I.FAST

Innovation Fostering in Accelerator Science and Technology

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DELIVERABLE REPORT

Coating facility built and tested at STFC, USI and INFN

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ABSTRACT

This Report is related to achieving I.FAST Milestone 39 (Coating facility built and tested at STFC and INFN). There is a significant change from depositing superconducting films on flat surface with a planar magnetron that have been used in past within ARIES collaboration to applying all earlier obtained knowledge to coating 6-GHz cavities.

Two deposition facilities for coating 6-GHz cavities have been built and tested at STFC. First facility is using an outer coil for providing magnetic field for the magnetron and used for depositing the seamless 6-GHz cavities delivered from INFN. Another deposition facility is using an internal permanent magnet cylindrical magnetron and used for coating co-called split cavities developed in a collaboration between the University of Lancaster and UKRI/STFC. The results of first cavity coating on both facilities are described in this report.

A deposition facility for coating 6-GHz cavities has been built and tested at INFN/LNL in a post magnetron configuration with external coil. A second deposition system has been built and tested for the coating of QPR cavities and planar samples with 4” planar magnetron source.

Thus, the I.FAST Milestone 39 (Coating facility built and tested at STFC and INFN) has been met.

For more information on IFAST, its partners and contributors please see https://ifast-project.eu/

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Delivery Slip

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Executive summary

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A deposition facility for coating 6-GHz cavities has been built and tested at INFN/LNL in a post magnetron configuration with external coil. A second deposition system has been built and tested for the coating of QPR cavities and planar samples with 4" planar magnetron source.
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1 Introduction

Copper cavity coated with a thin Nb film (1.5 to 5-µm) on 500 MHz has been operational since 1980 in various particle accelerators [1]. However, their performance has only matched those of bulk Nb cavity at moderately accelerating gradient up to 8.6 MV/m. In the past two decades, due to advancement in thin film deposition technology and better understanding of surface preparation there has been coordinated effort by SRF community to push the performance of thin film SRF cavity to a level that can compete with bulk Nb at high-accelerating gradient of up to 20 MV/m.

Furthermore, this advancement allowed materials with a critical temperature (T_c) higher than one for Nb to be synthesised on copper substrates with matching superconducting properties of their respective bulk materials.

Using materials with T_c higher than for Nb, such as NbN, Nb_3Sn, NbTiN, MgB_2, etc., as well as multilayer structures allows to reach parameters that are unreachable for existing RF cavities:

- Increasing a quality factor Q reduces a heat produced and, hence, the electricity consumption of the cryogenic system during the RF cavity operation.
- Using high T_c superconducting materials allows to operate RF cavities at 4.2 K instead of 1.9 K used for the high-performance Nb cavities, more than doubling the efficiency of the cryogenic system.
- Increasing the cost-effective acceleration field E (at present the minimum cost is achieved at just over 30 MV/m) will result in massive saving in the infrastructure (tunnel, LHe supply and He recovery lines, electric cables, controllers, cryostats, pumps, etc), for example 20% increase in the acceleration field allows 20% reduction the acceleration line (compare: a 4-km long tunnel for EU-XFEL instead of a 5-km long one, or 50-km long tunnel for ILC instead of 60-km).

This improvement goes by a few routes.

(1) Other materials forms of Nb, known as A15, such as Mg_2B and Nb_3Sn or Nb_3Ti with a higher T_c, a potentially higher critical field H_c, leading to potentially significant cryogenics cost reduction if the cavity operation temperature is 4.2 K or higher. Single crystal high quality films have been achieved on single crystal MgO, sapphire and single crystal copper substrates,

but more effort is needed for translating these remarkable results onto real 3D geometry cavity.

(2) Multilayer film of Superconductor/ Insulator/Superconductor (SIS) that should provide much higher E_a than a single layer films.

(3) Improving or developing various deposition method to deposit desired quality films on curved cavity surfaces.

Currently the thin film SRF cavity production is based on PVD process where sputtering is the preferred method due to its ease of scalability, moderate conformability and above all its ability to control the film morphology and microstructures. The SRF thin film synthesis by sputtering process owes much of its success to being a single element thin film (mostly Nb). Synthesis of an alloy SRF thin film on a 3D geometry such as cavity is much more challenging.

At ASTeC (UKRI/STFC Daresbury Laboratory), alloy-superconducting material such as Nb_3Ge, Nb_3Sn, V_3Si, NbTi, NbTiN and NbN have been successfully synthesized on various flat substrates either using alloy target or by co-sputtering, i.e., by sputtering simultaneously two constituents on a temperature-controlled substrate. In co-sputtering the achieved composition is dependent on the relative positions of the target and the substrate. The perfect stoichiometry can then be obtained by manipulating these positions. However, the control of the stoichiometry may be difficult over the large areas of accelerating cavities, especially if the stoichiometry range for the A15 phase is narrow.

