ISBN: 978-3-95450-168-7 doi:10.18429/JACoW-IPAC2015-WEPHA022

CHARACTERIZATION OF NB COATING IN HIE-ISOLDE QWR SUPERCONDUCTING ACCELERATING CAVITIES BY MEANS OF SEM-FIB AND TEM

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Abstract

The Quarter Wave Resonators (QWR) high-β cavities (0.3 m diameter and 0.9 m height) are made from OFE 3D-forged copper and are coated by DC-bias diode sputtering with a thin superconducting layer of niobium. The Nb film thickness, morphology, purity and quality are critical parameters for RF performances of the cavity. They have been investigated in a detailed material study.

INTRODUCTION

In the frame of the High Intensity and Energy (HIE) ISOLDE project [1], the niobium coating of high beta superconducting quarter-wave resonators (QWRs) has entered series production. The first cryomodule is upon to be finalized [2], however development activities continue to improve the understanding and optimization of the sputtering setup and process. One major focus is the thin film characterization and its relation with the functionality of the layer. This paper presents the results of the microscopic approach to characterize the Nb layer of the cavity coating baseline process.

EXPERIMENT

A dummy copper cavity (Q4) [3] has been designed as a sample holder to characterize the niobium layer. Samples supports are 35 mm long, 10 mm wide and 1 mm thick copper plates. Samples are identified by their position in internal (i) or external (e) conductor and their distance from the bottom to the top of the cavity. Top band (TB) sample placed over the weld on the top of the cavity was observed as well.

The applied DC-bias sputtering baseline parameters are: 0.2 mbar Ar pressure, 8 kW powered Nb cathode, -80 V biased cavity, and a substrate temperature rising from 300°C up to 630°C (below the bake out temperature of 650°C) on the inner conductor during a coating step. The whole coating process lasts 4 days and is done in 14 steps of 25’ coating + 5h35’ cool down to 300°C each, leading to a net coating time of 6h.

The film morphology, its thickness and quality from the selected places along the cavity’s inner and outer conductors were studied by dual beam high-resolution field emission Scanning Electron Microscope (SEM) with Focused Ion Beam (FIB).

Transmission Electron Microscopy (TEM) observation were performed together with Energy Dispersive Spectroscopy (EDS) measurements and orientation and phase mapping with a spatial resolution of 2-3 nm. TEM lamella sample from top part of the cavity was prepared to study chemical composition, structure and grain orientation of coating grains.

X-ray florescence measurements (XRF) were performed to cross check the coating thickness.

CROSS-SECTION IMAGING

A standard sample preparation of the cross-section consists of 4 steps on a selected area of interest [4, 5]: (i) a deposition of protective layer (in our case carbon of 1.5 μm thickness) onto the sample surface in order to protect it from the ion beam damage during further milling, (ii) the removal of the material by sputtering using the staircase-like pattern with high Ga^+ beam current to form the large trench, (iii) reduction of the beam current and milling to reduce redeposition of sputtered material onto the sample surface, (iv) imaging of the final surface using either secondary or back-scattered electrons or secondary ions depending on the goal of the study. All SEM images presented in this paper were acquired using in-column secondary electron detector and low accelerating voltage in order to visualize grain structure at different places in the cavity and possible presence of porosity.

The niobium layer thickness, its structure and the presence of porosity

The coating thickness was measured by two techniques – XRF and from the cross-section images after the FIB preparation. It is evident from Table 1 that the inner conductor coatings i4 and i7 are much thicker comparing with the same positions on external conductor e2 and e7. The measurements by FIB shows higher values than by XRF.

Table 1: Comparison of Coating Thicknesses Characterized by FIB and XRF Techniques

Thickness [μm]	FIB	XRF
i4	7.25	6.68
i7	5.74	5.41
i9	2.66	2.21
e2	1.79	1.64
e7	2.27	2.07
e9	2.53	2.16
TBi	2.78	2.50

