

# NIBIUM COATINGS FOR THE HIE-ISOLDE QWR SUPERCONDUCTING ACCELERATING CAVITIES

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## Abstract

The HIE-ISOLDE (High Intensity and Energy at ISOLDE) project is the upgrade of the existing ISOLDE (Isotope Separator On Line DEvice) facility at CERN, which is dedicated to the production of a large variety of radioactive ion beams for nuclear physics experiments.

A new linear accelerator made of 20  $\beta=10.3\%$  and 12  $\beta=6.3\%$  quarter-wave resonators (QWR) superconducting (SC) accelerating cavities at 101 MHz will be built, and in a first phase two cryomodules of 5 high- $\beta$  cavities each are scheduled to accelerate first beams in 2015. The cavities are made of a copper substrate, with a sputter-coated superconductive niobium (Nb) layer, operated at 4.5 K with an accelerating field of 6 MV/m at 10W Radio-Frequency (RF) losses ( $Q=4.5 \cdot 10^8$ ).

In this paper we will discuss the baseline surface treatment and coating procedure which allows obtaining the required performance, as well as the steps undertaken in order to prepare series production of the required number of cavities guaranteeing their quality and functionality.

## INTRODUCTION

The HIE-ISOLDE project is the upgrade of the ISOLDE facility located at CERN, which is dedicated to the production of radioactive nuclei for a number of applications covering nuclear-, particle-, and solid-state-physics, but also biophysics (radiobiology) and astrophysics.

The construction of the new superconducting linear accelerator (linac) for the energy increase of the beam (from 3 MeV/u to 10 MeV/u) requires the production of superconducting accelerating QWRs of two geometries, 12 with a geometrical  $\beta=0.063$  and 20 with a geometrical  $\beta=0.103$ , including an adequate number of spares.

The technology chosen for the production of HIE-ISOLDE superconducting cavities is the Nb/Cu technology [1],[2]. The reason of this choice is to combine the superconducting characteristics of niobium with the stiffness and high thermal conductivity of the thick Cu substrate, offering a valid alternative to bulk niobium resonators [3].

Of course, this was done at the expense of the added complication of the sputtering process of the niobium film, which was a challenging task in such a complex cavity geometry.

In this paper, we will describe the production steps of the first prototype  $\beta=0.103$  SC cavities which achieve the required RF-performances (given in [2]), from the cavity surface preparation to the baseline niobium coating recipe.

## 09 Cavity preparation and production

### I. Basic R&D New materials - Deposition techniques

## CAVITY PREPARATION STEPS

The baseline procedure to produce a sputtered QWR lasts about 4 weeks – 2 for the Nb coating and 2 for the RF-measurements.

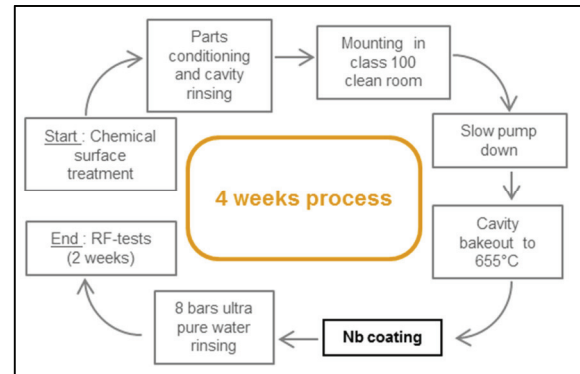


Figure 1: Summary of the 4 weeks process steps for the production of one cavity.

The production sequence comprises the following steps, also illustrated in Fig.1:

- Copper substrate surface treatments : Chemical polishing (SUBU) and passivation (Sulfamic acid)
- Dust-free copper substrate treatment : low pressure (8 bars) ultrapure water rinsing in clean room class 100
- Dust-free system assembly in clean room class 100
- Pre-heating of the copper substrate under vacuum : cavity bakeout temperature (635-655 °C), with  $T_{\text{bakeout}} > T_{\text{coating}}$
- Activation of NEG pump inside the chamber
- 8kW-power biased diode coating in several steps
- Dust-free sputtered cavity treatment : low pressure (8 bars) ultrapure water rinsing
- RF measurement

## Surface Treatments

The substrate surface preparation prior to coating is carried out by a 20 minutes chemical etching. The polishing agent (SUBU) is a mixture of sulfamic acid ( $\text{H}_3\text{NO}_3\text{S}$ , 5 g/l), hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 5% vol), n-butanol (5% vol) and ammonium citrate (1g/l) [4]. Its working temperature is around 72 °C, and the typical removal rate is 0.75  $\mu\text{m}/\text{min}$ , as confirmed by a test recently made. The cavity is then pre-rinsed with a solution of sulfamic acid, in order to pickle the surface. The typical roughness measured after a 20 minutes SUBU is 0.8  $\mu\text{m}$  [5].

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The cavity then undergoes a low pressure (8bars) rinsing with ultrapure water in a class 100 clean room.

