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SAMPLES FOR 3RD HARMONIC MAGNETOMETRY ASSESSMENT OF NbTiN-BASED SIS STRUCTURES*

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Abstract

In the quest for alternative superconducting materials to bring accelerator cavity performance beyond bulk niobium (Nb) intrinsic limits, a promising concept uses superconductor-insulator-superconductor (SIS) thin film structures that shields accelerator cavities from magnetic flux at higher fields [1]. Candidate materials for such structures are NbTiN as the superconductor and AlN as the insulator. We have demonstrated high quality NbTiN and AlN deposited by reactive DC magnetron sputtering (DCMS), both for individual layers and multilayers. Interface quality has been assessed for bi-layer stacks with 250 nm NbTiN layers and AlN thicknesses from 30 nm down to 1 nm. These SIS structures show continued sharp interfaces with total average roughness under 2 nm.

The H_{fp} enhancement of the films will be examined with a 3rd harmonic magnetometry. The system is being designed and built in a continuing collaboration with CEA Saclay. It can measure 25 to 50 mm samples on a temperature controlled stage. This contribution presents an overview of the design of the 3rd harmonic magnetometer and the material properties assessment of standalone films and multilayer nanostructures.

INTRODUCTION

In the search for a superconductor capable of surpassing the theoretical material limits of Nb, Gurevich [1] proposed the use of SIS layers to reach fields beyond the material limits of bulk Nb. When a superconducting layer S is thinner than its London penetration depth, it sees an enhancement of its lower critical magnetic field H_{c1}, causing the applied field to be attenuated as it passes through the layer. The insulating layer I needs to be thick enough to inhibit Josephson coupling. Then, the layered SIS structure attenuates the magnetic flux into the underlying superconductor. This may allow reaching higher cavity accelerating gradients.

NbTiN and AlN are promising materials for the SIS structures. NbTiN has a superconducting transition temperature (T_c) of 17.3 K for bulk like films. AlN has a lattice parameter of 4.08 Å which is close to NbTiN (4.34 Å). This paper discusses the properties of monolayers of NbTiN and SIS structures with AlN deposited by reactive DC magnetron sputtering. Also, the 3rd harmonic magnetometer is described .

SIS THEORY

SIS structures shield SRF cavities from vortex penetration generated by high magnetic fields. The penetration of a vortex can return the superconductor to its normal state by creating a thermomagnetic avalanche. The insulating layer in the SIS structure helps to disconnect magnetic vortices in the superconducting layer from the bulk superconductor.

The magnetic penetration shielding of SIS structures can be measured by magnetometry. The shielding is dependent on the thickness of the superconducting layer. A thinner superconducting layer can withstand higher fields but more of the applied field will reach the bulk superconductor. A thicker superconducting layer can not withstand high fields but will attenuate the applied field more before it reaches the bulk superconductor. The optimum thickness of the superconducting layer and the insulating layer can be extracted from the contour plot of the achievable peak surface-field without vortex dissipation for NbTiN-AlN on Nb (see Fig. 1). The contour plot was calculated using the equations from [2] and assumes for NbTiN a London penetration depth of 240 nm [3] and a coherence length of 2.2 nm.

DEPOSITION

The films are deposited on different substrates in a UHV system with a base pressure of 10^{-10} Torr. During deposition, the working gas is a mixture of Ar and N. The system is equipped with several DC magnetron sputtering guns with rotatable shutters. A 80/20 (at. wt.%) NbTi target and a pure Al target are used to deposit the SIS structures. The sample stage rotates to face each magnetron for sequential depositions of the different layers.

The substrates used are MgO, Nb and AlN ceramic. MgO is a single crystal with a lattice parameter of 4.36 Å which closely matches NbTiN (4.34 Å). This substrate provides an excellent surface for film growth, yielding high quality NbTiN films. Nb is the substrate of choice for the SRF application of SIS structures. The Nb surfaces are prepared by buffered chemical polishing (BCP) or electropolishing (EP). Films on AlN ceramic represent a worst case scenario

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because of the high substrate roughness (≈200 nm) and distorted grain orientations. Although the NbTiN films with the highest T_c are produced at 600°C, for SIS structures the substrate is heated only to 450°C in order to limit Al diffusion into Nb and NbTiN [4].

