Superconducting niobium coated copper accelerating cavities: investigation of cylindrical magnetron sputtering configurations

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I. INTRODUCTION

Niobium sputtered superconducting cavities for e⁺ e⁻ accelerators have great advantages respect to bulk niobium ones because of their lower cost and better performances, but there is a big limit on their use due to the decaying of the Q-value \( Q = \frac{\omega U}{P} \) when increasing accelerating field.

Studies made at CERN for medium \( \beta (\beta = v/c) \) LEP[1] cavities and at INFN-LNL for thin films coated at different angles[2] showed that arrival angle of niobium depositing atoms seems to have an influence on film properties; those works indicate 28° as limiting arrival angle to achieve satisfactory superconducting properties of niobium films.

This paper presents the results obtained on superconducting niobium films prepared by two magnetron sputtering set-up designed to coat 1,5 GHz cavities. This two configurations allow to obtain films growing at different orientations to the substrates, especially on the cell zone.

Electrical, Morphological and microstructural properties are tested focusing on their relation with films position along the cavity and atoms arrival angles. Results are compared so as to evaluate the two configurations and underline the problems of each techniques.

II. EXPERIMENTAL SET-UP

The sputtering system used to coat 1,5 GHz copper cavity has been reorganized from the old version[3]: the baking circuit, the electrical circuit and the by-pass zone has been renewed and optimized. A stainless steel cavity-shaped deposition chamber has been mounted and glass or copper substrates are positioned along two cavity shaped sample holders.

![Figure 1: Multiangle sampleholder and simulated film porosity for different target-substrate angles [2].](image)

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![Figure 2: CPM Cylindrical Post-Magnetron configuration and CM Cylindrical Magnetron configuration.](image)

The vacuum chamber is evacuated by a pumping system consisting on a rotary pump for a primary vacuum and a oil free turbo molecular pump with magnetic bearings. An ultimate pressure of the order of \( 10^{-9} \) mbar is obtained after 30h bake-out at 150°C. The niobium films, typically 2 μm thick, are grown on glass substrates by sputtering using a cylindrical magnetron configuration (CM) or a cylindrical post-magnetron configuration.
(CPM). For both configurations 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.

The magnetic field of the CM configuration is produced by a NdFeB permanent magnet located inside the cathode (Figure 2CM). On the other side in the CPM configuration four coils positioned like in Figure 2CPM produce a cavity shaped magnetic field around the cathode. End losses are limited by two cathode wings for the electrostatic confinement and by the two outer coils that close the bottle neck and act like magnetic mirrors.

III. DEPOSITION TECHNIQUES

Before coating, the chamber is baked at a temperature of 150°C for 30h; at the end of the operation the ultimate pressure is measured to reach typically 10^{-9} mbar at room temperature. Substrates positioned in the central cell and at the extension tubes are coated in a single operation.

In the CM configuration the extension tubes are coated first in a argon atmosphere of 1x10^{-2} mbar; a stable current of 1A is set between the cathode and the grounded sampleholders, corresponding to a voltage of about 400V. The magnet is moved in 10 steps over the cathode length in order to coat each tube uniformly. The cell is coated with slightly different discharge parameters, leaving the magnet fixed in its center for 15 minutes: the argon pressure is reduced to 1.5x10^{-3} mbar so as to increase the mean free path of the atoms ejected from the cathode and the current is increased to 3A to rise the sputtering rate. With this operations the film thickness is uniform over the whole length of the cavity.[4,5].

In CPM configuration niobium films are coated all along the cavity in one step. The procedure is more rapid but films in the extension tubes are thicker than in the cell. The gas discharge is established in argon atmosphere at 3.5x10^{-2} mbar and a current of 7A[6].

IV. RESULTS

The produced samples are characterized by measure of resistance versus temperature (critical temperature and residual resistivity ratio), x-ray diffraction pattern (cell parameter and grain dimension), x-ray texture (preferential orientation), AFM images (roughness).

Superconducting properties

Films coated with both configurations have Tc values from 9.25 K to 9.54 K with transition widths from 0.004 K to 0.1 K.

