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# THIN NIOBIUM SUPERCONDUCTING FILM PREPARED BY MODIFIED CYLINDRICAL MAGNETRON

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## Introduction

For future large superconducting RF-accelerators, technology of the Nb coated cooper cavities appears as an interesting alternative to the bulk-Nb cavities. Coating technology is advantageous compared to bulk niobium since copper is cheaper than niobium. Sputtering is a well-known technique for coating copper RF cavities with superconducting thin film [1]. The cylindrical magnetron-sputtering configuration for coating 500 MHz cavities with thin Nb films was developed for the first time at CERN [2]. In this system, the magnetic field for a plasma confinement is produced by a coil or permanent magnets placed inside the cylindrical niobium cathode. The sputtering discharge is realized in a noble gas atmosphere between the central cathode and the grounded cavity.

The technology of thin niobium film coating was successfully used for the production of the 350MHz LEP accelerating cavities. For high-Q, high gradient 1.5GHz cavities, a further progress in this technology is still needed.

For the coating of 1.3 GHz cavities that are 3 times smaller in dimension it was proposed at the University of Roma Tor- Vergata the cylindrical magnetron sputtering system with the magnetic field produced by external coils placed on the outside of the vacuum chamber [3]. Such sputtering system was realized in early 90's, the set-up was put into operation in the mid 90's and since 1997, in the frame of a collaboration with SINS Swierk, more systematic studies of the deposition of Nb films have been undertaken. The studies are aimed on optimization of the sputtering conditions as well as on characterization of the produced Nb films. The paper presents the recent results obtained with modified magnetron set-up. Glass and sapphire samples coated with 1-2μm niobium film were characterized by measurement of resistance versus temperature and by x-ray diffraction.

## Sputtering set-up

The sputtering system is schematically shown in Fig.1. The stainless steel deposition chamber has the shape of the 1.3 GHz cavity.

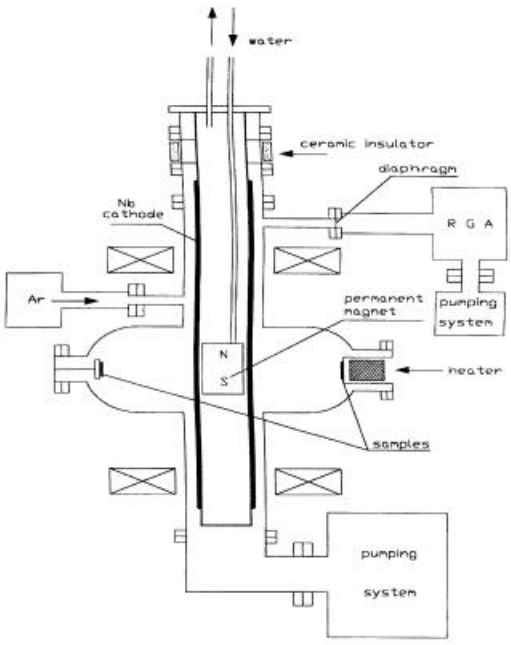


Fig.1 Sputtering system scheme

The vacuum chamber is evacuated by an ultraclean pumping system consisting of a diaphragm pump for a primary vacuum and a 180l/s turbomolecular pump. An ultimate pressure, of the order of $10^{-10}$ mbar and practically total absence of water and hydrocarbons are obtained after a 20 hours bake at 150°C. The system is equipped with a residual gas analyzer (RGA) to study the ultimate pressure gas composition and to monitor the percentage of gases produced during sputtering. The RGA

is equipped with another pumping system and it is connected to the cavity chamber through a 0.6 mm diaphragm. Pure Argon (99,9999%) is introduced as working gas for the sputtering process.

The cathode of magnetron is located on the axis of the system. It consists of a vacuum tight stainless steel tube surrounded by a high purity (RRR=150) niobium tube (20/24mm inner/outer diameters). The magnetron cathode is cooled by distilled water.

It is well known that sputtered films contain atoms of the noble gas used in the sputtering discharge. Energetic neutrals reflected from the cathode may be trapped in growing film. It was also shown in many papers [4], [5] that a discharge gas trapped in film leads to a deterioration of superconducting film properties. For this reason a working gas pressure and a discharge voltage should be kept as low as possible during ofthe deposition. To reduce this effect the study of a proper magnetic field configuration in magnetron system is needed.

