Nb SPUTTERED QUARTER WAVE RESONATORS FOR HIE ISOLDE

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Abstract

The HIE-ISOLDE superconducting linac will be based on quarter wave resonators (QWRs), made by niobium sputtering on copper. The operating frequency at 4.5 K is 101.28 MHz and the required performance for the high beta cavity is 6 MV/m accelerating field for 10 W maximum power dissipation. These challenging specifications were recently met at CERN at the end of a vigorous development program. The paper reports on the progress of the cavity RF performance with the evolution of the sputtering process; it equally illustrates the parallel R&D which is on-going at CERN and at INFN in the quest for even higher performances.

INTRODUCTION

The technology of Nb sputtering on copper was invented at CERN [1], and initially developed there for the LEP cavities [2]. The state of the art SRF surfaces made at CERN were magnetron sputtered Nb films on elliptical cavities, for LEP and for future Linac Colliders. In the case of Quarter Wave Resonators (QWR) for heavy ions, different in shape, bias diode sputtering was developed and is used at INFN-LNL [3]. In 2007 Nb sputtering on copper was selected for the QWRs needed for the HIE-ISOLDE project at CERN [4], making particular reference to the sputtered QWR made at INFN-LNL for the energy upgrade of the ALPI linac [5].

The HIE-ISOLDE specifications call for an average surface resistance of 65 nΩ at 6 MV/m accelerating field, corresponding to the performance achieved in the best ALPI cavities. This is quite challenging in consideration of the much larger surface of the HIE-ISOLDE resonator (a factor 2.5), with the consequent higher risk of spurious defects on the substrates. On the other hand, the lower operating frequency (101 MHz vs. 160 MHz) and the use of clean rooms for cavity preparations should provide some safety margin.

Work at CERN to establish a complete production chain for Nb sputtered QWR started in 2008 [6].

By the end of 2009 the chemistry, coating and RF testing infrastructures were operational, the first prototype cavity had been produced, and it was ready to be coated.

COPPER CAVITY SUBSTRATES

The geometry of the HIE ISOLDE high beta cavity is optimized for the sputtering process, avoiding sharp edges on the high RF current regions, after LNL experience [4]. The cavity design and electromagnetic parameters are described in [7]. The substrate material is OFE copper. A first design relying on rolled sheets and extensive electron beam welding was initially adopted (with 4 prototypes built) for the early phases of the program. In a second time a new version, based on machining from bulk with minimal number of EB welds, was produced and 3 prototypes were manufactured. The shape of the helium reservoir was also modified. As a result, the sensitivity to the helium pressure fluctuations was decreased by two orders of magnitude down to 0.02 Hz/mbar. A detailed report on the copper substrates is in these proceedings [8].

The standard procedure for cavity surface preparation prior to coating, adopted in all prototype tests, is made of the following steps: chemical polishing (SUBU), high pressure water rinsing and drying with ethanol in a clean room class 100 [9].

Nb SPUTTERING METHODS

In the initial phase of prototype cavity development at CERN, two methods for Nb coating were pursued in parallel: bias diode sputtering and magnetron sputtering. The bias diode technique relies on high substrate temperatures and on the negative bias to release impurities and to promote mobility of the Nb atoms during film growth. Magnetron sputtering was considered to be promising as it provides high deposition rates at lower pressures, reducing the level of impurities and allowing keeping the process temperature low. This feature in particular was attractive to minimize cavity deformations. Since the end of 2011 the HIE-ISOLDE team at CERN concentrated the efforts on adapting the bias diode method (the system is shown in Fig. 1) to the HIE-ISOLDE cavity. Metrological controls and beam dynamics simulations were done to verify that the impact of the mechanical deformations induced by high cavity process temperatures was tolerable [10].

Early in 2013, after a sustained development program, the HIE-ISOLDE specifications were reached for the first time and later exceeded. More details on the diode coating procedure at CERN are given in a dedicated paper in these proceedings [9].

In the meanwhile, in the frame of EuCARD and in collaboration with CERN, INFN-LNL carried out R&D

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on magnetron sputtering on the HIE ISOLDE cavity geometry. In the second half of 2013, R&D on the magnetron method was also resumed at CERN, aiming at optimizing the coating time. Coating of one cavity with the diode method takes 4 days, while with the magnetron method it would take less than one day. This endeavour can take advantage of the experience accumulated during the previous phase and it is progressing quickly. One cavity has performed just below the HIE ISOLDE specifications. These new developments are reported in another paper in these proceedings [11].

