Influence of substrates and buffer layers on the quality of NbN ultra thin film for THz HEB

B. Guillet, V. Drakinskiy, R. Gunnarsson, O. Arthursson, L. Méchin, S. Cherednichenko, Y. Delorme, J.M. Krieg

Abstract— In order to improve the crystalline quality of NbN ultra thin film for THz HEB applications, several buffer layers have been selected and investigated. The influence of the buffer layers on thermal boundary resistance of membrane-type Hot Electron Bolometer (HEB) devices and on their IF bandwidth is discussed. The influence of substrates and buffer layers on the quality of NbN ultra thin film has been studied by performing Atomic Force Microscopy (AFM) and low reflectometry measurements on NbN films on different substrates (3 μm SOI substrate and MgO buffered 3 μm SOI substrate). In particular, the physical properties (roughness and thickness) of NbN film layers have been carefully measured.

I. INTRODUCTION

NbN Hot Electron Bolometer (HEB) mixers are the device of choice for low noise heterodyne receivers for future astronomical and Earth’s science space missions for the frequency range above 1 THz. Currently, the mixer noise temperature is approximately 10 to 15 times higher than the quantum limit \( (hν/k_B) = 48 \text{K}/\text{THz} \), where \( h \) is the Planck constant, \( ν \) is the frequency, \( k_B \) is the Boltzmann constant \([1,2,3,4]\) and the IF gain bandwidth on bulk substrate is up to 4.5 GHz \([1,5,6]\).

The goal and motivations of this work is to fabricate a multipixel heterodyne receiver for 2.5 THz based on NbN superconducting hot-electron bolometer (HEB) mixers with quasi-optical design. The main membrane advantages are related to the RF and LO coupling efficiency. A 16 pixel heterodyne camera has already been built, which will be operated at 2.5 and 4.7 THz for deuterated hydrogen (HD) and neutral atomic oxygen (OI) lines observations. Nevertheless these values are sufficient for some radioastronomy applications.

The performances of NbN HEB mixer rely very much on the substrate the HEB is placed on (in contrast to SIS and Schottky mixers). As the result of the finite electron relaxation rate, the 3 dB HEB mixer gain bandwidth is limited by \( f_{\text{lim}} = (2\pi\tau_{\text{mix}})^{-1} \), where \( f_{\text{lim}} \) is the finite electron relaxation rate and \( \tau_{\text{mix}} \) is the electron-phonon interaction time.

The gain bandwidth of NbN mixers is related to the « intrinsic » properties of the NbN material (the temperature derivative of the resistance, the electron-phonon interaction time, the electron and phonon thermal capacitances and the speed of sound), to the substrate (the film/substrate phonon transmission) and to the measurements setup (bias current, IF load resistance, etc.) \([8]\).

Since the electron-phonon interaction time in NbN films is very short (~12 ps \([9]\), the limiting parameter of the hot electrons relaxation rate \( \tau_{\text{mix}} \) is the phonon escaping time (from the NbN film into the substrate). Figure 1 shows state of the art of the IF bandwidth using NbN mixers. Assuming that all these devices have the same thickness (expected thickness is 3.5 nm), the mixer gain bandwidth is related to the substrate or the buffer layer used. Most of the publications deal with HEB on bulk substrates but there are a few reports of how the HEB gain bandwidth is affected by replacing a bulk substrate by a thin membrane \([7,10,11]\). HEB devices (with 600 GHz design) have been fabricated on 1.4 μm thick Si₃N₄/SiO₂ stress-less membrane or Si₃N₄/SiO₂/bulk-Si. The difference of gain bandwidth measured between these two types of devices was not significant and was in the 0.6-0.9 GHz range \([7,12]\). These values are narrower than for NbN on bulk-Si \([13]\). The reduction of the gain bandwidth compared to bulk Si substrate is probably due to the material on which NbN is grown (Si₃N₄/SiO₂), and not to the membrane effect \([12]\). Nevertheless these values are sufficient for some radioastronomy applications.

