Open St Com meeting, 15-16 Nov 2021

Oleg B. Malyshev (UKRI) / Claire Antoine (CEA)

WP9 coordinators

WP9 main goal and objectives

➢Main goal:

- **Improving the performance and reducing the cost of acceleration systems**
  - both production and operation

➢Objectives:

- Define a strategy for innovative superconducting RF (SRF) cavities coated with a superconducting film.
  - Deposition techniques: PVD and ALD
  - Superconducting films: Nb, NbN, Nb_3Sn, V_3Si (and others) and SIS
  - Optimization of flat SRF thin films production procedure
- Optimise and industrialise the production
  - of seamless copper cavities and
  - of the deposition techniques.
- Produce and test prototypes of SRF (single-cell elliptical) cavities:
  - Initially with pre-prototypes with f = 6 and 3 GHz
  - Scaling up for f = 1.3 GHz.
- Test a new laser treatment of Nb coated cavity.

IFAST WP9:

- 9 countries
- 15 institutes
- >50 participants

	IFAST WP9 Partners	Leading	Participating
1	CEA (Saclay, France)	WP, Tasks 1 and 4	Task **1**, 2, **4**, 6
2	CNRS/IN2P3/IJCLab (Orsay, France)		Task 1, 4
3	IEE-SAS (Bratislava, Slovakia)		Tasks 2-6
4	INFN/LNL (Legnaro, Italy)	Task 2	Tasks 1, **2**, 3, 5, 6
5	*INFN/LASA (Milano, Italy)*		Tasks 2, 3
6	*Piccoli S.r.l. (Noale (VE), Italy)*		Tasks 2, 3
7	Helmholtz-Zentrum Berlin (Berlin, Germany)	Task 6	Tasks 1 and **6**
8	RTU (Riga, Latvia)	Task 5	Task **5**
9	University Siegen, (Siegen, Germany)		Tasks 2, 3, 6
10	UKRI/STFC/ASTeC (Daresbury, UK)	WP, Tasks 1 and 3	Tasks 1, 2, **3**, 5, 6
11	*Lancaster University (Lancaster, UK)*		Tasks 1 – 3, 6
12	*Jlab (Newport News, Virginia, USA)*		Tasks 1, 2
13	*PTI (Physics-Polytechnic Institute, Minsk, Belarus)*		Tasks 1, 2
14	*MEPHI (National Research Nuclear University, Moscow, Russia)*		Tasks 1 - 3
15	*Helmholtz-Zentrum Dresden-Rossendorf (Dresden, Germany)*		Tasks 1 – 3, 5

WP9 tasks

- **Task 9.1**: Coordination and strategy for innovative superconducting accelerating cavities
  - *CEA, INFN, HZB, UKRI, USI, JLab MEPHI, PTI.*
    - *Task Leaders: C. Antoine (CEA), O. Malyshev (UKRI)*
- **Task 9.2**: Innovative SC accelerating cavity prototype
  - *INFN-LNL, INFN-LASA, PICCOLI, UKRI, USI, CEA, IEE, HZB, PTI, MEPHI*
    - *Task Leader: C. Pira (INFN)*
- **Task 9.3** : Optimisation of process parameters and target development for SRF cavity coating with A15 material
  - *UKRI, INFN, IEE, USI, HZB, MEPHI, HZDR*
    - *Task Leader: R. Valizadeh (UKRI)*
- **Task 9.4**: Surface engineering by atomic layer deposition (ALD)
  - *CEA, CNRS*
    - *Task Leader: T. Proslier (CEA)*
- **Task 9.5**: Improvement of mechanical and superconducting properties of RF resonator by laser radiation
  - *RTU, UKRI, INFN, IEE, HZB*
    - *Task Leader: A. Medvids (RTU)*
- **Task 9.6**: Optimization of flat SRF thin films production procedure
  - *HZB, INFN, UKRI, USI, CEA*
    - *Task Leader: O. Kugeler (HZB)*

Task 9.1: Coordination and strategy for innovative superconducting accelerating cavities

(CEA, INFN, HZB, UKRI, USI, JLab MEPHI, PTI). Task Leaders: C. Antoine (CEA), O. Malyshev (UKRI)

- 1st IFAST WP9 (kick-off) meeting took place on 5th May 2021:
  - Started with an introduction of each partners (i.e. institute, laboratory or university).
    - main interest of his team in this collaboration and
    - what expertise, equipment and samples (cavities) they bring to the IFAST WP9 collaboration.
  - Then each Task leader reported a vision on the Task followed by a discussion on
    - Task, milestones and deliverables,
    - A role of each partner,
    - Communication with other tasks,
    - A provisional plan for 1st year.
- 2nd IFAST WP9 meeting took place on 24th Sep 2021
- 3rd IFAST WP9 meeting will take place on 18th November 2021
- Other informal meetings whenever is necessary by the partners

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This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under GA No 101004730.

Task 9.2

Innovative SC accelerating cavity prototype

Task Leader: Cristian Pira (INFN)

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Task 9.2 Objectives

- Optimize and industrialize the **manufacturing of seamless elliptical copper cavities**

MS38: First seamless copper 1.3 GHz cavity produced as substrate for the coating of the SC film (Report)	**M12**

- Demonstrate the possibility to replace the current Nb bulk technology with an innovative **SRF cavity coated with a superconducting film**

D9.2: RF test on coated resonant cavity. *Resonant cavity coated and tested with an alternative material to Niobium with a $Q_0 > 10^9$ at 4.2 K and 1.3 GHz.*	M46

Strongly connected to Task 9.3

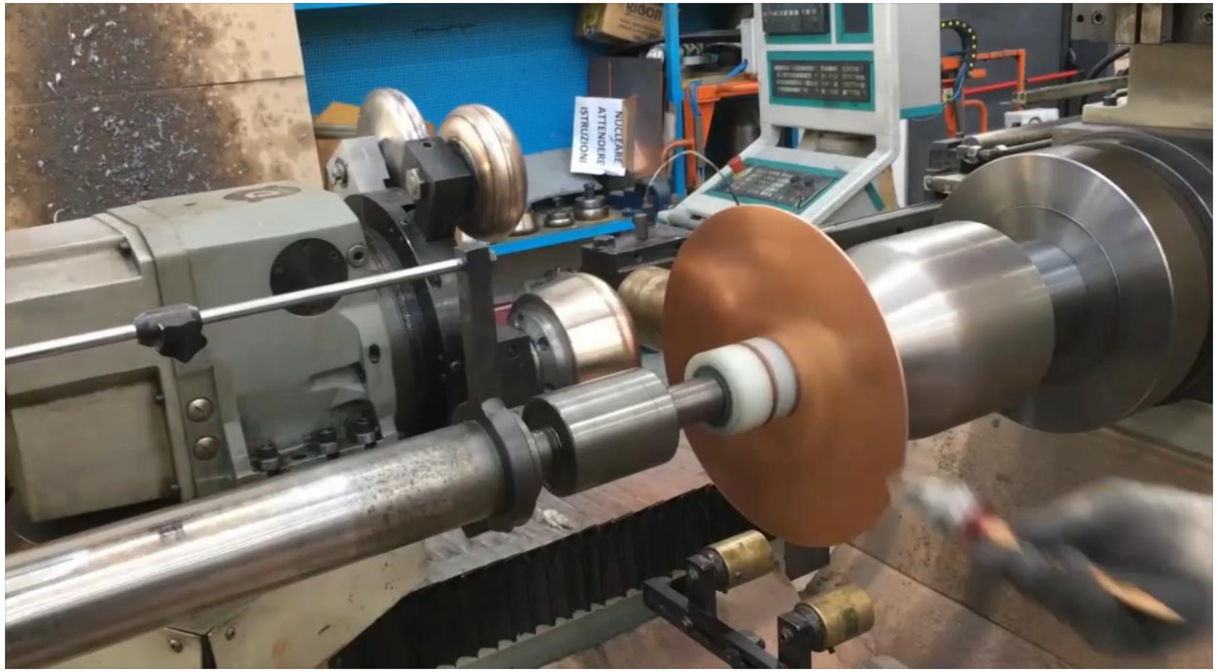
Seamless elliptical copper cavities

GOALS:

- Move cavity forming process from semi-automatic to fully automatic using CNC machine
- Study annealing temperature effect on formability
- Test reproducibility (in progress)

