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"Surface Treatments for Industrial Applications"

A miniaturized 6 GHz infrastructure for cutting down the cost of RF superconducting research

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INTRODUCTION

Since the International Committee for Future Accelerators recommended that the Linear Collider design has to be based on the superconducting technology, the scientific world interest is now focused on further developments of new resonant cavities fabrication techniques and cost reduction.

It is important to pursue research on new materials: the goal will be the achievement of superconducting cavities working better than the Nb ones at 4.2 K.

However the high beta 1.5 GHz resonant structure research, especially in this time of international economic recession, would become prohibitive and onerous both for the material costs, of production and treatments, and the cryogenic expense.

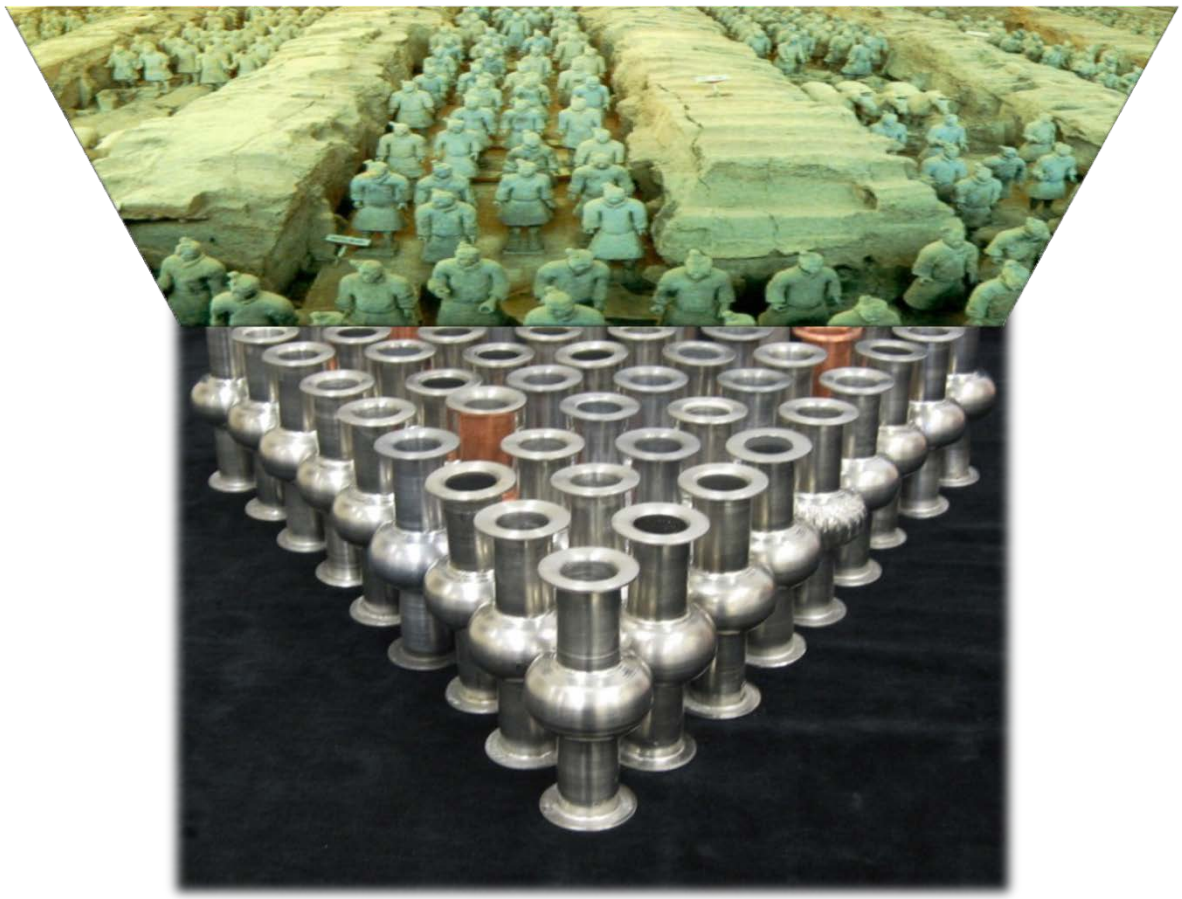
For this reason it is mandatory for the future of superconducting resonant cavities to cut down the costs introducing a new research concept. The idea is to build micro cavities completely equal in shape to the real scale model. The RF characterization of samples is an useful diagnostic tool to accurately investigate local properties of superconducting materials. But, a common limitation of systems used for this, often consists in the difficulty of scaling the measured results to the real resonator. In this work we will proof that 6 GHz resonators can simply become our cavity shaped samples.

We will proof that a mini low cost laboratory could be set up to study our 6 GHz samples in all the aspects of interest. The mentioned mini-lab consists in:

- A reduced size mechanical polishing bench
- A chemical/electrochemical minilab for BCP EP
- A mini oven for thermal treatments
- A miniaturized sputtering system
- An inexpensive cryogenics and quick RF measurements

We will show that with a tool like this, it is possible to study traditional and innovative surface treatments and performing RF tests on a large amount of cavities. A research budget much lower will allow to treat and tests a high number of cavities. One can study traditional surface treatments on Nb like: mechanical polishing, buffer chemical polishing, electro polishing, high pressure rinsing (HPR), alcohol rinsing and thermal treatments. It is also possible to study alternative thin film superconducting materials grown for example by sputtering, thermal diffusion or ALD on the inner surface of the 6 GHz samples.

With such a large “army of small soldiers”, as the 100 spun resonator fabricated every new idea would be easily tested at a reduced price and pushing the limit over will become real!!!



Chapter 1

TRADITIONAL SURFACE RESISTANCE MEASUREMENTS

In this chapter a brief review of some of the main methods developed to measure superconductor surface resistance (R_s) is reported.

1. An inverted microstrip ring resonator technique was produced by a collaboration between Naples and Salerno Universities in order to measure the RF properties of superconducting films in the framework of the search for new materials for sputter coated superconducting RF cavities for particle accelerators [1].

Advantages: high sensitivity, any kind of sample could be measured.

Disadvantages: approximative evaluation of the radiative losses, difficulties in photolithography preparation of the samples and of the antennas.

2. A 1.5 GHz superconducting niobium triaxial cavity has been prepared by Jefferson Lab Researchers (Newport News, Virginia, USA) to study residual surface resistance of superconducting materials at 2 K[2].

Advantages: high sensitivity 0,02 nΩ, value of R_S at the frequency of interest.

Disadvantages: possibility to have field emission and parasitic losses at the sample edge, difficulties to reach the sample/cavity ideal thermal contact (through calorimetric method).

3. A 400 MHz niobium quadrupole resonator has been manufactured at CERN to study the RF properties of superconducting bulk and thin film samples at low temperatures. Surface resistance of a bulk niobium sample as a function of temperature and applied RF field has been investigated using a calorimetric "RF-dc-compensation" method.

Advantages: high sensitivity.

Disadvantages: possibility to have field emission and parasitic losses, necessity to weld the sample to the cavity, value of R_S obtained at a frequency of 400 MHz.

1.1 Microstrip resonator technique

The inverted microstrip ring resonator is a technique that allows surface impedance measurements on films deposited on different, small area, substrates as a function of temperature and dc and RF fields in the range 1-20 GHz. The idea is to create a micro shaped ring resonator made of the material under investigation. The surface resistance is measured through the quality factor of the resonator.

The resonator is formed by the superconducting thin film under test, deposited on a substrate, where a narrow ring geometry is defined by a standard photolithographic process, placed on top of a thin (d = 130 μm), low loss, sapphire substrate in an "inverted microstrip" configuration. The resonator is completed by a ground plane made of the same superconductor (or of a well characterized superconductor) and is enclosed in a gold-plated copper box (*Figure 1-1*).

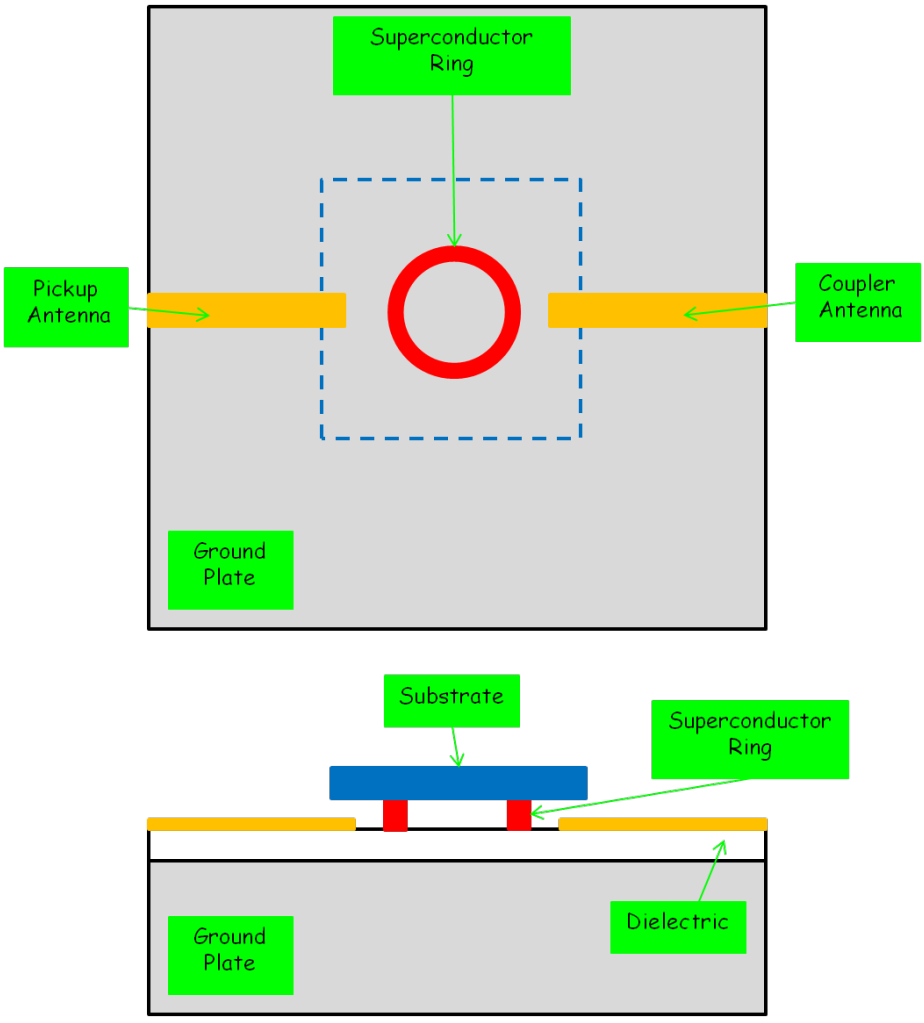


Figure 1-1 The inverted microstrip ring resonator assembly. The resonator is formed by the ring shaped superconducting thin film under test on a suitable substrate, placed on top of a sapphire dielectric, in an "inverted microstrip" configuration. The resonator is completed by a ground plane and by two 50 Ω microstrip antennas.

Two 50 Ω microstrip line antennas are used to excite and pickup the microwave signal from the resonator. Standard launchers are used to connect the antennas to 50 Ω coaxial cables. The RF power supply used for the measure has an upper frequency limit of 20 GHz (-20 - 10 dBm). The system temperature can be easily varied and controlled in the range 1.4 - 300 K. A uniform dc field (< 10 mT) can be applied by means of a pair of Helmholtz coils.

The resonator working frequency is determined by the ring diameter D and the order of resonant mode *n* through the relation:

$$\omega_n = \frac{2nc}{D\sqrt{\varepsilon_{eff}}}\left[1 + \left(\frac{\lambda_1+\lambda_2}{d}\right)\right]^{-\frac{1}{2}} \qquad 1\text{-}1$$

where $\lambda_{1;2}$ are the effective magnetic field penetration depths in the film and in the ground plane and ε_{eff} is the effective dielectric constant (ε_{eff} is related to the dielectric constant ε_r of the dielectric layer).

Equation *1.1* is valid in the limit $t_{1;2} \gg \lambda_{1;2}$ where t is the film thickness. Since ring diameters up to 20 mm can be used, given the sapphire dielectric constant $\varepsilon_r \sim 10$, measurements can be performed down to ~ 1 GHz, in the range of interest for accelerating cavity applications.

The quality factor Q_T of the resonant circuit is determined by fitting the output power versus frequency curve around the resonant frequency ω_n by a Lorentian curve of half width $\Delta\omega = \omega_n = Q_T$ (amplitude technique).

For symmetric coupling, the intrinsic quality factor of the ring resonator is Q_i and it is related to Q_T by:

$$Q_i = \frac{Q_T}{(1-R_v)} \qquad 1\text{-}2$$

with $R_v = 10^{-IL/20}$, where IL is the insertion loss measured in dB. Adjusting the gaps between the resonator and the antennas it is possible to achieve an "unloaded" situation:
IL > 20 dB, $R_v \ll 1$, in this situation is valid $Q_i \sim Q_T$.

The intrinsic quality factor of the resonator can be in turn expressed as:

$$\frac{1}{Q_i} = \frac{1}{Q_0} + \frac{1}{Q_D} + \frac{1}{Q_R} \qquad 1\text{-}3$$

where *Qo* is the quality factor related to the surface resistance of the resonator (what we are interested in), Q_D and Q_R describes the parasitic losses (respectively Q_D the dielectric losses and Q_R the radiation losses that must be minimized) and it is temperature independent.

If two identical superconductors are used for the ring and the ground plane

$$Q_0 = \frac{G}{(R_S)} \qquad 1\text{-}4$$

where R_S is the surface resistance of the superconductor and G depends on geometrical factors only and can be calculated.

If a different superconductor is used as a ground plane

$$Q_0 = \frac{G(1+\alpha)}{(R_S+\alpha R_{SGP})} \qquad 1\text{-}5$$

where R_{SGP} is the surface resistance of the ground plane, and α is a geometrical factor related to the field distribution in the resonator.

To determine the surface resistance of the superconductor under test by the measured quality factor of the resonant circuit Q_T, one can, in principle, use previous equations, if R_v and Q_D and Q_R are known, or directly equations *(1-4) or (1-5)* if the condition $Q_T = Q_i = Q_0$ is satisfied ($R_v \ll 1$, $Q_0 \ll Q_D$ and $Q_0 \ll Q_R$).

Unfortunately, while R_v is easy to determine and Q_D depend on the dielectric chosen, an exact evaluation of Q_R is practically impossible. It is therefore essential to achieve the condition $Q_c \ll Q_R$.

