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P. Fabricatore, P. Fernandes, G.C. Gualco, F. Merlo, R. Musenich, R. Parodi:

**STUDY OF NIOBIUM NITRIDES FOR SUPERCONDUCTING R.F.  
CAVITIES**

**Study of Niobium Nitrides for Superconducting R.F. cavities**

P.Fabbricatore, P.Fernandes\*, G.C.Gualco,  
F.Merlo\*\*, R.Musenich, R.Parodi

I.N.F.N. Sezione di Genova, via Dodecaneso, 33 16146 Genova

(\*) I.M.A., via L.B.Alberti 4 Genova

(\*\*) Istituto di Chimica-Fisica, Universita' di Genova,  
Corso Europa 26 Genova

**Abstract**

The paper deals with the first results of a study on Nb-N system having as main goal the performance of niobium nitride R.F. cavities. The nitrides were obtained by diffusion of nitrogen in bulk niobium at high temperature. Metallurgical and resistive measurements have been done to determine the phases and the critical temperature of the samples. Furthermore in order to study the R.F. behaviour of the material, a 4.5 GHz niobium nitride cavity has been built and tested. The R.F. measurements showed, above 4 K, a surface resistance in good agreement with the computed values ( $1.15 \mu\Omega$  at 4.2 K), i.e. about a factor of ten better than the value obtained for niobium at the same temperature and frequency.

At temperatures below 4 K the residual resistance became prevalent and its value was found to be  $0.69 \mu\Omega$ .

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## Introduction

In new particle accelerators reaching high accelerating fields the use of superconducting cavities becomes necessary. At present the superconducting material used in radio-frequency (R.F.) applications is niobium, with critical temperature  $T_c=9.2$  K, but to reach very high accelerating fields (50-100 MV/m) other materials, having  $T_c$  and critical field higher than niobium, could be used. The interest in the materials with critical temperature higher than niobium is connected to the relation between  $T_c$  and the R.F. surface electrical resistance (that is an exponential function of  $-T_c/T$ ) and to the fact that the accelerating field increases, other factors being equal, as the surface resistance decreases.

Therefore high values of  $T_c$  could allow to reach higher accelerating fields besides reducing the thermal losses. Niobium nitrides are potential useful materials for superconducting applications due to their high critical temperature (the maximum value of  $T_c$  founded is about 17 K) and to the relatively easy preparation process.

As in R.F. applications the electrical and magnetic fields penetrate into the surface of superconductors only for a thin depth (about 20 nm for Nb at 4.5 GHz), the niobium nitrides have a great development as thin films obtained by reactive sputtering<sup>1,2,3</sup>. An alternative method is the gas-metal reaction by bulk diffusion, that seems to be simple and reliable for the cavity production; for this reason we investigated the binary Nb-N system analyzing samples prepared by bulk diffusion in nitrogen atmosphere (about 100 hPa) at temperature ranging from 1700 to 2000 K. Aiming to R.F. applications, a complete study of the material requires the measurement of R.F. properties such as the surface BCS resistance, the residual resistance, the secondary emission coefficient, the breakdown mechanism and the maximum achievable fields. In order to have a complete R.F. characterization of niobium nitrides a R.F. cavity at resonant frequency of 4.5 GHz, made by reacting a niobium cavity in nitrogen atmosphere, was tested.

## The Nb-N system

As regard the binary system Nb-N several phases are known, in a range of composition from 0 to 1.2 of N per Nb atom ratio<sup>4,5</sup>. These phases are not stoichiometric compounds but for each of them a range of composition is allowable. The variation in the composition of a phase influences its physical properties as the superconductive critical temperature.

The phases known as  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$  and  $\text{Nb}_4\text{N}_5$  show superconductive behaviour, but the most interesting for R.F. applications are the  $\gamma$  phase and the  $\delta$  one, having critical temperatures ranging from about 13 K to more than 17 K<sup>1,6</sup>. Though the superconducting properties of niobium nitrides were extensively studied, it is not clear the relation between the measured critical temperature and the crystallographic structure, but it seems that  $T_c$  mainly depends on the N content of the nitride without discontinuity due to the phase changing<sup>6</sup> (the upper limit of the nitrogen content of the  $\gamma$  phase being close to the lower limit of the  $\delta$  phase).

