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DOUBLE CATHODE CONFIGURATION FOR THE Nb COATING OF HIE-ISOLDE CAVITIES*

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Abstract

The Quarter Wave Resonator (QWR) cavities for HIE-ISOLDE project at CERN have entered their ending phase of production. Some R&D is still required to improve the uniformity of the Nb layer thickness on the cavity surface. In order to improve this behaviour one approach which has been proposed is to replace the single cathode with a double cathode and test the suitability of different deposition techniques. With this change it is possible to control the plasma and power distribution separately for the inner and outer part of cavity and thereby potentially improve film uniformity throughout the cavity and coating duration. In this study a comparison between the deposition rates obtained using a single cathode and a double cathode using Direct Current (DC)-bias diode sputtering, DC-magnetron sputtering (DCMS) and Pulsed DC-magnetron sputtering (PDCMS) is presented. The morphology of the thin film samples were compared using Focused Ion Beam (FIB) cross section milling and Scanning Electron Microscopy (SEM) analysis.

INTRODUCTION

The technology of Nb sputtering on copper cavities was developed in early 1980's at CERN [1]. This technology was selected for QWR cavities for HIE-ISOLDE project in 2007 [2] while the work for establishing the setup and method for the series production was started in 2008 [3]. After passing this R&D stage, the first cryomodule cavities were produced between 2013 and 2014 [4]. With the completion of the fourth cryomodule [5] the project has reached its final stage, in which five spare cavities are being produced. Despite reaching its final production stage, R&D on QWR coatings continues in parallel, as advances in RF performance are still possible.

Coating layer thickness uniformity remains one of the aspects which requires further improvement. For example, current baseline cavities are known to show discrepancies between the inner and outer conductor thickness profiles [6]. Typically the minimum thickness required for a coating is defined by the penetration depth of the RF field (~ 40 nm) and must be thick enough to minimize losses with respect to material quality (dense layer, defect and contaminant free) and substrate influence. In practice we target to a thickness of tens of penetration depths with an additional safety margin resulting to a minimum 1 µm film. The maximum thickness is primarily limited by the residual stress induced in the film [7] and the associated adhesion issues between the film and the copper surface. It mostly depends on the coating method and surface preparation and can be validated by high pressure rinsing test up to 100 bars.

In order to achieve the desired cavity performances for the HIE-ISOLDE project [8] with the current baseline process we have to tune the outer conductor minimum thickness by increasing the overall coating time and consequently increasing the inner conductor thickness (up to 4 times larger [6]) and potentially the amount of impurities in the film.

To facilitate improved control of the coating process, and thereby ensure a more optimal coating thickness and deposition time, a new double cathode system has been designed. The increased flexibility of this two component system facilitates independent optimization of the inner and outer conductors coating rates and helps in reducing the amount of impurities.

In this paper the double cathode design will be introduced and comparisons will be drawn between the deposition rates obtained with double cathode and those obtained using conventional single cathode with different coating techniques. For each technique, the film morphology at different locations within the cavity will also be presented and discussed, along with the limitations of DC-magnetron sputtering technique for this geometry.

EXPERIMENTAL SETUP

Double Cathode Scheme

In this scheme two cathodes are used instead of a single one as used in production baseline process. Fig. 1 shows the comparison of the single and double cathode schemes.

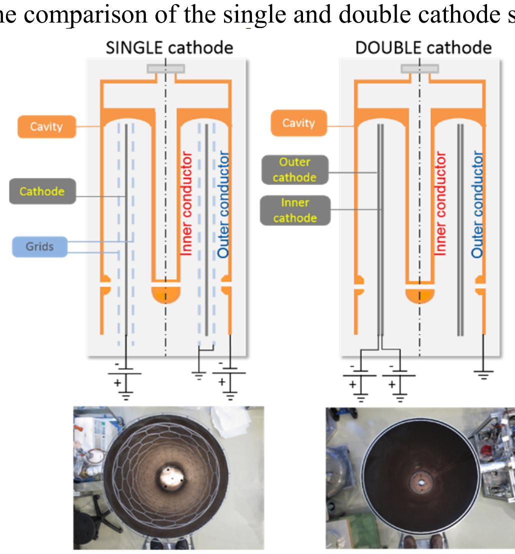

Figure 1: (a) single cathode scheme for DC-bias diode baseline, (b) double cathode scheme.