CNC Machine

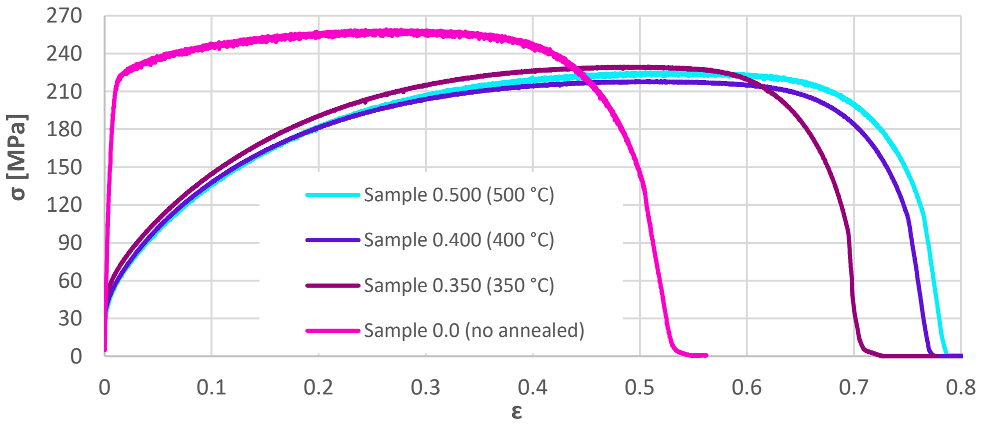
Demonstrated the possibility to use CNC machine to spun a 1.3 GHz cavities



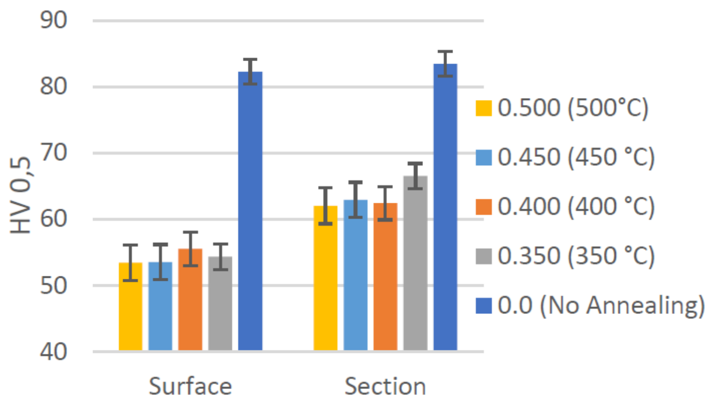
4 step process

Annealing Temperature

- T = 500 °C is a standard LNL annealing treatment
- **Reduce T →** Reduce grain dimension → **Increase mechanical properties**
- Test on small sample first



stress strain curve @ different annealing T



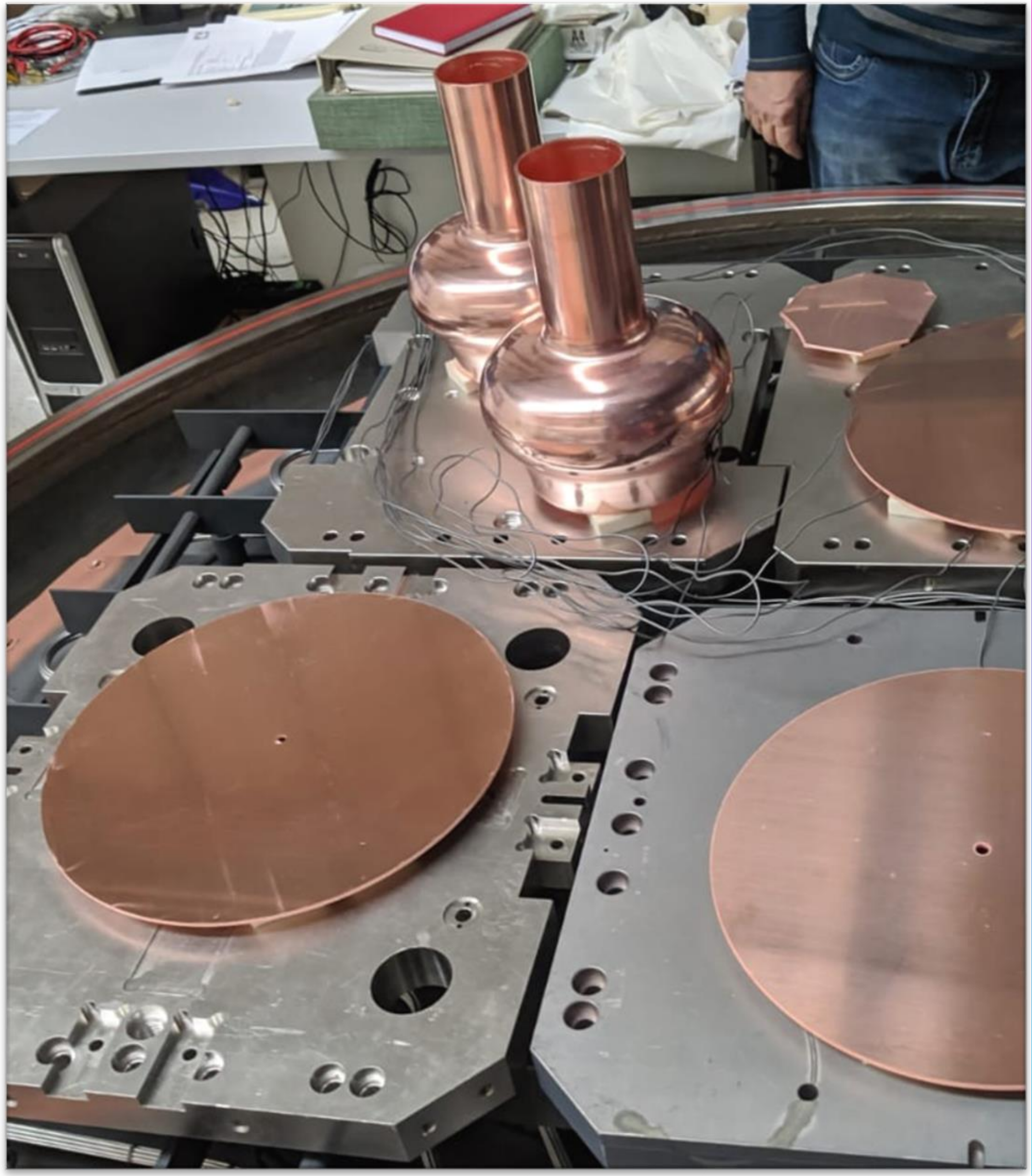
Micro hardness @ different annealing T

Results:

400 °C comparable to 500 °C

Test on real cavities

- 18 cavities realized
- 2 annealing T: 500 and 400°C
- No visible difference in forming processes: **no intermediate annealing necessary**
- Waiting characterizations (mechanical and metrology)



Next steps (6 months)

→Optimize CNC program and procedure for a full cavity

→Buy Cu OFE sheets for cavity production @ Piccoli/INFN (seamless) and PTI (EBW)

  →Waiting Piccoli pre-financing

→New polishing implant commissioning @ INFN

→Preparing MOU with PTI (cavity production)and JLab (EBW/polishing)

→Planning of coating equipment and development and CAD Design of 1.3 GHz coating facility @ INFN, UKRI, UniSiegen

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Task 9.3

Optimisation of process parameters and target development for SRF cavity coating with A15 material

Task Leader: Reza Valizadeh (UKRI)

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Task 9.3 Part 1: Cavity Coating and Evaluation

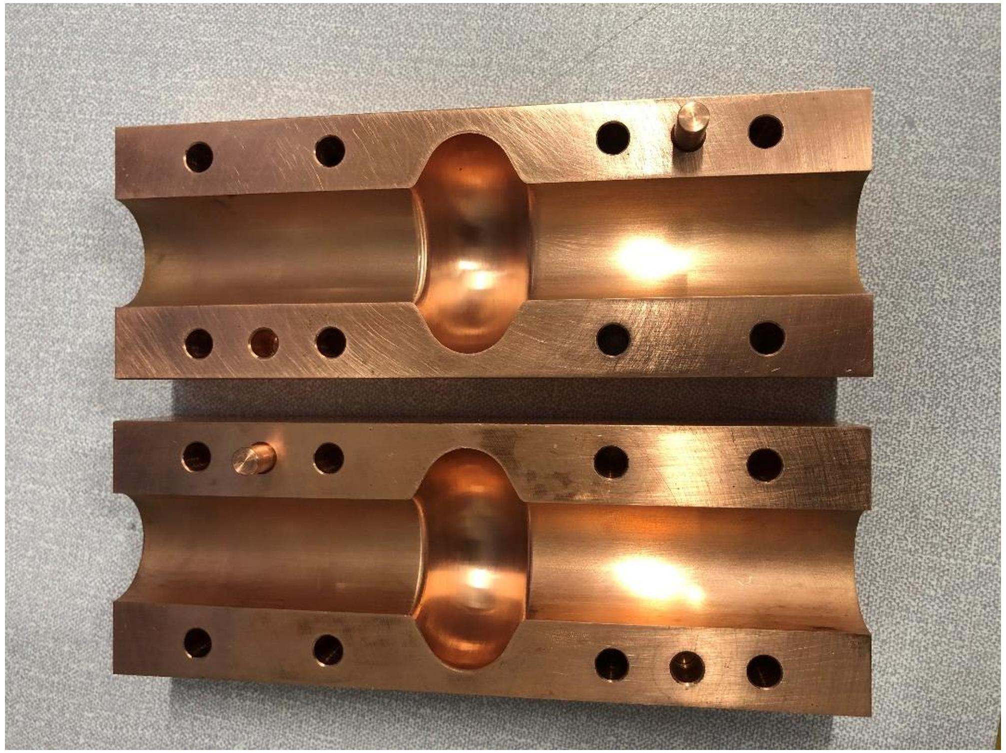
Aim:

