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Investigation of thin films for fabrication of Nb/AlN/NbN tunnel junctions and microstrip lines of NbTiN-SiO$_2$-Al

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Abstract: **The surface of thin films of Nb, Al, NbTiN, SiO$_2$, Al$_2$O$_3$ is investigated in this work. These films are necessary for the fabrication of high-sensitive devices of THz range. The fabrication processes of such devices are described briefly. All films were fabricated using a Kurt J. Lesker magnetron sputtering system. The study of the film surface roughness was carried out using a Bruker Ikon atomic force microscope. The surface quality of films is determined not only deposition mode, but plasma etching process also. The best values of the root-mean-square deviation of the surface profile R$_q$ = 2 nm were obtained for the used NbTiN film with a thickness of 325 nm. Thin Al-layers that is used for tunnel barrier formation is studied. It is shown than Al films with a thickness of more than 6 nm are already continuous. The surface roughness of the single-layer and multilayer films has been studied**

Keywords: **superconductivity, tunnel junctions, magnetron sputtering, thin films, surface roughness**

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1. INTRODUCTION

It is important to know and control the parameters of the thin films used for fabrication of receiving structures for the terahertz (THz) range. Thin films of the needed parameters (surface roughness, resistivity, critical temperature) are required for the microstrip lines and for the superconductor-insulator-superconductor (SIS) Josephson junction. Nb and NbN are used as superconductors for SIS-junctions: Nb-AlO_x-Nb with a limiting frequency of 700 GHz and Nb-AlN-NbN with a limiting frequency of 1.4 THz [1,2]. NbTiN and Al are used as electrodes for the microstrip line and SiO_2 as dielectric.

Niobium-based tunnel SIS-junctions are the main elements for fabrication of high-sensitive detectors for radio astronomy tasks [3,4]. The surface quality of the films used to make devices directly affects their properties. For example, in [5], it was demonstrated that an NbTiN film with a more developed crystal structure (the film roughness is determined by larger crystallites) also has a lower Tc and a higher resistivity, which negatively affects on the operation of the device at high frequencies (more than 1 THz). This article is a continuing work on optimisation of fabrication of thin films for superconducting devices [5] and is aimed to determining the quality of their surface.

2. FABRICATION TECHNOLOGY OF SIS JUNCTIONS

All structures are formed on two types of substrates: high-ohmic polished silicon or quartz. The latter is necessary to achieve the best parameters for waveguide SIS-structures since it's dielectric constant is significantly less than that of silicon. Quartz with a thickness of 200 μm after sample preparation is polished to a thickness of about 40 μm for receiving structures with an operating frequency of 1 THz. A 100 nm thick Al_2O_3 layer is deposited onto the selected substrate over the entire surface. This buffer layer acts as a stop-layer in the subsequent etching of NbTiN in fluorinated gases and avoids etching of the substrate. All films were deposited by magnetron sputtering.

The first layer of the working structure is a bottom electrode from NbTiN 325 nm thick. This thickness is due to the London penetration depth in this material [6]. The NbTiN layer is sputtered over the entire area of the substrate, and then etched over a resistive mask using plasma-chemical etching in tetrafluoromethane (CF_4). The same etching process is used for the Nb and NbN etching. The etching process provides a flat electrode edge, in contrast to the «lift-off» lithography technology, which, under conditions of magnetron sputtering and at similar thicknesses, can leave "walls" of metal along the edges. In addition, this layer is the lower layer of the NbTiN-SiO_2-Al microstrip line, in which high-frequency currents flow. Since the penetration depth of the field is large, inhomogeneities of the order of 10 nm are not very important. For aluminum, which is the upper electrode of the microstrip line, currents flow along the lower surface; therefore, the upper film roughness is also not important.

The next step in fabrication is deposition of three-layer Nb-AlN-NbN (80 nm-7 nm-80 nm) structure over the entire area of the substrate and etching over a resistive mask right through to the NbTiN layer. As a result, "columns" of a three-layer structure are formed on the surface of the lower electrode, which are SIS junctions. The area of the tunnel junction can be from 0.5 to 40 μm^2, which is determined by the tasks. At this step, it is important to stop the etching of the bottom layer in the three-layer structure

(Nb) in time to avoid etching of the NbTiN layer. After that the process of anodisation in a solution of ammonium tetraborate with glycol is happens. It's necessary to protect the sides of the SIS-junctions and surface of NbTiN from the short connection with upper electrode. The same resistive mask is used to deposit an insulator layer 250 nm thick, which is necessary both as a dielectric layer in a microstrip line and as an insulating layer from the shorts between the SIS junction and the upper electrode. The upper electrode is formed from a 450 nm thick aluminum layer.