CVD and PECVD of either single (Nb) or alloy superconducting material (NbTiN and NbN) has also been used at ASTeC to deposit mainly on flat substrates. In this process, one or more precursors, present in vapor phase, chemically react and form a solid film on a substrate at the appropriate temperature. The deposition rate and the structure of the film depend on the temperature and the reagent concentration. The control of the temperature and gas flow uniformity over the entire cavity surface may be difficult with complex geometries.

Combination of the two-deposition process of PVD and CVD can overcome the shortfall of each individual process. The hybrid physical chemical vapor deposition (HPCVD) which combines physical and CVD has been shown to produce high quality MgB_2 thin film on various 2D flat substrates. The high temperature used in HPCVD favors excellent epitaxy and crystallinity, yielding RRR values in excess of 80.

Recently, we extended the HPCVD process to synthesis alloy superconducting thin film MgB2 on 3D geometry substrate by using magnetron sputtering for one of the elements of the alloy (Mg) using single element wire and provide the remaining element of the SC alloy (B) in vapor form [2]. The plasma from the magnetron sputtering facilitated the decomposition of the precursor and hence allow the chemical reaction to take place at much lower permitted temperature for a copper cavity.

Based on the success above the next step is to synthesis superconducting films on inner surface of RF cavity. For this purpose, we have designed a new set up to be able to deposit inner surface of a 6 GHz copper cavity at ASTeC.

At ASTeC, two type of 6 GHz copper cavity is used for this project. One is the traditional close copper cavity made by spinning at INFN and is delivered to ASTeC after being etched and polished and ready to be deposited. Another one is an open structure cavity designed by Lancaster University and ASTeC jointly and machined from a copper block. After production for the time being the cavities are sent to INFN for polishing and then returned to ASTeC for deposition and RF characterization.

Hence, at ASTeC we have designed two different deposition facilities for each cavity design. As well as the vacuum chamber design we had to design new type of cylindrical magnetron for variety of cavity sizes and shape. These magnetrons use either internal permanent magnet or use a magnetic field provided by DC coil.

At INFN cavity deposition facility exist for the past 2 decade [3, 4, 5] and been explored for Nb deposition routinely. The 6 GHz coating system has been updated and optimized last year for the coating of A15 materials. During the first year of iFAST a proof of concept of the possibility to realize a Nb_3Sn single use target by tin dipping has been successfully done [6] and will be scaled to 6 GHz cavities in the continuation of the project.

A new coating system for the deposition of Nb_3Sn on QPR samples has been designed, built and tested at INFN. First small samples present a Tc∼16 K measured by inductive method.

2 Deposition facilities

A total of four different facilities for superconducting film deposition in 6 GHz and QPR cavities have been realized and tested so far, two at ASTEC and two at INFN, respectively.

2.1 UKRI facility: Close 6 GHz copper cavity

Figure 1 shows the picture of the cavity deposition chamber (a), the closed cavity assembly flange (b and c). Due to a small aperture of the cavity iris (which is about 19 mm diameter), the magnetic field for magnetron is provided by an outer coil (see Fig. 1a). The entire deposition chamber is confined within the coil gap. The flange assembly is consisting of a donut plate shape which clamp the cavity to the CF200 flange. Six halogen lamps are mounted at the outer periphery of the plate to provide a heat for a high temperature deposition, a K-type thermocouple is also clamped to the cavity to measure the cavity temperature while is being heated. A heat shield consisting of three concentric stainless-steel cylinders separated by a 1-mm gap between adjacent cylinders. A 3-mm diameter Nb rod is inserted in the centre of the cavity which provide as deposition target. The deposition parameters are shown in Table 1.

The deposition carried out using Advance Energy pinnacle plus in pulsed DC mode to provide higher concurrent ion bombardment during deposition to produce high density thin film. Krypton was used for deposition gas.

The preliminary visual inspection showed that cavity was internally entirely coated with good adhesion. The cavity has been sent to INFN for the RF characterisation and waiting for the results.

