

SURFACE CHARACTERIZATION STUDIES OF GOLD-PLATED NIOBIUM*

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Abstract

The native niobium oxide layer present on niobium has been shown to affect the performance of superconducting RF cavities. Extremely thin layers of gold on the surface of niobium have the potential to suppress surface oxidation and improve cavity performance. However, depositing uniform layers of gold at the desired thickness (sub-nm) is difficult, and different deposition methods may have different effects on the gold surface, on the niobium surface, and on the interface between the two. In particular, the question of whether gold deposition actually passivates the niobium oxide is extremely relevant for assessing the potential of gold deposition to improve RF performance. This work builds on previous research studying the RF performance of gold/niobium bilayers with different gold layer thicknesses. We here consider alternative methods to characterize the composition and chemical properties of gold/niobium bilayers to supplement the previous RF study.

INTRODUCTION

Niobium is a standard choice of superconducting metal for the construction of superconducting radio frequency (SRF) cavities. As cavity fabrication and preparation procedures have improved, resulting in corresponding improvement in cavity performance, the surface properties of the niobium used have become a focus of research and development. Since an RF field excited in a cavity interacts with the cavity most strongly at the surface (within the first few tens of nanometers [1]), changes to the surface can dramatically impact performance of the cavity [2].

The native niobium oxide is the outermost layer present on niobium, and its properties are relevant to RF applications and research. The oxide is thin (less than 10 nm in total) and is dominated by an Nb₂O₅ layer, followed by a layer of NbO₂, then NbO_x ($x \leq 1$) phases [3]. A diagram of the oxide layers, taken from [4], is shown in Fig. 1.

This work is an extension of previous work carried out at Cornell which uses thin gold layers to study changes to surface resistance as a result of replacing the native niobium oxide with a non-oxidizing normal conducting layer. The full extent of that work can be found in [4] and [5]. The goal of the previous work was to study the effect of changes to surface resistance on cavity performance. Rather than trying to manipulate the properties of the niobium oxide directly (a time-consuming and challenging process, owing to the many subtle ways that the oxide can be manipulated through procedures such as heat treatments, chemical treatments,

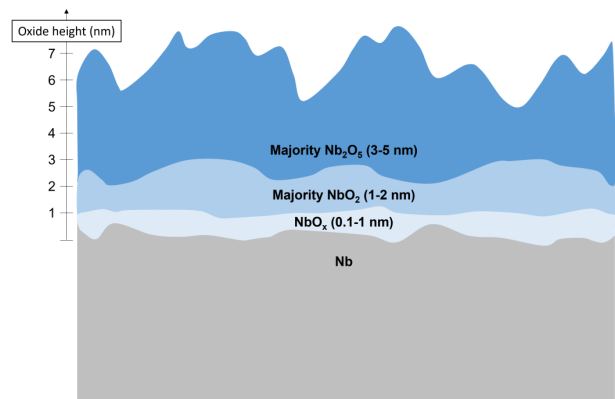


Figure 1: Cartoon of the native niobium oxide structure, taken from [4].

atmospheric environment, and more), the authors aimed to replace the native oxide with a thin layer of gold. Since gold is a non-oxidizing normal conductor that adheres well to niobium even at thin layers, this effectively allows for the isolation of the oxide as an experimental variable. RF performance and surface resistance could then be directly analyzed without concern for the intervening oxide.

In this study, we introduce two main extensions to the original work. The first is the use of X-Ray Photoelectron Spectroscopy (XPS) to study the composition of the surface of the niobium directly. The second is the development of alternative deposition methods for gold on niobium. The use of XPS is explained fully in the **Analysis and Results** section, and the development of alternative deposition methods is explained fully in the **Gold Deposition** section, specifically the *Electroplating* subsection.

GOLD DEPOSITION

Sample Preparation

The samples utilized for this study were 1 cm by 1 cm by 2 mm high-RRR (Residual-Resistance Ratio) niobium samples, which are shown in Fig. 2. The samples were prepared using a standard niobium preparation procedure, consisting of an approximately 75 μ m electropolish, followed by a five-hour ultra high-vacuum bake at 800 $^{\circ}$ C, then a short electropolish of approximately 2 μ m. Following the short electropolish, the samples were loaded into an inert-atmosphere glovebox with a nitrogen atmosphere with less than 3 ppm of oxygen content and less than 5 ppm of water content. The samples were submerged in 1-2% hydrofluoric acid for 30 minutes, then were rinsed with methanol and allowed to dry. Unfortunately, it should be noted that due to technical issues with the glovebox, the oxygen content did fluctuate up to 15-20 ppm of oxygen during the procedure. As this is still superior to atmospheric oxygen content by a

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factor of approximately 10,000, it was determined sufficient for this initial study, however the oxygen content present for sample preparation is an area of substantial focus for improvement in subsequent studies.

Lastly, the samples were either attached to 4" diameter aluminum pucks using double-sided tape and sealed in plastic bags (for use with the evaporation deposition system, discussed in the following section), or left in the inert-atmosphere glovebox (for use in development of the electroplating system). In each case, a control sample was kept aside and was exposed to atmosphere instead of receiving gold deposition.

