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# The HIE-ISOLDE Superconducting Cavities: Surface Treatment and Niobium Thin Film Coating

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# THE HIE-ISOLDE SUPERCONDUCTING CAVITIES: SURFACE TREATMENT AND NIOBIUM THIN FILM COATING

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## Abstract

CERN has designed and prepared new facilities for the surface treatment and niobium sputter coating of the HIE-ISOLDE superconducting cavities. We describe here the design choices, as well as the results of the first surface treatments and test coatings.

## INTRODUCTION

For the post-accelerator of radioactive ion beams at CERN a major upgrade will take place in the next 4-5 years. The upgrade consists of boosting the energy of the machine from 3MeV/u up to 10 MeV/u with beams of a mass-to-charge ratio of  $2.5 < A/q < 4.5$ .

In order to match the higher energy requirement a modular superconducting linac based on quarter wave resonators (QWRs) is planned to be installed downstream the present normal conducting linac. Part of the present normal conducting linac will be replaced by new superconducting cavities in order to allow the full energy variability between 1.2 and 10 MeV/u [4]. The new accelerator is based on two gap independently phased 101.28MHz Nb sputtered superconducting Quarter Wave Resonators (QWRs). Two cavity geometries, "low" and "high"  $\beta$ , have been selected for covering the whole energy range.

An R&D program has started at CERN in 2008. The basic technological choice for the HIE-ISOLDE cavities lies in the use of the Nb/Cu technology and more details about this choice are given elsewhere in this Proceedings [2]. In this paper we will describe the first surface treatment of the copper QWR prototype, the coating facility and the two sputtering configurations tested up to now.

## SURFACE TREATMENTS

The chosen QWR cavities production sequence comprises the following steps:

- Machining and electron beam (EB) welding [2].
- Warm RF test for frequency measurement.
- Chemical polishing (SUBU) and passivation.
- Low pressure ultrapure water rinsing.
- Coating.
- Low pressure ultrapure water rinsing.
- RF warm and cold test (TRIUMF, later on at CERN) [7].

Surface treatments play a fundamental role in cavity performances. With improvements in fabrication and ultra cleanliness techniques, the limitation on superconducting cavity performance now seems to be the surface state generated by the etching process.



Figure 1: The three tanks in line for the QWR chemical polishing.

Surface preparation prior to coating will be carried out by SUBU chemical etching. This polishing agent (SUBU) is a mixture of sulfamic acid ( $\text{H}_3\text{NO}_3\text{S}$ , 5g/l), hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 5% vol), n-butanol (5% vol) and ammonium citrate (1g/l) and the working temperature is around 72°C. The SUBU is preceded and followed by washing with a dilute solution of sulfamic acid [6].

The effectiveness of the SUBU was tested on the EB welding between two copper plates. After 20  $\mu\text{m}$  removal the surface presents an average roughness  $R_a$  of 0.8  $\mu\text{m}$ . It is an acceptable value, as verified several times at CERN with EB welding of the  $\beta_0 = 1$  elliptical resonators.

A closed circuit system with dedicated tubes and a pump has been built for the cavity treatment. Three tanks in line are made of stainless steel and polypropylene for respectively the SUBU (thermo controlled tank), the passivation and the final rinsing (Fig.1). The acid enters the cavity through four tubes and it flows out from the top and the beam aperture. Simulations of the fluid velocity of the

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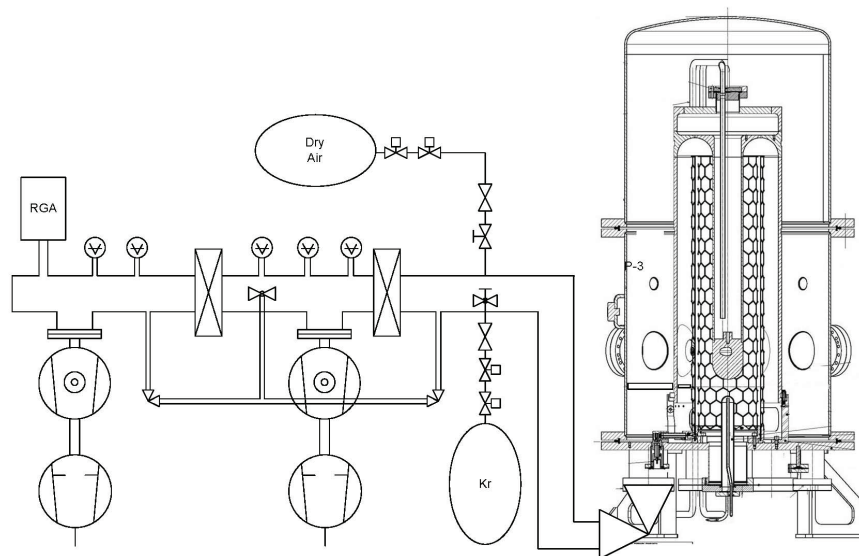


Figure 2: Scheme of the pumping system System.

