NbN films have lower order symmetry about the growth axis than does the substrate. Therefore, two orientations of the NbN crystal differing

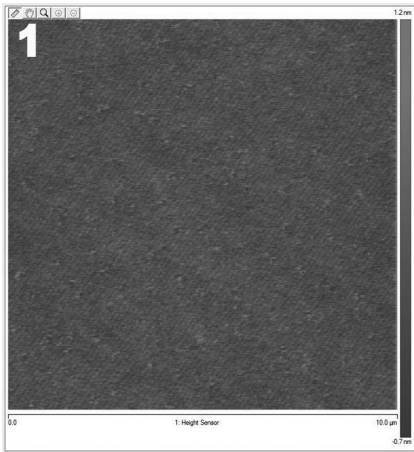


Figure 1: $10\ \mu\text{m} \times 10\ \mu\text{m}$ AFM height scan of a 55 nm thick NbN film grown on 6H-SiC. RMS roughness is measured to be 0.32 nm. Barely visible are parallel stripes that are explained by depressions in the films at the site of boundaries between NbN grains whose crystal orientation differ by a 60° rotation about growth axis.

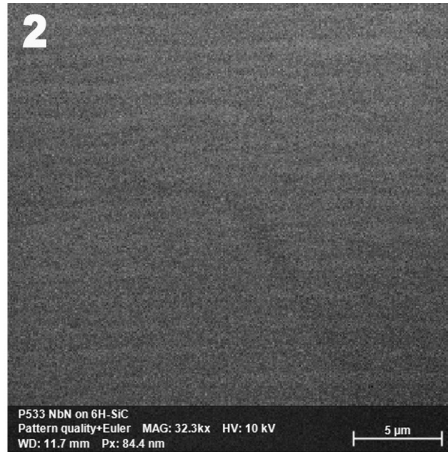


Figure 2: Crystal orientation map of a $25\ \mu\text{m} \times 25\ \mu\text{m}$ area of the surface of an 89 nm NbN film on 6H-SiC produced by EBSD using an SEM with an accelerating voltage of 10kV. The color indicates the relative angle of the crystal structure. The parallel array of grains is found to span the entire $1\ \text{cm} \times 1\ \text{cm}$ sample. This image shows grains that are approximately 750 nm in width. See full color version on pages xxviii-xxix

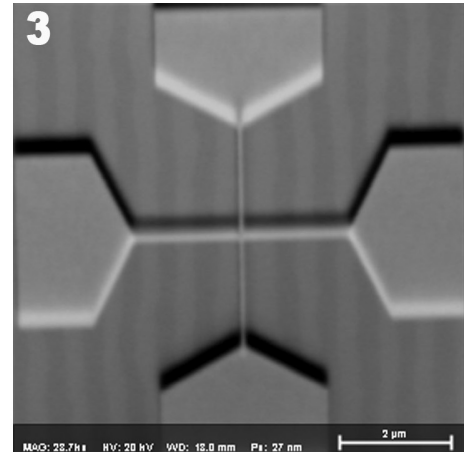


Figure 3: SEM image of a developed resist mask that is used as an etch mask to create structures to test the effect of grain boundaries on the electronic transport properties. The mask, created through EBL, is aligned to the grain structure of the sample. One arm of the cross is parallel to the grains and crosses no grain boundaries; the other arm is perpendicular and crosses several grain boundaries.

by a 60° rotation about the growth axis are equivalent with respect to the substrate crystal structure.

We have found using EBSD techniques to determine the orientation of the NbN crystal about the out of plane direction that the NbN crystallizes with two orientations corresponding to 60° rotation about the out of plane axis. For NbN films on 6H-SiC, we have found that the boundaries between grains of different crystal orientation are parallel and span at least hundreds of microns across the surface of the film, as shown in Figure 2. The width between boundaries has been found to vary, with the largest grains found to be approximately $1\ \mu\text{m}$ wide. No such parallel grain orientations have been observed for NbN films grown on GaN.

In an effort to determine the effect these grain boundaries have on both superconducting and metallic electron transport, a combination of e-beam lithography,

photolithography, and plasma etching were used to etch the NbN film, leaving small crosses of NbN oriented with respect to the grain boundaries, as seen in Figure 3. One arm of the cross lies within a single grain, and the perpendicular arm crosses many grains. Processing and characterization of these structures is ongoing.

References:

- [1] Oya, G., et al., "Transition temperatures and crystal structures of single crystal and polycrystalline NbN films", *Journal of Applied Physics*, 1974 vol: 45 (3) pp: 1389-1397.
- [2] Katzer, D., et al. "Epitaxial metallic β -Nb₃N films grown by MBE on hexagonal SiC substrates", *Applied Physics Express* 8 (2015), no. 8, 085501.
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