DEVELOPMENTS ON SRF COATINGS AT CERN

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Abstract

The thin films techniques applied to Superconducting RF (SRF) have a long history at CERN. A large panel of cavities has been coated from LEP, to LHC. For the current and future projects (HIE-ISOLDE, HL-LHC, FCC) there is a need of further higher RF-performances with focus on minimizing residual resistance Rres and maximizing quality factor Q_0 of the cavities.

This paper will present CERN's developments on thin films to achieve these goals through the following main axes of research:

The first one concerns the application of different coating techniques for Nb (DC-bias diode sputtering, magnetron sputtering and HiPIMS). Another approach is the investigation of alternative materials like Nb_3Sn.

These lines of development will be supported by a material science approach to characterize and evaluate the layer properties by means of FIB-SEM, TEM, XPS, XRD, etc.

In addition a numerical tool for plasma simulation will be exploited to develop adapted coating systems and optimize the coating process, from plasma generation to thin film growth.

MOTIVATIONS

The technical and financial challenges set by the next generation of accelerators rely on the availability of the SRF thin film technology for the acceleration of the particles.

Superconductive thin film materials on copper remain one of the best options in terms of thermal stability, material cost, potential for higher T_c coatings and low sensitivity to Earth's magnetic field, the latter allowing simpler and cheaper cryostat design [1].

The main drivers for the SRF thin film developments at CERN are:

- Short term: HIE-ISOLDE high-beta Nb/Cu Quarter Wave Resonators (QWR) cavities (100 MHz), 20 cavities in total [2].
- Medium term: HL-LHC Crab Nb/Cu Wide Open Waveguide (WOW) cavities (400 MHz), 16 cavities [3]
- Long term: LHC upgrade, ERL, FCC elliptical cavities (400/800 MHz) [4]

The bottleneck of the Nb thin films is the strong increase of R_s with RF-field [1].

R_s is composed of the BCS surface resistance (R_{BCS}), the residual resistance (R_{res}) and the fluxon-induced surface resistance.

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R_{BCS} is a material property, which is found to depend both on temperature and frequency. Factors governing R_{res} are still not well understood.

To reduce the R_s increase with RF-field different paths related to the choice of the superconductive (SC) material or its treatment or the substrate treatment are under investigation at CERN.

The thin film developments at CERN are oriented along three axes referring to the different coating process steps:

1. Substrate preparation, surface and interface
2. Thin film production
3. Top layer surface properties

The target is to grow a smooth, pure, defect free, dense layer of uniform thickness.

SUBSTRATE PREPARATION, SURFACE AND INTERFACE

The first axis concerns the substrate properties, its surface preparation and the resulting interface with the thin film. These parameters will influence the way the layer grows, its purity and composition. The surface preparation will influence the adhesion of the layer and its thermal contact with the copper substrate.

Substrate Preparation

The substrate chemical preparation by electropolishing or chemical polishing are currently used at CERN. Understanding of the key parameters, beyond the roughness, towards the ideal surface conditions is still in progress. The duration and uniformity of the chemical polishing in complex substrate geometry can affect the resonant frequency of the cavity [5]. The surface etching map will be characterized in the case of HIE-ISOLDE cavity geometry for further understanding and optimization of the process.

Substrate assembly in the coating system in a dust free environment is mandatory to guarantee reproducible and reliable coatings conditions. These actions are well controlled at CERN using ultra-pure high pressure rinsing of the substrate and assembly in an ISO 5 clean room. An upgrade of these facilities is foreseen in the next future to match the new substrate shapes and dimension requirements.

Surface and Interface

Thermal annealing at 650°C of the copper substrate is done in between two chemical polishing steps as it was observed that this thermal cycle combined with the chemical surface etching helps to reduce the sulphur content and its segregation at the surface of the copper [6]. XPS surface analysis of OFE copper samples chemically

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polished and annealed following the same process as for cavities are currently under study to understand the role of each step in the segregation/removal of surface impurities process. A second annealing of the substrate at the same temperature, right before coating and without vacuum break in between is used to degas the substrate, and thus lowers the hydrogen content and limits the amount of impurities during the coating. This annealing dissolves as well the native oxide layer.

The contamination by copper diffusion in the layer during the coating at high substrate temperature must also be considered, especially with new materials like Nb_3Sn. Diffusion barrier layer, like tantalum thin film, is under test for A15 superconductive materials [7].

