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# SURFACE CHARACTERIZATION OF NbTiN FILMS FOR ACCELERATOR APPLICATIONS*

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*Abstract*

The development of next-generation SRF cavities requires the deployment of innovative material solutions with RF performance beyond bulk Nb. Theoretical interest has stimulated efforts to grow and characterize thin multilayer superconductor/insulator/superconductor (SIS) structures for their potential capability of supporting otherwise inaccessible surface magnetic fields in SRF cavities [1]. The ternary B1-compound NbTiN is among the candidate superconducting materials for SIS structures. Single crystal NbTiN films with thicknesses below 10 nm are also of interest for the development of high resolution, high sensitivity (SNSPD) detectors for particle physics application. Using DC reactive magnetron sputtering, NbTiN can be deposited with nominal superconducting parameters. This contribution presents the on-going material surface and superconducting properties characterization in order to optimize the NbTiN films for each application.

## INTRODUCTION

Of the Nb B1-compounds, NbTiN has the highest critical temperature, 17.3 K for bulk material [2]. B1-compounds have a NaCl crystal structure. In NbTiN, the FCC sites (Cl) are occupied by metallic atoms, and the octahedral sites (Na) are occupied by non-metallic atoms. The stoichiometry of B1-compounds greatly affects the superconductive properties of the material. In NbTiN, Ti acts as a nitrogen getter which helps to remove vacancies, improving the quality of the film. NbTiN is a type-2 superconductor with a $H_{c1}$ of 30 mT. A theory by Gurevich [1] predicts that multilayer SIS (superconductor/insulator/superconductor) structures composed of alternating superconductor and insulator layers could provide magnetic screening for the cavity, allowing the reach of once unfeasible magnetic fields. One of the bases of the approach is that type-2 superconductors can have their lower $H_{c1}$ enhanced at thicknesses smaller than their penetration depth. NbTiN based SIS structures could give accelerators the potential to operate effectively at liquid He temperature, lowering cost of operation. The chosen insulator for the SIS structures, AlN, can be grown as a cubic crystal with a similar lattice parameter to NbTiN. AlN has been shown to improve $T_c$ of NbTiN , in Terahertz applications [3].

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Superconducting Nanowire Single Photon Detectors (SNSPDs) provide a combination of high detection sensitivity to infrared radiation, fast reset times, and low dark count rates. These benefits are largely due to quantum sensitivity to photons disrupting the superconducting state and the cryogenic temperature of the nanowire. A thin film of about 10-5 nm is required for SNSPDs to allow the absorption of a single photon to transition the superconductor into the normal state. The lack of grain boundaries in a single crystal NbTiN SNSPD may decrease the dark count of the detector.

## EXPERIMENTAL METHOD

The deposition of NbTiN thin films was carried out in a UHV chamber equipped with 3 DC/RF magnetron guns (see Fig. 1). The magnetron guns can also be used in HiPIMS mode. This system is customized to enable deposition of many metallic compounds and dielectric thin films. The vacuum is maintained at a base pressure of $10^{-9}$ Torr by a CTI-10 cryopump via a variable gate valve. To improve the vacuum quality, baking, UV light and plasma cleaning are available between deposition cycles. To monitor the UHV deposition system's base pressure, Bayard-Alpert ion gauges are used. During deposition, high pressure ion gauge is used in combination with an RGA in differential pumping mode to monitor the gases in the chamber.

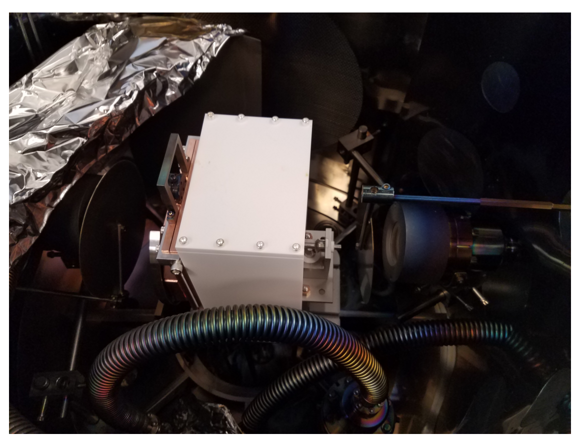

Figure 1: Two of the magnetron guns in the deposition system with the sample stage in the center.

