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CERN BASED T_C MEASUREMENT STATION FOR THIN-FILM COATED COPPER SAMPLES AND RESULTS ON RELATED STUDIES

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Abstract

In the framework of the Future Circular Collider (FCC) Study, the development of thin-film coated superconducting radio-frequency (SRF) cavities capable of providing higher accelerating fields (10 to 20 MV/m against 5 MV/m of LHC) represents a major challenge. The development of a test stand commissioned at CERN for the inductive measurement of the critical temperature (T_cc) of SC thin-film deposited on copper samples for SRF application is presented in this work. Based on new studies for the production of Non Evaporable Getters (NEG) coated chambers, the first results of an alternative forming method for seamless copper cavities with niobium layer integrated in the production process are also presented.

INTRODUCTION

The station for the measurement of the T_c of SC thin films deposited on copper substrate presented in this paper was commissioned at the Central Cryogenic Laboratory (Cryolab) at CERN. A consistent R&D program for the development of SC thin-film coated copper cavities is ongoing at CERN, which implies the synergy of the Vacuum, Surfaces and Coatings, Radio Frequency, Cryogenics and Mechanical and Materials Engineering groups. The measurement of the T_c is needed as first assessment of the film quality, and can turn out to be costly in terms of both time and financial resources. Hence the measurement station at the Cryolab has the role of providing a service with fast feedback in the initial part of the production process. The test station is described in "T_c MEASUREMENT". The results obtained with the first reproduction of the procedure described in "REVERSE COATING STUDY" for the production of NEG coated chambers, although applied to niobium and copper to investigate the effectiveness of the *reverse coating* concept for the production of niobium-coated seamless copper cavities, are then presented. Additional measurement series were performed on niobium and A15 films deposited on copper with the T_c measurement station, according to methods established by addressing different goals (which were to study the optimisation Nb film quality for ion incidence at grazing angles, find the optimal recipe for A15 coatings and understand the influence of the substrate preparation on the film quality) and in cooperation with other institutes such as INFN-LNL (Italy) [1], HZB and University of Siegen (Germany).

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T_C MEASUREMENT

The measurement station consists of a contactless two-coil system operated inside a large neck, liquid helium vessel cryostat. The samples[1] to be measured can be inserted and extracted via a dip stick into a chamber that lies above a helium bath, in helium vapour environment. The vapour can flow into the chamber (and hence thermalise the sample) thanks to an inlet at the bottom of the chamber. The temperature of the vapour is adjusted via a heater wound around the inlet and connected to the temperature sensor through a PID feedback loop.

The measurement principle is based on an inductive technique sensitive to the magnetic field expulsion occurring in the film when it turns superconducting, due to Meissner effect. As depicted in Fig. 1.1, the system consists of two coils arranged in front of each other, with the coil planes being parallel but having opposite orientations. The sample is placed between the coils so that its faces are also parallel to the coil planes. The geometry of the system requires the sample to have the standard size and shape of $11 \times 35 \times 1$ mm^3, as Fig. 2 shows. The coil facing the film, namely the *drive* coil, is excited with a sinusoidal AC current which in turn generates an alternating magnetic field that can be measured as a voltage induced across the coil facing the sample substrate, addressed in this context as *pickup* coil, which remains passive throughout the measurement. Before the *drive* coil is turned on, the sample is cooled down below its critical temperature to avoid magnetic flux trapping. When the magnetic field is turned on, the film is in the SC state, the screening currents prevent the field lines from entering the film, and a base background signal is detected in the *pickup* coil to which both external noise and the field leaking around the sample contribute. Starting from this initial state, a temperature ramp is run until the sample turns normal conducting. The data acquisition system logs the temperature and voltage amplitude data during the ramp. The transition of the sample to the normal conducting state results into a step-like voltage signal in the *pickup* coil, due to the increased amount of magnetic field crossing the sample and reaching the *pickup* coil as superconductivity breaks and the screening currents cease to exist.

Figure 1.2 shows a schematics of the complete experimental apparatus, including the generator for the drive current (I_D), the controller to set and read the temperature of the vapour, the lock-in amplifier for the measurement of the am-

[1] In this context, by the term "sample" we refer to the "copper substrate *plus* SC film" system.