3. INVESTIGATION OF FILMS SURFACE

The morphological analysis has been carried out through a Bruker Dimension Icon AFM, equipped with a Nanoscope V controller operated in PeakForce Tapping® mode. The measurements have been performed using N-doped Si probes (Bruker Scanasyst-air).

The main parameters in determining the film roughness are the arithmetic mean deviation of the surface profile R_a and standard deviation of the surface profile R_q.

The surface roughness of a thin film depends significantly on the quality of the surface and the material on which it is deposited. We investigated the surface of Al_2O_3 100 nm thick, deposited with an RF magnetron on a silicon substrate. For this film R_q = 1.02 nm, R_a = 0.8 nm were obtained. The AFM image is shown in **Fig. 1**. For comparison, for the surface of optical quartz polished by the mechanical-chemical method R_a = 0.2 nm [7].

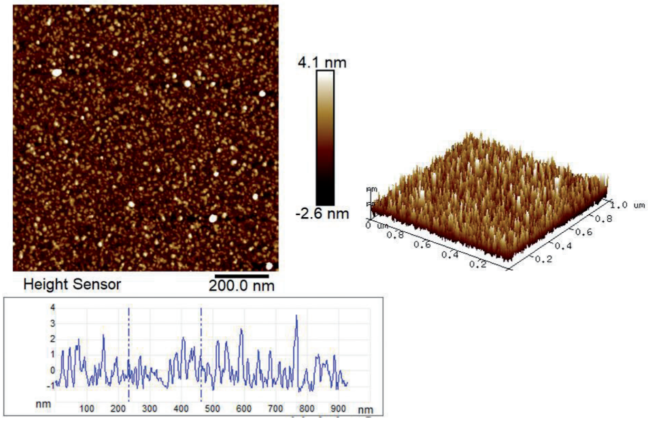

Fig. 1. *AFM images of an 100 nm Al_2O_3 film. Scanning area 1×1 μm². At the top left is a 2D scan view, at the top right it is in 3D, at the bottom is a cross-section in the center of the scan.*

A 325 nm thick NbTiN layer was deposited on a silicon substrate. For the scan area 90×90 μm² R_q = 2 nm, R_a = 1.3 nm were obtained. For the same sample R_q = 2.24 nm, R_a = 1.81 nm were obtained for the scan area 4 μm². The scan profile is shown in **Fig. 2**.

The NbTiN surface was also monitored after plasma chemical etching procedures. A sample was prepared, on which an 80 nm thick Nb layer was deposited, and

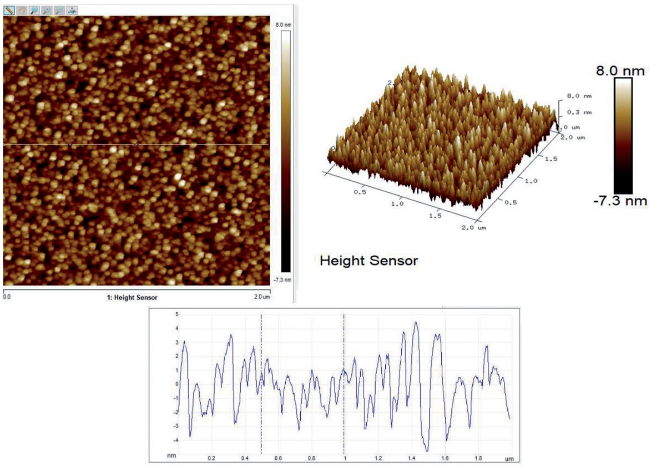

Fig. 2. *AFM images of NbTiN with a thickness of 325 nm. Scanning area 1×1 μm². At the top left is a 2D scan view, at the top right it is in 3D, at the bottom is a cross-section in the center of the scan.*