The phase diagram of the binary system Nb-N<sup>4</sup> shows that the  $\delta$  phase is stable at temperatures higher than 1600 K: as the temperature is lowered, a transition occurs giving  $\gamma$  or  $\epsilon$  phase depending on the reaction conditions<sup>4</sup>. Nevertheless the  $\delta$  phase can be stabilized at low temperatures by impurities as carbon or oxygen<sup>1</sup>. The  $\delta$  phase can be also obtained by reactive sputtering in atmosphere of argon and nitrogen but, as proposed by Gavalier et al.<sup>1</sup>, also in this case the presence of the  $\delta$  phase can be related to the presence of impurities.

The diffusion of nitrogen in bulk niobium does not allow to obtain  $\delta$  Nb-N, if very low impurities are present in the reagents or are added by the reaction environment: this is confirmed by our experience as we shall show in next sections.

We shall give a short review of  $\gamma$  and  $\delta$  phases together with the  $\alpha$  and  $\beta$  ones, not interesting for R.F. applications, but often observed in our samples.

The  $\alpha$  phase is a solid solution of nitrogen in niobium having a body centered cubic niobium lattice with the nitrogen atoms in interstitial places. The critical temperature of this phase ranges from less than 4 K to 9.2 K (pure niobium) and decreases as the niobium content increases<sup>9</sup>.

The  $\beta$  phase has hexagonal Nb lattice with the nitrogen atoms in the tetragonal interstices. The composition, given in N atoms number per Nb atom, ranges from 0.4 to 0.5. The s.c. critical temperature is about 8.6 K, very close to the niobium one.

The  $\gamma$  phase has higher N content, ranging from 0.75 to 0.79 N atoms per Nb atom, as reported by Brauer<sup>4</sup> (other studies showing a range from 0.7 to 0.85<sup>10</sup>) The lattice has a tetragonal structure having lattice parameters ratio  $c/a$  of 1.96-1.97, depending on the N content.

The  $\delta$  phase is the most interesting from the superconductivity point of view, giving the maximum obtained  $T_c$  of about 17 K. Its lattice has a slightly tetragonally distorted face centered cubic (NaCl type) symmetry with  $c/a=0.998$ . The composition of the  $\delta$  Nb-N ranges from 0.88 to 0.98 (atom ratio). As previously said, this phase is unstable at low temperatures (if not stabilized by impurities) and was never observed in our samples.

## Reaction method

As our aim is the study of the R.F. behaviour of superconducting materials we designed the reaction environment foreseeing the reaction of resonant cavities and, therefore, taking into account the cleanness condition requested for a cavity surface: we avoided the use of oil diffusion pumps and the presence of objects that can generate dust or pollution by unwanted elements (for instance carbon). Moreover we used pure reacting materials.

To prepare the niobium nitrides samples we started from specimens of reactor grade niobium: the samples, 0.01 cm thick, have a U-shape suitable for the resistive measurements of  $T_c$ .

The nitrides are prepared by heating the niobium specimens in pure nitrogen atmosphere: the nitrogen used in the reaction has a total content of impurities of 10 ppm (1 ppm of oxygen and 3 ppm of water). The reaction occurs in a furnace where the pressure can range from U.H.V. conditions ( $10^{-6}$  Pa) to atmospheric pressure and the maximum temperature available is about 2300 K. The furnace is evacuated by a turbomolecular pump and the U.H.V. conditions are guaranteed by six ion pumps having a total pumping speed of 240 l/sec.

The heating element is a cylindric resistor (10 cm in diameter) made of tantalum as the inner shields, while the outer shields are made of molybdenum. The sample holder is a tungsten disk.

In order to remove the impurities adsorbed on the surfaces of the samples and of the furnace elements (oxygen, water, carbon oxide, hydrocarbons) an annealing, at 1700 K and at pressure of  $10^{-6}$  Pa for about two hours, is done before starting the nitriding reaction.

Then the furnace is filled with nitrogen, warmed up and kept at the reaction temperature for several hours. We prepared samples in several conditions of temperature and pressure, ranging from 40 to 200 hPa and from 1700 to 2000 K, and for reaction times from one hour and a half to three hours and a half: in table I temperature, pressure and time of reaction for the samples we prepared are given.

The reaction time elapsed, the furnace is cooled down (about 1000 K) in few minutes in nitrogen atmosphere to prevent the loss of nitrogen at the surface of the samples and the building of unwanted phases. Then the furnace is evacuated at  $10^{-6}$  Pa and kept in this environment until the measurements are done.