MAGNETOMETRY

Magnetometry is used to evaluate the magnetic penetration. Superconducting quantum interference device (SQUID) magnetometry applies a magnetic field to both sides of a sample. This field configuration induces edge effects and is different from the field inside of a cavity. 3^{rd} harmonic magnetometry applies a parallel magnetic field to only one side of the sample and edge effects can be minimized with a large enough size ratio between the sample and the coil used to apply the magnetic field.

3^{rd} harmonic magnetometry systems have three main components: a coil, a sample holder and a cold reservoir. The coil is used to generate a magnetic field parallel to the sample's surface when in the Meissner state. The field producing coil is 5 mm in diameter so a 25.4 mm diameter sample's edge effects will be minimal, and with a 50.8 mm diameter the edge effects will be further minimized. The sample holder maintains a narrow gap of tens of microns between the sample and the coil to detect the 3^{rd} harmonic signal. The cold reservoir protects the sample from heat generated by the coil.

The wire in the coil is 38 american wire gauge (AWG) (about 100 um diameter) and is coated in polyimide (Kapton). The coil generates an AC magnetic field at 1kHz and simulations of the coil show the maximum field around 250 mT [5]. It also acts as a pickup coil to measure the 3^{rd} harmonic induced voltage. The onset of non-linearity in the induced voltage, shows the H_{fp} value has been reached, and allows the reconstruction of the Meissner transition curve over a large set of fields and temperatures.

MONOLAYER AND MULTILAYER FILMS

The samples prepared for measurements with 3^{rd} harmonic magnetometry are monolayers and SIS structures. The monolayers of NbTiN are from bulk like thickness (≈1.5 um) to 52 nm. The substrates used are the different orientations of MgO and electropolished polycrystalline Nb (25.4 mm diameter). The prepared SIS structures have ≈250 nm of NbTiN. AlN layers range in thickness from 22 nm down to 5.6 nm. The EP Nb sample sizes are 25.4 mm and 50.4 mm in diameter. The contour plot shows previous measurements collected from SQUID and 3^{rd} harmonic magnetometry (Fig. 1).

The T_c for NbTiN films deposited on insulating substrates are measured with a four point probe (Kelvin technique) that can measure batches of 32 samples. Films on bulk conductive substrates can not be measured [6] due to the overwhelming contribution of the substrate to the measurement. For these films, we will rely on other methods like magnetometry to estimate T_c.

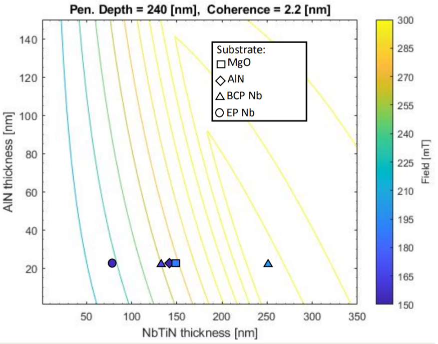

Figure 1: Theory showing the H_{c1} for SIS structures of NbTiN-AlN on Nb [2]. The calculation, for NbTiN, assumes a London penetration depth of 240 nm and a coherence length of 2.2 nm and for the Nb bulk, a London penetration depth of 40 nm. The shapes are previous SQUID and 3^{rd} harmonic measurements with the fill color corresponding to the H_{fp} of the sample.

The T_c of the NbTiN witness films deposited on MgO are around the δ-phase T_c of 16 K (from 16.6 K to 15.6 K). The witness samples of the bilayers have T_c ranging from 14.88 K to 14.39 K. The 3^{rd} harmonic magnetometer, under construction at JLab, will directly measure the T_c of the films on conductive substrates.