The dielectric losses of sapphire are indeed quite low ($Q_{dielectric} \sim 10^7$ at liquid helium temperatures) while radiative losses are difficult to evaluate for a ring resonator. It is possible to consider as an upper limit the radiative losses of an equivalent linear microstrip obtaining $Q_0 \geq 10^7$ if $R_S \gg 0{,}1\ \mu\Omega$. So the ultimate resolution of this method is related to this last condition and it is therefore estimated to be $\sim 10^{-7}\ \Omega$.

The main drawback is connected to the fact that parasitic losses are indistinguishable from residual superconducting losses. Their evaluation is $Q_0 \geq 10^7$ (0.1 $\mu\Omega$ resolution), but they cannot rule out the possibility that some unpredicted parasitic losses are more severing limiting their resolution.

1.2 The triaxial cavity

To determine the RF surface impedance of a superconductor (instead of the microstrip resonator technique) a method that consists in putting the samples under test into a large cavity is widely used. The most popular cavity shape adopted to test the samples is a cylindrical shaped cavity used in its TE_{011} mode due to the fact that there are no currents crossing indium vacuum sealing joints.

There are two way to place a sample in a TE_{011} cavity.

One is the end plate replacement method, which replaces one end plate with a disk sample. But, its resonant frequency is usually high due to the limited sample size.

The other is the Q-perturbation method, which places the sample on the maximum magnetic field location within the cavity. Disadvantage of this method is that the entire sample has to be placed at the maximum magnetic field location for the sake of achieving highest sensitivity. This might result in same unwanted losses on the sample edge. Another disadvantage is that the sample location usually breaks the cylindrical symmetry introducing measurement error on the geometrical factor.

At Jefferson Laboratories a 1.5 GHz superconducting triaxial niobium cavity working at 2 K has been designed [2]. It is a Q-perturbation method on a TEM cavity.

This device permits to measure RF surface losses of a 25.5 to 40 mm diameter superconducting sample using a Nb tapered cone resonant structure. It also permits to measure the RF losses of the sample using a calorimetric method.

Calorimetry methods are based on numerical simulation. The one used in this infrastructure is able to exclude losses caused by the niobium cavity, indium joints, coupling probes, and by field emission impacting at places other than the sample.

The niobium triaxial cavity consists of three separate pieces *(Figure 1-2)*.

Piece I: it is the bottom of the triaxial cavity, which consists of a centre rod, a tapered cone, a cylindrical wall, two RF coupling ports, and one vacuum pumping port (not shown).

Piece II: it is formed by the cavity top plate, which completes the cavity and separates its vacuum from the temperature sensor chamber vacuum. On the cavity side, a 25.4 mm or larger diameter (until to 40 mm) thin film sample can be put in the centre for surface resistance measurement. On the other side of the plate, 16 temperature sensors are located on four concentric rings to measure the temperature distribution caused by RF losses on the sample when electromagnetic energy is stored in the cavity. The plate provides a thermal resistance between the sample and the liquid helium bath. Piece II is made form rector grade niobium because it has

a lower thermal conductivity than high RRR niobium and thus enhances the sensitivity of the calorimetry.

Piece III: it is the other part of the temperature sensor chamber. Stainless steel is used because of the ease of machining and welding for vacuum ports and instrumentation feedthroughs. The central coaxial cone is excited in the TEM mode at 1.5 GHz. This TEM cavity is more compact due to its coaxial line structure and more sensitive than the commonly used TE_{011} because the magnetic field of this mode has its maximum on the bottom surface of the cone , where the probes couple and pick up the signals. The magnetic field is circumferential and gradually decays along the cone, dropping dramatically at the open end.

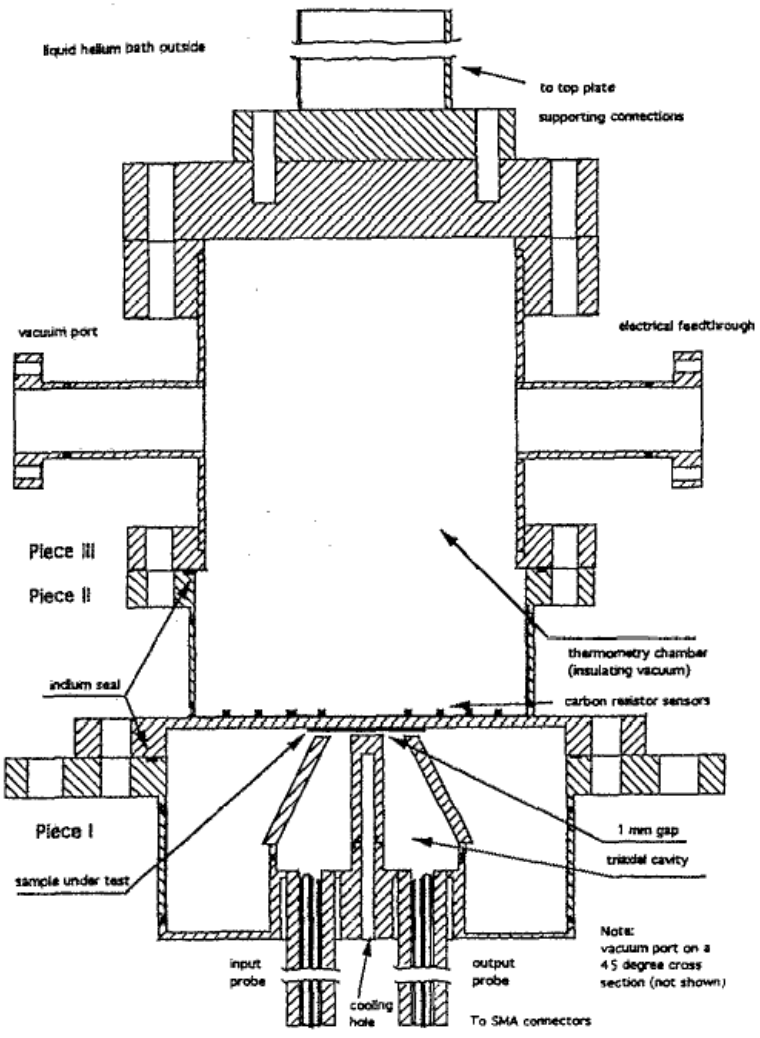


Figure 1-2 The assembly consists of three pieces. Pieces I and II are made from reactor grade niobium, and Piece III from stainless steel. Vacuum port of the cavity is not shown.

To have the measurement sensitivity as large as possible, a small gap of 1.5 mm has been chosen. The displacement current flowing from the centre conductor to the sample, and from the sample to the conic conductor, results in a real radial current on the sample. When the sample's

diameter is between 25.4 and 40 mm, the RF loss at the edge is greatly reduced, and is zero in the latter case. The outer volume of the cavity vacuum space is an anti-resonant volume, which has a low field distribution and has been designed to make the indium joint loss small. In addition, the triaxial cavity has a tuning mechanism to adjust the gap, which changes the resonant frequency and can be used to shift the zero point of the field.

RF measurements at 2 K is a manually controlled system in which phase, frequency and amplitude could be adjusted. Through incident, reflected and transmitted power could derive the power loss P_{loss} within the cavity and the coupling factor β_1 of the input probe. So it is possible to calculate Q_0 through the relation

$$Q_0 = \omega\tau(1+\beta_1) \qquad 1\text{-}6$$

by measuring the resonant frequency ω and the decay time constant τ . The maximum fields in the cavity can be derived from

$$U = C_1 E_{max}^2 = C_2 H_{max}^2 = Q_0 + P_{loss} \qquad 1\text{-}7$$

where C_1 and C_2 are constants determined by numerical calculations.

The surface RF resistance of the superconductor under test can be calculated through the relation

$$R_s = \frac{G}{Q_0} \qquad 1\text{-}8$$

where G is the geometrical facto of the triaxial cavity and it is known.

In conclusion the surface resistance at 1.5 GHz of thin superconducting film grown on a 25.4 to 40 mm disk substrate could measured using a niobium triaxial cavity. This design greatly reduces edge losses and substrate interface losses of thin-film samples. An extremely low detection threshold for surface resistance, 0,02 nΩ, can be achieved.

1.3 A 400 MHz coaxial resonator

As discuss in previously a common way to study RF properties of a superconductive sample is to test it, in a cylindrical cavity, through the Q-perturbation method. An example of this method is represented by the triaxial Nb cavity at CEBAF. However, since the sample disk is placed at a maximum of the electric field two problems come out. The first problem is represented by the enhanced field emission from the sample. A second problem is that the RF current passing the sample edge could be create uncontrollable spurious dissipative effects.

To avoid these two major problems of the triaxial cavity an infrastructure to measure the surface resistance of superconducting samples at 400 MHz has been built at CERN in 2003 [5]. It is based on the thermal substitution technique to measure the R_S of a sample.
The designed apparatus is schematically sketched in *Figure 1-3.*

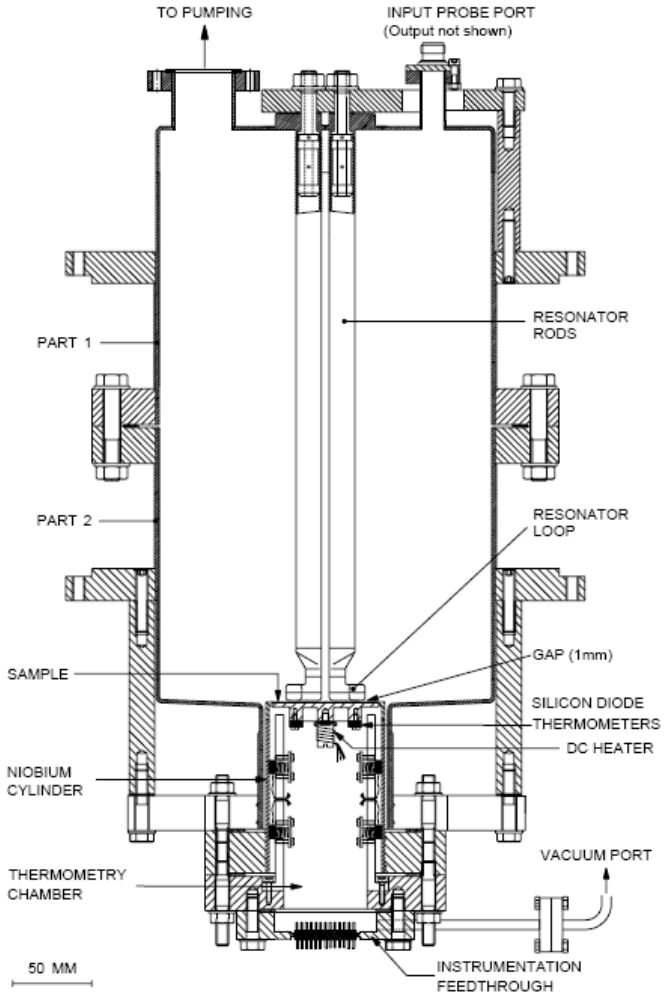


Figure 1-3 Layout of the niobium quadrupole resonator with mounted sample cylinder and thermometry chamber housing a dc heater and temperature sensors.

The niobium quadrupole cavity consists of three separate parts, as shown in *Figure 1-3*. A 2 mm thick niobium cylinder (Ø 210 mm, l = 361 mm) is composed of two separate niobium

cans (parts 1 & 2) which are electron beam welded and vacuum brazed to stainless steel flanges. Inside this screening container, a four-wire transmission line built from niobium rods (Ø 16 mm) is welded to the upper cover plate of the resonator. At the bottom end of the rods the niobium tubes are bent to form half rings of 25 mm radius. The resulting loop "illuminates" the front disk (Ø 75 mm) of the investigated sample, which is positioned 1 mm below the niobium half rings. The test cylinder is fixed to a 6 inch Conflat flange (part 3) and mounted into a tubular port of 2 mm bigger radius and equal length to ensure that the sample surface flushes with the lower cover plate of the niobium cylinder to which the port is welded.

The sample is a 4 mm thick disk made of the superconductor under test. It is electron beam welded to a niobium cylinder which has been welded to a Conflat flange. The front disk of the sample carries, on its inner side (under vacuum), three small stubs where temperature sensors (silicon diodes) and a heater (controlled by a current source and a voltmeter) are mounted. In order to measure the temperature of the sample cylinder, up to eight thermometers can be fixed too.

A three dimensional cavity code (MAFIA) has been used by the outhors to study the field distribution inside the quadrupole resonator. Since the dissipation is measured by calorimetry, no determination of the unloaded quality factor is needed. The coupling to the resonator is realized by two strongly over coupled loops, which are mounted on the upper cover plate. This allows the approximation $P_{total} = 2P_t$ (where P_t is the transmitted power). Taking the following expressions

$$Q_{sample} = \frac{\omega U}{P_{diss}} = \frac{G_{sample}}{R_{S,sample}} \qquad 1\text{-}9$$

$$Q_L = \frac{\omega U}{P_{total}} = \omega \tau_L \qquad 1\text{-}10$$

where Q_L is the loaded quality factor (superconducting cavities with couplers), and

$$P_{total} = \sum_{i=0}^{4} P_i = P_{diss} + P_1 + P_2 + P_{beam} + P_{mult,efe} \qquad 1\text{-}11$$

where P_1 e P_2 are losses in the couplers, P_{beam} are losses induced in the presence of a beam and the term $P_{mult,efe}$ describes the losses due to multipacting and enhanced field emission in the cavity, we see that

$$\frac{1}{Q_L} = \frac{1}{\omega U}\sum_{i=0}^{4} P_i = \sum_{i=0}^{4}\frac{1}{Q_i} \qquad 1\text{-}12$$

Using *1-8* with the equations *1-12* and *1-13*

$$Q_L = \frac{\omega U}{P_L} \qquad 1\text{-}13$$

one finds the final result for the sample surface resistance inside the quadrupole resonator:

$$R_{S,sample} = \frac{G_{sample}}{\omega\tau}\frac{P_{diss}}{P_{total}} \cong \frac{G_{sample}}{2\omega\tau}\frac{P_{diss}}{P_t} \qquad 1\text{-}14$$

Knowing that $P_{diss} = (1/2)R_S SH^2$, where S is the surface area of the sample, the average magnetic field on the sample surface can be written as

$$\langle H_{rf}^2 \rangle = 2\frac{\omega\tau}{G_{sample}}\frac{P_{total}}{S_{ill}} \cong 4\frac{2\omega\tau}{G_{sample}}\frac{P_t}{S_{ill}} \qquad 1\text{-}12$$

There was a small arbitrariness in the choice of the illuminated sample surface S_{ill}. Finally could be took a value of $S_{ill} \sim 12\ cm^2$.

The surface resistance *R*s(T) of the sample could be measured by using a rf-dc compensation method. Therefore, the temperature of the quadrupole resonator is stabilised at 1.85 K and the dc heater was switched on to increase the sample temperature by a well defined ΔT with $\Delta T\text{min} \cong 2.9$ mK. When the thermal equilibrium – defined as no temperature change ($\Delta T < 2.9$ mK) within one minute – is reached, the heater is switched off and the dissipated heater power P_{diss} is compensated by switching on and increasing the rf power until the condition *T*sample (rf) = *T*sample (dc) is fulfilled. By measuring P_{diss}, P_t (*eq. 1-12*), ω, and τ one gets with equation *1-14* the sample surface resistance $R_{S,sample}$ (T).