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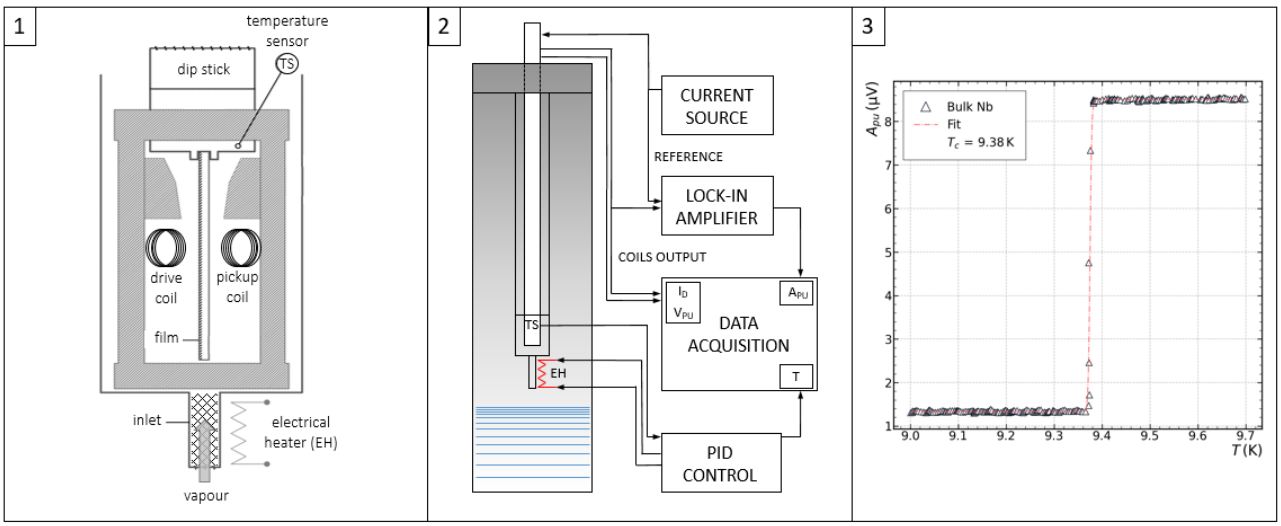

Figure 1: (1) Schematics of the system for the contactless, inductive measurement of the critical temperature of superconducting films deposited on copper for SRF applications. (2) Schematics of the complete experimental setup. (3) A typical measurement of the SC transition of niobium. Data from a bulk niobium sample measured during the calibration phase of the system.

plitude (A_{PU}) of the voltage across the *pickup* coil (V_{PU}). TS and EH respectively indicate the temperature sensor and the electrical heater. By defining the average T_c of the sample as the temperature corresponding to the inflection point of the measured data, it is finally possible to extract this value as the maximum of the fitted data derivative. A representative example of this is shown in Fig. 1.3, with data obtained from a calibration measurement performed with bulk niobium (RRR $\geqslant$ 300). In the plot, the height of the curve, the flat *plateaus* and the narrow step are representative of a good superconducting transition.

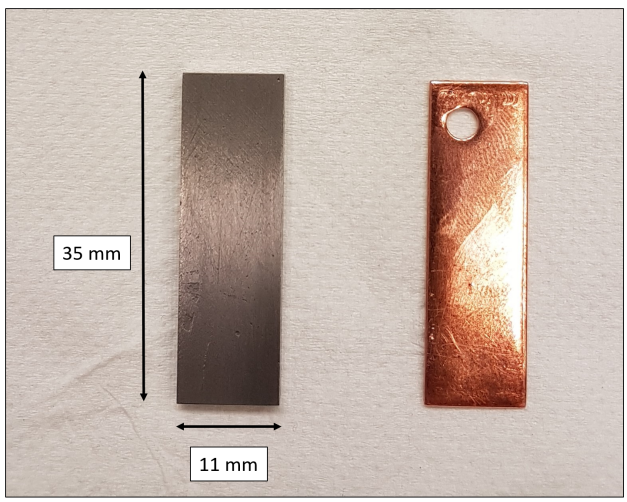

Figure 2: Bulk niobium (left) and copper substrate (right) samples in the standard size of 11 × 35 × 1 mm^3 for the Cryolab T_c measurement station.

REVERSE COATING STUDY

The quality of the substrate has been already proven to be of utter importance when it comes to the performance of niobium-coated copper SRF cavities [2]. Among the parameters to be optimised, the presence of seams and welds is a factor that cannot be completely avoided with standard cavity fabrication methods. However, it has been shown that the electroforming of seamless copper cavities, whose properties are comparable to OFE-grade bulk copper [3], is a real possibility. Based on the idea of electroforming copper vacuum chambers with integrated NEG film coating [4], the same production steps were applied to explore the possibility of integrating the niobium film in the electroformed cavity. The idea consists in building the cavity by copper electroforming around a sacrificial aluminium mandrel, whose shape and size emulate the ones desired for the final cavity. The mandrel is degreased and first, coated with a niobium layer, then with a copper layer. The latter serves as adhesion layer for the subsequent electroforming step. At last, the aluminium mandrel is chemically etched so that only the electroformed cavity with the integrated niobium film is left. A successful outcome of this process would not only lead to better adherence of the SC film to the copper substrate, but also make the chemical treatment of the substrate in preparation to the coating no longer be needed.