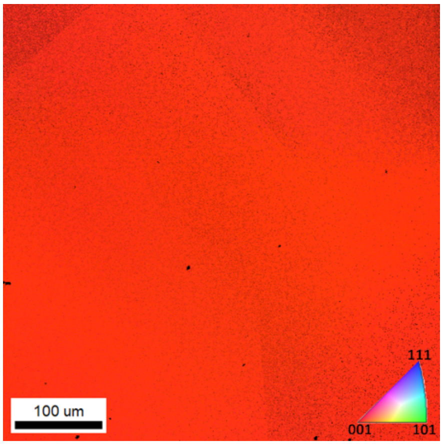

Figure 2: Inverse pole figure (IPF) map from EBSD of a 57 nm NbTiN film on MgO (100). The IPF map is filtered for confidence index above 0.1.

For monolayers, electron backscatter diffraction (EBSD) shows a single crystal film on MgO (see Fig. 2) but lower quality of crystallinity (average confidence index ≈0.30) is observed for NbTiN films on EP Nb, where for films deposited on BCP Nb shows better crystallinity. Multilayer films show the same trend, films on EP substrates show less crystallinity then BCP substrates. This suggests that there are some issues with preparation of the EP Nb surfaces and the EP procedure needs to be revised.

Transmission electron microscopy (TEM) cross sections show the NbTiN and AlN in multilayers on MgO have sharp interfaces (Fig. 3). This multilayer film is not a SIS structure

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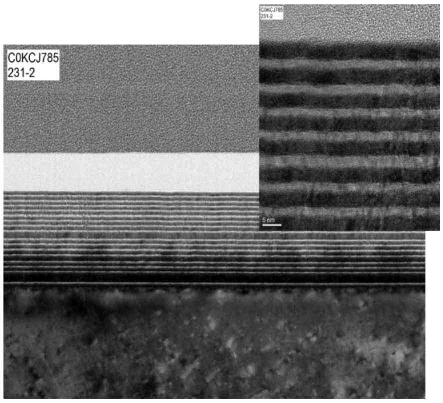

Figure 3: TEM cross section of a multilayer (16 bilayers) sample showing the interface quality and total roughness.

nevertheless, it has similar roughness to bilayers (NbTiN-AlN) on MgO (Fig. 4). Also, the number of layers does not increase the roughness of the films.

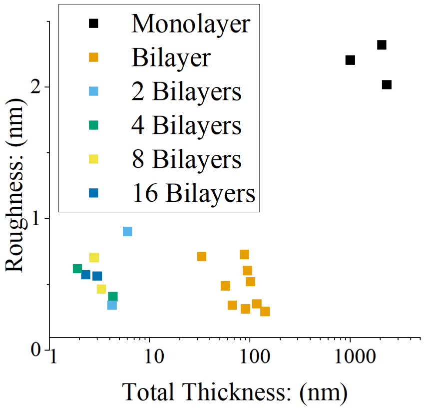

Figure 4: Roughness of NbTiN-AlN multilayers and NbTiN bulk like films on MgO. The number of layers does not increase the roughness of the films.

Bilayers deposited at JLab on MgO, AlN ceramic and EP Nb were measured with SQUID at William & Mary. Also, two of our bilayers on BCP Nb (50.8 mm diameter) were measured at KEK with 3rd harmonic magnetometry. The thicker of the two SIS structures (248 nm of NbTiN with 20 nm AlN) measured the highest increase in H_{fp}, 30% higher than Nb. Using the empirical formula, $H_c(T) = H_c(0)(1-(T/T_c)^2)$ a H_c of 231 mT is roughly estimated (Fig. 1).

CONCLUSION

Thin films for measurement by 3rd harmonic magnetometer have been prepared. The film quality is heavily dependent on the substrate and on the presence of an AlN layer. The prepared SIS samples will be used to investigate the effect of thickness of the insulator, from 22 nm to 5.6 nm, on the H_{fp} of SIS structures based on NbTiN and AlN. H_{fp} enhancement has been observed for SIS structures. In collaboration with CEA Saclay, JLab is building a 3rd harmonic magnetometer in order to measure the H_{fp}. This instrument will also allow T_c measurements across thicknesses and substrates, especially for Nb substrates. Upon completion of the 3rd harmonic magnetometer the films presented here will be measured. Future system improvement will address the material for the coil (annealed Cu wire, more thermally conductive coil coating) to reach higher magnetic fields.

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