CHARACTERIZATION OF SUPERCONDUCTING MULTILAYERS SAMPLES

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Abstract

Best RF bulk niobium accelerating cavities have nearly reached their ultimate limits at rf equatorial magnetic field H ≈ 200 mT close to the thermodynamic critical field H_c. In 2006 Gurevich proposed to use nanoscale layers of superconducting materials with high values of $H_c > H_c^{Nb}$ for magnetic shielding of bulk niobium to increase the breakdown magnetic field inside SC RF cavities [1].

Depositing good quality layers inside a whole cavity is rather difficult but we have sputtered high quality samples by applying the technique used for the preparation of superconducting electronics circuits and characterized these samples by X-ray reflectivity, dc resistivity (PPMS) and dc magnetization (SQUID). Dc magnetization curves of a 250 nm thick Nb film have been measured, with and without a magnetron sputtered coating of a single or multiple stack of 15 nm MgO and 25 nm NbN layers. The Nb samples with/without the coating clearly exhibit different behaviors. Because SQUID measurements are influenced by edge and shape effects we propose to develop a specific local magnetic measurement of H_{C1} based on ac third harmonic analysis in order to reveal the screening effect of multilayers.

INTRODUCTION

Bulk niobium cavities have proven to provide the highest accelerating gradients in superconducting RF cavities for particle accelerator application with values around 40 MV/m. When the accelerating field reaches this value, the magnetic component near the equator is close to the thermodynamic critical field $H_c \approx 200$ mT where niobium ceases to be superconducting.

There are several evidences that the dissipation observed at high field has a magnetic origin because of the BCS term in the surface resistance at high field. As HRF approaches HC, the normal electrons density and RBCS increase due to the effect of current pair-breaking on thermal activation which in turn increases heating, making RBCS nonlinear at high field. So far this nonlinear rf response has only been evaluated for type II superconductors in the clean limit and at low frequency. It shows that at high field the non linear correction increases exponentially with field and temperature, and can give rise to thermal runaway [2, 3].

This model can in particular explain the hot spots observed on cavities where bundles of trapped vortices can produce localized dissipative regions from which heat spreads over several tens of mm. The magnetic/vortex origin of the hot spots have been recently demonstrated [4].

High field non linear dissipation could explain the monopoly of niobium in SRF applications since Nb has the highest H_{C1} value (180 mT at 0 K) among all superconductors: high H_{C1} material is mandatory to prevent early vortex penetration on surface defects (asperities, grains boundaries…). Attempts to use higher T_C and H_{C2} superconductors have failed so far, probably due to their low H_{C1}, that allows early penetration of magnetic vortices resulting in high surface dissipation (for a recent review on that topic see [5]).

MULTILAYERS

A. Gurevich proposed to use composite structures specifically designed for RF accelerating applications based on nanoscale multilayers of superconducting materials with values of $H_C \gg H^{Nb}{}_C$ for magnetic shielding of bulk niobium.

Very high H_{C1} can indeed be achieved with films whose thickness d is smaller than the magnetic penetration depth λ, at least in a configuration where the field is parallel to the surface of the film [1]. So we could use such films to screen bulk niobium and allow much higher field to be reached inside cavities. Bulk niobium is still necessary to prevent perpendicular vortices to penetrate the film and an insulating layer (~ 15 nm) is needed to prevent Josephson coupling between coating layers and Nb substrate. Such structure would be particularly efficient in the case of RF elliptic cavities where the magnetic field is concentrated well inside the cavity and is parallel to the surface.

Deposition techniques

Thin film deposition on curved, large surfaces like the cavities is difficult as it has been widely shown recently (see e.g. [6]). Thus, we are searching for a deposition technique suitable for cavity geometries, i.e. which could provide uniform coating of nano-layers, with sharp interfaces, low densities of defects, including grains boundaries and impurities. As well as low residual stress are necessary. Testing such nano-structures deposited inside cavities would be fairly easy since in this field configuration no side effects are expected.

As a first step we have prepared high quality samples and characterize those using standard measurements. Several deposition techniques can achieve very good quality films in specific conditions, but as usual in the SRF community, their characterization raises several difficulties: most of the classical techniques do not allow predicting their RF behavior. Nevertheless, demonstrating the effective screening effect of nanometer scale NbN films (high H_{C1}) on good Nb samples could initiate the interest of scientific community in searching alternative deposition techniques like the one presented in [7, 8]. To reach this goal, the evaluation of the first penetration field for layered samples as compared to the bulk niobium is of fundamental importance.

In order to produce high quality layered films, we applied the magnetron sputtering technique, an asserted techniques well developed for the preparation of superconducting electronics circuits particularly for Josephson junctions and detectors fabrication.

EXPERIMENTAL DETAILS

We have grown respectively one single layer (SL) R-Al_2O_3/Nb(250 nm)/MgO(y nm)/NbN(x nm), one multilayer (ML) R-Al_2O_3/Nb(250 nm)/[MgO(y nm)/NbN(x nm)]x4. NbN was deposited by dc magnetron sputtering from a 6-inches diameter niobium target in a reactive (nitrogen/argon) gas mixture at 300°C. The same target is used for Nb deposition applying only argon pressure, whereas the MgO layer is RF-magnetron sputtered from a MgO target.. More details on the technique can be found in [9]. The NbN top layer is further RIE etched on a part of the wafer to provide the bulk niobium reference sample (R-SL).

