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THICK FILM MORPHOLOGY AND SC CHARACTERIZATIONS OF 6 GHz Nb/Cu CAVITIES

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Abstract

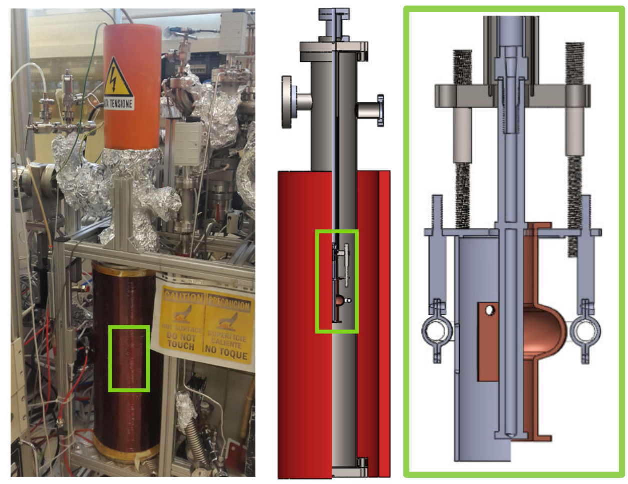
Thick films deposited in long pulse DCMS mode onto 6 GHz copper cavities have demonstrated the mitigation of the Q-slope at low accelerating fields. The Nb thick films (~40 microns) show the possibility to reproduce the bulk niobium superconducting properties and morphology characterizations exhibited dense and void-free films that are encouraging for the scaling of the process to 1.3 GHz cavities. In this work a full characterization of thick films by DC magnetometry, computer tomography, SEM and RF characterizations are presented.

INTRODUCTION

At Legnaro National Laboratories (INFN), an innovative approach for prototypes cavities has represent an important part of the SRF research. The approach includes high temperature coatings, by placing the substrates inside the vacuum chamber, and depositing thick films (<100 µm). These films, promote a structure that pushes the superconducting properties and performances close to the niobium bulk ones. In this work will be shown different characterizations on prototype cavities and samples that support the hypothesis mentioned before, as well as the last results on a QPR sample deposited with similar conditions to the 6 GHz cavities.

COATING TECHNIQUE

For the deposition of the thick films (~45µm), post magnetron configuration has been used (Fig. 1). The source of the magnetic field that allows the sputtering process to develop, is located outside the vacuum chamber. The copper substrates (cavities or samples) are deposited by long pulse DC magnetron sputtering. Single layers of hundreds of nanometres grow consecutively in order to avoid stress in the thick film that might affect the adhesion of the niobium to the copper and reduce the film performances. The coating technique includes also the high-temperature coating at 550°C in ultra-high vacuum conditions. The substrates are located inside a vacuum chamber, this permits the implementation of a heating system for the baking and coating processes [1].



(a) (b) (c)

Figure 1: (a) Deposition system by post-magnetron sputtering technique. (b) Design of system. (c) Cavity and sample coating configuration inside the system.

THICK FILM MORPHOLOGY

Thorough analysis by Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD) characterizations were made at STFC to samples from cut 6 GHz cavities coated at LNL. The thick film approach was studied in two different deposition modes: long pulse and one-shot (coating without any pauses in the process) modes. The coating technique by long pulse deposition showed a higher grain structure than the one-shot deposition. Furthermore, the dispersion of the histograms representing the grain sizes, was also lower. In the Fig. 2, it is possible to observe the EBSD characterization for cavity 16 deposited by long pulse mode where each pulse thickness is 500 nm (a), and cavity 7 deposited in one pulse mode (one-shot).

It is evident that close to the Cu-Nb interface, the grain size is small and gradually the size increased with thickness. After approximately 30 µm, the structure is homogeneous with a columnar growth of the Nb grains.