Finally, the thin film surface adhesion and consequently the thermal properties of the film/substrate interface are crucial for the cooling efficiency of the SC layer [8] and can impact the RF performance of the cavity as discussed in [9].

THIN FILM PRODUCTION

The second axis of development concerns the coating itself. The usual ways to control and optimize the film growth, its structure and morphology are the adjustment of the substrate temperature, the sputtered atoms/ions flux and energy and their angle of incidence on the substrate.

Substrate Temperature

The SC coatings are usually done at substrate temperature of about 150°C when the cavity is used as vacuum chamber (1.3 GHz and LHC 400 MHz cavities for instance). When the substrate cannot be used as a vacuum chamber, it is mounted in a dedicated vacuum system where the cavity can be baked and coated up to 650°C (HIE-ISOLDE cavities).

Figure 1 shows Focused Ion Beam (FIB) cross section and Transmission Electron Microscopy (TEM) images illustrate the impact of high temperature cycles of the baseline DC-bias diode sputtering HIE-ISOLDE cavity coating. At this position (middle of the antenna) it shows a dense smooth layer [10]. The contrasts in the TEM image highlight the 15 successive coated layers.

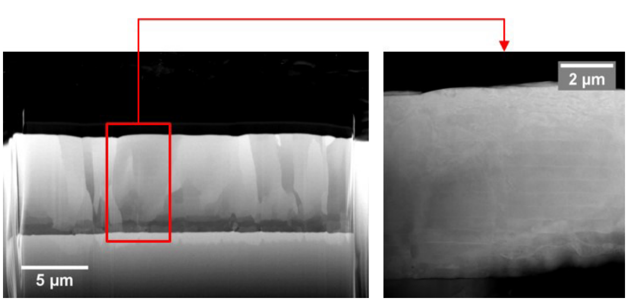

Figure 1: FIB-SEM cross section image of HIE-ISOLDE coating at the middle of the antenna (left) and corresponding High Angle Annular Dark Field (HAADF) TEM image showing the 15 successive layers deposited (right).

Further analysis on a thinner TEM lamella (Fig. 2) revealed the origin of this contrast: dislocation concentration gradient induced by the 15 high temperature coating/cool down cycles of the HIE-ISOLDE coating process [11]. This particular film structure is still under investigation from both materials and SC (vibrating sample magnetometry and susceptometry) points of view.

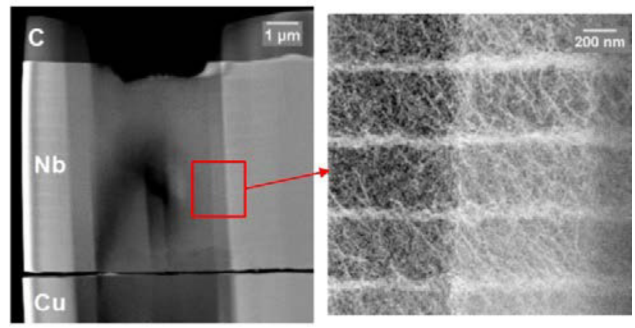

Figure 2: STEM HAADF images show (left) overview of TEM lamella (right) layers 5 to 7 from Cu-Nb interface and gradient of dislocations in between these layers.

The impact of the substrate annealing and of the substrate temperature during the coating will be further assessed by RF and materials characterization of 1.3 GHz cavities baked and coated at high temperature (650°C) and compared with reference low temperature (150°C) coated cavity.

Coating Techniques

The different coating techniques will influence the flux and energy of ions and atoms impinging the substrate.

In DC-magnetron and DC-bias diode sputtering the substrate bias is used to control the energy of the buffer gas ions towards the substrate. These techniques are commonly used for production at CERN. Developments are focused on bias tuning and cathode geometry optimization, as in the case of HIE-ISOLDE using DC-bias diode sputtering. In this example, the baseline coating at the top of the cavity is not as dense as on the antenna [10], as presented in Fig. 3.