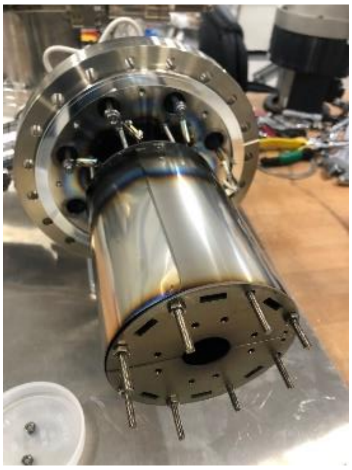
(a)  (b)  (c) 

Figure 1: A 6 GHz closed cavity deposition facility at UKRI: closed cavity deposition system (a), a cavity assembly flange without (b) and with a heat shield (c).

Table 1: Deposition parameter for Nb coating of first 6 GHz closed cavity.

Power	150 W
Current	0.23 A
Voltage	650 V
Pulse frequency	350 kHz
Duty cycle	1.1 μs
Deposition time	1 hour 30 min
Deposition pressure	2.6×10^{-2} mbar
Mag current	7.54 A
Deposition temperature	650 °C

2.2 UKRI FACILITY: OPEN 6 GHZ COPPER CAVITY

The cavity has been designed at Lancaster University in a collaboration with ASTeC. The advantage of such cavity is that it can be coated internally in both closed and open structure allowing variety of target to substrate geometry. It can be coated both with internal permanent magnet cylindrical magnetron or use the outer coil magnet magnetron construction. It can also be coated with planar magnetron used in flat sample substrate studies.

The first trial of deposition was done using an internal permanent magnet cylindrical magnetron as shown in Fig. 2(a). The magnetron is placed in the centre of cavity which is assembled on a CF200 SS flange in open structure with a gap of 60 mm between the two parts of the cavity. The magnetron is constructed with 99.95 Nb tube of 16 mm OD and 13 mm ID purchased from Goodfellows. One end of the tube is capped with Nb disk and the other side is welled to CF 38 SS flange. The tube is electron beam welded at both ends. Inside the Nb tube a series of 12 mm OD ring magnet is placed which is water cooled during deposition to keep the integrity of the magnet. The deposition parameter is shown in Table 2. Figure 2(b) shows visually the plasma distribution around the magnetron during deposition.

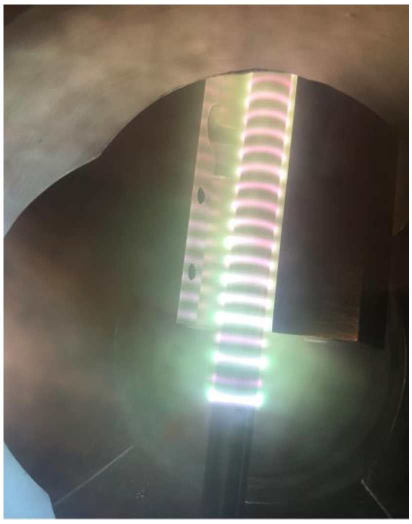
(a)  (b) 

Figure 2: A 6 GHz open cavity deposition facility at UKRI: (a) the flange assembly and (b) the plasma distribution around the magnetron during deposition.

Table 2: deposition parameter for Nb coating of first 6 GHz open cavity.

Power	300 W
Current	2.1 A
Voltage	143 V
Pulse frequency	350 kHz
Duty cycle	1.1 μs
Deposition time	3 hours
Deposition pressure	3.8×10^{-2} mbar
Deposition temperature	Room Temperature

Figure 3 shows the coated cavity after the deposition. Another advantage of this type cavity is that condition of the deposited film can be assessed visually immediately after deposition without any destruction to the cavity. It can be seen that the cavity is uniformly coated with high degree of adhesion in the cavity area.

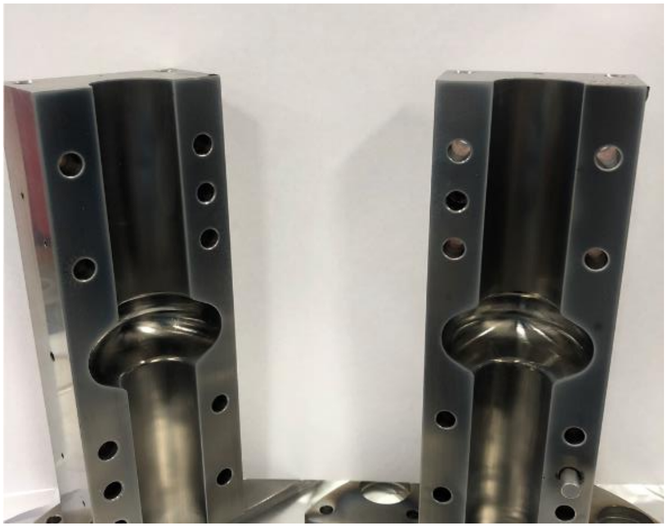


Figure 3: Nb coated open cavity.