- Evaluation of SRF performance of high T_c superconductor thin film by deposition of high T_c superconductor inside a 6-GHz copper cavity.
  - Nb_3Sn, NbTiN, NbN, MgB_2

6 GHz copper cavity

- Two type of cavity is going to be explored at UKRI/STFC/DL

INFN seamless standard elliptical copper cavity

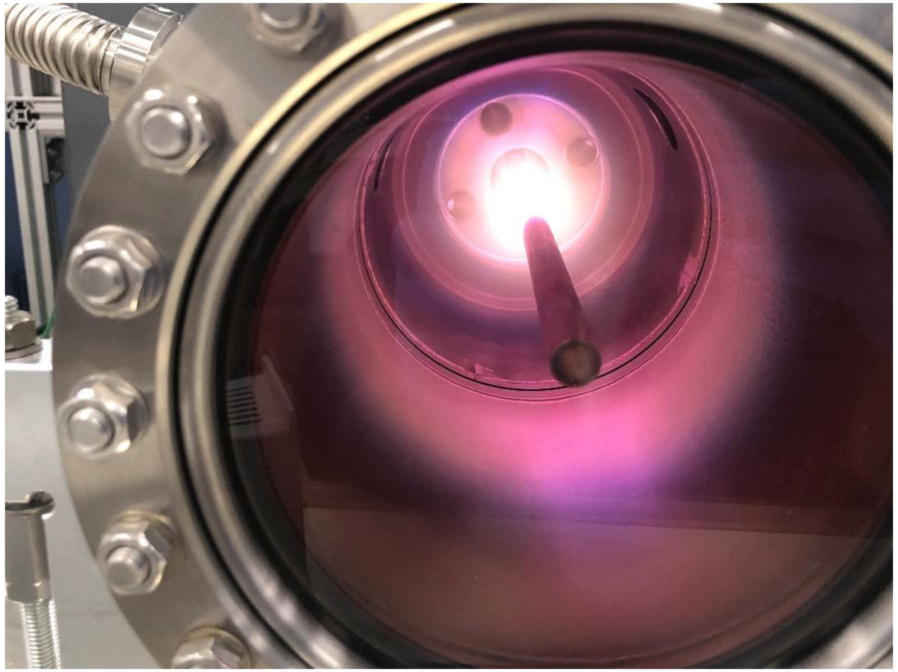


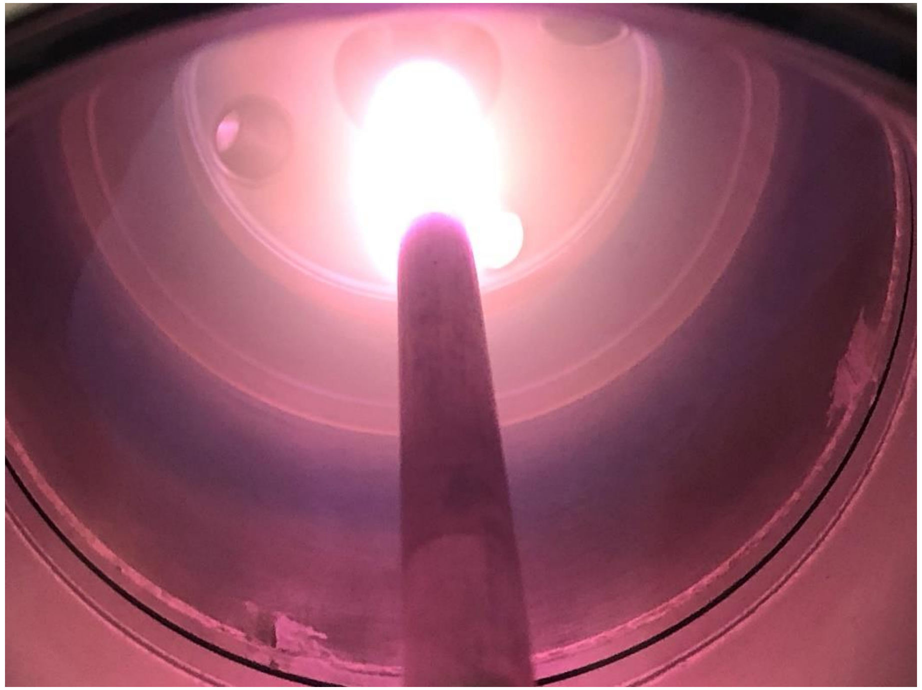
Lancaster University / STFC spilt cavity design

Deposition facilities: Hybrid physical chemical vapour deposition (HPCVD)

- Single or twisted multiple wires with outer magnetic field.

Deposition facilities:
Permanent Magnet Cylindrical Magnetrons





- Two niobium cylindrical magnetrons with water cooled internal permanent magnet are manufactured (OD = 13 and 19 mm) which were tested and ready to be implemented.

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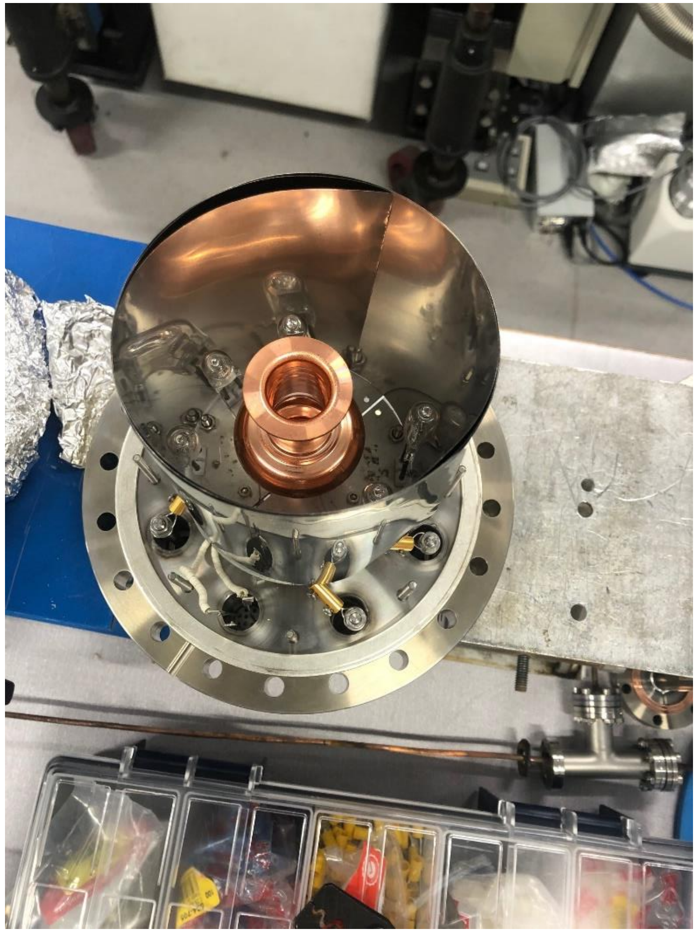
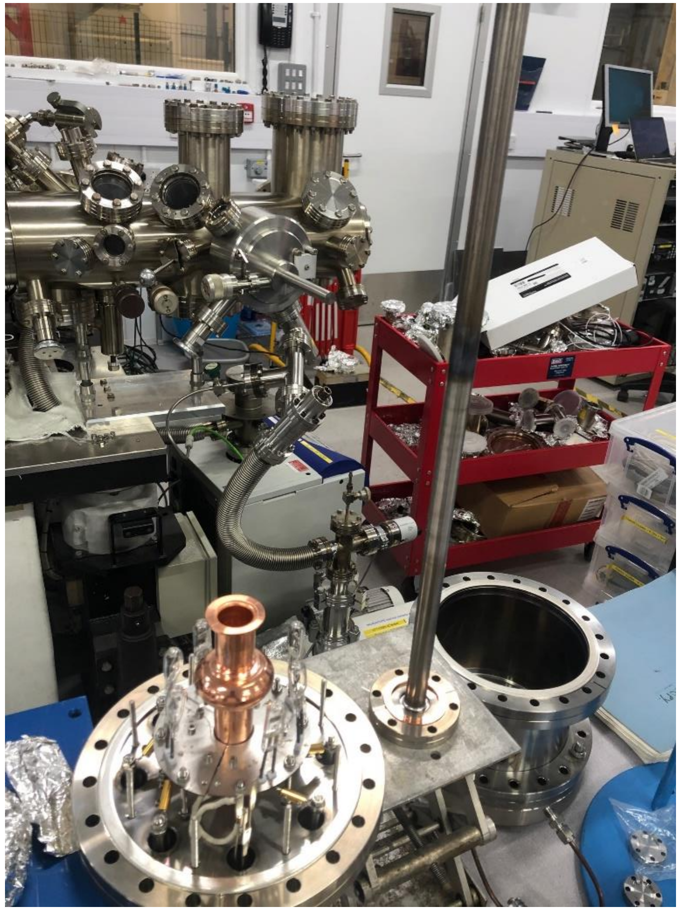
Deposition facilities:
6 GHz cavity mount for deposition





