NB COATING DEVELOPMENTS WITH HIPIMS FOR SRF APPLICATIONS

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Abstract

In the last few years the interest of the thin film science and technology community on High Impulse Power Magnetron Sputtering (HIPIMS) coatings has steadily increased. HIPIMS literature shows that better thin film morphology, denser and smoother films can be achieved when compared with standard dc Magnetron Sputtering (dcMS) coating technology [1]. Furthermore the capability of HIPIMS to produce a high quantity of ionized species [2,3] can allow conformal coatings also for complex geometries. A study is under way at CERN to apply this technology for the Nb coating of SRF 1.3-1.5 GHz Cu cavities, and in parallel at SHU the plasma physics and its correlation with film morphology are being investigated [2]. Recent results achieved with this technique are presented in the paper.

INTRODUCTION

DC magnetron sputtering (dcMS) has been extensively used for producing thin film Nb/Cu RF cavities for particle accelerators such as LEP and LHC, and in future HIE-ISOLDE, at CERN. Surface resistance at high field is however trailing that of state of the art bulk Nb cavities, behaviour often attributed to defects inherent in the dcMS process itself. In the last years the community started to focus on the High Impulse Magnetron Sputtering (HIPIMS). This recent deposition technique, given its properties such as the high target ions content in the plasma, allows achieving a denser and smoother film if compared with the dcMS ones. The main aim of this paper is to show the first results achieved at CERN in studying HIPIMS coated cavities, and the correlation obtained studying different working condition with the morphology of the film and with the plasma composition.

EXPERIMENTAL SETUP

Coating Systems

The coating system used at CERN is a vertical cylindrical magnetron configuration, the same used for the usual dcMS coatings, in which the vacuum chamber is the cavity itself, and is shown schematically in

Figure 1. The whole system is all-metal UHV and baked for 24 hours out prior to coating in order to reach base pressures in the 10^{-10} mbar range. The central stainless steel tube supports one central and two peripherals niobium targets and allows the insertion of a SmCo magnet which can be moved up and down in order to create the plasma in the most suitable region of the target, and guarantee uniform coating. The magnet is air cooled. The peripheral targets are insulated from the central tube, such that it is possible to either bias or to keep them grounded while the central target is kept at high voltage for sputtering. The magnet, with a tangential component of the magnetic field equal to 100 mT on the magnet surface, giving a value of around 50/60 mT on the target surface, confines the plasma in a specific region about 5 cm in length. The cavity cell (central part of the cavity) is the most critical region for accelerator performance. It faces the central cathode and is coated in one single coating run, keeping the plasma in the central cathode region. The cavity cut-off tubes are instead coated in five different deposition steps in order to obtain a better coating uniformity. The cut-offs were coated by dcMS, while the cell was coated either by HIPIMS or by dcMS for comparison.

The coatings were obtained in a Kr atmosphere at 4.3×10^{-3} mbar.

Figure 2 represents the typical current and voltage waveforms of a HIPIMS discharge. The power supply used at CERN is a Hüttinger Electronics TruPlasma Highpulse DC Unit, capable of 10 kW max average power, and voltage up to 2 kV, with pulse width up to 200 µs and frequency up to 500 Hz.

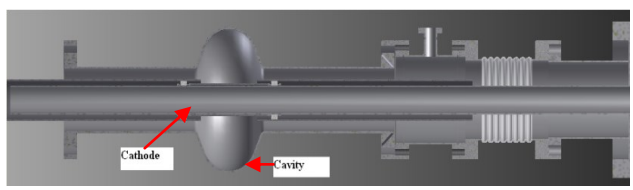

Figure 1: System used at CERN.