2.3 INFN FACILITY: 6 GHZ COPPER CAVITY

In Fig. 4, the coating system for the 6 GHz cavities at LNL in a post-magnetron configuration is visible. The cavity inside the vacuum chamber is placed approximately at the centre of the coil, where the magnetic field lines are homogeneous and parallel to the cathode surface. The target (Nb or Nb_3Sn) is provided with its correspondent active water-cooling system that runs during the baking and sputtering processes. The system allows to work in an ultra-high vacuum (UHV) environment, in pressures lower than 5×10^{-9} mbar.

The dimensional constrain of the 6 GHz cavities imposed the design of a compact sputtering source, where cathode, 6 GHz cavity and IR lamp, are mounted together (with the appropriate electrical insulation) on a CF100 flange. The cavity is installed in half-disks and fix to the sputtering stand as showed in Fig. 5. The cavity is heated during the baking and the coating process by a circular infrared lamp. A post magnetron sputtering coating configuration has been chosen, since in the cylindrical symmetry it allows a high plasma confinement, fundamental for the low mean free path of electrons inside the 6 GHz cavity: in the cut off, distance between cathode and cavity is less than 5 mm.

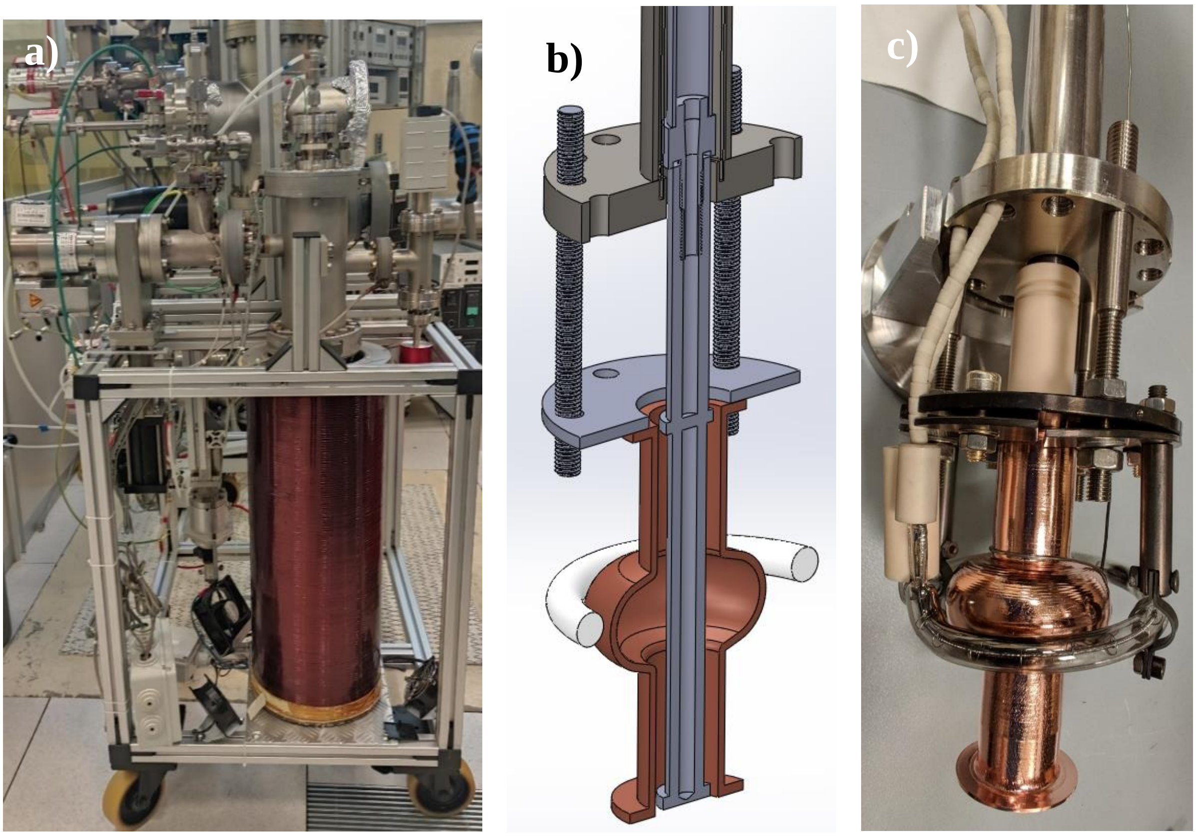


Figure 4: 6 GHz cavities coating facility at LNL. (a) Vacuum system and coil. (b) 3D section of the post magnetron. You can recognize the cooled cathode coaxial to the cavity and the IR ring lamps to heat the cavity (c) Cu cavity installed in sputtering stand.