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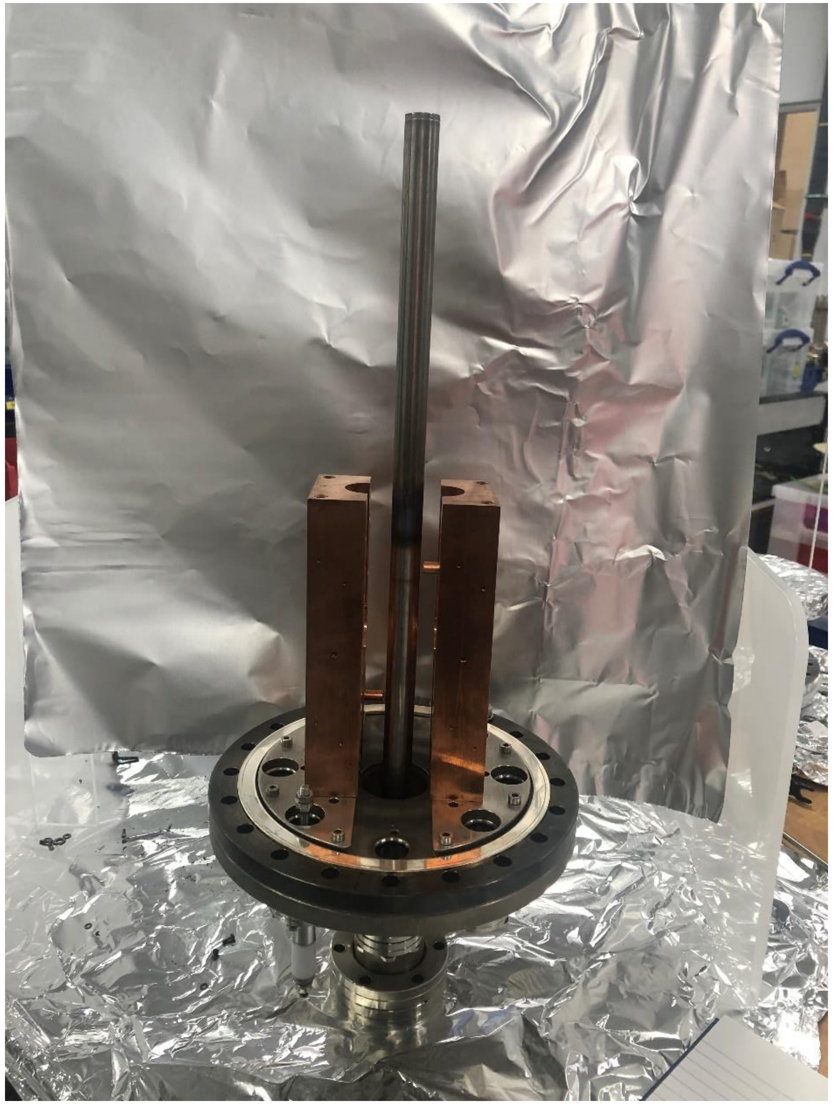
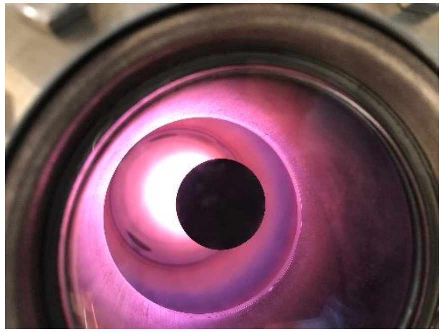
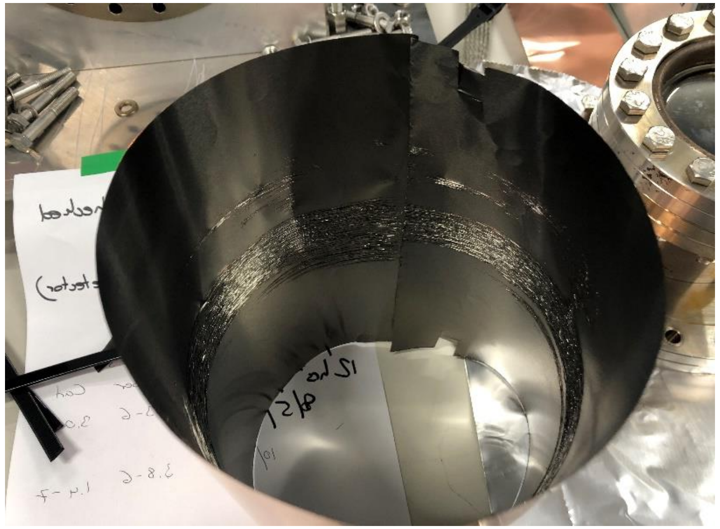
Deposition facilities:
Open 6 GHz Cavity assembly with Magnetron



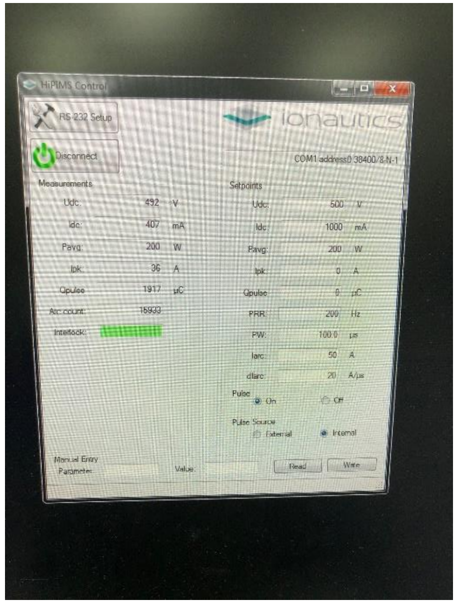
Deposition facilities:
Large cylindrical Magnetron

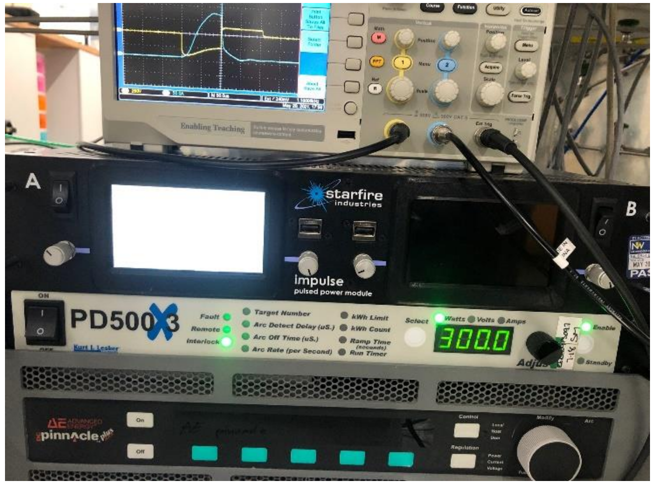


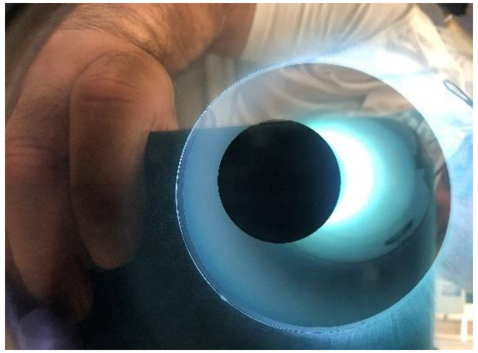
DC and pulse DC sputtering

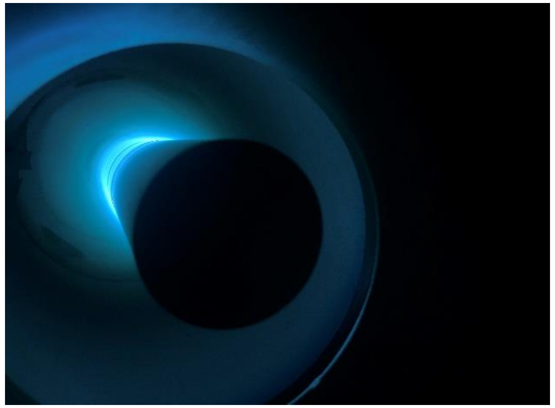


Film deposited along the length of active magnetron area. Some condition the centre delaminated









