ARCO PROJECT STATUS REPORT

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Abstract

We designed and realized a planar arc source working in UHV conditions to study the deposition of superconducting Nb films. The vacuum arc technique could become a good alternative to magnetron sputtering in trying to improve the performance of superconducting Nb coated Cu cavities. First results obtained with Nb films deposited on Cu and sapphire substrates using the planar arc source are very promising. The films have been analysed by x-ray diffraction and optical and electron microscopy and their T_c, J_c and RRR have been measured. The results are "bulk-like" with RRR~50, T_c =9.26K, ΔT_c = 0.02 K, J_c~$3*10^7$ A/cm^2 even for thickness as low as 0.1μm. The roughness of Nb films on Cu substrates is mainly due to substrate preparation, while on sapphire substrates it is mainly due to the presence of microdroplets.

1 INTRODUCTION

Superconducting cavities for particle acceleration are mainly based on Nb bulk technology[1]. The Nb/Cu technology was demonstrated to be a valid alternative for relatively low accelerating fields (up to 8MV/m) by successful operation in LEP [2] and presents several advantages respect to Nb bulk cavities, namely better mechanical stability, insensitivity to external magnetic fields, lower cost, better thermal stability, easier conditioning on the machine, easier connection to the cryostat.

The present technology mainly consists in depositing Niobium films on the inner surface of a Cu cavity using a cylindrical magnetron-sputtering configuration in UHV conditions [3,4]. In recent years considerable R&D has been undertaken at CERN to improve the Q behavior and the top field value of 1.5 GHz Nb/Cu cavities [5]. Despite the effort, problems such as fast Q degradation at fields higher than ≈15 MV/m [6] and difficulty in reaching fields higher than 25 MV/m with Q_0 of $1x10^{10}$ required for SC linear colliders like TESLA [7], are not solved yet.

On the other hand, vacuum arc coating is known to be a powerful technique to produce films on several materials. Its main advantages, compared to the standard sputtering process are the highly ionized state of the evaporated material, the absence of gases to sustain the discharge and the high energy (about 50eV) of atoms reaching the substrate surface. The technique is thus a candidate for replacing magnetron sputtering in trying to produce films having bulk–like properties on Cu cavities. Its main disadvantage is the production of microdroplets (or macroparticles), emitted from the region of the arc spot, consisting of molten cathode material with typical dimensions in the range from 0.1 to 10 micron. Microdroplets become charged with electrons during their passage trough the plasma region near the cathode and accelerated to high energy. They solidify during their flight to the anode and can become embedded in the growing film. Several methods, some patented, are described in literature on how to filter-out microdroplets to obtain smooth films [8-11]. While planning to study filtering in a near future we present here preliminary work done with an unfiltered arc and aimed at characterizing films produced in different conditions so as to find the best parameters for coating.

2 EXPERIMENTAL SET-UP

Our planar arc source is mounted inside a UHV chamber pumped down to 10^{-10} Torr by an oil-free pumping system consisting of a membrane pump on the foreline of a drag turbo molecular pump.

The system is equipped with a Quadrupole Mass Analyzer (QMA) to check presence of contaminant gases before and during coating. The base pressure of $1x10^{-10}$ Torr is reached after one night baking at 200^{o}C. A schematic drawing of the system is shown in Fig. 1.

We apply 6 kV pulses to the triggering electrode (see Fig.1) to ignite the discharge under UHV conditions. The discharge starts between cathode and trigger and propagates to the anode while the arc spot moves to the top cathode surface. The conical cathode is obtained from a 50mm diameter high purity Niobium rod and fastened to a water-cooled Cu support. The vacuum chamber around the Nb cathode, also conical, with a final inner diameter of 90mm, was fabricated from a single stainless steel rod.

The coil surrounding the vacuum chamber generates the magnetic field needed to confine the arc spot to the cathode surface on which the spot moves randomly with a speed between 1and 10m/s. A floating potential screen surrounds the cathode to prevent the discharge from moving downwards, towards the bottom part of the chamber.