The obtained nitrides have a light gold colour and are very hard and brittle. Due to the brittleness of the material, the samples must be handled with a great care.

The same technique we have described was used to react the niobium cavity. The cavity from which we started, described in the section "R.F. measurements", has walls 0.1 cm thick with large niobium grains (0.3-0.5 cm) visible on the surface. Before the reaction the cavity was cleaned by removing the external material layer (several microns) by chemical etching in the standard mixture of nitric, hydrofluoric and phosphoric acid, then was rinsed in distilled water and in methanol.

### Metallurgical analysis

A characteristic of the niobium nitrides obtained by bulk diffusion is the inhomogeneity of the composition (and of the structure) along the direction perpendicular to the external surface, i.e. the nitrogen content decreases from the surface to the centre of the samples, so that either a single phase with variable composition or several phases can be obtained. Aiming to the R.F. behavior of niobium nitrides we are interested in the phase and its composition at the samples surface for a depth of few tens nanometers.

As the  $\gamma$  phase has critical temperature higher than the other phases, to the exclusion of the  $\delta$  one, and because of its  $T_c$  increases with the nitrogen concentration, the best situation at the surface is to have the  $\gamma$  phase with the highest nitrogen concentration.

As said previously and as confirmed by our analysis the  $\delta$  phase was not obtained by the method we used, i.e. by diffusion of nitrogen in bulk niobium, without carbon or oxygen impurities.

In order to have informations on the crystallographic structure of the nitrides we made three different and complementary metallurgical analyses: X-ray diffraction from the sample surface, X-ray diffraction from powders and micrographic analysis. All these analyses allowed to know the distribution of the phases in the samples and the lattice parameters which are related to the nitrogen concentration<sup>10</sup>.

The X-ray diffraction showed that the  $\gamma$  phase was obtained in all the samples, often together with the phases  $\alpha$  and  $\beta$ , while the  $\delta$  phase was never observed. The phases  $\alpha$  and  $\beta$  at low nitrogen content were present in all the samples with the exclusion of the N. 5 reacted at 2000 K. These phases were seen by diffraction by powders and were not present at the surface of the samples where was observed the  $\gamma$  phase only. As it is shown in table II the lattice parameters of the  $\gamma$  phase are different on the surface and into the bulk of the samples. Furthermore, confirming the X-ray diffraction, we observed, by micrographic analysis of the samples Ns. 1, 2, and 3, the presence of several layers corresponding at the three phases  $\alpha, \beta$  and

$\gamma$ : the thickness of the outer layer ( $\gamma$  phase) and of the middle layer ( $\beta$  phase) does not present great variations for the three samples while the thickness of the inner layer depends on the sample and decreases increasing the reaction time and the nitrogen pressure. By the comparison with the critical temperature measurements it came out that the nitrogen pressure is not a critical parameter over 100 hPa.

A micrograph of the sample N. 2 is shown in fig. 1.

Table II shows the results of the X-ray analysis for samples obtained in different conditions.

As it is shown it comes out that the differences among the values of the lattice parameters are very little and the comparison with the critical temperature values, also reported, is not feasible.

In order to have a complete check of the process we analyzed, after the R.F. measurements, the nitride of the cavity. Before the reaction the dimension of the niobium grains of the cavity were about 0.3-0.5 cm and on the nitride it was possible to observe the shape of the niobium old grains. Nevertheless the micrographic analysis in polarized light and the X-ray diffraction have shown, inside the old grains, a structure of smaller nitride grains having dimensions of about 10 microns. For comparison the dimension of the grains obtained by reactive sputtering ranges from 10 to 100 nanometers<sup>1,11</sup>.

Looking at the section of the cavity wall we observed that the thickness of the  $\gamma$  phase is about 30 microns from the surface but a complete diffusion occurred along the niobium grain edges. This fact could explain the brittleness we observed in the cavity: though the bulk has metallic mechanical properties, the fracture occurs along the grain boundaries completely reacted.

#### D.C. measurements

The first test we made on the superconducting properties of the niobium nitride samples had the goal to determine the critical temperature of the material. In order to obtain these values we made measurements of the resistance of the samples as function of the temperature.

The samples were mounted in a variable temperature cryostat and cooled down to 4.2 K. Besides the temperature was increased very slowly. The resistance of the sample was determined by a four wires measurement and the temperature was monitored by a calibrated cryogenic thermometer (Lakeshore CGR 1000).

