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RF CHARACTERIZATION OF NOVEL SUPERCONDUCTING MATERIALS AND MULTILAYERS*

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Abstract

Cutting edge SRF technology is likely approaching the fundamental limitations of niobium cavities operating in the Meissner state. This combined with the obvious advantages of using higher critical temperature superconductors and thin film depositions leads to interest in the RF characterization of such materials. A TE mode niobium sample host cavity was used to characterize the RF performance of 5" (12.7 cm) diameter sample plates as a function of field and temperature at 4 GHz. Materials studied include MgB_2 and thin film atomic layer deposition (ALD) NbN and NbTiN on Nb substrates. These higher critical temperature superconductors all have coherence lengths on the order of a few nm. It is therefore likely that defects on the order of the coherence lengths will cause early flux penetration well before the theorized superheating field of an ideal superconducting surface. Superconductor-insulator-superconductor (SIS) multilayers have been proposed as a mechanism of arresting these early penetration flux avalanches and are therefore studied here as well, using the same NbN and NbTiN films, but over thin layers of insulating AlN on Nb substrates.

INTRODUCTION

For decades niobium has been the sole choice for accelerating SRF cavities. In this time it has been pushed to what are currently believed to be near its intrinsic bulk limitations of efficiency and maximum Meissner state magnetic field. Alternative materials with different properties could provide a means of pushing beyond these limitations. Achieving such a breakthrough could allow for building and operating future accelerators with greatly reduced cost. Materials with properties that make them candidates for this application are difficult to deposit such that these properties are maintained, especially on the large area and curved surface required of an accelerating cavity. Due to limited data available for how these materials respond to the high RF field conditions of an SRF cavity it is difficult for researchers to choose ideal paths for the long and costly path to develop the equipment and techniques needed to obtain these materials on a cavity.

In this paper a specialized resonator capable of exposing flat 5" diameter sample disks to similar conditions to that of an accelerating cavity is used to investigate some of these candidate materials. In contrast to depositions on a curved cylindrical cavity surface it is possible to obtain samples of these materials on flat sample plates using existing equipment and techniques and is done via collaboration with industry specialists. The specialized resonator is not capable of driving the materials at the field strengths that would be necessary to surpass niobium or currently with enough resolution to comment on its agreement with theoretical predictions (The identification of the sources of measurement error and their elimination to improve resolution enough that this is possible are both active research [1]). But it can indicate if a material has the potential to compete in this regime and is worthy of investing in a more costly and rigorous investigation on a cylindrical cavity.

In this work surface resistance measurements as a function of sample magnetic field and temperature at 4 GHz are presented for five samples of promising materials. Plasma enhanced atomic layer deposition (PE-ALD) at Veeco-CNT was used to deposit SIS' and SS' multilayer structures of NbN and NbTiN. These samples have good results and are likely promising candidates for future study. A single MgB_2 sample deposited by reactive vapor deposition at STAR Cryoelectronics was investigated. This sample is one of the first grown by a new deposition system and needs further investigation and development.

MEASUREMENT

All measurements discussed in this work were performed with a sample host cavity shown in Fig 1. The cavity is a modified pillbox designed to achieve maximum field on the sample assuming limitation by thermal quench of the low mean free path niobium host cavity and can theoretically achieve up to 120 mT in a 4 GHz TE_{011}-like mode [2]. The quality factor is measured by observing the decay of energy by monitoring the power emitted from the fundamental power coupler after driving the cavity at resonance and turning off the signal generator. This quality factor is converted into sample surface resistance by combining measurements from two independent sample plates. The first is the sample of interest and the second is a niobium sample plate. This second measurement is used to obtain the dissipation on the host cavity which should be independent of sample plate material and allows for isolating the dissipation corresponding to the sample plate. For a complete discussion of how the measurements are performed and how sample surface resistance and sample magnetic field values are obtained see [1].