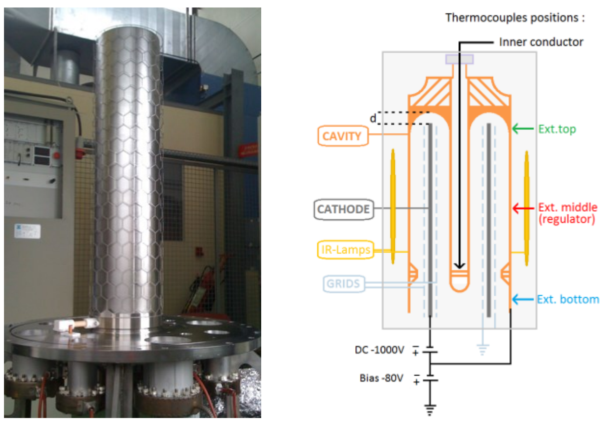

Figure 1: HIE-ISOLDE sputtering system (right: bias diode scheme, left: Nb cathode and external grid).

BIAS DIODE CAVITIES AT CERN

The production of Nb/Cu resonators is a complex process and the final result can be influenced by a huge number of factors, in part correlated. These go from the quality of the copper substrate (material purity, roughness, possible defects and porosities, cleanliness during and after the chemical steps, etc.) to the many sputtering parameters (geometric factors, temperatures, base vacuum and residual gases, sputtering pressure, bias voltage, sputtering voltage and current, nature of the process gases, etc.). The preparation for the RF test is also essential, in particular the manipulations to remove the sputtering gear, the ultra-pure water rinsing and drying, and the mounting of the bottom plate with the RF contact. The exact relative weight of each of these parameters in a given protocol which empirically proves to be effective is generally not completely known, due to the lack of time to carry out systematic studies.

The progression of cavity performance at CERN in 2012/2013 was accomplished in 13 iterations on four different prototypes. The cavities are named Qx_y where x is the serial number of the copper substrate and y is the progressive number of Nb coating. The work was guided by the knowledge of the ALPI procedure [12], by basic physics considerations and by an evolving understanding of the most influential parameters. It is well established that Nb films can display good RF properties provided that they are sufficiently pure and ordered. Moreover, a “good” microstructure is required, with well-connected grains and absence of voids. A useful (necessary but not sufficient) figure of merit used in sample studies to assess the film quality is the standard RRR. Furthermore the Nb film must adhere well to the substrate and resist the high pressure water rinsing without peeling off.

Differently from elliptical cavities, which are coated in a configuration where the cavity itself is the vacuum chamber, the quarter wave resonators are installed for coating in a large vacuum system, where the size of outgassing surfaces is much larger, increasing the risk of film contamination. On the other hand this configuration allows bringing the cavity to high temperatures without oxidizing it, and even the oxide layer on the copper substrate is dissolved during bake-out. An important step forward in our development was installing inside the vacuum chamber infrared lamps, by means of which higher temperatures could be reached during bake-out, thus allowing increasing the substrate temperature during the coating. High bake-out temperatures reduce the level of contaminants, notably hydrogen, and high deposition temperatures favour the desired microstructure [13]. Once high temperatures could be reached the attention was turned to the Nb deposition rate, which controls the final level of impurities in the film. The deposition rate could be increased by gradually pushing up the sputtering power (from the initial 2 kW to 8 kW). However, since the maximum substrate temperature and the total film thickness were fixed, and because the cavity heats up faster during coating at higher power, it was necessary to start carrying out the coating in steps, letting the cavity to cool down for several hours between the runs. As a result, the film is composed of “layers”. The nature of the interfaces between these layers and their possible influence on the film properties are unclear. Actually the number of layers and the peak deposition power cannot be dissociated in our setup.

Another variant introduced during the development was the nature of the working gas and of the gas used for the venting after sputtering. We started by using krypton and dry air respectively, following CERN experience [14]. In July 2012 we have changed to argon and nitrogen, according to LNL practices. It is likely that argon is better adapted than krypton to the bias technique because it preferentially re-sputters impurities which have a more similar atomic weight, while the re-sputtering of Nb is enhanced by Kr, reducing the effective coating rate.