Niobium sputtered with the CM configuration shows RRR between 40 and 10 while films coated in the CPM configuration present RRR from 22 to 7.

Figure 3: Example of resistivity versus temperature curve acquired with a four contact method. The sample was positioned on the cell iris; its transition is 0.005 width around 9.401 K.

Both RRR and Tc results get worse going down from the beam tube to the cell equator. Lower Tc and RRR are localized near both iris of the cell and this effect is stronger for the CPM configuration.

Pure bulk niobium has a transition temperature of 9.26 K so Tc values are all greater than Nb bulk one due to the compressed crystal lattice.

Crystal structure

X-ray diffraction spectra don’t show important variations of cell parameters and grain dimensions along the cavity walls and down to the cell. Films coated with the CM and CPM configurations show grain size of the order of 300 Å.

Niobium lattice is body centered cubic with a cell parameter of 3.303 Å; film deposited on quartz are mainly in a compressive state of stress in fact their cell parameters range between 3.285 Å and 3.271 Å.

Films grown on copper, on the other side, shows a slight tensile stress caused mainly by different thermal expansion coefficients of niobium on copper. Unfortunately the substrate temperature hasn’t been controlled because of the poor thermal contact of the sample holder with the external cavity chamber.

Texture analysis[7] is performed on (110) peak, the most intense in Nb powder diffractogram, in order to investigate the occurrence of preferential orientation during film growth. Films tend to grow with the normal to 110 crystal plane aligned with the atoms arrival direction.

All films coated with the CM configuration grow inclined to the substrates except the film on the equator whose texture pole figure is a perfect circle centered on the origin.

Surface morphology

The appearance of film surface at submicrometer scale is clearly seen by using Atomic Force Microscope (AFM) imaging. AFM analysis was performed in contact mode,
constant force, on an area 5x5 \( \mu m \) wide.

Figure 4: plot of average roughness measured from AFM images of niobium films coated with with CM configuration.

Figure 4 is a plot of measured average roughness along the cavity. Inside the cell films at the equator are rougher than on the iris, probably due to an increasing of the cathode-substrate distance.

In addition film roughness is higher in the beam tube than in the cell due to the deposition procedure: the magnet moves along the cathode during beam tube deposition so films deposited at different inclinations grow one over each other. In the end substrates expose on the surface the film with the worst inclination, coated when the magnet is in the further position.

**V. CONCLUSIONS**

Compressive stress is mainly due to ions and atoms bombardment of the film during the growth process and less to contaminations trapping, in fact RRR higher than 20 all along the cavity means high level of cleanliness maintained during all depositions.

Interpolating all the results of Tc and RRR obtained during 13 deposition on more than 140 samples we found the following relation:

\[
T_c = (9.46 \pm 0.02) - (0.117 \pm 0.0042) \frac{1}{RRR} + (300K) \rho_{ph} \frac{1}{RRR} - 1
\]

Summarizing:

- Best superconductive properties are obtained on films placed parallel to the cathode.
- Both configuration cannot produce films with the same superconductive and morphological properties all along the cavity walls.
- Texture analysis demonstrate that fixing the magnet at the center of the cell reduce the problem of angular deposition but it doesn't solve it completely. Moving the magnet up and down during beam tube coating, on the other side, produce textured films along the tubes. On the contrary, post-cylindrical magnetron sputtering grants good properties for films along the beam tube but films on the cell are affected by inclined deposition.
- AFM measures show that films with the best electrical property (high RRR) has rougher or even porous surface. Probably the resistive method is not adequate to establish the film applicability for RF use because the direct current can percolate trough a porous film. It may be necessary to use different analysis techniques like inductive Tc measurements, magnetic moment measures and Electrochemical Impedance Spectroscopy.

Magneton sputtering could be improved changing the cathode shape or the magnet's disposition or both. A cavity-shaped cathode could help atoms to reach the substrates in perpendicular directions so as to avoid the grown of textured films. The sputtering process must be efficient, so a magnetic field, which is parallel to the target surface and perpendicular to the electric field lines is needed too.