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Table 1: Parameters Used for the Different Coating Techniques

Technique	Cathode scheme	Pressure [mbar]	Magnetic field[G]	Cathode(s) power [kW]	Duration [min]	Energy [kWh]
Diode	Single	$2x10^{-1}$	none	8 (5.4 in, 2.6 out)	345	43 (29 in, 14 out)
DCMS	Single	1.3×10^{-2}	95	2 (1.6 in, 0.4 out)	421	14 (11.2 in, 2.8 out)
DCMS	Double	1.3×10^{-2}	111	2.2 in, 3.5 out	158	5.8 in, 9 out
PDCMS	Double	1.6×10^{-2}	118	2 in, 4.5 out	145	4.4 in, 9.8 out

The distance between cathodes has been set to 2 mm in order to keep $p\times d$ (pressure x gap between electrodes) small enough [9] to avoid parasitic plasma between the cathodes. Each cathode is controlled with a separate power supply which gives the advantage of independent control of plasma and power distribution for inner and outer conductors in this scheme and as a consequence the resulting local coating rate.

Coating Techniques

Double cathode scheme is adapted for DC-magnetron sputtering (DCMS) and Pulsed DC-magnetron sputtering (PDCMS) techniques. The results will be compared with single cathode DCMS [10] and DC-bias diode baseline [8] taken as reference.

DC-bias diode process with double cathode will not be discussed in this study as the current grid setup is not completely compatible with the double cathode design.

DCMS regime can operate at lower pressure than diode thanks to the magnetic confinement of electrons. This allows higher coating rate with larger transmission of atoms to the substrate. The higher coating rate and low pressure regime will reduce the amount of impurities in the layer. Due to the larger transmission (larger mean free path/less collisions) argon ions and niobium atoms have a larger energy with respect to the diode process, impacting the way the film grows [11].

Pulsed DCMS is usually of interest for dielectric coating and to avoid target poisoning. In our application the pulsed power supply has a reverse pulsed feature to quench the plasma. This feature could be used to repel Ar ions towards the substrate and potentially densify the growing Nb layer, having a similar effect to a bias. Pulse frequency has been set to 60 kHz to obtain the longest reverse pulse possible of 5.4 µs.

Table 1 summarizes the parameters used with the different coating techniques and cathode schemes adopted so far.

All the coatings have been performed at high temperature to get better film properties as discussed in [6]. For the DCMS coatings, plasma instabilities on the outer conductor side were observed and mitigated by increasing the process pressure. Therefore the process pressure is 20% higher in the case of PDCMS.

The double cathode enables to access the distribution of power on inner and outer side of the single cathode in diode and DCMS regime. For this purpose the double cathode has been run as single cathode by connecting both cathodes together and measuring the current flowing in each of them separately. The measured values are mentioned in Table 1.

From this measurement we can observe that power is not evenly distributed between inner and outer part. This is expectable because of the difference in geometry and surface aspect ratio between the cathode and substrate, inducing a different plasma impedance and thus power distribution.

In the production baseline DC-bias diode configuration a total equivalent energy of 43 kWh for the coating is applied. This corresponds to about 14 kWh with the single cathode DCMS process in order to achieve the minimum 1 µm thickness. Therefore14 kWh was the target value for the double cathode magnetron processes distributed among the two cathodes. The power distribution between inner and outer part allows as well to calculate the distribution of energy between the two sides of the cathode. For the double cathode the first approximations have been estimated to balance the coating rate inside and outside using these power/energy distributions. Furthermore, the power has been increased with respect to the single cathode DCMS process in order to overall increase the coating rate and reduce the coating duration (Table 1).

