SPUTTERING OF NIOBIUM THIN FILMS ONTO COPPER QUARTER WAVE RESONATORS

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Abstract

Niobium Sputter-coated Copper Cavities can represent an elegant solution for high performance resonators.

Respect to bulk Niobium, the improved thermal conductivity of 1 μm Niobium sputtered on OFHC Copper, together with an important reduction of material costs, make the sputtering solution an attracting feature even for heavy ions resonators.

The Sputtering of Niobium in a DC Biased Diode configuration has been investigated for Legnaro OFHC Copper QWRs. By such a method high purity superconducting Niobium thin films have been deposited onto the internal surface of resonator with a uniform thickness of 1 ± 0.15 μm and a critical temperature $T_C \geq 9.2$ K.

The sputtering feasibility of high quality superconducting Niobium thin films onto Copper QWR has been demonstrated.

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Introduction

Thanks to the results obtained at CERN by Benvenuti et al. [1-3], the sputtering of Niobium onto Copper resonators is now a well-established technology for 500 MHz and 350 MHz electron cavities.

The better rf performances respect to bulk Niobium resonators, the improved thermal stability of the Nb/Cu system and the experimental evidence that residual losses are not affected by the earth magnetic field trapped during cooling into the superconductor [4,5], together with a considerable costs reduction due to the material saving are the advantages of the sputtered Niobium solution for electron cavities.

Some of these benefits are assumed to hold also at lower frequencies. That makes the technology of Niobium sputtering very attractive even for heavy ions cavities.

Unfortunately the tricky shape of the several kinds of heavy ions cavities is often a limitation to the development of sputtered resonators.

For sputtered electron cavities there are already about ten years of research [3]. Even superconducting materials having Tc higher than the one of Niobium, for instance NbTiN [6-8], are investigated at the moment.

On the contrary the status of art for heavy ions resonators is not so advanced: Lead is still the favourite superconductor whenever superconducting cavities are needed in a rather short time and cheap way. Even bulk Niobium is not easy to apply to all kinds of heavy ions resonators, and the sputtering technology only recently has been considered for investigation [9,10].

From this point of view the 160 MHz Quarter Wave Resonator (QWR) adopted in the ALPI booster of LNL have a shape simple enough to be sputtered (fig. 1).

Hence in 1988 a research program on Niobium sputtered Copper QWR started and a laboratory fully dedicated to this topic has been created. The main philosophy [9,11] was that the production for the LINAC started with Lead electroplated OFHC Copper QWR adopting just the same shape of cavity studied for the sputtering of Niobium.

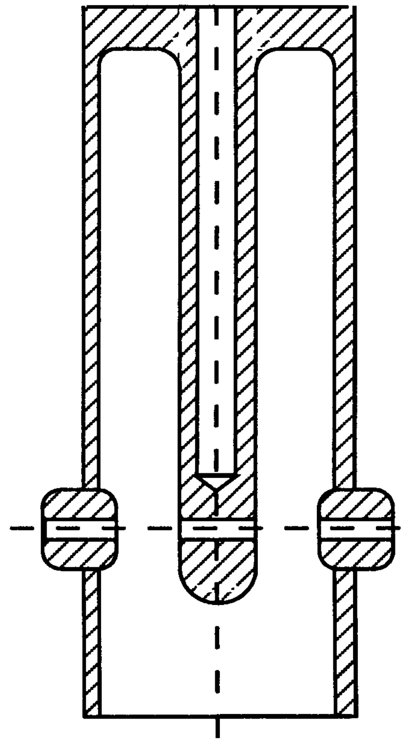

Fig.:1 *The 160 MHZ QWR adopted for ALPI.*

The investigation phases will terminate and the sputtering technology will be defined well-established when it will exist a good statistics on curves of the Q factor versus the accelerating field. Hence we will establish a precise "recipe" so that the technology can be ready to be transmitted also to industries or to other institutions. At that moment the ALPI Lead cavities can be stripped and, four by four, they can be coated with Niobium.

