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Properties of ultrathin epitaxial NbN_x film on C-cut sapphire

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Abstract. Here we report on the results of obtaining and study of epitaxial ultrathin superconductive films of niobium nitride grown on a C-plane sapphire substrate. The films were deposited from metal-organic precursor using the plasma-enhanced atomic layer deposition. We employed X-ray diffraction, and high-resolution transmission electron microscopy techniques to study the structural properties of the films. We also determined the quasiparticle diffusion constant, the coherence length, the superconducting transition temperature, the critical current density, and the non-uniformity of the resistance distribution of niobium nitride films.

Keywords: atomic layer deposition, niobium nitride, epitaxy, superconductivity, critical current density

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Свойства ультратонких эпитаксиальных пленок нитрида ниобия на сапфире с C–cut ориентацией

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Аннотация. В данной работе были получены эпитаксиальные сверхпроводящие ультратонкие пленки нитрида ниобия на C-cut сапфире. Пленка нитрида ниобия осаждалась методом атомно-слоевого осаждения, усиленного плазмой из металлорганического прекурсора. Были изучены структурные свойства эпитаксиальной пленки и некоторые сверхпроводящие характеристики.

Ключевые слова: атомно-слоевое осаждение, нитрид ниобия, эпитаксия, сверхпроводимость, критическая плотность тока

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Introduction

NbN is a type II superconductor whose superconducting transition temperature is close to 17 K. Because of this, NbN is used to manufacture superconducting nanowire single-photon detectors (SNSPD) with quantum efficiency above 90% [1], photon number resolution (PNR) detectors [2], wideband hot-electron bolometers (HEBs) [3], and other types of devices. Extended defects, such as multiple grain boundaries of polycrystalline films, can limit the critical current value [4], degrading detector performance, for example, leading to temporal instability of the SNSPD leading edge [5, 6]. This has generated considerable interest in the research community to grow NbN epitaxial films on various single-crystal substrates and epitaxial buffer layers with a lattice constant close to the NbN lattice constant. Here, we report an ultrathin NbN epitaxial film grown on a sapphire C-cut substrate using the plasma-enhanced atomic layer deposition (PEALD) technique at 350 °C. The resulting ultrathin NbNx epitaxial film was characterized by X-ray reflectometry, X-ray diffraction, and high-resolution electron microscopy. The NbNx film was found to have a high-quality crystal structure according to [111]. For the obtained epitaxial film, we determined the superconducting transition temperature, critical current density, coherence length, diffusion constant and non-uniformity of sheet resistance distribution.

Materials and Methods

We chose 2-inch sapphire C-cut substrate wafers (0001) for deposition. The ALD process of the NbN film consisted of five main steps. In the first step, the organometallic precursor TBTDEN was fed into the chamber. After exposure to the precursor, the chamber was purged with Ar. Then, the NH_3/Ar reaction gas mixture was fed into the chamber and the plasma was ignited. After the plasma exposure to the NH_3/Ar gas mixture was completed, the H_2/Ar gas mixture was fed into the chamber and the plasma was ignited. At the end of the cycle, the chamber was purged with Ar.

Structural analysis was performed using an Empyrean diffractometer from Malvern Panalytical with Cu Kα1 radiation. The diffractometer was equipped with a primary hybrid monochromator, a parallel analyzer, and a 4-round 5-axis goniometer for single crystal and epitaxial layer analysis. Scanning curves (2θ-ω) were obtained with a primary parabolic X-ray mirror. The structure of the NbN film sample was examined by HRTEM using a JEOL JEM-2100 Plus microscope at an accelerating voltage of 200 kV.

Magnetoresistance was measured using the four-point probe method over a range of temperatures and a magnetic field varying from 0 to 7 T applied both parallel and perpendicular to the sample. The critical current was measured using a precision Keithley 6221A low-noise current source using a two-point technique. The uniformity of the resistance distribution was measured with a 4-probe resistance meter.

