CERN-based T_c Measurement Station for Thin-film Coated Copper Samples and Results on Related Studies

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Introduction

Background: development of thin-film coated superconducting radio-frequency (SRF) cavities capable of providing higher accelerating fields (10 to20 MV/magainst5 MV/m of LHC) in the framework of the Future Circular Collider (FCC) Study.

T_c Measurement Station: commissioned at the Central Cryogenic Laboratory (Cryolab) at CERN for the inductive measurement of the critical temperature (T_c) of SC thin-film deposited on copper samples for SRF application.

Reverse coating: study of an alternative forming method for seamless copper cavities with niobium layer integrated in the production process, based on new studies for the production of Non Evaporable Getters (NEG) coated chambers.

Critical temperature measurement station

The development of SC thin-film coated copper cavities at CERN implies the synergy of the Vacuum, Surfaces and Coatings, Radio Frequency, Cryogenics and Mechanical and Materials Engineering groups:

- T_c as first assessment of the film quality, can be costly in terms time and financial resources;
- the station provides a free service with fast feedback in the initial part of the production process;
- inductive technique sensitive to the magnetic field expulsion occurring in the film when it turns superconducting, due to Meissner effect.

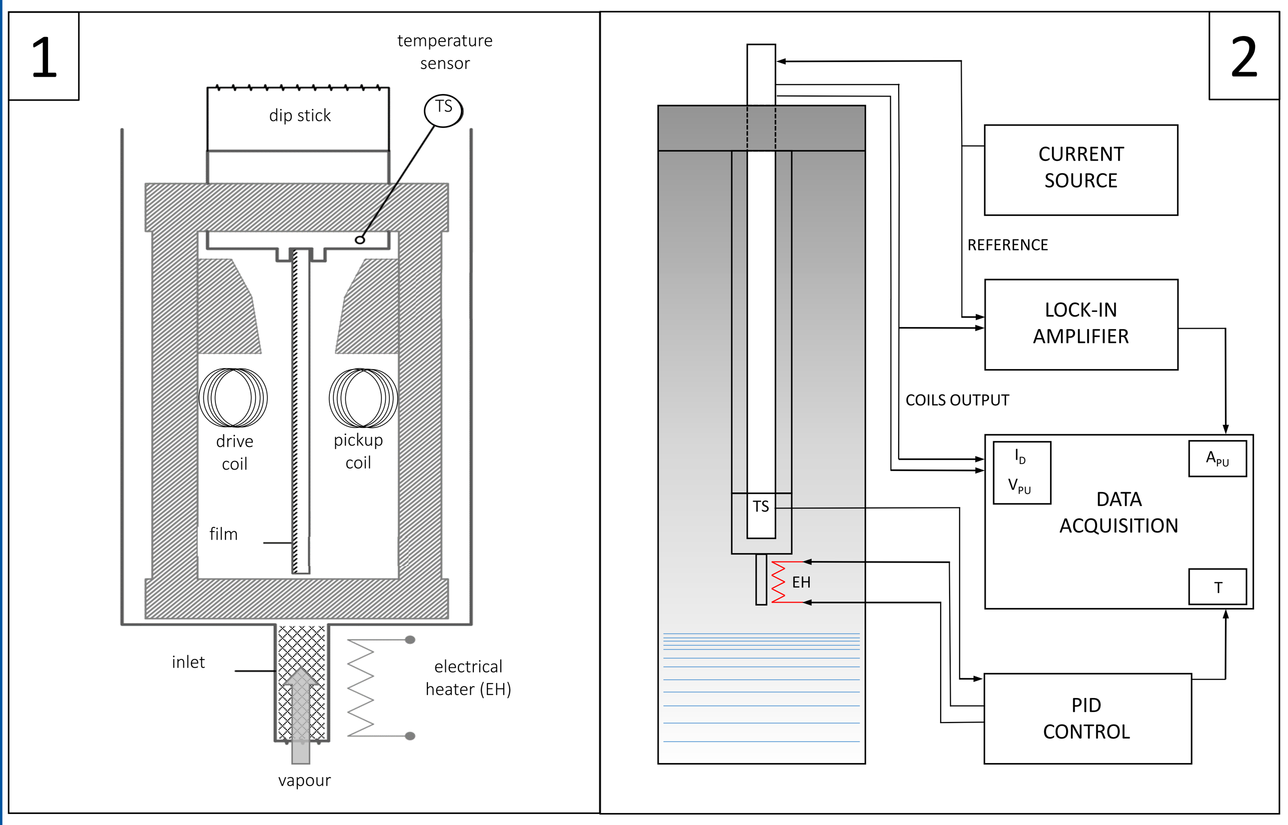
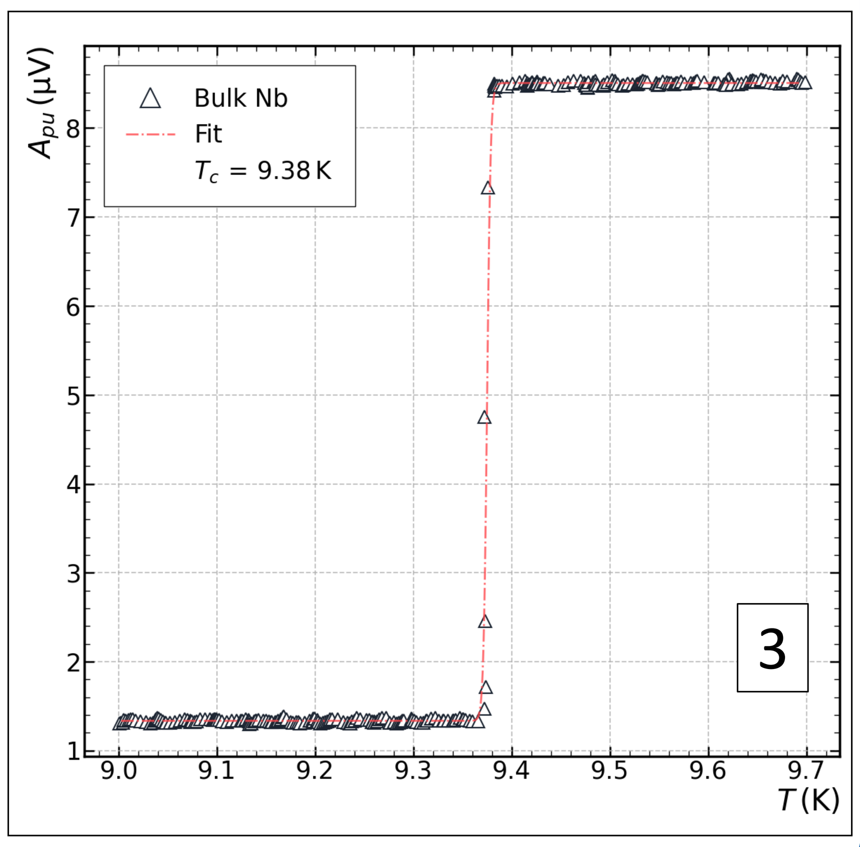