Various HIPIMS supplies with + kick and no bias

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Task 9.3 Part Two: Planar Samples& QPR deposition

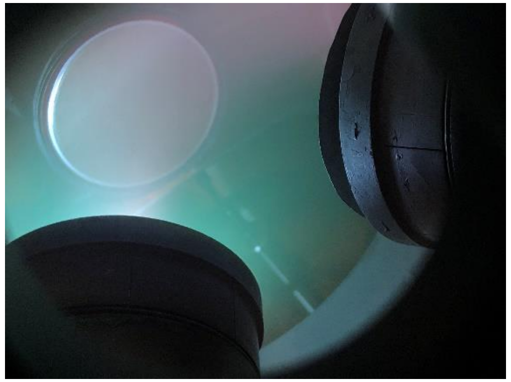
- Aim: optimise deposition parameters for other high T_c superconductor and provide sample for other partners for SRF evaluation of the SRF thin Films

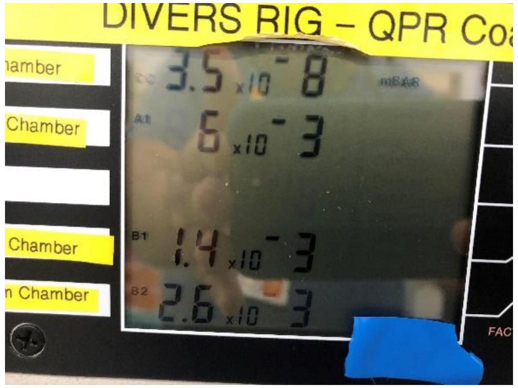
QPR Deposition Nb/AlN/NbN

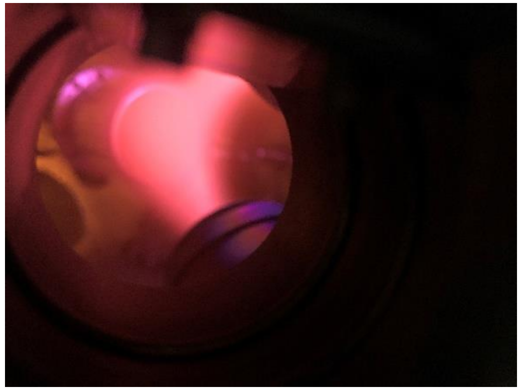
Nb Layer:
Power: 400W Pulsed DC
Current: 1.54 A Voltage: 260 V
F: 350 KHz and DT: 1.1 µs
Time: 5.5 hours
Thickness: 4 µm
Gas: Kr Pressure: $2x10^{-3}$ mbar

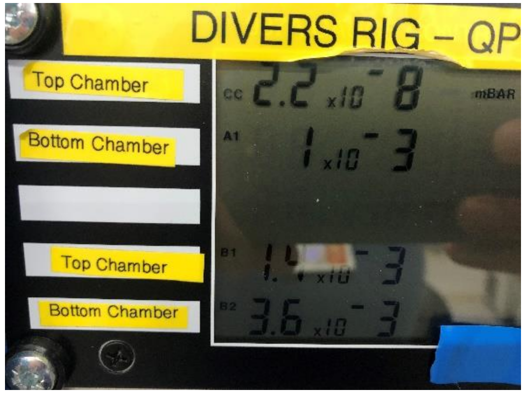
AlN Layer
Power: 200 W Pulsed DC
Current: 0.56 A Voltage: 357 V
F: 350 KHz and DT: 1.1 µs
Time: 1.5 min
Thickness: 3nm
Gas: Kr/N2 Pressure: $3.6x10^{-3}$ mbar

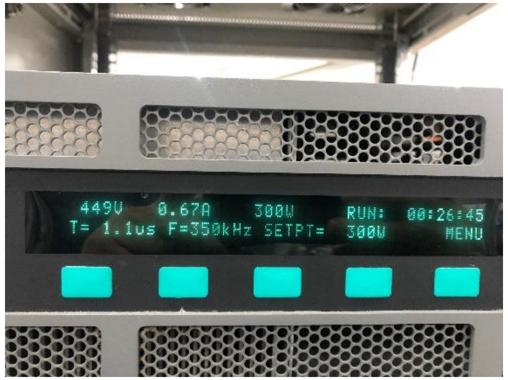
NbN depositon:
Power: 300W Pulsed DC
Current: 0.67 A Voltage: 449 V
F: 350 KHz and DT: 1.1 µs
Time: 40 min
Thickness: 180 to 200 nm
Gas: Kr/N2 Pressure: $3.6x10^{-3}$ mbar