For the first trial these steps were reproduced using aluminium disks of both EN-1050 and EN-6082 alloys. The study steps and the relative results obtained with aluminium EN-1050 are presented below.

1. **Coating of Al disk**
   The aluminium disk was first degreased with a detergent solution in ultrasonic bath, then mounted into the coating chamber equipped with both the Nb and Cu cathodes, as shown in Fig. 3.a. The mounting system presents an axis that allows the disk position to be changed from one cathode to the other without open-

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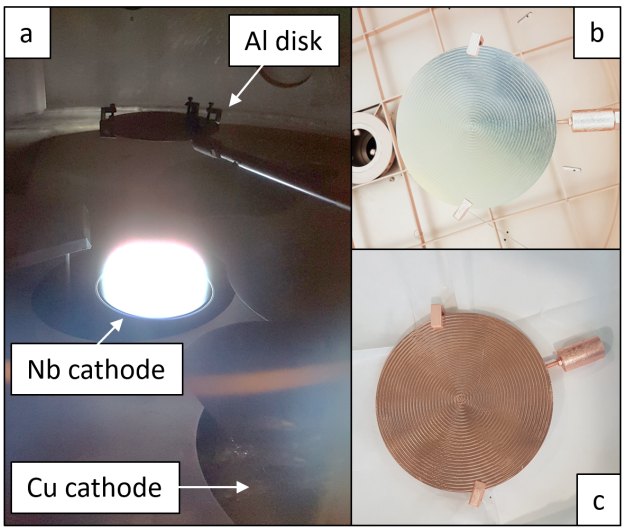

Figure 3: (a) Aluminium disk mounted inside the coating chamber. The niobium and copper cathodes are also visible. (b), (c) Aluminium disk before and after the coating of the niobium and copper layers.

ing the coating chamber, in such a way the coating of the Nb and Cu layer can be performed consecutively without venting the system. The two layers were deposited via bi-polar HiPIMS [5] and corresponded to a final thickness of 1 µm of niobium and 2 µm of copper. Figure 3.b and Figure 3.c show the disk as it appeared before and after the coating respectively.

2. **Electroplating of Cu**
   A layer of 0.5 mm of copper was electroplated on the disk on top of the deposited layer, following the procedure described in [4]. From this disk the first samples were cut for T_c measurement, in order to have an indication of the quality of the film before proceeding with the etching of the aluminium.
3. **Etching of Al and final tests**
   After the first T_c measurement, the etching of the aluminium layer was performed. A 5 M NaOH solution was used, as in the NEG study [4], and again the T_c was measured.

RESULTS AND CONCLUSION

The plot in Fig. 4 presents a sharp, well-defined transition from which it was possible to extract a critical temperature of 9.4 K, which is an indication of a good quality niobium film in terms of superconducting performance, and confirms that the niobium layer is in compressive stress as it is usually observed for sputtered niobium films [6]. For ease of comparison, the measurement after the etching is shown in Fig. 4 together with the previous one in which the film was still sandwiched between the aluminium and the copper. The SC transition in the etched sample measurement shows an amplitude which is nearly a factor 5 smaller and a rising offset, both indicating early flux penetration. The width of the transition is such that it is not possible to identify the

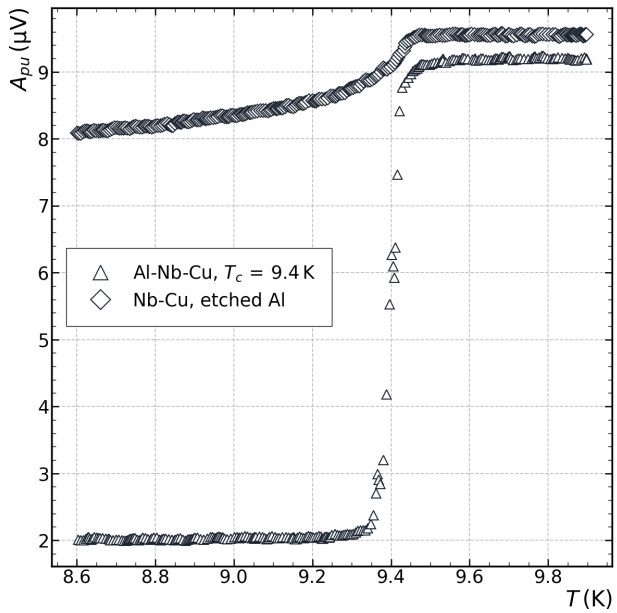

Figure 4: Measurement of the SC transition of the niobium film before the etching of the aluminium (bottom graph) and after (top graph). The etching of the aluminium in NaOH solution results in the degradation of the superconducting performance of the niobium film.