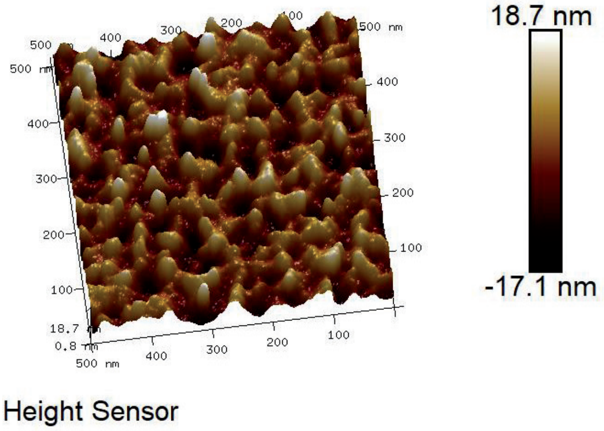

Fig. 3. *Surface of NbTiN after Nb etching. Scanning area is 0.5×0.5 μm².*

then etched into CF_4. **Fig. 3** shows the NbTiN surface after etching the Nb layer. We note that the film roughness became significantly higher: R_q = 5.2 nm, R_a = 4.14 nm.

The NbTiN surface was also studied after etching the Nb/AlN/NbN structure. For the scan area 10×10 μm² R_q = 11.6 nm, R_a = 8.2 nm were obtained (see **Fig. 4**). We conclude that the NbTiN surface is significantly damaged by etching through the overlying layers, which may be associated with anisotropic etching of the material. We are planning to use a thin Al layer (up to 5 nm) as a stop layer to prevent etching of NbTiN in fluorine-containing gases in further studies. In addition, there is information in the literature that the Al layer can wet the NbTiN or Nb layer, which will reduce the surface roughness [8].

According to the technological map, the Nb layer is deposited on top of the NbTiN layer. The fabrication of this layer on a magnetron sputtering system is possible using both a DC magnetron and an RF magnetron. It was shown in [9,10] that in the case of using a DC magnetron, the surface of the films is smoother, which is better suited for fabrication of high quality Josephson junctions. In this work, the roughness of an Nb film with a thickness of 200 nm deposited on a silicon substrate was investigated, as well as the roughness of an Nb film with a thickness of 80 nm deposited over the NbTiN layer. For the first case, the values R_q = 0.78 nm and R_a = 0.62 nm were obtained. The AFM images (2D and 3D) are shown in **Fig. 5.** In the second case, R_q and R_a were 1.86 nm and 1.48 nm on a scan area of 1 μm² (see **Fig. 6**). The surface roughness of Nb film deposited on top of the NbTiN layer has better R_q and R_a values in comparison with the roughness of the NbTiN film.

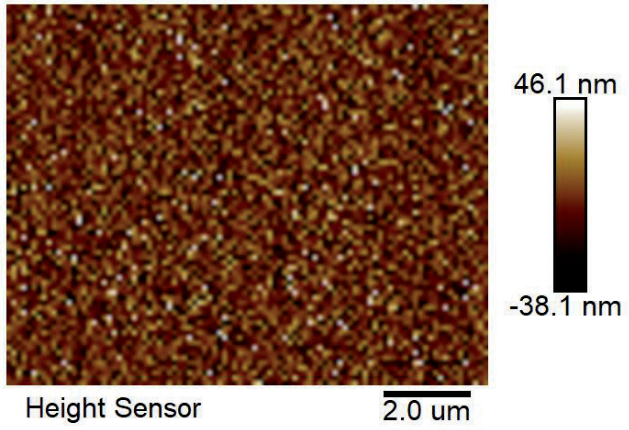

Fig. 4. *NbTiN surface after Nb/AlN/NbN etching. Scanning window area 10×10 μm².*