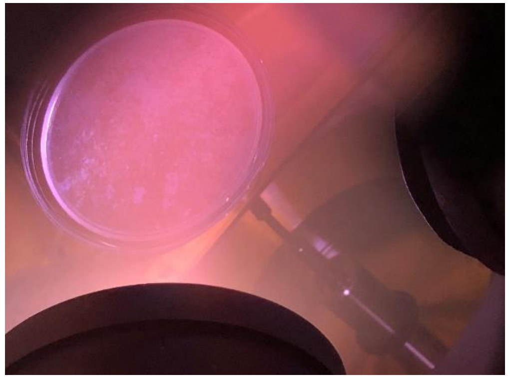


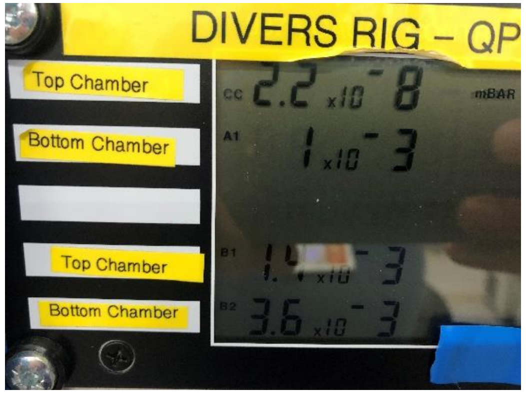




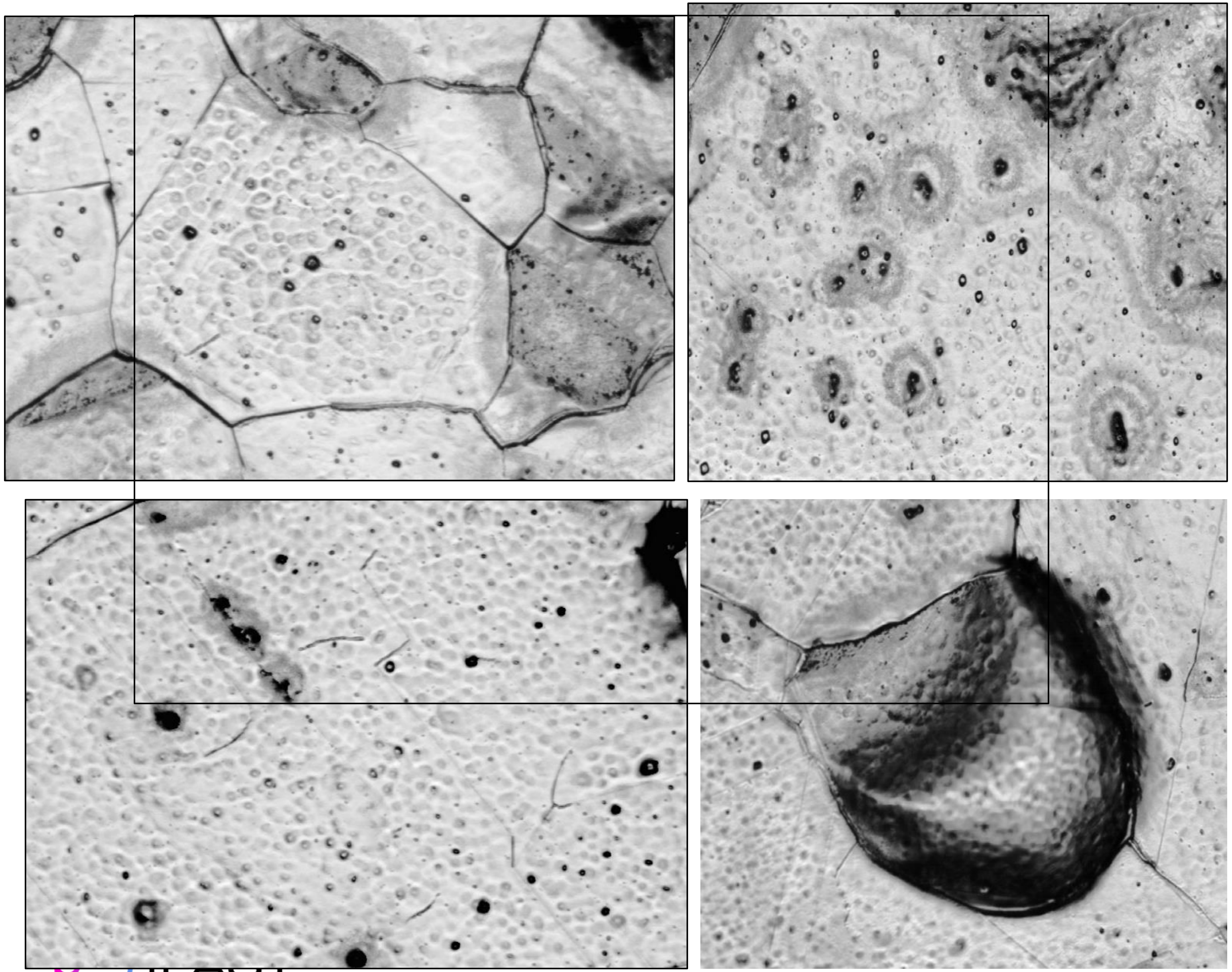








QPR after deposition

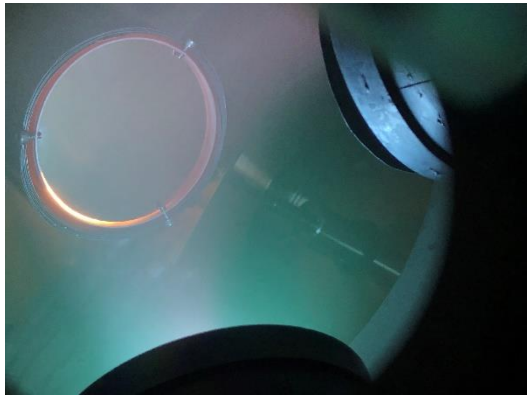


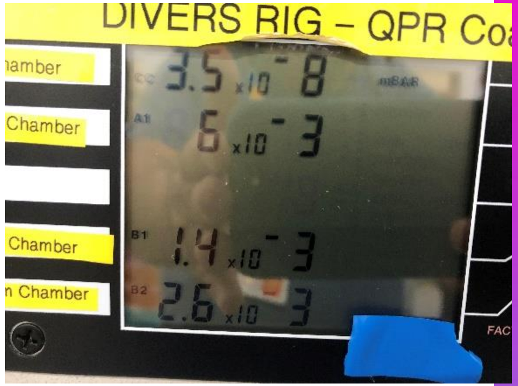
Diamond turned Cu Nb/AlN/NbN

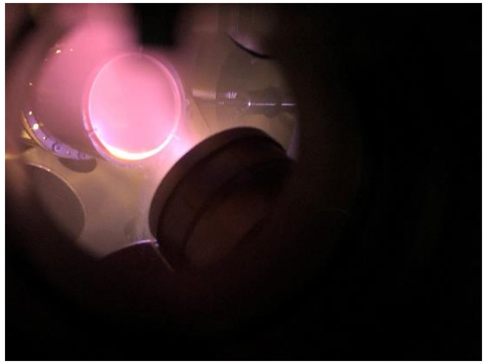
Nb Layer:
Power: 400W Pulsed DC
Current: 1.69 A Voltage: 237 V
F: 350 KHz and DT: 1.1 μs
Time: 5.5 hours
Thickness: 4 μm
Gas: Kr Pressure: 2.6x10^{-3} mbar

AlN Layer
Power: 200 W Pulsed DC
Current: 0.56 A Voltage: 357 V
F: 350 KHz and DT: 1.1 μs
Time: 1.5 min
Thickness: 3nm
Gas: Kr/N2 Pressure: 4.3x10^{-3} mbar

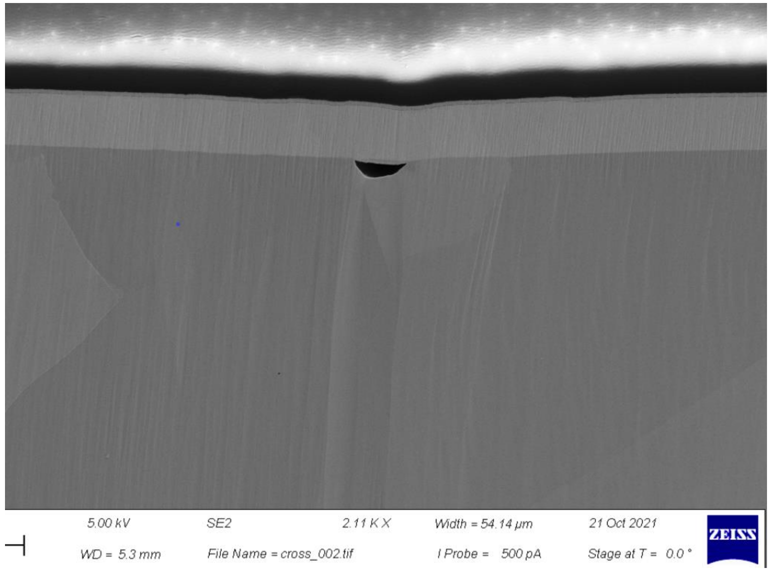
NbN depositon:
Power: 300W Pulsed DC
Current: 0.77 A Voltage: 392 V
F: 350 KHz and DT: 1.1 μs
Time: 1h 20 min
Thickness: 180 to 200 nm
Gas: Kr/N2 Pressure: 4.3x10^{-3} mbar