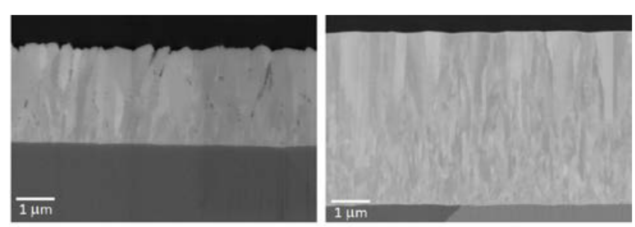

Figure 3: FIB-SEM cross section images of HIE-ISOLDE coating at the top of the cavity with -80V bias (left) and -120V bias + wide cathode (right) showing the densification of the layer.

Increasing the substrate bias voltage by 50% and adjusting the cathode geometry facing the top of the cavity (4 mm wide instead of 2 mm) improved consequently the density and thickness of the layer at this position, Fig. 4, left. But this was at the expense of the antenna part where the thin film analysis by FIB-SEM cross section the layer showed delamination of the layer, Fig. 4, right.

Combination of thickness non-uniformity along the cavity and stress induced by the larger bias can explain this issue. Finer tuning of the bias should lead to a densification of the layer without adhesion issue.

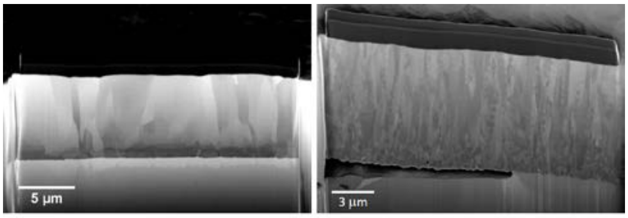

Figure 4: FIB-SEM cross section images of HIE-ISOLDE coating at the middle of the antenna with -80V bias (left) and -120V bias (right) showing the delamination of the layer.

To remedy to the thickness non-uniformity, a new “double cathode” has been fabricated. It will make it possible to decouple the inner and outer conductor plasmas and get an independent control of the coating for each part of the cavity. The first test of this cathode is planned for the end of 2015.

Impinging Angle

The impinging angle can be controlled by ionising the sputtered atoms using High Power Impulsed Magnetron Sputtering (HiPIMS) technique and biasing of the substrate [1]. This method, under active development at CERN on 1.3 GHz cavities substrate, is particularly interesting for coating complex geometries, like low-beta RF cavities, to obtain a conformal deposition with normal angle of incidence all over the substrate.

TOP LAYER SURFACE

The third axis of development focused on the quality of the top most layer which is penetrated by the RF. Critical parameters are: film roughness, grain size, grain boundaries impurities, surface contamination and oxide layer. They depend mainly on the two first points and can barely be controlled afterwards.

The losses related to the surface oxidation and intergranular oxidation will be investigated. If needed, two approaches to cure them are foreseen: either using HiPIMS to grow very dense film, or by protecting the deposited SC surface with an additional passivation layer.

COATING COMPLEX GEOMETRIES

Complex geometries imply specific coating constraints that can be mitigated by an adapted RF and mechanical design of the cavity: avoid sharp edges, minimize potential shadowing area and welds.

Nonetheless, the coating setup and more specifically the cathode have to be properly designed to match the desired layer properties described above. An example of the actual HIE-ISOLDE QWR coating system [11] and possible adaptation of the later for WOW cavity coating [3] is presented in Fig. 5.

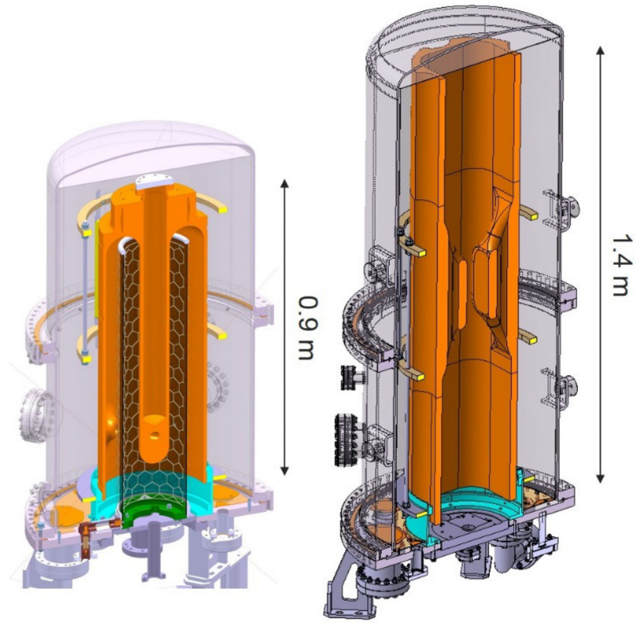

Figure 5: QWR (with single cathode and grids, left) and WOW (right) coating setups.

