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Growth and characterization of epitaxial ultra-thin NbN films on 3C-SiC/Si substrate for terahertz applications

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Abstract

We report on electrical properties and microstructure of epitaxial thin NbN films grown on 3C-SiC/Si substrates by means of reactive magnetron sputtering. A complete epitaxial growth at the NbN/3C-SiC interface has been confirmed by means of high resolution transmission electron microscopy (HRTEM) along with x-ray diffractometry (XRD). Resistivity measurements of the films have shown that the superconducting transition onset temperature ($T_C$) for the best specimen is 11.8 K. Using these epitaxial NbN films, we have fabricated submicron-size hot-electron bolometer (HEB) devices on 3C-SiC/Si substrate and performed their complete DC characterization. The observed critical temperature $T_C = 11.3$ K and critical current density of about 2.5 $\text{MA cm}^{-2}$ at 4.2 K of the submicron-size bridges were uniform across the sample. This suggests that the deposited NbN films possess the necessary homogeneity to sustain reliable hot-electron bolometer device fabrication for THz mixer applications.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Hot-electron bolometer (HEB) mixers based on superconducting ultra-thin NbN films are largely used for THz spectroscopy for space [1] and ground-based [2–4] observations. To date, the state-of-the-art HEB mixers are used for the frequency range of 1.2–5.2 THz [1, 5–7] with the noise performance ranging from 10 to 15 times the quantum noise at the respective frequency.

The intermediate frequency (IF) gain bandwidth for HEB mixers is determined by their ability to cool down quickly. The currently used phonon-cooled HEB mixers are vastly dependent on the heat transfer rate between the superconducting film and the substrate. The material parameters such as the thickness ($d$) of the superconducting film, and its acoustic match ($\alpha$) with the substrate largely define this process [8]. The critical temperature ($T_C$) is important to ensure fast cooling of hot electrons through interactions with phonons. A variety of available single crystal substrates such as quartz, silicon, sapphire and MgO are employed with the NbN films when it comes to HEB mixers.

Crystal quartz is usually the preferred choice when it comes to waveguide THz mixers because of its low dielectric permittivity and RF loss. However, HEBs on quartz substrates suffer from relatively narrow IF gain bandwidth of about 2 GHz [9], which could be insufficient for, e.g., radio astronomy applications. Silicon material is also an attractive substrate candidate due to its low loss at THz frequencies and the maturity of the processing technology. Nevertheless, the IF
bandwidth is typically measured to be 3–4 GHz [10]. Cubic MgO substrate demonstrates a good lattice match with B1-NbN, which results in an IF bandwidth of 5.2 GHz with a MgO buffer layer on Si [11] and 3.7 GHz with a MgO buffer layer on crystal quartz [12], respectively. Still, the relatively large dielectric constant \( \varepsilon_r = 9.6 \), hygroscopic properties [13] and sensitivity to acids and alkaline solutions on its surface [14] hinder the usage of MgO as an ultimate substrate material for THz waveguide mixer applications. Sapphire is also known to be a good substrate material for epitaxial growth of NbN [15]. However, its relatively high dielectric constant \( \varepsilon_r = 9.3–11.5 \) and processing challenges compared to those of quartz are of major concern for waveguide HEB mixers.

Another possible solution to further increase the IF bandwidth and overcome the existing problems is to use a monocrystalline 3C-SiC substrate [16], thus providing the necessary lattice match (\( \sim 1\% \)) and, hence, proper conditions for epitaxial growth of NbN thin films. Earlier, NbN deposition over 3C-SiC has been reported [14, 17]. However, no attempts have been made either to fabricate practical HEB devices out of NbN ultra-thin films prepared over 3C-SiC substrate, or to systematically study the device electrical properties.

In this paper, we present the results of material characterization of epitaxially grown ultra-thin NbN films on 3C-SiC/Si substrates, deposited by means of DC magneton reactive sputtering. Results of complete DC characterization of fabricated NbN HEB devices with various bridge sizes on 3C-SiC/Si substrate are reported. These results provide evidence of the deposited NbN film uniformity, which makes it useful for fabrication of practical devices for THz electronics, e.g., hot-electron bolometers.