1.4 Sample limits

As already shown the RF characterization of superconducting samples would be an useful diagnostic way to accurately investigate local properties of a bulk superconductor, of a grown superconducting films and given surface treatments on them.

However, common limitations of systems used for RF characterization of superconducting samples, often consist in the difficulty of samples preparation and scaling up the measured results to the real resonator. Each device previously described has intrinsic limits and drawbacks: sample size and position, uncontrollable dissipative effects at sample edges, field emission, difficulties to reach the ideal thermal contact and difficulties to assemble the experimental devices. The RF performance testing of a sample and its extrapolation to the frequency of a cavity is and will always remain an indirect way of measuring superconducting RF properties.

Obviously the most direct way to test RF properties of a superconductor would be the use real size cavities, but 1.5 GHz resonant structures would be too onerous both for costs and time consuming.

- For chemical surface treatments it is necessary to use a huge quantity of acids: they are expensive and dangerous.
- Experimental infrastructures are big and pricy, in particular the cryogenic apparatus. It is complex and it has to be filled with more than 400 litres of liquid helium for a single RF test.
- Moreover the RF testing procedure takes a long time. It includes the cavity pumping , bake out, cooling at 4,2 K and cooling at 1,8 K. Generally to perform only one RF test one week is not enough!

Therefore the idea is to build micro-cavities completely equal in shape to the real scale model. In *Figure 1-4* a visual of accordance to reality of RF properties of superconducting samples and superconducting 6 GHz cavities is reported. Any sample extrapolation will be far from the accuracy obtainable with a real superconducting resonator like a 6 GHz cavity.

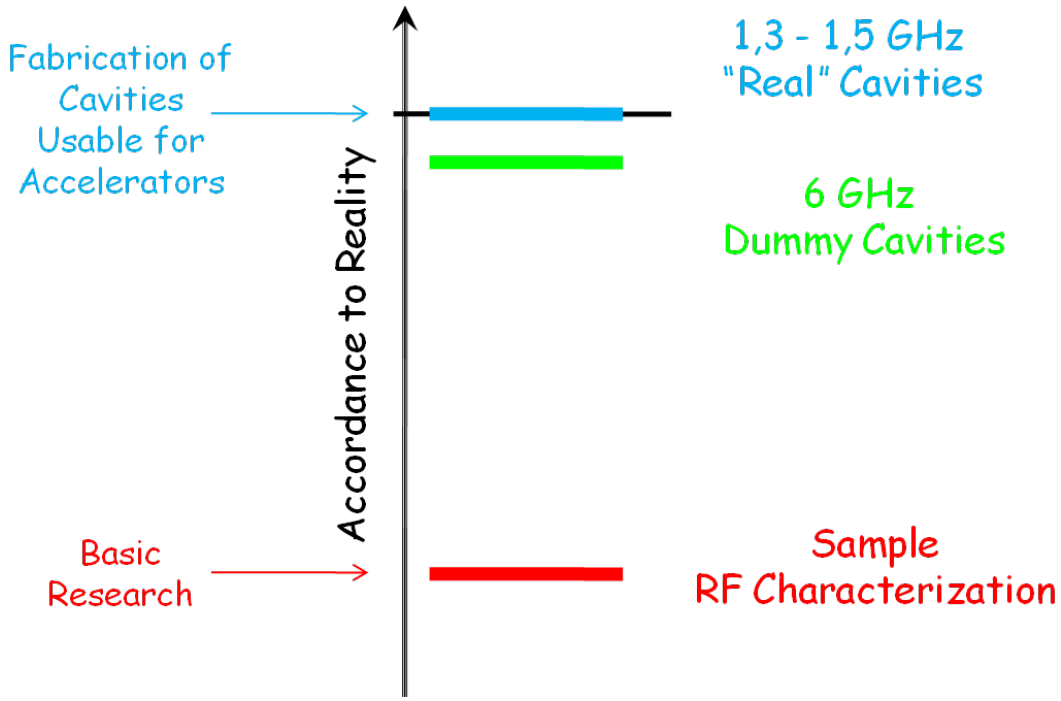


Figure 1-4 A visual of accordance to reality of RF properties of superconducting samples and superconducting 6 GHz cavities. Any sample extrapolation will be far from the accuracy obtainable with a real superconducting resonator like a 6 GHz cavity.

Chapter 2

6 GHz CAVITIES: A DENSE RF TEST STATISTIC AND COST REDUCTION

The idea, to perform a high numbers of RF tests on superconducting materials and at same time to reduce research budget, is to build 6 GHz cavities. RF measured samples will never be comparable to a real large cavity. It is always an indirect measurement. Vice versa 6 GHz cavities are at the same time easy to handle as a sample but they are "real" cavity.

They are made from larger cavities fabrication remaining material using spinning technology, they don't need welding (even for flanges) and finally they can be directly measured inside a liquid helium dewar. While 1,3 – 1,5 GHz cavities need for the RF test no less than 1 week time preparation. With 6 GHz cavities it is possible to perform more than one RF test per day.

With a tool like this it is possible to study traditional and innovative surface treatments and to perform RF tests on a large amount of cavities with a research budget much lower than the one necessary to treat and tests real cavities. One can study traditional surface treatments on Nb like: mechanical polishing, buffer chemical polishing, electro polishing, high pressure rinsing (HPR), alcohol rinsing and thermal treatments. It is also possible to study new thin film superconducting materials grown for example by sputtering or thermal diffusion.

In this chapter is shown how is possible, with a reduce budget, to set up a Mini-Laboratory able to perform all the traditional treatments necessary on niobium high gradient large cavities and not only:

- A reduced size mechanical polishing bench
- A chemical/electrochemical minilab for BCP EP
- A mini oven for thermal treatments
- A miniaturized sputtering system
- An inexpensive cryogenics and quick RF measurements

2.1 Geometry and fabrication technique

6 GHz cavities are made trough the spinning technology *(Figure 2-1)* that represents a valid alternative to electron beam cavities production, also for real resonators.

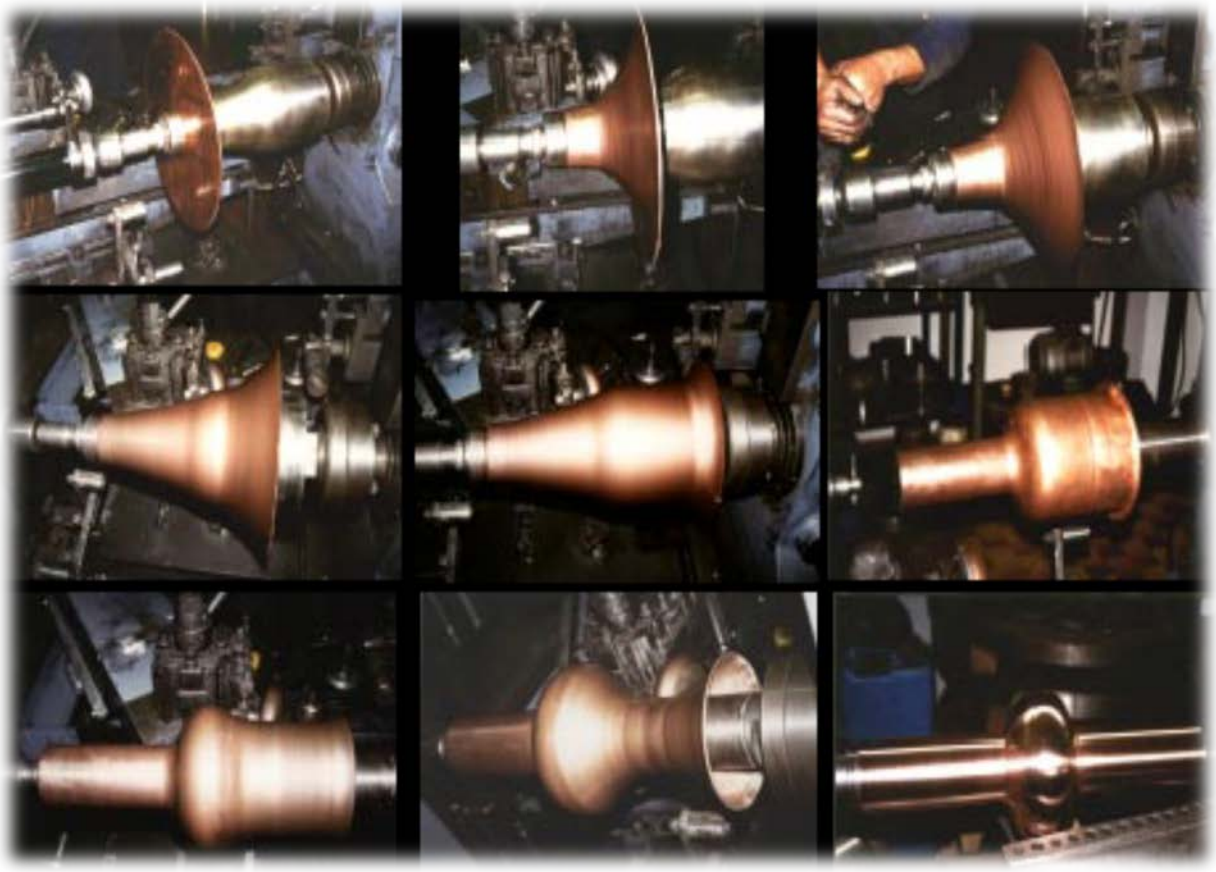


Figure 2-1 Snapshots of a large Cu cavity during the spin moulding.

High beta superconducting accelerating resonators of both bulk niobium and niobium sputtered onto copper types are commonly manufactured by spinning two half-cells, which are then electron-beam welded together from the inside. Welding is a complicated and costly operation that places severe limitations on the fabrication of high frequency cavities due to the narrow size of the bore.

At the National Institute of Nuclear Physics in Legnaro (LNL-INFN) [4] have adapted the well-known spinning technique to form a fully seamless resonator without electron beam welding. In this way, starting from a disk or a seamless tube, it is possible to build seamless cavities with no intermediate annealings, more rapidly, simply, and with more uniform thickness than through hydroforming. Both 1,5 GHz niobium and copper cavities can be easily manufactured with high reproducibility and significant savings in manufacture costs.

The 6 GHz cavities produced by spinning are obtained using larger cavities fabrication remaining material “scraps” as shown in *Figure 2-2*, they don't need any kind of electron beam welding (even for flanges) as shown in *Figure 2-3*, finally their production require a short fabrication time, a half day per cavity. For these reasons it possible to control the costs production and to realize a large amount of cavities with a low research budget as shown in *Figure 2-4*.

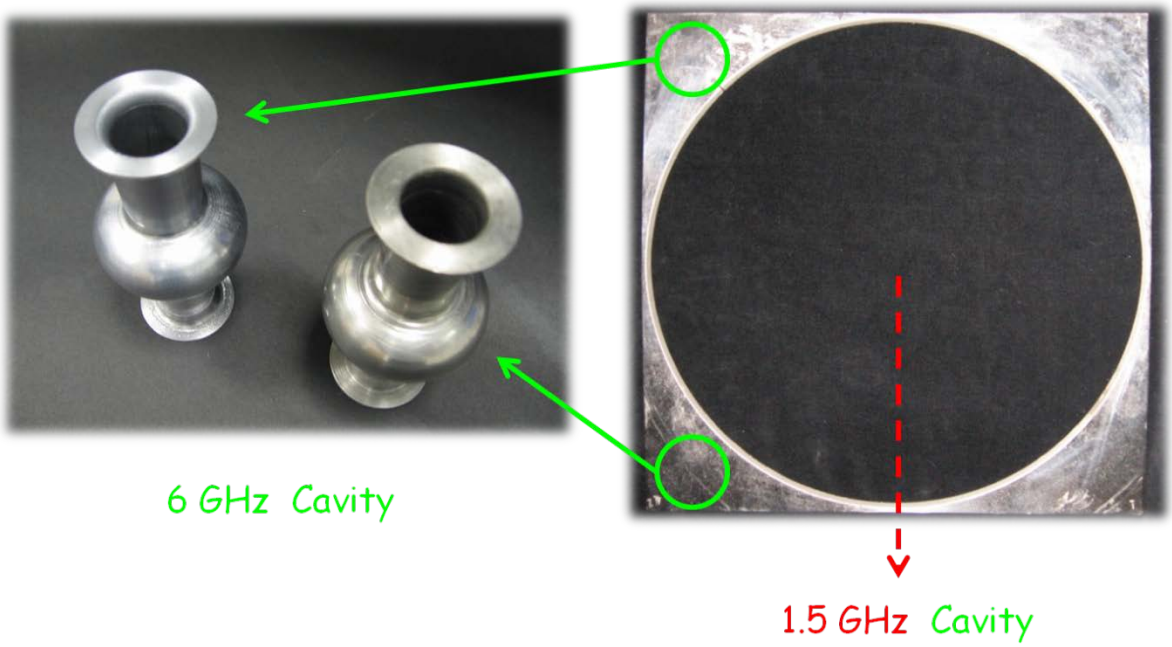


Figure 2-2 On the right a scrap, of a large Nb cavity, from which are obtained 4 small 6 GHz resonators from the 4 corners.

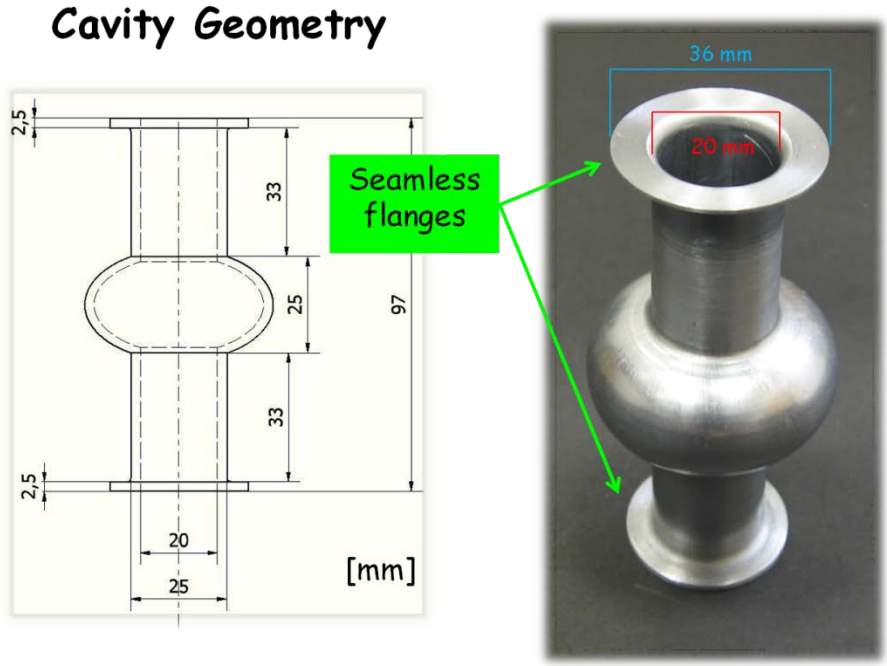


Figure 2-3 The 6 GHz cavity geometry.