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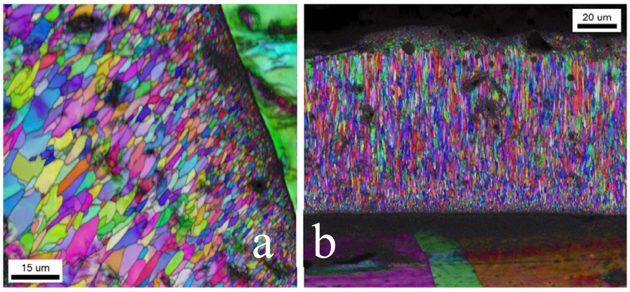

Figure 2: EBSD characterization: (a) Long pulse deposition (cavity 16). (b) One pulse deposition (cavity 7).

The SEM characterization showed a dense, void-free, and columnar growth of the Nb thick film onto the Cu cavities. It is possible to observe in Fig. 3, the homogeneity of the morphology when approximately 30 µm of niobium are deposited in the equator of the cavity.

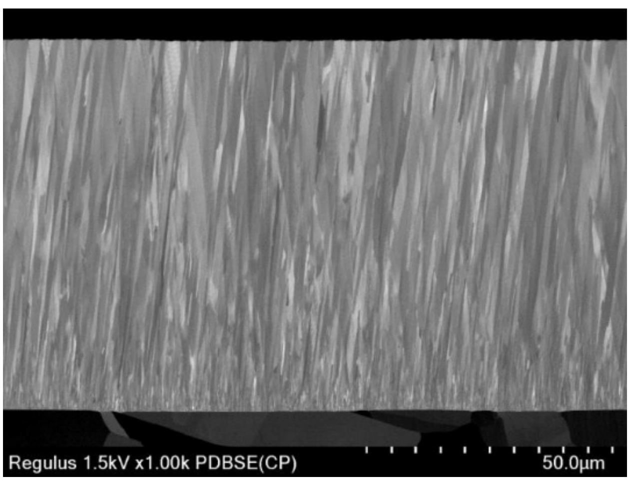

Figure 3: SEM characterization of Nb film on Cu cavity.

The characterization also shows a differential grain growth dependence on the relative angle between the source (Nb target) and the substrate (Cu cavity). At the equator the grains grow perpendicular to the surface while for the sample extracted from the iris, the grains grow oblique to the Cu surface (Fig. 4). The preferential growth affects the critical temperature locally as reported in [2].

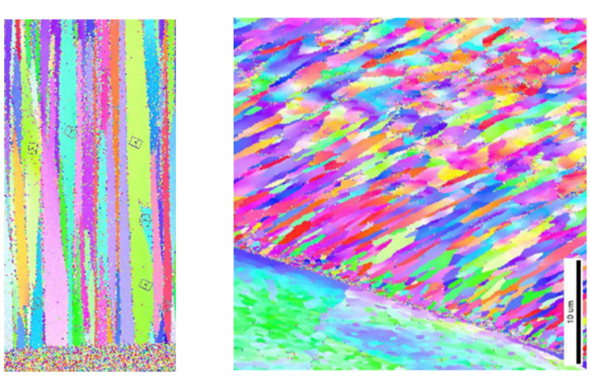

Figure 4: EBSD images from thick film samples extracted from equator (left) and iris (right) of a 6 GHz cavity.

The reproducibility of these results is confirmed by the analysis of samples extracted from cavity 21. This cavity was deposited with the same coating parameters as cavity 16, where the long pulse thickness is 500 nm and maintaining the pulses pauses the same. In Fig. 5, the columnar growth and the size of the grains are visible and reached tens of micrometres.