Since a membrane is needed to reduce losses but it may be detrimental for epitaxial growth of NbN, there are two ways to increase the IF bandwidth of HEB devices. The first method is to put the HEB outside the membrane and place it on a bulk silicon surface by etching the Si₃N₄/SiO₂ buffer layer. Preliminary study has shown that a NbN film with a high Tc can be deposited on silicon surface after Si₃N₄/SiO₂ etching. Moreover a careful study of the interconnexions between the different devices must be performed.

This work was supported by ESA contract 16940, by the European Community's sixth Framework Program under RadioNet R113CT 2003 5058187, and by the MC2 ACCESS contract 026029.

B. Guillet was with LERMA Observatoire de Paris, 61 Avenue de l’Observatoire, 75014 Paris, France. He is now with GREYC (UMR 6072), ENSICAEN & Université de Caen Basse Normandie, 6 Bd Maréchal Juin, 14050 Caen, France. Corresponding author, phone: 0033231452693 fax: 0033231452698; E-mail: bguillet@greyc.ensicaen.fr.

V. Drakinskiy, R. Gunnarsson, O. Arthursson, and S. Cherednichenko are with Microtechnology and nanoscience (MC2), Chalmers University of Technology, 412 96 Göteborg, Sweden.

L. Méchin is with GREYC (UMR 6072), ENSICAEN & Université de Caen Basse Normandie, 6 Bd Maréchal Juin, 14050 Caen, France.

Y. Delorme and JM Krieg are with LERMA Observatoire de Paris, 61 Avenue de l’Observatoire, 75014 Paris, France.
antenna on the membrane and the deported HEB mixer has to be done. The second way is to choose the “right material” for the membrane fabrication and NbN deposition. This technique is required in order to obtain epitaxial NbN thin films. Epitaxial thin films could exhibit similar properties as bulk material. So far it has not been thoroughly studied which buffer layers that are most suitable to get such films. The purpose of this work is to study the influence of various layers on NbN properties.

This paper presents some results of this « buffer layer approach ». Five different structures have been studied: NbN/Si3N4/SiO2/Si, NbN/MgO/Si3N4/SiO2/Si, NbN/SOI, NbN/MgO/SOI and NbN/MgO/Si. The NbN films on these different structures have been deposited by dc reactive magnetron sputtering at Moscow State Pedagogical University.

II. STRUCTURAL CHARACTERIZATION OF NbN FILMS

The choice of the most appropriate substrate for achieving the epitaxial growth of NbN is governed by several criteria [14]: (i) the lattice parameter mismatch between NbN and the substrate (or the buffer layer) has to be low (amorphous material is then prohibited); (ii) the NbN material and the substrate (or the buffer layer) have to be chemically inert; (iii) an ideal substrate would have a flat dense surface and be free of twins and other structural inhomogeneities; and (iv) the thermal expansion mismatch between NbN and the substrate (or the buffer layer) has to be low. Moreover the pressure and the substrate temperature during deposition have to be properly chosen in order to satisfy thermodynamical conditions during growth.

According to Table I, the most suitable material are 3C-SiC, Al2O3 and MgO. Their lattice mismatch with NbN material is approximately zero. Si3N4/SiO2 buffer layer is not the most suitable substrate for achieving the epitaxial growth of NbN. The Si3N4 layer described here and used for the previous realisation of NbN HEB devices on membrane was amorphous. It is possible that the reduced IF bandwidth measured in NbN/Si3N4/SiO2 devices was related to reduced quality of the NbN films.

Samples have been studied by x ray diffraction (XRD) in grazing 2θ configuration so that the signals from the substrate is minimized compare to those of the ultra thin film. Figure 2 shows the results for NbN/SOI and NbN/MgO/SOI. In the latter case all possible MgO orientations are observed indicating a polycrystalline MgO buffer layer. This was expected since the MgO layer was deposited at room temperature. In both cases reflections from the ultra thin NbN films are weak and no preferential orientation could be established, thus revealing the polycrystalline structure of NbN. The peaks are also quite wide compared to reflections from epitaxial layers.