Two coils placed outside of the cavity chamber produce the magnetic field in our magnetron. The coils are surrounded by soft iron shield of 4mm thickness. The magnetic field, well known from thermonuclear researches as "magnetic bottle configuration" was produced.

In order to obtain a high efficiency of magnetic confinement of electrons in magnetron the magnetic field lines have to die onto the cathode. The mirror ratio of the presented magnetic bottle configuration is about 2 and this value was limited by the dissipated power in the coils since the system of the coils cooling was inefficient.

For improving of the magnetic configuration of the magnetron, 2 SamCo permanent magnets (small cylinders 8mm diameter 16mm long) have been introduced into the stainless steel tube. This mixing of magnetic fields produced by coils and permanent magnet has improved the electron confinement leading to an increase of maximum discharge current of about a factor 2 respect to the previous configuration. Obtained I-V characteristics with improved configuration of magnetic field are shown in Fig.2.

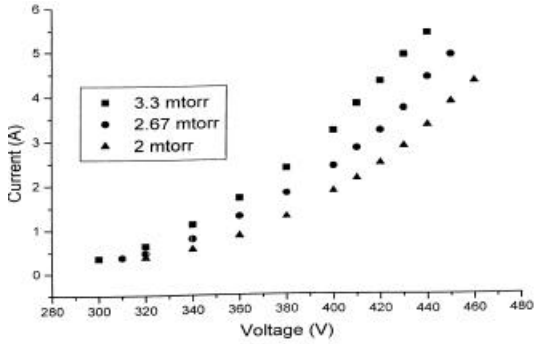


Fig.2. Discharge characteristics at different gas pressure

**Film deposition**

Small (14x19mm) sapphire or glass samples are placed on two sample holders located on the equator of the cavity. Prior the mounting on the sample holder, substrates are cleaned in an ultrasonic bath with acetone and rinsed with de-ionized water.

Due to heaters located inside the holders it is possible to keep samples during deposition at higher temperature, up to 300°C.

After the 20 hours bake-out at 150°C the ultimate pressure of $10^{-10}$mbar is reached at room temperature.

Argon is then injected at a pressure of about $7x10^{-3}$ Torr to start the discharge and then set at the desired value, usually between 1 and 4 mtorr. The discharge voltage is usually kept constant, while current in the coils is adjusted in order to obtain the maximum discharge current at the fixed pressure.

The cavity and the system are at room temperature when discharge starts, while samples can be heated and kept at higher temperatures (up to 300C) before and during the coating. In order to compare films deposited under different conditions, the sputtering time is chosen to obtain films of 1 μm thickness.

Table 1 lists the deposition parameters: working gas pressure, cathode voltage, discharge current, time of deposition, substrate temperature and measured values of Residual Resistivity Ratio.

**Table1**

| *Samp. #* | *Press. [mTr]* | *Volt. [V]* | *Curr. [A]* | *Time [min]* | *Temp. [C]* | *RRR* |
|---|---|---|---|---|---|---|
| 1.99 | 1.7 | 430 | 1.0 | 60 | | 8.6 |
| 3.99 | 2.0 | 430 | 1.0 | 60 | | 9.3 |
| 4.99 | 2.0 | 430 | 1.0 | 60 | | 10.8 |
| 5.99 | 2.0 | 420 | 0.98 | 60 | 275 | 27 |
| 6.99 | 2.0 | 420 | 0.98 | 60 | | 11.1 |
| 7.99 | 1.6 | 410 | 1.0 | 30 | | 7.0 |
| **11.99** | **1.6** | **410** | **2.1** | **30** | **300** | **28** |
| **12.99** | **1.6** | **410** | **2.1** | **30** | | **18.9** |

**Film characterization**

The produced samples are mainly characterized by measurement of resistance versus temperature and by x-ray diffraction pattern.

The R (T) curves are carried out with a standard four-lead technique and most of them are obtained in a cryocooler reaching 12K as minimum temperature, not low enough to measure the critical temperature of produced films, but sufficient to obtain the Residual Resistivity Ratio (RRR). RRR (also called $\beta_{10}$) is defined as the ratio of the resistivity at room temperature ($\rho_{300K}$) and the resistivity at 10 K ($\rho_{10K}$). This ratio gives an estimation of impurity and lattice defect content in the film.