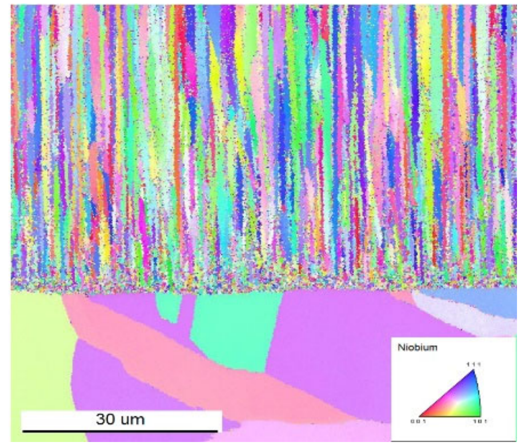

Figure 5: EBSD characterization to equator of cavity 21.

PERFORMANCES OF 6 GHz CAVITIES

At LNL the research on the niobium on copper cavities has been an important contribution for the last decades, for the elliptical cavities as for the quarter wave resonators. The 6 GHz cavities represent a low-cost research that has been a key step to go through from the prototypes into the real cavities in the framework of the accelerator technology. In this context, several cavities coated with thick films have proofed the possibility to mitigate the Q-slope for low accelerating fields [1]. In Fig. 6 are shown two cavities that were discussed previously, cavity 16 and 21.

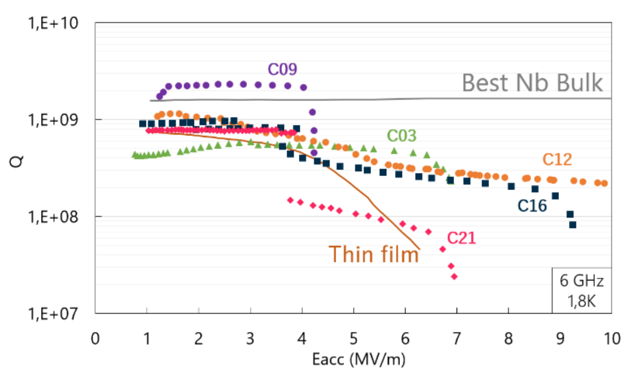

Figure 6: RF performances of thick film coated 6 GHz cavities at 1.8 K.

The cavities 09, 16, and 21, which possessed a uniform and columnar growth of Nb grains that reached tens of microns, mitigated the Q slope for low accelerating fields with remarkable constant performances. At approximately 4 MV/m, these cavities presented Q-switches or quench, that may be produced by detachments at the microscopic level of the niobium film. Considering the high thickness of the films, these possible defects may cause a local in-

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crease of the temperature that induce the Q-switch behaviour. It is worth to mention that in the cells of the cavities was not observed any type of defects regarding the non-proper adhesion of the films.

The influence of the single pulse thickness was studied by coating eight different cavities with single layers from 100 nm o 500 nm. In Fig. 7, may be seen that the cavities with the thicker single layer of 500 nm presented systematically higher performances, in which are included cavities 16 and 21 mentioned in the previous sections.

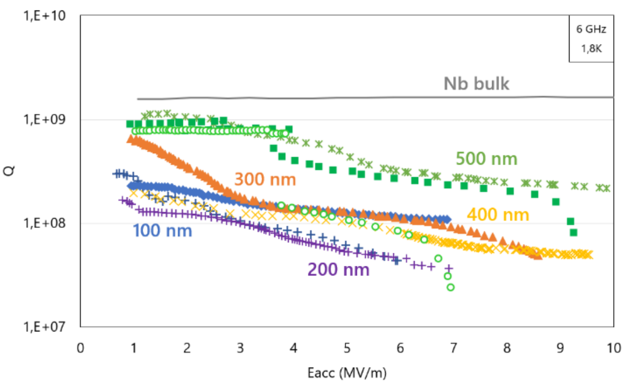

Figure 7: Thick film coated 6 GHz Cu cavities characterization by different single pulse thickness.

DC MAGNETOMETRY

The DC magnetometry permits a further study of the response of samples under a magnetic field (performed at STFC). Estimations of the superconducting properties is given by the change of the magnetic moment of the sample respect to the applied magnetic field.