SUBU, injected into the cavity by four tubes, have shown that the velocity of the fluid close to the bottom wall of the cavity is uniform: the values obtained vary between 0.04 m/s and 0.06 m/s. They correspond to a Reynolds number lower than the limit for turbulent flow.

The first chemical treatment was tested on copper plates placed into the dummy stainless steel cavity. The whole procedure was tested and, after a visual inspection of the samples, the treatment procedure was approved. The first copper cavity was chemically treated. The whole procedure took about two hours. The cavity low pressure rinsing was performed in a class 100 clean room with ultrapure water at 6 bar. One copper cavity is now stored in a class 10 clean room and it is ready to be coated.

## NEW FACILITY FOR QWR COATING

The history of Nb/Cu QWR resonators starts at LNL INFN for the super-conducting linac ALPI for heavy ions, operating since 1994. At the moment 52 quarter wave Nb/Cu resonators are mounted and the success of the development of higher  $\beta$  cavities opened the possibility to apply the sputtering technique to the medium  $\beta$  section as well [1].

The construction of a high  $\beta$  cavity prototype started at CERN in the middle of 2008 and the copper body which makes the substrate for the niobium sputtering was completed in April 2009. CERN has designed and prepared new facilities for the surface treatment and niobium sputter coating of the HIE-ISOLDE superconducting cavities. The LNL experience has been the starting point for the cavity design, the development of the bias diode sputtering configuration and the design of the coating chamber.

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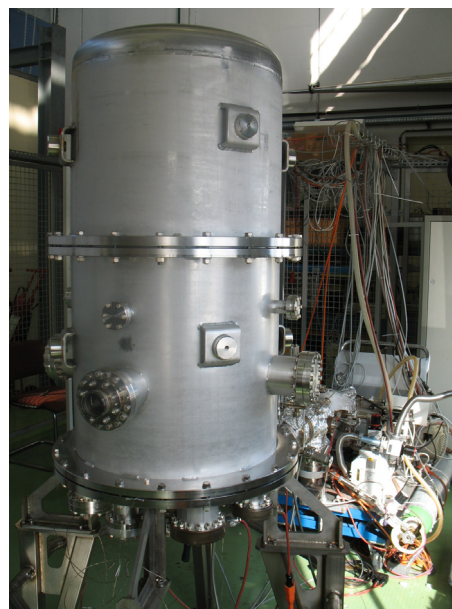


Figure 3: QWR Sputtering System.

The coating chamber is pumped by a turbomolecular pump and a primary pump and it is connected by a by-pass to a Residual Gas Analyzer (RGA) system (Fig. 2). Since the chamber is 600mm diameter wide, it is sealed with two viton o-rings. This fact limits the heating temperature during baking but after a two days baking the base pressure is around  $5 \cdot 10^{-9}$  mbar in the coating chamber.

The cathode-grids structure and the cavity are assembled inside a class 10 clean room and placed into the vacuum