The Sputtering Configuration

The goal of the project consists in the production of few QWR prototypes sputter-coated by an high purity superconducting Niobium film having Tc not less than 9.25 K, the Nb bulk Tc. Good thickness uniformity of the Niobium coating is also requested: the film indeed must be thicker than about 0.5 µm to avoid any penetration of rf fields into the substrate, but thinner than about 4÷5 µm to avoid that peeling of the coating from the substrate arises due to the different thermal expansion coefficients of Copper and Niobium. A thermal excursion of about 700 K occurs indeed since the cavity is cooled in the sputtering chamber from about 400°C (the substrate temperature during deposition) to room temperature and from this to the liquid Helium temperature. Although the thickness range is rather wide, a thickness of 1 µm is usually deposited since the thinner is the film the higher is the thermal stability of the Nb/Cu system.

In order to build a cathodic structure that would be the most simple and would provide high thickness uniformity, the technique of DC Biased Diode sputtering (cathodic sputtering) has been preferred to the one of magnetron sputtering. A magnetic confinement of the discharge indeed has the advantage of high rate sputtering, but many difficulties arise whenever high thickness uniformity on large substrate areas is requested. On the contrary such problem can be solved by cathodic sputtering since sufficient thickness uniformity is insured, provided that the cathode follows rather faithfully the shape of the cavity i.e. the distance between target and substrate is approximately the same in any region of the cavity.

The final cathode configuration is represented in fig. 2. In its extreme simplicity, it presents few peculiarities [12] that make it preferable among all the others considered. The target is indeed just a Niobium cylinder depositing simultaneously onto the inner stub, the external conductor and the top plate.

If the cathode radius R_c is approximately equal to

$$R_c = \frac{R_i + R_e}{2}$$

where R_i and R_e are respectively the radius of the inner shaft and that of the external cylinder, one gets approximately equal thickness on the two cylinders.