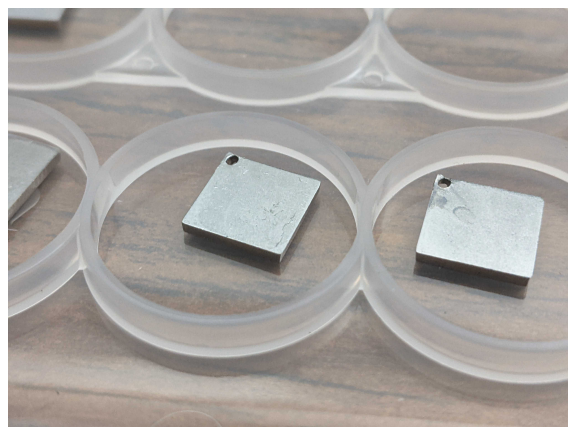


Figure 2: Niobium samples used in the study, in a plastic container for transport.

Evaporation Deposition

This study, following the original work, used a CVC SC4500 evaporation deposition chamber to deposit thin layers of gold on niobium [5]. While the deposition chamber is exposed to atmosphere during sample loading, the nitrogen purge was kept active and sample loading time was minimized in order to minimize the chance of exposing the samples to atmospheric oxygen before gold deposition. One sample at a time was loaded into the deposition chamber before pumping down to 1.5×10^{-6} Torr. Once at low pressure, 1.5 nm of gold was pre-deposited to clean the source before depositing 1 nm, 1.5 nm, or 2 nm of gold onto the sample. The deposition rate used was 0.01 nm/s, and the thickness was confirmed using calibrated Quartz Crystal Control. The chamber was then returned to atmospheric pressure and the samples were removed and taken for XPS imaging.

Electroplating

This study also furthered development of an electroplating system for gold, based on previous work by Z. Sun (displayed at this conference, [6]). While thin-layer gold deposition has yet to be confirmed for the setup, it has allowed for deposition of thicker layers. The setup is shown in Fig. 3. The plating solution used was a commercially available gold electroplating solution. The niobium sample hanging on a niobium wire is the cathode, and a platinum wire is used as the anode. The solution is heated to 50 °C before plating.

Low current (below 5 mA) and short plating times are used to keep the deposition rate low for thin-film deposition.

While development of the setup for thin-film is still in progress, the setup has been confirmed to deposit thicker layers of gold at higher currents and deposition times, and is a promising method for future Nb-Au studies.

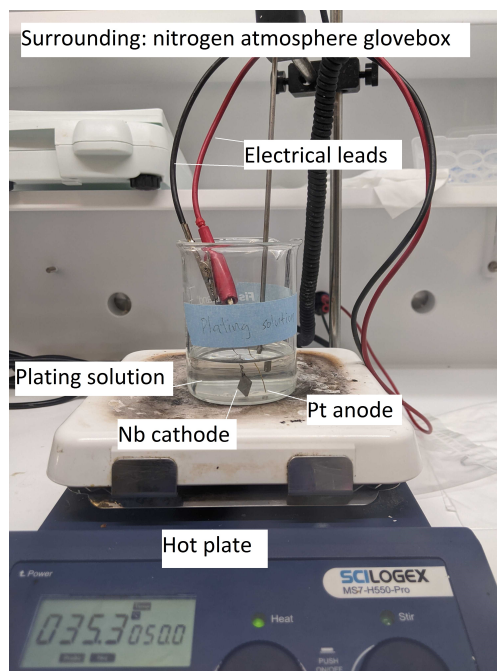


Figure 3: Electroplating setup. Development and optimization are still in progress.

ANALYSIS AND RESULTS

X-Ray Photoelectron Spectroscopy for Niobium

XPS allows not only for identification of various elements present on a sample, but can scan at smaller resolutions of binding energies to allow for analysis of different phases of elements and compounds. This allows for a niobium scan to be performed that can reveal the different oxide phases present. The intermediate phases are more difficult to identify, and this analysis will focus only on Nb and Nb₂O₅.

The XPS system used for sample analysis in this study was a Thermo Scientific Nexsa G2 X-Ray Photoelectron Spectrometer System. Survey scans were performed, as well as element-specific scans of niobium and oxygen for the control, and niobium, oxygen, and gold for the 1 nm gold-deposited sample. Both survey scans and gold scans confirmed the presence of gold on the niobium. CasaXPS analysis software was then used to fit the niobium scan data to a combination of five niobium oxide phases, based on the phases in [7].

Results

Fits are shown in Fig. 4, with the control sample results in subfigure (a) and the gold sample results in subfigure (b). As can be seen, the oxide has not been completely eliminated, but the structure has changed substantially.