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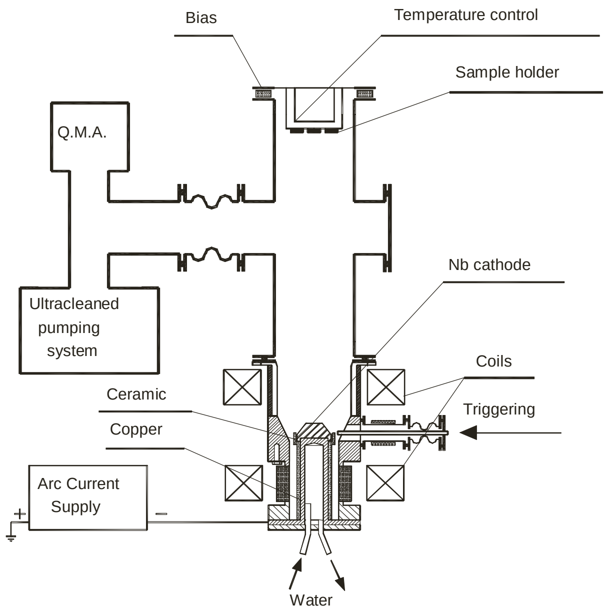

Figure 1: Layout of the UHV system with the planar arc source.

Since the discharge is sustained in the vapors of the cathode material, there is a minimum arc current below which the discharge is unstable. In our configuration and with a Nb cathode the minimum current is ≈ 90A. However, since the arc spot ignition is a random process, and the rate of evaporation is not exactly constant, the arc can extinguish even at slightly higher currents. For the production of Nb samples safe arc currents ≥120A have therefore always been used.

Substrates to be coated are secured to a massive Cu flange placed at a distance of about 50cm from the cathode and kept at a constant temperature of 100°C. The flange is electrically insulated from the vacuum chamber in order to have the possibility of biasing the substrates. Applying a negative bias voltage to the latter can be very useful for several reasons: a) electrons are reflected away thereby reducing the current through and the heating of the samples (more than 90% of the discharge current is supported by electrons) b) Nb ions are further accelerated thus reaching the growing film with higher energy c) a fraction of charged microdroplets is also reflected away.

3 RESULTS AND DISCUSSION

Several samples have been produced with arc currents in the range from 120A to 200A and bias voltages in the range from 0 to 100V. When applying a voltage bias to the sample holder, the bias current has been recorded.

3.1 The bias current

The current for negative bias voltages higher than 20V mainly consists of ions while for lower voltages electrons dominate. A graph of current versus bias voltage is shown in Fig.2 for an arc current of 140A. It can be seen that at a bias of about -18 V the current fluctuates around zero, indicating that the same number of ions and electrons arrive on the sample holder. If we let the sample holder potential floating, its potential soon reaches ≈ -18 V, while when it is connected to ground a net electron current of about 10A is observed to flow in the connection. With applied negative biases higher then 40V, the ion current saturates at a value of about 700mA. The measurement however shows large fluctuations, indicated in Fig 2 by error bars about the average value.

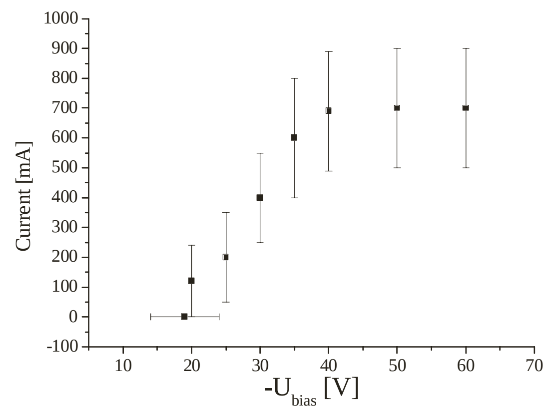

Figure 2: Bias current as function of the bias voltage.

At an average ion current of 700mA the instantaneous current values vary in between 500mA and 900mA indicating large changes in the quantity of evaporated material and/or in the mean ionization. It is worth recalling that during arc deposition Nb is mainly ionized 3 times (Nb^{3+}), but that Nb^{2+} and Nb^{4+} are also present [12]. Such large current fluctuations justify the requirement to lay sufficiently above the minimum current to reduce arc breaks to a minimum. The different values of ion and electron currents are attributed to the larger mobility of the latter. The result also confirms that electrons carry the major part of the plasma current and are responsible for heating of the system during deposition. Without a negative voltage bias and in the absence of cooling the temperature of the sample holder can reach 300°C in a few minutes. When a negative bias is applied external heating must be supplied to keep the coating temperature at 100°C. The deposition rate, ranging from 10 to 20A/s, depends mainly on the arc current value and is not influenced by the value of the applied bias.

3.2 Vacuum conditions

The base pressure in the chamber is $1x10^{-10}$ Torr, increasing to about $1x10^{-9}$ Torr when the sample holder is heated to 100°C prior to the arc discharge.