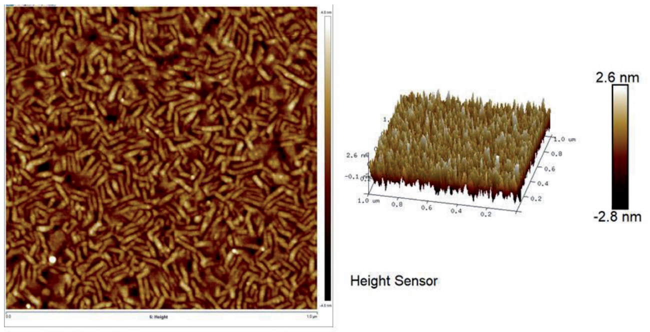

Fig. 5. *Nb 200 nm thick, scan area is 1×1 μm² and its 3D image.*

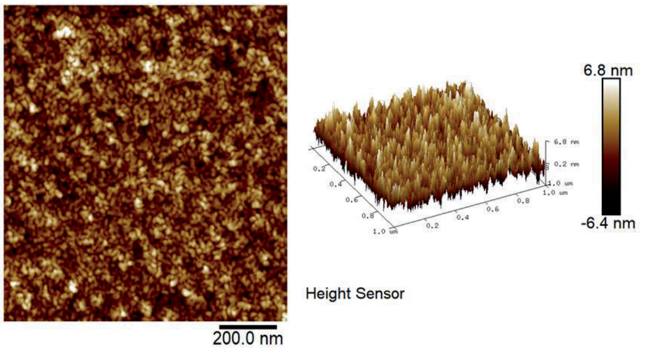

Fig. 6. *Nb 80 nm thick, scan area is 1×1 μm² and its 3D image.*

We studied thin Al films with a thickness of 3 nm, 6 nm, 20 nm, 124 nm, deposited on a silicon substrate with a buffer layer of 100 nm Al_2O_3. Thin films with thicknesses of 3, 6 and 20 nm is sputtered with a low deposition rate (0.2 nm/s) and with a reduced power supplied to the magnetron (300 W), thicker Al films sputter at a magnetron power of 500 W, the deposition rate is 1 nm/s.

Typical thickness of the barrier layer in the SIS junction is 1-1.5 nm, and its formation occurs in the vacuum chamber without breaking the vacuum between the superconducting layers. In these conditions we can't studied surface of AlO_x or AlN directly because additional oxidation occurs at the atmospheric pressure. First, a thin layer of Al 5-7 nm is deposited [8,11], then either oxidation in an oxygen atmosphere at a characteristic pressure of 10^{-1} mbar, or nitridisation in a high-frequency nitrogen plasma (pressure $6 \cdot 10^{-3}$ mbar, power 75 W). **Fig. 7** shows a AFM image of 1×1 μm² of an Al film with a thickness of 3 nm. For this film vertical peak-to-peak distance is 3 nm. It means that this film has island structure. In this case $R_q = 0.9$ nm, $R_a = 0.75$ nm, but we should keep in mind that due to island structure this film repeats the relief of the lower layer-Al_2O_3.