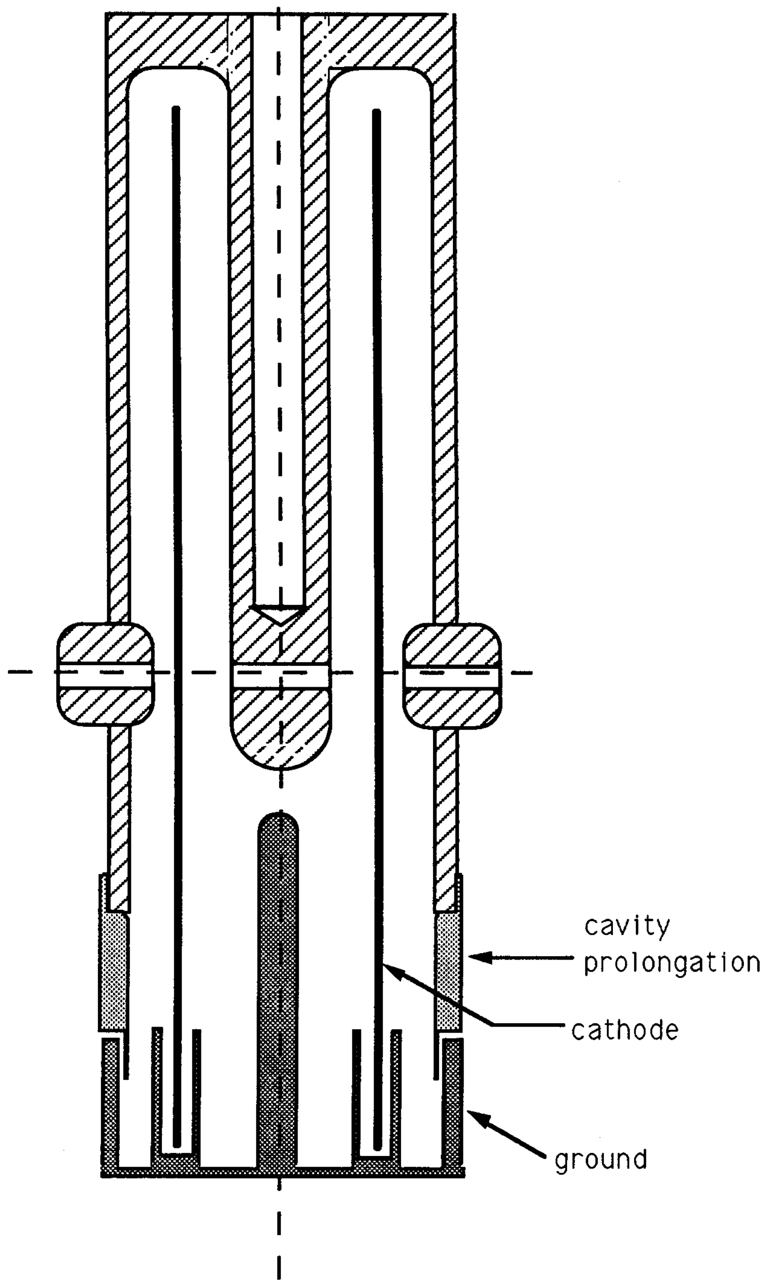

Fig. 2 *The Biased DC Diode Sputtering configuration.*

That is not totally obvious since while almost all the material leaving the external surface of the target reaches the copper substrate, not all the Niobium leaving the internal surface of the target is collected by the copper shaft: some material falls again back to the target and from there it is resputtered again (fig. 3). Really the problem is more complicated since a certain fraction of the Niobium atoms is back-sputtered to the target. Moreover the sputtering from the internal and external cathode surfaces correspond to different discharge regimes.

The thickness distribution has been obtained plugging several samples of Corning glass or Copper inside a dummy resonator. The thickness of samples was after measured by a surfometer.

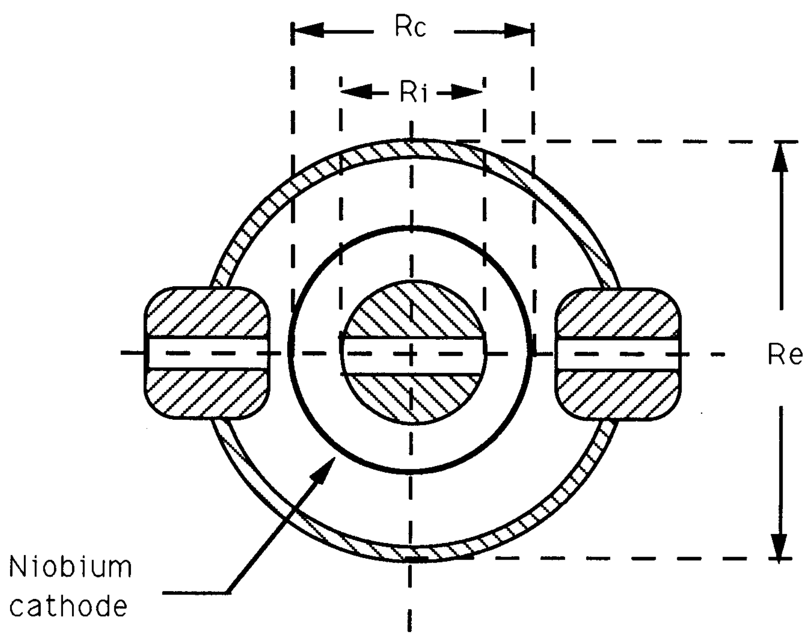

Fig. 3 *Cross section of the cavity and of the Niobium cathode.*

The right radius of the target has been found studying the difference in thickness between the coatings on the internal and on the external cylinders versus the target diameter. This curve has been obtained trying, run by run, several cathodes of different diameter. Such cathodes were made of stainless steel, indeed it is enough to scale the obtained results by the Niobium sputtering yield. The final cathode was done by electron beam welding a 2 mm rolled Niobium slab. RRR of the slab was 250.

The deposition on the top plate is simply provided by the sharp edge on the top of the target. Indeed the tremendously high concentration of electric field lines at this edge results in a much higher sputtering rate. Once having fixed the radius of the Niobium target, we sputtered different runs varying the distance **d** between the edge of the target and the top plate. The best thickness uniformity for the top plate is obtained for

$$d = R_e - R_c$$

Following such a methodic a uniform thickness of 1 ± 0.15 μm all over the cavity has been achieved by a single step deposition.

It must be remarked that the target is not cooled. This implies a considerable simplification from the technological point of view. In fact the target cooling can be useful but is not compulsory.

One interesting sputtering configuration of the several tested is shown in fig. 4. The idea of having separated cathodes for the different regions to coat is to "decouple" the different discharge regimes, splitting so a complex and manifold problem in some elementary and more easy. Moreover it is possible to work with lower currents, that means a better stability of the discharge.

Therefore the three cathodes variant has been investigated driving them both simultaneously and subsequently, both with and without ground. The variant of only two cathodes without hat on top and without ground has been also studied, both in the case of electrodes driven separately and driven at the same potential.