The measured critical temperature values are shown in table II together with the results of the metallurgical analysis. The resistance measurements did not allow to obtain the residual resistance ratio (RRR) of the  $\gamma$  nitride because of



the presence of  $\alpha$  and  $\beta$  phases. The sample N. 5, composed by the  $\gamma$  phase only, was not measured due to its extreme brittleness and its critical temperature was determined via magnetic susceptibility measurements. These measurements showed also that some different phases with low  $T_c$  (8.6 and 6.4 K) are on the surface of the sample, and probably they are placed in thin layers at the grain boundaries. Only this geometrical disposition could explain how an amount of material, well below the sensitivity of the X-ray diffraction (one percent) could be responsible for large variations of the AC susceptibility as the ones measured in our case.

### R.F. measurements

The R.F. properties of niobium nitride were obtained by measuring the R.F. characteristic of a Nb-N cavity operating at 4.5 MHz in the  $TM_{010}$  mode.

The cavity was an old niobium one, used in a previous research<sup>12</sup> to state the limit field and  $Q_0$  for a niobium superconducting accelerator.

The maximum  $Q_0$  obtained for the niobium cavity was  $5.7 \cdot 10^7$  at 4.2 K and  $2 \cdot 10^8$  at 1.8 K at a driving power of few mW. The R.F. parameters for the computation of the fields and the surface resistance were obtained by using the OSCAR2D code<sup>13</sup>. A cross section of the cavity with superimposed the electric field lines is shown in fig. 2. The relevant R.F. parameters of the cavity are reported in table III.

The cavity was reacted together with the sample N.3, which showed a critical temperature of 14.6 K.

After the reaction and without any further treatment, the cavity was mounted on the test apparatus, connected to a 60 l/sec ion pump and evacuated to  $1 \cdot 10^{-6}$  Pa at room temperature: during this operation the cavity was exposed to the atmospheric air for half an hour only. The cavity was continuously pumped also during the cryogenic tests, as in our previous experiments on niobium cavities, in order to simulate the operating condition of a superconducting accelerator. The cavity test was performed in a standard parallel cryostat which could be cooled down to 1.3 K by lowering the pressure. At pressure of 20 hPa corresponding to 1.8 K, the available maximum refrigeration power is about 5 W, more than enough for compensating the 100 mW steadystate heat losses of the cryostat and cooling the cavity driven by 5 W of R.F. power, corresponding to a theoretical field level of 1570 gauss or 35 MV/m of accelerating field, far over the maximum value of 20 MV/m or 1080 gauss achieved in the previous runs.

The cavity was shielded against the local earth magnetic field using a cryogenic  $\mu$ -metal shield reducing the load field to  $1 \cdot 10^{-3}$  gauss, in order to reduce the added losses due to flux trapping in the superconducting material at the transition.

The cavity was cooled down very slowly to avoid thermal stresses on the brittle structure and thermoelectric currents in the cavity due to differences of temperature.

The  $Q_0$  measurement was performed in the usual way by the standard decrement method.

The R.F. power to feed the cavity is generated by a voltage tuned oscillator, locked to the resonant frequency of the cavity under test by an automatic frequency control (AFC) feedback loop.

The signal coming from the generator, amplified by a TWT power amplifier up to a level of 20 watts, is feeded to the cavity via a R.F. switch. The modulated R.F. power is applied to the cavity.

The layout of the used R.F. system is shown in fig. 3. By measuring the transmitted, reflected and applied power to the cavity, together with the decay time  $\tau$  of the R.F. power radiated by the cavity when the driving R.F. power is turned off, the values of the coupling coefficient  $\beta$ , the loaded quality factor  $Q_L$ , and the power losses of the cavity are measured. The  $Q_0$  is related to the measured quantities by the relations

$$Q_L = 2\pi f_0 \tau$$

where  $f_0$  is the resonant frequency of the cavity.

$$Q_0 = (1 + \beta)Q_L$$

The knowledge of the  $Q_0$  and power losses at the breakdown of the cavity allows to compute, by using the parameters of cavity reported in table III, the maximum values achieved by the magnetic and electric fields in the cavity. The measurements at 4.2 K on our niobium nitride cavity showed a  $Q_0$  value of  $2 \cdot 10^8$  corresponding to a surface resistance of  $1.15 \mu\Omega$ , a factor of two better than the corresponding value of the niobium at the same temperature.