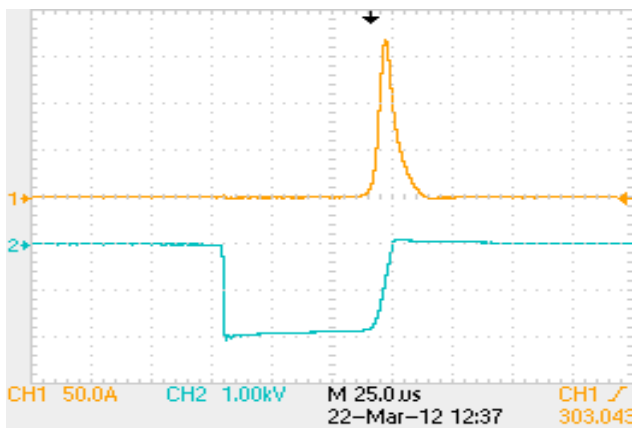

Figure 2: Typical HiPIMS current and voltage behaviour.

The cavity analyzed in this paper was coated with a peak current of 200 A (current density of 2 A/cm^2), a potential of 570 ± 10 V, an average current of 2.6 A with a pulse width of 200 µs and a frequency of 106 Hz. Motivation for this parameter choice is given in [2].

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Coupons were placed in the central part of a stainless steel dummy cavity, in order to replicate as well as possible coating conditions on a real copper cavity. For every coating test two samples were coated. Copper samples were used for Scanning Electron Microscope (SEM) analysis to get information on the morphology, the texture and the grain size of the niobium film deposited, as well as for cross-checking the thickness of the deposits with measurements by X-Ray Fluorescence (XRF). Quartz samples were used for RRR measurement. For all these analyses the reader is referred to [2].

RF Measurements

During the RF measurements, cavities are helium bath cooled in a vertical cryostat, with temperature sensors attached directly on top and on the bottom of the cell using kapton tape and cable binders. It was found that that the temperature gradient between the upper and lower part is only about 0.2 K at the moment of transition. The quality factor is measured at 4.2 K and 2 K, reached by pumping on the helium bath, see Figure 3**Erreur ! Source du renvoi introuvable.**. It should be noted that during measurement of the HIPIMS cavity at 4.2 K the pressure was in the 10^{-7} mbar range, rising up to the 10^{-5} mbar range in super-fluid He.

Above 11 MV/m there was radiation up to 200 uS/h measured at the bottom of the cavity. The interlock system prevented measurements at higher fields. A quality factor of $6x10^9$ was obtained at low field and 2 K, corresponding to a surface resistance of 43 nΩ. The BCS resistance at 2 K and 1.3 GHz is about 8 nΩ. The obtained residual resistance is therefore about 35 nΩ. Several dcMS cavities have been produced and tested as well. The best performing one had weaker performance compared to the best HIPIMS cavity, and is also shown in Figure 3. This cavity has a resonant frequency of 1.5 GHz and for direct comparison with the 1.3 GHz HIPIMS cavity its quality factor was quadratically scaled. Helium processing with 150 W (11 MV/m) was performed for about 1 h at 4.2 K with no effect on the Q-value.

So far all results obtained concerning residual resistance and Q-slope remain however below the best values obtained with standard dcMS coatings reported in literature [4], thus indicating that some tune-up is still needed in the whole production chain.

During cool down of the HIPIMS cavity to 2 K the surface resistance as a function of temperature was measured. The data can be used to derive the superconducting energy gap Δ/k_B, see. The value of 1.51 lies below literature values. The reason might be that the measurements were not performed at constant field, but constant input power. Also the field level was rather high E_{acc}=2.5-3 MV/m, where a Q-slope is already observed.

The resonant frequency was measured as a function of temperature. A new procedure for this measurement was applied.

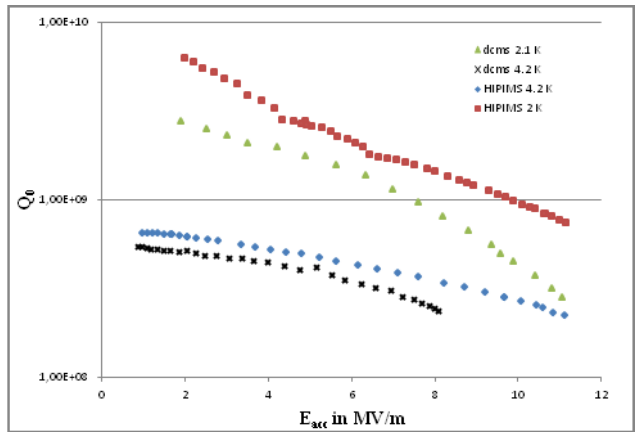

Figure 3: Quality factor of two elliptical single cell cavities, produced by dcMS and HIPIMS sputtering.