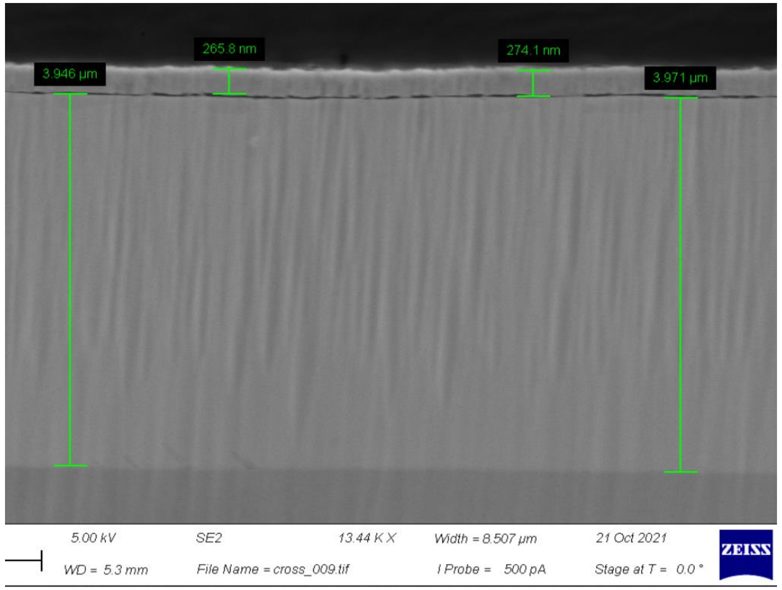


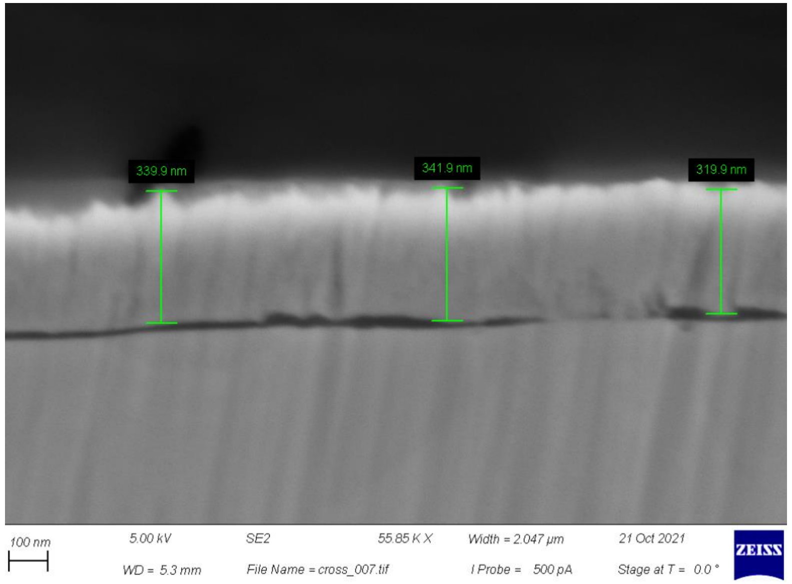


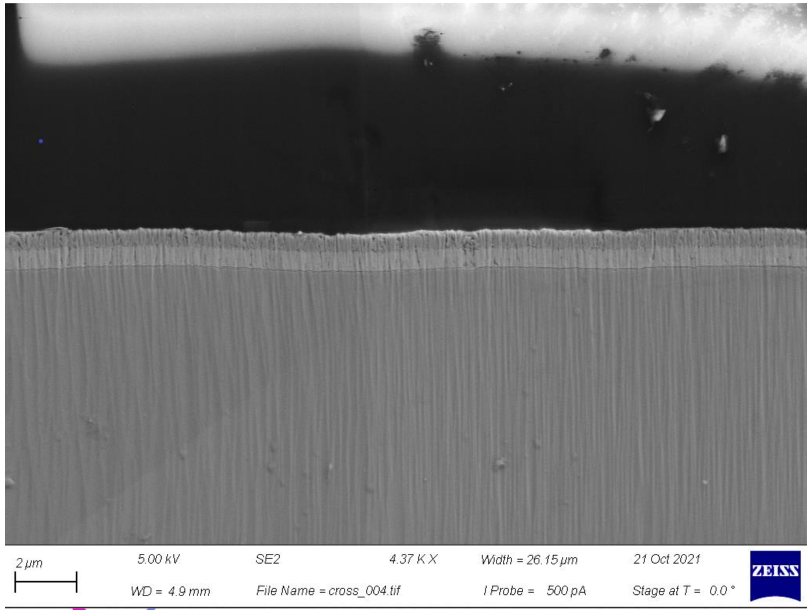


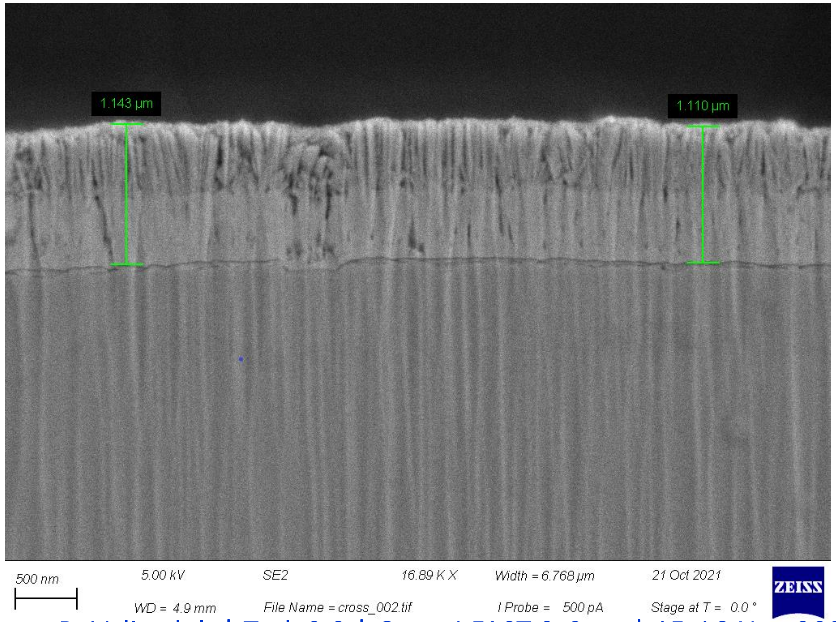
Cross section SEM of triple and double layer NbN