The deposition parameters for the Nb coating on 6 GHz cavities are described in Table 3. The Argon process pressure of 5×10^{-2} mbar is the optimal working pressure demonstrated during stress tests performed, where Kapton foils exhibited zero-stress behaviour and overall, the best RF performances during previous studies [7]. For Nb, the substrate temperature during the sputtering process was selected to be 550 °C, and baking temperature 600 °C. Higher temperature can cause copper softening and damage the cavities. For the deposition of A15 SC it will be necessary to increase the temperature of the substrate and find the right balance between the best coating conditions and the preservation of the mechanical properties of the copper cavity. Due to the small space between the cathode and the anode (cavity) an unusual high magnetic field of 830 G is required for an appropriate plasma confinement.

Table 3: Coating parameters for Nb on Cu 6 GHz cavities at LNL.

Baking temperature	Coating temperature	Magnetic field	Ionization current	Argon pressure
600°C	550°C	830 G	1 A	5×10^{-2} mbar

A proof of concept of the possibility to realize a Nb_3Sn single use target by tin dipping has been successfully done and will be scaled to 6 GHz cavities in the continuation of the project. The results have been already presented as poster at last SRF conference [6]. The dipping system has been realized in 2005 [8] and upgrade during this project with a new Nb furnace (Fig. 5).

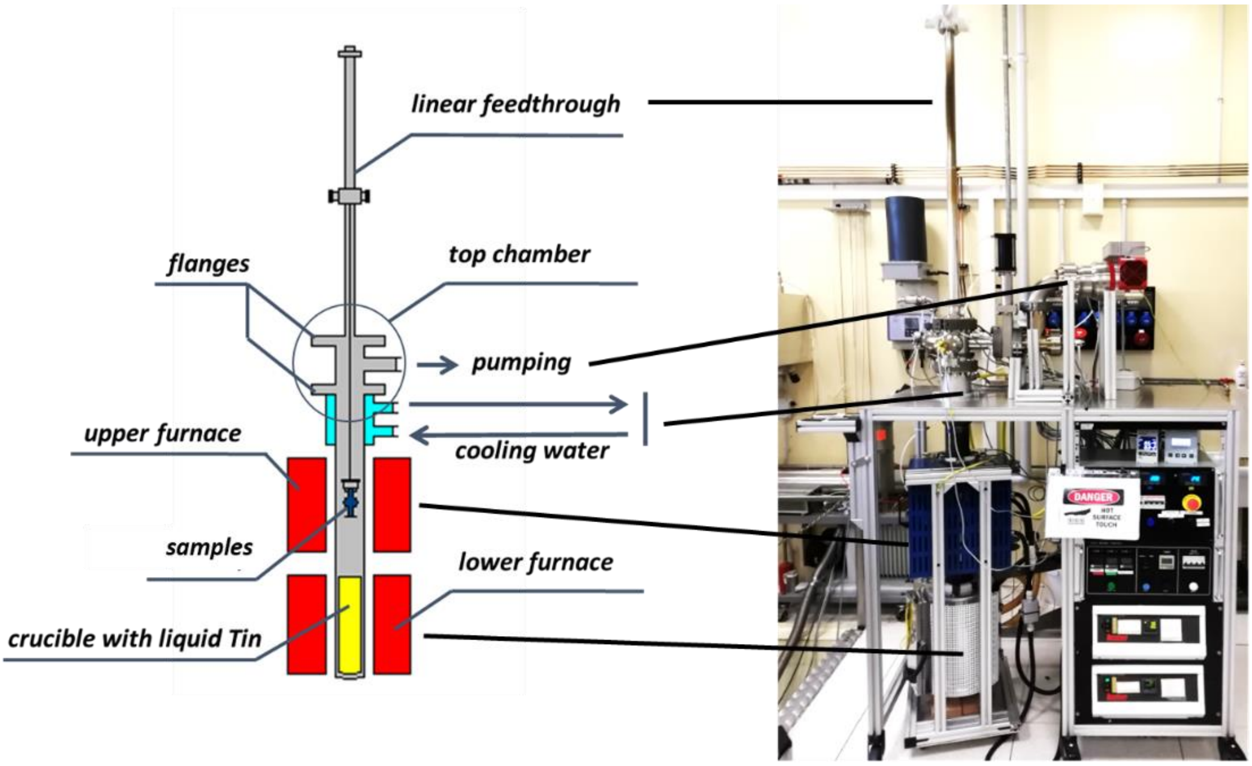


Figure 5: Dipping system used for the preparation of Nb_3Sn magnetron sputtering targets.