We emphasize that the R.F. surface resistance value at 4.2 K for the niobium nitride is in very good agreement, as for niobium, with the BCS predicted value showing no effect due to the residual resistance. By lowering the temperature to 1.8 K the  $Q_0$ -value changes smoothly reaching the limit of  $3.3 \cdot 10^8$  due to the high value of R.F. residual resistance of  $6.9 \cdot 10^{-7} \Omega$ .

This limit value of residual resistance is reached at a temperature of 3 K and does not change by changing the bath temperature nor the R.F. level till the breakdown is reached.

The surface resistance of the niobium nitride was measured on the whole range of temperature from 1.8 K to the transition temperature of 14.6 K by monitoring the cavity temperature by a carbon glass resistor (Lake shore CGR-1000).

The usual plot of the surface resistance versus  $T/T_c$  (fig. 4) shows a good agreement for  $T > 4$  K with the value computed by using the BCS theory. Fig. 5 shows a plot of the BCS theoretical value of  $R_s$  up to 1.8 K.

The cavity quenched at a power dissipation limit of 100 mW, showing a slow transition due to the thermal heating on the whole cavity, in agreement with the similar behavior of niobium with high value of residual resistance (in the  $1.5 \mu\Omega$  range).

### Discussion

The results of our analysis on the Nb-N samples, prepared by diffusion of nitrogen in bulk niobium, point out that only the  $\gamma$  phase, together with phases having lower nitrogen content ( $\alpha$  and  $\beta$ ), can be obtained by the method we described. The maximum critical temperature observed in our samples was 15.6 K. The other superconducting phase of niobium nitride ( $\delta$  Nb-N), having higher critical temperature in respect to the  $\gamma$  Nb-N, was not observed.

These results are in agreement with previous metallurgical analysis: in fact the  $\delta$  phase is not stable at low temperatures if not stabilized by carbon or oxygen impurities and it is generally obtained either by bulk diffusion in presence of impurities or by reactive sputtering. Nevertheless also in the sputtered Nb-N films the presence of the  $\delta$  phase seems to be related to the presence of carbon impurities<sup>1</sup>.

As we are interested to the performance of R.F. cavities, our aim is to reduce the surface electrical resistance, i.e., according to the BCS theory, to increase the critical temperature. In this case the increasing of critical temperature is related to the addition of impurities making the  $\delta$  phase stable at low temperatures (for instance by introducing small amount of methane in the reaction environment). Nevertheless, at the present time, the effect of the impurities on the residual surface resistance (not BCS) is not known: so it is not clear whether to obtain the  $\delta$  phase by adding impurities would produce a real improvement and this problem must be carefully studied.

We made a R.F. characterization of the niobium nitride ( $\gamma$  phase) by measuring the R.F. properties of a cavity. Our measurements showed a good agreement

with the BCS theory at temperatures higher than 4 K while at lower temperatures the effect of the residual surface resistance becomes prevailing. From the experimental data, the cavity being in liquid helium bath at atmospheric pressure, it results that a gain of a factor ten is reached for the nitride in respect to the niobium but, due to the high residual resistance of the nitride, the niobium R.F. resistance in superfluid helium bath is better than the nitride one.

High residual resistance is generally related to the contamination of the cavity surface: in this case we would exclude the hypothesis of contamination effect because the nitride cavity was carefully handled and was submitted to all the treatments that in niobium cavities results in good R.F. surfaces (residual surface resistance in the  $10^{-7} \Omega$  range). Furthermore the furnace used to react the cavity was used in the past to anneal niobium cavities that, after this treatment, showed  $Q_0$  of few  $10^9$  and surface field up to 40 MV/m and 1000 gauss.

Starting from the previous considerations we suppose that the high residual resistance we observed in the nitride cavity does not depend on the surface condition.

Another hypothesis can be advanced: the residual resistance could be mainly related to the presence of phases having low critical temperature or not superconducting at all.

This explanation of the high residual resistance is suggested by the analysis on samples of sputtered Nb-N films reported by Wagner et al.<sup>14</sup>. In their samples the Nb-N grains are found to be separated by amorphous material few nanometer thick. We can evaluate the amount of material having poor R.F. behavior in the hypothesis that its electrical conductivity similar to the one of the copper: dividing the copper surface resistance (about  $2 \cdot 10^{-2} \Omega$ ) by the measured residual resistance ( $7 \cdot 10^{-7} \Omega$ ) we obtain the surfaces ratio  $3 \cdot 10^4$ . Therefore the amount of not superconducting phase is very low in respect to the  $\gamma$  phase and cannot be observed by X-ray analysis whose sensitivity allows to distinguish phases in quantity ratio higher than one percent.