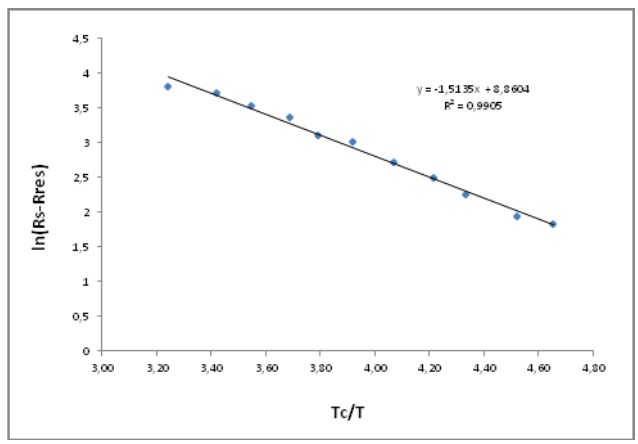

Figure 4: Surface resistance as a function of temperature for the HIPIMS cavity produced at CERN.

The measurement was performed in the following way. First the helium was evaporated until only a few litres were left in the cryostat. Then the cryostat was pumped down to 1 mbar. During the consequent warm up f vs T was measured.

The resonance frequency shift is related to the change in penetration depth in the superconducting state and was fitted (Figure 5) with the Gorter-Casimir temperature dependence [5].

$$\Delta\lambda = \lambda_0 \cdot \frac{1}{\sqrt{1-\left(\frac{T}{T_c}\right)^4}} - \lambda(T_0)$$

Where λ_0 is the penetration depth at zero Kelvin, correlated to the mean free path l, the London penetration depth λ_L, and the BCS coherence length ξ via

$$\lambda_0 = \lambda_L \sqrt{1+\frac{\pi\xi}{2l}}$$

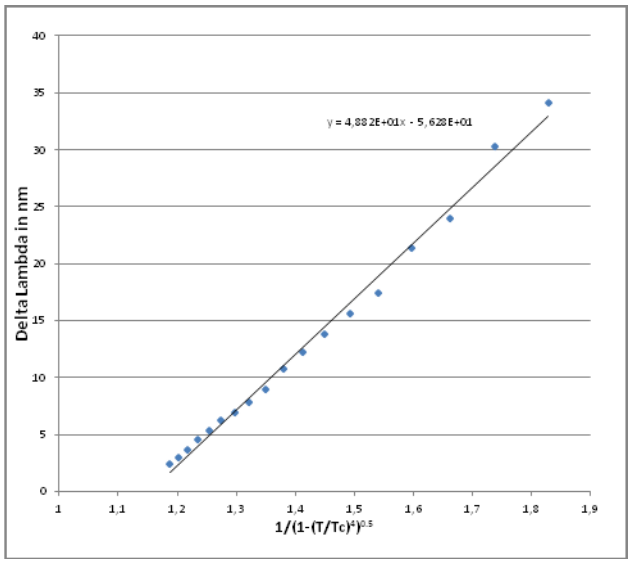

Figure 5: Change in penetration depth as a function of temperature.

The fit allows to extrapolate the penetration depth at 0K, which can in turn be related to the mean free path and therefore to the RRR of the film using (RRR-1)=2.7 l (nm). For the input parameters λ_L=32 nm and ξ_0=39 nm were chosen.

The best fit parameters are listed in the table below.

Table 1: Best fit parameters

Tc (K)	λ_0 (nm)	l (nm)	RRR
9.4	50	42.5	16.7

XRD Analysis

In order to have a deeper understanding of the texture of the thin film produced XRD (X-Ray Diffraction) analysis has been done using the grazing incidence angle technique. The two samples examined were produced with a peak current value of 110 and 160 A respectively (peak current density of 1.1 and 1.6 A/cm^2). The analysis of the XRD spectra is shown in Figure 6.