6 GHz cavities are 97 mm long and have a 45 mm diameter cell, an *electrical length* of 25 mm and the same *geometrical factor* of a large resonator, 287. They have two large flat flanges at the ends. For each of them the available surface to ensure the vacuum sealing is equal to 7 cm^2.

Due to the spinning process, material mechanical stress is considerable and the imprints caused by the internal collapsible mandrel must be removed.

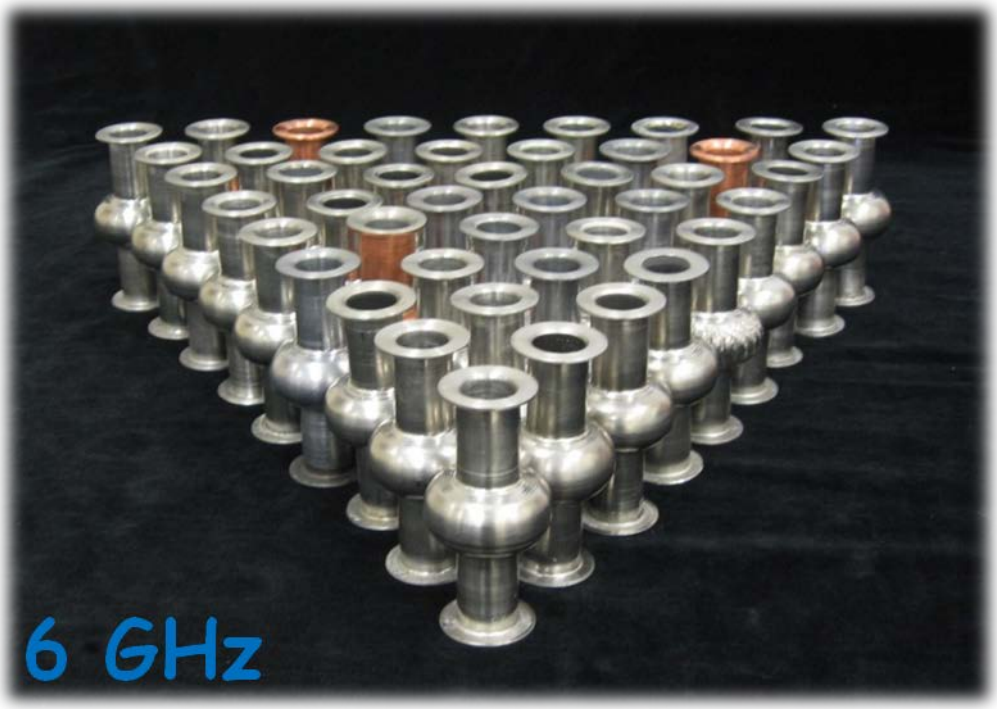


Figure 2-4"A great army of small soldiers"

2.2 Low cost and reduced size mechanical polishing bench

The spinning process implies material surface defects, stress and dislocations. The cavity cell is characterized by the presence of evident vertical scratches due to the used mandrel. Obviously the internal surface finishing of a resonant structure is directly correlated to its performance, especially at high fields. Moreover the lubricant, necessary for the metal mechanical processing can contaminate the used material. The idea is to make the surface smooth and free from contaminants.

In order to remove surface roughness and contaminations introduced during the spinning process, we use a compact and portable tumbler in spite of the bigger and complicated designed ad hoc for large cavities.

Figure 2-5 Compact tumbling system for small resonators.

The 6 GHz cavity is filled with a certain number of abrasive agent pieces (media) *Figure 2-6*, plugged up and fixed to the machine. The tumbler makes the cavity rotate so that the small media pieces can erode the metal surface in a uniform, controllable and reliable way.

Different materials could be used for this kind of mechanical polishing: for example small SiC triangular shaped blocks, 5 mm sphere of yttria stabilized zirconium dioxide and flakes of Al_2O_3 and SiO_2 powders embedded in a polyester matrix.

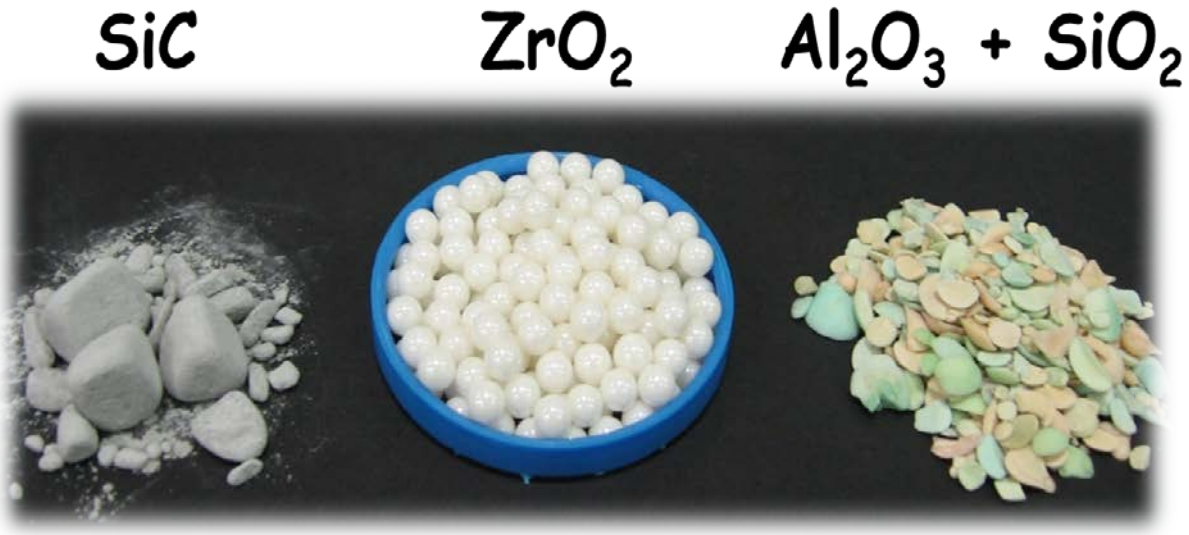


Figure 2-6 Three types of different abrasive media.

Silicon carbide is a very hard material and it can be used for the first low level mechanical polishing. ZrO_2 is a high density material and can be used for the intermediate smoothing. Al_2O_3 plus SiO_2 (in PET) flakes are soft and can be used for the final surface finishing.

During a mechanical treatment, for each cavity, it is easy to stop the process and monitor the smaller resonator weight change with a balance and internal surface finishing with the help of a miniature camera (visible on *Figure 2-7*).

The idea is to check an eventual defect evolution after each polishing treatment without any risk to touch the small cavity internal surface. It is composed by two different parts: a computer interfaced firm base and a mobile tool equipped with the optical and lighting devices. They are connected by an optical fibres cable. The acquired images magnification is around 60x. Positioning the mobile tool near the point of interest, focusing and eventually correcting the lightening are the only necessary operations to obtain the desired image. To make the tool positioning on defects reliable a support system has to be used. The latter is equipped with a fixed protractor, so the resonator rotation angle (relative to its axis) can be easily measured. The miniature camera tool can be moved forward, backward, up and down inside the cavity. The tool displacement along the cavity axis can be easily measured with the ruler fixed to the system basis.

Using this procedure it's easy to choose the right media and to establish the right treatment duration to perform the best internal surface finishing in a few steps.

Figure 2-7 The miniature camera tool used for the cavity inspection. On the top part on the right a picture of the cavity cell before the mechanical polishing. On the bottom part on the left a picture of the same place of the cell after the mechanical polishing. The surface after the treatments appears smoother than before. The vertical scratches due to the used mandrel have disappeared.

2.3 Low cost chemical/electrochemical minilab for BCP EP

Following the classical and well known surface treatments general protocol of large cavities, after the mechanical polishing the procedure counts a chemical polishing. Chemical treatments are performed to smooth further on the cavity surface, to remove the possible niobium sub-oxide and contaminants.

Chemical treatments for large resonators require tens of litres of sulphuric acid, hydrofluoric acid, phosphoric acid and nitric acid handled with big and complex infrastructures. On the other hand, just a few litres of acids (2-3l) are needed for 6 GHz cavities using a simple and easy to handle infrastructure. This means a drastic reduction of acids and materials costs and also a considerable reduction of human risks and time processing.

To perform the traditional surface chemical treatments, buffer chemical polishing (BCP) and electrochemical polishing (EP), on a 6 GHz cavity can be used a small system as the one reported in *Figure 2-8*.

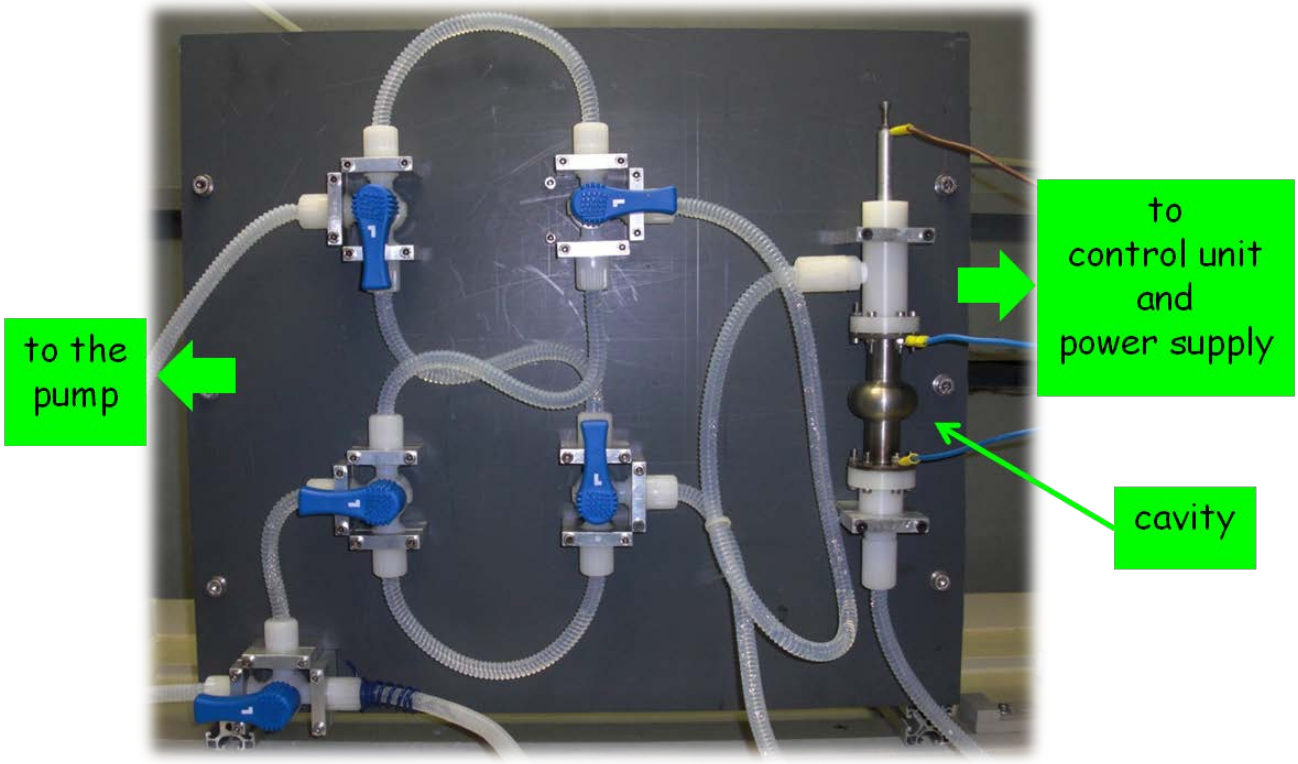


Figure 2-8 In this picture is visible the mini-chemical system set up for BCP and EP. In particular can be seen on the right a 6 GHz cavity installed in vertical position, equipped with special flanges for EP. The acid flux is directed from the bottom to the top of the cavity in order evacuate the hydrogen, produced during the process, quickly. The 3-way valves are useful to invert the flux direction.

BCP AND EP FLANGES

For buffer chemical polishing are used simple holed PVDF (poly-vinil-dene-fluoride) flanges as reported in *Figure 2-9.*

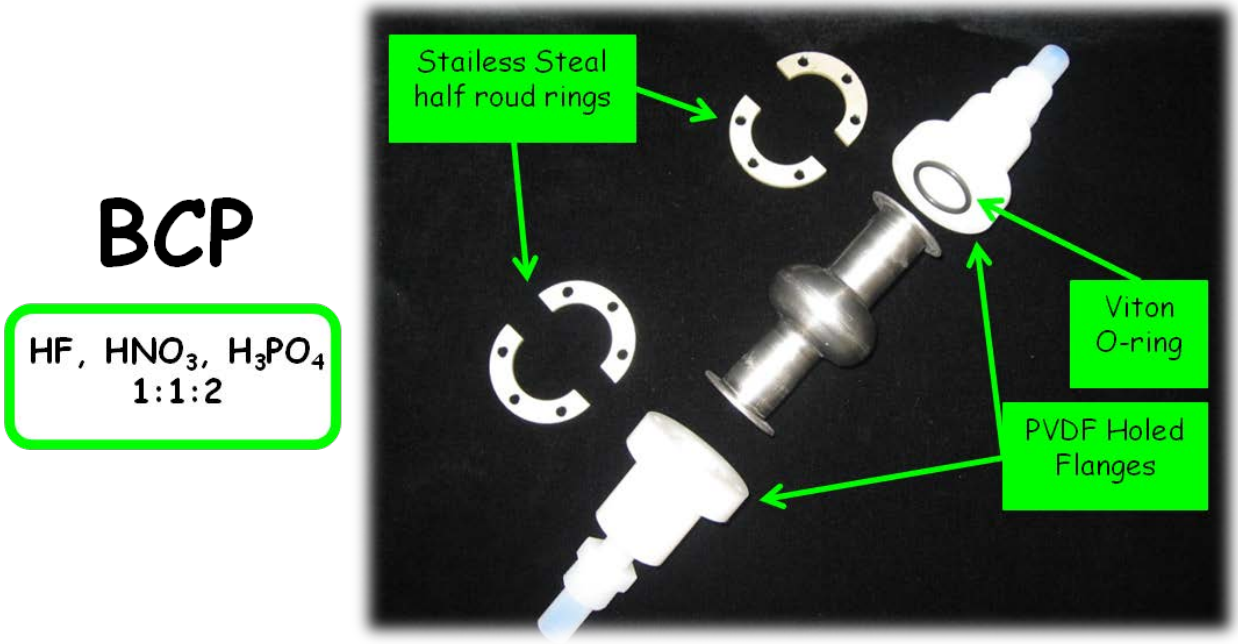


Figure 2-9 Details of the cavity closing system for buffer chemical polishing.