With the triggering electrode sparking, to ignite the arc discharge, the pressure increases further to $\approx 10^{-7}$Torr from outgassing of the chamber walls caused by the formation of the plasma.

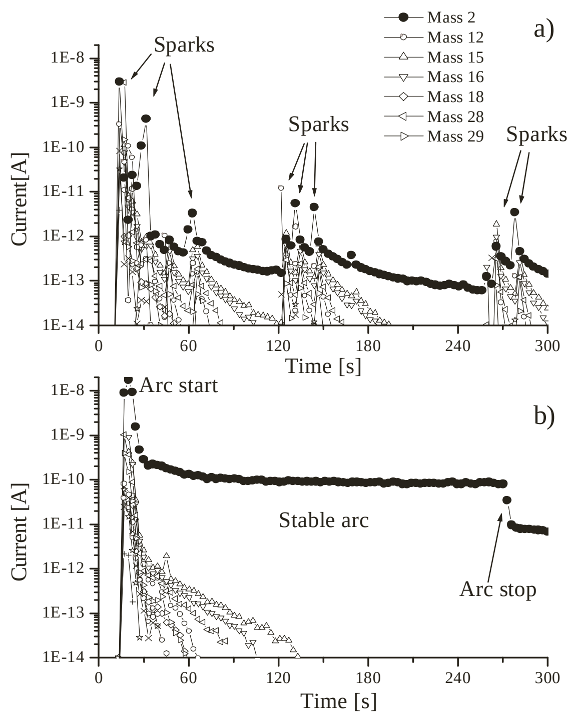

Figure 3: Ion current density versus time for different mass gases. 3a: triggering generates sparks (and outgassing) but arc doesn't start; 3b arc starts with the first spark. All gases, but hydrogen, drop below the detection limit of our instrument after 1-2 minutes of arc operation.

In fact all residual gas partial pressures increase in approximately the same ratio, since the corresponding gas species - in particular hydrocarbides, carbon monoxide and water - are all desorbed from the surface during sparking. Outgassing reaches its maximum when a stable arc current is established. Then, as Nb starts being evaporated from the cathode and the chamber walls become coated with a fresh Nb film, outgassing decreases rapidly.

A typical behavior of partial pressures of residual gas species versus time is shown in Fig. 3. In particular Fig.3a shows the partial pressure rise due to triggering sparks in a situation when the arc discharge does not start. Fig 3b shows the behavior in time of partial pressures when a stable arc current is established. The total pressure increases up to 10^{-6} Torr as soon as the arc discharge starts, and stays almost stable during deposition. Note though that in such conditions the residual gas is almost exclusively hydrogen, its partial pressure being usually more than 3 orders of magnitude higher than that of other contaminants. This excess of hydrogen can be understood as generate by Nb bulk cathode, providing an essentially 'infinite' source of this gas. All other gases are present only on the chamber wall surface which makes their partial pressures drop below the detection limit of our instrument after only a few minutes of operation. It is planned to outgas our next cathodes for several hours at 600^{o}C in UHV condition prior to use, to drastically reduce their hydrogen content.

3.3 Samples Characterization

The substrates used were sapphire (r-plane) and Cu. They were fixed on the holder Cu flange by means of screws and springs and/or with conducting silver paint. The Cu substrates were prepared at CERN using the standard recipe used for chemistry of 1.5 GHz Cu cavities (electropolishing +SUBU) and mounted without any additional treatment. Sapphire substrates were cleaned in an ultrasonic bath, using acetone, alcohol and de-ionized water, and dried in nitrogen. Sapphire substrates are mainly used to perform fast RRR checks using a cryocooler system that can reach a temperature of 12 K. Some results are shown in Fig. 4 where the film resistance normalized to its room temperature value is plotted versus temperature. The top, flat curve corresponds to one of the first samples produced, in which Fe, Ni and Cu impurities were present. After several improvements to the system samples with RRR in the range from 10 to 50 even for film thickness of a few thousand Å only were obtained. The result is very encouraging since it is higher by a factor of 2 to 5 than what usually obtained under similar conditions (same thickness and coating temperature) by magnetron sputtering (RRR usually between 5 and 10). It is likely that our improvement is due to epitaxial growth of the Nb film on the sapphire substrate. This hypothesis is being checked by X-ray diffractometry of the samples.

