DIFFERENT SPUTTERING CONFIGURATIONS FOR COATING 1,5 GHZ COPPER CAVITIES

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Abstract

In the framework of the attempts to densify the sputtering discharge, two different sputtering configuration are reported:

- a mixed bias magnetron sputtering technique has been explored for depositing niobium into 1,5 GHz copper cavities. Results are presented and compared with the standard CERN technique. The superconducting and high resolution morphological and mechanical properties of niobium films sputtered onto the inner walls of electropolished cavities, have been studied as a function of the deposition technique. Even if niobium films coated with the bias technique show a higher density and Tc similar to the other films, they don't present higher RRR values. Preliminary RF tests are presented.
- post Magnetron sputtering in thermoelectric emission regime have been investigated and is under improvement in order to increase the RRR values of sputtered Nb: superconducting and structural properties of the obtained films have been measured to check the technique capability and its possible application for coating cavities.

INTRODUCTION

In order to save building costs of superconducting resonant cavities for particle accelerator, the development of sputtering technique for the deposition of Niobium (Nb) superconducting films onto Oxygen Free High Conductivity Copper (OFHC Cu) cavities has started in the 1980's at CERN [1].

While bulk niobium is usually adopted for conventional RF superconductiong cavities [2], LEP (CERN) [3] and ALPI (INFN-LNL) [4] cavities consist of OFHC Cu, internally sputter-coated with a thin niobium layer (about 2μm). Further induced advantages of thin film approach are the better thermal stability of the OFHC Cu, the insensitive to the trapped earth magnetic field and possibility of applying high T_c [3].

The possibility to enhance the plasma density have beeen explored in order to increase the sputtering rate and then to reduce the impurities in the Niobium sputtered film to finally obtain a high purity Niobium coating. First of all a positive electrode has been inserted in the standard magnetron configuration in order to increase the plasma voltage, the plasma density and promote the ion bombardment of the growing film. This configuration has been called Bias Magnetron Plasma technique (BMP) [5,6].

Furthermore, in order to enhance the plasma density, we tried to booster the plasma with the injection of external electrons. We first planned to utilize an external source of electrons for the plasma density enhancement using a hollow cathode source [7] coupled to a standard planar 2" magnetron [8]. Then we improve the cylindrical sputtering configuration in order to apply the post magnetron sputtering in thermoelectronic emission regime to the cavity coating. This is the High Ionized Plasma technique (HIP).

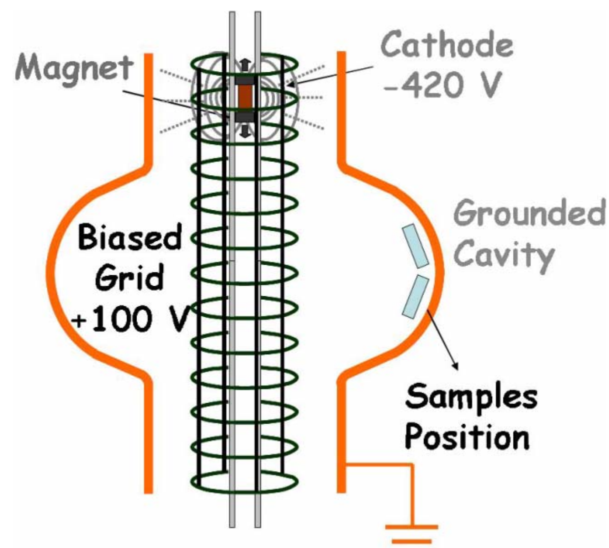

Figure 1: Scheme of the Bias Magnetron Plasma configuration

Sample have been coated at the Superconductivity Laboratory of the National Institute for Nuclear Physics in Legnaro (Padua, Italy) while morphological and microstructural analysis and Coatings Intrinsic Hardness and Elastic properties evaluation have been performed at Mechanical and Industrial Engineering Department of University of Rome "Roma Tre" (Rome, Italy).