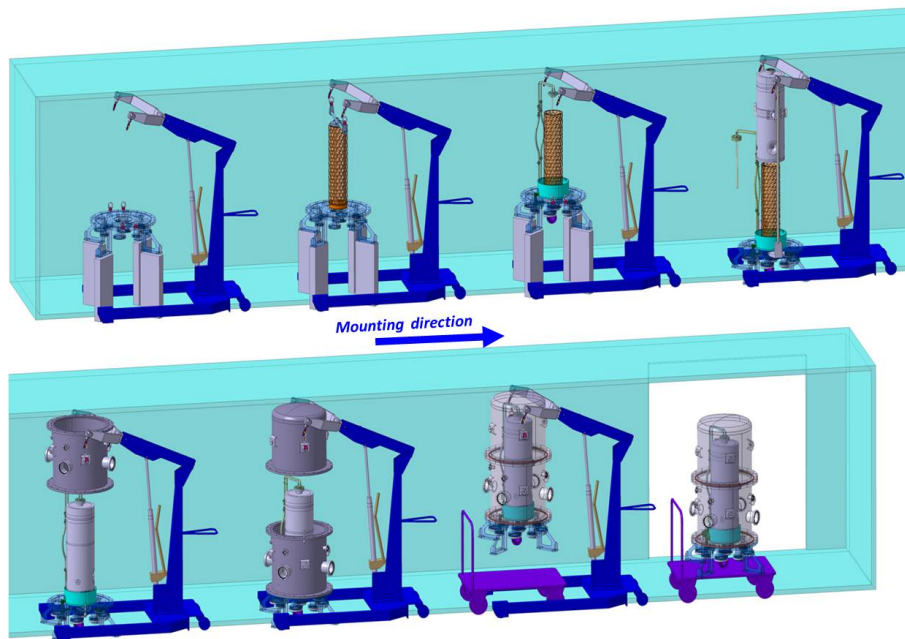


Figure 4: Mounting Sequence: inside a class 10 clean room the cathode and the grids are assembled and closed into the vacuum chamber.

chamber. The closed chamber is then connected to the pumping system outside the clean room. Due to the low height of the clean room, the chamber is modular (Fig. 3) and the whole system is mounted in several step, as shown in Fig. 4. Cathodes and grids are easily demountable as coating is foreseen for both high  $\beta$  and low  $\beta$  cavities.

### Bias Diode Sputtering

On the basis of the LNL experience with heavy ion cavities for LINAC ALPI, the technology for niobium on copper QWRs was started developing the DC Bias Diode Sputtering technique: the cylindrical cathode is surrounded by an external and an internal grid. The cathode is biased negatively, the grids are grounded and the cavity is slightly negative (around 80 V) in order to assure a soft resputtering of the growing film.

The main problem was encountered as soon as the cathode temperature raises: after 20-40 min the plasma disappeared from the outer part of the cathode. This gave rise to a non homogeneous distribution of the plasma. As a consequence a different sputtering rate was measured: after 2 hours of sputtering a thickness of 250nm was measured on the inner antenna while there was no measurable film on the outer wall. Even in the inner part the sputtering rate was too low ( $\sim 2$  nm/min).

### Magnetron Sputtering

To overcome the problem encountered with the DC Bias Diode Sputtering technique it was decided to test a cylindrical magnetron configuration.

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Figure 5: The multilayer coil, 1m diameter, surround the sputtering chamber. Calculations were run to simulate the axial magnetic field and optimize the coil heights.

Two coating test, developed in a smaller system, confirmed the difference in thickness distribution between the diode and the magnetron configurations. The magnetic field assures an acceptable thickness on both sides of the cathode.

Calculations were run to simulate the axial magnetic field and optimize the coil heights. A multilayer coil of



Table 1: Coating parameters

Parameters	test8	test9	test10	test11	test13
Pressure (mbar)	0.015	0.015	0.01	0.008-0.15	0.01
Cathode I (A)	3	3	3	3	3
Coil I (A)	50	40	40	40	40
Time (min)	240	410	420	420	410

1 m diameter, was built. Its dimensions and the number of layers were calculated in order to obtain a magnetic field which is homogeneous, higher than 100G and parallel to the cathode. More than six tests were run with it and the results are shown in Figs. 8 and 9.

The main advantages of this configurations are: stable plasma, improvements on the thickness, more homogeneous distribution of the plasma between the external wall and the internal antenna.

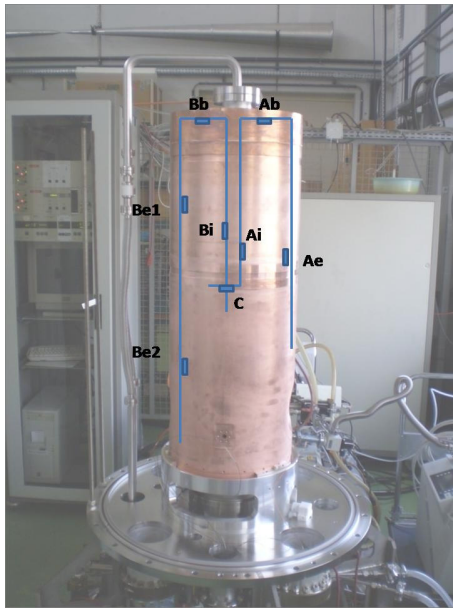


Figure 6: A sample holder, with a shape that follow the cavity walls, is placed inside the stainless steel cavity. Quartz samples are positioned along the sampleholder, on representative places of the cavity.

## RESULTS

During the first part of the R&D program the tests are performed with a stainless steel cavity with a shape similar to the QWR. A sample holder, which follows the cavity walls, is placed inside the stainless steel cavity. Quartz samples are positioned along the sampleholder, on representative places of the cavity [7]. For the sake of simplicity the cavity is divided into four areas: external wall (Ae and Be samples in Fig. 6), inner conductor (Ai and Bi), bottom part where the inner conductor is welded to the cavity outer wall (Ab and Bb) and central part on the top of the

inner conductor (C).

