NB COATING OF COPPER CAVITIES BY UHV CATHODIC ARC*

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Abstract

Niobium thin film coated copper RF cavities are an interesting alternative to niobium bulk cavities mainly because copper is cheaper, has higher thermal conductivity and better mechanical workability and stability than niobium. Unfortunately the observed degradation of the sputter-coated cavities quality factor with increasing accelerating voltage prevents their use in future accelerators specified to work at field values higher than 15MV/m. To try and overcome this limitation we have developed an alternative coating technique based on a cathodic arc system working under UHV conditions. Main advantages of this technique compared to standard sputtering are the ionized state of the evaporated material, absence of gases to sustain the discharge, high energy of atoms reaching the substrate surface and possibility to have high deposition rates. Recent results on the characterization of niobium film samples produced by UHV cathodic arc are presented, showing that the technique can produce high quality films under different angle of deposition. The system to demonstrate the deposition of a single cell cavity have been commissioned and first tests will be presented and discussed.

INTRODUCTION

The majority of superconducting RF resonators presently operating are made of bulk niobium[1]. Among pure elements, niobium has the highest critical temperature (9.25K in its bulk form) and the highest thermodynamic critical field H_{th} [2]. Its mechanical properties, even if not excellent, are good enough to allow manufacturing resonating devices with the required accuracy and reliability. However, at high accelerating fields local defects can start dissipating energy thereby causing local heating, which, because of the poor thermal conductivity of niobium may trigger thermal instabilities and eventually make the cavity quench (thermal runaway) [1]. The production of bulk niobium cavities has been the object of intense research and development, particularly in high-energy physics laboratories, and a considerable improvement of their performances during the last decades has been achieved [1]. Thermal stability has been improved by using high purity Nb to improve the cavity thermal conductivity. In recent years considerable progress has also been made towards increasing the maximum rf field of Nb bulk cavities, mainly by a) improving the Nb purification techniques at high temperatures, b) baking-out the cavities to move quenches towards higher fields c) minimising surface defects by raw material inspection methods d) using electropolishing instead of chemical polishing and e) producing Niobium cavities from very large grains material, ultimately from single crystal Nb sheets.

For most applications, where only few, relatively small cavities are needed, bulk niobium is a valid choice but in the case of future accelerators, using a large number of cavities and/or low frequency (below ~500MHz), the cost of bulk niobium of adequate purity (usually RRR > 300) and the fabrication cost, mainly machining and Electron Beam welding, can become an issue.

For such applications it has been recognized and proven, by the pioneering work done at CERN, that resonators built by depositing a thin superconducting layer on a high conductivity, relatively low cost supporting structure does provide much better thermal stability at a lower cost. Copper is an obvious choice for the resonator wall material because it has excellent thermal conductivity, is relatively inexpensive, easy to machine and commercially obtainable in high purity grade. It has been proven at CERN that it can be relatively easily coated with high quality Nb films using the magnetron sputtering technique.

On the other hand, the quality factor Q, of magnetron sputtered Nb/Cu cavities has been shown to decrease with increasing RF field. The best 1.5GHz cavities produced at CERN did reach accelerating fields up to 27 MV/m, but the Q degradation was still present, with Q dropping below 10^{10} at around 15MV/m and reaching $5x10^9$ at around 20MV/m [3].

The reasons for such a behaviour, also clearly observable on a different, much less pronounced scale [4,5] in today's highest performing bulk Nb resonators, has not been satisfactorily explained so far by the

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several models presented since. To have new insight in the phenomenon we proposed in the early 2000's, we proposed an alternative coating technique namely the arc coating in ultra-high vacuum (UHVCA) [6-8] to try and produce ultra-pure, more bulk-like Nb films. The main features of UHVCA are the absence of the auxiliary gas needed for sputtering and the absence in the plasma of neutral atoms of the material to be deposited, the plasma being fully ionised. It is easy to apply bias on the substrate and the film properties can be modified by changing the frequency and duty cycle of a pulsed bias. Details of the arc source and coating apparatus can be found elsewhere[8].