Our $\beta_{10}$ value range from 7 to 30 among the best reported in literature produced by sputtering. Few samples are measured in a cryostat to check the superconducting critical temperature. The measures showed a very sharp transition ($\Delta T_c$<0.1K) and $T_c$ between 9.5 and 9.6K. The fig. 3 shows R (T) measurements. Fig.4 shows the detail of the transition region.

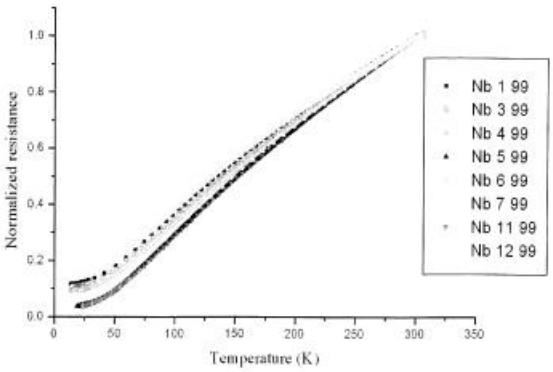


Fig.3. R (T) characteristics

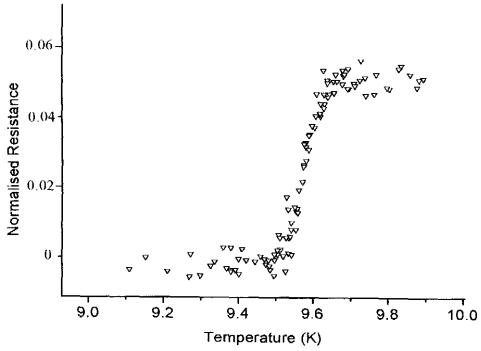


Fig.4.R (T) characteristics (transition region)

Data on critical temperature are in agreement with existing data on niobium films of high quality in presence of a compressive stress [6-7].

Samples were also analyzed by X-ray Diffraction in the Bragg-Brentano configuration ($\theta/2\,\theta$) using a Cu $K_\alpha$ source. A typical X-ray diffraction spectrum is reported in fig. 5 and it shows that Niobium films grow with the (110) plane parallel to the film surface.

For most of the explored coating conditions, the peak positions are shifted to angles lower than the nominal values, corresponding to a lattice constant about 0.5% larger than the bulk (Å = 3.303Å) in the growing direction. The produced niobium samples are in a compressive stress in the substrate plane mainly due to the bombardment of high energy neutral and to self-bombardment of the sputtered material during deposition [7][8].

This compressive stress is responsible for the observed increase of the critical temperature.

While the changes in the lattice parameters and stress are within the error of our measurements, we observe a significant increase of the $\beta_{10}$ obtained with the mixed magnetic configuration on samples coated at room

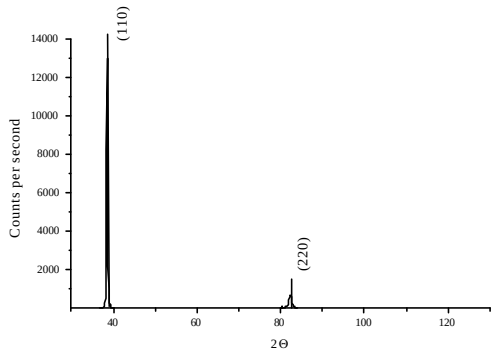


Fig.5 X-Ray diffraction spectrum in the $\theta/2\,\theta$ configuration for a typical niobium sample on sapphire. Only the reflections relative to the Niobium planes (110) and (220) are present

temperature, which rise from about 7 in the previous configuration up to 19 in the present one. Higher $\beta_{10}$ values can be obtained by the heating of the substrate with a maximum of $\beta_{10}$=30 for a coating temperature of 300°C.

## Conclusions

We have presented the results obtained on niobium films produced by magnetron sputtering in a UHV system. The use of a mixed magnetic configuration improves the efficiency of magnetic confinement of electron leading to higher discharge current. Very good film quality is obtained with critical temperature of about 9.5K and RRR values up to 30.

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