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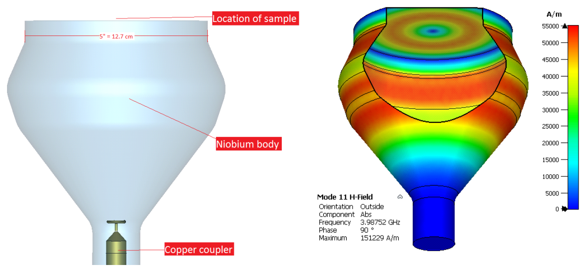

Figure 1: Sample host resonator used for measurements in this paper. The host cavity is made of low mean free path niobium. The sample is a 5" diameter disk that completes the closed cavity boundary at the top of the host and is thick enough to maintain high vacuum in the cavity without imploding (usually 3 mm is used). The cavity is designed for operation in a TE_{011}-like mode excited by a copper coupler. The right image shows the surface magnetic field strength on the surface of the resonator in the 4 GHz TE_{011} mode (the plate area is shown through the cut-out).

MULTILAYER NbN & NbTiN

Interest in NbN & NbTiN

NbN and NbTiN have similar superconducting properties that could theoretically allow for Meissner state operation of an SRF cavity with less dissipation. The superconducting properties are sensitive to growth conditions and can vary over a wide range, making it challenging to obtain a large-area film of high enough quality for use in an SRF resonator. Sputtering has been used to obtain high quality films of both materials and has been used to grow small flat NbTiN samples that have reached surface resistance lower than niobium for small fields [3–5]. This is promising but obtaining these high quality films on the large curved surface of an SRF cavity could be technologically challenging. An alternative deposition process is plasma-enhanced atomic layer deposition (PE-ALD) which has the advantages of atomic-level thickness control, high uniformity over large-area substrates, low temperature depositions, and a more direct extension to deposition onto the curved surfaces of an SRF cavity. Both NbN and NbTiN have been deposited with PE-ALD. The best superconducting properties observed are promising, with higher critical temperatures than Nb, but are lower than the best of other deposition methods [6, 7].

Interest in Multilayers

Superconductor-insulator-superconductor (SIS') multilayers were proposed for SRF applications mainly as a means of increasing maximum SRF cavity accelerating gradient [8]. They consist of a thin superconducting layer S with thickness less than its penetration depth ($d < \lambda_s$), an insulating layer (I) of thickness $\sim 1 - 10$ nm , and a bulk superconductor (S'). The configuration is such that the field is applied to layer S and propagates into the rest of the structure due to the small thickness providing incomplete screening. Screening currents are induced in the bulk layer (S') that produce magnetic fields in the thin layer (S). If $\lambda_s > \lambda_{s'}$ the screening currents in the bulk layer overwhelm those in the thin layer and suppress magnetic field screening in this layer. It is easy to show that this can lead to a modest increase in superheating field (defined as the magnetic field where it becomes energetically favorable for a vortex to enter a superconductor in the Meissner state including the energy barrier associated with the vortex being inside the material) [9–11]. This effect also occurs in SS' bi-layers

The superheating field predicted is relevant for DC fields but for RF fields it is likely that the superheating field is not necessarily the maximum field in which a low-loss Meissner state can be maintained. Indeed the high critical temperature and superheating field materials of interest for SRF applications (and would be desired for use as the thin S layer) are all high κ materials having large penetration depths and small coherence lengths. This small coherence length can cause a defect on the same spatial order as the coherance length to disrupt the phase of the BCS condensate and act as a Josephson junction where Josephson vortices can enter at a Josephson penetration field that seems to be far lower than the superheating field. This has been observed experimentally and agrees with simple theories for YBCO in RF fields [12–16] and there is strong evidence that it should be involved in the determination of current maximum field for bulk Nb_3Sn [17, 18]. Vortices that enter the body of the superconductor dissipate energy of the applied RF field and can trigger a heating avalanche effect resulting in loss of the superconducting phase. The main potential role of an SIS' structure in SRF is for the insulating layer to prevent vortices that enter these high κ materials from penetrating into the bulk and causing such an event [19]. While this can be visualized somewhat intuitively we have not found a rigorous argument for this behavior. Even if the insulating layer does block the flow of vortices there is debate about if it will cause less or more heating [11].

Samples & Results

Multilayer NbN and NbTiN structures were desposited at Veeco-CNT using plasma enhanced atomic layer deposition (PE-ALD) [6, 7].

