

RF CHARACTERIZATION OF NIOBIUM FILMS FOR SUPERCONDUCTING CAVITIES *

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Abstract

The surface resistance R_S of superconductors shows a complex dependence on the external parameters such as temperature, frequency or radio-frequency (RF) field. The Quadrupole Resonator modes of 400, 800 and 1200 MHz allow measurements at actual operating frequencies of superconducting cavities. Niobium films on copper substrates have several advantages over bulk niobium cavities. HIPIMS (High-power impulse magnetron sputtering) is a promising technique to increase the quality and therefore the performance of niobium films. This contribution will introduce CERNs recently developed HIPIMS coating apparatus. Moreover, first results of niobium coated copper samples will be presented, revealing the dominant loss mechanisms.

INTRODUCTION

Niobium coated copper cavities have several advantages compared to bulk niobium cavities: The higher thermal conductivity of copper avoids thermo-magnetic break downs, the BCS resistance is lower due to a shorter electron mean free path and niobium films are less sensitive to trapped magnetic flux. In addition, demanding less niobium reduces material costs [1]. The current limitation is that niobium film cavities usually show satisfactory Q_0 below accelerating gradients of 10 - 15 MV/m, although it has been shown that higher gradients can be achieved. In addition, niobium film cavities exhibit usually a stronger Q decrease with accelerating field compared to bulk niobium cavities [2]. Therefore, the application of niobium film cavities is attractive for relatively low accelerating gradients of a few MV/m, as used at CERN for LEP II and the LHC due to the lower R_S at 4.2 K. The aim of this study is to expand the possibility of using coated cavities instead of cavities made from niobium sheet for accelerator applications demanding gradients above a few MV/m.

SPUTTERING TECHNIQUES

In the basic configuration, plasma is created applying a voltage between an anode (substrate) and a cathode (target). This electric field is necessary to attract ions towards

the target and obtain a bombardment which will create an emission of target atoms. Changing the working parameters (such as the working pressure, the plasma gas, the applied voltage, etc.) and using a magnetic field combined with the electric field we can optimize the sputtering yield. In this case, it is referred to as Magnetron Sputtering (MS) and an increase in the deposition rate and the ionization efficiency can be achieved. The MS machine can be controlled by DC or RF depending on the target (the former for conductive targets while the latter for insulator target). Figure 1 represents schematically a usual dcMS configuration.

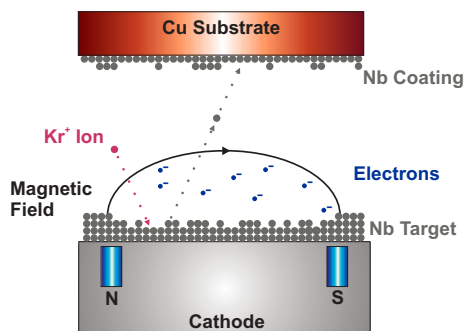


Figure 1: Dc magnetron configuration.

As it can be seen in the picture, the magnetic field helps keeping the electrons trapped close to the target surface. Doing this we obtain a denser plasma and a higher power density on the surface of the target, enhancing the ionization of the sputtering gas and the deposition rate. The energies of the sputtered neutrals are in the order of chemical bonds (1 - 10 eV) which allow the creation of a denser film compared to the diode configuration. The main characteristic of this technology is the I-V curve:

$$I_d = k_d V_d^n \quad (1)$$

where I_d is the cathode discharge current; k_d is a constant depending on the working parameters and V_d is the cathode discharge voltage; n is a constant between 3 and 15 and depends on multiple factors, such as the working parameters. A critical point for the deposition of thin films is how to correlate the structure of the film with the plasma parameters. This has been clarified by Thornton [3] (and later by A. Anders) in his widely used diagram, which can

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be seen in Figure 2. By this diagram it can be understood how the energy of the impinging ions or the temperature of the substrate can influence the film growth. Since in dcMS we obtain very few target ions (fraction $< 10^{-2}$), in order to reach a fine-grained structure, we must increase the substrate temperature.

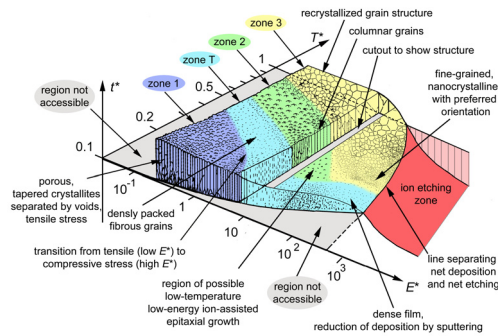


Figure 2: Structure zone diagram [4]. Copyright A. Anders, with kind permission of the owner.