X-ray characterization

Large angle X-rays Diffraction measurements provide information on the crystalline relations between the substrate and the deposited layers. For instance, for sample SL we observed that Nb, NbN, and MgO were all (200) textured at 100% although it was not possible to determine if they were polycrystalline or monocrystalline. In addition the NbN layer was slightly expanded (0.5%) in the (200) direction. For sample ML we observed also a (200) texture for each layer, but the (200) Nb texture is only partial (~ 89 %).

Low angle X-rays reflectivity gave information about thicknesses and interface roughness of the different layers. The measured signal was fitted using Paratt formalism that takes into account the existence of several layers of various electronic densities. Resulting data are summarized in table 1.

Table 1 : summary of the X-rays reflectivity analysis

Sample **SL**	Thickness (nm)	Roughness (nm)	Sample **ML***	Thickness (nm)	Roughness (nm)
Nb	250	1	**Nb**	250	1
MgO	14	1	**MgO**	5	1
NbN	25	1.5	**NbN**	12	1.5

* Sample B has 4 NbN/MgO layers of the same thickness

T_c measurement

The superconducting critical temperature T_c of each sample was measured using a Quantum Design PPMS facility. Measurements of the resistive transition (Fig.1) show that ML exhibits a higher T_c=15.4K than the 250nm Nb substrate layer (H-SL, T_c=8.9K) but lower than SL (T_c = 16.4K). T_c values of NbN films, close to the bulk T_c = 17K), indicate good quality of the films..

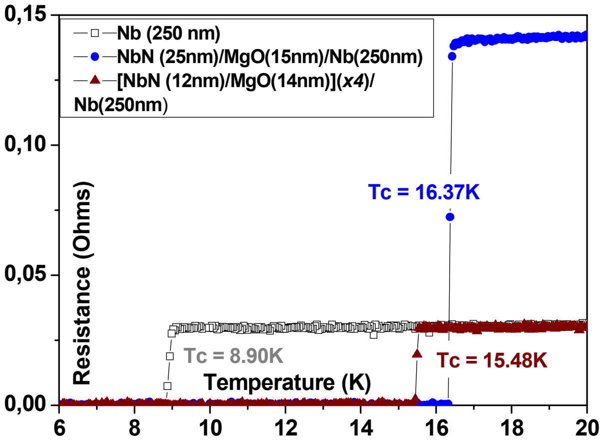

Figure 1: Resistive transition of an Nb film covered or not by a NbN/MgO multilayer

SQUID

SQUID samples were cut to 5x5 mm^2 and glued to high purity quartz thread to be either parallel or perpendicular to the magnetic field. DC magnetization curves M(H) parallel to the sample plane have been measured using a Quantum Design MPMS equipped with a setup which enables simultaneous measurements of M(H) for both transverse and longitudinal field orientation.

SQUID measurement of thin superconducting films with H parallel to the sample plane is fairly difficult to analyze because of the existence of a strong transverse signal as shown by Zhukov *et al.* [10]. Moreover, regardless of any sample anisotropy, a purely geometric effect is to be expected due to edge effects. Magnetic moment is very sensitive to any small disorientation of the sample, which will have a dramatic effect on the signal intensity. This angle sensitivity has been confirmed in ref. [11]. The origin of the transverse signal is clarified as due to strong quadrupolar component that arises from a large perpendicular dipole moment generated by superconducting edge currents confined within the layer's plane. As the quantum design apparatus relies on a dipolar signal, therefore the fitting seems always poor.

The data presented hereafter were optimized for the best possible fit on longitudinal moment that allows, for the moment, qualitative estimation of the magnetic moment. Figures 2 and 3 show the DC magnetization curves of sample SL at 4.5 K and 12 K, and sample SL compared with the reference R-SL at 4.5 K. Curves were normalized such that **M** = -**H** at low field. At 12 K only the 25 nm NbN layer is still superconducting while in at 4.5 K both Nb (250 nm) and NbN are superconducting. The more striking difference lies in the hysteresis behavior: the combination of an NbN layer on Nb obviously strongly reduces vortex penetration compare to Nb single layer sample.

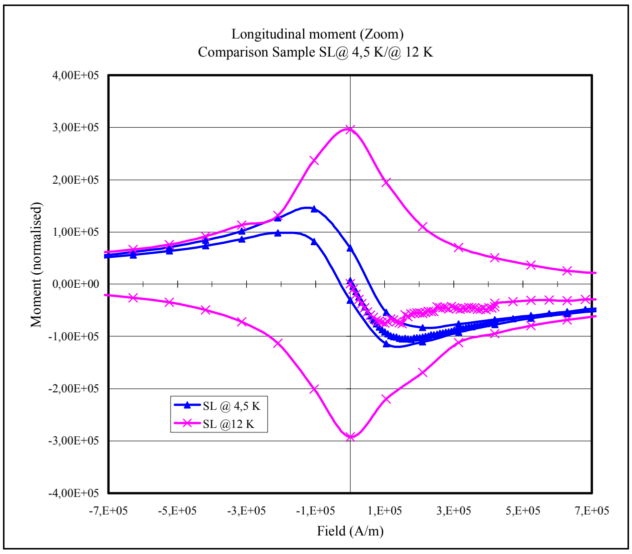

Figure 2: Sample SL at 4.5 K (blue) and 12 K where only NbN is superconducting (pink).