EXPERIMENTAL DETAILS

Biased Magnetron Plasma apparatus

The Biased Magnetron Plasma technique coatings under examination have been obtained by a Cylindrical Magnetron Sputtering plant. All details about the standard Cylindrical apparatus and its modification to apply the Bias Magnetron Plasma techniques are well described in references 6 and 9. Coating parameters are summarized in Table 1.

Coating on ultrasound cleaned quartz substrates have been performed in several runs in order to obtain superconductive properties measurement. During the same runs OFHC copper substrates have been coated. Copper 5x10 mm samples were preliminary cleaned by standard electro-polishing in a Phosphoric acid and Buthanol mixture, then etched in a Sulfammic acid, Ammonium Citrate, Buthanol and Hydrogen Peroxide mixture (SUBU). All samples have been passivated in the end. This etching sequence is the cavity usual treatment and it has been reproduced on small samples.

The same copper 1,5 GHz monocell cavitiy has been chemical treated and coated with the two techniques: first time with the CM parameter and the second time with the BMP parameter collected in Table 1.

Hereinafter the cavity treatment sequence is listed:

- Stripping from the previous coating
- 1 hour electropolishing
- 10 minutes SUBU
- 10 minutes passivation
- High Pressure Water Rinsing (HPWR) 1 hour at 100 bar [10]
- Coating CM or BMP keeping the cavity at 150°C.
- HPWR 1 hour at 100 bar
- RF test [11,12]

High Ionized Plasma apparatus

For setting the High Ionized Plasma apparatus first a study of the hollow cathode and cylindrical cathode in the thermoelectronic emission regime has been performed. The Hollow Cathode is a system made of a low working function metal that use the thermoelectronic emission to ionize the gas that flux inside it, creating a plasma. A detailed description of all preliminary tests that drove to the building of the High Ionized Plasma apparatus is described in reference 13.

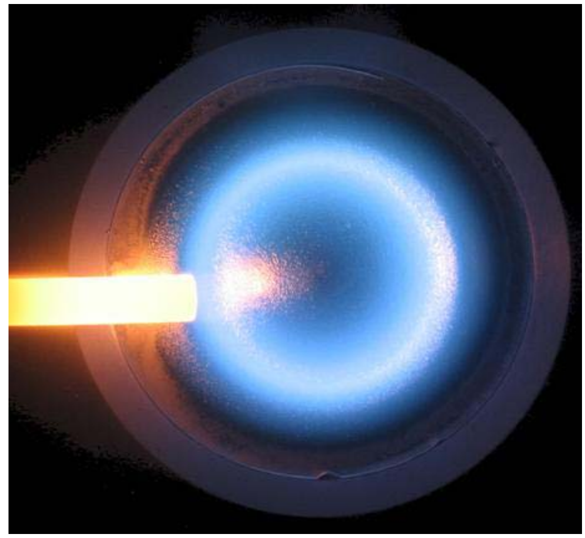

Figure 2: Picture of the inject hollow cathode magnetron. The hollow cathode directly injects electrons inside the plasma ring of the planar magnetron.

For the deposition we used quartz samples fixed in a sample holder with the same shape of the cavity. In this first experiment we've deposited Nb thin film only in the centre of the cavity (the iris). We are currently studying the feasibility of a deposition system where a moving cathode will sputter the entire inner surface of a 1,5 GHz Cu cavity. The sputtering parameters are collected in Table 1. For the high temperature reach, the cavity is cooled by a flux of compressed air.

	CM	BMP	HIP
Ultimate pressure (10^{-9} mbar)	2	2	1
Deposition pressure (mbar)	$2x10^{-3}$	$2x10^{-3}$	$2x10^{-3}$
Cathode current (A)	7	7	15
Cathode power (kW)	1,54	1,86	4,05
Bias Voltage (V)	0	100	0
Process time (min)	20	20	16

Table 1: Deposition parameters for Cylindrical Magnetron Standard technique, Biased Magnetron Plasma technique and High Ionized Plasma technique.