A surface ratio of  $3 \cdot 10^4$  corresponds to about 1 nm of resistive material at the edge of the 10  $\mu\text{m}$  nitride grains, again in good agreement with the thickness of the amorphous material found by Wagner. This our assumption is backed by the analysis of the magnetic susceptibility measurements of our fifth sample.

### Acknowledgements

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Table I - Reaction conditions for the niobium nitride samples. The cavity was nitrated together with the sample N. 2.

SAMPLE NUMBER	REACTION CONDITION		
	T (K)	p (hPa)	t (hours)
1	1700	40	3 1/2
2	1700	130	3
3	1700	200	1 1/2
4	1700	200	3
5	2000	130	3

Table II - Lattice parameters obtained by X-ray diffraction and critical temperature of the niobium nitride samples.

SAMPLE NUMBER	LATTICE PARAMETERS DIFFRACTION BY SURFACE			LATTICE PARAMETERS DIFFRACTION BY POWDERS			T <sub>c</sub> (K)
	a (Å)	c (Å)	c/2a	a (Å)	c (Å)	c/2a	
1	4.382	8.653	0.987	4.385	8.622	0.983	13.1
2	4.384	8.658	0.987	4.385	8.625	0.983	14.6
3	4.381	8.656	0.988	4.384	8.625	0.984	14.3
4							14.9
5	4.382	8.646	0.987	4.384	8.615	0.983	15.6

Table III - Relevant cavity parameters where  $E_p$  and  $H_p$  are the maximum electrical and magnetic fields on the surface,  $E_a$  is the effective accelerating field,  $P$  is the dissipated power,  $Q$  is the quality factor and  $G$  is the geometrical factor.

Resonant mode	TM <sub>010</sub>
Resonance frequency	4587 MHz
$E_p/(PQ)^{1/2}$	742 V/mW <sup>1/2</sup>
$E_a/(PQ)^{1/2}$	416 V/mW <sup>1/2</sup>
$H_p/(PQ)^{1/2}$	0.0157 gauss/W <sup>1/2</sup>
Shunt impedance	2.63 10 <sup>11</sup> Ω
G	229 Ω

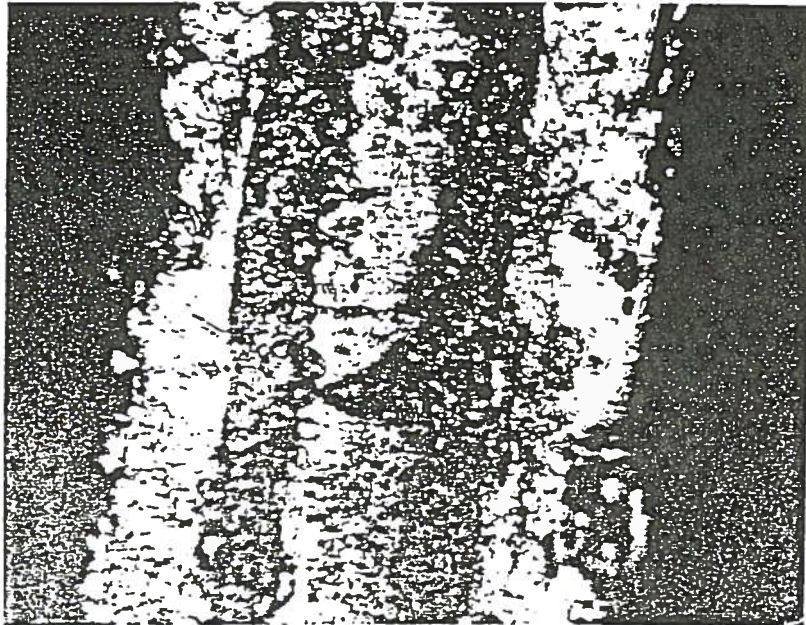


Fig. 1 - Micrograph of the section of a niobium nitride sample (N.2). Three phases are present:  $\gamma$  Nb-N (external layers),  $\beta$  Nb-N (middle layers) and  $\alpha$  Nb-N (internal layer).