2. Experiment

2.1. Fabrication

Single crystal 3C-SiC films were grown in a hot-wall chemical vapor deposition reactor (CVD) [18] on 100 mm diameter p-type boron-doped (100) Si wafers. Compared to the cold-wall reactor employed in [17], the concept of a hot-wall reactor provides a higher heating- and cracking-efficiency of the hydrocarbons [19]. The carrier gas was hydrogen purified through heated palladium cells mixed with 2% of Ar. Silane (SiH\(_4\)) and propane (C\(_3\)H\(_8\)) were used as precursors. The Si/H\(_2\) ratio was typically 0.024% and the C/Si ratio 1. The growth temperature was always 1350°C and the pressure 300 mbar. The low pressure allows us to achieve a high quality epilayer with low background level of impurities and excellent uniformity compared to atmospheric pressure growth, as in [17], since higher flow velocity is obtained. In addition, a carbonization layer prior to the 3C growth was made using maximum propane flow, allowed by the system through the susceptor, before and during the temperature ramp up. The 3C-SiC films were undoped and had a thickness of about 2 \( \mu \)m.

The NbN films were grown on preheated (\( \sim 800^\circ C \)) 3C-SiC/Si substrates by means of reactive DC magnetron sputtering in an Ar/N\(_2\) gas mixture using a Nb target with a diameter of 2 inches. Prior to loading in the sputtering system, the substrates were chemically cleaned from organic contaminants in a 5:1:1 mixture of deionized water, NH\(_4\)OH and H\(_2\)O\(_2\). The native oxides were treated by means of buffered oxide etch (BOE) solution. The background pressure prior to deposition was 2.7 \( \times 10^{-6} \) Pa. During the reactive sputtering the Ar/N\(_2\) gas mixture with 10:1 flow ratio was used at a total pressure of 0.68 Pa. The DC magnetron current to the target was set to 0.5 A and the resulting deposition rate for the NbN film was 75 \( \AA \) min\(^{-1}\). The deposition rate was verified by film thickness measurement via transmission electron microscopy (TEM) and x-ray diffractometry on satellite NbN/Si specimens.

The HEB devices were fabricated using a 12 \( \times 6 \) mm\(^2\) 3C-SiC/Si substrate, on which NbN film was deposited. The bolometer length was defined by the separation between its contact pads, in our case ranging from 100 to 400 nm; the pads were defined by electron-beam lithography (EBL) using a bilayer PMMA/copolymer resist system. Ti/Au (3/30 nm) contacts were then evaporated followed by a lift-off. Subsequent EBL and DC magnetron sputtering steps were used to pattern the Nb/Pd lines together with the DC contact pads. The NbN bridge width (800 nm) between the contact pads was defined by a negative e-beam resist etch mask and further reactive etching in CF\(_4\)/O\(_2\) gas mixture. The negative resist mask was left on top of the HEB bridge as a protection against degradation factors.

2.2. Characterization

In order to determine the microstructure, the crystallographic orientation and the composition of the layer system NbN/3C-SiC, including the interface region, an aberration corrected (Cs) Titan 80–300 high resolution transmission electron microscope (HRTEM) equipped with a scanning module, a high angle annular dark field (HAADF) detector and an energy dispersive x-ray detector (EDX) was used.

X-ray diffraction (XRD) measurements were utilized for verification of the epitaxial relation between the lattice parameters of the NbN film and the 3C-SiC substrate. For these purposes, a Phillips X’Pert diffractometer with Cu Ka source (\( \lambda_{Cu} = 1.540598 \) \( \AA \)) operated at 40 mA and 40 kV was employed. For two-dimensional mapping, we have used a Ge(220) four-crystal monochromator with crossed slits as primary optics, while for secondary optics, a triple axis module was applied. For x-ray reflectometry measurements of the film thickness, we used line focus and a thin film collimator module with a 1/16\(^\circ\) fixed slit as secondary optics.