Results and Discussion

Fig 1, *a* shows the experimental scanning curve (ω-2θ) near sapphire symmetric reflection (0006). The peak (111) of NbN_x and its satellites, which occur only if the monocrystalline film has a uniform thickness composition and a coherent film-substrate interface, are also shown in Fig. 1. The calculated curve for a monocrystalline NbN_x epitaxial layer with a thickness of 3.8 nm is shown below the experimental curve in Fig. 1. This curve is in good agreement with the experimental one, which shows that the NbN_x epitaxial layer grows in the plane (111) parallel to the sapphire plane (0001). The inset to Fig. 1, *a* shows the swing curve on NbN_x reflection (111). The FWHM value is low and equals 0.02°, indicating that most planes have minimal deflection from the growth direction (111). According to the TEM study, the film is also epitaxial. The planes parallel to the surface of the substrate surface without discontinuities within the analysed area can be seen in Fig. 2, *b*. The NbN_x lattice parameter was determined from high resolution TEM images as 4.41 ± 0.02 Å. The cubic phase with spatial group Fm3m is the main phase. The presence of satellites in the ω-2θ curve indicates significant structural perfection of this layer.

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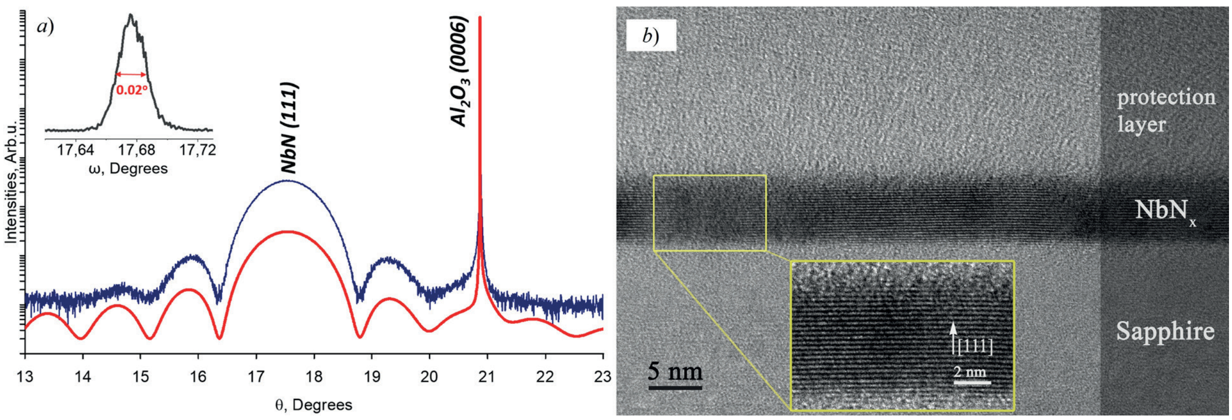

Fig. 1. Experimental scanning curve (ω−2θ) on symmetric reflections (111) NbN_x and (0006) sapphire (blue) and calculated curve for the single crystalline NbN layer 3.8 nm thick (red) (*a*). HRTEM image of NbN_x film (111)/Al_2O_3 (0001) (*b*)

The value of the superconducting transition temperature for the 6 nm thick film was 12 K. The upper perpendicular and parallel critical fields of $H_{c2}(0) \perp$ and $H_{c2}(0) \parallel$ were 10.7 T and 234 T, respectively. A temperature dependence of sample resistance in perpendicular magnet field was analyzed to determine the diffusion constant (D) and coherence length (ξ_{GL}). This data for epitaxial NbN in applied perpendicular field is given in Fig. 2, *a*. The data was taken in the magnet field ranging from 0 to 7 T. It is demonstrated that during the increase in a magnet field value. We show that for increasing values of magnetic field the value of superconducting transition temperature decreases and the width of the transition increases. This is the typical behavior of superconductors in a magnetic field [7]. The electron diffusion constant was determined using the derivative of the critical field value with respect to the temperature, taken at $T = T_c$, according to the expression in the paper [8]. We obtained a value of the diffusion constant of 0.6 cm^2/s. The coherence length ξ_{GL} at 0 K was determined according to the formula from the article [9]. According to the expression, the value of the coherence length is 4.7 nm. Temperature dependences of the resistance of the epitaxial NbN_x film in the applied perpendicular magnetic fields are shown in Fig. 2, *a*. The critical current value was 525 μA at 4.2 K, which corresponds to a current density of 5.7 MA/cm^2. The temperature dependence of J_c for this bridge is shown in Fig. 2, *b*.