starting point, another indication that the film is already in the mixed state well below T_c. Overall the shape of the SC transition appears to be degraded, suggesting that the etching process damaged the niobium film. The films were analyzed by focused ion beam scanning electron microscopy (FIB-SEM) in order to asses the damage to the niobium layer. Two distinct cases are shown here: in the first one the sample was removed as soon as the etching process finished (27 hours), while in the second one the sample stayed longer (28 hour) in the solution after the end of the etching process. Longer exposure times lead to the formation of a niobium oxide layer on the surface of the film which is not present in the sample that spent 27 h in the solution. This effect is visible in Fig. 5, where the niobium layer that was exposed to the etching solution for a shorter time (a) shows a smooth surface reproducing the machining lines present on the surface of the aluminium disk. The film cross section appears regular too, with the columnar growth pattern typical of physical vapour deposited (PVD) niobium. The sample exposed for longer time (b) instead appears rough at the surface and its section presents damaged structures and voids corresponding to the niobium film.

The strategy for the next iteration of the film-integrated copper cavity forming steps has been now defined based on the observations from the first trial. In order to prevent the etching process from attacking the niobium layer, a protective copper layer will be deposited on the aluminium mandrel *before* the deposition of the niobium film, resulting into a Al-Cu-Nb-Cu sandwich at the end of the coating process [4]. On this last copper layer the final copper thickness will then be electroplated. The aluminium mandrel will be

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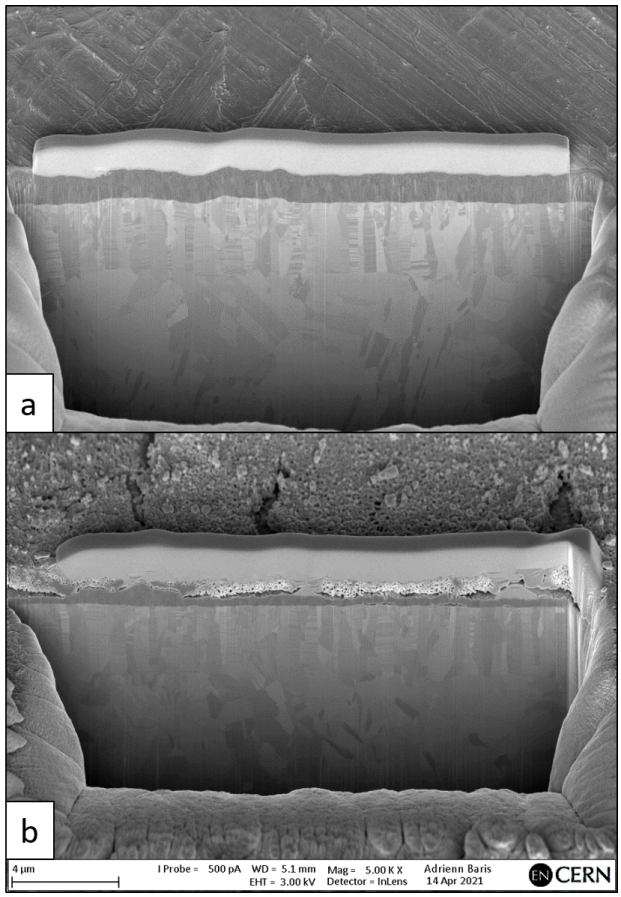

Figure 5: FIB-SEM images of a Nb-Cu sample after the etching of the aluminium. From top to bottom of each image the visible layers are: the surface of the Nb film, the platinum layer required for the FIB ion milling, the cross section of the niobium film and the cross section of the copper layer. Sample (a) was exposed to the NaOH etching of the aluminium for about 27 h. Sample (b) was exposed for a longer time of about 28 h and the damage due to the NaOH water solution is visible on the niobium surface and across its thickness.

again etched in a NaOH solution. Finally, the copper protective layer will be etched in an ammonium persulfate solution, which should not attack the niobium layer and should act as a diffusion barrier against the free hydrogen released during the aluminium mandrel dissolution in NaOH.

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