Measurements of the temperature dependence of the deposited NbN films’ resistivity, as well as current–voltage characteristics of the fabricated HEB devices were made through a standard four-probe technique in an LHe dewar. The samples were glued on a fiberglass fixture and the individual HEB devices were connected through wire bonding. The temperature during the DC measurements was read from a calibrated Si-diode temperature sensor.
3. Results and discussion

3.1. Structure of the NbN/3C-SiC interface

High resolution transmission electron microscopy (HRTEM) cross-section images of a NbN sample grown on 3C-SiC substrate confirmed the complete epitaxial nature of the deposited films. The HRTEM presented in figure 1 evidences the crystalline quality of the 5 nm thick NbN film (cf the included diffraction pattern, figure 1). Along the NbN/3C-SiC interface, one could though notice a partially amorphous interlayer, with a thickness of about three atomic distances. We suggest that this is due to applied RF sputter-cleaning with argon ions prior to deposition, which might have caused radiation damage to the 3C-SiC substrate. However, optimization of the plasma cleaning conditions may eliminate this problem. It is worth mentioning that although causing some stacking faults in the NbN layer, the amorphous interlayer did not preclude the epitaxial growth of NbN. The reason for this behavior is the fact that not all of the interface in question is amorphous, as in certain parts a direct contact between crystalline NbN and 3C-SiC is made, enabling the observed epitaxial growth. The thickness of the film was also confirmed to be 5 nm, which was expected from our deposition rate calibration.

The NbN films have been also examined by x-ray diffraction (figure 2). In comparison with the XRD results presented in [14, 17], where relatively thick films were studied, we present the XRD data obtained on the same 5 nm thick NbN studied by HRTEM and with \( T_c \) as further shown in figure 3. Figure 2(a) shows the XRD pattern of a 3C-SiC film deposited on a Si substrate with (100) orientation. The broad 3C-SiC substrate peak indicates that this layer has a wide range of lattice parameters. An XRD area scan of the NbN and the underlying 3C-SiC layer is shown in figure 2(b). The broadening of the contours in \( 2\theta \), just as in the single scan of figure 2(a), points towards the large variation of the lattice parameter of the substrate. Note that both the substrate and the NbN layer diffraction peaks of the (200) reflection have not been resolved as they virtually occupy the same location at about 41.4°, corresponding to a lattice parameter...
Figure 3. Critical temperature for an ultra-thin NbN film grown on a 3C-SiC/Si substrate.

Figure 4. Measurements of the normal-state resistances at 20 K as a function of the bridge length. The dashed line represents the best linear fit to the measured values. The error bars represent the standard error of the measured data.

Figure 5. Dependence of the critical temperature and critical current on the bridge length. The error bars represent the standard error of the measured data.

$a = 4.3584 \text{ Å}$, indicating that on average this film is strained to the 3C-SiC. Moreover, the overlapping of the peaks agrees with the diffraction patterns presented in figure 1. These data largely coincide with the results presented in [17].

3.2. Electrical and superconducting properties of NbN/3C-SiC HEBs

The measured temperature dependence of the resistivity of the unpatterned ultra-thin NbN film with the structural properties described above is shown in figure 3. The critical temperature for the presented unpatterned ultra-thin NbN film on 3C-SiC/Si substrate, estimated from the middle of the transition (10%–90%), corresponds to 11.8 K, which is similar to the $T_c$ of the NbN film presented in [14]. We observed a transition width ($\Delta T_c$) of 0.8 K for our NbN film, which is about 0.3 K narrower than that estimated from figure 3 from [17]. We attribute the sharper transition to the enhanced structural properties of the NbN film which could be a consequence of the different 3C-SiC growth method, produced by the hot-wall CVD technique. The narrower superconducting transition width directly improves the conversion efficiency and noise performance of the HEB mixer [20].

In order to assess the properties of the epitaxially grown ultra-thin NbN films on 3C-SiC for further possible use for HEB devices, a number of HEB structures with various bridge lengths have been fabricated.

Further, we discuss the DC properties that can be used as performance indicators to verify the quality and uniformity of the film. We have found that the fabricated devices provide consistently similar DC characteristics, which proves that our analysis is representative. Figure 4 shows the average resistance at 20 K of 24 measured devices in this batch, as for each bridge length up to seven devices were measured. The measured normal-state resistance takes into account not only the resistance of the NbN film between the contact pads but also includes the contribution of the resistance at the NbN and Ti/Au interface. Commonly, the Ti/Au contact pads are formed ex situ and without additional cleaning of the NbN film prior to evaporation. This largely defines the contact resistance between the NbN film and the Ti/Au contact pads [21–23]. Additionally, due to a noticeable conductivity of the 3C-SiC/Si substrate, the film resistivity at room temperature is difficult to estimate. Therefore, the measurements were performed at 20 K where the conductivity of the substrate is considered negligible due to carrier freeze-out. The measured bridge resistance ($R$) at 20 K scales linearly with the device length ($l$), figure 4. The total bridge resistance according to the transmission line model (TLM) [24] is $R = 2R_C + R_{\square}l/w$, where $w$ is the bridge width, $R_{\square} = 500 \pm 50 \Omega$/square is the film sheet resistance and $R_C = 50 \pm 5 \Omega$ is the contact resistance. We assume the $R_C$ remains the same for all bridge lengths by keeping the same device geometry and applying the same processing technique.