As a result of the whole analysis we have found that the single target configuration is among all the least complicated and the most functional. Moreover a target structure the less thick as possible is needed in order to get sputtering in the holes of beam ports and in the hole of the donut. Indeed there is a risk that the plasma can be suppressed wherever the target-substrate distance becomes comparable to the Crookes dark space (fig. 3).

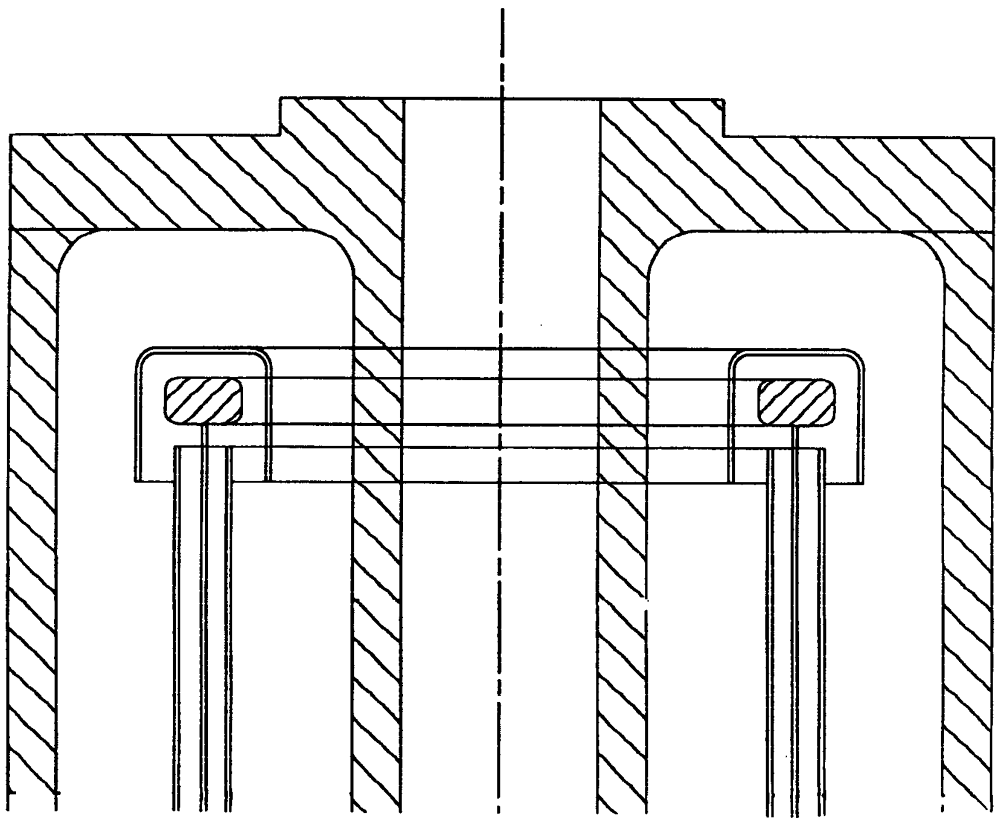

Fig. 4 *Top detail of the three cathodes variant.*

In order to get a high quality Niobium film inside the holes of beam ports many different cathodic configurations have been tried [12]. Except the hole, no particular problem exists to coat the beam port region since it gets sputtered with reasonable uniformity during the normal deposition.

The coating of the beam port hole is a separate problem. We desired to get the coating inside the hole while sputtering the cavity. A single step deposition indeed is preferable to multi-step depositions since the interfaces among Niobium layers deposited in different runs are always "dirty". Moreover in some regions it is unavoidable the presence of Niobium sputtered at low rate, that usually has not good superconducting characteristics.

Many variants have been explored and among them two ,seem to be the most meaningful.

The former requires a Niobium rod at the same potential as the Niobium target laying along the beam axis and crossing orthogonally the target (fig. 5b). This geometry will provide sputtering along the whole beam port.

The latter variant does not foresee any supplementary target. The plasma indeed is extracted into the beam port hole by a simple Niobium wire (or even a wire ending with a small grid) inserted into the hole parallelly to the beam axis (fig. 5c) and positively polarized respect to the ground. On the basis of our investigation both solutions are extremely interesting and each one of them presents advantages and drawbacks. This latter one indeed is very simple to construct and to work with; moreover no further modify must be added to the target, but it needs an additional power supply. The film in the last case has good superconducting properties for about one diameter along the hole.