In this paper we report some recent results on the properties of films deposited by UHVCA on sample substrates at different bias values and cathode-to-substrate angles. We will also present and discuss the system to deposit a copper cavity using the UHVCA.

FILM STRUCTURAL PROPERTIES

The structure of films deposited on substrates may significantly differ from that of bulk material. Films have usually smaller grains, more defects and are more stressed than the corresponding bulk material. To investigate these structural properties the best technique is X-Ray diffraction, which is based on the reflection of an X-ray beam by the material crystallographic planes, according to the well known Bragg relation.

The position of the maximum of the reflected radiation line determines the value of the interplane distance, the line width and shape contain information on grain size and microstrain whereas its intensity is related to the presence of texture.

Typical results for the lattice constant of UHVCA Nb films on copper and sapphire substrates are in the range in the range of 0.3300nm to 0.3315nm. These values, compared to the lattice constant of bulk niobium (0.3303(6)nm) indicate that the lattice constant in UHVCA coated films is less distorted than in sputtered ones which present a lattice constant in the range 0.3315nm to 0.3330nm.

The lattice distortion is responsible for the critical temperature increase in the sputtered film[10] and disappears when the copper substrate is chemically dissolved and the Nb film lattice parameter approach the bulk value [11].

Information on the average micro-strain in films is also obtained from XRD measurements. In particular, spectra obtained at grazing incidence provide more information than those in the Bragg-Brentano configuration, since in the latter configuration the peaks diffracted at large angles (if present) are noisier due to the finite film thickness. Quantifying the microstrain requires (provided the shape of the peak is sufficiently well defined) evaluating the 2θ value of the peak of each line and the line width, β conventionally defined as its FWHM value expressed in radians. The data can then be arranged as a Williamson-Hall plot, which in principle enables to separate the contributions to peak broadening due to microstrains and to finite grain size respectively [12].

A linear fit of data from Nb films grown by UHVCA gives an intercept on the Y axis at about $0.005nm^{-1}$ so that, the grain size *D* can be estimated to be about 200nm [13], in agreement with the AFM results. For the sputtered films the intercept on the y axis is too close to zero to extract the grain size. For more details on film analysis see ref. [13]).

FILM MORPHOLOGICAL PROPERTIES

The film morphology is studied mainly by SEM (Secondary Emission Microscopy), but also by AFM (Atomic Force Microscopy), optical microscopy and roughness measurements. Note that the roughness and large scale morphology of the films are largely determined by the roughness of the copper substrate since the film mainly follows it.

The main problem, of morphological nature, with the UHVCA technique is the fact that "macroparticles" (MP) of the material to be deposited are explosively emitted from the arc cathode spot in the form of liquid droplets during the arc discharge [14] and deposited on the film after having (mostly) cooled-down in flight thus affecting the film roughness and possibly its density and its electrical properties. The number and size distribution of Nb macroparticels have been studied in the case of Nb [15,16]: it is found that the number depends on the arc parameter and on the particular geometry used, both when using a planar cathode [8] and a cylindrical one[16].

It is not clear at the moment, for lack of data, whether residual MPs will limit the RF cavity voltage performance, due to either field emission, RF field enhancement and/or creation of voids in the film. Recent results obtained at Wuppertal University indicate that pure Nb spherical macroparticles resting on the film surface should not be expected to field-emit at least up to RF fields of ~40MV/m [17] but other effects, such as that of voids left in the film (due to MP shadows) on the RF cavity response, still need further investigation. For these reasons several magnetic macroparticle filters have been developed and commissioned in Rome and in Swierck to remove most of the MPs from the plasma before they reach the surface to be coated. Results on filtered coated samples can be found in [8,9].

While the first cavity coating apparatus in Rome is unfiltered in order to keep it as simple as possible, magnetic filtered apparata will be assembled and used as soon as first RF tests will provide the data on the coated cell performance.

TRANSPORT AND SUPERCONDUCTING FILM PROPERTIES

In many laboratories a first estimate of the film purity is obtained based on measurement of the transport and superconducting properties of niobium films since it is well know that impurities act as electron scattering centres thereby significantly increasing the film resistivity [18]

RRR values for Nb films deposited on glassy quartz and on copper substrates by magnetron sputtering, at a substrate temperature of 150C, typically range from 5 to 30, depending on the discharge gas used and the voltage applied to the cathode [19].