In the case of electrochemical polishing are used particular PVDF flanges able to hold a aluminum cathode conveniently designed as reported in *Figure 2-10.*

Figure 2-10 Details of the cavity closing system for electro polishing. The aluminum cathode, that enters inside the cavity, is shaped to maintain constant the cathode – internal surface cavity distance.

The flanges are expressly designed to obtain the highest acid flow through the cavity to allow hydrogen bubbles, produced during the oxi-reduction reaction, to escape freely.

The evaluations of damaged layer thickness produced by the spinning process could be vary from 150 to 250 μm. For this reason it is convenient to remove with BCP/EP at least 300 μm: in average this thickness corresponds to about 30 g of removed material.

Again, using the miniature camera previously described, the internal surface finishing can be easily observed after each step of chemical treatment.

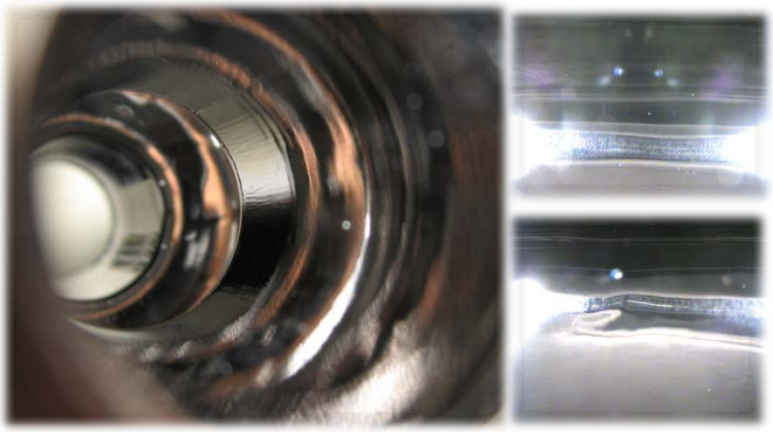


Figure 2-11 Some pictures of the inner surface of a cavity after EP step.

2.4 A mini oven for Thermal Treatment

As delivered, commercial niobium typically has less than 1 wt ppm of dissolved hydrogen because the material in its final form is usually annealed for recrystallization. But hydrogen concentration can increase during chemical etching, especially if the temperature of the acids etch is allowed to rise above 20±C and/or if hydrogen bubbles are not allowed to escape freely and quickly.

According to the phase diagram of the Nb-H system [5], the required concentration of hydrogen to form the Nb hydride phases is very high at room temperature ($4.6*10^3$ wt ppm for the μ phase and $7.5*10^3$ wt ppm for the ε phase). Therefore these phases do not form. As the temperature is lowered, the hydrogen concentration needed to form the hydride phases decreases. Above 150 K the danger of hydride formation is still not very serious, because the concentration required is still relatively high. The cavity can be cooled as slowly as desired to 150 K. As the temperature is lowered below 150 K, the hydrogen concentration required to form the hydride phases decreases to a dangerous level, so that islands of the hydride phase may form even when the concentration is as low as 2 wt ppm. The hydride precipitates at favourable nucleation sites. If these are at the surface they increase the residual loss. The diffusion rate of hydrogen between

150 and 60 K remains quite significant, so that hydrogen can move to accumulate to critical concentration at nucleation sites. Only when the temperature is reduced to below 60 K does the diffusion of hydrogen slow down enough that hydrogen can no longer accumulate at hydride centres. Therefore when a cavity with a large bulk hydrogen concentration is cooled to liquid helium temperature, the length of time it is kept between 150 and 60 K will determine the extent of hydride formation and the accompanying residual losses. The "Q disease" (common way to call this residual loss) can be mitigated by rapid cooling of the cavity through the dangerous temperature regime but the best remedy is to degas the hydrogen entirely by heating the niobium cavity in vacuum (< 10^{-7} mBar) at 700 to 900±C. Although the danger of oxygen pickup at this temperature is minimal, it is advisable to protect the RRR of the niobium by surrounding the outside of the cavity with titanium [6].

For the reason mentioned above, 6 GHz Nb cavities must be annealed in a dedicated vacuum chamber: a mini oven that consists of a titanium box, an Inconel® vacuum chamber (furnace) and an external heater.

Figure 2-12 All the parts that compose the stand that enter into the furnace.

The cavity is mounted into a Ti tube supported into the Inconel® cylindrical vacuum chamber *(Figure 2-12)*.

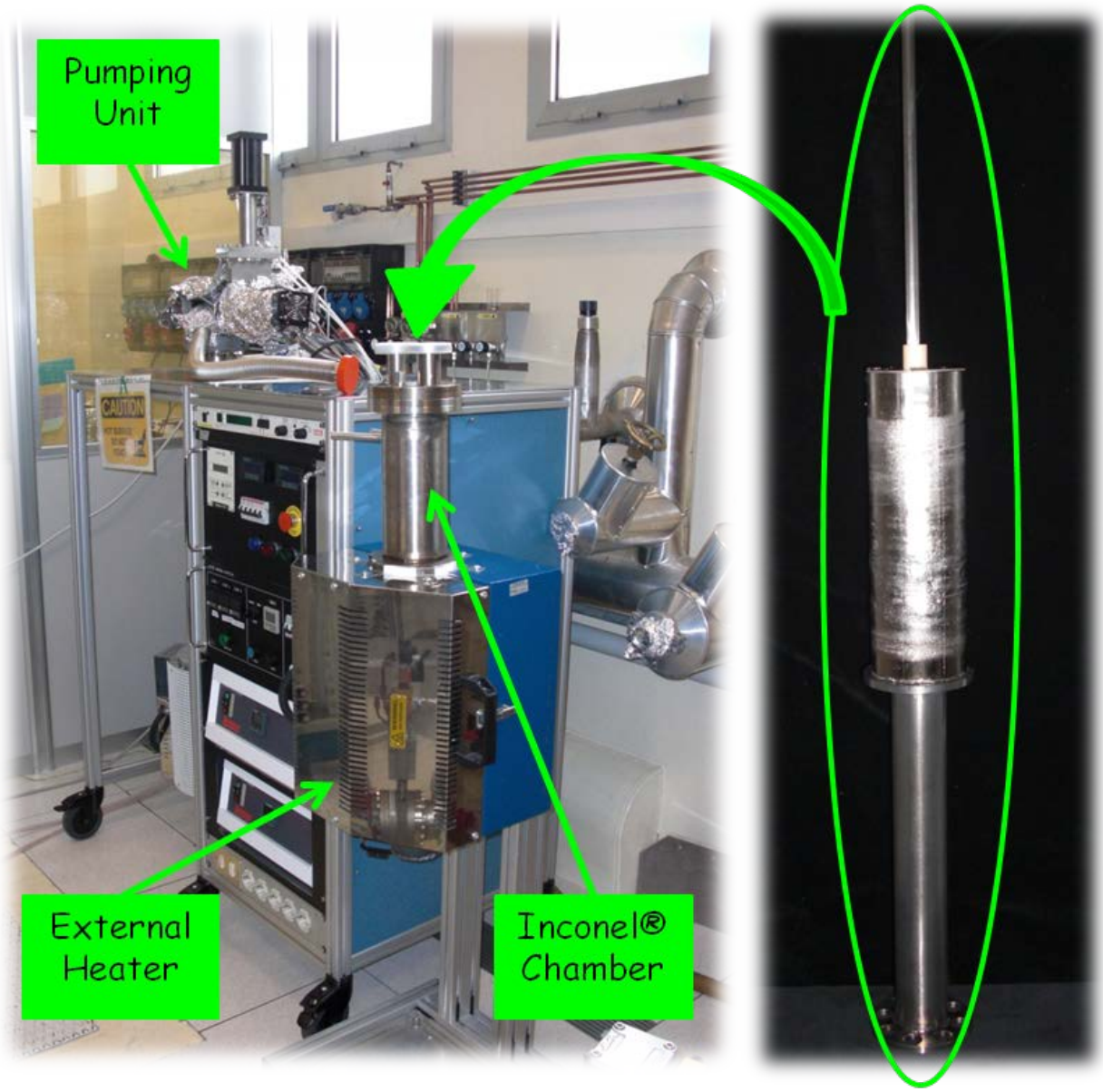


Figure 2-13 On the right the cavity is mounted inside the Ti box fixed on the Inconel® holder. The entire stand is placed into the chamber (left picture) in order to perform the thermal treatment.

The apparatus temperature is measured with two thermocouples: one of them is fixed to the Ti tube internal part, while the second T sensor monitors the temperature externally (Ti tube outer wall). Titanium behaves as a getter: at high temperature it bonds to reactive gases such as O_2. After reaching a ~10^{-8} mBar base pressure, the external heater is switched on. The optimal thermal treatment temperature, ~800±C, could be hold for 3 hours and Hydrogen.

Titanium and oxygen contaminations, into the cavity walls, are unavoidable with this technique. The external layer has to be removed (with a quick 1:9 EP, at least 5 minutes to remove just few μm).

2.5 A miniaturized sputtering system

With 6 GHz resonators one can study any kind of superconducting material that could be deposited inside it. From this point of view it is possible to build systems like the two depicted in *Figure 2-14* and *Figure 2-17*.

This miniaturized sputtering system allow to coat the inner surface of a 6 GHz cavity with multilayers of Nb and Sn. The resonator is fixed to a linear feedthrough and during the deposition is moved up and down toward the cathode to assure the complete and homogeneous coating.

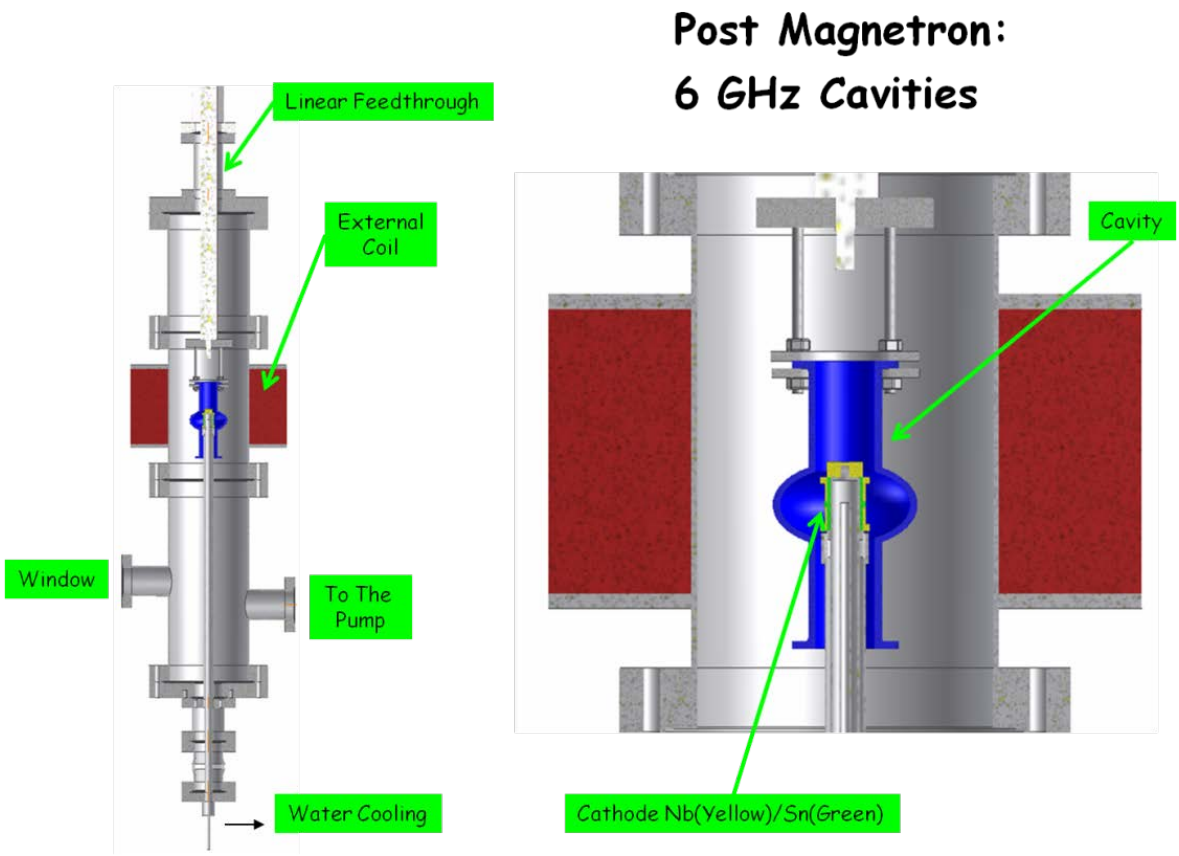


Figure 2-14 A schematic of the system built to coat 6 GHz cavities using the post magnetron sputtering technique. On the left the complete vacuum chamber is shown, on the right the detail of the deposition zone. The coil is out of the chamber.

As shown in *Figure 2-14* the cathode consist in a fix long stainless steel cooled tube ended with a composite Nb/Sn target. It is, except for the target, completely screened with a grounded second external tube. The cathode diameter is 17 mm while the diameter of the cavity is 20 mm so it can be moved coaxial to the cathode without touching it. The target, that is equipped with two Nb wings, is positioned in the centre of a strong axial magnetic field produced by the coil. The wings, *Figure 2-15-A*, along with the magnetic field enhance the sputtering rate.

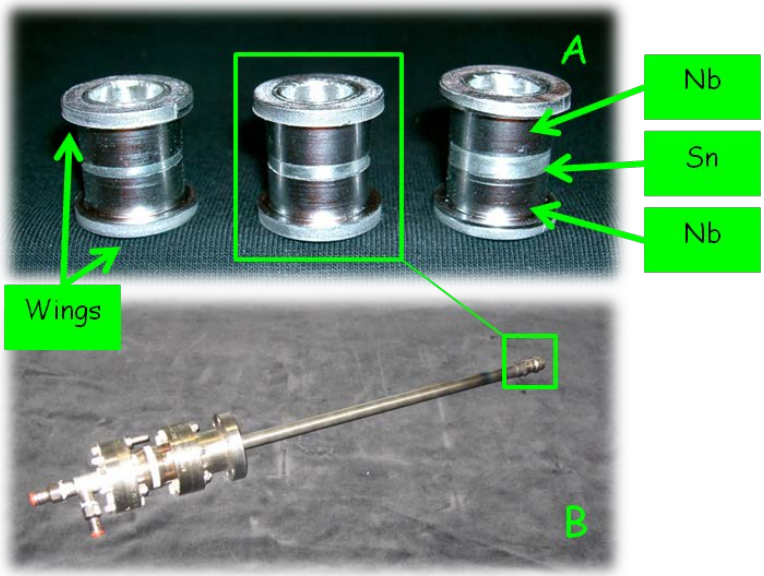


Figure 2-15 In A the details of three composite target with different tin amount. In B the entire cathode.