III. THICKNESS MEASUREMENTS

The thicknesses have been investigated with a spectroscopical ellipsometer (from J.A. Woollam Co.), with x ray reflectometry, or with high-resolution transmission electron microscopy (HRTEM). The details of the modelling of the studied structures will be published seperately. The NbN exhibits a metallic behaviour in ellipsometry terms. The analysis also gives that the optical constants of the NbN are slightly different in the different samples, which may indicate that the stoichiometry of NbN could vary between the samples.
The SOI substrate exhibits a 3 mm silicon device layer (high resistivity), a buried oxide (BOX) 500 nm ± 5% thick and a 520 μm thick handle silicon wafer. Thin low stress Si3N4/SiO2 membranes could be obtained with 0.6 μm thick Si3N4 buffer layer and 0.8 μm thick SiO2 buffer layer.

As shown in Table II, the thicknesses from ellipsometry measurements are quite consistent with the substrate specifications. An important fact is that the measured NbN thickness is larger than expected for all samples. This thickness is between 5 to 10 nm and seems to be substrate dependent. Electrical properties of the NbN films and therefore of the devices are related to the quality and the thickness of the NbN film. For example, it is known that for a given substrate the critical temperature decreases with the thickness of the NbN film (thinner film gives lower critical temperature) [5]. Therefore, thickness measurements have to be considered important for the further development on devices based on such ultra thin films. Previously Gao et al. using HRTEM found a thicker NbN film than expected from deposition on bulk Si and on 3C-SiC buffer layer [15]. Moreover, ellipsometry revealed the presence of native silicon dioxide. A quite thick oxide (6.7 nm) has been measured on top of the SOI structure. One reason why this layer is much thicker than expected (1 to 2 nm usually) could be linked to the NbN deposition process that oxidises the Si surface. The vacuum is never perfect (oxygen remains) and the sample surface heats up to 800 °C during the deposition process. HRTEM measurement has to be done to check the thickness of the native silicon dioxide.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Expected structure</th>
<th>Measured structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbN/ SOI</td>
<td>NbN 3.5 nm ± 5%</td>
<td>NbN 5.9 ± 0.4 nm</td>
</tr>
<tr>
<td></td>
<td>SiO2</td>
<td>SiO2 6.7 ± 0.7 nm</td>
</tr>
<tr>
<td>SiO2 (BOX)</td>
<td>500 nm ± 5% SiO2</td>
<td>489.9 ± 1.1 nm</td>
</tr>
<tr>
<td>Bulk-Si</td>
<td>520 μm</td>
<td>Bulk-Si</td>
</tr>
<tr>
<td>NbN/MgO/ Si</td>
<td>NbN 3.5 nm ± 5%</td>
<td>NbN 7.8 ± 0.1 nm</td>
</tr>
<tr>
<td></td>
<td>MgO 200 nm</td>
<td>MgO 206.7 ± 0.5 nm</td>
</tr>
<tr>
<td>Bulk-Si</td>
<td>520 μm</td>
<td>Bulk-Si</td>
</tr>
<tr>
<td>NbN/Si3N4/SiO2/Si</td>
<td>NbN 3.5 nm ± 5%</td>
<td>NbN 10.1 ± 0.1 nm</td>
</tr>
<tr>
<td></td>
<td>Si3N4 600 nm ± 5%</td>
<td>Si3N4 602 ± 1 nm</td>
</tr>
<tr>
<td></td>
<td>SiO2 800 nm ± 5% SiO2</td>
<td>784.9 ± 1.1 nm</td>
</tr>
<tr>
<td>Bulk-Si</td>
<td>520 μm</td>
<td>Bulk-Si</td>
</tr>
</tbody>
</table>