### System Assembly and Coating Setup

The assembly of the system is made in a clean room class 100. The cavity is mounted coaxially with the cylindrical Nb cathode, which is itself surrounded by an inner and an outer grid for plasma polarization (see Fig.2). A heating system made of 3 Philips Infrared lamps of 2 kW power each, is then mounted outside the cavity.

The system is then sealed in a two-segments vacuum chamber with 2 DN630 Viton O-Rings. The use of Helicoflex gaskets has been tested for several cavities but these experiments showed that the improvement of vacuum induced by the metallic seals had no visible effects on the RF-performances of the cavities.

The closed and tight chamber is then connected to the pumping system outside the clean room, where the connection line is purged 3 times with pure nitrogen before opening the chamber via a manual valve. The system is then pumped at constant low flow (0.5 L/min) by means of a flow-meter, thus avoiding turbulences, preventing dust motion inside the chamber.

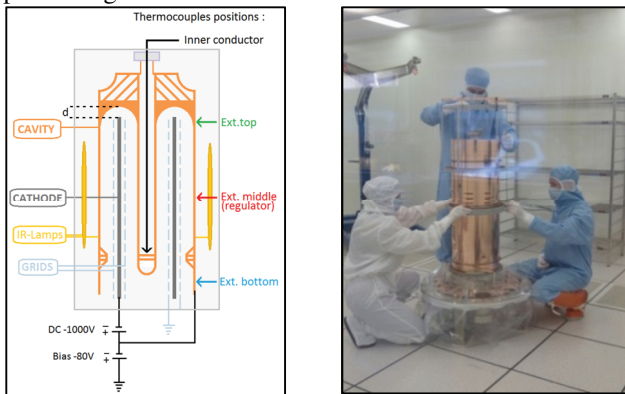


Figure 2: Coating setup (left) and system assembly in clean room class 100 (right).

### Bakeout and NEG Pumping

Once the system is pumped, it undergoes a phase of bakeout. A first phase of 6 hours, by means of an external bell surrounding the whole vacuum chamber, allows reaching 120 °C on the entire vacuum vessel and degases its surfaces.

An internal bakeout is then performed for 48 hours with the lamp system, allowing the cavity to be heated up to 655 °C on the external conductor and 635 °C (lower value) on the internal one. The purpose of this internal bakeout is to overcome the maximal temperature reached during the coating (about 635 °C), thus insuring that there is no niobium film contamination due to vacuum deterioration during the high-temperature coating.

After bakeout, a Saes Getter CapaciTorr D1000 NEG cartridge is activated, allowing a local pumping inside the chamber itself, thus improving the vacuum quality. The chamber is connected via a bypass to a residual gas analyser (RGA) allowing the species present in the

vacuum chamber to be identified and monitored at every moment of the bakeout, NEG activation, and coating.

### Coating Procedure

An example of copper cavity before and after the niobium coating is shown in Fig.3.

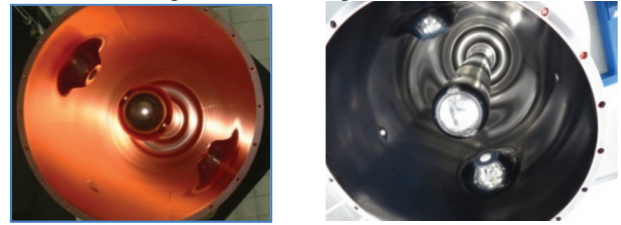


Figure 3: QWR before (left) and after (right) Nb coating.

The baseline coating is based on DC biased diode sputtering. The cathode is negatively polarized ( $\approx -1000$  V), the grids are grounded, and the cavity is negatively biased to  $-80$  V (Fig.2). Biasing the cavity allows a low-energy bombardment of the surface of the Nb film by the Argon (Ar) ions, thus densifying and smoothing the thin layer.

The sputtering parameters found to be the most suitable for the coating are; 0.2 mbar Ar pressure, 8 kW power, and a substrate temperature between 315 °C and 625 °C for the internal conductor, corresponding to an external conductor temperature varying from 300 °C to 435 °C (taken in the middle of the latter). All these parameters are automatically monitored during the process as shown in Fig.4.

The process is made in 14 runs, of about 23 minutes each (3 min for ramp-up at 8 kW, 20 min at nominal power), alternated with pauses in between, in order to prevent the cavity temperature overcoming the bakeout temperature. The limiting factor in our case corresponds to the inner conductor temperature that rises faster than the one on the external conductor due to the system configuration. Therefore, the coating is stopped before reaching 635 °C, and the cavity cools down naturally before the next cycle. Each cycle lasts 6 hours and the total process duration is 4 days.

The average film thickness after these 14 runs coating process reaches typically 7.5  $\mu\text{m}$  / 2.5  $\mu\text{m}$  / 1.7  $\mu\text{m}$  on the internal/ top/external conductor respectively [6].