The central sample stage can be heated to 700 °C. The stage is mounted on a differentially pumped rotating conflat system allowing to position the substrates in front of the magnetrons and thus allowing in-situ sequential deposition of different layers. The stage is equipped with a rotatable shutter which allows variation of multiple deposition parameters

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within the same environmental conditions. In this system, NbTiN films are deposited with nominal superconducting properties ($T_c$ up to 17.25 K for bulk-like films).

## CHARACTERIZATION

The topography and roughness of the films were characterized by atomic force microscopy (AFM). An Al coated tip was used for the measurement to prevent the mirror-like character of the films from interfering with the measurement. The roughness of the thinner films (<5 nm) is that of the substrate (see Fig. 2). The thick films (≈ 2000 nm) have up to 5 nm RMS roughness (see Fig. 3).

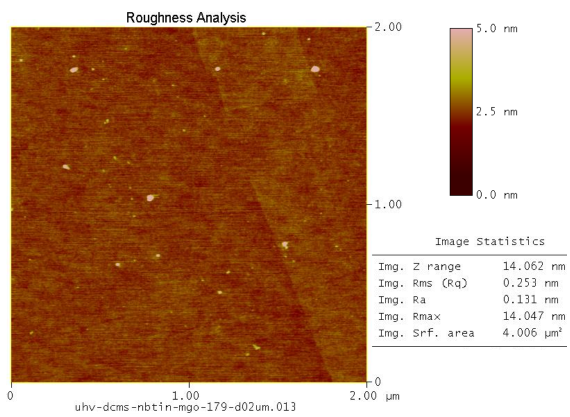


Figure 2: 2 µm x 2 µm AFM scan of a 4.5 nm thick film with a $T_c$ of 14.1 K of NbTiN on MgO substrate.

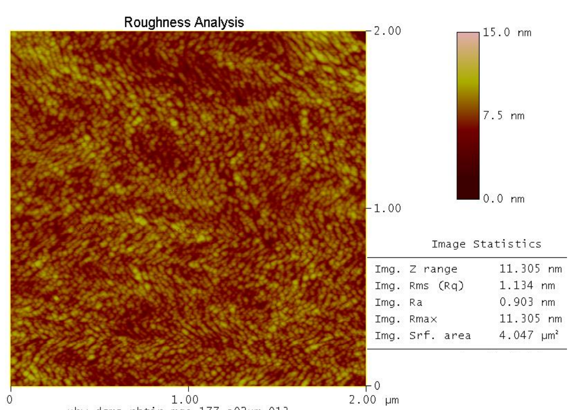


Figure 3: AFM scan of a ≈2 µm thick NbTiNAlNMgO film with a $T_c$ of 16.93 K.

The crystal orientation of the grains was determined with electron backscatter diffraction (EBSD). EBSD IPF (inverse pole figure) maps show the films are single crystals as in Fig. 4. The crystallographic texture of the samples represented by pole figures was measured by X-ray diffraction (XRD) and EBSD as shown in Fig. 5. The XRD technique samples an area $2{\times}10^5$ larger than EBSD. The two techniques show the samples have no texture and confirm they are single crystals.

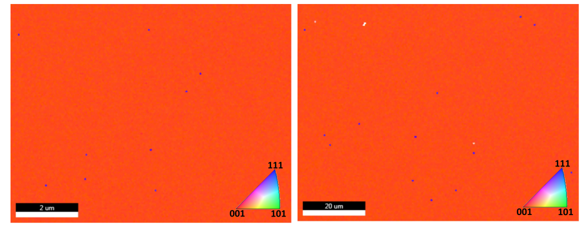


Figure 4: EBSD IPF map of a 5.8 nm thick film with a $T_c$ of 13.36 K of NbTiN on MgO substrate.

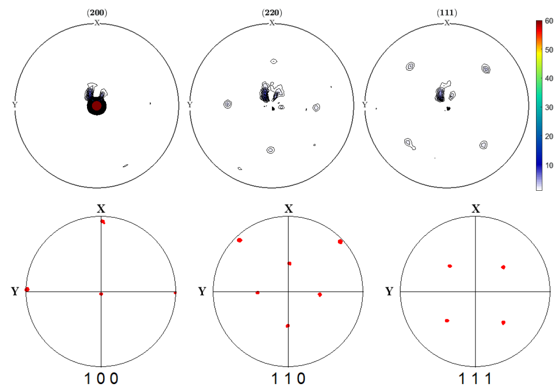


Figure 5: Pole figures measured with XRD (top) and EBSD (bottom). The butterfly like shape at the center of the XRD figures is an artifact from the x-ray beam over projecting the sample and illuminating the stage of the XRD instrument. Background subtractions of a blank substrate were inadequate to fully remove the artifact. The intensity of the diffracting planes at high angles was too low for defocusing corrections to be effective.