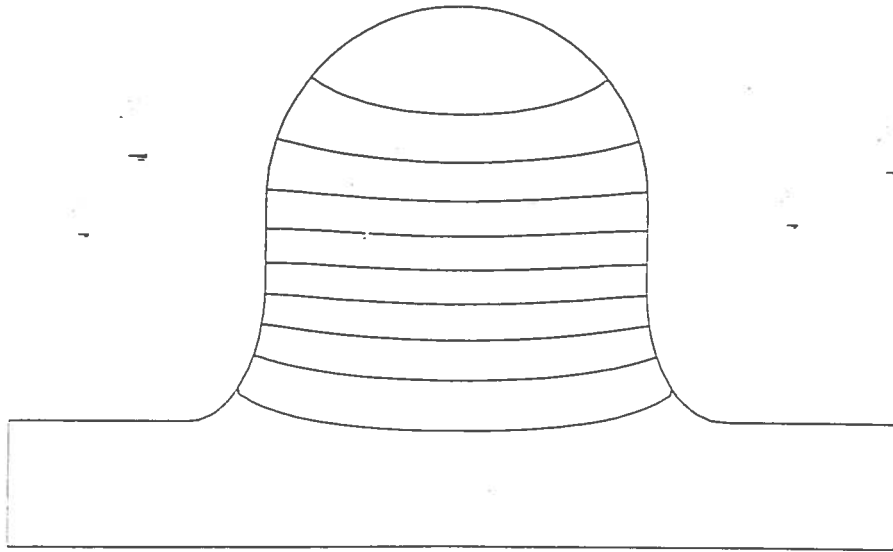


Fig. 2 - Cross section of the niobium cavity<sup>12</sup> nitrided to measure the R.F. properties of niobium nitride. The electric field lines are superimposed<sup>13</sup>