The RRR of Nb films deposited at temperatures below 100C by UHVCA instead stays consistently in the 20 - 50 range [7,8], in agreement with what one expects, given that there are no auxiliary gases present to poison the film.

Another important information on the Nb film quality is given by the superconducting critical temperature value, T_c. In Nb films, T_c is very sensitive to impurity and stresses; in fact small amounts of impurities can lower the film T_c whereas compressive stresses can raise it, up to ~9.6K [11,21], compared to the bulk value of 9.26K. The superconducting transition temperature of magnetron sputtered Nb films is reported to be 9.5K-9.6K due to the compressive stress present in the film[10].

T_c inductive measurements on UHVCA Nb films deposited in different conditions have been reported elsewhere [7,8] to be in the 9.21K to 9.28K range, with ΔT_c less than 0.02K. This proves that high quality films, with properties close to the bulk ones can be consistently obtained with the UHVCA technique.

INFLUENCE OF THE DEPOSITION ANGLE

In the case of sputtered films, if the incidence angle of ions (or atoms) on the substrate is not normal to the surface, irregularities of the substrate may produce shadows and therefore film inhomogeneities. In fact, at low incident angles the sputtered film structure changes and the roughness increases [22,23]. Results obtained from measurements of the permeability of niobium sputtered films to helium gas show a significant increase of the film porosity with decreasing incidence angle. The fraction of surface permeable to He gas, in the case of sputtered (CERN type) SC cavities has been shown to increases from ≅4.4 ppm at the equator, where incidence is close to normal, to ≅ 25 ppm at the iris [3] where the incidence angle is much closer to grazing.

Using UHVCA has therefore the advantage that, because the deposition is via ions, by negatively biasing the substrate the ions incidence angle can be made almost normal to the substrate. Films produced by cathodic arc deposition have accordingly consistently shown low roughness, almost independently from the cathode-to-substrate angle [23].

We have deposited several samples using the planar arc system, without using the magnetic macroparticle filter described in [9]. The transport and the superconducting properties of such films, deposited at room temperature on sapphire negatively biased at -40V (constant), are also almost independent from the incidence angle up to 60 degree with RRR values from 30 to 40, decreasing to 18 and 13 at 75 and 90 degrees respectively.

FEG-SEM images of UHVCA deposited Nb films on negatively biased substrates oriented at different angles with respect to the cathode show aflat surface up to 60 degrees. Some small defects start to appear at 75 degrees incidence and become more evident close to 90° [24,25]. The deposition angle does influence the deposition rate, however even at 90° the deposition rate is higher than 100 nm/min for all investigated bias values, which allows depositing thick films in a relatively short time [24,25].

INFLUENCE OF THE VOLTAGE BIAS

RRR and X-ray results on samples, are summarized as a function of DC bias in Table I showing that the RRR is not strongly influenced by biases ranging from 26 to 50 and that the lattice parameter *a* is quite close to the Nb bulk value (0.3306) in the same range.

Table I Summary of the results obtained at negative constant bias

Bias (V)	Film Thickness (μm)	RRR	a_{Nb} (nm)
-23	0.9-2.8	26	0.3308
-40	0.9-2.6	40	0.3312
-60	1.0-1.7	30	0.3313
-80	0.7-1.0	50	0.3310

The effect of pulsed bias on Nb films is also under investigation. First tests indicate that applying a pulsed bias better morphological and structural properties of Nb films can be obtained: pulsed bias deposited Nb films have larger grains (up to microns) with less defects and flat surface as seen in figure 1 showing a 1.4μm thick Nb film UHVA deposited on a sapphire substrate with a -60V bias, pulsed at 10KHz with a 50% duty cycle.