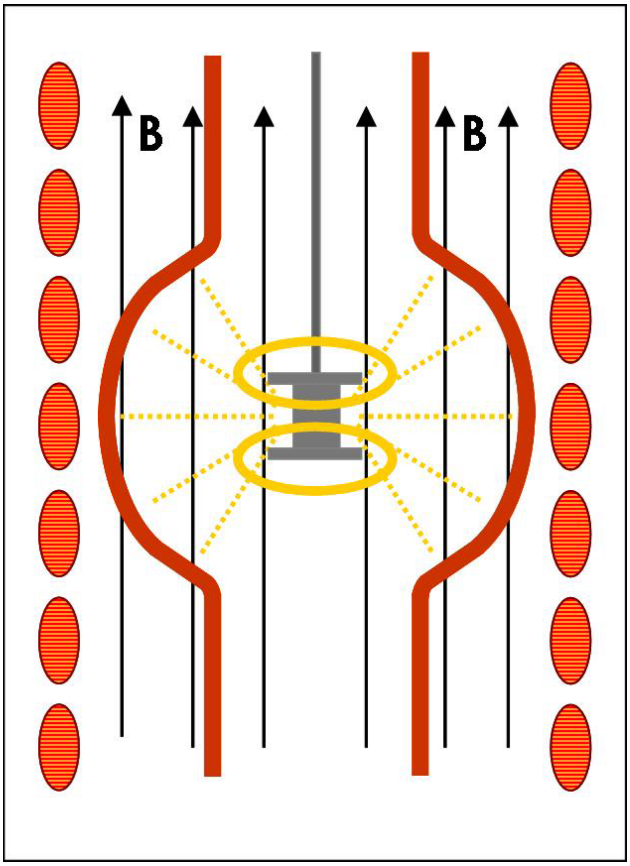

Figure 3: Scheme of the High Ionized Plasma apparatus.

Superconducting properties measurements

The Residual Resistivity Ratio β and the transition temperature Tc of sputtered films on insulating quartz is measured with the four point method to eliminate the effect of possible contact resistance at the contact point between niobium films and the pins of the measuring device. A Visual Basic program has been written to acquire the resistance curve from 300 K to the transition temperature. Applying to the sample a current of 10mA the voltage is measured. The same method is used for the temperature probe, applying a direct current of 10mA the resistance is measured and the temperature is obtained from the calibration curve. The low temperature resistance of the sample is fixed at 10K and the RRR calculated with the following formula:

$$\beta = \frac{R(300K)}{R(10K)} = 1 + \frac{R_{ph}(300K)}{R_{res}} = 1 + \frac{\rho_{ph}(300K)}{\rho_{res}} \quad (1)$$

$\rho_{ph}(T)$ is the resistivity due to the electrons-phonons collision, decreasing with the temperature; ρ_{res} is the residual resistivity and it doesn't depend on the temperature. ρ_{ph} (300K) is 15 μΩ·cm and it is a costant while the phononic resistance at 10 K is negligible.

RRR is an immediate estimate of the film quality: the higher is the value, the more pure is the material. In addition it is an adimensional parameter that doesn't care of the geometric dimensions: keeping them constant with the temperature, they get eliminated in the ratio.

The transition temperature Tc ± ΔTc is calculated from the resistance curve with the following formula

$$Tc = \frac{T_{(90\%)} + T_{(10\%)}}{2}$$
$$\Delta Tc = T_{(90\%)} - T_{(10\%)} \quad (2)$$

T(90%) is the temperature corresponding to the 90% of the resistance before the transition;

T(10%) is the temperature corresponding to the 30% of the resistance before the transition.

ΔTc is the error on the critical temperature estimation.

RF measurements have been performed with the cryogenic and electronic apparatus located at the Superconductivity Laboratory of the National Institute for Nuclear Physics in Legnaro (Padua, Italy). A control program for Q-factor measurement [14,15],developed in Visual Basic, has been updated to control the several RF apparatus connected to the computer [15] (i.e. Peak Power Analyzer, RF Generator, etc.) to measure cavity at 1.5 and 6 GHz. Heads and system calibration has been carried out using the E4417A EPM-P Series Agilent Power Meter Head as reference.

The cryogenic apparatus enables to execute RF test at 4.2 K and 1.8 K.