As we can see from the image it's clear how the orientation of the grains is modified by the peak current value. The peak of the substrate, the Cu <111>, is getting more and more relevant compared to the others, indicating a reducing thickness of the films as result of increasing peak current. This could be partially attributed to a compacting effect on the film. It's also quite clear that the presence of Nb <211> and Nb <220> is getting less and less relevant with respect to Nb <110> and leading to a more ordered texture film.

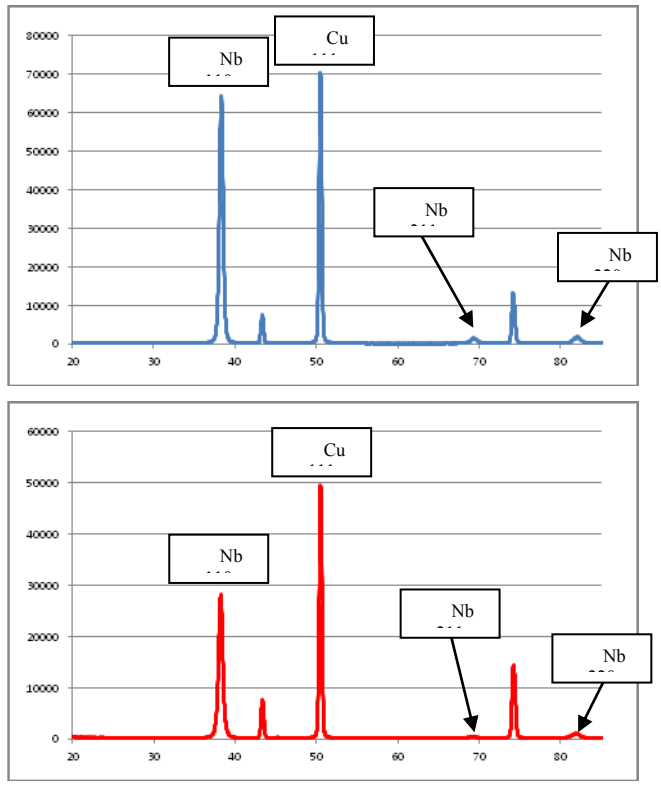

Figure 6: XRD spectra. Sample 1 (110 A) is shown in blue while sample 2 (160 A) is shown in red.

SEM Analysis

Figure 7 shows SEM micrographs of the surface of samples produced at CERN in cavity geometry. The samples were produced by a baseline dcMS process and three HIPIMS processes at various current densities (from 0.5 to 2 A/cm^2).

The samples were produced without biasing voltage. It can be seen that the morphology of the film for the low current case is more "disordered" and closer to the dcMS case. For the high current case the morphology seems to be much more ordered, following a preferential disposition. Furthermore, increasing the current density, it was possible to achieve a smoother surface and a bigger grain size as shown by the SEM measurements.

The HIPIMS samples also showed an improved adhesion. This became apparent when bending the samples in order to create a crack in the film for cross-sectional SEM analysis, the dcMS samples lifted out from the substrate. With the samples produced with HIPIMS that lift out did not occur independently of the coating parameters used indicating qualitatively stronger adhesion.