According to the results of reference 13 after one diameter the quality of the coating becomes no significant. Further investigation is needed to establish which solution is the most convenient. No particular problem subsists for the deposition into the donut hole, since the distance from target to donut is larger than the one from target to beam port (fig. 4) and since there is sputtering from both sides.

DC biased diode sputtering in planar configuration is used for coating the cavity bottom plate with Niobium. Values of RRR around 12 and T_c between 9.25 K and 9.45K are found in such a case. Obviously the thickness uniformity is higher than in the cylindrical configuration.

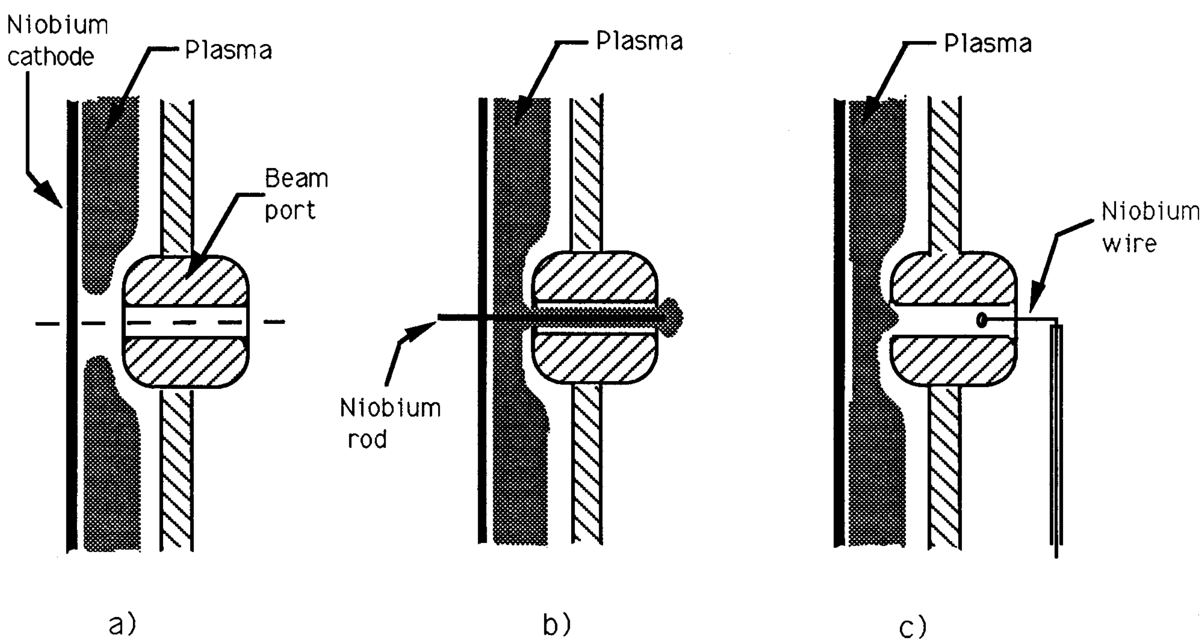

Fig. 5 *The sputtering in the beam port region:*
a) due to the short distance between cathode and beam port, a hole in the plasma appears if no precaution is taken.
b) An additional cathode (just a Niobium rod) and the right adjusting of the Argon pressure suppress the plasma hole.
c) A grid positively biased in front of the cathode, but inside beam port, also promotes plasma there.

The Niobium sputtering

High purity superconducting Niobium thin films all over the cavity surface have been deposited during a single step process with a thickness of 1 ± 0.15 µm obtained in a run of approximately half an hour.

The sputtering discharge is performed at the following parameters:

Initial Pressure	≈	$10^{-9} \div 10^{-8}$ mbar
Argon pressure	≈	$5 \times 10^{-2} \div 2 \times 10^{-1}$ mbar
Discharge Voltage	≈	$-1.4 \div -2$ KV
Discharge Current	≈	$3 \div 5$ A
Bias voltage	≈	$-80 \div -130$ V
Bias Current	≈	$1 \div 1.5$ A
Substrate temperature	≈	300 K $\div$ 700 K
Grid potential (if used)	≈	$+5 \div +25$ V
Grid current	≈	$0 \div 2$ A

Samples show Residual Resistivity Ratio (RRR) values between 8 and 18 and critical temperatures $T_C \geq 9.2$ K (fig. 6). Diffractometric studies have been performed showing highly oriented films. Scanning microscopy reveals an average grain size around 500 Å. RBS analyses, within the limits of its sensitivity, has revealed no impurity inside the Niobium film.

