# PRELIMINARY RESULTS OF NB THIN FILM COATING FOR HIE-ISOLDE SRF CAVITIES OBTAINED BY MAGNETRON SPUTTERING

A. Sublet\*, I. Aviles, S. Calatroni, A. D'Elia, N. Jecklin, I. Mondino, S. Prunet, M. Therasse, W. Venturini Delsolaro, P. Zhang, CERN, Geneva, Switzerland

## Abstract

In the context of the HIE-ISOLDE upgrade at CERN, several new facilities for the niobium sputter coating of QWR-type superconducting RF accelerating cavities have been developed, built, and successfully operated. In order to further optimize the production process of these cavities the magnetron sputtering technique has been further investigated and continued as an alternative to the already successfully operational DC bias diode sputtering method. The purpose of this poster is to present the results obtained with this technique. The Nb thickness profile along the cavity and its correlation with the electromagnetic field distribution inside the cavity are discussed. Film structure, morphology and Residual Resistivity Ratio (RRR) will be considered as well and compared with films obtained by DC bias diode sputtering. Finally these results will be compared with RF measurement of a production-like magnetron-coated cavity.

## INTRODUCTION

In parallel to the established niobium coating baseline for the production of the high beta superconducting quarter-wave resonators (QWRs) [1,2], CERN is continuing its magnetron coating activities. On track to achieve the HIE-ISOLDE specifications (6 MV/m accelerating gradient with a total maximum power dissipation of 10 W), this technique is a good candidate to optimize the production cycle of the cavities.

#### SETUP AND PROCESS WINDOW

The experimental setup used is very similar to the one of the "diode" coating [2] except that the grids facing the coaxial cathode are removed and a solenoid is placed around the vacuum chamber to generate a uniform magnetic field across the cavity to be coated. Additionally a donut ring is placed on top of the cathode to reduce the top gap between cathode and cavity to 19mm and to follow the cavity top curvature.

Magnetron studies were started few years ago at CERN for QWR coating but the pre-heating of the cavity was not wished due to mechanical limitation of the substrate.

The current design and facilities allow us to pre-heat the substrate to 650 °C to degas the large amount of hydrogen trapped in the bulk copper before the niobium coating and thus rexduce the self-contamination of the film during the deposition.

#### Plasma ignition monitoring

The process window boundaries have been determined

\*alban.sublet@cern.ch ISBN 978-3-95450-143-4 Spectroscopy (OES) to collect light emitted by the argon plasma from the outer discharge. As the cathode is coaxial and used to sputter both inside (inner conductor) and outside (outer conductor), the plasma ignition and stability must be controlled to guarantee a balanced discharge and thus a uniform coating. However, due to the complex geometry of the substrate surrounding the cathode, the plasma ignition and expansion varies with the discharge parameters. 



by monitoring the plasma ignition using the electrical

characteristics of the discharge and Optical Emission

Figure 1: Typical plasma monitoring curves, 5 min 0-3 kW cycle,  $1.2x10^{-2}$  mbar, 93 G, 300 °C.

For a given condition:  $1.2 \times 10^{-2}$  mbar, 93 G, substrate temperature of 300 °C, Figure 1 illustrates plasma ignition and extinction within one power cycle composed of a ramp-up of 2 min from 0 to 3 kW, a 20 s plateau at 3 kW and a ramp-down from 3 kW to 0 in 2 min.

A first threshold voltage is reached shortly after power on (Figure 1 (1)), followed by a second one (Figure 1 (2)) at 775 W corresponding to outside plasma ignition as Ar emission is detected at the same time. Argon emission intensity steadily increases with power ramp-up. The plasma extinction outside is observed (Figure 1 (3)) at a power of 275 W, showing hysteresis with the ignition.

The voltage-current characteristic of the discharge, Figure 2, highlights this hysteresis (patterned area), where first (1) and second (2) thresholds are observed.

This basic plasma ignition study has been conducted to determine the magnetron process window boundaries with our setup as a function of pressure and magnetic field (Table 1). It shows that in these conditions, under  $8 \times 10^{-3}$  mbar it is not possible to ignite plasma outside.

#### Cavity preparation and production



Figure 2: Voltage-current characteristic of magnetron discharge, 5 min 0-3 kW cycle,  $1.2 \times 10^{-2}$  mbar, 93 G, 300 °C.