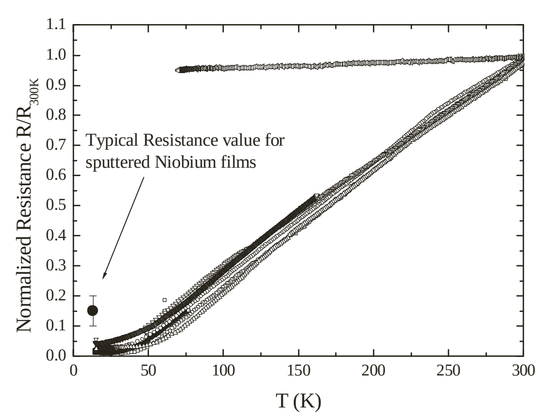

Figure 4: Resistance versus temperature for different samples. The resistance values are normalized to their value at room temperature.

Critical current density (J_c) and T_c of the film were measured on both Cu and sapphire substrates using an inductive method. The sample is placed in contact with a primary coil used to both to send a frequency signal and to detect a response on the signal third harmonic, the latter being only present when the sample is in the transition state. Typical results are shown in Fig.5: T_c is defined as the temperature at the transition step half height (in most cases coincident with the maximum of the curve) and the transition width as the difference in temperature 90% below the curve maximum. It can be seen that results are rather scattered, T_c varying from 8.7K to 9.26K even for samples with high RRR but with the best sample showing values identical to bulk metal. The latter result was obtained with low film thickness (0.1µm) and low deposition temperature (100°C) but could not be so far reproduced on thick samples.

The reason is, we believe, contamination arising from arc breaks occurring when the deposition time gets long, a problem we are working to solve. J_c measurements follow the same pattern, giving values ranging from $2x10^6$A/cm^2 to $3x10^7$/A/cm^2. T_c and J_c measurements are consistent (higher T_c correspond to higher J_c) but in no definite correlation, so far, with RRR or coating conditions. This hints at the presence of metallic contaminants that do not affect resistivity but only the superconducting properties.

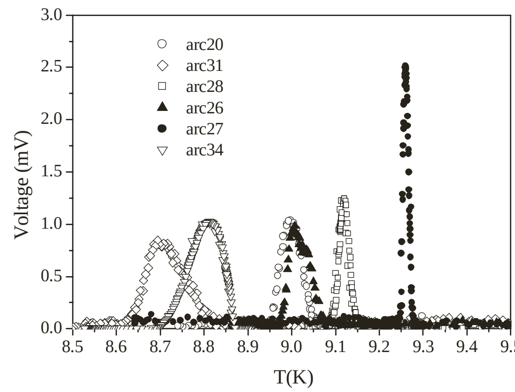

Figure 5: Results of inductive T_c measurements on some Niobium films produced using our planar arc source.