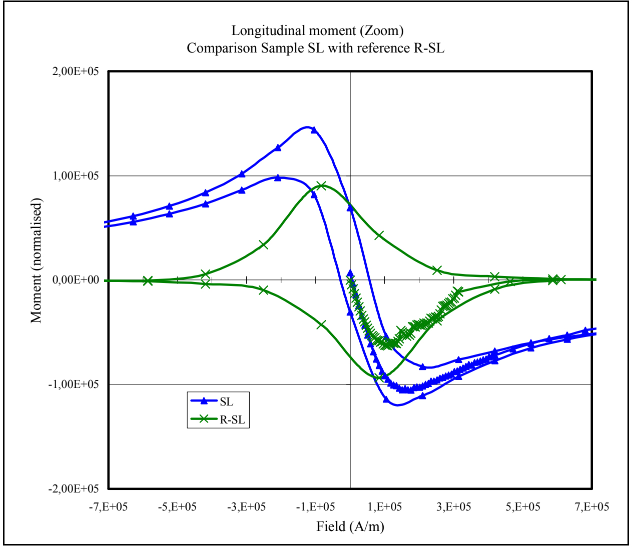

Figure 3: Sample SL at 4.5 K (blue) compared with reference R-SL (green).

Note that the apparent H_{C1} of monolayer appears systematically higher than the reference one (about a factor of two). Nevertheless they do not reach the very high values predicted for d<λ thin films [1]. Several explanations are possible: first we obviously measure the global magnetic behavior of the total composite structures but not directly B_{C1} of the nanometric layer (NbN). Secondly, side, shape and orientation effects are very strong in the SQUID measurement geometry. Moreover, the bulk layer is “protected” only on one side whereas it is immerged inside a uniform field that is configuration somewhat different from the initially proposed model.

Nevertheless it seems possible to fit the results with a more suitable algorithm [12] and we hope to present the processed data in a forthcoming article.

PERSPECTIVES

3rd harmonic analysis

Since SQUID measurements are strongly influenced by orientation, edge and shape effects, we propose to develop a specific local magnetic measurement of H_{C1} based on ac third harmonic analysis as developed in ref.[13]. This technique is based on the hysteretic behavior of the magnetization in the critical state, that gives rise to non zero odd harmonics in the spectrum of the electrodynamic response of superconductors exposed to an ac magnetic field. In fig. 4 (a), $V_3(T)$ is strictly equal to zero in the Meissner phase, but it acquire finite values with a bell-shape temperature dependence in the mixed state below the irreversibility line and it comes back to zero in the flux flow and normal state regimes.

First measurement on 80 nm niobium multilayers seems to show a clear increase of B_{C1} in perpendicular field configuration [14]. In our case we need to develop in addition an experimental set-up where the field configuration is similar to cavities, i.e. parallel to the surface.

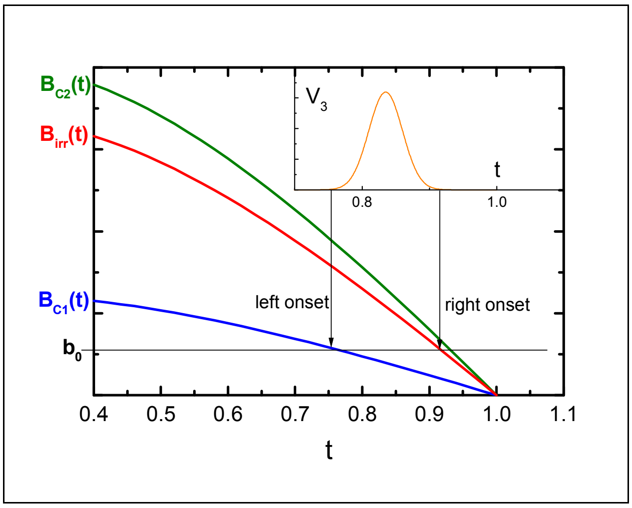

Figure 4: principle of third harmonic analysis (see text for details). t =T/Tc

Depositing technique

We need to determine how the screening properties evolve in more realistic situations. We plan to test samples deposited on bulk monocrystalline and polycrystalline niobium with the same technique or with alternative techniques like ALD, which might be easier to develop for full cavity deposition.

CONCLUSION

We have presented the superconducting properties of composite structures specifically designed for RF accelerating applications. In particular we have analyzed SL and ML multilayer by DC SQUID magnetization measurement in a parallel field configuration. We have shown a very promising behaviour of these composite structures: the first critical field of a multilayer is enhanced by a factor ~ 2 and the vortex penetration is partially suppressed as it has been inferred from the observed reduction of the area of the hysteresis magnetization curves.

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