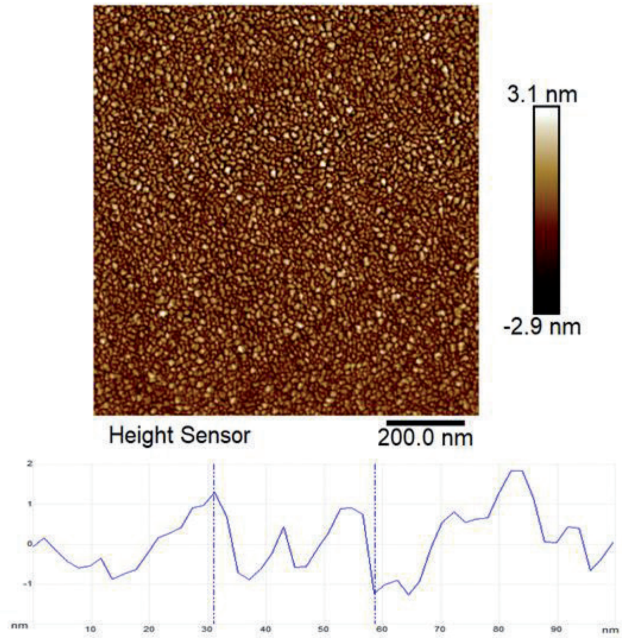

Fig. 7. *Al film, 3 nm thick and its cross section, scan area is 1×1 μm².*

The AFM image of a 6 nm Al film is shown in **Fig. 8**. Here, as in the 3 nm Al

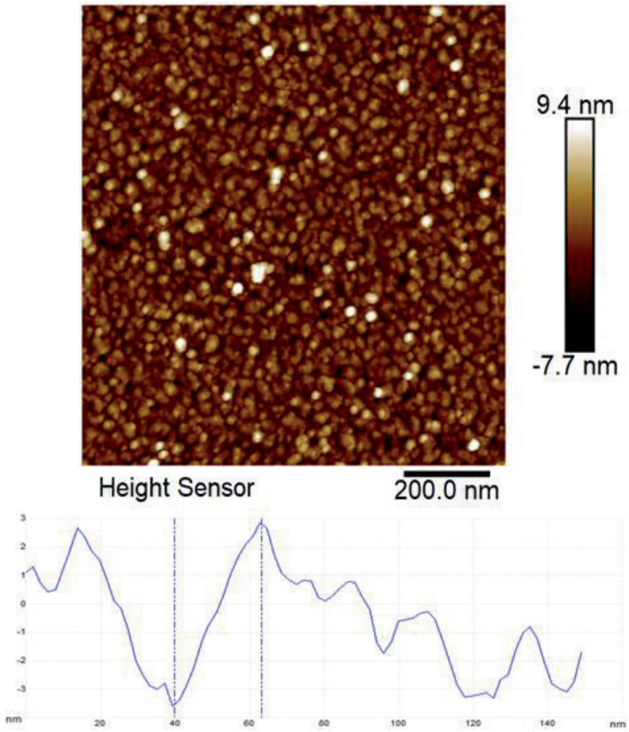

Fig. 8. *Al film, 6 nm thick and its cross section, scan area is 1×1 μm².*

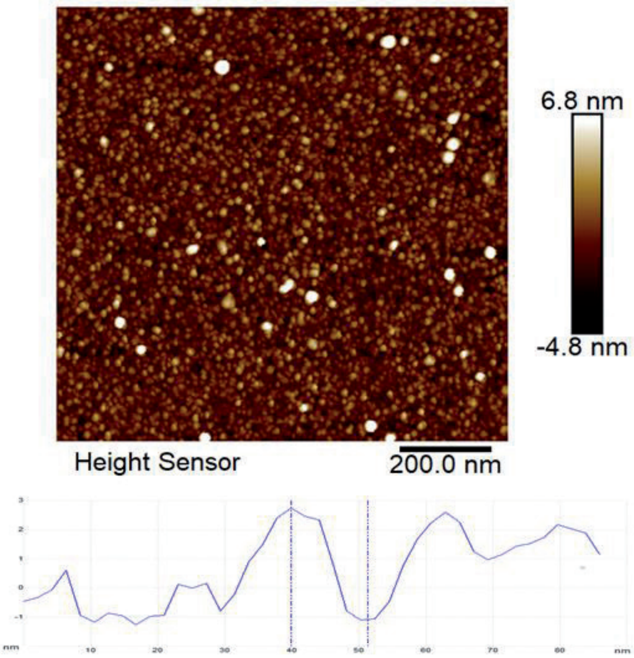

Fig. 9. *Al film, 20 nm thick and its cross section, scan area is 1×1 μm².*

film, it can be seen that the distance between the peaks reaches 6 nm, but the film grains are already much larger. For this film the values R_q = 2.3 nm and R_a = 1.7 nm were obtained.

The AFM image of a 20 nm thick Al film is shown in **Fig. 9**. The maximum peak-to-peak vertical distance for this film is 4 nm, R_q = 1.5 nm and R_a = 1.1 nm, that is less than for 6 nm films. It means that this film has already continuous.

Aluminum films deposited at higher speeds and thicknesses have significantly greater roughness. For Al with a thickness of 124 nm on a silicon substrate covered by a 100 nm Al_2O_3 film R_q = 2.57 nm, R_a = 2 nm (**Fig. 10**, top), but if we take into account a strong feature that can manifest itself at large thicknesses, then R_q = 4.5 nm, R_a = 3 nm (Fig. 10 bottom).