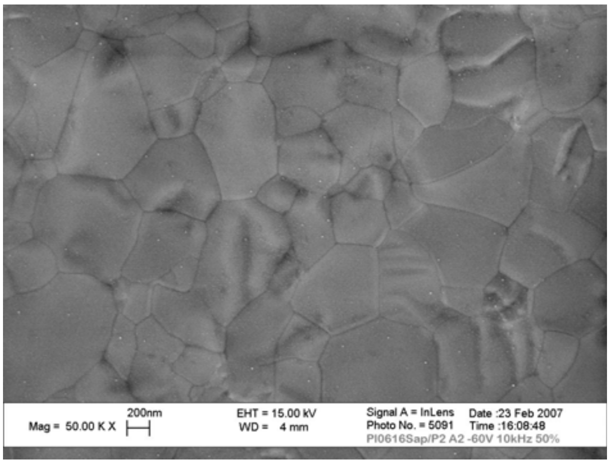

Figure 1 FEG-SEM image of a Nb film deposited on a sapphire substrate using the pulsed bias.

X-ray spectra on the same film show that grains are random oriented, i.e. that no epitaxy between film and substrate is present and that not only grain size is increased but also that the strain in the grain is reduced to the point that, in some cases, it is even possible to resolve the Cu-$K_{\alpha 1}$, Cu-$K_{\alpha 2}$ doublet of the X-rays source. In pulsed bias operation the Nb deposition rate is higher than with constant bias since during the bias-off phase it is bombarded by electrons which, unlike energetic ions, do not produce back-sputtering of the deposited film. Moreover the high electron current does heat the substrate to relatively high temperature. Adhesion of Nb films as thick as 40µm to the substrate also confirms the superior adhesion and lower stress properties of UHV coating compared to magnetron sputtered ones.

CAVITY COATING SYSTEM

The present , unfiltered apparatus to study the coating of single cell RF cavities, a schematic view of which is shown in Fig.8, consists of a cathode plus anode assembly(A) connected to a ceramic insulator (B) on top of which sits the cavity to be coated (C). The other side of the cavity is connected to a second CF100 ceramic insulator (D) and, by a standard CF-100 flanged T-junction piece (F), to a 180 l/s turbo-pump unit.

Several dc-current fed coils (in air), distributed along the system (Fig.2) generate the magnetic field guiding the plasma. The lowest coil surrounds the cone-shaped Nb cathode to stabilize the position of the cathode hot spots. Other identical coils (26.5 mm high, with 350 turns each and inner diameter of 226 mm) provide the uniform magnetic field to magnetize the electrons and transport the plasma to the cavity cell. A special stainless steel cavity, flanged at the equator, has been used to study the plasma transport in to the cavity cell. This cavity allow to easily place samples in his interior and to control deposition rate and film quality, also the low thermal conductivity of stainless steel allow to control the plasma position by monitoring the temperature using an infrared camera. A third cylindrical insulator mounted on top of the cavity supports a flange (G) equipped with low-voltage feedthrough pins to one of which an insulated, horizontal, 7 cm diam stainless steel disc suspended inside the vacuum chamber above the cavity (E) is connected. The disc, is, serves as a plasma ions collector, to optimise the plasma transport into the cavity cell when the slant and exit coils are switched off.

The first tests performed in this configuration has shown that only the upper half of the cavity and the lower cut-off pipe could be satisfactorily coated can be coated. This implies that two sources, one from each side of the cavity, are needed to coat the cavity in a single run without opening it to air.

A series of tests have been performed in order to optimize the plasma transmission and to optimize film thickness by varying the coil positions and magnetic field configuration. The coil arrangement of figure 2 has proven more efficient than the cusp geometry previously reported [26], allowing to transport several amperes of ion currents into the cavity and to adjust the plasma beam position at the cavity surface by adjusting the current in the slant and exit coils.

More work is needed to optimise the deposition rate and the niobium film quality. However a first copper cavity has been coated using the system illustrated in figure 2. The cavity, produced and chemically cleaned at CEA-Saclay, was sent to Rome under Ar pressure. After bakeout its upper half-cell and lower cut-off pipe were coated first; the estimated Nb film thickness ranged from ~2 to ~5µm. The system was than vented to dry nitrogen, and the cavity turned upside down, baked-out and evacuated to coat the other half-cell and cut-off pipe. This procedure is clearly not optimal since contamination can occur during venting, turning over and pumping. After deposition the cavity was sent back to CEA-Saclay to be high pressure water rinsed (HPWR) and RF tested. Unfortunately during the HPWR portions of the Nb film deposited on the last coated half-cell and cut-off did peel-off, whereas film deposited during the first coating run did not. This result suggests that adhesion problem originated from the exposure to air after the first deposition and that the deposition needs to be made in a single run. In addition, since macroparticles present near the cavity iris can cause field emission, the final apparatus should also include filtering. Macroparticle filter and such a second, macroparticle-filtered apparatus, schematically shown in Fig.3, has been designed and is being assembled.