Test Facilities

To assess the layer properties depending on the coating parameters and substrate geometry, dedicated test substrates and facilities are available at CERN.

For HIE-ISOLDE geometry, a cavity moke-up used as sample holder at 1:1 scale has been developed and used for process and hardware development [12]. A similar approach is foreseen for crab cavities with a WOW prototype cavity under design for coating tests [3].

The 1.3 GHz cavities and sample moke-up are used for coating techniques developments (magnetron sputtering, HiPIMS and bias). The SC material investigations are conducted at a first stage using planar magnetron systems.

Each setup has a dedicated vacuum system, including basic vacuum diagnostics (vacuum gauges, residual gas analyser - RGA) and the possibility to mount external plasma diagnostics, Quadrupole Resonator (QPR) sample [13] and copper/quartz samples for material and RF characterization.

In addition to these facilities, CERN developed a plasma test bench to investigate the effect of basic sputtering and plasma parameters, study transport phenomena as a function of process pressure, as well as characterise the different coating techniques. The test bench uses a simple cylindrical substrate as anode and a niobium rod as cathode [12]. The setup has been adapted over the years to integrate plasma diagnostics tools like Optical Emission Spectroscopy (OES), Retarding Field Energy Analyser (RFEA), quartz balance and Langmuir probe. This setup will give direct inputs and benchmarking for the plasma simulations studies.

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Plasma Simulations

Faced with the geometrical complexity of the new projects cavities, and with the need for coating process optimization, an effort has been started to rely on numerical simulation tools. The aim is to increase the understanding of coating processes from plasma generation to thin film growth and converge in a faster way to the best coating setup design for a given cavity geometry.

The first step towards this goal has been to compare these numerical simulations obtained with a DSMC-PIC (Direct Simulation Monte Carlo – Particle in Cell) code [14] with the aforementioned dedicated plasma test bench experiments, both in DC-bias diode and DC-magnetron sputtering modes. The Fraunhofer IST has completed this case study and CERN will soon use this simulation code under license.

Interesting results have been found regarding plasma ignition and development in this simple setup, by simulating reactions between charged and metastable species in an argon buffer gas, providing ionized argon bombardment profiles on the niobium cylindrical cathode rod with the plasma code, and niobium deposition profiles on the substrate with the transport code (Fig. 6).

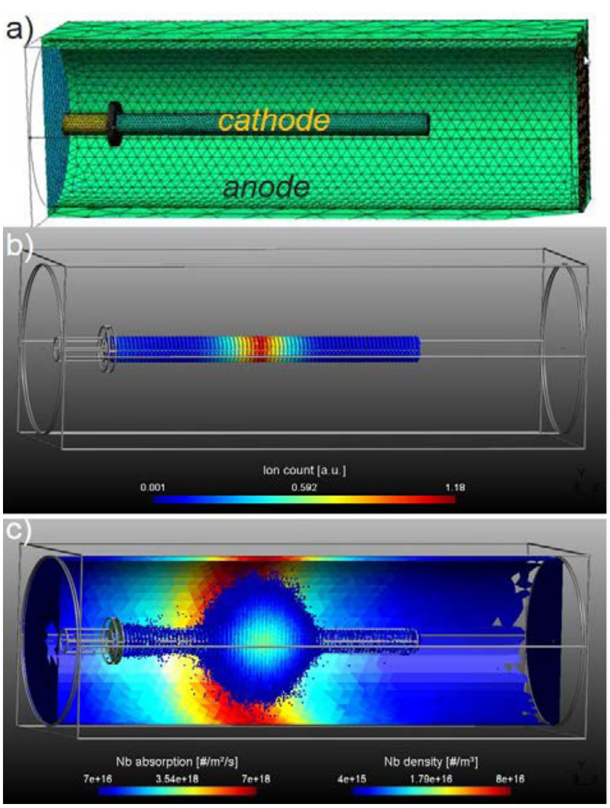

Figure 6: Experimental setup and mesh a), accumulated Ar+ ion flux onto the cathode during PIC-MC plasma simulation b) and resulting DSMC transport simulation, where Nb is sputtered from the target with a distribution according to the ion bombardment profile c) (DC-magnetron sputtering, 0.01 mbar).