Surface Morphology and Coatings Microstructure

Thickness measurements were performed using a Dektak® 8 Advanced Development Profiler Veeco. The profiler scans the surface with a constant load fixed at 10 mg. Six different scans, 2 mm long, have been executed for each sample in three different positions near the area where the quartz substrate is exposed. Here each step from niobium to quartz gives the film thickness. The average of the six measurements is considered as the film thickness and the root mean square as its error.

In-plane morphological characterisation of coatings were performed using Atomic Force (AFM) and Scanning Electron Microscopy (SEM) techniques.

Surface roughness measurement was also carried out by Contact-Mode AFM analysis: roughness parameters (Roughness Average, Ra, Root Mean Square, RMS, Peak to Peak) were obtained after statistical analysis on a total scanned area of about 750 μm^2.

Microstructure of coatings (in-plane and cross-section coating investigations, coating thickness, crystallite size and growth direction analysis, surface and layer interface analyses) was investigated by using Focused Ion Beam (FIB) techniques: FIB systems [16] utilize a finely focused beam of gallium ions (Ga+) operating at low beam currents for imaging and at high beam currents for site-specific milling. While FIB techniques therefore appear to be an ideal tool for microstructural investigations of thin films, such as superconducting Nb coatings, they have so far been used for microstructural investigations of sputtered Nb thin films only rarely.

One example of FIB applications is the study to reveal the difference in the structure of 'standard' polycrystalline Nb films grown on oxidized copper surface and epitaxially grown ones deposited on oxide-free copper [1].

In the present study, FEG-SEM microstructural investigations have been performed after FIB sectioning, while interfaces structure and microstructure and thickness of the surface oxide layer have been investigated by TEM after FIB sample preparation.

Coatings Intrinsic Hardness and Elastic properties evaluation

Micro-Hardness measurements were performed using Vickers standard indentation tests, with loads ranging from 100g (1N) down to 0,5g (0,005N). The obtained value for each load corresponds to the mean of six measures. In order to extrapolate film intrinsic hardness, the Jonsson & Hogmark [17] and the Chicot & Lesage [18] models have been used. Work hardening behaviour was taken into account as well, by adopting the Indentation Size Effect (ISE) Meyer's law [18,19], which describes the relation between load and size of indentation:

$$P = k \cdot d^n \qquad (3)$$

Where P is the applied load, d is the indentation size, k and n are constants.

Eq. (3) can be rewritten as a function of Vickers hardness:

$$HV = HV_0 \cdot d^{n-2} \qquad (4)$$

Coefficient n is known as Meyer's or hardening index, generally lower than 2,0 for pyramidal indenters [20], while constant HV_0 has the physical meaning of the Vickers hardness for an infinitesimal applied load.

Coating Elastic recovery was evaluated by 3D AFM reconstruction [21] of residual indent volume and applying Lesage model (all details reported in [22;23]), by which an evaluation of the elastic recovery is performed after comparison between the actual volume of the residual indent, measured by AFM analysis, and its theoretical value on Vickers indentation marks.

The main objective of the mechanical characterisation was to determine statistically significant differences in surface properties for Biased and Unbiased MS-PVD Nb films, in order to find out their correlation with coating microstructures.

It has to be underlined that elastic properties of coatings are always strongly microstructure-dependent [24], being elastic response correlated to columnar grain dimension and orientation, film density, pores, micro-cracks, residual stress. For these reason calculated values have to be considered as an estimation of the Elastic response of the coatings. In the case of MS-PVD Nb coatings for superconducting application, the evaluation of coating Elastic properties is performed with the main aim of describing the correlation between coating Elastic properties, microstructural aspects (such as density and grain dimension) and coating functional properties.

RESULTS

Biased Magnetron Plasma

Only niobium films on quartz have been analyzed with the resistive methods. Two runs of the bias technique show a similar values of RRR for all films around the equator (Figure 4). Unbiased technique shows such a difference in RRR due to the different time of sputtering (15 min for Run 30 and 20 min for Run 31). Time of sputtering has a strong influence on the film purity and consequently on RRR: the higher is the sputtering time, the purer is the gas in the chamber and the cleaner is the atoms flux arriving on the substrate, due to the getter property of the growing niobium.