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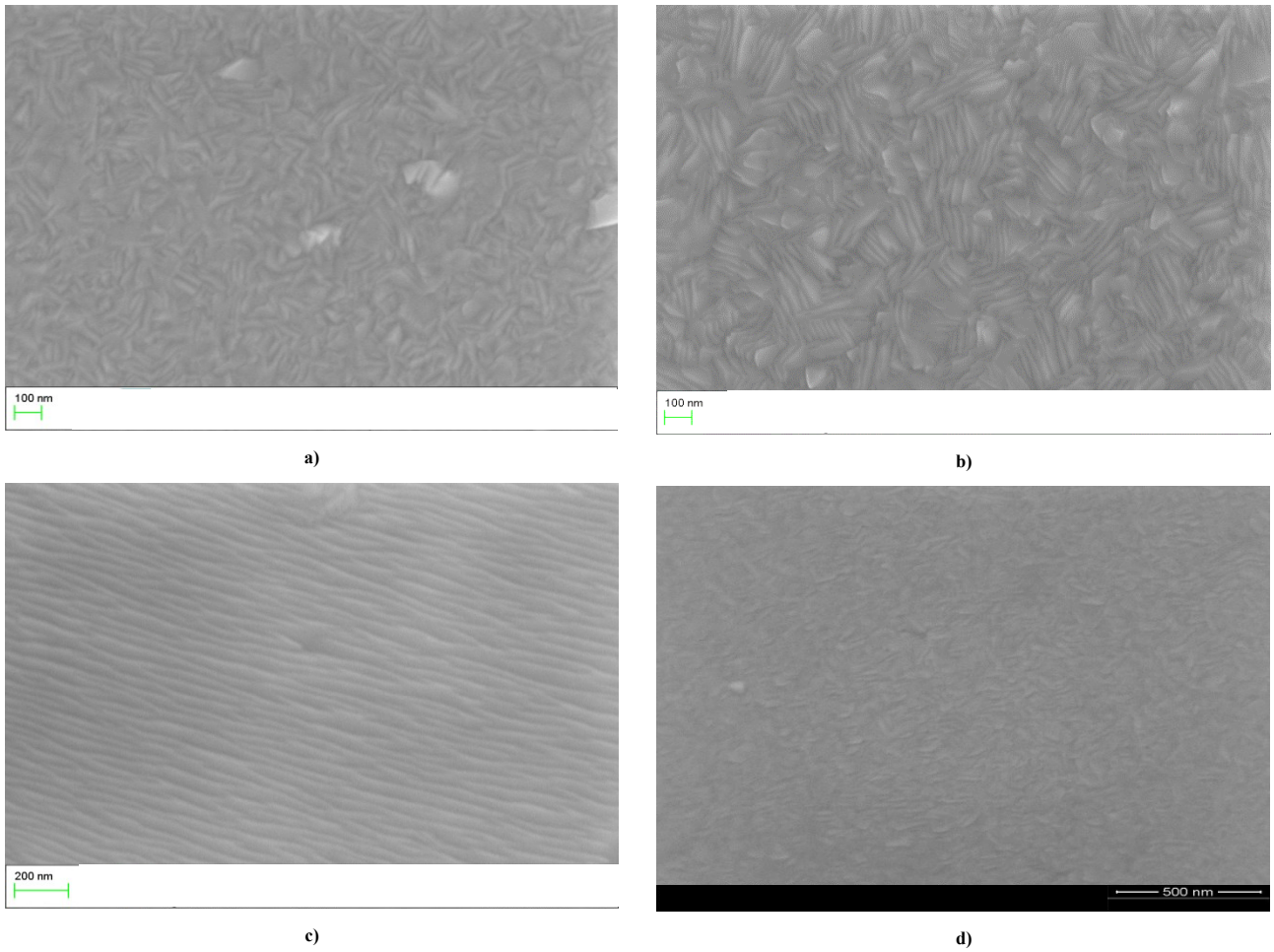

Figure 7: Clockwise from left: dcMs sample (a), HIPIMS sample produced with 50 A peak current (b), HIPIMS sample produced with 150 A peak current (c), HIPIMS sample produced with 200 A peak current (d).

CONCLUSIONS

The preliminary study described in this paper has shown that an HIPIMS coating in an accelerating cavity can perform as a dcMS coated cavity. This can be given by the improved morphology of the coating (density, smoothness) and with a further investigation the limits reached with this tests might eventually be overcome. HIPIMS is confirmed being an interesting and promising technology to enhance performance of the thin film coated cavity.

It has been shown also that deposition with HIPIMS will improve adhesion and allows the possibility of creating coatings with a preferred orientation. Furthermore, given the energy imparted to the Nb ions [2], it is possible to achieve higher density structured film, with the aim of increasing the superconductive properties of the film itself.

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