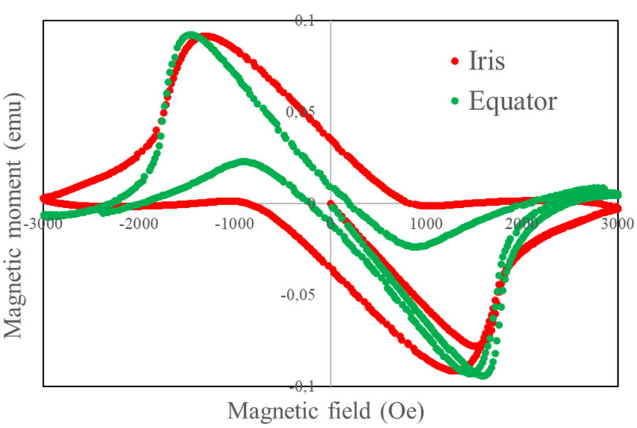

Figure 8: Magnetization loop of samples cut from cavity 21.

The magnetization loop shown in Fig. 8, shows the different responses depending on the position of the cavity. From this characterization, it is possible to infer qualitatively that there is less magnetic flux trapped in the equator respect to the iris. This statement is due to the capability of the sample to return to its initial conditions that may be described as well, comparing the internal areas of the loops. Furthermore, the full penetration field (H_{fp}) and the upper critical field (H_{c2}) can be estimated from the graph [2] to be H_{fp} ~ 148 mT and H_{c2} ~ 330 mT. While for bulk Nb H_{c1}= 180 mT and H_{c2}= 280 mT. It is possible to compare with thin films produced within the ARIES collaboration to be H_{fp} ~ 12-20 mT and H_{c2} ~ 280-300 mT. The comparison between these values implies the improvement of the superconducting properties of Nb thick films of Niobium with respect to the ones of standard Nb thin films for the coated cavities technology. Considering as well, the different orientation of the crystal growth due to the relative angle between the substrate and the cathode, described above. It is important to highlight that the measurements were done on samples cut directly from cavity 21, which presented a Q-slope mitigation (constant performances) at low accelerating fields.

To understand the systematic improvement of the superconducting properties for the thick films, the same characterization was made to different films. These films were stripped from the cavities 07, 16 and 21 and placed onto Kapton for the estimation of H_{fp}. These three cavities were coated using different parameters and presented different performances during the RF test, with cavity 07 showing the lowest performances. No corelation between the DC magnetometry and the RF performances is present. Although the three samples presented a similar behaviour of H_{fp} being 154 mT, 114 mT, and 148 mT respectively [3].

THICK FILMS ON PLANAR SAMPLES

The copper planar samples used in this work were OFC (oxygen-free copper) with dimensions 11 mm x 35 mm x 1,5 mm. The samples were treated by electropolishing (EP), electropolishing and chemical polishing (EP + SUBU5) and plasma electrolytic polishing (PEP) [4] to study the effect of the different treatments on the morphology and on the critical temperature of the thick films. The samples were coated with the cylindrical coating technique and equipment described in the previous section with a thick film of 45 µm. The Tc characterization was done by the CERN Central Cryogenic Laboratory by inductive method [5], and the SEM characterization by Siegen University.

Regarding the critical temperature measurements, the samples showed a sharp and clean transition for all the treatments (Fig. 9). The critical temperature values are for EP and PEP 9,36 K while for the sample treated by EP and SUBU the Tc presented was the highest with 9,38 K. No significant differences in the critical temperature exist in this study for the different surface treatments, and more studies are needed to understand the film stress contribution.

The shape of the SC transition in the Tc measurements of these samples is comparable to the one of a high purity (RRR >= 300) bulk niobium sample measured with the same technique [5], both for its strong symmetry between the start and end point of the transition step and narrow width in temperature [6]. More samples will be coated and characterized for a better description.