- 100 nm NbN deposited on bulk Nb
- 100 nm NbN deposited on 2 nm of AlN deposited on bulk Nb
- 100 nm NbTiN deposited on bulk Nb
- 100 nm NbTiN deposited on 2 nm of AlN deposited on bulk Nb

The bulk Nb used in these depositions was electropolished, high pressure rinsed and baked at 800° C to remove gaseous impurities introduced from the acid. The critical temperatures of various materials were measured by flux expulsion. The Nb was all near 9.2 K, NbN was 13.3 K in the

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bi-layer and 13.5 K in the SIS' structure. No flux expulsion was observed for the NbTiN samples which could indicate their deposition conditions caused them to be in a non-superconducting phase or that their critical temperature was less than that of Nb. Material properties were investigated for the NbTiN depositions using witness samples that were grown simultaneously to the sample used for RF testing. Results are displayed in Fig. 2. XPS measurement indicate that the NbTiN layer deposited over the AlN is titanium-rich while the NbTiN layer deposited directly on Nb is niobium-rich. XRD reveals shifts in diffraction peaks. SEM shows the samples have similar morphology. The conclusion of this characterization is that the superconducting properties of the two NbTiN samples could differ significantly.

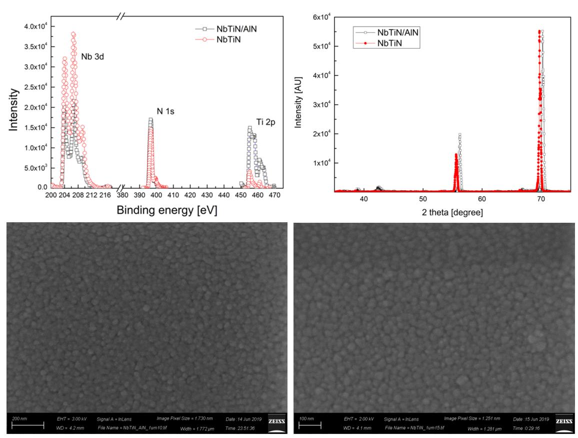

Figure 2: (Top left) XPS data indicating the sample of NbTiN deposited on Nb is niobium-rich while the layer deposited over AlN is N-rich. (Top right) XRD data showing shifted diffraction peaks between the two NbTiN samples. (Bottom left) SEM image of NbTiN on AlN. (Bottom right) SEM image of NbTiN on Nb.

Surface resistance measurements are shown in Fig. 3. Note that reported uncertainties are not complete due to omitting an important systematic error resulting from differences in extra dissipation between the calibration and sample measurements [1]. The measured surface resistances at 4.25 K as a function of peak sample magnetic field are shown including the calibration measurement. Simple estimates of low field surface resistance in multilayer structures predict the measured surface resistance should be very near that of the niobium substrate [9]. The measurements of both NbTiN samples and the SIS' NbN sample are all close to expected values of niobium. The NbN bi-layer is higher than expected and may indicate poor superconducting properties of the NbN or impurities at the Nb - NbN interface. Note that this is speculation and the higher resistance could result from countless possibilities.

The measured residual resistance is displayed in the center image of Fig. 3. The NbN samples were both measured in conditions of higher ambient magnetic fields [1] and it is possible the residual resistance is due to dissipation from flux vortices interacting with the RF fields. The measured