This is one of the reasons that brought about the development of HIPIMS. The goal was to provide a higher energy to the target in order to increase the plasma density. HIPIMS offered the perfect solution, since only a change in the power supply is necessary to transform a dcMS system into a HIPIMS, without any need of major hardware redesign. By biasing the substrate we can control the direction of the particle flux and increase their energies, and enhance the possibility to coat complex substrate geometries. Furthermore, the highly energetic ions produced by HIPIMS have a dramatic effect on thin film growth, improving the adhesion and densification of the film [5, 6]. Working in a pulsed mode allows to keep the average power low (in the same range of dcMS), while the low duty cycle allows the use of very high power (in the order of a few kW/cm²) for very short time. This improves the ionisation of the sputtered flux (ionized fraction in excess of several 10%) and film quality. For these reasons HIPIMS is one of the most promising techniques for improving common magnetron sputtering used in many industrial and research processes for thin film deposition.

SAMPLE PREPARATION

So far, there are no RF results available on HIPIMS coated cavities or samples. In a first step, we produced and tested a dcMS sample in order to have a reasonable reference. Prior to the dcMS coating an oxygen free copper substrate was degreased, chemically polished with SUBU (H₃NO₃S, H₂O₂, n-butanol and ammonium citrate) and electron-beam welded to a BCP treated bulk Nb sample body. After the coating, the sample was rinsed with ultra-pure water and ethanol. In order to keep oxidation of the surface at a minimum, the sample was dried and then stored under filtered nitrogen until it was mounted in the Quadrupole Resonator.

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THE QUADRUPOLE RESONATOR

The Quadrupole Resonator displayed in Figure 3 is a compact apparatus designed for RF characterization of superconducting samples of 75 mm diameter over a wide parameter range. It is a four wire transmission line resonator exiting TE_{2x} modes at 400, 800 and 1200 MHz.

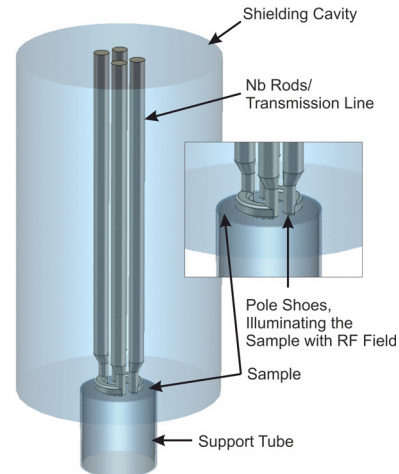


Figure 3: The Quadrupole Resonator.

The magnetic contribution is almost identical for all resonance frequencies, but the electric field increases linearly with the resonance frequency. The samples are equipped with a dc heater enabling to heat them up to 12 K and four temperature diodes. Details concerning the measurement capabilities can be found in [7]. The measurements are based on an RF-DC compensation method: Starting at the helium bath temperature, the sample is heated up to a certain temperature of interest and the heater power P_{DC1} is measured. When the RF is switched on, the sample temperature increases due to dissipation. The heater power (P_{DC2}) is reduced until the temperature of interest is reached again. The dissipated power from the RF field is the difference between the heater powers with RF on (P_{DC1}) and off (P_{DC2}) and the surface resistance R_S is proportional to it:

$$R_S = \frac{2 \cdot (P_{DC1} - P_{DC2})}{\int_{\text{Sample}} |\vec{H}|^2 dS} \quad (2)$$

The integrated magnetic field \vec{H} over the sample surface S in the denominator is directly proportional to the transmitted power P_t with a factor derived from a simulation of the RF field configuration on the sample surface [8]. P_t , P_{DC1} and P_{DC2} are measured directly.

RESULTS

In the following, results on the first dcMS sample will be presented. R_S was measured as a function of temperature with an applied RF magnetic field of 12 mT. It is displayed in Figure 4.

$R_S(T)$ was fitted as a sum of a temperature independent residual part R_{res} and a BCS contribution:

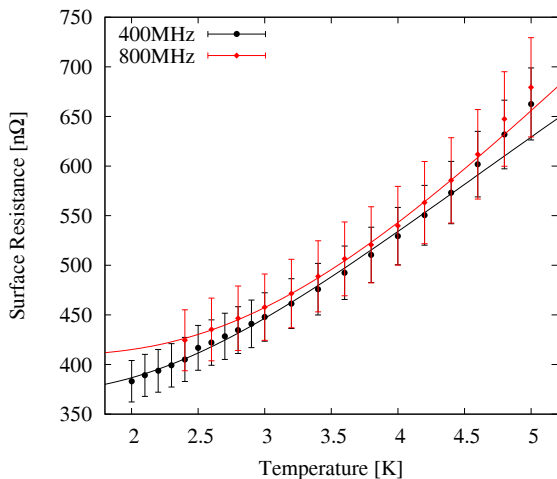


Figure 4: $R_S(T)$ for 400 and 800 MHz.

