# THIN FILM COATING OPTIMIZATION FOR HIE-ISOLDE SRF CAVITIES: COATING PARAMETERS STUDY AND FILM CHARACTERIZATION

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*Abstract*

The HIE-ISOLDE project at CERN requires the production of 32 superconducting Quarter Wave Resonators (QWRs) in order to increase the energy of the beam up to 10 MeV/u. The cavities, of complex cylindrical geometry (0.3m diameter and 0.8m height), are made of copper and are coated with a thin superconducting layer of niobium. In the present phase of the project the aim is to obtain a niobium film, using the DC bias diode sputtering technique, providing adequate high quality factor of the cavities and to ensure reproducibility for the future series production. After an overview of the explored coating parameters (hardware and process), the resulting film characteristics, thickness profile along the cavity, structure and morphology and Residual Resistivity Ratio (RRR) of the Nb film will be shown. The effect of the sputtering gas process pressure and configuration of the coating setup will be highlighted.

## INTRODUCTION

In the frame of High Intensity and Energy (HIE) ISOLDE project [1], the niobium coating of the high beta superconducting quarter-wave resonators (QWRs) is about to enter series production. Development, understanding and optimization of the sputtering setup and process has been conducted along the last 4 years at CERN to exceed the specifications (6 MV/m accelerating gradient on the beam axis with a total maximum power dissipation of 10 W) [2, 3].

## TESTS FACILITIES

The general coating system is detailed in [3]. For the purpose of R&D tests, two different setups were used:

a) A dedicated production-like cavity has been designed with a door access to the inside and 50 samples positions along the outer and inner conductors as sketched in figure 1 (a). This setup allows having access to the film properties by sample characterisations prior to make a coating on a cavity for RF-test, and thus measure the impact of process or hardware modifications on the quality of the Nb film in a reproducible way.

b) A specific test bench with scaled-down setup (figure 1 (b)) has also been developed to investigate in particular the effect of the pressure on the effective sputtering yield and thickness of deposited film. This downscaled setup is very versatile thanks to its easy access and fast sample changes and parameter settings while respecting the original distance of substrate to cathode of 52 mm.

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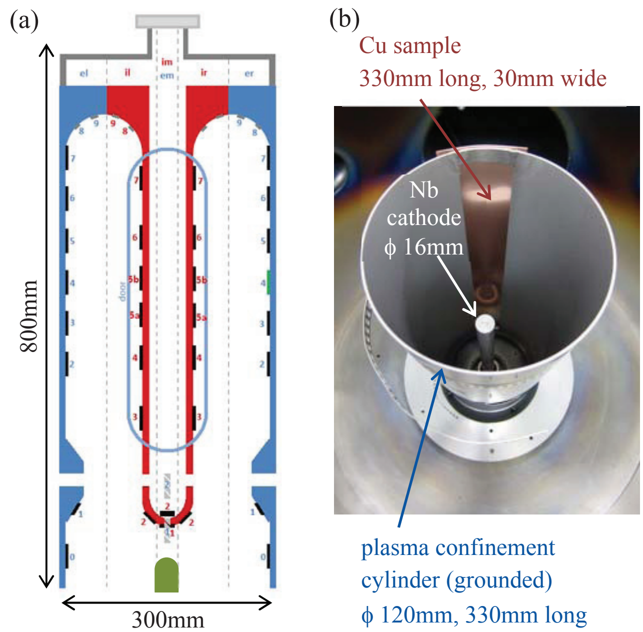


Figure 1: (a) cross section drawing of test cavity with position of samples, inner conductor in red, outer conductor in blue and centre electrode in green; (b) top view of the test bench setup with a copper band sample.

## COATING PARAMETERS

Several parameters of the hardware and of the process have been varied to optimize the coating uniformity and properties. The cathode shape – length, geometry and position relative to the substrate, the centre electrode facing the inner conductor – shape, distance to inner conductor tip, DC power and pressure were varied and tried in test cavity as summarized in table 1 below.