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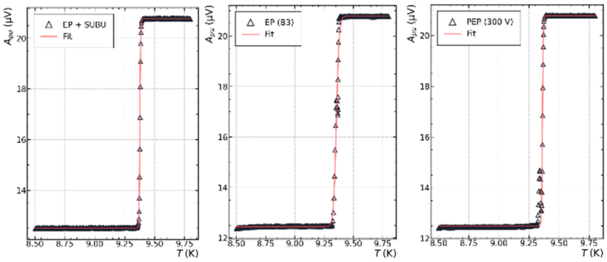

Figure 9: Tc measurements of thick film coated samples with different surface treatments applied.

Respect to the SEM characterization to the surface of these samples, a substantial difference was not present for the different treatments. This may indicate the non-contribution of the sample preparation to the morphology and to the superconducting properties of the thick films. In Fig. 10, the SEM images show the surfaces of the samples mentioned before.

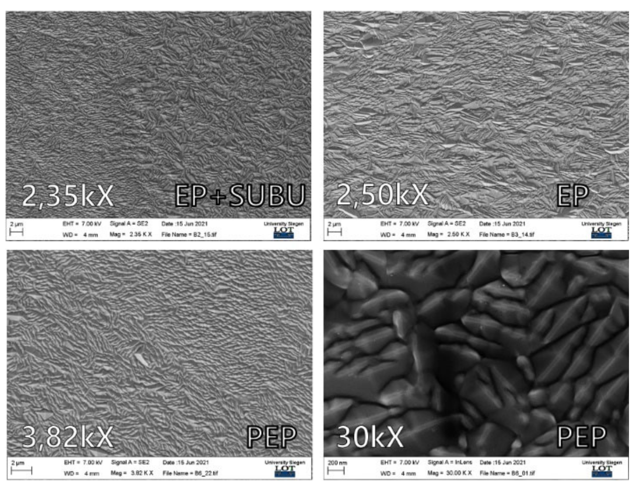

Figure 10: SEM analysis of planar substrates with different surface treatment prior the thick film coating.

Further analysis will be needed in cross-section to understand the effect of the substrate preparation on the niobium crystal development through the whole thickness.

PERFORMANCES OF THICK FILM ON QPR SAMPLE

Taking into consideration the advantages that the RF characterization by the Quadrupole resonator (QPR) has proven in the past years, a QPR sample (B-5) was coated with a niobium thick film. This characterization, performed at the Helmholtz-Zentrum Berlin für Materialien und Energie facilities [7,8], includes the possibility of testing the RF properties on planar samples by calorimetric technique and provides a unique opportunity to directly compare the performances of the thick films with different coating configurations, typologies, and techniques. In the framework of the QPR samples research, sample B-5.11 was treated by electropolishing using the standard protocol used in the ARIES collaboration [9]. The QPR sample was coated with similar parameters to the usually used for the thick films 6 GHz copper cavities in a planar configuration. A 4-inches target of niobium RRR 300 was used for the long-pulsed DC magnetron sputtering process. The approach applied for the coating was a thick film of 45 µm with single layer thickness of 500 nm and high temperature process 550°C in an argon atmosphere under ultra-high vacuum conditions. The degassing process (prior to the coating process) was applied for 42 hours at 600°C.

The aim of the B-5.11 sample by thick film, was to scale the 6 GHz coating process and to obtain a RF characterization that confirmed the results obtained for planar samples and elliptical prototype cavities.

The RF characterization was done at 417 MHz. In Fig. 11, the change of the surface resistance respect to the temperature is compared with a niobium bulk baseline (JN5 baseline) for $B_{sample} = 10$ mT.