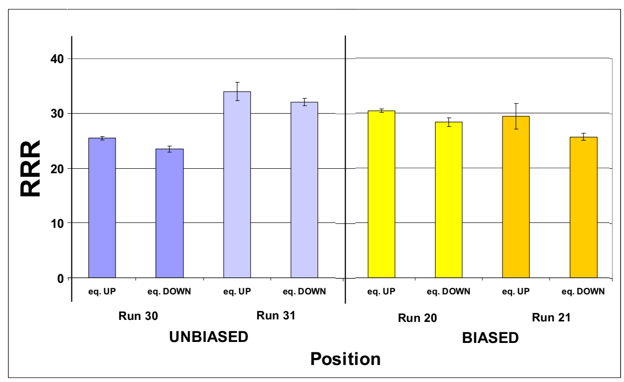

Figure 4: RRR values of the niobium film on quartz sample, for different positions around the equator (eq. UP, eq. DOWN) and two different run for each deposition techniques (Biased, Unbiased).

Comparing the results for the two techniques and the same sputtering time (Run 20, Run21 and Run31), unbiased samples shows higher RRR around 34.

In addition resistive measurements show that Biased films have higher critical temperature than unbiased films even if most of them are included in the error bars. Equator up and down films don't show significant differences for all tests. All film reveal a critical temperature higher than the bulk niobium one.

In-plane SEM (Figure 5) analysis at different magnitudes shows a needle-shaped grain structure from both biased and unbiased coatings, with grain average longitudinal dimension of 350 nm.

AFM images confirm a crystallite elongated structure with an average dimension of 300-350 nm. Superficial roughness results are reported in Figure 8: bias technique produce rougher film than the standard one[23].

Figure 6 show FEG-SEM observation after FIB sectioning of (a) biased and (b) unbiased (CERN) thin Nb films on oxidized copper [25]. A coarser microstructure have been observed for the unbiased coating, with large columnar grains that extend all the way through the film depth; finer columnar grains are visible in the case of the biased coating, which also directly influence the surface roughness.

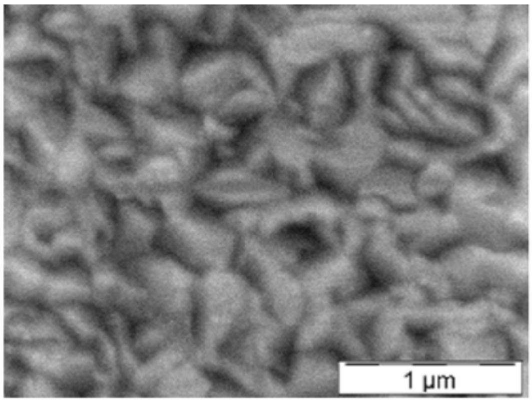

Figure 5a: Nb on Cu BIAS type (SEM SE 20 kV 40000x)

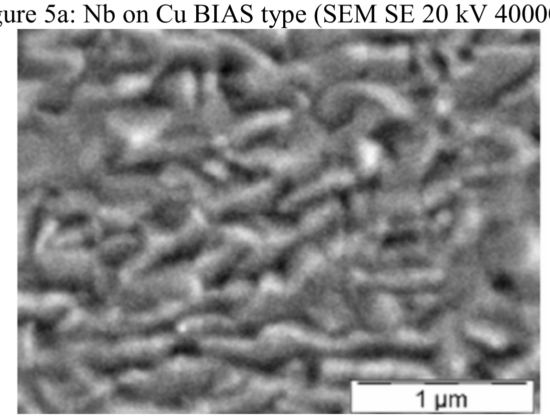

Figure 5b: Nb on Cu CERN type (SEM SE 20 kV 40000x)

Figure 7 shows a detail of the biased coating on Cu substrate, detected by TEM after FIB lamella thinning, of the surface oxide layer, whose thickness of ~12 nm is slightly higher than previously reported results [23]. It has to be underlined that at present no other technique can give equivalent morphological and microstructural thin films characterization, including speed of analysis, site-specific morphology and composition and crystal orientation.