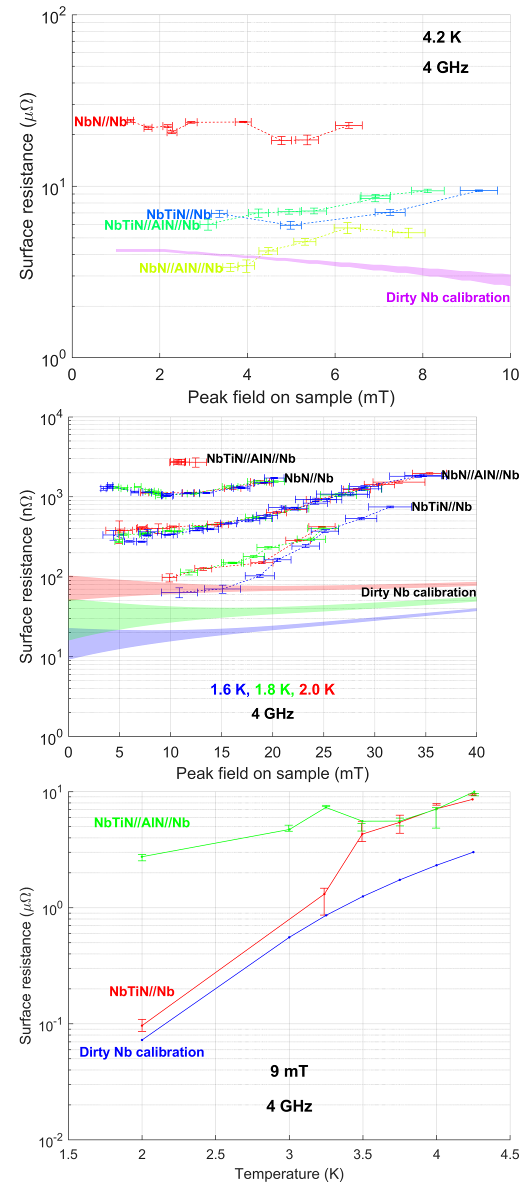

Figure 3: Surface resistance measurements for NbN and NbTiN mutlilayer samples at 4 GHz and the low mean free path Nb calibration measurement. The top and center figures show surface resistance as a function of peak magnetic field on the sample at a temperature of (top) 4.2 K showing the BCS component and (center) 1.6 K - 2.0 K showing the residual resistance. The bottom figure shows surface resistance as a function of temperature at a fixed peak sample magnetic field of 9 mT. Note that the error bars do not account completely for systematic errors and notably ignore uncertainty due to differing amounts of extra dissipation between sample and calibration measurements [1].

surface resistance of these samples increases linearly with applied field which is characteristic of such dissipation [20]. The NbN bi-layer surface resistance shows a slight negative slope at low fields but this could be a measurement artifact.

The NbTiN bi-layer surface resistance increases with field but appears to be less linear and approaches that of the of

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the niobium calibration. It is interesting that 2.0 K and 1.8 K low field measurements are identical while the low field surface resistance at 1.6 K is smaller. It is unclear if this is a true effect of the material or just unaccounted measurement error. A measurement mistake was made on the NbTiN SIS' sample test resulting in the decay data of the 1.8 K and 1.6 K measurements having too few data points for acceptable resolution for reliable decay constant extraction and are omitted in this plot. Despite this it is clear that the low temperature surface resistance of this sample is relatively poor even though its cooling conditions are similar to that of the low resistance NbTiN bi-layer sample.

The bottom plot of Fig. 3 displays the measured surface resistance as a function of temperature at a peak sample magnetic field of 9 mT and again shows curious results for the NbTiN samples. The two are similar at 4.25 K – 3.75 K. At 3.25 K the surface resistance of the SIS' sample jumps while that of the bi-layer drops. Again it is not clear if this is truly an effect of the samples and not a measurement artifact. Reports from the HZB quadrupole resonator measurements on a similar sample of sputtered NbTiN on AlN on Nb describe a similar effect [4, 5].

No comments can be made on the role of multilayers in maximum Meissner state field due to limitations of maximum power input achievable in the test system preventing observations of the limit in the sample. The resonator should be able to operate at higher powers than were used in these measurements but reliable measurements are currently limited to low power due to power dependent issues that are not understood. In this work the maximum field observed on the NbTiN SIS' structure is ~ 20 mT, for the NbTiN bilayer ~ 35 mT, for the NbN SIS' structure ~ 50 mT, and for the NbN bi-layer ~ 22.5 mT. These measurements indicate that PE-ALD NbN and NbTiN seem like viable options to further explore for SRF accelerator application because the surface resistance measurements are not negatively impacted by their presence (if the simple theories are believed) and their field limits have not been observed.

MgB_2

Interest in MgB_2

MgB_2 is a relatively novel superconductor with unique superconducting properties such as the occurrence of two energy gaps of different symmetry. Cooper pair formation is thought to be mediated by phonon interactions. It has a critical temperature of up to 40 K making it the largest of any material realistically considered for SRF application. MgB_2 has displayed promising surface resistances surpassing that of niobium at 4.2 K. For further discussion on MgB_2 see references such as [3, 21].