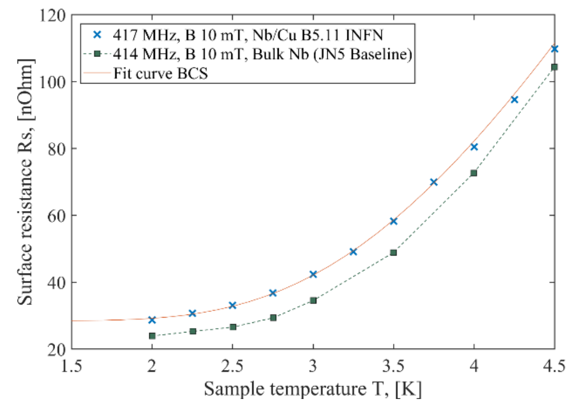

Figure 11: Surface resistance vs temperature plot for the thick film coated QPR sample B-5.11.

Furthermore, the characterization of the surface resistance at 4.5 K, 2.5 K, and 2 K (Fig. 12) presented more similitudes to the Nb bulk baseline. It is worth to highlight the flat-Q characteristics for low peak fields, similar to the results presented for 6 GHz cavities. In addition, the sample presented at 4.5 K and 2.5 K Q-switches at 30 mT, comparable with the results on prototype elliptical cavities for 4 MV/m. However, the sample achieved remarkable high peak field of 70 mT at 4.5 K.

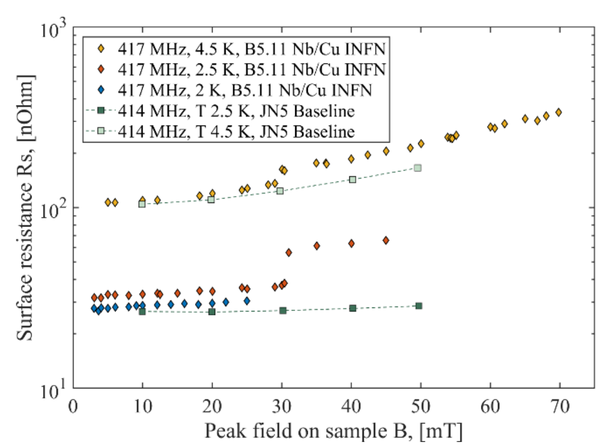

Figure 12: Sample B-5.11 surface resistance vs peak field at 417 MHz.

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The low values of the surface resistance are shown in a linear scale, with the corresponding linear fit for surface resistance values up to 30 mT for 2 K and 2.5 K, in Fig. 13.

In Fig. 13 may be observed the significance of the Q-switch, where the surface resistance increased from 37 nOhm at 30 mT to 56 nOhm. The flat Q or nearly constant surface resistance at low fields makes it worth it to further investigation and research on thick films for accelerating cavities.

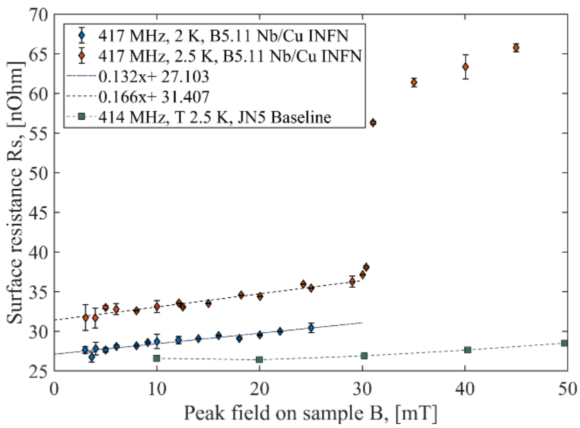

Figure 13: Linear scale of surface resistance vs peak field.

CONCLUSION

In this work, the different characterizations performed on thick films, coated at high temperature by long-pulse DCMS, demonstrate the possibility to obtain dense and homogeneous films. The thick film morphology promotes bulk niobium-like superconducting properties that are confirmed by the DC magnetometry results. The RF performances of these thick films on an elliptical configuration, as well as on a planar configuration by the QPR, showed a significant improvement of the performances with respect to the niobium thin film and a mitigation of the Q-slope for low peak fields.

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