# New Magnetron Configurations for Sputtered Nb onto Cu

G.Lanza+\$, J.Bermudez+\$, A.Frigo+\$, H.Padamsee\*, V.Palmieri+\$, D.Tonini\$

+ Laboratori Nazionali di Legnaro, ISTITUTO NAZIONALE DI FISICA NUCLEARE, Italy
\* Newman Laboratory, Cornell University, Ithaca, USA
\$ Science Faculty, University of Padua, Italy.

#### I. INTRODUCTION

The adoption of the Nb/Cu technology at CERN for the LEP cavities and the successful operation of ALPI Linac at INFN-LNL have demonstrated the feasibility of using large-scale copper accelerating cavities coated with a thin superconducting niobium film.

Niobium sputtered film microstructure and morphology and consequently its superconducting properties, strongly depend on target-substrate deposition angle. One limit of the standard cylindrical magnetron sputtering deposition technique is the coating geometry, in fact assuming a cosine law for the atom emission mechanism, the incidence angle of the atoms on the cavity wall varies from  $9\pm4^{\circ}$  around the equator to  $50\pm10^{\circ}$  near the iris.

In order to improve the Nb film quality for 1,5GHz cavity coatings, we investigated the application of two main ideas to the sputtering process: i) promoting the effect of plasma bombardment of the growing film, ii) increasing the sputtering rate.

The effect of Nb atoms arriving perpendicular to the substrate is explored by increasing the plasma confinement efficiency by means of a target parallel to the magnetic field lines. The removal of adsorbed impurities from the film surface and the increase of the film density are investigated by a biased third electrode that promotes the positive ions bombardment of the growing film. A mixed bias-magnetron has been built using a positively charged metal grid positioned all around the cathode.

### **II. NEW MAGNETRON CONFIGURATIONS**

The CERN standard film coating procedure consists of covering the inner wall of copper resonator using the Cylindrical Magnetron (MC) sputtering technique [1]. The aim of the present work is to improve the film quality modifying or completely changing the coating configuration starting from two main ideas:

- promoting the effect of plasma bombardment of the growing film in order to remove impurities weakly bonded to the surface;
- increasing the sputtering area and the plasma ionization efficiency in order to increase the sputtering rate.

## **Original** Apparatus

A stainless steel cavity-shaped deposition chamber has been mounted on a vacuum system and quartz substrates are positioned along two cavity shaped sample holders. An ultimate pressure of the order of  $10^{-9}$  mbar is obtained after 30h bake-out at 150°C.

The cathode is located on the axis of the system. It consists of a vacuum tight stainless steel tube surrounded by a high purity niobium tube. The magnetron cathode is cooled by compressed air.

## Cavity shaped magnetron sputtering cathode

A niobium ring, 48.5 mm maximum diameter and 27 mm high, has been positioned around the cathode tube in the middle of the cavity cell. The magnetic field is produced by a 2 cm high NdFeB permanent magnet located in the middle of the cell, inside the cathode, centred with the niobium ring

This configuration aims to reduce the deposition angle and to increase the area where the magnetic field is perpendicular to the electric field.



FIG. 1:Plasma all around the niobium ring and magnetic field lines simulations for the 2cm high NdFeB magnet (Pandira Software[2]).

Film thickness analysis shows that this configuration doesn't work as expected. The sputtering rate at the equator is higher than standard MC sputtering rate, but films are thicker in the equator than in the iris so the deposition is not homogeneous all over the ring.

Typical RRR (Residual Resistivity Ratio) and Tc (Superconducting Critical Temperature) results at the equator are 13 and 9.36K. Tc higher than Nb bulk means a compressive stress of the crystal lattice, confirmed also by X-ray diffraction cell parameter measure a = 3.29 Å respect to the bulk value  $a_0=3.303$  Å. In addition texture analysis is performed on (110) peak, the most intense in Nb powder diffractogram, in order to investigate the occurrence of preferential orientation during film growth. Texture polar figures show the inhomogeneous grain orientation growth for all the positions along the cell.

Several additional simulations have been run to optimize the best combination of magnet configuration and cathode shape.

#### **Biased Magnetron Sputtering Configuration**

If the film is given a negative potential with respect to the plasma, the resulting technique is referred to as bias sputtering. In this way films are subjected to a certain amount of resputtering. Advantages of bias are mainly the removal of most impurities during resputtering and rearrangement of atoms during film growing.

Considering the bias process Eq.1 is modified as

$$f_i = \frac{(\alpha_i N_i - \beta)}{(\alpha_i N_i - \beta) + R} \quad \text{Eq. 1}$$

where  $N_i$  is the number of atoms of species i bombarding unit area of film in unit time during deposition,  $\alpha_i$  is the effective sticking coefficient of the species i during deposition and R is the deposition rate of the film.  $\beta$  is a function of the bias current due to impurities ions [3]. The bias technique is highly reliable in fact over 40 Quarter Wave Resonators are installed and working at LNL for ALPI.

In our case the bias technique is added to the magnetron cathode as showed in Figure 2.



FIG.2: Scheme of the magnetron bias configuration with six rods. Detail of isolators and springs.

An efficient grid configuration uses stainless steel rods: six vertical stainless steel rods, isolated from the cathode by ceramic cylinders, surround the niobium tube. Twelve springs keep the rods tight and avoid heating deformations. Tests with rods positively biased from 100V to 200V have been executed.

Pulsing the cathode with a 50 kHz square wave pulsed voltage is a way to increase the plasma ionisation degree and to promote the surface ionic bombardment during the positive voltage period.

RRR of 11 in the cell and 20 above and below the iris have been obtained after pulsed sputtering with the

SS rods configuration. Maximum Tc obtained at the equator is 9.3K, meaning a compressive stress of the crystal lattice, confirmed also by this X-ray diffraction cell parameter measure a = 3.30 Å. These results show no improvement or worsening of the film stress respect to MC standard deposition.

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.

Film thickness measurements show that the grid shadowing doesn't affect the sputtering rate in any position all along and around the cell.

Probable limiting factors are impurities coming from the Stainless Steel rods immersed in the plasma; in fact they could partly mask the benefit effects of the applied bias. Impurity content analysis hasn't been executed yet.

## **III. CONCLUSIONS**

Several approaches have been studied to improve the sputtered niobium film properties and different magnetron sputtering configurations has been built and tested:

- Cavity shaped magnetron sputtering cathode
- Biased Magnetron Sputtering Configuration

Best results have been obtained with pulsed biased magnetron sputtering with the six rods configuration but improvement of the film purity is compulsory for emphasize the benefit of the ion bombardment.

#### **IV. ACKNOWLEDGMENTS**

The authors are grateful to S. Stark, G. Keppel, A. Minarello and S. Marigo.

- [1] C. Benvenuti et al., Physica C 351 (2001) 421.
- [2] J. H. Billen and L. M. Young; Poisson Superfish Pandira; LA-UR-96-1834 Revised November 5, 2003; Los Alamos National Laboratory
- [3] "Coating Deposition by Sputtering", Hanbook of plasma processing technology, S. M. Rossagel, J. J. Cuomo and W. D. Westwood eds, Noyes Publications, 196 (1990)