The interesting question which gas is better to use when exposing for the first time to atmosphere the fresh Nb film after sputtering was not yet fully addressed.

The next parameter that we looked at was the film thickness. It is known that a minimum value, likely at least 1 µm, is required to have good RF performances. It was then tried to increase the coating time to get a globally thicker Nb film. This gave encouraging results, as it is visible in Fig. 2 (cavity Q2_7).

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A dummy copper cavity (Q4) had been designed as a sample holder with realistic thermal properties. Once that became available, the thickness distribution could be investigated on samples. It was realized that, as the plasma parameters evolved, films had been growing thinner than assumed from the total electric energy of the process, and that there was a dependence of the effective coating rate on slight changes of pressure. More importantly, it was apparent that the coating rate at the cavity top was much lower than anticipated. The sample study conducted with the Q4 mock-up is reported elsewhere in these proceedings [15].

Realizing the importance of the *local* sputtering rate opened the way to the next breakthrough. What was needed was to change the distribution of deposition rate (thickness), in order to increase it at the cavity top, where the maximum RF current is. This was done by reducing the distance between the cathode and the cavity top.

In summary, the main steps undertaken during the development were: increasing temperatures, increasing deposition rate and thus number of Nb layers, changing the working gases, increasing the global thickness, and increasing the local deposition rate at the cavity top.

RF TEST RESULTS

The evolution of the RF performance in vertical cryostat at 4.5 K is displayed in Fig. 2 below.

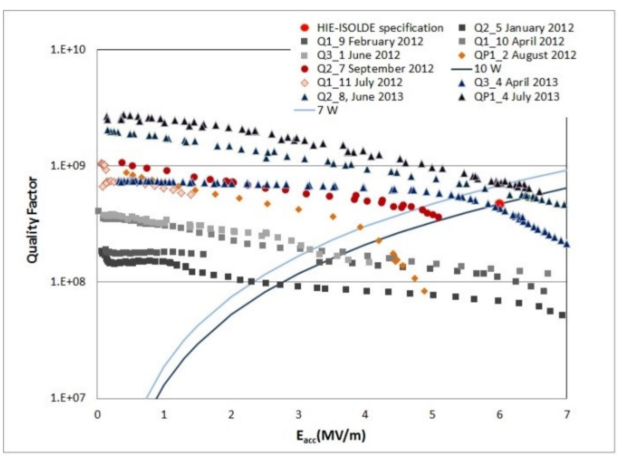

Figure 2: Q vs. E_{acc} curves of bias diode coated cavities.

Shown in the plot are the results after He processing, when applied. In some cases maximum performance was reached after having warmed the cavity up to 30 K, followed by a second cool down to 4.5 K. The present understanding of this phenomenon is in terms of thermoelectric currents trapping flux in the Nb layer during the superconducting transition. The vertical temperature gradient is reduced during the second cool down thereby reducing the trapped flux.

Along with the usual Q vs. E curve, in few cases the shift in resonance frequency as a function of temperature was measured. The shift is related to the change of penetration depth in the superconducting state and was fitted with the Gorter-Casimir temperature dependence [16], using a procedure described in [17]. The fit allows extrapolating the penetration depth at 0 K, which in turn is related to the mean free path and thus to the average RRR of the film. The results, shown in Table 1 below, document the evolution of the RRR over time.

Table 1: Evolution of RRR During Cavity Development

Coating test	λ_0 (nm)	RRR
Q2_3 April 2011	188	1.9
Q1_5 June 2011	83	6.8
Q1_10 Feb. 2012	62.3	13.5
Q2_8 April 2013	50.7	26.4
Q3_4 March 2013	45.7	41. 8

SPECIFIC ISSUES

A few more specific problems had to be tackled on the way to higher RF performance. They are documented for completeness hereunder.

Quality of the Inner Conductor Tip Surface

As the power of the sputtering discharge got increased we faced a degradation of the sputtered film at the inner conductor tip (Fig. 3, left). The surface displayed macroscopic roughness (orange-peel texture); adhesion of the coating was poor and the Nb layer did not withstand low pressure water rinsing.