RESULTS AND DISCUSSION

Nb Deposition Rate Profile

Figure 2 shows the comparison of the Nb deposition rate profile along the cavity for DCMS configuration with single cathode scheme (filled symbols) and for DCMS, PDCMS configurations with double cathode scheme (unfilled symbols). The deposition rate is an average value obtained by dividing the thickness by the total deposition time. The thickness is measured *in-situ* and on samples *ex-situ* using X-Ray Fluorescence (XRF) technique. The error bars along deposition rate axis are indicating the spread measured between points at different azimuth (rotating the cavity by 90° steps about a fix reference side), while those along x-axis are showing the precision in the vertical position of the measurement. It can be seen that for DCMS with single cathode, the difference between the inner and outer conductor deposition rate profile is up to one order of magnitude. This difference is reduced for DCMS using double cathode configuration by balancing the power between the two cathodes (Table 1). The results of DCMS (double cathode) enables to refine the power distribution between inner and outer cathode for PDCMS configuration (double cathode) and a comparable deposition rate profile for outer and inner conductor has been achieved.

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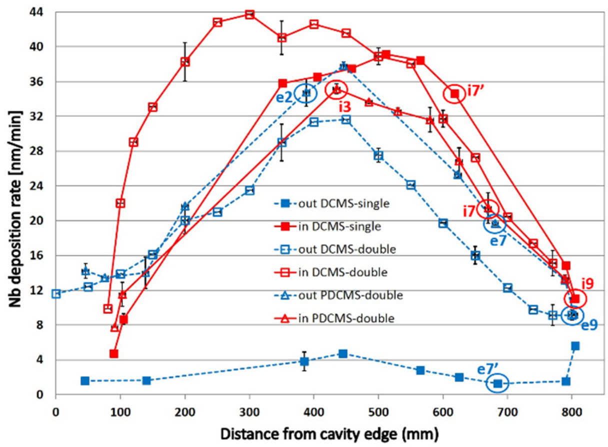

Figure 2: Comparison of Nb deposition rate profiles of iner and outer conductor for DCMS and PDCMS using single and double cathode schemes.

Figure 3 shows the comparable coating rate profiles for inner and outer conductors that can be achieved using optimized balanced power with PDCMS double cathode process with respect to DC-bias diode baseline process.

A constant problem of all the configurations remains at the top of the cavity, a sensitive position due to the high magnetic component of the RF-field [6], where the deposition rate is low compared to the other positions in the cavity. Further tuning of the coating process and cathode design is needed to improve the coating rate in this region.

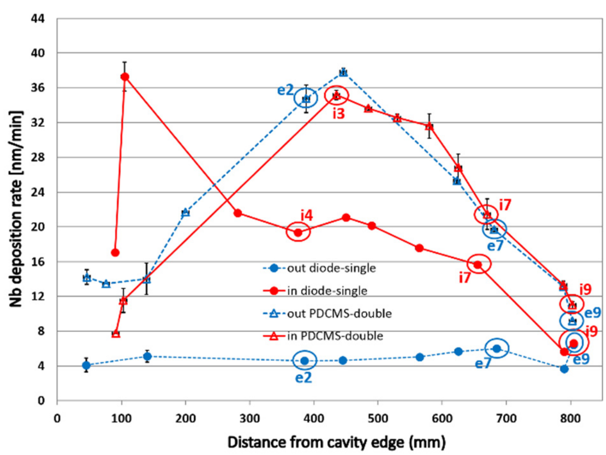

Figure 3: Comparison of Nb deposition rate profiles of DC bias diode baseline using single cathode and PDCMS using double cathode.

Nb Thin Film Morphology

The RF properties of Nb thin films are improved by increased purity and increased order within the grain distribution. Furthermore a microstructure free of voids is required to improve superconducting characteristics and film adhesion.

FIB cross sectional analysis of the single and double cathode at different positions in the inner and outer conductors are shown in Fig. 4 and Fig. 5. The corresponding locations at which this analysis was performed are circled in Fig. 3. Fig. 4 [12] shows that for the DC-bias diode sputtering configuration the film layer is dense through the whole cavity with nanoscale voids present only at positions e9 and i9. In comparison, Fig. 5 shows that moving from sample e2 towards the top and then towards the inner conductor of cavity till sample i9 the film is dense and only a small number of nanoscale voids are present in samples e7 and i9, which suggests an appropriate film growth in this region.