2.5 INFN FACILITY: QPR AND PLANAR SAMPLES

In INFN a new coating system has been designed, built and tested for the coating of QPRs and planar samples, fundamentals for the coating R&D in order to test new solutions in a simple configuration prior to scale in the complex cavities 3D geometries.

The coating system has been mounted in a new structure in which we have also included the system for the coating of 6 GHz, a second system with magnetron 4 "for the deposition of buffer layers of Nb (to prevent Cu contamination in Nb_3Sn films) and a chamber for post-annealing of the 6GHz cavities in UHV (see Fig. 6). Particular attention has been paid to the sample holder, that thanks to the use of 3 IR lamps allows to deposit at high temperature (up to 1000 °C) both QPR and small samples of copper, quartz or sapphire. Two different 4" magnetrons are available: a commercial one from MeiVacAnd an in-house magnetron developed in 2008 at LNL to provide a uniform target erosion, using two coaxial coils aligned to the target. First Nb_3Sn samples coated on sapphire show T_c~16 K measured by inductive methods (Fig. 7).

Figure 6: A15 Coating facilities at LNL dedicated to i.FAST project. (a) 3D drawing of the structure containing the 3 coating systems used (6 GHz cavity, Nb3Sn QPR, 4" Magnetron coating system used for Nb buffer layer coatings). (b) Picture of the coating facilities installed at LNL. (c) Cross-section of the sample holder used for QPR coating. You can see the 3 lamps that allow substrate temperatures up to 900°C.

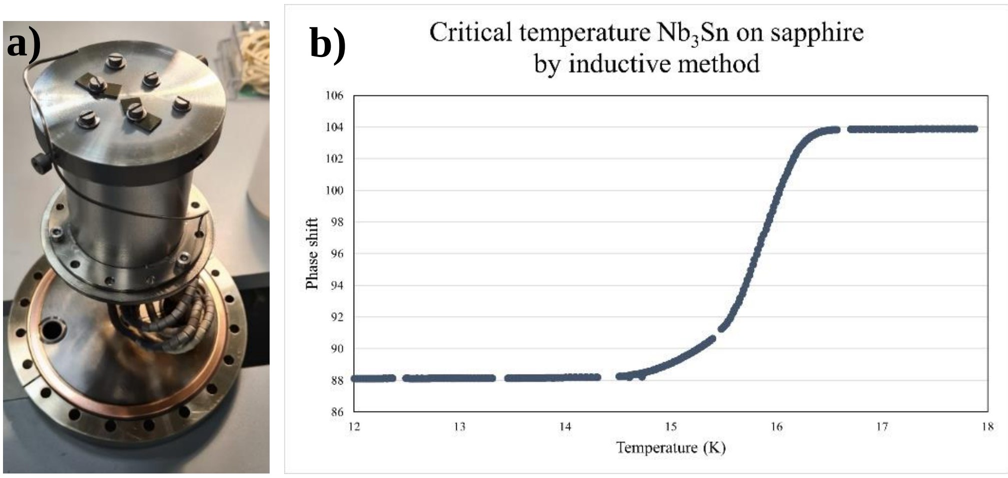


Figure 7: (a) First coatings of Nb_3Sn on sapphires. (b) Tc of the first samples by inductive methods.

3 A deposition of 6 GHz closed cavity with NbTiN

The main objective of IFAST project is to produce copper cavity coated with superconducting alloy A15 or B1 film with higher Tc than Nb. In line with this objective, we have deposited a closed cavity with NbTiN using a Nb rod target similar to one described in Section 2.1 but this time is raped with 1 mm diameter Ti wire. The deposition was done in a reactive environment of Kr (60%) and nitrogen (40%). The deposition parameters are shown in Table 4.

Table 4: Deposition parameter for NbTiN coating of first 6 GHz close cavity.

Power	150 W
Current	0.16 A
Voltage	644 V
Pulse frequency	350 kHz
Duty cycle	1.1 µs
Deposition time	1 hour 30 min
Deposition pressure	3.6×10^{-2} mbar
Mag current	7.51 A
Deposition temperature	650

The cavity originally was used for commissioning the deposition facility. Hence prior to NbTiN deposition a thin layer of Nb was deposited. The second layer of NbTiN was deposited several weeks after on Nb coated copper cavity. After deposition, the cavity was cut in two section and is shown in Fig. 8. It can be seen that the cavity is uniformly coated with golden colour which is signature colour related to transition metal nitride.