These cavities, although they may show high Q values at low field and low Q slopes, were limited by strong field emission, which was understandable as the damaged zone corresponds to the peak electric field.

Figure 3: Surface of the inner conductor tip before and after removing the central electrode.

This problem had been known during the development phase of the ALPI cavities and it had been solved by optimizing the geometry of the central electrode facing the cathode [12]. Several attempts were therefore made at CERN, with electrodes of different shape and diameter, until it was noted that the size of the damaged zone was directly correlated with the diameter of the electrode. Removing the electrode completely produced a smooth surface (Fig. 3, right). A cavity (Q2_8) was then made which was free from field emission up to 7 MV/m.

Q Switches

Some cavities displayed so-called Q switches, whereby at a given threshold in field the intrinsic Q dropped dramatically. This phenomenon is due to the sudden transition of some portion of the cavity surface to the normal conducting state. In LEP and LHC times, small Q switches were observed when parts of the coating lost contact with the substrate and got heated above Tc by the RF field [18]. Investigations were conducted by simulating the effect in the hypothesis of quenching at various cavity locations, and by placing thermometers and heaters on the bottom plate to induce and observe the transitions. Finally a cavity, which had been plagued by Q switches, was re-measured, having only changed the tuning plate fittings, and it was found that the Q switch had disappeared. It was then concluded that the problem was located in the bottom plate and was therefore not intrinsic to the coating.

Quality of the RF Contact Surface

The relatively short distance (70 mm) between the inner conductor tip and the bottom plate created the risk of losses in the RF contact, which is not superconducting. The magnetic field at that location would reach ~1.7 mT at the working point, which was unsatisfactory [19]. A dedicated experiment with an indium gasket led to exclude that the low *Q* at the time were due to the RF contact. However, to be on the safe side, the next prototypes were made with longer tip-gap distances (80 mm and 90 mm). This in turn created some problems of adhesion of the Nb film on the cavity lower edge (Fig. 4, left), due to the less favourable exposure of this surface to the sputtering cathode (smaller integral Nb flux and grazing angles). The problem could be mitigated (Fig. 4, right) by using a longer cathode, even though the need for Nb coating in this area is unclear.

Figure 4: RF contact surfaces after rinsing: adhesion improved using a longer sputtering cathode.

Cavity Conditioning Issues

Multipacting levels at low field are usually conditioned during the cool down of the cavity. Helium processing was applied whenever clear signs of field emission appeared (X rays and heating of the bottom part of the cavity). We used helium pressures of about 10^{-5} mbar and carried out the conditioning in CW trying to maximize the cavity power compatibly with the available cryogenic power. The procedure was effective in most cases, shifting the onset of emission to higher fields, but sometimes we also observed degradation of the performance after strong processing events. The limited time available for the RF tests did not allow checking whether such performance losses would be reversible by applying more conditioning.

TUNING PLATE

The bottom plate is made of OFE copper with a magnetron sputtered Nb layer. It is clamped to the cavity by means of stainless steel collars housing 72 M6 screws closed with a 5 Nm torque, which push on titanium rings compensating the differential thermal contraction between copper and stainless steel. The required tuning range is at least 30 kHz and the tuning resolution should be less than 1 Hz. The detailed design of the plate and of the tuning system mechanism is being finalized; a prototype was recently tested and fulfilled the specifications. The tuning system and tuning strategy are the subject of a dedicated paper in these proceedings [20].

DISCUSSION AND OUTLOOK

As it is shown in Fig. 2, the cavity performance constantly progressed over time. Changes in the procedure which gave good results were kept in for all the subsequent cavities. For a tentative interpretation, we divide the test cavities in four groups: the square symbols correspond to tests at increasing coating temperatures and deposition rates, but with bake-out temperatures still lower than those reached in the sputtering process. Diamonds correspond to cavities with $T_{(baking)}>T_{(coating)}$ but also higher deposition rates/number of layers and different sputtering gas. The cavity Q2_7 (circles) was sputtered with all the previous changes implemented but adding 25% more thickness. Finally triangles correspond to cavities coated with progressively lower distances between the cathode and the cavity top. In this configuration, the specifications for HIE-ISOLDE have been exceeded reaching an average surface resistance of 43 nΩ at 6 MV/m accelerating field. This translates in 7 W power dissipation, which guarantees 30% margin with respect to the available cryogenic power.