Table 1: Parameter Table (baseline Parameters in **bold**)

| Test # | top gap [mm] | Centre electrode ϕ | Power [kW] | Pressure [mbar] |
|---|---|---|---|---|
| 1 | 52 | 40 mm | 8 | 0.25 |
| 2 | 46 (donut) | 40 mm | 8 | 0.23 |
| 3 | 32 | 40 mm | **8** | **0.22** |
| 4 | 32 | **none** | 1.23 CW | 0.077 |
| 5 | **22** | 20 mm | 1.33 CW | 0.081 |




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### *Geometrical (Hardware) Parameters*

The hardware parameters like cathode and centre electrode helped to address the film uniformity and thickness issues. Thus we managed to obtain a uniform well adhering layer on the tip of the inner conductor by removing the centre electrode. The longer cathode allowed us to obtain a well adhering layer on the edge of the outer conductor. Finally, by reducing the distance between the top of the cathode and the top of the substrate down to 22 mm, at the position where the RF magnetic field is the most intense, we managed to gain a factor two in film coating rate.

### *Process Parameters*

On the other hand, the process parameters like cathode power, sputtering gas pressure or substrate temperature were varied to obtain the film properties needed for the RF performances of the cavity. Pre-heating of the cavity substrate up to temperature of 650 °C certainly plays a role in the niobium film purity by releasing most of the hydrogen trapped in the bulk copper substrate before the coating process starts [4]. Therefore the temperature of the substrate during the process should not exceed the maximum reached during the pre-heating phase. Pressure and power are two other process parameters intimately linked together, and they are actually hardly decoupled from the substrate temperature. Low pressure may increase the deposition rate due to larger mean free path and higher ions energy driven by the lower collisions rate. High power may help to increase the sputtering yield in DC bias diode sputtering regime, and helps growing a high quality film with closer grain boundaries and dense, smooth layer.

### *Limitations*

These parameters are not independent and for instance one cannot ignite a high power discharge at low pressure due to the limited 1kV voltage output of the DC-power supply system. Thus a low pressure coating will take place at low power as well. The coating sequence itself is also dependent on the power. At high DC-power, the substrate temperature will increase in a very short time up to the pre-heating temperature and one must allow sufficient cooling time before continuing the process. As a consequence such coating is done in several runs (up to 15) to obtain the desired film thickness for a net coating time of 6 hours. At low power (< 1400W) one can work either in continuous (CW) or in several runs mode.

## TEST CAVITY RESULTS

### *Niobium  eposition Rate Profile in  est  avity*

The thickness of the niobium film deposited on copper samples is measured by X-Ray Fluorescence (XRF).

The effect of the cathode to cavity top distance ("top gap") on the film thickness is shown in figure 2 below. The inner and outer conductor deposition rates do not vary too much in both setups (up to ~ 50% larger). On the contrary, the deposition rate at the top of the cavity (zoom in figure 2) shows a clear improvement. A factor two gain at this position is obtained by reducing the cathode to top distance of 20 mm from 52 mm to 32 mm top gap.

This top gap distance has a big impact on RF performances of the cavity. Decreasing this distance has the effect of increasing the coating rate, thus the thickness, locally on the top of the cavity (region of highest magnetic field). This increase of rate in this particular region is correlated to the increase of the quality factor of the cavity. As shown in [3], a further improvement was obtained with 22 mm top gap distance.

