HIGH QUALITY Nb NANOLAYERS AS A BASE FOR SUPERCONDUCTING SPINTRONICS

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Abstract

Advanced technological process for high quality Nb film preparation of large area (~600 mm$^2$) and constant nanoscale thickness is developed. Homogeneity and proper thickness of the Nb layer were proved by the target-holder movement during the DC sputtering. The special protection cover Si-layer was used for a long-term stability of the superconducting properties of the prepared Nb films. The increase of superconducting critical temperature (> 1.5 K for films with comparable thickness) in comparison with films prepared by conventional technique is reported.

Introduction

Superconducting hybrids based on thin films are the object of intense investigations for recent decades as a base element for superconducting electronics [1]. Niobium is a traditional material for superconducting electronics. Meanwhile, Nb has a high getter capacity with adsorbed gases which intensively affects the superconducting properties especially for nanoscale thick films. To develop a reliable producing of the most important for applications superconducting Nb layer with thicknesses of $d_{Nb} \sim 5$-15 nm (keeping $d_{Nb} \approx \xi_{Sc}$ - superconducting coherence length) and with critical temperature $T_c$ close to its bulk material value, using a method compatible with multilayered structures production, is a challenge for constructing of superconducting devices based on proximity effect [1]. The general approach to increase $T_c$ for deposited Nb films is to deepen vacuum in the deposition system, to heat the substrate or to increase the deposition rate [2]. However, this approach restricts applicability of thin Nb films in hybrid electronic devices. The presented method improves the quality of Nb films in the range of thicknesses of 5-15 nm avoiding standard restrictions.

Experimental details

Nb samples were prepared by magnetron sputtering on flame-polished glass substrates or on commercial (111) silicon substrates kept at room temperature. The base pressure in the “Leybold Z400” vacuum system was about 2×10$^{-6}$ mbar; pure argon (99,999%, “Messer Griesheim”) at pressures of 8×10$^{-3}$ mbar used as a sputter gas. Targets of 75 mm in diameter, from Nb (99,99%) and Si (99,999%) were pre-sputtered for 3-5 minutes to remove contaminations as well as to reduce the residual gas pressure in the chamber during the pre-sputtering of Nb. As a next step, we deposited silicon buffer layer with RF magnetron to obtain clean interface for the subsequent niobium layer. To provide homogeneity and proper thickness of the Nb layer the target-holder was moved during the DC sputtering using specially constructed arrangement based on controllable DC motor with a gear. Using this setup result in
the average growth rate of the Nb layer ~ 1.3 nm/sec (the steady-state deposition rate would be about 4 nm/sec), the deposition rate close to optimal value for usual deposition technique [3]. To prevent the deposited films against degradation in an ambient atmosphere we protected the samples with ~5 nm silicon cap layer.

![Fig. 1. The design of resulted nano-sized layered structure based on Nb-film.](image)

Figure 1 demonstrates the design of fabricated nanosize layered structure. The resulting long samples (length = 80 mm, width = 7.5 mm) were cut equidistantly and subsequently perpendicular to the long side of the sample onto 2.5 mm wide strips for resistance measurements using a diamond cutter (dashed lines in Fig. 1).

The standard DC four-probe method was used, applying a sensing current of 10 μA with alternating polarity. The Rutherford backscattering spectrometry (RBS) gives a possibility to determine the absolute thickness of the layers at the level of 1 nm with an accuracy of 0.03 nm.

### Results and discussion

The thickness measurements and the elementary analysis of the films were performed by RBS after $T_c$ detection by resistive measurements. The properties of the Nb films are presented in Table 1. The elementary analysis performed by RBS did not detect any impurities inside the Nb layer in the range of RBS accuracy (1.5÷2 at %). Figure 2 presents Nb layer thickness determination for the series S15 and S16. The numbers within series correspond to cut sequence of strips. The dashed lines in Fig. 2 are averaged values of Nb films thicknesses 7.3 and 8.3 nm for each long sample (S15 and S16). The accuracy of thickness determination by RBS method for such range of thicknesses is within 0.4-0.5 nm.

![Residual resistance $\rho_N$ (µΩcm)](image)

Table 1. Properties of the deposited Nb films.

<table>
<thead>
<tr>
<th>Deposited material</th>
<th>Thickness, (nm)</th>
<th>Critical temperature $T_c$ (K)</th>
<th>Residual resistance $\rho_N$ (µΩcm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nb, 99.99%</td>
<td>5.4</td>
<td>5.7</td>
<td>20.2</td>
</tr>
<tr>
<td></td>
<td>6.8</td>
<td>6.36</td>
<td>18.5</td>
</tr>
<tr>
<td></td>
<td>9.3</td>
<td>7.44</td>
<td>12.6</td>
</tr>
<tr>
<td></td>
<td>28.3</td>
<td>8.25</td>
<td>7.9</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>8.91</td>
<td>5.4</td>
</tr>
</tbody>
</table>

Thickness deviation did not exceed 0.5 nm from strip to strip for each series, being in the accuracy of the measurements range. Anyway, this value of thickness deviation corresponding to 0.1-0.15 K difference in $T_c$ from strip to strip is quite suitable for proximity effect investigation and for technical applications.
The critical temperatures of the samples with Nb thicknesses 5.5 - 100 nm are presented in Fig. 3. The slope of the dependence of superconducting critical temperature on thickness changes at about 10 nm corresponding to the residual resistance. The critical temperatures of 5.7-7.5 K are close to the ones detected for the best of the Nb thin films in the same thickness range prepared by molecular beam epitaxy process (MBE) in much better vacuum conditions [6]. The increase of superconducting critical temperature is >1.5 K for films with comparable thickness in comparison with films prepared by magnetron sputtering using common technique [3].

The shape of typical superconducting transition is presented in the insert in Fig. 2. The width of transition (criteria 0.9 $R_N$-0.1 $R_N$, $R_N$ is resistance in normal state before transition) is only 0.05 K.

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**Fig. 2.** Thicknesses of Nb samples (“strip”) cut from wedges S15 and S16. The data are based on RBS measurements [5]. Sample number in series corresponds to its position along the wedge.

**Fig. 3.** Critical temperature of the Nb samples with the thickness in the range of 5.5 – 100 nm. Dashed line indicates the value of the “thick” Nb film with thickness 6.8 nm, $T_c = 8.91$ K. Inset: Typical superconducting transition for Nb film with thickness 6.8 nm, $T_c=6.37$ K (criteria 0.5$R_N$).
Conclusions

The presented technological approach yields significant improvement in superconducting properties (>1.5 K for films with comparable thickness) of large area nanoscale Nb films in comparison with common method of DC- magnetron deposition [3]. The thickness deviation of Nb layer along the sample does not exceed 5-6% for all strips. This value is in general within the accuracy of the thickness determination method (RBS). The increase of superconducting critical temperature (>1.5 K for films with comparable thickness) opens the possibility of proximity effect investigation and spintronic devices construction based on large area superconducting films with the thicknesses of a few nanometers. The proposed technology is patented [4] and used for preparation of the superconducting spin-switch, the base element of spintronics.

Acknowledgements

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References