Fig. 1: measurement system.
Co-planar coils into a chamber that lies in He vapour environment. The inlet lets the vapour flow in. The heater (EH) sets the temperature of the vapour to thermalize the sample to be measured. The temperature sensor (TS) reads the temperature and regulates the EH power in a PID loop.
Fig. 2: complete experimental set-up.

T_c Measurement

1. The film-on-Cu sample is placed between the coils, the film faces the *drive* coil.
2. The sample is cooled down below T_c with field off, to avoid flux trapping.
3. The *drive* coil is excited with a sinusoidal current that induces an AC magnetic field.
4. Due to the screening currents in the SC film only a background signal is detected in the *pickup* coil.
5. A temperature ramp is run until the sample turns normal conducting (NC), temperature and voltage amplitude data are logged.
6. The SC to NC transition results into a step-like signal in the *pickup* coil, due to the increased amount of field crossing the sample.
7. Sample average T_c defined as the temperature at the inflection point: extract this value as the maximum of the fitted data derivative (Fig. 3).



Reverse coating study

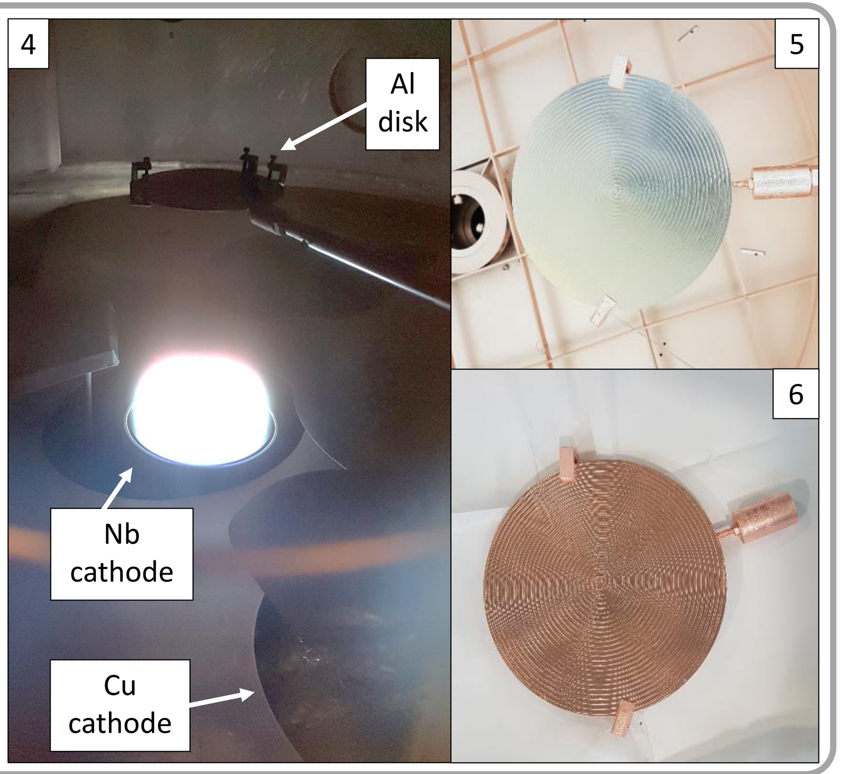
The quality of the substrate is of utter importance when it comes to the performance of niobium-coated copper SRF cavities [1]. The presence of seams and welds cannot be completely avoided with standard cavity fabrication methods, but it has been shown that the electroforming of seamless copper cavities is a real possibility [2]. Based on the idea of electroforming copper vacuum chambers with integrated NEG film coating [3], the following shows the same production steps reproduced 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;
- coating the mandrel via bi-polar HiPIMS with a niobium layer, then with a copper layer;
- chemically etching the aluminium mandrel so that only the electroformed cavity with the integrated niobium film is left.