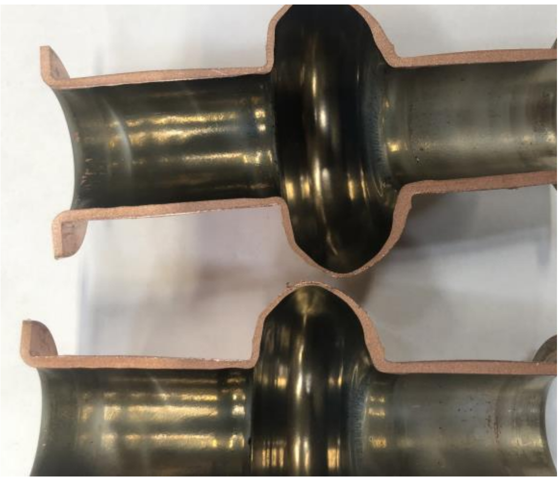


Figure 8: A 6-GHz closed copper cavity deposited with double layer Nb/NbTiN.

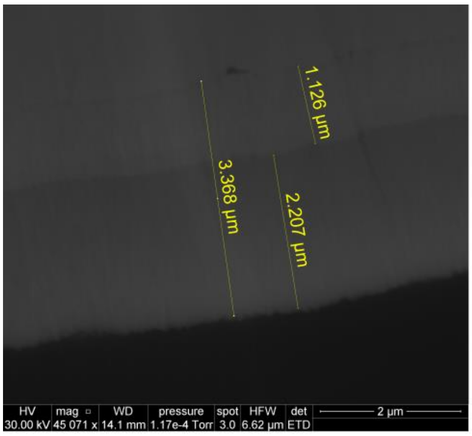


Figure 9: X-section SEM of dual Layer Nb/NbTiN

Consequently, several pieces from the cavity equator is cut for analysis. The X-section SEM of such section is shown in Fig. 9. It illustrates that the film transition between each layer is smooth and no significant damage layer at Nb and NbTiN interface is observed taking despite fact that the NbTiN layer was deposited post deposition of Nb and after the Nb layer was exposed to air. The Nb layer thickness is estimated to be about 1.1 μm and the NbTiN layer is about 2.2 μm.

The ratio of Nb to Ti determined by EDX analysis was 50:50. The critical temperature determined by squid magnetometer is found to be around 15 K with slow transition which can be consequence of non-uniform composition this shown in Fig. 10 (b). The first field of penetration is about 30 mT and the H_{c2} is higher than the range set in the squid measurement. The magnetisation curve is free of any flux jumping illustrating the film is free for any deep level defects as shown in Fig. 10 (a).

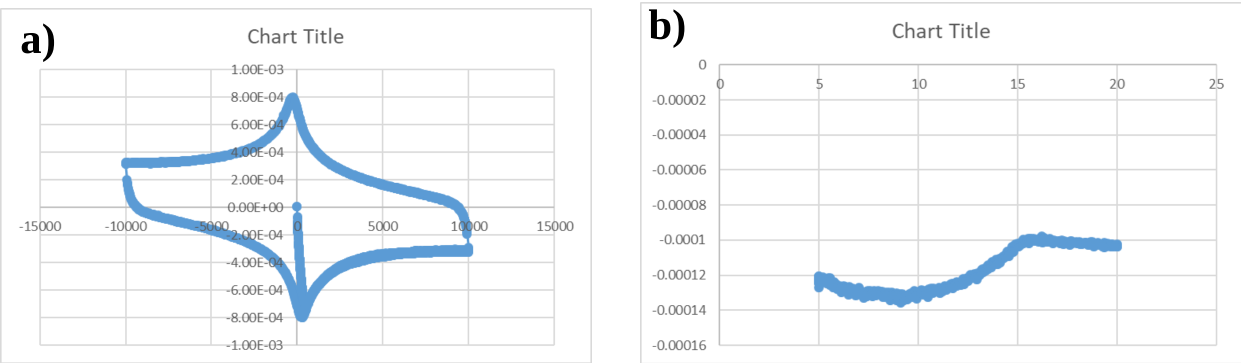


Figure 10: (a) magnetization curve measured at 4 K and (b) magntization under contant 100 Oe for varing temperture from 20 to 5 K.

4 Future plans / Conclusion / relation to other IFAST work

4.1 Conclusion

Report is related to achieving I.FAST Milestone 39 (Coating facility built and tested at STFC and INFN). Two deposition facilities for coating 6-GHz cavities has been built and tested at STFC. First facility is using an outer coil for providing magnetic field for the magnetron and used for depositing the seamless 6-GHz cavities delivered from INFN. Another deposition facility is using an internal permanent magnet cylindrical magnetron and used for coating co-called split cavities developed in a collaboration between the University of Lancaster and UKRI/STFC. The results of first cavity coating on both facilities are described in this report.

