Ultra-thin film NbN depositions for HEB heterodyne mixer on Si-substrates

Sascha Krause, Vincent Desmaris, Alexey Pavolotsky, Denis Meledin, Victor Belitsky, Eckhard Pippel

Abstract—**The key of improving hot-electron bolometer (HEB) mixer performance lies inevitably in the quality of ultra-thin NbN films itself. This work presents a thorough investigation of crucial process parameters of NbN films deposited by means of reactive DC-sputtering on Si-substrates at elevated temperatures up to 750°C.**

The polycrystalline NbN films with thickness of 4 to 10nm were characterized by DC resistivity measurements, ellipsometry and high resolution transmission electron microscopy (HRTEM) in order to confirm thickness and film structure. Since the macroscopic properties such as critical temperature, thickness as well as the transition width to the superconducting state are directly linked to HEB mixer noise temperature and IF bandwidth, a set of experiments were conducted to enhance aforementioned properties. We considered deposition temperature, RF biasing, nitrogen and argon partial and total pressure during deposition as major process variable parameters. Careful optimization of the deposition conditions allowed setting up a process resulting in high-quality NbN ultra-thin films with thickness of 5.5nm exhibiting Tc of 10.5K. Moreover, the transition width could be kept as low as 1.4K. The produced films were stored at ambient conditions and re-characterized over a period of 4 month without measurable degradation.

Index Terms—**NbN ultra-thin films, HEB, reactive DC magnetron sputtering**

I. Introduction

THE key technology for low-noise sub-millimeter and THz heterodyne receivers lies in the employment of super-conductive material such as the widely used NbN with critical temperature (Tc) of 16-17K of the bulk [1]. However, this material also exhibits short electron-phonon interaction and phonon escape times [2], which favors its applicability in hot electron bolometers HEB as most sensitive heterodyne receiver above 1.3THz [3].

The use of silicon as a substrate to deposit onto NbN ultra-thin films is still most attractive from a processing point of view, especially for thin membrane-like structures where bulk substrates cannot be employed. The rather large lattice mismatch of NbN to Si hinders the growth of epitaxial ultra-thin NbN films, however, careful optimization of various deposition parameters when deposited at elevated substrate temperatures, yields to high-quality poly-crystalline NbN films as presented in this work. The crystallographic structure and interface of NbN and Si were investigated by means of high resolution transmission electron microscopy (HRTEM) and the super-conducting properties such as Tc and residual resistance ratio (RRR) extracted from resistance-temperature measurements.

II. Experiment

Widely available silicon and silicon-on-insulator (SOI) substrates with (100) – orientation have been used to grow onto ultra-thin NbN films by means of reactive DC magnetron sputtering. The surface of the substrates was ultra-sonically cleaned and pre-sputtered in argon plasma prior the deposition of NbN. Several process parameters such as partial pressure of Ar and N_2, the total pressure, the presence of RF bias and the substrate holder temperature were investigated and their interaction evaluated by R(T) measurements in a calibrated four-point-probe setup. The system used is an AJA ATC Orion 5 DC magnetron sputtering tool, able to maintain a base pressure in the process chamber of $1.8x10^{-8}$mTorr.

The employment of HRTEM confirmed both expected thicknesses and provided structural clarification on an atomic level, important for deducing most suitable deposition parameters.

III. Results and Discussion

The NbN film seen in Fig. 1 was deposited for 60s and served as a reference for deducing the deposition rate of 1.2Å/s. The depicted film is clearly poly-crystalline with differently sized grains. The interface across the native SiO_x and the NbN film appears to be sharp within a few atomic layers, desirable for HEB since the escape of phonons at the interface is not additionally obstructed [4].

Sascha Krause is working as a PhD student in the Group for Advanced Receiver Development at the Earth and Space Science Department at Chalmers University of Technology, Gothenburg, Sweden. (e-mail: sascha.krause@chalmers.se)

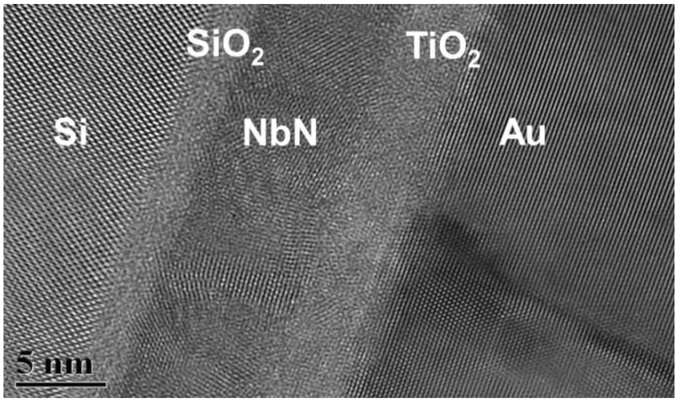

Fig. 1. HRTEM showing the cross-section of the NbN ultra-thin film grown onto Si substrate with native oxide layer. The thickness amounts to approximately 7nm for a deposition time of 60s and appears uniform across the entire specimen.

A. Atmosphere

The ratio of argon and nitrogen combined with a certain sputtering rate of Nb is determining the stoichiometry of the NbN film. Furthermore, it was observed that the optimal N_2/Ar ratio is dependent on the total pressure and decreases with increasing pressure. This behavior was also reported in [5]. Thus, keeping the DC magnetron current constant, yielded to following dependence of films' critical temperature, as illustrated in Fig 2.