Sample & Results

In this work a large-area 5" diameter niobium disk was coated with MgB_2 using reactive vapor deposition [22] at STAR Cryoelectronics with a newly developed system. The first sample was a 800 nm thick layer coated on electropolished niobium. SEM images were obtained on a witness sample coated with this deposition. Figure 4 displays one of these images showing deep divisions between MgB_2 regions. This sample had a critical temperature of 40 K measured by flux expulsion but its surface resistance was not measured. The layer was removed using nitric acid and a 400 nm layer was deposited after solvent cleaning and rinsing the niobium plate. This second deposition had a critical temperature of 35 K.

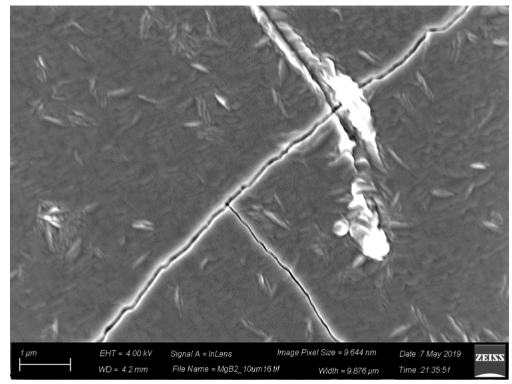

Figure 4: SEM image taken on 800 nm thick MgB_2.

The second deposition surface resistance was measured to have a high residual resistance and is shown at 2 K in Fig. 5. The large dissipation on the sample required moving the coupling antenna further into the cavity where it dissipates more power than in the calibration measurement. This mismatch causes a large systematic error in the sample surface resistance calculation [1] that is not included in the displayed error bars. Despite this it is clear that this sample had a large residual resistance.

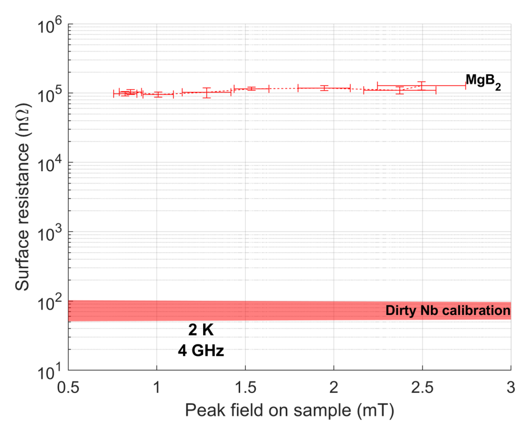

Figure 5: Measured surface resistance of 400 nm thick MgB_2 at 4 GHz and 2.0 K. Error bars do not include a large systematic error resulting from mismatch between extra dissipation in calibration and sample measurements [1].

It is expected that this surface resistance can be improved significantly. The deposition system is still under development and and it will likely improve in the quality and consistency of its films in time. The superconducting prop-

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erties of MgB_2 degrade if it is exposed to moisture. This requires not high pressure rinsing the sample before it is assembled onto the host cavity and it is possible this introduced contamination that caused extra dissipation. In addition to skipping the high pressure rinse the sample was exposed to cleanroom air for two hours during the assembly process and the degradation in material quality during this time could contribute to the resistance. To enable high pressure rinsing and maintain the properties of the MgB_2 film during the assembly procedure the implementation of a passivization layer of atomic layer deposition AlN is being investigated. The SEM image in Fig. 4 reveals deep trenches dividing regions of MgB_2 by distances that are likely on the same order as the coherence length. It is possible the additional residual resistance comes from Josephson coupling through these divisions [13].

CONCLUSION

The surface resistance of PE-ALD NbN and NbTiN multilayers was measured and is found at high temperature to be similar to that of the niobium substrate as is expected by basic theories. At low temperatures resistance seems to become dominated by residual but can approach values similar to niobium. No limiting field was observed on any sample due to apparatus power issues. This indicates PE-ALD NbN and NbTiN multilayers could be viable for SRF accelerator applications. Preliminary results on MgB_2 are presented but have poor surface resistance. It is expected that the use of a passivation layer, further development of the deposition system, and more careful substrate preparation could improve its performance.

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