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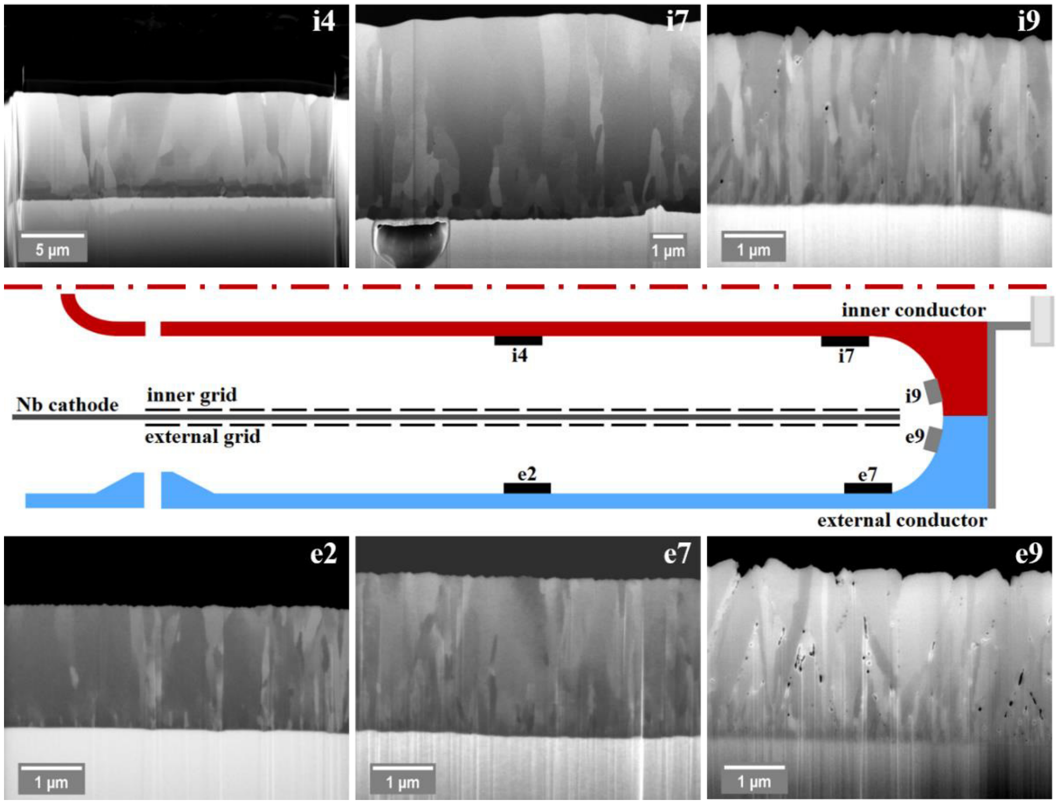

Figure 1: Schema of the Q4.8 cavity. The black boxes represent the positions of the samples with deposited Nb coating along the inner and external conductor. The cross-section images from selected positions revealed the grain structure, in certain samples the presence of porosity.

The Nb coating grain structure is comparable at the similar positions in the inner and external conductor. At the top of the cavity grains of few nanometers are formed at the interface with copper for samples e9, i9 see Fig. 1 and TBi, see Fig. 2. During the further growth elongated Nb grains of different width are developed. Samples e9 and i9 are rougher than the other samples.

The unexpected presence of porosity was revealed for e9, i9 and TBi samples at the top of the cavity. Whereas for the TBi sample, the round nanometer size pores were observed only close to the Cu substrate, samples i9 and especially e9 porosity goes along the grain boundaries through the whole thickness of deposited layer towards the surface. For the superconductive functionality of the Nb layer, such porosity could lead to its larger residual resistance (*Rres)*, which consequently may affect the RF performance of the cavity. During the preparation of the cross-section µm size porosity in the Cu substrate was observed as well. The defect in the substrate is presented for the i7 sample in Fig. 1.

The structure of thicker coatings revealed the presence of 14 deposited layers that corresponds to 14 deposition steps and goes across adjacent grains formed in the upper part of the coating, particularly in sample i4. Fig. 3 shows the contrast of the first 3 layers as seen during the SEM imaging on prepared cross section. TEM lamella prepared by Ar^+ milling showed the layers contrast over the whole thickness of the coating, Fig. 4. Unfortunately this lamella was too thick to acquire high resolution images to reveal the nature of this contrast.

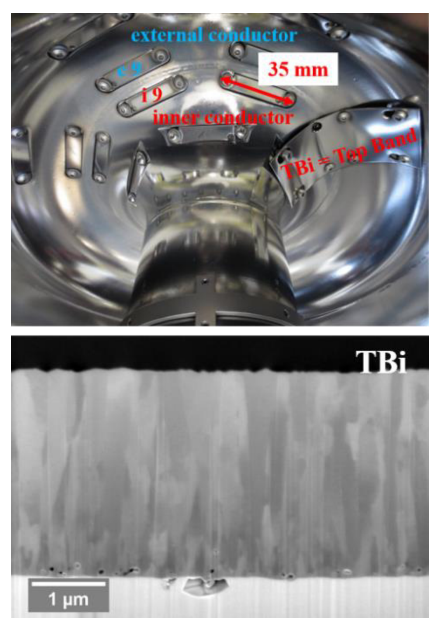

Figure 2: The image shows the real sample positions in the cavity as well the location of Top Band sample that is not marked in Fig.1 scheme. Corresponding cross-section image reveals grain structure and porosity presence close to the Cu/Nb interface.

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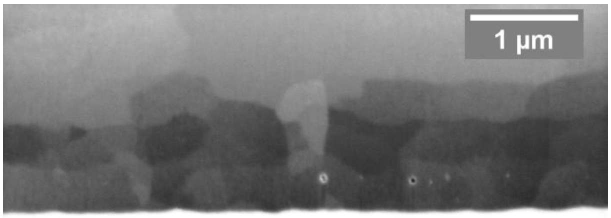

Figure 3: A layered structure of i4 sample close to the copper interface. The rare presence of nm size pores was observed.