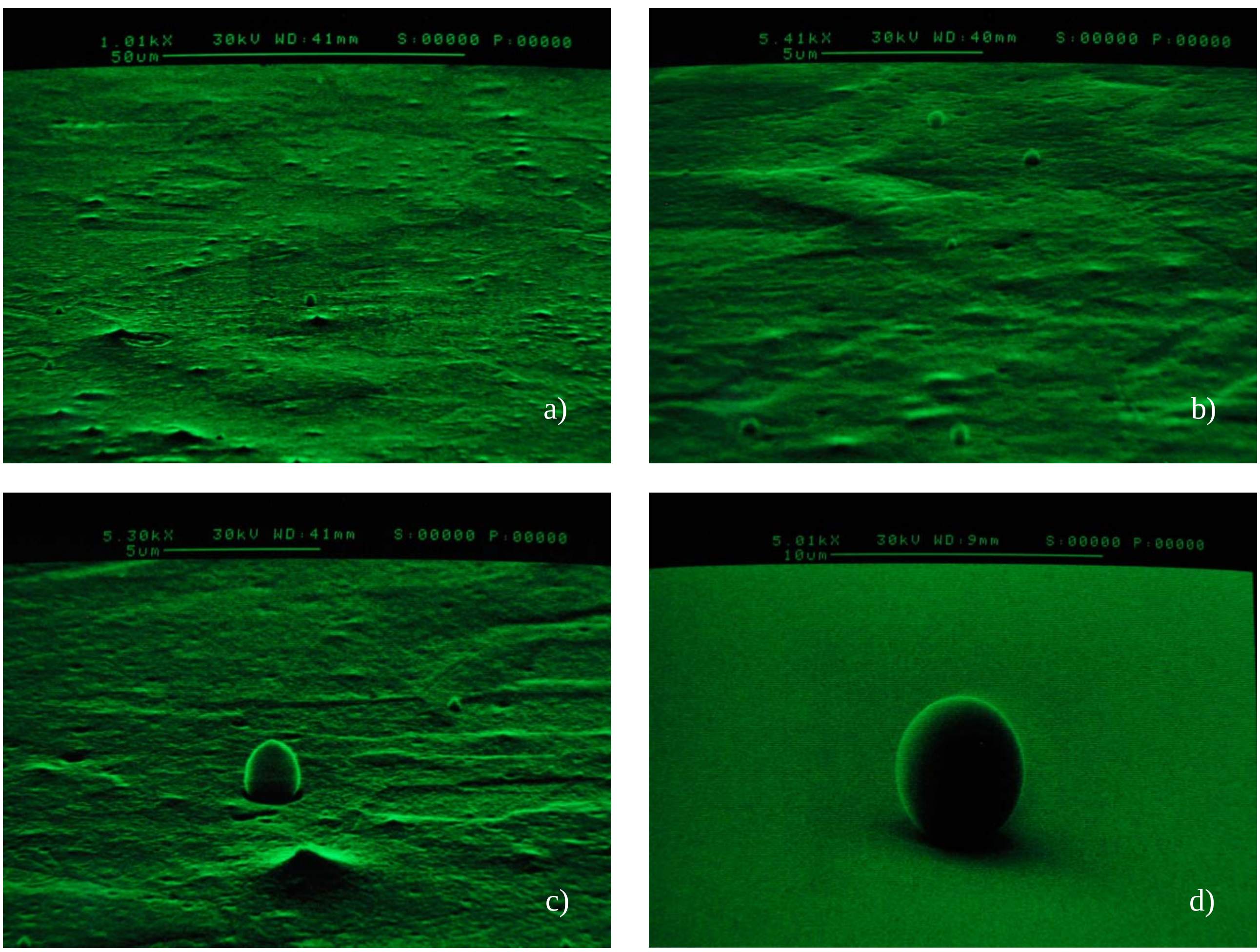

Fig 6: SEM images of the film surface in tilted view (about 45 degrees) a) Niobium on copper, general view b) Small microdroplets on a Nb film deposited on copper c) Details of a microdroplet (about 2microns) and a defect on Nb/Cu surface d) A large microdroplet on a Sapphire surface

3.4 Film surface conditions

The main disadvantage of arc coating is the production of microdroplets that are embedded in the growing film. Microdroplets are made of high purity molten Nb and, while not contaminating the film, increase its surface roughness. The presence of microdroplets in our films was studied by optical and electron microscopy and by roughness measurements. In Fig.6 SEM pictures show a general view of a film on Cu substrate and evidence the presence of microdroplets on its surface. SEM images and roughness measurements indicate a surface roughness similar to that of the Cu substrate: Ra = 0.15μm for the Cu alone and 0.18μm for 1μm Nb film on Cu.

The situation is of course very different when Nb is deposited on sapphire, which has a roughness ≤20Å, not measurable by our instrument. In this case, the film roughness of 0.1μm is mainly due to the presence of microdroplets. SEM images at higher magnification show some spherical microdroplets seemingly just resting on the film surface.

The diameter of the observed spheres ranges from 0.5μm to 5μm, the upper limit being established with good confidence since a defect large than 5μm is easily seen with SEM, while the presence of microdroplets with diameter smaller than the quoted lower limit cannot be excluded. The larger microdroplets on the surface could be easily removed by HPWR or ultrasonic bath. Nb samples on sapphire were also looked at with an optical microscope in transmission. Films were completely opaque just after coating, but after having been passed in an ultrasonic bath for 1 minute several bright spots did appear, corresponding to microdroplets with radius larger than the film thickness having been removed.

4 CONCLUSIONS AND FUTURE PLANS

We designed and realized a planar arc source working in UHV conditions to study the deposition of superconducting Nb films. First results obtained with Nb films deposited on Cu and sapphire substrates using the planar arc source are very promising. Niobium films with "bulk-like" properties (RRR~50, T_c =9.26K, ΔT_c = 0.02K, J_c~$3*10^7$A/cm^2) were produced even for thickness as low as 0.1μm. The roughness of Nb films on Cu substrates is mainly due to substrate preparation, while on sapphire substrates it is mainly due to the presence of microdroplets. The results are encouraging given that they have been obtained with no special macroparticle filtering.

Linear and filtered arc sources to study vacuum arc coating in actual cavity geometry are under development.

5 ACKNOWLEDGEMENT

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