In contrast, porosity is revealed in samples i3 and i7 where large numbers of micro to nanoscale voids are present. The region around i7 position is particularly critical due to the presence of the highest magnetic component of the RF-field there [6]. This would lead to higher losses if the layer quality is poor. Thus the PDCMS process using double cathode needs to be improved in this region in order to achieve the same morphology as the diode baseline process.

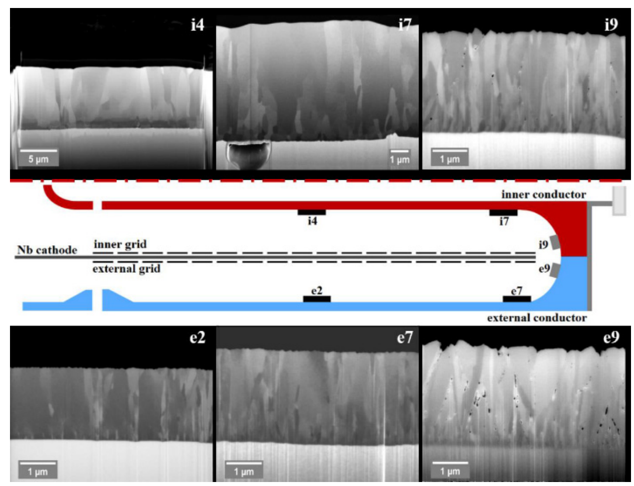

Figure 4: FIB cross-section of samples coated with DCbias diode baseline (single cathode) from [12].

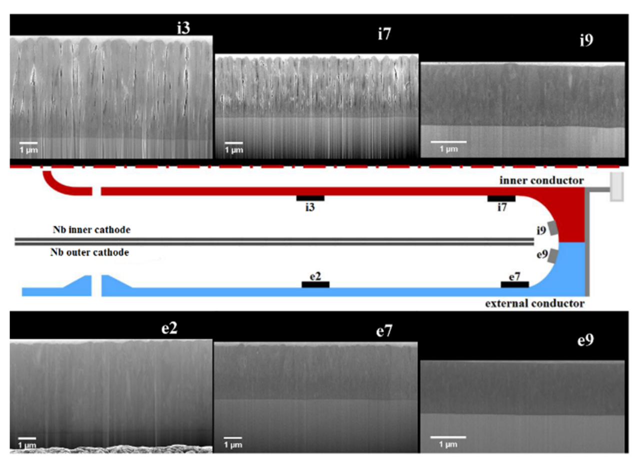

Figure 5: FIB cross-sections of Samples coated in PDCMS configuration using double cathode scheme.

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Figure 6 shows the FIB cross-section of two samples, e7' and i7', coated with DCMS using the single cathode. These samples are taken at a similar position as e7 and i7 samples from PDCMS double cathode process (Fig. 3). For the outer conductor sample e7' the layer is much thinner and features a larger number of nanoscale voids as compared to e7 from the PDCMS process. The inner conductor sample i7' is much thicker and has larger surface features and voids than those observed in the corresponding i7 sample produced with PDCMS double cathode.

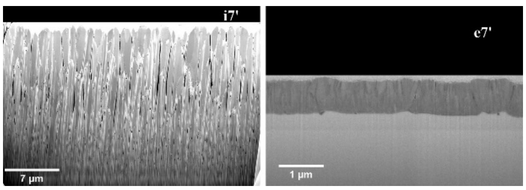

Figure 6: FIB cross-sections of Samples coated in DCMS configuration using single cathode scheme.

Both results from magnetron sputtering process at this location show a layer quality potentially detrimental for the functionality of the superconducting film, although less severe in the case of the double cathode PDCMS process. This is however more related to the improved coating rate with the double cathode rather than a densification of the layer due to the reverse pulsed feature of PDCMS process. Thus we cannot draw conclusion at this stage about a potential bias-like effect for PDCMS process.

From sputtering and transport prospective at low process pressure (magnetron) in this particular geometry for inner conductor part, the large porosity in the film (i7/i7'/i3) might be explained by the concave and convex shapes of the cathode and substrate respectively. Such configuration leads to larger grazing angle for impinging niobium atoms at the surface.

Working at high pressure (diode baseline) sputtering atoms suffer collisions with buffer gas (smaller mean free path) which might help these atoms to have a more favourable angle of incidence to the inner conductor. Combined with the additional energy given by ion bombardment from the biased substrate it helps to obtain a denser layer.