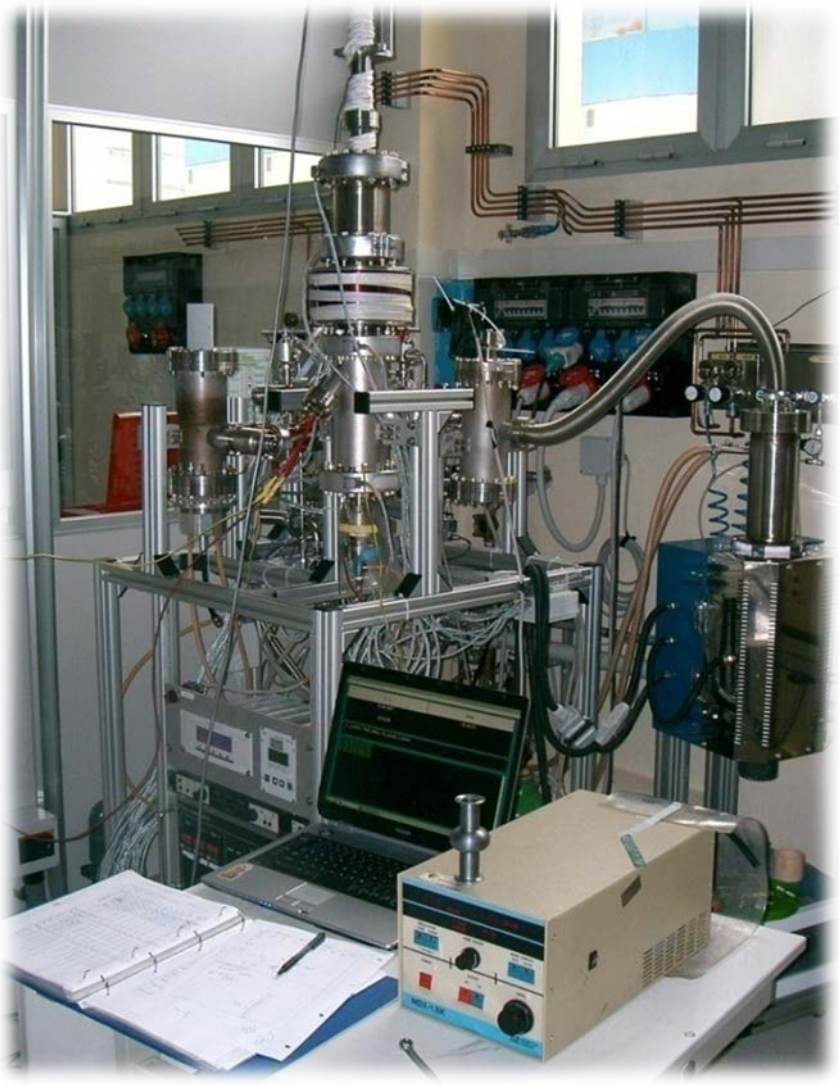


Figure 2-16 The sputtering system.

Otherwise, the system shown in *Figure 2-17* allows to produce Nb_3Sn through the method of liquid tin diffusion on the surface of 6 GHz niobium cavity[6].

The system consist in: a ultra high vacuum cylindrical reaction chamber made of Inconel, an Alumina crucible for the tin bath (99.99% nominal purity), an UHV linear manipulator that permits to move the cavity from the top to the bottom (and vice versa). The lower part of the system can be heated by an irradiating external furnace (hot zone) while, to avoid Sn vapour condensation in the manipulator and in the pumping systems, the upper section ("cold zone") can be cooled trough a water jacket.

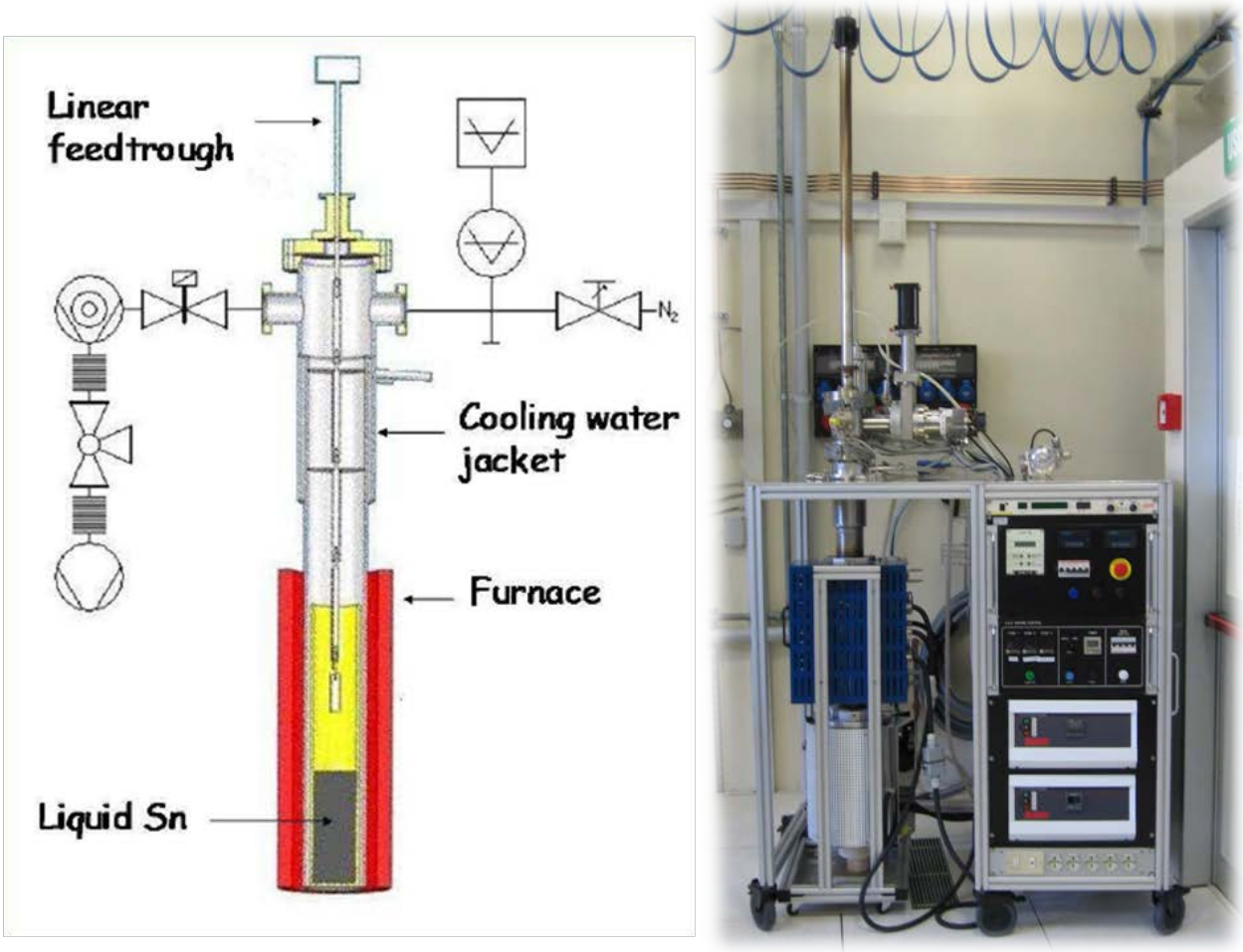


Figure 2-17On the left schematic representation of the system. A high vacuum cylindrical reaction chamber is used, it contains an Alumina crucible for the tin bath and a linear manipulator to move the cavity (not drown) from the top to the bottom and vice versa. The lower part of the system can be heated by an irradiating furnace, while the upper zone can be cooled trough a water jacket or heated by a second irradiating furnace

This technique consists in dip the Nb cavity into the tin bath at a temperature of ~930°C for some minutes. After that the cavity is moved outside the bath and annealed in the upper part of the chamber at a temperature above 1000°C for some hours in order to achieve the right Nb/Sn phase.

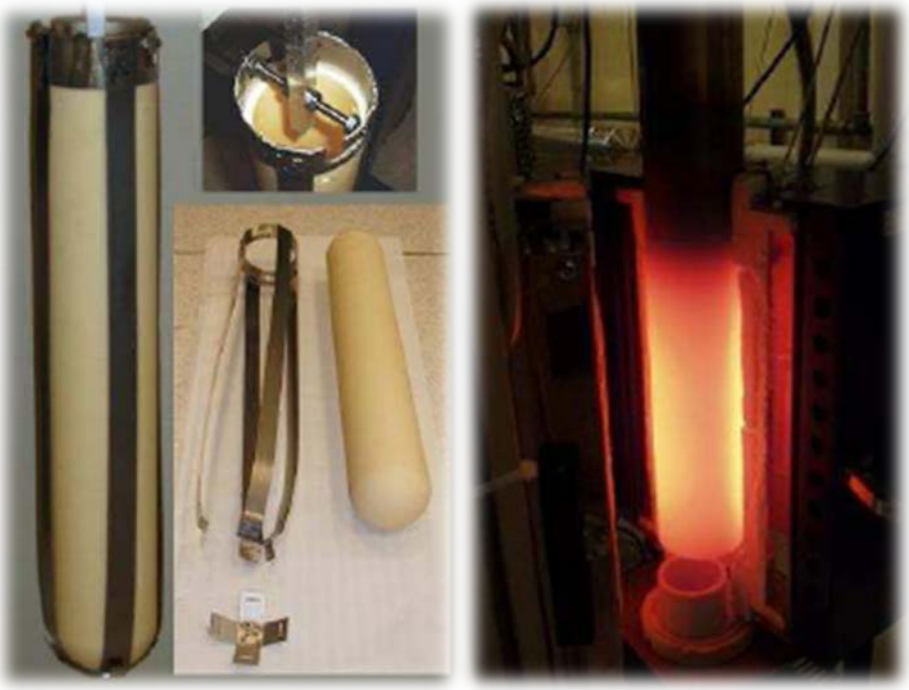


Figure 2-18 On the left the Al2O3 vessel containing Sn can be extracted with a long metallic rod which can be hooked up to the inconel jacket that enclosesthe crucible. On th right the lower part of the system can be heated by an irradiating furnace

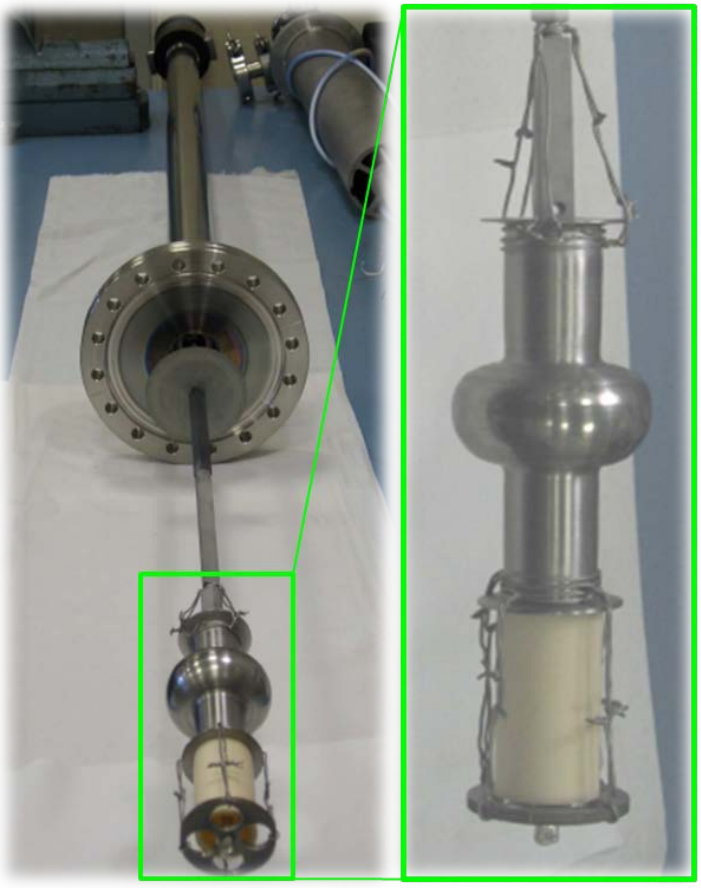


Figure 2-19 A 6 GHz cavity connected to the linear feedthrough

2.6 Inexpensive Cryogenics and Quick RF Measurements

To perform the 6 GHz cavities RF tests has been designed and built a compact and user friendly cryogenic infrastructure. It has been conceived to enter a 450 or 250 l liquid helium dewar. Its global appearance is depicted in *Figure 2-20*.

Such an infrastructure permits to reduce the cryogenic power consumption, avoiding any loss during a helium transfer into a cryostat for common large cavities: a measurement performed at 4.2 K requires roughly 20 litres of liquid He. Furthermore, pumping directly over the bath, it is possible to achieve 2 K consuming about 70 litres more to complete the measurement at low temperature. The procedure to cool down and to warm up the system is very fast.