A successful outcome of this process would lead to better adherence of the SC film to the copper substrate and make the chemical treatment of the substrate in preparation to the coating no longer be needed.

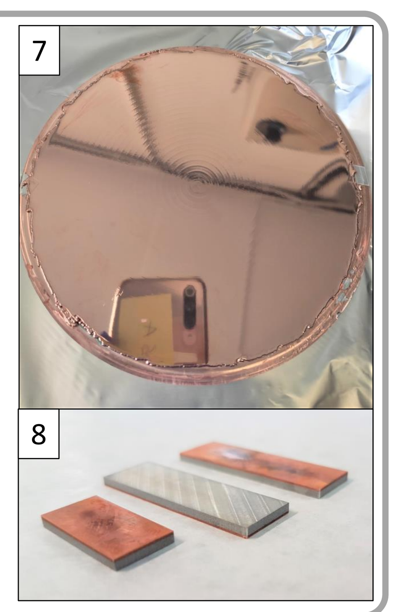
Coating of Al disk

- An EN-1050 aluminium disk was first degreased then mounted into the coating chamber (Fig. 4, 5).
- Without venting the system, the coating of the Nb and Cu layer is performed consecutively.
- The two layers are deposited (1 um (Nb) and 2 um (Cu)).
- The disk the way it looks after the coating in (Fig. 6).



Electroplating of Cu

- The deposited layer serves as adhesion layer for the electroplated copper.
- A layer of 0.5 mm of Cu is electroplated on the disk on top of the deposited layer (Fig. 7).
- From this disk the first samples are cut (Fig. 8) for T_c measurement (Fig. 9 bottom), to have an indication of the quality of the film before proceeding with the etching of the aluminium.



Etching of Al and final tests

- After the first T_c measurement the aluminium layer is etched in a 5 M NaOH solution.
- T_c is measured again (Fig. 9 top).

[1] Benvenuti C. *et al.*, "CERN studies on niobium-coated 1.5 GHZ copper cavities", SRF 2001, conference proceedings
[2] Rosaz G. *et al.*, "Copper electrodeposition for the manufacturing of seamless SRF cavities", SRF 2019, conference talk
[3] Lain Amador L., "Production of ultra-high-vacuum chambers with integrated getter thin-film coatings by electroforming", doctoral dissertation (2019)
[4] Russo R., Sgobba S., "Influence of coating temperature on niobium films", Part. Accel. 60 (1998)