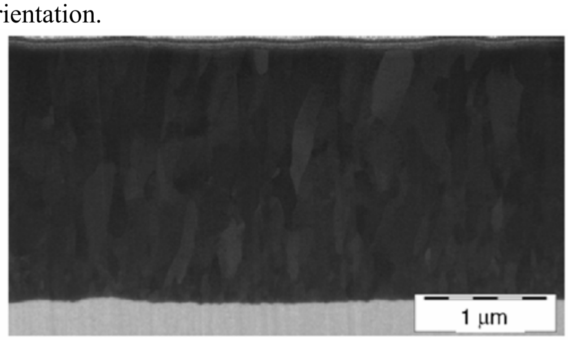

Figure 6a: Nb on Cu BIAS type (FEG-SEM SE 3 kV 80000x, after FIB sectioning)

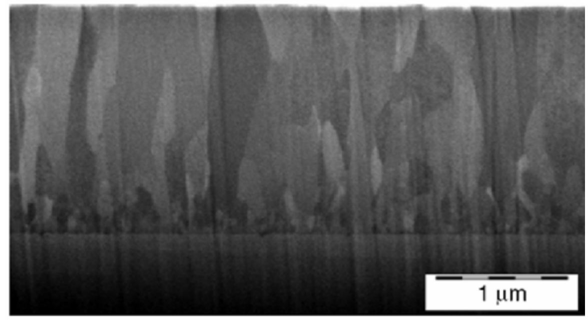

Figure 6b: Nb on Cu CERN type (FEG-SEM SE 3 kV 80000x, after FIB sectioning)

Results for mechanical characterisation are reported in Table 2 and Figure 9. BMP films show in average more than 50% higher MHV hardness than unbiased.

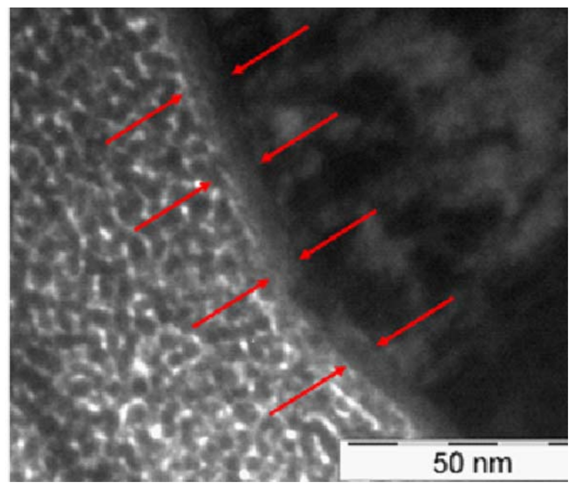

Figure 7: Detail of the surface oxide layer, Nb on Cu BIAS type (TEM BF, 660000X after FIB lamella thinning): thickness of 12 nm

RF test carried out on two cavites get the results shown in Figure 10. The Standard CM receipe applied on a copper cavity show a Q_0=2.6x10^9 , the cavity quenches at 13 MV/m. The second coating with the BMP technique has been applied on the same cavity after stripping, elctropolising, chemical etching and HPWR. The cavity positioned on cryogenic stand undergoes several leak problem and in fact it quenched at very low field.

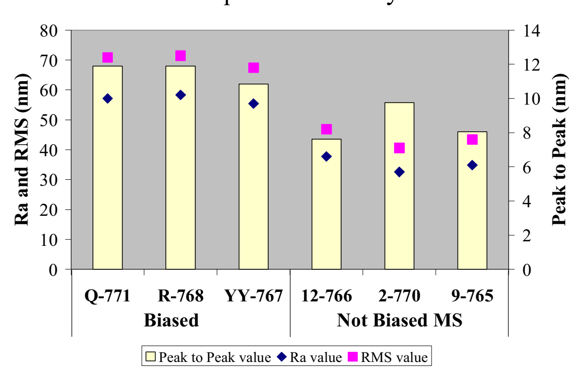

Figure 8: Roughness measurement results for all investigated samples.