Note that the plasma ignition threshold shifts towards higher power with higher cathode temperatures, probably due to gas rarefaction in the vicinity of the cathode.

Table 1: Outer plasma threshold power (in **bold** configuration illustrated in figure 1 and 2)

		Magnetic field [G]		
		70	93	117
kr pressure [mbar]	0.004	No ignition	No ignition	No ignition
	0.008	800W	2000W	No ignition
	0.012	275W	775W	2300W
	0.016	50W	110W	300W
¥	0.02	0W	90W	250W

### **TEST CAVITY RESULTS**

#### Niobium Deposition Rate Profile

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

The test cavity was coated using the parameters from Figure 1 (4), i.e. 93 G magnetic field, 2 kW power and a process pressure of 1.1e-2 mbar, cavity biased to -80 V. Deposition was done in two runs of 2h10min and 1h20min, with 2h10min cool down in between.

Figure 3 shows the deposition rate profile of the niobium layer deposited by magnetron sputtering in the test cavity and by DC bias diode sputtering, together with the RF magnetic field distribution along the cavity.

As compared to the DC bias diode sputtering baseline coating, the deposition rate of the film obtained in these conditions is very similar for the outer conductor (blue curves), though at the top of the cavity, the magnetron coating has a 50% faster rate.

Regarding the inner conductor (red curves) this effect is even more pronounced with a rate up to four times larger with the magnetron method. Additionally, the rate difference between the inner and outer conductor in these conditions is also larger than for DC bias diode coating. This misbalanced rate could be viewed as a drawback; however the fact that it matches well the RF-field distribution in the cavity might be of interest. Having a higher growth rate at position where RF magnetic field will be larger might help for the RF performances because of potentially larger, cleaner grains and thicker layer.

## Film Structure and RRR

The film structure is analysed by SEM on the coated copper samples (Figure 4). Samples from tip of the inner conductor show some voids but no blister and a very good adhesion of the layer on the substrate.



Figure 3: Niobium film deposition rates profiles along cavity outer conductor (in blue) and inner conductor (in red) for magnetron and diode coating and simulated magnetic field distribution in the cavity (dashed lines). opyright

**09** Cavity preparation and production



Figure 4: 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.

Compared to DC bias diode coated samples, the grain size of magnetron coated samples looks smaller though deposition rate is higher. The slower thermal cycle during the magnetron coating might explain this observation.

An average RRR value of 18 (Figure 4) was measured on magnetron coated samples, which is comparable to RRR measured on DC bias diode coated samples [3].

#### **RF TEST RESULTS**

Two production-like cavities have been produced for RF measurements with this setup. Figure 5 shows RF-test result of the first one together with the diode baseline reference cavity. The second one is under measurement.



Figure 5: RF tests results for DC bias diode baseline coating (O) and magnetron coating ( $\Delta$ ).

This cavity was coated in the same conditions as for the test cavity described above but with a third run of 1h20min to obtain a 30% thicker layer. This first measurement of a magnetron coated cavity is very close to HIE-ISOLDE specification. The  $Q_0 = 2x10^9$  is almost at the level of DC bias diode coating cavity.

In terms of coating time the advantage of magnetron is also remarkable as a cavity can be coated within one day while it lasts four days with DC bias diode method.

# CONCLUSION

This preliminary study defined the process window for magnetron coating. RF-measurement of first magnetron coated cavity shows very encouraging results, close to the HIE-ISOLDE project specifications.

## ACKNOWLEDGMENT

We would like to acknowledge the receipt of fellowships from the CATHI Marie Curie Initial Training Network: EU-FP7-PEOPLE-2010-ITN Project # 264330.

Authors are especially thankful to the BE-RF and TE-VSC teams at CERN for their flexibility and precious support.

#### REFERENCES

- [1] W. Venturini et al., "Nb Sputtered Quarter Wave Resonators for the HIE-ISOLDE", in SRF2013, Paris, September 2013.
- [2] N. Jecklin et al., "Niobium Coatings for The HIE-ISOLDE OWR Superconducting Accelerating Cavities", SRF2013, Paris, September 2013.
- [3] A. Sublet et al, "Thin Film Coating Optimization for HIE-ISOLDE SRF Cavities: Coating Parameters Study and Film Characterization", in SRF2013, Paris, September 2013.

# ISBN 978-3-95450-143-4

# **09** Cavity preparation and production

0