Fig.4 shows the evolution of the parameters during the coating. On the left, the parameters of the entire deposition are depicted – so that the similarity of each run can be noticed –, whereas on the right, only a zoom of run 9 is magnified for clarity. One can see that the coating process is stopped when the inner conductor is at around 625 °C, but an overshoot of 10 °C happens. This overshoot is indeed taken into account not to overcome the 635 °C-bakeout temperature.

The cavity tuning plate is also niobium sputtered in another UHV coating chamber, with a planar magnetron setup at  $9 \cdot 10^{-4}$  mbar Krypton pressure, 150-200 °C temperature, and 600 W power. The plate is mounted on the cavity, and the RF-contact between cavity and plate is ensured by mechanical clamping with 72 screws tightened at 5Nm each.

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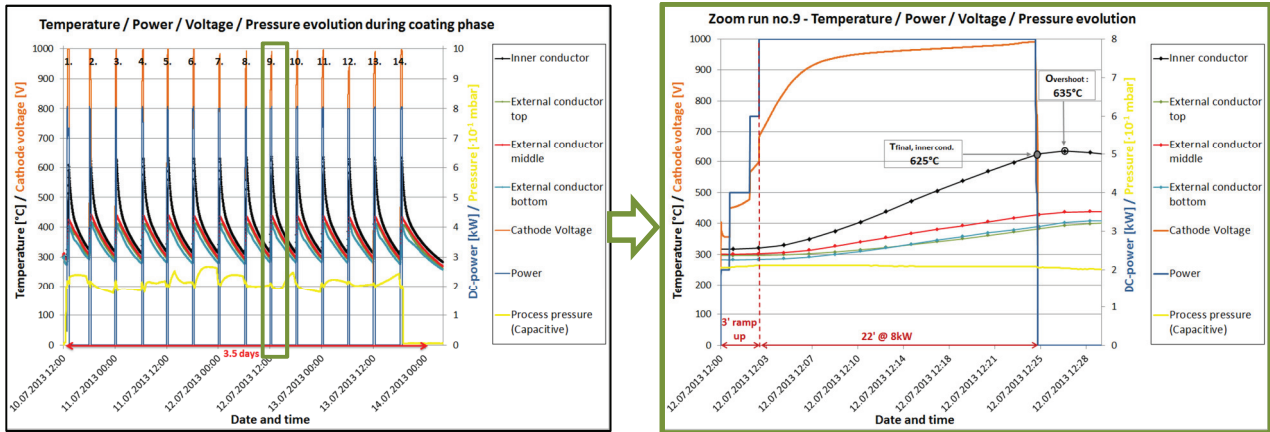


Figure 4 : Temperature, power, voltage, pressure evolution during the coating for the 14 runs (left) and a magnified zoom (green box) for a single run (right). The intervals over which the temperature rises correspond to the coating phases, and the ones where the temperature decreases correspond to the cooling down phases during which no coating is made.

### CAVITY RF-PERFORMANCES

The 101.28 MHz QWRs produced by the coating recipe mentioned above show a  $Q_0$  in excess of  $2 \cdot 10^9$  and overcome the specified performances at the nominal accelerating field of 6 MV/m at 10 W – with a margin of 30% on the cryogenic power. This baseline RF-result (orange diamonds) is illustrated in Fig.5, on which results of previous coatings setups are also depicted (blue/green).

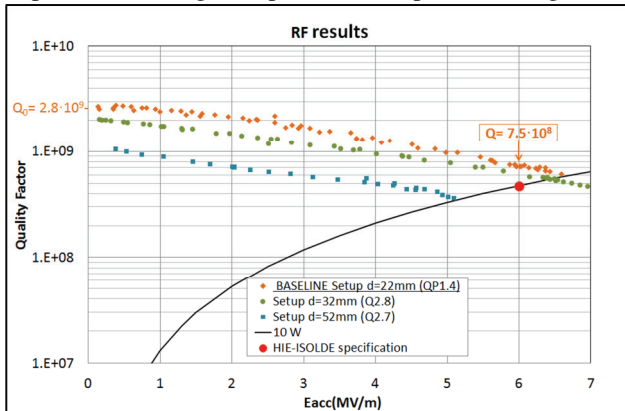


Figure 5 : RF results for 3 different cavities made with the same baseline coating recipe, but for different setups : distance  $d$  between cathode and top cavity is modified.

The baseline coating was actually chosen to be the one with the setup giving the best results – i.e. using the distance  $d=22$  mm between cathode and cavity top ( $d$  is defined in Fig.2 left). It is indeed noticeable that there exists a correlation between the increase of quality factor to the decrease of distance  $d$  [6].

### SUMMARY

Applying the coating procedure given in this paper, we are now ready to start the production of the 20 required high- $\beta$  cavities for the new HIE-ISOLDE linear accelerator. A new coating system is also under construction to start the optimization phase of the low- $\beta$

cavities and to continue the research and development of magnetron sputtering technique started recently [7] in parallel with the production.

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