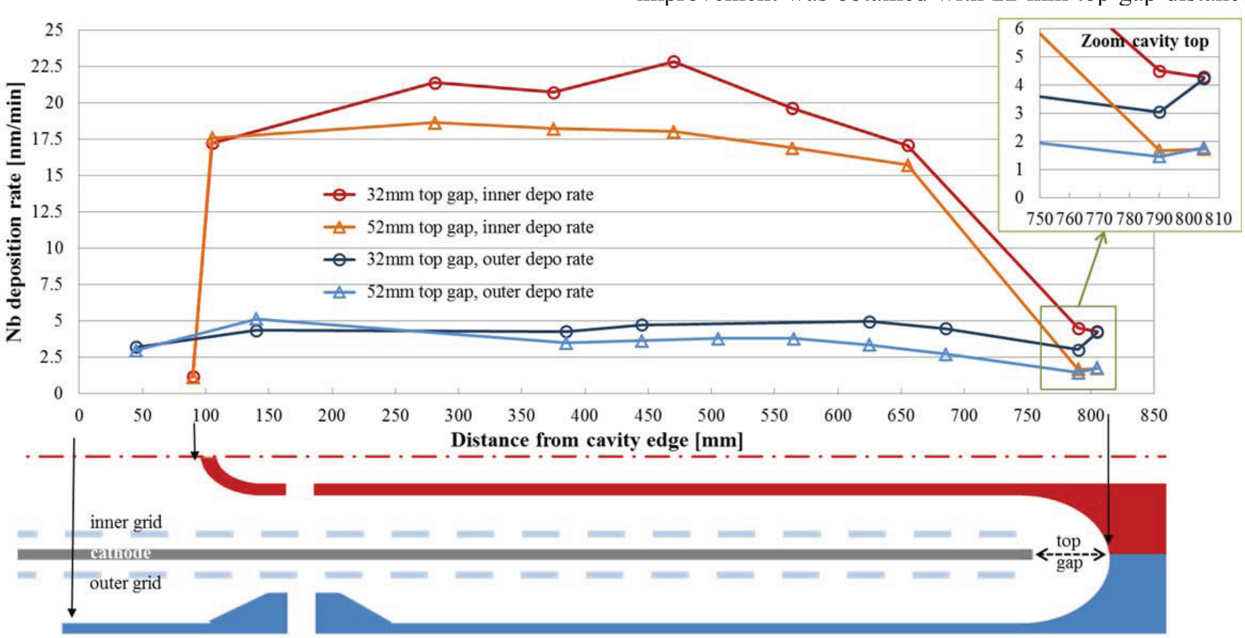


Figure 2: Niobium film deposition rates profiles along cavity outer conductor (in blue) and inner conductor (in red) for test #1 with top gap of 52 mm (Δ) and test #3 with top gap of 32 mm (O).

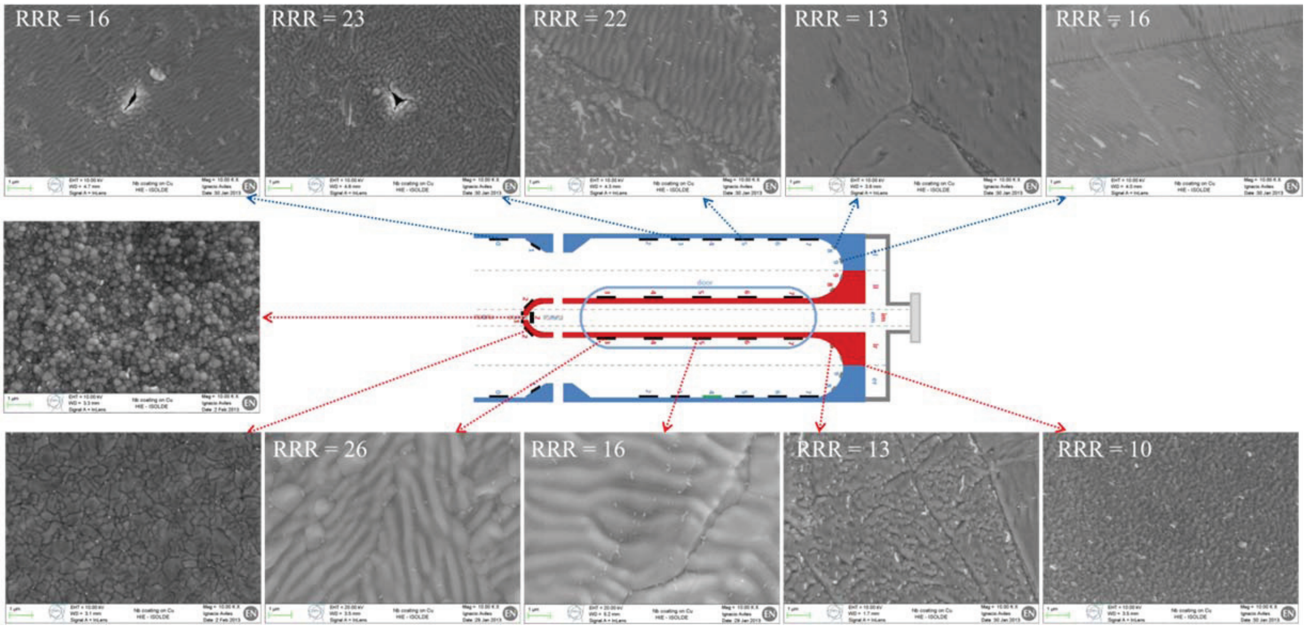


Figure 3: SEM pictures (magnification x10000) of niobium coated copper samples taken along the cavity outer conductor (in blue) and inner conductor (in red) and RRR measured at corresponding position, test #1, 52 mm top gap.