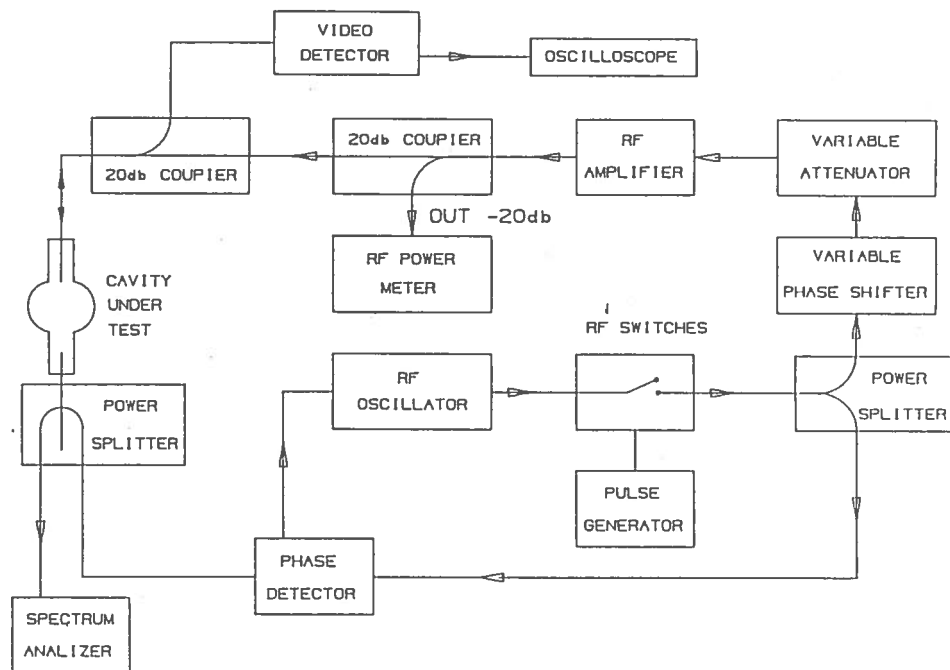


Fig. 3 - Scheme of the R.F. system



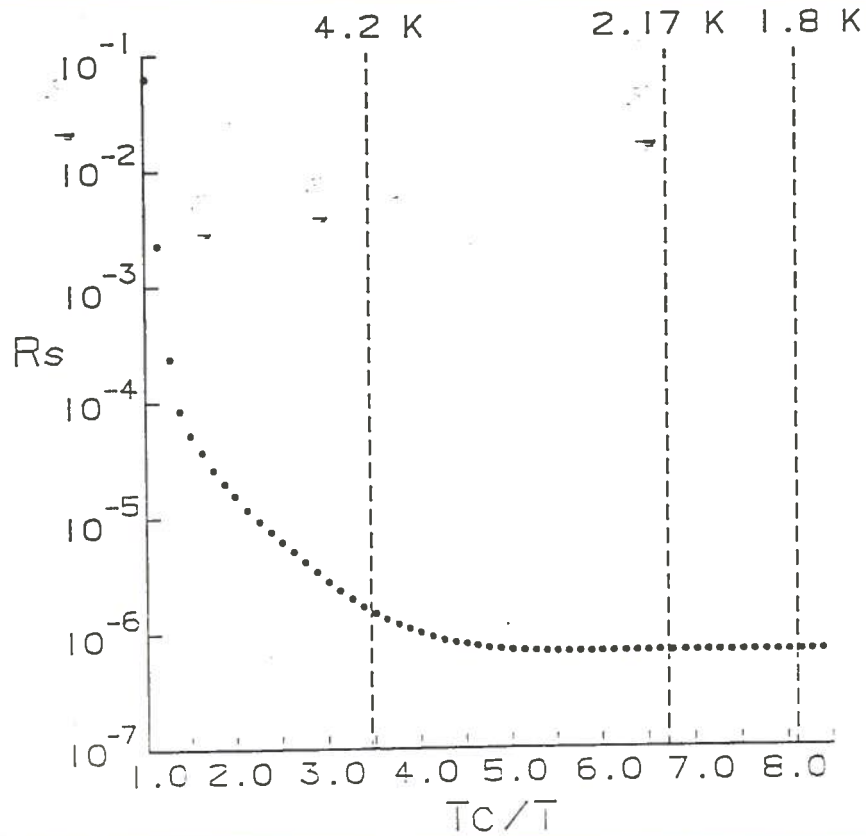


Fig. 4 - Experimental surface resistance vs. reduced temperature  $T/T_c$  for the niobium nitride cavity

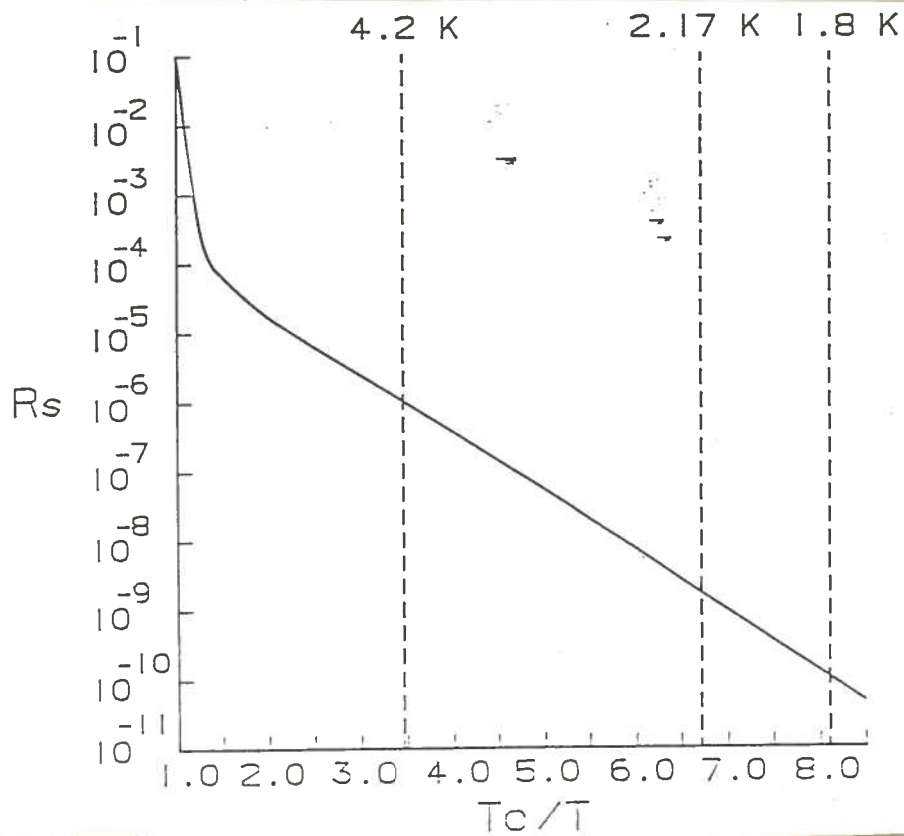


Fig. 5 - Theoretical BCS surface resistance vs. reduced temperature  $T/T_c$  up to 1.8 K for the niobium nitride cavity