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Thus, the I.FAST Milestone 39 (Coating facility built and tested at STFC and INFN) has been met.

4.2 FUTURE PLANS / RELATION TO OTHER IFAST WORK

Achieving this Milestone is demonstrating a significant change from depositing superconducting films on flat surface with a planar magnetron that have been used in past within ARIES collaboration to depositing curved surface of real RF cavity. This allows the Task team to go to the next stage: depositing various superconducting thin films onto 6-GHz cavities and testing the RF properties of these cavities at cryogenic temperatures. Following the Task 9.3 programme, different superconducting materials with a critical temperature T_c higher than the one for Nb will be explored, for example Nb_3Sn, NbTiN and the other materials that were investigated in ARIES WP15 and beyond. SIS structures are also going to be explored. These studies will lead to the Task 9.3 delivery (D9.3**:** First 6 GHz cavity coated and characterized) And the main purpose of the task: finding the coatings that allows to produce cavities with the RF properties better than for bulk or coated Nb.
Task 9.3 (Development) is directly connected to Task 9.2 (Prototype). Optimising the deposition process with 6-GHz cavities allows to reduce the costs in comparison to 1.3 GHz cavity: smaller cavity requires less copper, quicker production, less chemistry use for surface polishing, smaller deposition chamber, less usage of the deposition target and less use of expensive LHe for the RF test. After defining the most proposing film coating with 6 GHz cavity, these results will be applied to deposit a 1.3 GHz cavity within Task 9.2.

5 References

[1] Benvenuti C. et al., (1984) Niobium films for superconducting accelerating cavities, *Appl. Phys. Lett.* (45) 583.

[2] Misra N., Valizadeh R., Kumar V., (2020) Hybrid Physical Chemical Vapour Deposition (HPCVD) of Superconducting MgB_2 Thin Films on three-Dimensional Copper Substrates, *Adv. Mater*. *Lett.* (11)11, 1-6.

[3] Palmieri V. et al., (1991) Sputtering of Niobium Thin Films onto Copper Quarter Wave Resonators, Proceedings of SRF91, Hamburg, Germany, SRF91E11.

[4] Lanza G. et al., (2006) New magnetron configurations for sputtered Nb onto Cu, *Physica C: Superconductivity*, (441), 102–107.

[5] Garcia V. et al., (2021) Thick film morphology and SC characterizations of 6 GHz Nb/Cu cavities, Proceedings of SRF2021, East Lansing, MI, USA, SUPCAV007.

[6] Zanierato M. et al., (2021) Nb_3Sn films depositions from targets synthesized via liquid tin diffusion, Proceedings of SRF2021, East Lansing, MI, USA, TUPCAV006.

[7] Palmieri V. et al., (2017) The way of thick films toward a flat Q-curve in sputtered cavities Proceedings of SRF2017, Lanzhou, China, TUYBA03.

[8] Deambrosis S. M. et al., (2006) A15 superconductors: An alternative to niobium for RF cavities, *Physica C: Superconductivity*, (441), 108–113.

Annex: Glossary

Acronym	**Definition**
AFM	Atomic Force Microscope
CEA	Saclay Nuclear Research Centre - Commissariat à l'Energie Atomique
CERN	European Council for Nuclear Research
EDS	Energy Dispersive X-ray Spectrometry
EP	Electropolishing
HIPIMS	High power impulse magnetron sputtering
HZB	Helmholtz-Zentrum Berlin
IEE	Institute of Electrical Engineering Slovak Academy of Sciences, Bratislava
INFN-LNL	Italian Institute of Nuclear Physics - Legnaro National Laboratories
OFE	Oxygen Free Electronic copper
OFHC	Oxygen-Free High thermal Conductivity copper
PPMS	Physical Property Measurement System
QPR	Quadrupole Resonator
QWR	Quarter Wave Resonator
RF	Radio Frequency
RTU	Riga Technical University
SC	Superconductivity
SEM	Scanning Electron Microscope
SRF	Superconducting Radio Frequency
SUBU	Chemical Polishing of Cu with a solution of Sulphamic Acid and Butanol
UKRI/STFC/DL	United Kingdom Research and Innovation / Science and Technology Facilities Council / Daresbury Laboratory
T_c	Critical temperature of superconducting transition (thermodynamic)
TF	Thin films
XRD	X-Ray Diffraction