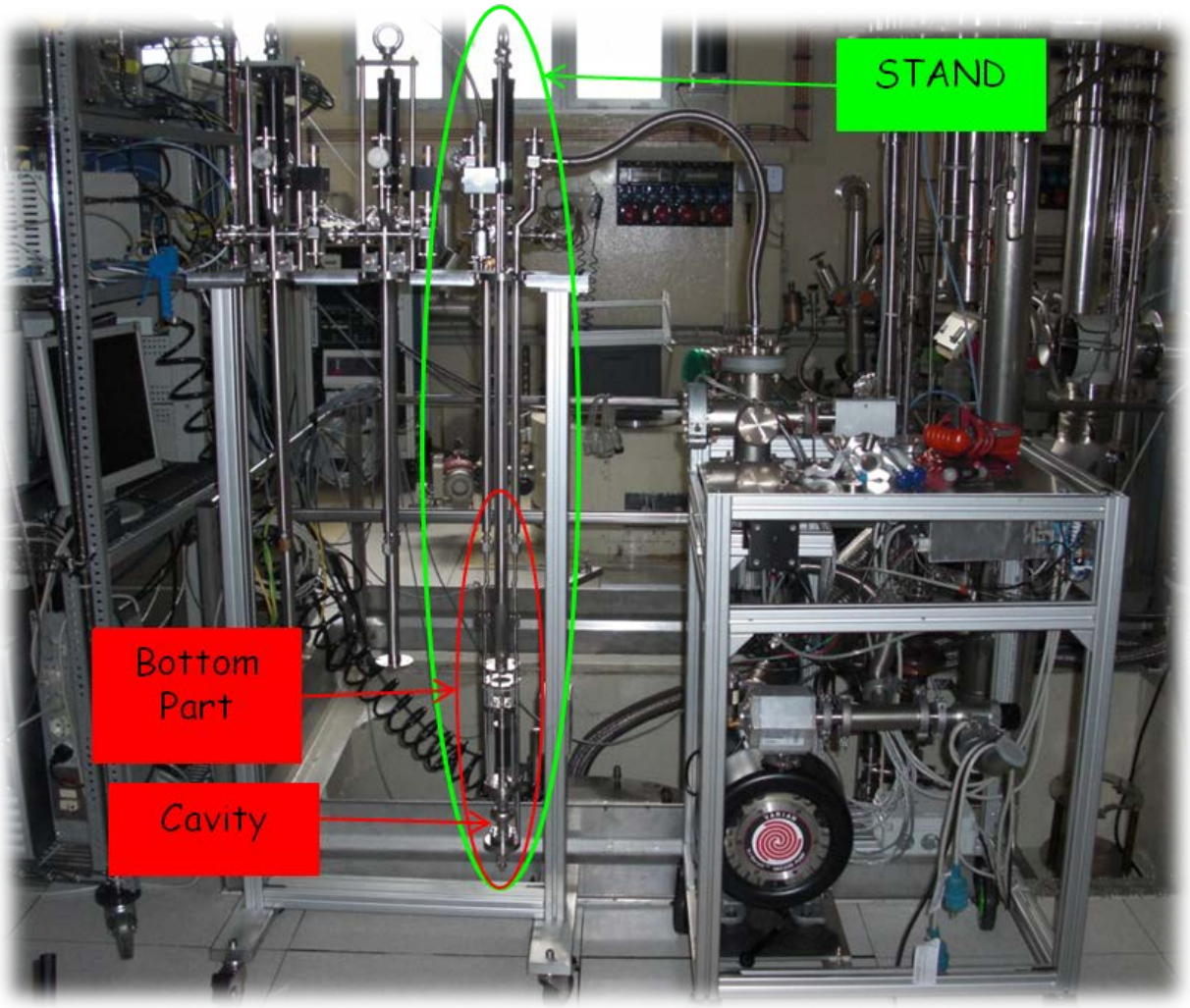


Figure 2-20 Green circled one of three stands, connected to the pumping control unit (on the right).

To oversimplify the system assembling, the bottom part of the stand has been made completely independent *Figure 2-20*. The small resonator can be mounted, in clean room, without the presence of the complete stand that would make the work complicated.

The bottom part consists of two main elements that close the cavity: the Coupler Flange and the Pick-up Flange. Referring to *Figure 2-21*, Coupler Flange is a stainless steel disc with 8 holes for M4 s.s. screws and a CF 16 knife edge on one side. It is equipped with a 8 mm pumping line (Coupler-Pipe) and a bellow that permits the motion of the coupler antenna. The last is connected to a CF 16 - SMA coaxial feedthrough. Four teflon coated vertical s.s. bar lines preserve the system alignment and prevent blockage problems due to the freeze-over of the bellow upper flange during the liquid helium insertion step.

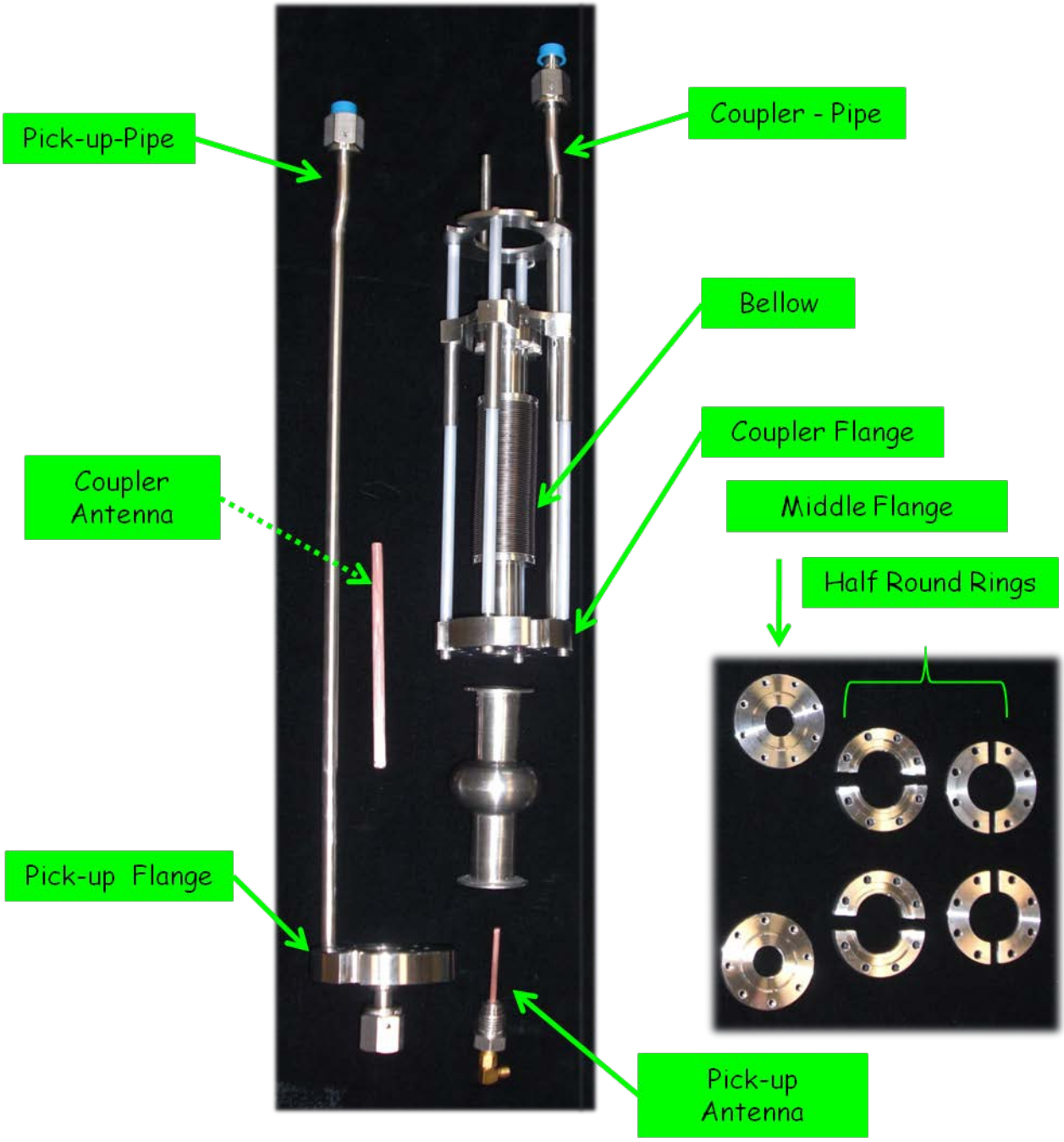


Figure 2-21 The bottom part completely dismounted.

Pick-up Flange is again a stainless steel disc with 8 holes for M4 s.s. screws and a CF 16 knife edge. It is equipped with a 8 mm diameter pumping line (Pick-up-Pipe) and a Swagelock assembly for the pick-up antenna insert (SMA ended).

The cavity is closed at both sides through the Cp/Pk Flanges with a particular double system depicted in *Figure 2-22*.

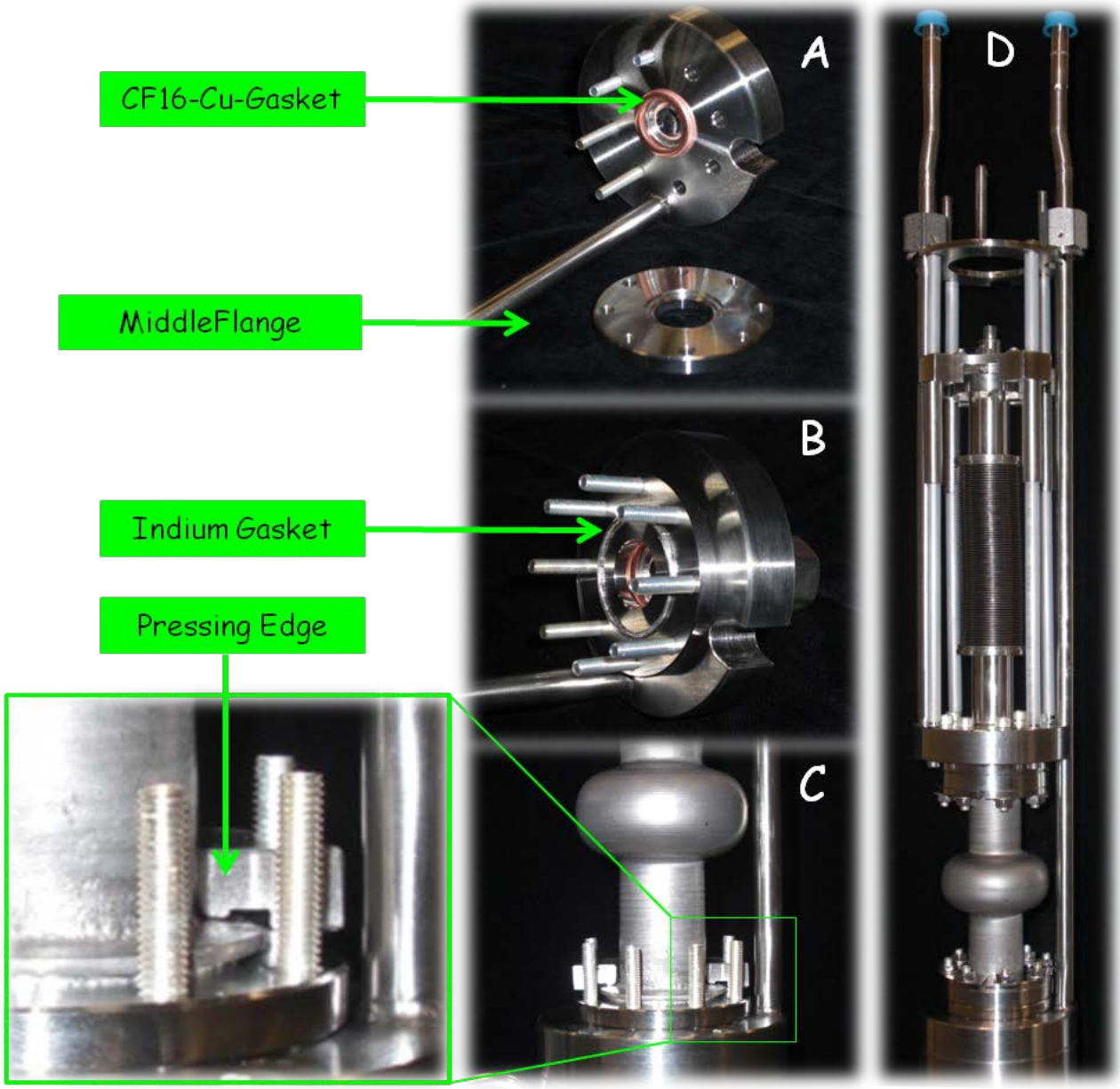


Figure 2-22 Assembling details of the Pick-up Flange to the cavity. The same setting up is adopted for the Coupler Flange. In figure C is represented the entire bottom part assembled.

The vacuum between the Pick-up Flange and the Middle Flange (equipped with 8 threaded holes) is guaranteed using a CF-16 copper gasket using 8 s.s. screws (figure A). Vacuum between the Middle Flange and the cavity flat borders is guaranteed using a 1.5 mm diameter indium wire squeezed with four stainless steel half round rings pressed through 8 nuts

screwed to the same screws (for each flange, when the first two half round rings are positioned, the others must be rotated of 90 degrees around the cavity axis before to be placed) (figure B and C).

The two pipes, of the bottom part, are connected to the double pumping line of the stand through two small bellows in order to contain thermal contractions during cooling down of the system.

The stand has been also conceived to execute special surface treatments like in flux helium conditioning and atmospheric plasma treatments. For this reason the top part of the stand is equipped with four all metal CF-16 valves and two different pumping lines connected through a bypass to guarantee the best pumping as shown in *Figure 2-23*.

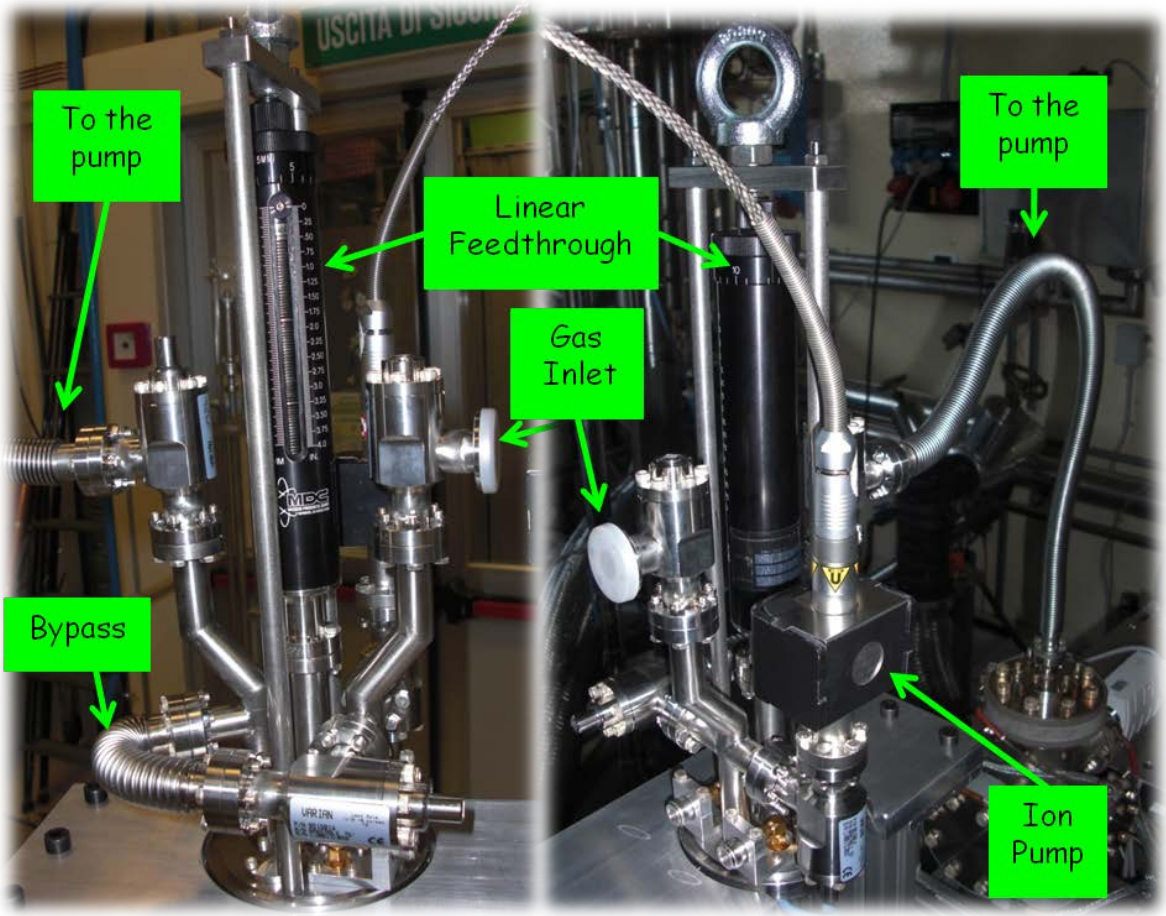


Figure 2-23 Two different views of the top part of the stand. In the left picture the bypass is visible. It is open during the RF measurement and close during the surface treatments to let the process gas to flow in the cavity. In the right picture is visible the 2 l/s ion pump.