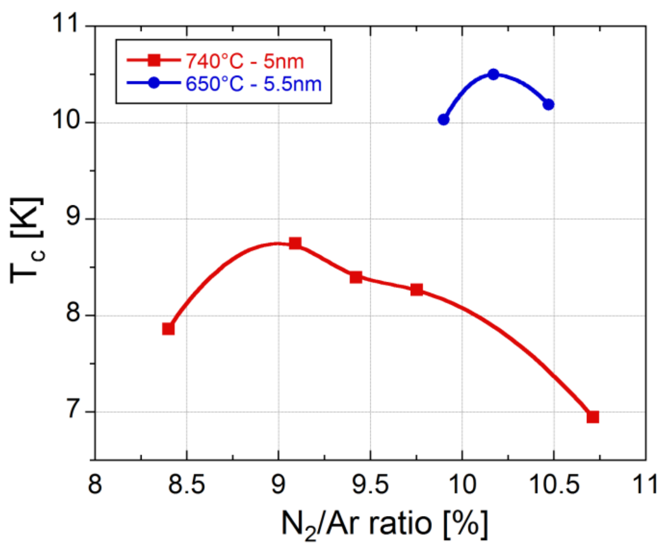

Fig. 2. The stoichiometry of the NbN film is linked to its superconducting properties and quickly degrades apart from the optimal N_2/Ar ratio.

High quality NbN films with Tc as high as 10.5K on SOI substrate exhibit a narrow allowable range of the optimal N_2/Ar ratio and degrade quickly apart from this. The optimal ratio may shift due to deterioration of the Nb target over the course of time.

B. Temperature

Increasing the temperature of the substrate by means of intentional substrate heating is known to increase the mobility of arriving atoms on the surface and enables the arrangement of NbN in a more ordered manner. Thus, the Tc and RRR of 5.5nm ultra-thin NbN films grown under equal conditions, show the dependence, illustrated in Fig. 3 as a function of substrate temperature.

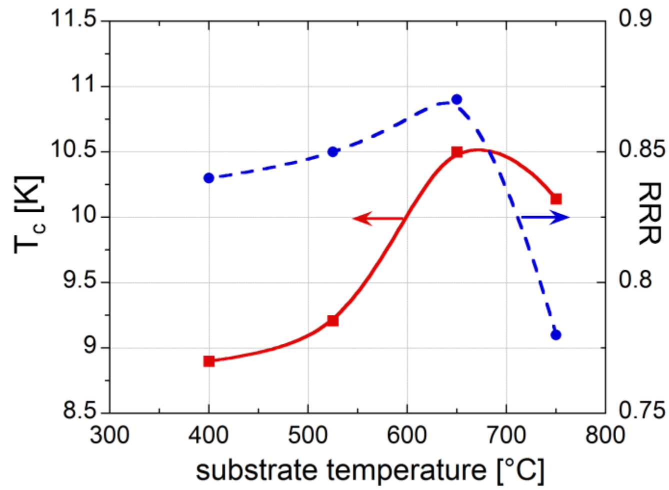

Fig. 3. Tc and RRR as a function of substrate temperature for an optimal chosen N_2/Ar ratio. The thickness is approximately 5.5nm, according to a deposition time of 45s.

The critical temperature gradually increases with rising substrate holder temperature and peaks at approximately 650°C, accompanied with highest RRR of 0.87, which confirms the excellent film quality and low disorder. Apart from this point, both the Tc and RRR degrade quickly for temperatures beyond 750°C. We presume the contamination of the film at those high temperatures due to increasing outgassing.

C. RF bias

The introduction of RF power throughout the sputtering process is motivated by adding surface energy to the film and the increase in mobility of arriving atoms in form of argon ion bombardment. Different experiments were conducted, revealing that the presence of additional RF bias is essential for high Tc films. The absence of RF power degraded films' Tc to 6K compared to 9K under equivalent conditions but applied 4W of RF power. However, no improvement in Tc has been observed, when increasing the RF power even further.

D. Annealing and long time storage

The eventual enhancement of NbN super-conducting properties was investigated by applying rapid thermal annealing (RTA) to electrical characterized films. We observed that the Tc for all annealed films was suppressed by 1K to 3K for annealing times ranging from 10s to 30s and temperatures from 700°C to 950°C.

The effect of storage and aging is an important concern for the practical employment of NbN ultra-thin films. Thus, monthly re-measuring of the Tc over a period of 4 month for samples stored at ambient conditions, showed a scarcely measurable degradation of Tc by approximately 0.1K and similar sheet resistance.

IV. Conclusion

We thoroughly studied the optimization of process parameters to obtain high-quality ultra-thin NbN films by means of reactive DC magnetron sputtering. Fine-tuning the films' stoichiometry by adjusting the N_2/Ar ratio improved the critical temperature by at least 1K compared to initial depositions. Similarly, increase in the substrate temperature gradually promoted the growth of NbN ultra-thin films up to 650°C, followed by subsequent worsening of the Tc beyond 700°C. Furthermore, additional RF power during the sputtering process seems to be essential for achieving high Tc.

The optimization led to high-quality poly-crystalline NbN films with thickness of 5.5nm, exhibiting a Tc of 10.5K on SOI substrates, which can be employed in high-performance HEB heterodyne receiver utilizing a Si fabrication process with vast integration possibilities.

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