High Ionized Plasma

The Hollow Cathode and Planar Magnetron coupling is well described in Figure 11.where the I-V curve with and without the electron injections of the Hollow Cathode are traced: the plasma is characterized by a lower impedance, as a consequence there is an increased current at constant voltage while the Hollow Cathode current rises. The hollow cathode magnetron shows an increase of 25% in the sputtering rate compared to the magnetron alone.

X-ray shows grains dimension in the order of 25-30 nm instead of 15 nm typically of the CERN standard method. Texture analysis is performed on (110) peak. Texture figures show an homogeneous grain orientation growth, mainly perpendicular to the substrate, all along the cavity cell. The RRR values are still low compared to CERN standard system..

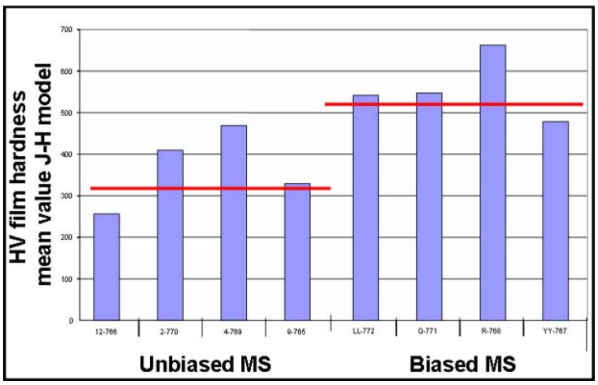

Figure 9: Film intrinsic Vickers hardness (kgf/mm^2) (applied load 0.5gf, J-H model) for all samples

DISCUSSION

RRR directly depends on film thickness (grain dimensions), [26], defects, impurities and grain boundary scattering. Standard Cylindrical Magnetron films show higher RRR than Biased Magnetron films. This effect doesn't coincide with higher thickness and bigger grain dimensions.. This probably suggests a lower impurities concentration.

	ID	Intrinsic hardness (C-L) HV_0 ; n	Intrinsic hardness (J-H) HV_0 ; n	Measured Apparent Elastic Modulus
Deposition mode	#	[GPa] #	[GPa] #	[GPa]
Unbiased MS-PVD Nb film	2-770	0,61 1,972	0,61 1,939	-
	9-765	0,34 2,030	0,3 2,004	-
	12-766	0,49 1,880	0,47 1,804	54,33
Biased MS-PVD Nb film	Q-771	1,36 1,706	1,25 1,803	-
	YY-767	1,09 1,819	1,14 1,725	88,95
	R-768	0,77 1,842	0,71 1,976	-

Table 2: Results summary for mechanical characterisation of MS-PVD Nb films (Vickers Hardness is reported in accord to Meyer ISE law, eq. 3)

After morphological and mechanical surface characterisation, higher surface roughness, lower mean crystallite size and higher Vickers intrinsic hardness have been found for Biased Nb films, compared to the unbiased ones.

At a sub-microscopic scale, it has been observed that tailoring of coating microstructure directly reflect on its surface roughness: as showed in Figure 6, the biased Nb film is characterised by a finer columnar microstructure and lower crystallite size, which directly influences measured surface roughness: this microstructural aspect surely influences coating residual resistivity and *Q* factor, as underlined by other authors [27,28].

By hardness measurement and the use of models to extrapolate true film hardness and Elastic Modulus, some useful information on coating microstructure can be obtained: the lower mean intrinsic hardness for Unbiased film it is likely due to lower coating density and a grain boundary weakening effect.

Elastic response, evaluated after shape analysis of static Vickers indentation marks [29], resulted to be more than 60% higher for BMP films than unbiased films, while both values are significantly lower if compared with the theoretical one for bulk Niobium.

This results is not obviously related to a change in the intrinsic Elastic properties of Niobium, but most likely due to changes in coating density and microstructure; in fact several experimental studies have already underlined that Elastic response of thin films is strongly influenced by coating density and microstructure [24].