One of them connects the stand to the main pumping unit, the second permits the process gas injection. The third valve opens and closes the bypass and the fourth one is used to isolate

the 2 l/s ion pump. It has to be turned on (after being baked out) for the RF test when the main pumping unit is disconnected.

In *Figure 2-23* are also visible: the 4 Inches linear feedthrough (fixed to a CF-16) able to move the coupler antenna through a bar and the structure to hang up the stand to the overhead travelling crane. Moreover on the top flange of the cryogenic infrastructure there are 4 holes to let RF cables and thermometer wires pass. (*Figure 2-24*)

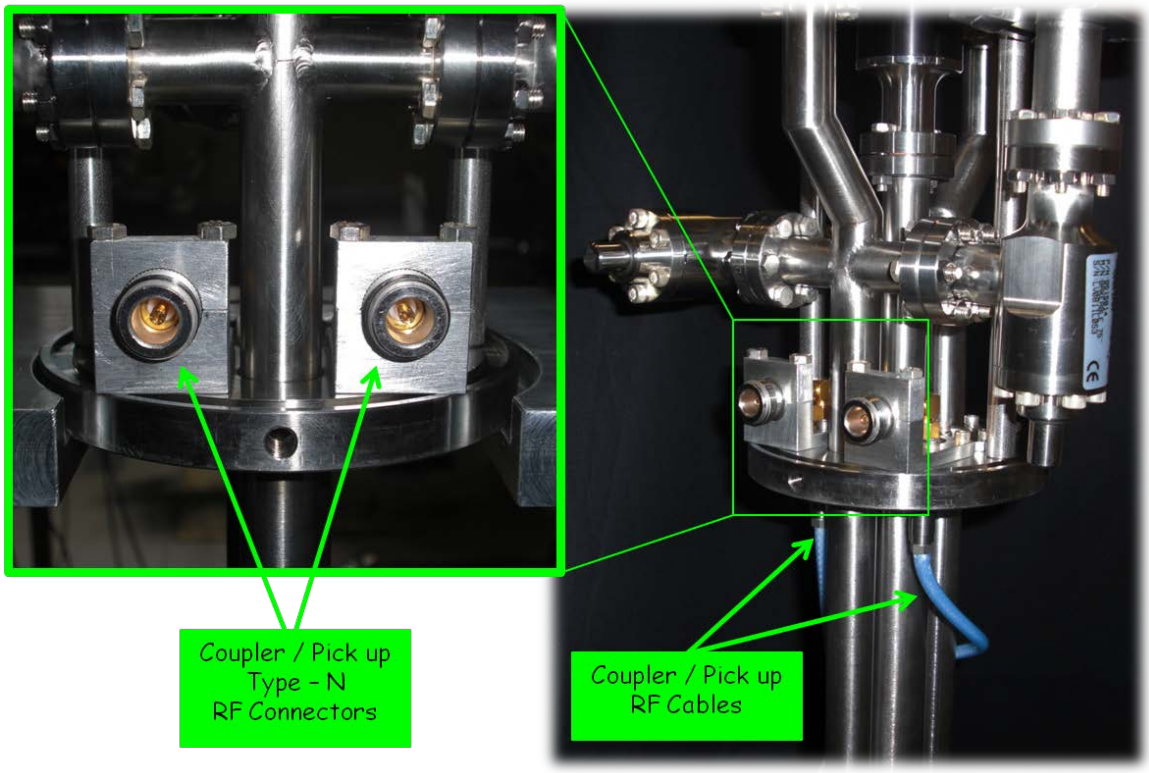


Figure 2-24 The RF cables are clearly visible: they are embedded into two supports, thought to avoid their stresses or motions during the cavity measurement.

Ones the cavity is evacuated and the whole stand is backed out, to reach a base pressure lower than 10^{-8} mbar, the main pumping unit is disconnected and the ion pump is turned on. After that cavity is shielded with a μ-metal screen *(Figure 2-25)* to avoid magnetic field entrapment during the cooling procedure.

At this point we are ready for the RF test.

The stand is slowly inserted into the helium dewar using the overhead travelling crane *(Figure 2-26)* and cooled at the temperature of the liquid helium 4,2 K. The surface resistance value of the superconductor under test is obtained through the measure of quality factor of the cavity using the equation

$$R_S = \frac{G}{Q}$$

where, as mentioned in the first chapter, *G* is the geometrical factor. It is the same for 1,5 GHz and 6 GHz cavities and it is equal to 287.

Figure 2-25 Detail of μ-metal screen used during the RF test to avoid the parasitic magnetic fields.

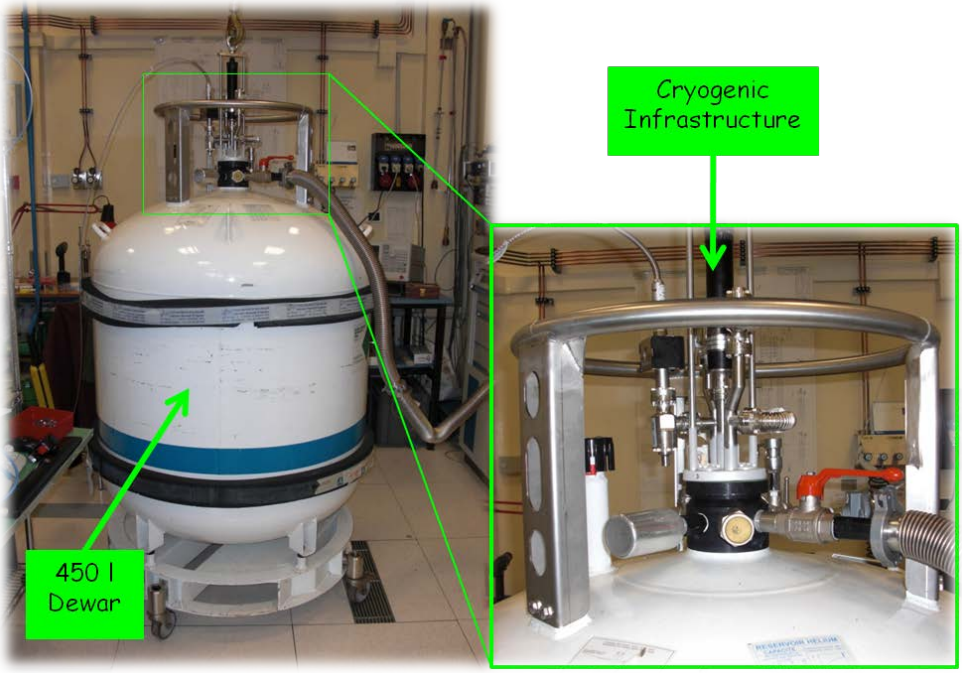


Figure 2-26 The 450 l helium dewar containing the cryogenic infrastructure during the cooling procedure before the RF test.

Figure 2-27 RF test apparatus.

In the last figure a photograph of the apparatus to test the cavity is shown.

The Q measurement can take advantage of computer-controlled procedures, which control the devices, collect data and assist the operator during the measurements. The procedures are similar for different resonators operating in a wide range of frequencies (160 MHz, 1,3-1,5 GHz, 6 GHz). Also most of the costly part of the equipment can be shared, the only things that differ being elements that have a small frequency range.

The computer is interfaced with a RF signal generator, two power meter, a frequency counter, two He level meters, temperature sensors, and stepping motor control.

The control program, developed in Visual Basic, allows to calibrate the RF lines, to find the resonant frequency, to set the loop phase, to lock the generator to the resonator frequency, to adjust suitable coupling conditions and the forward power level [6]. It measures the levels of pick-up, forward and reflected power signal.

The program computes the Q, plots Q as a funtion of the accelerating field allowing both a fast data analysis and recording *(Figure 2-28)*. Another set of similar programs controls the motion of the coupler and monitors the cavity temperature, the level of liquid He and other useful parameters.

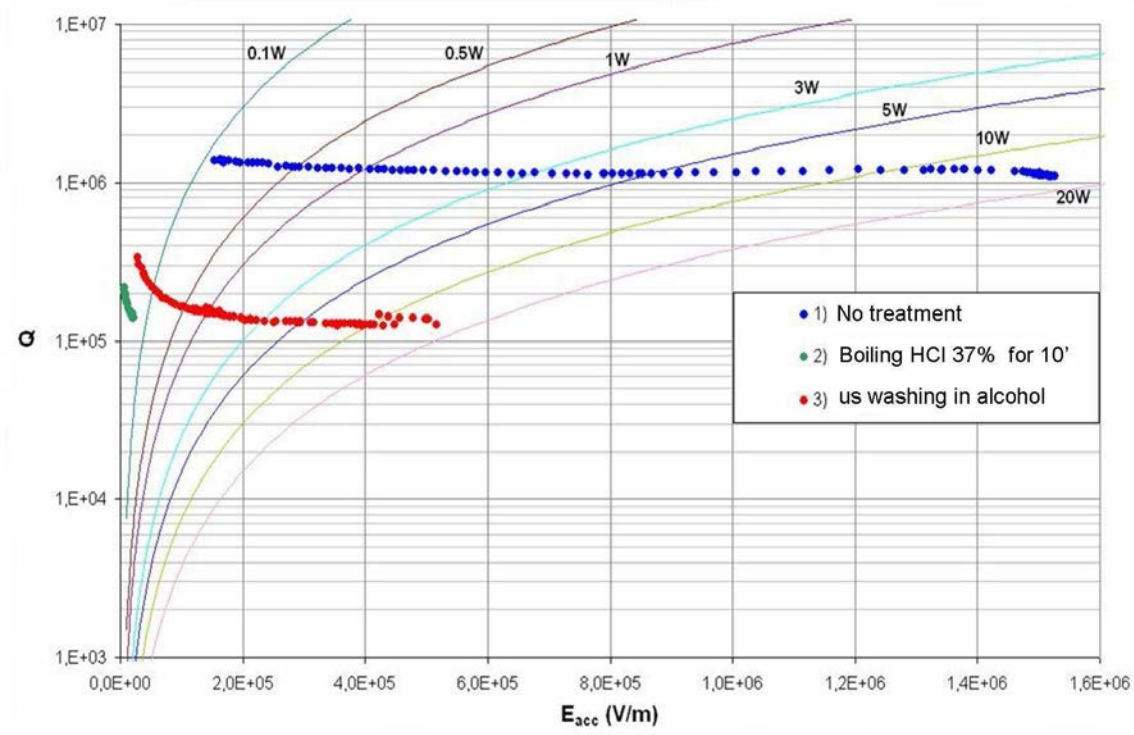


Figure 2-28 A tipical graph of the Q versus accelerating field E_{acc} for a 6 GHz cavity after each treatment.

2.7 Low cost application of innovative surface treatments

In addiction to the traditional surface treatments discussed before, it is possible to start a wide study of alternative surface treatments. Examples:

- Ionic Liquids / EP

  There is the necessity to reduce the risks and the costs of using a large quantity of dangerous acids in the traditional Nb chemistry. Thus the SRF community attention is now focused on alternative, HF free, chemical polishing treatments. A promising solution is represented by Ionic Liquids *(urea + choline cloride)*.

- Atmospheric plasma cleaning

  Plasma based atmospheric processes provide an excellent opportunity to achieve a cavity surface preparation process which is superior in terms of cost, performance, and safety, to the wet chemical process currently in use. Plasmas are chemically active media. Depending on the way they are activated and their working power, they can generate low or very high "temperatures" The fascinating possibility to perform etching and cleaning processes of RF cavities without the need of any vacuum pumping system has to be deeply explored realizing different atmospheric configurations as corona plasma, RF resonance plasma, plasma jet and torch.

- ALD - Atomic Layer Deposition

  ALD is a self-limiting (the amount of film material deposited in each reaction cycle is constant), sequential surface chemistry that deposits conformal thin-films of materials onto substrates of varying compositions. It is similar in chemistry to chemical vapor deposition (CVD). Due to the characteristics of self-limiting and surface reactions, ALD film growth makes atomic scale deposition control possible. ALD can be used to deposit several types of thin films, including various oxides (e.g. Al_2O_3, TiO_2, SnO_2, ZnO, HfO_2),metal nitrides (e.g. TiN, TaN, WN, NbN), metals (e.g. Ru, Ir, Pt), and metal sulfides (e.g. ZnS).

- Protective Coatings

  In example insulating over-layers or under-layers like Al_2O_3 etc.

- Vanadium Silanization

  It is a technique that allows to create thin films of V_3Si on V using silane (SiH_4)[6].

- Niobium Nitrurization

  It is a technique that allows to create thin films of NbN on Nb using nitrogen.

Chapter 3 CONCLUSION

3.1 Revolution in technology approach

The last chapter being so, 6 GHz cavities could represent a powerful tool to drive and push the superconducting radio frequency (SRF) community towards a new research approach. With a low research budget a large numbers of small cavities could be produced. A mini-lab infrastructure could be set up and with it the spun resonators can be used like 6 GHz samples to study alternative surface treatments and new superconductors.

Try to imagine 10 different laboratories all around the world that will test 10 different surface treatments per day for 10 days. You will have tested 1000 different cavities or treatments at the end of the 10^{th} day. In only one year you will have obtained such a dense statistics that SRF research would be revolutionized.

With 6 GHz resonators now a wide zoology of innovative surface treatments on superconductors would become accessible. The actual SRF limits could be maybe surpassed because of the low costs of small cavities processing and the large number of cavities treated and tested.

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