$T_c$ is measured with a four point probe using differential conductance measurements. The samples are measured by batches (32) in a liquid He dewar [4]. The temperatures of the samples are measured with calibrated CERNOX thermometers with a sensitivity of 50 mK.

The crystallographic characteristics were measured with a Panalytic X'Pert Pro equipped with a four circle goniometer. To measure the crystal lattice parameter for thick films standard $\theta - 2\theta$ measurements were used. For the thinner films, the four circle goniometer is essential to achieving proper alignment and to optimizing signal intensity. Measurements for thin films used glancing or asymmetric incidence. The lattice parameter of NbTiN deposited on AlN is reduced from the bulk value as shown in Fig. 6.

X-ray reflectivity (XRR) was used to determine the film thickness and interfacial roughness. XRR curves were processed by X'Pert Reflectivity software. To determine the thickness of the film, the software used a FFT of the XRR curve (see Fig. 7). The software estimated the roughness of the samples and verified the thickness, found by FFT, with progressive segmented fit algorithm. The fitted XRR roughness is similar to the AFM RMS roughness but the latter is dependent on the individual area of the AFM scan.

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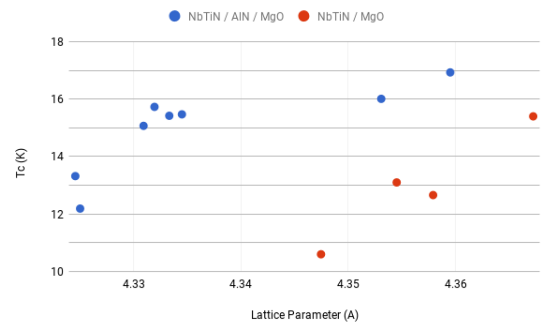


Figure 6: The bi-layer samples have a thickness of NbTiN 33 nm to 2 µm with an AlN layer of 15 nm. Single layer samples have a thickness of 7.5 nm to 15 nm.

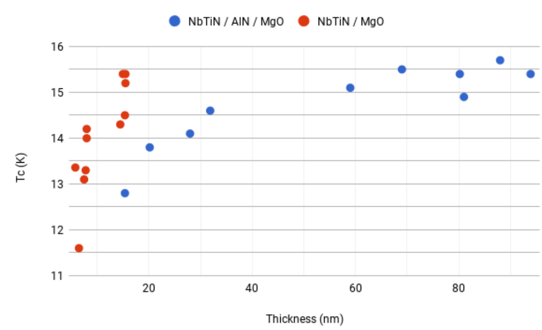


Figure 7: $T_c$ versus thickness for NbTiN/AlN bi-layers and NbTiN monolayers on MgO. Bulk-like (≈2 µm) NbTiN/MgO films exhibit a $T_c$ of 17.25 K and $H_{c1}$ of 30 mT [5].

## CONCLUSION

Deposition of high quality thin films of NbTiN with an interlayer of AlN on MgO and NbTiN on MgO was accomplished. The thickness of NbTiN in the bi-layer films are under 150 nm and single layer under 10 nm. High quality layers of about <150 nm thick NbTiN on AlN with good superconductive properties are required for SIS structures. The deposited bi-layer films have $T_c$ from 15.73 K (141 nm thick) to 12.19 K (33 nm thick). SNSPDs require thin films under 10 nm thick. Thus far, the thinnest deposited NbTiN films (4.5 nm) have a $T_c$ of 14.1 K. NbTiN films with thicknesses within the required range for SIS and SNSPDs structures have been deposited and are very high quality films.

## FUTURE WORK

The next steps will involve SQUID measurements to determine the first flux penetration field for films of various thicknesses and to describe how the number of layers affect the magnetic shielding properties of the SIS structures. For SNSPDs, a campaign of detector design and etching is needed to find an optimal device geometry.

## ACKNOWLEDGEMENTS

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