The sputtering parameters listed just above are critical for the good superconducting properties of the coating and some parameters are even more important than the others (the film microstructure, for example, strongly depends on the bias voltage). However it is remarkable that two different depositions, executed with the same sputtering parameters, give exactly the same distribution of RRR and Tc, showing the high reliability of the process.

Another important information given by samples, averaging over about fifteen depositions, is that the highest quality of the deposit is regularly found on the top part of the cavity, that is a critical place since the maximum of radiofrequency currents are located there. In fact any impurity floating in the sputtering chamber will not reach such deep region since it will be gettered by the film growing on the wall in proximity of the opened basis of the cavity. To avoid contamination there too, we plug the cavity to be sputtered on a stainless steel tube of the same diameter. In such a way any kind of impurity entering the cavity will be stopped by the film deposited onto such a "resonator prolongation".

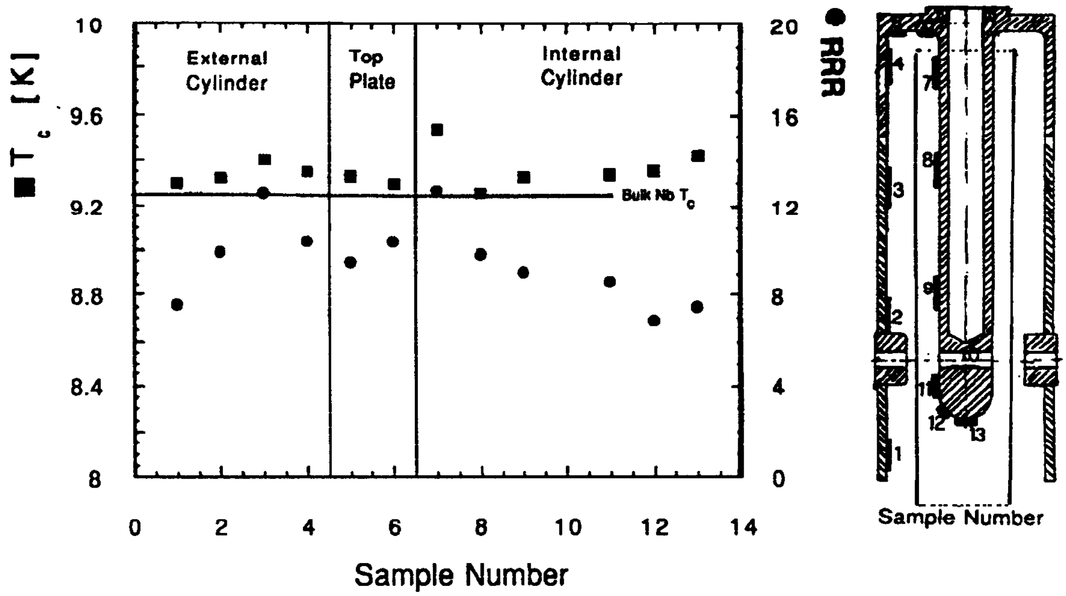

Fig. 6 *Distribution of T_C and RRR versus the sample position.*

No difference in sputtering regimes has been observed when sputtering onto the dummy cavity or onto the real Copper cavity. Before the deposition the Copper substrate is tumbled, after is chemically etched according to the standard CERN recipe [14], rinsed with ultrapure deionized water, dried with ethylic alcohol and baked under ultrahigh vacuum at 400°C.

The first OFHC Copper QWR prototype has been Niobium coated and performed for the rf measurement. For the first prototype we preferred to dial initially with a model exactly equal to the real cavity, but with the only difference that beam ports were absent.

When mounted into the cryostat, because of a failure of the temperature controller, the cavity was heated up to more than 200 °C and a leak appeared on an Indium sealing: the Niobium coating remained exposed to the atmospheric pressure for several hours.

Notwithstanding this, the cavity was still superconducting but not high values of Q-factor and accelerating field were obtained.

Conclusions

From such a status of art we can deduce one general consideration: the sputtering technology can be applied to QWR. The "difficult" shape of such a resonator is not at all an obstacle to the feasibility of sputtered Nb QWR. Samples show that high quality superconducting films are obtainable all over the surface.

Our current program consists in coating, stripping and coating again a certain number of resonators to see, apart of accidental problems, how rf performances are related to the sputtering parameters.

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