Thin Film Growth Discussion

Coating samples in the dummy cavity in order to optimize the process involves three weeks of preparation work, assembly and coating. Thus a trial and error approach is very time consuming. In order to understand the morphological issue along the inner conductor in the case of magnetron sputtering, transport and film growth simulations have been conducted to see the influence of pressure on film growth, starting with PDCMS process pressure (1.6×10^{-2} mbar). A strong assumption of uniform racetrack (sputtering profile) throughout the cathode surface is made to run the simulation. First using SRIM [13] one simulates the sputtering of Nb target with Ar ions for inner and outer cathode separately. The sputtering angular and energy distribution combined to the racetrack are then fed to SIMTRA [14] to obtain the atom energy and angular distributions (transport) on the substrate. The resulting file of SIMTRA is used as input to NASCAM. NASCAM (Nanoscale Modelling) is a 2D-3D Kinetic Monte Carlo code for the simulation of deposition, diffusion, nucleation and growth of films on a surface [15]. We have neglected the thermal diffusion and ion bombardment effect for simulation. Thus the results are only compared among each other and not directly with the FIB cross-sections.

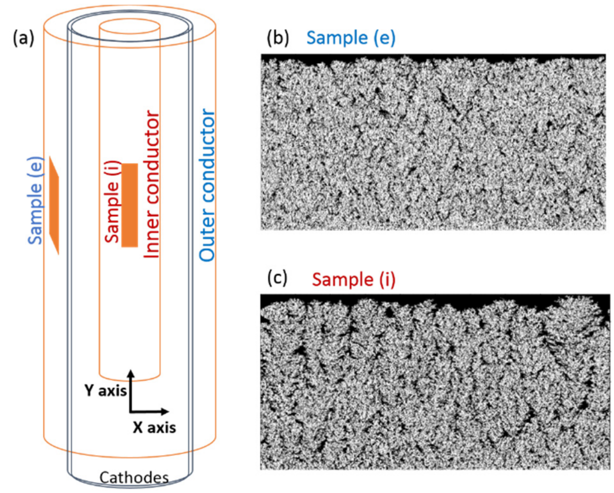

Figure 7: (a) position of samples on inner and outer coductor for which simulation is conducted. (b) outer and (c) inner samples cross-section of thin film growth otained from NASCAM simulation.

NASCAM simulation is conducted for samples (35 mm × 10 mm) placed at the centre (400 mm) of modelled cavity (800 mm) on inner and outer conductor as shown in Fig. 7(a). X and Y axis are supposed along the length and width of samples and simulation is conducted along X axis as FIB cross-section is also performed in this direction. Fig. 7(b) and Fig. 7(c) shows the thin film growth obtained from NASCAM for sample on outer conductor and inner conductor respectively. The results of our simulations indicates larger porosity as well as higher roughness for inner sample than the outer one which is qualitatively consistent with what we observed on the inner and outer FIB cross-sections.

The whole transport and growth simulations have also been conducted for the inner conductor part at a lower process pressure (4×10^{-3} mbar) to assess its effect on the film porosity. The result shows no improvement in the morphology of the film (Fig. 8).

Thus in this particular geometry, using magnetron sputtering, an additional active bias applied to the substrate might be needed to densify the layer.

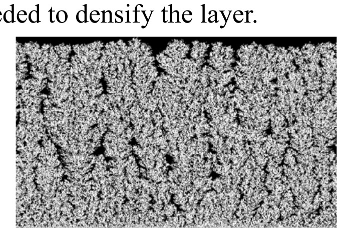
Figure 8: cross-section of thin film growth obtained at low process pressure (4×10^{-3} mbar) from NASCAM simulation for sample on inner conductor.

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CONCLUSION

The preliminary study shows the success of double cathode scheme for obtaining equal deposition rate profiles for inner and outer conductor. The overall coating time is reduced, however, porosity remains in film on the inner conductor when using magnetron sputtering. NASCAM simulation shows that changing process pressure would not help densifying the film on inner conductor part. Further development is needed to address this issue by implementing for instance an active bias to the substrate.

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