### *Film tructure and RRR*

The film structure is analysed by SEM on the coated copper samples. The outcome of these measurements is presented in Figure 3.

Due to higher deposition rate, thicker layer and higher temperature on the inner conductor, grains are larger than on the outer conductor and top of the cavity. On both inner and outer conductors the grains of recrystallized copper are visible and govern the crystallographic orientation of niobium film. On the tip of the inner conductor samples show equiaxial grains, the morphology of the very tip is much more granulated and exhibit blisters if observed at low magnification. Smoother films with proper adhesion have been obtained in this region by removing the centre electrode, as for the baseline setup. The rest of the film is dense and uniform along the cavity.

The RRR is defined as the ratio of resistances ($R$) at room temperature and just above the superconducting transition temperature: $R_{300K}/R_{10K}$. It is measured on quartz samples mounted in the test cavity. RRR measurements (Figure 3) show an average value of 17 and the distribution along the cavity shows no correlation with the coating rate.

## TEST BENCH RESULTS

Higher DC-power is of interest to obtain higher deposition rates and denser films but this implies to work at higher pressure not to be limited by the maximum voltage output of the power supply (1 kV).

The test bench, with its down scale design, allows the use of different power supplies and sputtering configuration (DC bias diode and magnetron).

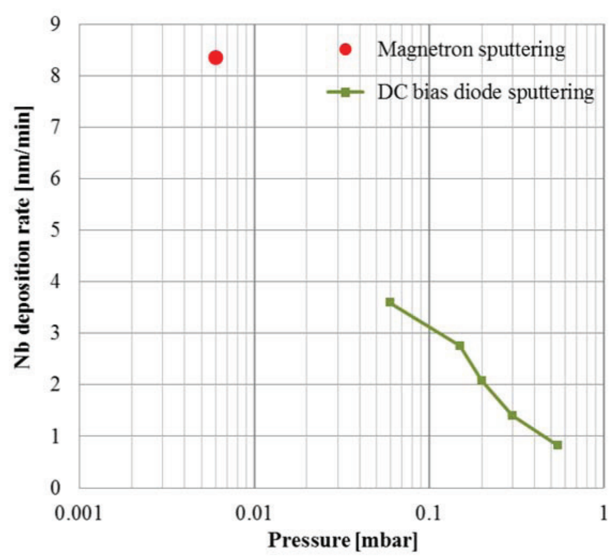


Figure 4: Average Nb film deposition rate from test bench study, as a function of pressure for the DC bias diode and magnetron sputtering, power = 155 W in both cases.

To investigate the effect of the pressure on the Nb deposition rate a given power, fixed at 155 W, was chosen to have a thermal behaviour comparable to the cavities.

Samples thickness measurements by XRF revealed an almost linear decrease of coating rate with the log of the increasing pressure (Figure 4). Magnetron sputtering allows having a measurement of the coating rate for a mean free path much larger than the cathode-anode distance.

One can observe that working with DC bias diode sputtering at high pressure regime is a limiting factor for the deposition rate and the film growth for three reasons:


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(1) the mean free path is smaller than the cathode-anode distance, increasing the number of collisions and thus reducing the sputtering rate;

(2) due to the larger collision rate, energy of Ar ions and Nb atoms is much smaller, changing the way the film grows (structure, morphology);

(3) the lower deposition rate at higher pressure increases the time needed to obtain the desired film thickness and exposes longer the growing film to contamination during the coating.

Consequently, in DC bias diode sputtering, raising the pressure to work at higher power is not an option to further enhance the film properties with this technique and a higher voltage power supply is necessary.

Therefore, the magnetron sputtering route is of interest [5] to overcome these drawbacks and to obtain potentially even better RF performances.

## CONCLUSION

CERN thin film coating test facilities are used to optimize both hardware and process parameters and define optimal film growth conditions.

This study, in parallel to real cavity RF measurements, resulted in the release of the DC bias diode sputtering baseline recipe for production of HIE-ISOLDE SRF cavities [3] exceeding project specifications.

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