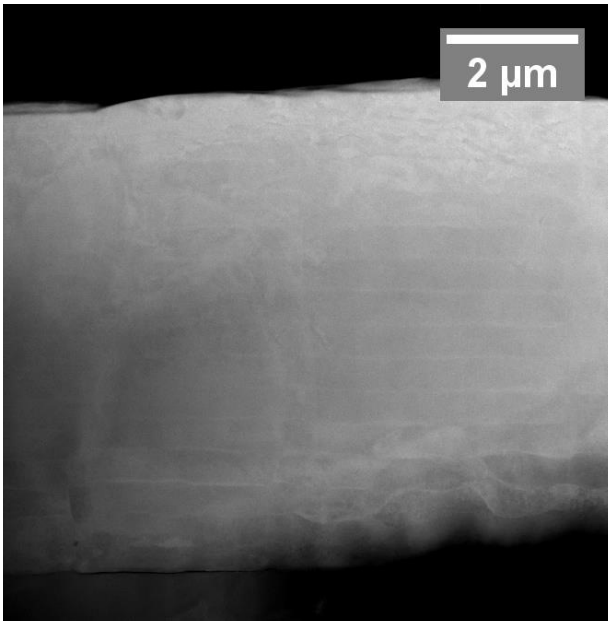

Figure 4: A layered structure of i4 sample seen through the whole thickness of the coating on Ar^+ polished sample.

EDS measurements excluded the presence of contamination or oxide layer. Further TEM study is needed to explain if contrast corresponds to the different grain orientation or atoms reordering while cool down process takes place. The upper part of the thicker coatings is formed with several µm size grains. The surface of the samples from the cavity sides is relatively flat in comparison with the pointed grains shape of e9 and i9 samples.

TEM MEASUREMENTS

The e9 sample was chosen for detailed TEM measurements. Standard TEM lamella was prepared using FIB. EDS mapping at low magnification excluded presence of contamination. Naturally formed 10 nm thick oxide layer was observed on the surface of Nb layer and around the porosity. The porosity oxide is formed after the thinning process is finished and the sample is transferred from the FIB to TEM.

Investigation of the interface between the Cu substrate and first layer of Nb at high magnification revealed the presence of nm size Cu precipitates that are randomly distributed along the interface, see Fig. 5. The precipitates were found only up to the distance of 200 nm far from the interface.

The orientation mapping experiment with ASTAR technique [6] based on collection of precession electron diffraction patterns and cross-correlation with the simulated template was performed on e9 sample. Grains are randomly oriented in the coating, see Fig. 6. Small grains close to the interface cannot be indexed because of the grain overlap. For the grain size quantification plain view sample at defined height of the coating together with X-Ray Diffraction measurements will be more precise.

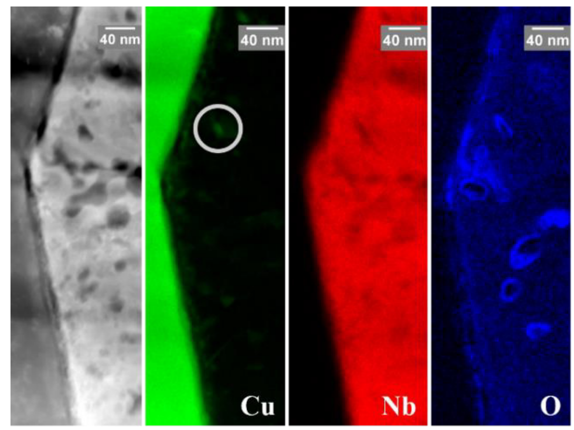

Figure 5: High Angle Annular Dark Field (HAADF) image of the e9 sample interface between copper and Nb coating with corresponding maps of Cu, Nb and O. The presence of 10 nm size precipitate is marked with the circle.

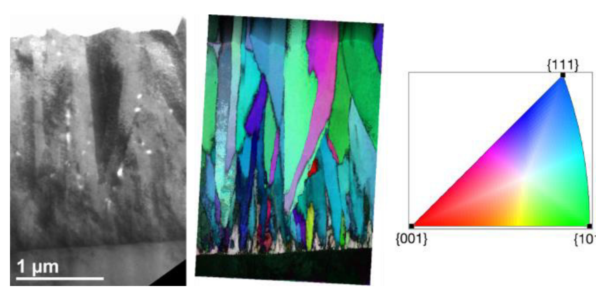

Figure 6: Bright field TEM image of e9 sample with corresponding ASTAR orientation mapping for Nb coating and orientation triangle.

CONCLUSIONS

The detailed material study revealed the grain growth structure of the Nb coating samples along the copper cavity and presence of porosity through a complete deposited layer in samples e9 and i9.

As shown for e9 sample, Nb layer is free of contamination, nm size Cu precipitates were found up to 200 nm far from Cu substrate. Grains are randomly oriented.

Insight in the Nb coating structure, composition and grain orientation might help together with other measurements to understand the behavior of the applied deposition recipe and the RF performance of the cavity.

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