Even if biased niobium on copper films are harder and denser than the unbiased one, at the moment their small grain dimensions and consequently their low RRR seem to make the biased technique still not competitive with the standard one from the RF point of view. RF measurements at very low electric field don't show noteworthy differences but test should be repeated for the BMP cavity due to leak problems.

On the other hand study of the High Ionized Plasma technique are still in progress and a more deep investigation on film morphology and properties will be handled.

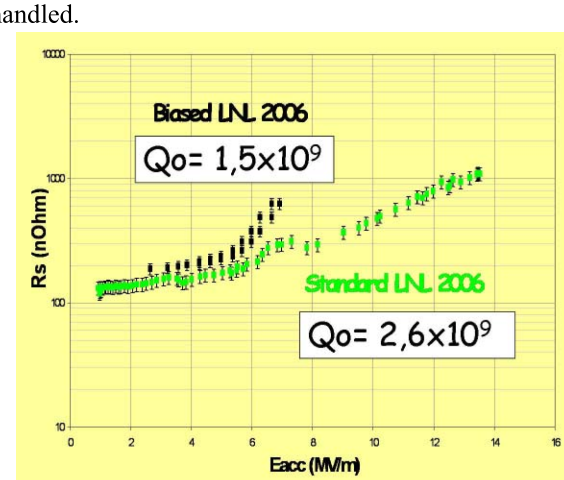

Figure 10: RF test results for 1,5GHz tesla type monocell copper on niobium cavities.

At the moment two probable limiting factors for the low RRR values has been found :

1) degassing of the cavity during the sputtering process because $T_{process} > T_{baking}$;
2) electronic bombardment of the growing film.

Solution for cooling the cavity and inserting a positive biased electrode in the coating system are on-going.

CONCLUSIONS

Two different coating techniques and apparatus have been built and tested for coating 1,5 GHz copper cavities with a thin film of niobium.

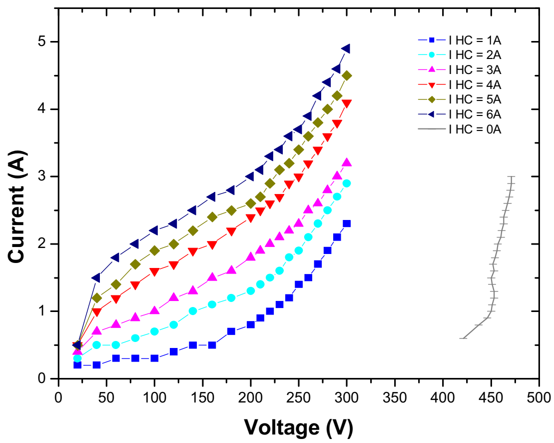

Figure 11: I-V curves of the Hollow Cathode and Planar Magnetron coupling at different current applied to the Hollow Cathode.

Morphological, microstructural and mechanical properties of coatings have been experimentally determined by FIB-SEM, AFM, TEM and nanoindentation techniques, and then correlated to Superconducting properties (critical temperature Tc and Residual Resistivity). A comprehensive chemical, morphological and mechanical surface characterisation has been applied on several samples before performing the RF test on cavities , in order to find out and improve the appropriate correlation functions among process parameters\microstructure \surface properties and superconductig properties.

Even if the High Ionized Plasma technique show some technical problems the idea of densify the plasma seem to be the necessary to improve the film quality. A Future development will be the combination of both High Ionized Plasma and Biased Plasma system in order to sum the potentialities of both techniques.

Biased Magnetron Plasma reveal finer columnar structure than unbiased film: this morphological evidence explain biased film lower RRR and is connected to film roughness and density. To solve this problem several studies changing coating parameters systematically is compulsory. In addition Krypton gas will substitute the actual Argon sputtering gas.

Combination of morphological and RF characterisation will provide a deeper understanding of thin film behaviour and could give useful indications for coating production process optimisation.

Niobium films have however not yet achieved their possible ultimate performance, contrary to what has been obtained with niobium sheet cavities, and this hinders at present their use for electron linacs although their cost is far inferior. Anyway several way haven't been explored yet, many studies are under development and niobium films on copper cavities are still a challenge for SRF applications.

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