$$R_S(T) = R_{res} + A_{BCS} \cdot \exp\left(-\frac{\Delta}{k_B T}\right) \quad (3)$$

with the Boltzmann constant k_B and the superconducting energy gap Δ . This fit is only valid for temperatures below $T_c/2$ and is therefore only applied to $T < 4.6$ K. The factor A_{BCS} originates from the BCS theory and should increase quadratically with frequency. In contradiction to an expected factor of 4, we found A_{BCS} for 800 MHz being only a factor of 1.75 higher compared to A_{BCS} for 400 MHz. A possible explanation is that the RF field interacts with the copper substrate. In that case the anomalous skin effect would apply, yielding a factor of about 1.59 ($\sim f^{(2/3)}$).

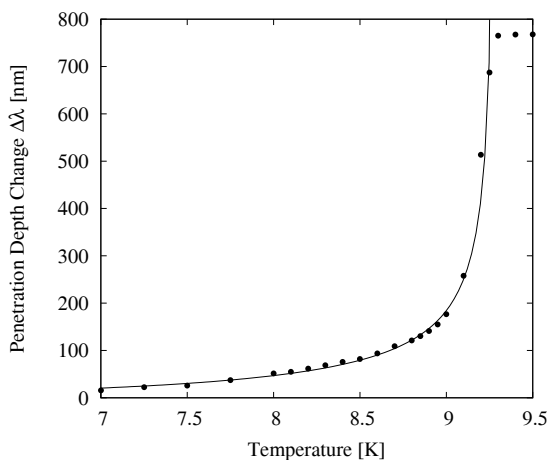


Figure 5: Change of the penetration depth $\Delta\lambda$ as a function of temperature.

In order to derive the penetration depth of the sample, it was warmed up with the dc heater and the resonance frequency was measured. The shift of the penetration depth $\Delta\lambda(T)$ can be calculated from the frequency shift and is shown in Figure 5. The penetration depth at 0 K, λ_0 , can be fitted via [8]:

$$\lambda(T) = \frac{\lambda_0}{\sqrt{1 - (T/T_c)^4}} \quad (4)$$

The fit yields $\lambda_0 = (93 \pm 3)$ nm for the penetration depth and a critical temperature of $T_c = (9.28 \pm 0.02)$ K. Both values are consistent with a superconducting niobium film, although a corresponding RRR of about 3 is a sign of a low quality film (typical RRR values are 10 to 20 [2]). A source of contamination could have been a not sufficiently clean handling between the SUBU treatment and the coating or originating at the critical Nb/Cu electron beam welding.

CONCLUSION

The development of niobium films has been recently relaunched at CERN. The aim is to extend the application space to higher accelerating gradients using the HIPIMS technology. The current work focuses on reproducing results achieved with the dcMS technique, for example 20 nΩ at 4.2 K for the 352 MHz LEP cavities [1].

The Quadrupole Resonator is an ideal tool to do fundamental studies on new coating technologies due to the wide accessible parameter range. It was used to test a first dcMS sample which was not performing up to state of the art. Adjustments concerning clean handling of the substrate have been already put in place.

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REFERENCES

- [1] S. Calatroni. *Physica C*, 441(1-2):95–101, July 2006.
- [2] V. Arbet-Engels, C. Benvenuti, S. Calatroni, P. Darriulat, M.A. Peck, A.-M. Valente, and C.A. Vant Hof. *Nucl Instrum Meth A*, 463(1-2):1–8, May 2001.
- [3] John A. Thornton. *J Vac Sci Technol*, 11(4):666, July 1974.
- [4] Andr Anders. *Thin Solid Films*, 518(15):4087–4090, May 2010.
- [5] A. P. Ehiasarian, A. Vetushka, Y. Aranda Gonzalvo, G. Safran, L. Szekely, and P. B. Barna. *J App Phys*, 109(10):104314, 2011.
- [6] A. P. Ehiasarian, J. G. Wen, and I. Petrov. *J App Phys*, 101(5):054301, 2007.
- [7] Tobias Junginger. PhD thesis, University of Heidelberg, Germany, July 2012.
- [8] T. Junginger, W. Weingarten, and C. Welsch. In *Proceedings of the 14th Conference on RF Superconductivity*, pages 130–136, 2009.