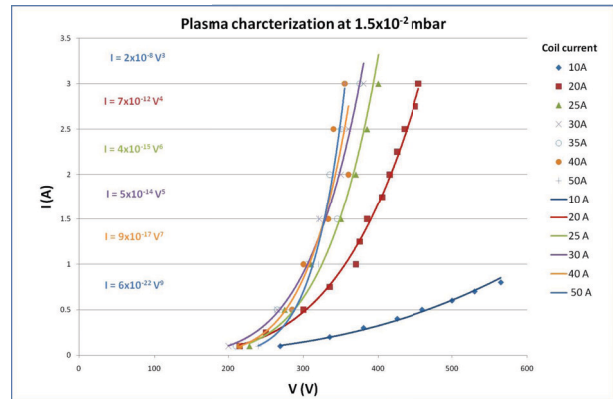


Figure 7: The plasma was characterized measuring the current as a function of the voltage for different coil currents and pressures. In this graphs the curves are recorded at  $1.5 \cdot 10^{-2}$  mbar: this pressure assures a uniform plasma distribution around the cathode.

To find suitable coating settings the plasma at different pressures and coil currents was characterized as shown in Fig. 7. Then some points of the I-V curves were selected and tested. The resistive properties (Residual Resistivity Ration RRR) of niobium on quartz samples were then measured and their dependence upon various coating parameters was estimated.

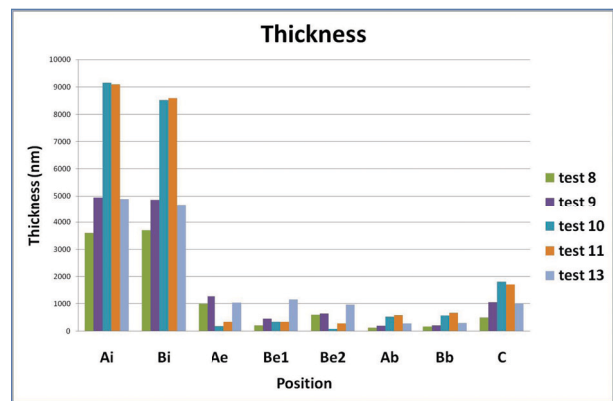


Figure 8: Niobium film thickness versus the position along the cavity wall (Figure 6).

The main aim is to obtain a homogeneous distribution of the film thickness along the cavity walls. Certain conditions of gas pressure, cathode current and coil current allow to obtain a good ratio between the film thickness on the external wall and on the inner conductor. Due to geometrical factors, this ratio cannot not be lower than four. Even if the magnetron sputtering guarantee a constant outer plasma, the thickness of the film on the bottom part is still low compared to the inner antenna. The problem of the coating on the bottom part of the cavity is magnified due to the fact that the magnetic field in that area is perpendicular to the cathode surface.

Several solutions for balancing the film thickness are under test. Solutions to modify the magnetic field shape or the cathode structure are under development.

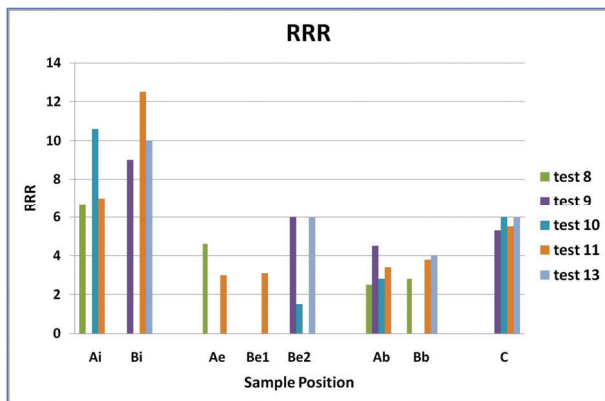


Figure 9: Niobium film thickness versus the position along the cavity wall (Figure 6).

### CONCLUSIONS

An R&D program to design and prepare new facilities for the surface treatment and the niobium coating of the HIE-ISOLDE superconducting cavities has started at CERN in 2008. Up to now the first copper QWR prototype was chemically treated and it is ready to be coated.

The DC Bias Diode Sputtering and the Magnetron Sputtering configurations were tested and niobium on quartz samples were characterized with thickness and RRR measurements. The sputtering conditions have still to be optimized to obtain a homogeneous coating. Solutions to modify the cathode structure and to balance the film thickness are under development.

### ACKNOWLEDGEMENTS

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