CasaXPS can also perform numerical analysis of the relative percentage concentrations of different phases of an oxide. This generates percentage concentrations, as a percentage of the scanned element (for example, the percentage of the Nb scan that was composed of Nb₂O₅). This calculation showed that Nb and Nb₂O₅ show a slight difference in concentration between the two samples (on the order of 1% for Nb and 4% for Nb₂O₅). However, this analysis is preliminary, and identification of the less prominent phases is difficult. The authors cannot yet report with confidence a specific difference in oxide passivation between the control and the gold-plated sample.

CONCLUSION

Given the exploratory and developmental nature of this study, the authors conclude only that XPS analysis shows the presence of gold likely has some impact on the structure of niobium oxide, and that electroplating shows promise as an alternative deposition method for thin films of gold on niobium. Further development and work is in progress for improvement of oxygen concentrations present during the sample preparation process, as well as on optimization of the electroplating setup for thin-film deposition.

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REFERENCES

- [1] G. D. L. Semione *et al.*, “Niobium near-surface composition during nitrogen infusion relevant for superconducting radio-frequency cavities,” *Phys. Rev. Accel. Beams*, vol. 22, no. 10, p. 103 102, 2019.
doi:10.1103/PhysRevAccelBeams.22.103102
- [2] H. Padamsee, J. Knobloch, and T. Hays, *RF Superconductivity for Accelerators*, 2nd ed. Wiley-VCH, 2011.
- [3] Z. Sun *et al.*, “Surface oxides, carbides, and impurities on RF superconducting Nb and Nb₃Sn: A comprehensive analysis,” *arXiv*, 2023. doi:10.48550/arXiv.2305.02467
- [4] T. Oseroff, “Advancing a Superconducting Sample Host Cavity and its Application for Studying Proximity-Coupled Normal Layers in Strong Microwave Fields,” Ph.D. dissertation, Cornell University, 2022.
- [5] T. Oseroff, Z. Sun, and M. Liepe, “Measurements of the amplitude-dependent microwave surface resistance of a proximity-coupled Au/Nb bilayer,” *arXiv*, 2023.
doi:10.48550/arXiv.2305.12035
- [6] Z. Sun, T. Oseroff, and M. U. Liepe, “Electrochemical Synthesis and Materials Design for Superconducting RF Cavities: Sn, Zr, and Au electroplatings on Nb,” presented at the 21st International Conference on Radio-Frequency Superconductivity (SRF’23), Grand Rapids, Michigan, United States, June 2023, paper TUPTB006, this conference.
- [7] J. Halbritter, “On the oxidation and on the superconductivity of niobium,” *Appl. Phys. A*, vol. 43, pp. 1–28, 1987.
doi:10.1007/BF00615201

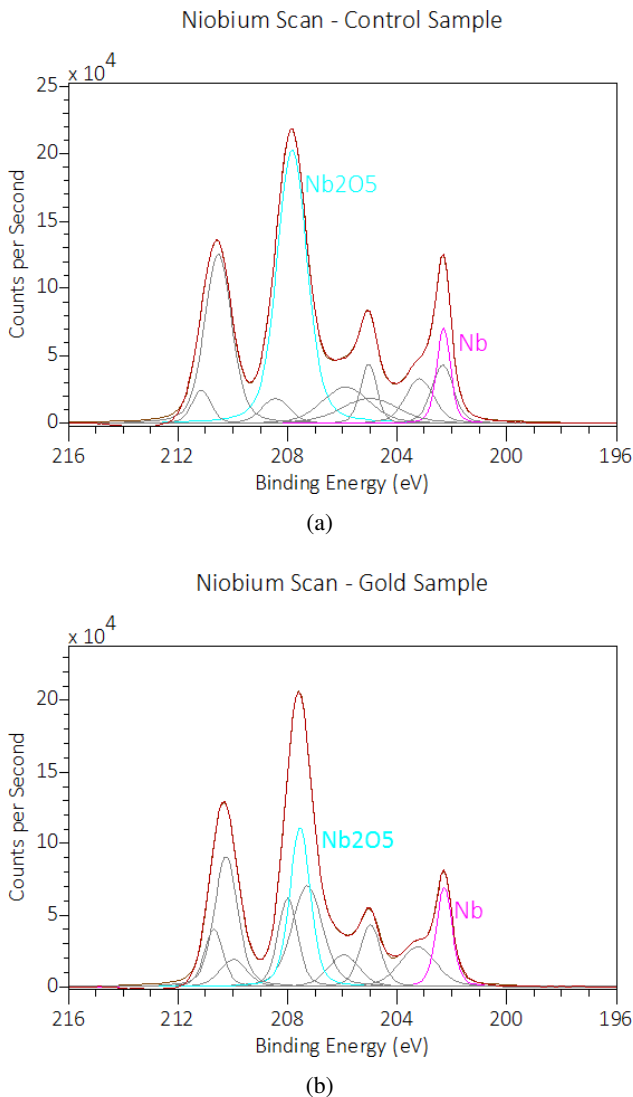


Figure 4: XPS niobium scan analysis of different samples. (a) Preliminary analysis of the control (bare niobium) sample. (b) Preliminary analysis of the 1 nm gold-deposited sample.