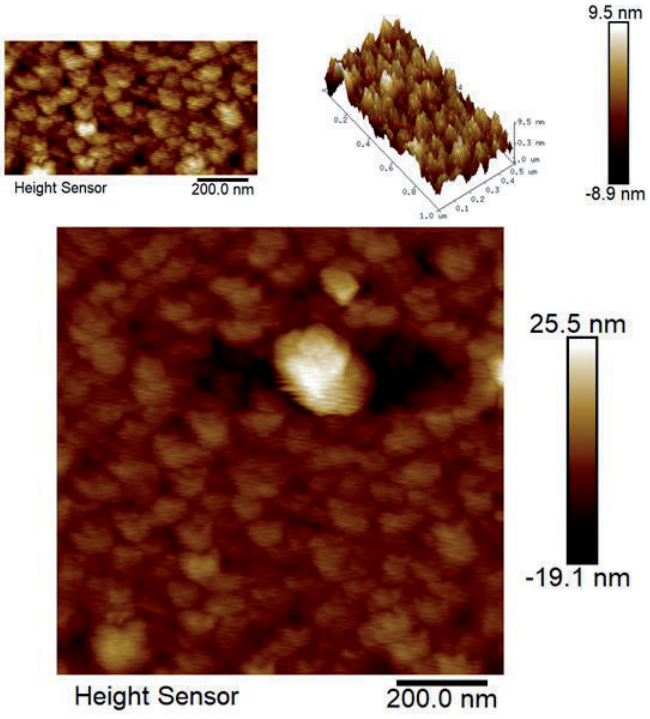

Fig. 10. *Al, thickness 124 nm. Top: scan area with a uniform surface, bottom - with a feature that appears periodically throughout the film.*

We studied the SiO_2 surface, deposited after etching a three-layer structure, on top of the NbTiN layer: R_q = 4.0 nm, R_a = 3.0 nm, which is better than the NbTiN surface after etching. The scan result is shown in **Fig. 11**.

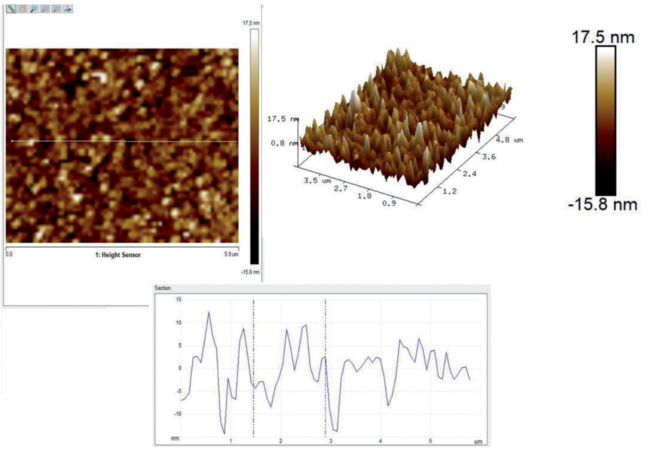

Fig. 11. *Scan 6×6 μm² of SiO_2 surface deposited on top of NbTiN. On the top left is a 2D view, on the right it is in 3D, below is a cross-section.*

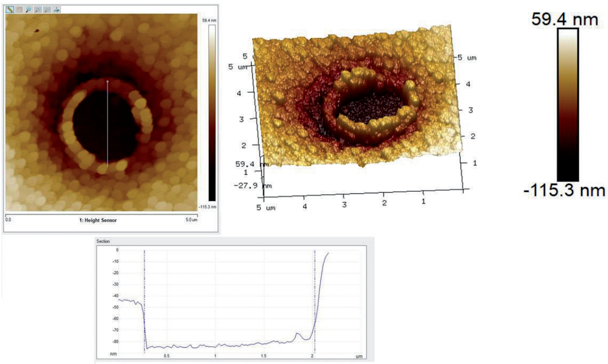

Fig. 12. *SiO_2 surface and SIS junction. Scanning window size 5×5 μm². Top left view in 2D, top right it is in 3D, bottom cross-section along the SIS-junction.*

We also studied the SiO_2 surface directly near the formed SIS junctions: the diameter of junction is 1.8 μm, the characteristic size of the SiO_2 granules is 250 nm (**Fig. 12**).

4. CONCLUSION

Atomic force microscopy was used to study surface roughness of films: Al, Nb, NbTiN, SiO_2, Al_2O_3, which are necessary to fabricate devices that is operates in the terahertz range. The measured values of R_q and R_a show that the surfaces of these films are suitable for further work on the fabrication of SIS junctions. It is noticed that the NbTiN surface strongly depends on the subsequent technological operations, namely, on the plasma-chemical etching. It is proposed for further work to use a thin aluminum layer, which should prevent the etching of NbTiN after etching of Nb. It is demonstrated that the required thickness for the formation of a continuous Al film is more than 6 nm.

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