Not shown in Fig. 2 are cavities affected by vacuum leaks or contamination during coating. Also not shown is the result of the cavity sputtered after Q2_7, when we tried to increase the sputtering power from 8 kW to 12 kW. In doing so the sputtering pressure had to be increased to sustain the discharge, as the maximum voltage of typical power supplies for sputtering is limited to 1 kV. Disappointingly, the RF performance of this cavity was inferior to that of Q2_7. Tests on samples indicated that the increase in pressure had the side effect of reducing the effective coating rate.

There was no time yet for a full optimization of the parameters which are considered to be relevant, for instance the bias voltage and the sputtering rate. In order to further increase the deposition rate with the bias diode method, a new power supply capable of supplying more than 1 kV is being purchased. Preliminary tests also showed that coatings at lower power and lower pressure show a larger sputtering rate, for a fixed maximum sputtering voltage.

More studies will be needed to disentangle the influence of sputtering rate, coating temperature and of layered coating on the surface resistance of the film.
The level of performance reached so far is sufficient to start the series production, but work will continue in order to secure more margins and cover possible performance losses from the vertical tests to the Linac operation.

DEVELOPMENTS AT INFN-LNL

A system for magnetron sputtering HIE-ISOLDE cavities was set up at INFN-LNL. The vacuum system and the Nb cathode are shown in Fig. 5 below.

Figure 5 HIE-ISOLDE sputtering system at INFN-LNL.

The Nb cathode is a double wall water cooled cylinder rounded at the top (Fig. 5) to follow the substrates shape. A systematic test campaign on samples, using a stainless steel cavity provided by CERN, was carried out to study the superconducting properties of the Nb film at various locations in the cavity, as a function of the sputtering configuration and parameters.

The setup achieved high sputtering rates (above 10 Å/s), and very good thickness homogeneity (2±1 µm).

The working pressure is in the range of 10^{-2} mbar, increasing the argon pressure improved the thickness homogeneity along the cavity.

Deposition temperatures are not easily measurable with the stainless steel sample holder and the quartz samples; however the cavity is heated up at 350 °C during the process by means of infrared lamps.

The measured RRR as a function of the sputtering power at half height of the external conductor is shown in Fig 6.

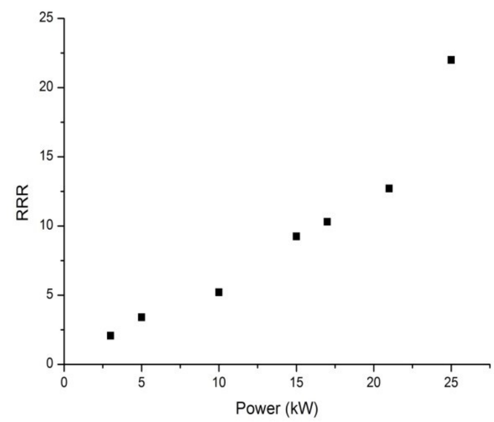

Figure 6: RRR vs. sputtering power of Nb samples at LNL.

This preliminary work allowed selecting a suitable range of sputtering parameters. The next step will be the sputtering and test of a real copper cavity substrate of the HIE ISOLDE type.

CONCLUSIONS

Besides its value for the nuclear physics community, the HIE-ISOLDE project, with its choice of the Nb/Cu technology for the QWR, acted as a test bed to rebuild the CERN infrastructure on SRF thin films for accelerating cavities, after the LHC era.

The project created an opportunity to advance the R&D on two different techniques of Nb sputtering, the bias diode method traditionally applied to this type of cavities, and the promising approach based on magnetron sputtering, investigated in parallel at CERN and at INFN.

A strong development campaign, focused on the bias diode method, led CERN to achieving and exceed in early 2013 the challenging HIE-ISOLDE specifications of 6 MV/m accelerating field at 10 W power dissipation.

While work continues to improve our understanding and to push the performance even higher, the currently developed procedure is mature to be applied, in time for the imminent start of the series cavities production for the first HIE-ISOLDE cryo-module

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