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Electron spectroscopy study in the NbN growth for NbN/AlN interfaces

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Abstract

NbN superconductor and wide band gap AlN thin films were deposited using sputtering at room temperature. Study of the nitride interfaces are forerunner to the growth Josephson junctions that are considered able to work in the terahertz frequency. We find that to be compatible with lithography technology and to have a high critical transition temperature, the substrate should not be overheated, and this means working in low power regime to limit the induced heating of the plasma. X-ray photoelectron spectroscopy and X-ray diffraction analysis were performed on samples deposited on crystalline, amorphous, flexible, and nanostructured substrates. The experimental results suggest us how to improve the deposition process in order to obtain the best nitride films as well as NbN/AlN/NbN trilayers for Josephson junction applications.

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1. Introduction

In the recent past, there has been an increased interest in low-temperature superconductors. The commercial lowtemperature (10 K) cryocoolers now available have made possible the growth and investigation on new materials, especially, concerning thin film technologies. Special attention has been focused on NbN films and on their characterization [1,2]. This nitride, whose critical temperature (T_c) is 16 K in the stoichiometric composition, presents high critical current density and high mechanical stability that makes it even more interesting than the high T_c superconductors, for the realization of layered heterostructures like Josephson junctions (JJ); indeed, these are the most popular superconducting devices used in micro-electronics. The combination of two nitrides (a few nanometers of AlN, deposited as an insulating gap, between two layers of NbN) produces a good superconductor-insulator-superconductor (SIS) junction in the range 13-15 K [3,4]. An increasing number of research efforts are aimed at finding the right sputtering parameters in combination with the constraints of the electronic industry, i.e., the use of photoresist for the photolithography masks and the use of different substrates such as crystals, glasses and even flexibles. In this paper we report (i) the sputtering parameters found during the growth of the NbN/AlN interface at room temperature; (ii) the X-ray photoelectron spectroscopy (XPS) results which give the electronic structures of the Niobium nitride and information on the stoichiometry; and (iii) the X-ray diffraction (XRD) data, to yield information on the structure of the interface. We found that even with

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the substrate at room temperature and limited sputtering DC power, it is possible to obtain NbN with T_c well above 14 K. Heating the substrate or increasing the sputtering power, procedures that normally improve the quality of the film deposition, is not suitable in the presence of material with low vapor pressure like the photoresist.

2. Experimental

Different T_c values have been obtained for thick NbN films grown, using the same parameters, on different substrates i.e., (1) Mylar, Kapton (and glass, not reported here), among the amorphous and flexible substrates; (2) Si, MgO as crystalline; and (3) nanostructured alumina (anodized Al_2O_3). The highest T_c (14.2 K) for NbN film is obtained using 200 W sputtering power (315 V, 0.64 A) and Si (100) as substrate. The sputtering parameters must be carefully chosen to obtain high quality films compatible with the constraints of photolithography technology. We also paid attention to (a) the DC or RF mode; (b) the gas composition; (c) the total pressure, and (d) the target-substrate distance. The adhesion on the different substrates has been checked, and we found that the structured Al₂O₃ substrate is the most resistant to mechanical scratching. The structured Al₂O₃ has been obtained from a sputtered aluminum film treated at room temperature in oxalic 0.5 M acid solution under a constant bias voltage. The distance between anode and platinum cathode was 10 mm. Different bias voltages induce different porous oxide growth on aluminium, in particular, the roughness of the film surface increases with the bias. In this experiment, we tried four different voltages: 10 V, 20 V, 30 V, and 40 V with a fixed exposure time (15 min), and we chose the film growth at 20 V. The roughness obtained with this bias gives a good adhesion without affecting the interfaces of the layered heterostructure. The NbN/AlN interface is realized by the alternate deposition of NbN and AlN [6]. The two nitrides are grown with different sputtering param-



Fig. 1. Superconductor behaviour for NbN semiconductor and Nb metallic films on Si(100).

eters, starting from Nb and Al target (4 in.). NbN was grown in DC mode with a gas mixture composition of 9:1 Ar/N₂ flux, while AlN was deposited in RF mode (100 W) with a gas mixture of 1:1 of N₂ on Ar, the total pressure in the deposition chamber being 10^{-1} Pa. In these conditions, the deposition rates for NbN and AlN were 0.6 and 0.06 nm/s, respectively (measured by an ex situ thickness profilometer). In a layered heterostructure deposition, typical thicknesses were 200 nm for NbN and 1 nm for AlN. Figs. 1 and 5 show the resistivity measurements of NbN films made by means of four-probe technique in a closed cycle helium cryostat apparatus. The XPS data as well as XRD diffractograms were collected, after exposure of the samples to air, by using standard dedicated laboratory equipments, with Mg-Ka and Cu-Ka, respectively as photon sources.

3. Data analysis

The resistivity of all the films was measured in order to verify their superconducting quality. It is interesting to note that a different concentration of N_2 in the Ar/N_2 gas mixture gives a semiconducting or metallic behaviour, and this is evident observing the high temperature side of the resistivity measurement (Fig. 1). The NbN film in Fig. 1 is grown under a high power condition (300 W DC, 360 V, 0.83 A) and shows the highest T_c but the growth temperature reached is too high for lithography technology.

In Fig. 2, a SEM image of the NbN thin film deposited on a Si substrate is shown in order to prove the uniformity and good quality of the film obtained by using the sputtering parameters reported above. Two sets of XPS spectra detected at the Nb core level $3d_{3/2}$ - $3d_{5/2}$ and $3p_{1/2}$ - $3p_{3/2}$ peaks have been reported to understand the surface composition of Nb and NbN samples varing the N₂/Ar ratio.

In Fig. 3, the top curve shows the spectrum of the Nb film ($T_c = 9.2$ K), where the presence of various NbO_x is evident. It appears as a $3d_{5/2}$ and $3d_{3/2}$ doublet shifted



Fig. 2. SEM image of the NbN thin film on Si(100) substrate.

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