TABLE III
PHYSICAL AND ELECTRICAL PROPERTIES OF DIFFERENT NbN SAMPLES

<table>
<thead>
<tr>
<th>NbN (3.5 nm expected)</th>
<th>Thickness [nm]</th>
<th>Roughness [Å] (2×2 μm²)</th>
<th>R300K [Ω]</th>
<th>Tc [K]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbN on Si3N4/SiO2/Si</td>
<td>10.1 ± 0.1</td>
<td>&lt;9</td>
<td>660-700</td>
<td>8.3</td>
</tr>
<tr>
<td>NbN on MgO/Si3N4/SiO2/Si</td>
<td>~5-7</td>
<td>440</td>
<td>11.1</td>
<td></td>
</tr>
<tr>
<td>NbN on SOI</td>
<td>5.9 ± 0.4</td>
<td>3 to 9</td>
<td>450-555</td>
<td>9.5</td>
</tr>
<tr>
<td>NbN on MgO/Si</td>
<td>~7</td>
<td>~5</td>
<td>500</td>
<td>10.2</td>
</tr>
<tr>
<td>NbN on MgO/3C-SiC</td>
<td>7.8 ± 0.1</td>
<td>250</td>
<td>13.0</td>
<td></td>
</tr>
</tbody>
</table>

aAll the samples are polycrystalline.
bMethod used: ellipsometry, HRTEM or x ray measurements

The SOI substrate exhibits a 3 mm silicon device layer (high resistivity), a buried oxide (BOX) 500 nm ± 5% thick and a 520 μm thick handle silicon wafer. Thin low stress Si3N4/SiO2 membranes could be obtained with 0.6 μm thick Si3N4 buffer layer and 0.8 μm thick SiO2 buffer layer.

As shown in Table II, the thicknesses from ellipsometry measurements are quite consistent with the substrate specifications. An important fact is that the measured NbN thickness is larger than expected for all samples. This thickness is between 5 to 10 nm and seems to be substrate dependent. Electrical properties of the NbN films and therefore of the devices are related to the quality and the thickness of the NbN film. For example, it is known that for a given substrate the critical temperature decreases with the thickness of the NbN film (thinner film gives lower critical temperature) [5]. Therefore, thickness measurements have to be considered important for the further development on devices based on such ultra thin films. Previously Gao et al. using HRTEM found a thicker NbN film than expected from deposition on bulk Si and on 3C-SiC buffer layer [15]. Moreover, ellipsometry revealed the presence of native silicon dioxide. A quite thick oxide (6.7 nm) has been measured on top of the SOI structure. One reason why this layer is much thicker than expected (1 to 2 nm usually) could be linked to the NbN deposition process that oxidises the Si surface. The vacuum is never perfect (oxygen remains) and the sample surface heats up to 800 °C during the deposition process. HRTEM measurement has to be done to check the thickness of the native silicon dioxide.

IV. SURFACE MORPHOLOGY

AFM measurements have been made on the films. The root mean square (rms) roughnesses are quite large compared to the NbN film thickness (Table III). The surface morphology is not homogeneous on a 5×5 μm² scale, which could cause problem for the HEB fabrication (cf. figures 3 and 4).

Table III gives the summary of the physical and electrical properties of the NbN films. The sheet resistance value at room temperature and the critical temperature for each sample are given. All the films are polycrystalline (from XRD and HRTEM analysis). Assuming that the NbN thickness is similar (around 6-7 nm), the MgO buffered bulk-Si seems to be the more promising: it gives a low sheet resistance and a high critical temperature.

Fig. 3. AFM image of NbN/ SOI sample: the associated rms roughness is 3 Å (5×5 μm² area).

Fig. 4. AFM image of NbN/MgO/ SOI sample: the associated rms roughness is 5 Å (5×5 μm² area).
V. CONCLUSION

Physical and electrical properties of NbN ultra thin films with different buffer layers and different substrates have been measured and studied. We have demonstrated that several physical and electrical parameters of the NbN film depend strongly on the choice of buffer layers and substrates. Thickness and roughness seem to be the most sensitive parameters. Meanwhile, the thickness measurements on devices based on such ultra thin films have to be considered very carefully. There are still big technological challenges to achieve ultimate NbN ultra thin films and devices based on them.

ACKNOWLEDGMENT

The authors thank KOSMA for providing SOI wafer, SIFCOM at ENSICAEN for x-ray measurement and Oriel GmbH for help in ellipsometry analysis.

REFERENCES