Results

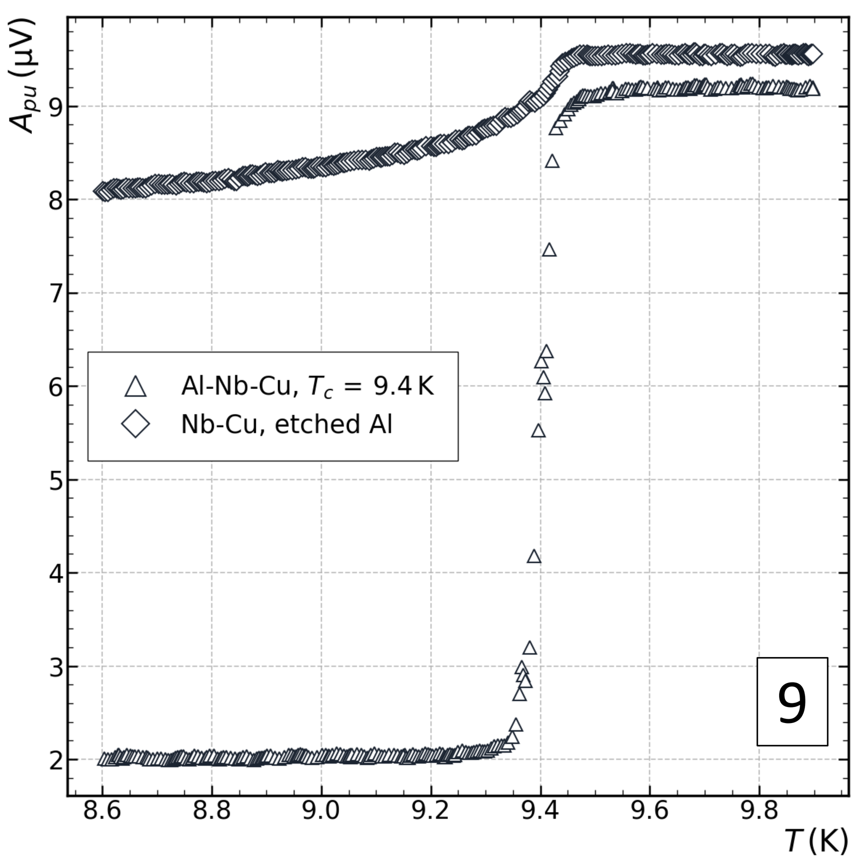
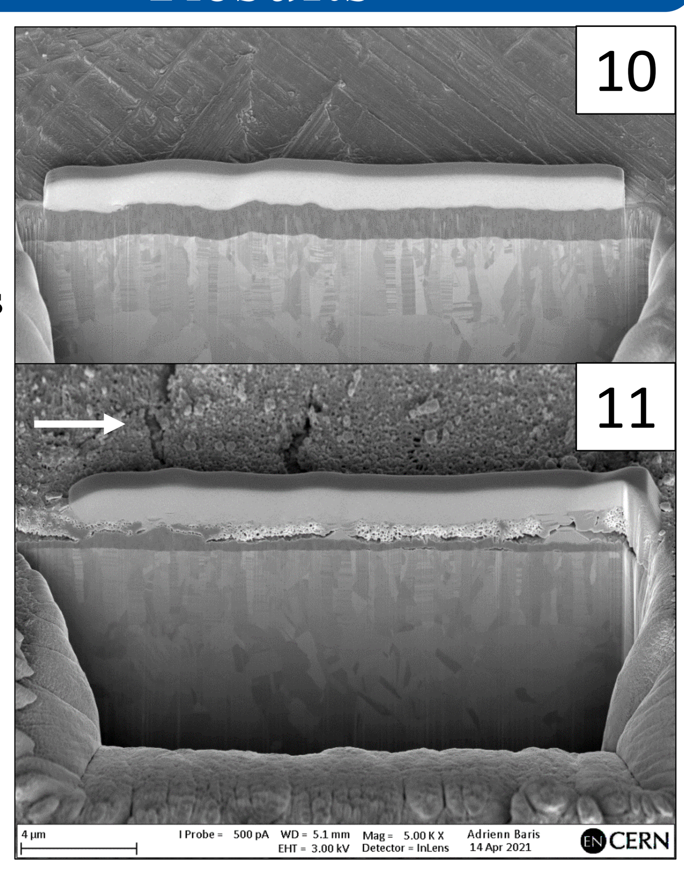


Fig. 9 (bottom): Al-Nb-Cu
- Sharp, well-defined SC transition, indication of a good quality niobium film.
- T_c = 9.4 K
- Nb layer in compressive stress (usual for sputtered Nb [4]).

Fig. 9 (top): Nb-Cu
- Transition amplitude ~5 times smaller and rising offset, due to early flux penetration.
- Width of the transition is broad, makes it impossible to identify the start point, the film is in the mixed state well below T_c.



FIB-SEM pictures - Two distinct cases:
(1) the sample was removed as soon as the etching process finished (Fig. 10). The Nb layer shows a smooth surface reproducing the machining lines from 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;
(2) the sample stayed longer (~1 hour) in the solution after the end of the etching process (Fig. 11), leading to the formation of a NbO layer. The surface (indicated by the arrow in Fig. 11) appears rough and the section presents damaged structures and voids in correspondence of the Nb film.

After the etching of the Al disk, the SC transition appears to be degraded suggesting damage to the Nb film. This is confirmed by the FIB-SEM pictures of two samples etched for different times. **Next iteration:** deposit a protective Cu layer on the Al disk *before* the deposition of the Nb film to prevent the etching of Al from attacking the Nb layer. The final Cu will then be electroplated on the resulting Al-Cu-Nb-Cu sandwich [3].