Good agreement between simulation and experiment is shown in Fig. 7.

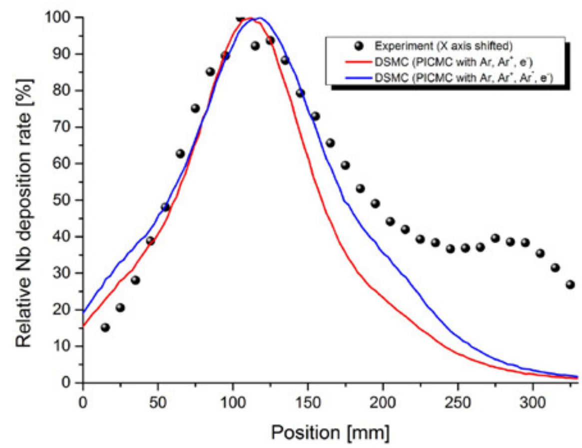

Figure 7: Relative experimental and simulated Nb deposition rate profile along the substrate facing the cathode (DC-magnetron sputtering, 0.01 mbar).

Once the benchmarking study on the plasma test bench is completed the code will be used to simulate complex geometries starting with HIE-ISOLDE cavity geometry and then extended to crab cavities case to optimize the coating setup.

A next step will be to use the deposition profiles as inputs for existing thin film growth simulation code like NASCAM developed by Namur University [15, 16].

SUPERCONDUCTING MATERIALS

The developments on niobium coatings concentrate on the coating techniques and surface preparation presented above.

To counteract the R_s limitation of niobium film an alternative is to use A15 compounds offering a higher Tc and smaller BCS surface resistance, Fig. 8 [17].

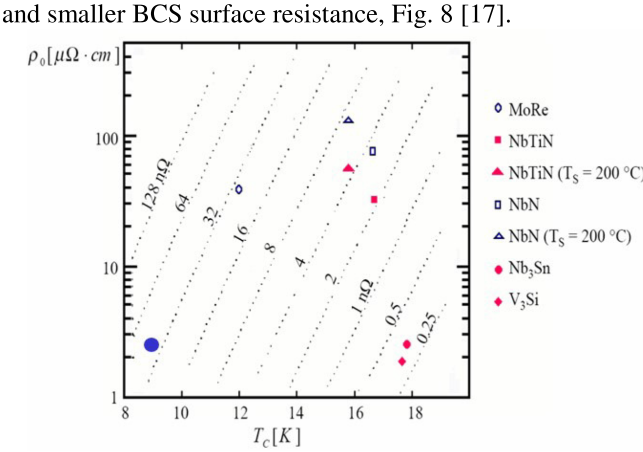

Figure 8: Lines of equal BCS surface resistance in (ρ, Tc) space for different SC materials, adapted from [17].

Nb_3Sn and V_3Si are the most promising compounds. A systematic study of Nb_3Sn has been launched at CERN using Nb_3Sn alloy target and DC-magnetron sputtering [7] on copper and quartz samples for layer characterization. V_3Si will be studied in the same way in a next future.

First trials at CERN were performed with ambient temperature sputtering and annealing of the samples up to 800°C under vacuum after coating. These experimental conditions, compatible with a copper substrate, were sufficient to obtain the desired A15 phase [7]. The setup is currently adapted to perform high temperature coating by heating the copper substrate and trying to get the A15 phase *in-situ*.

COLLABORATIONS

These challenging objectives can only be completed by a team work and collaborative efforts within CERN and beyond: STFC-CFR-CERN collaboration launched in spring 2015, European projects EuCARD-2 and EuCARD-3, collaboration with local universities - UNIGE and EPFL (CIME and CRPP), with research institutes - CEA Saclay and IPN Orsay and with the international SRF thin films community.

CONCLUSIONS

Thin film SRF is one of the most promising approach for large scale project like FCC. The recently started second run of LHC will determine the direction accelerator technology should take for its future developments.

As a consequence, SRF thin film technology must be mature by that time and extensive developments in collaboration with worldwide institutes started.

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