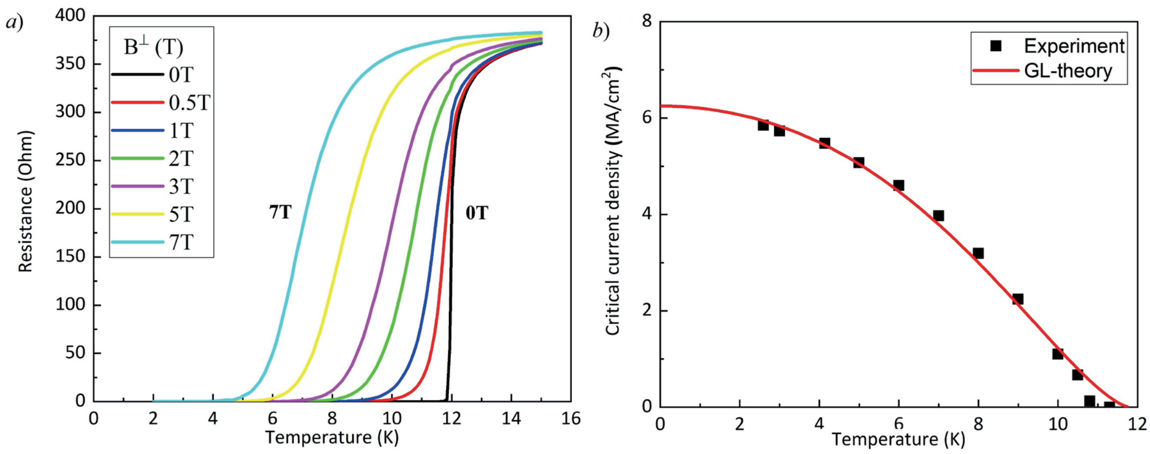

Fig. 2. Temperature dependences of the resistance of the epitaxial NbNx film in the perpendicular magnetic field (*a*). Temperature dependence of the critical current density in the NbN_x epitaxial film microbridge on a sapphire substrate (*b*)

The uniformity of the sheet resistance distribution measured with a 4-probe resistance meter is shown in Fig. 3. The non-uniformity of the sheet resistance distribution was 1.6 %. The non-uniformity of the obtained NbN_x films is largely determined by the diameter of the inductively coupled plasma source and the operating pressure at the plasma exposure stage in the process chamber.

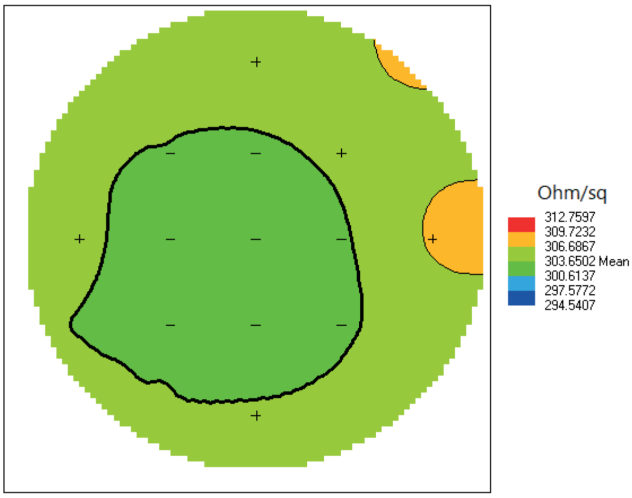

Fig. 3. Sheet resistance distributions of an epitaxial film of NbN_x on a sapphire C-cut substrate

Conclusion

We managed to deposit an epitaxial high quality ultrathin NbN_x film on the C-plane sapphire by the ALD method at 350 °C. We also have established the features of epitaxial growth of the NbN_x film we deposited. We have also determined the values of superconducting transition temperature and critical current density as 12 K and 5.7 MA/cm^2, respectively. In this case, the coherence length is equal to 4.7 nm and the diffusion constant is equal to 0.6 cm^2/s. The non-uniformity of the resistance distribution was 1.6%.

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