Figure 5 displays the measured critical current and critical temperature data. We observed that independently of the bridge length, the critical current values were in the range from
100 to 120 μA. Similarly, the critical temperature values for the processed bridges were about 11.3 K and independent of their length. This confirms the uniformity of the produced ultra-thin NbN film. The measured values of the critical temperature for the patterned HEB structures are close to the value of the unprocessed film suggesting that there is nearly no processing related damage to the NbN film.

We have studied the temperature dependence of the critical current density in the NbN bridges on 3C-SiC by measuring the current–voltage (I–V) characteristics in the temperature interval from 4.2 K to the critical temperature. Additionally, we have fabricated a batch of similar bridges (28 measured devices) made of thin NbN film (see table 1), grown on a crystal quartz substrate. The quartz substrate was used because it has the advantage of having low dielectric constant and low RF losses, a combination very suitable for THz waveguide applications [5]. We assume that both samples have the same contact resistance and both NbN films have equal specific resistance.

The measured values of the critical temperature of NbN film grown on 3C-SiC/Si substrate are shown in the Table 1. The quartz substrate was used because it has the advantage of having low dielectric constant and low RF losses, a combination very suitable for THz waveguide applications [5].

<table>
<thead>
<tr>
<th>Substrate</th>
<th>R_{20 K} (Ω)</th>
<th>w (nm)</th>
<th>d (nm)</th>
<th>T_C (K)</th>
<th>j_C(0) (MA cm⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3C-SiC/Si</td>
<td>213 ± 12⁸</td>
<td>800</td>
<td>5⁹</td>
<td>11.3</td>
<td>3.05</td>
</tr>
<tr>
<td>Quartz</td>
<td>135 ± 15⁵</td>
<td>800</td>
<td>8.6 ± 1.7⁷</td>
<td>9.3</td>
<td>1.14</td>
</tr>
</tbody>
</table>

Table 1. Normal-state and superconducting properties of the samples.

The values for the bridges on 3C-SiC/Si substrate are taken over seven samples.

a Obtained from the HRTEM image, figure 1.

b The average critical current density (j_C) on the quartz substrate is taken over all 28 samples.

c The film thickness is estimated based on scaling of the bridge resistance with the film thickness, assuming that the samples on both substrates have the same contact resistance and both NbN films have equal specific resistance.

d The film thickness is estimated based on scaling of the bridge resistance with the film thickness, assuming that the samples on both substrates have the same contact resistance and both NbN films have equal specific resistance.

e The value of the unprocessed film suggesting that there is nearly no processing related damage to the NbN film.

To our knowledge, the work presented here is the first to report on extensive DC characterization of fabricated HEB devices on epitaxial ultra-thin NbN films grown on a single crystal 3C-SiC layer. Therefore, we consider the data from the DC characterization as a useful complement to the work presented in [14, 17], where only temperature–resistance curves of plain NbN films have been shown.

4. Conclusions

The presented experimental work on deposition and characterization of ultra-thin NbN films on 3C-SiC/Si substrate demonstrated that the use of monocrystalline 3C-SiC/Si substrates provides good lattice matching with NbN, resulting in epitaxial growth of ultra-thin NbN films. The complete epitaxial growth of NbN results in an improvement of the superconducting film properties. The highest T_C value obtained for the 5 nm thick epitaxial films was 11.8 K. The processed NbN HEBs had a similar critical current density of ~2.5 MA cm⁻² at 4.2 K across the wafer. We conclude that the epitaxial NbN films grown on 3C-SiC/Si substrate have a good potential to be used in THz HEB mixers with the purpose of further IF bandwidth improvement.

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References