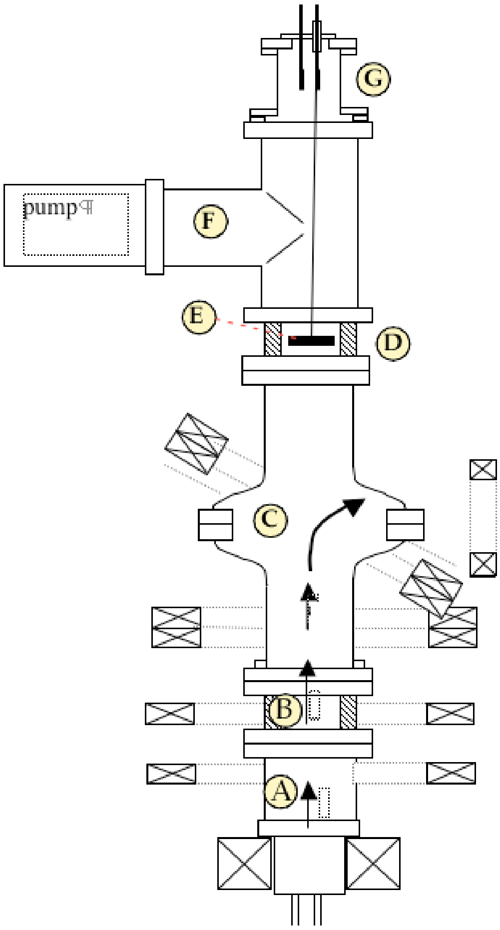

Figure 2 Magnetic configuration for cavity coating. The uppermost coil can rotate around the cavity axis to produce a uniform coating in the upper cell.

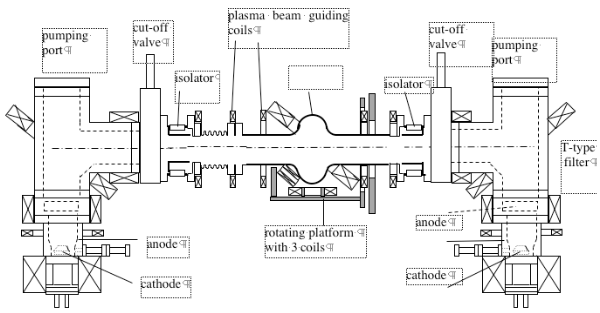

Figure 3 Schematic drawing of the system equipped with two filtered arc sources to deposit the cavity in a single run

CONCLUSION

A comparison has been made between properties of niobium films obtained by magnetron sputtering and by biased cathodic arc deposition in ultra high vacuum (UHVCA). UHVCA-deposited Nb films have been shown to have structural and transport properties closer to the bulk ones, thus providing a promising alternative for niobium coated, high voltage, high Q, copper RF cavities. Several sample films have been deposited with different biases and ion incidence angles; while a more complete characterization of the samples is in progress, it has been shown that there is no strong dependence of the film RRR from the bias potential and from the ion deposition angle up to 60 degrees incidence and that even at 90 degrees incidence the film RRR is higher than 10.

Several properties of the Nb films UHVCA deposited using negative bias pulsed at a proper frequency and duty cycle, such as grain size, stress and strain, have been shown to be superior to those routinely obtained by magnetron sputtering. A system to study the coating of single cell, TESLA type RF cavities, has been shown capable of transporting large ion currents into the cell using proper focusing magnetic field configurations and a first copper cavity has been coated, thus providing the UHVCA principle. A deposition system equipped with two arc sources is currently under construction to deposit the cavity in a single run and will be operation in the early 2008.

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