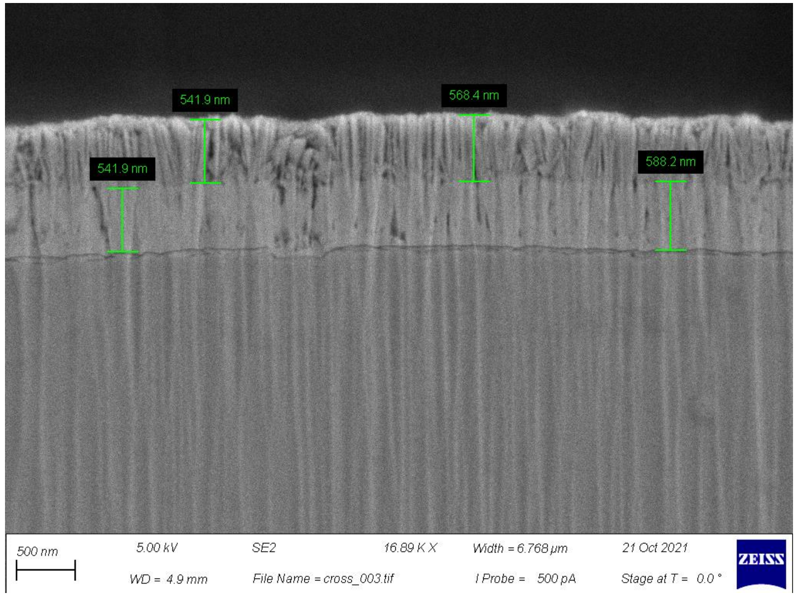






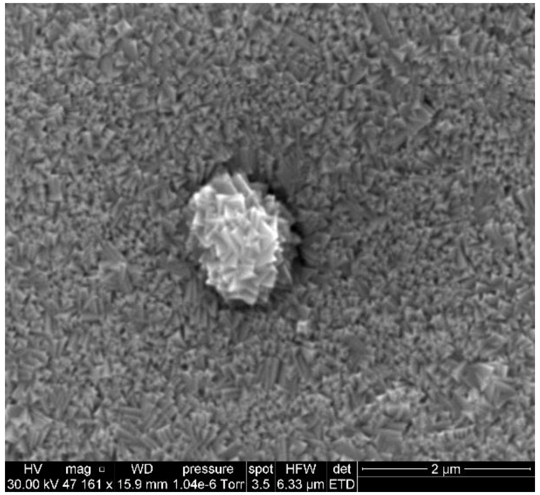




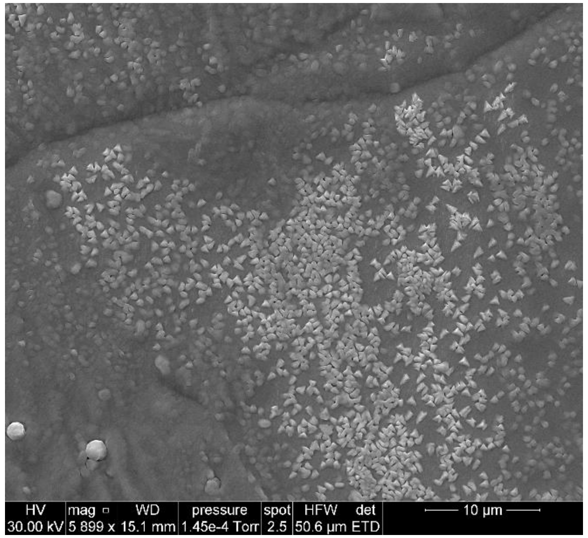


SEM and EDS

Addition of SEM with EDS analysis to increase our efficiency

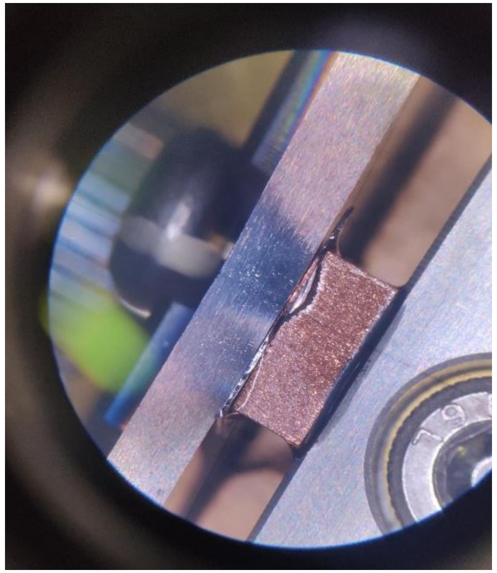


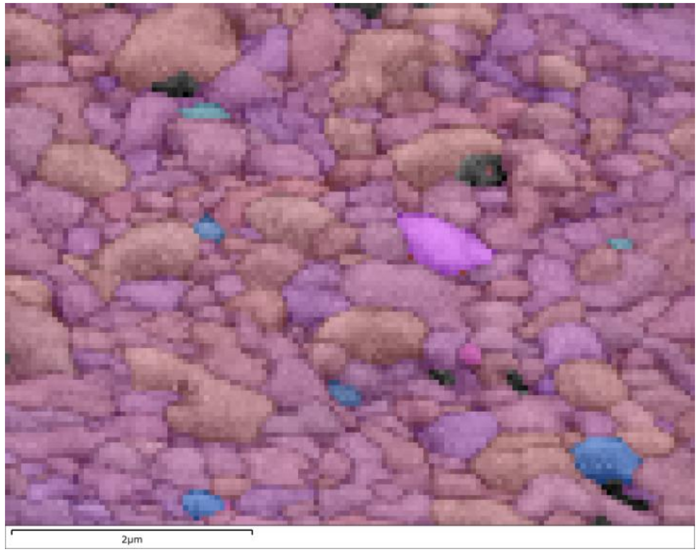
HIPIMS Multilayer Nb/NbN

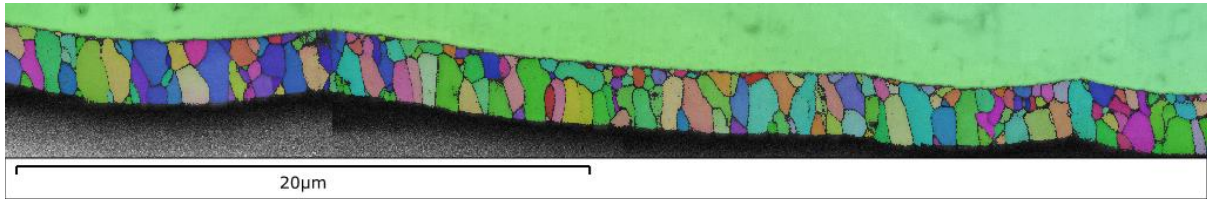


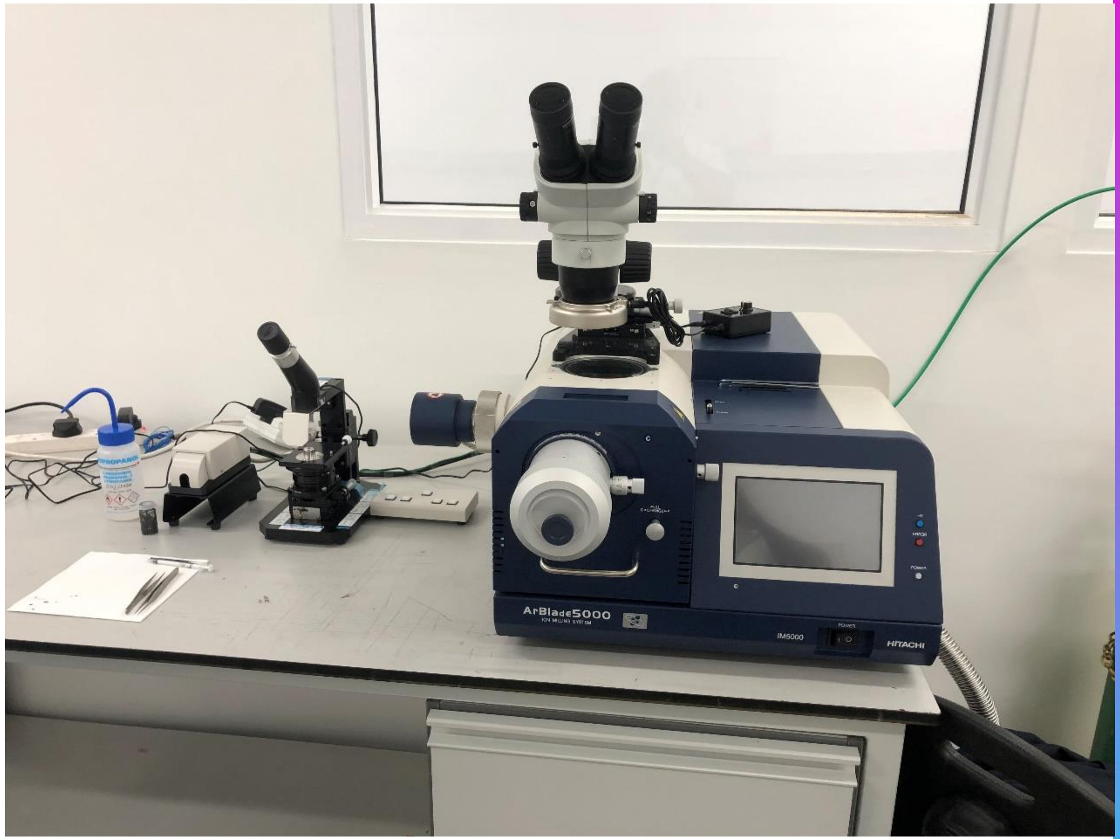
HIPIMS Nb

Ion Beam Miller for X-section SEM and EBSD





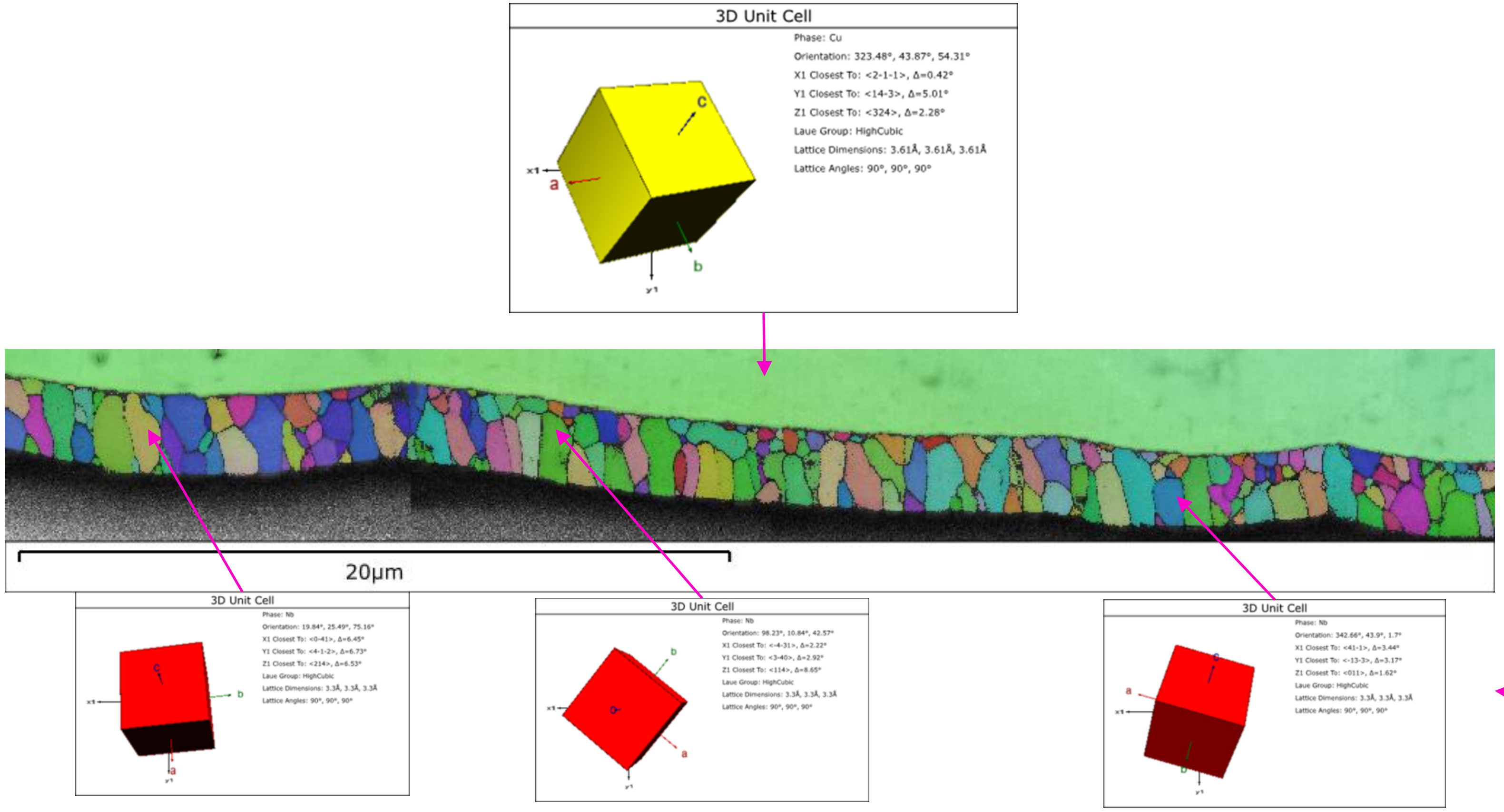


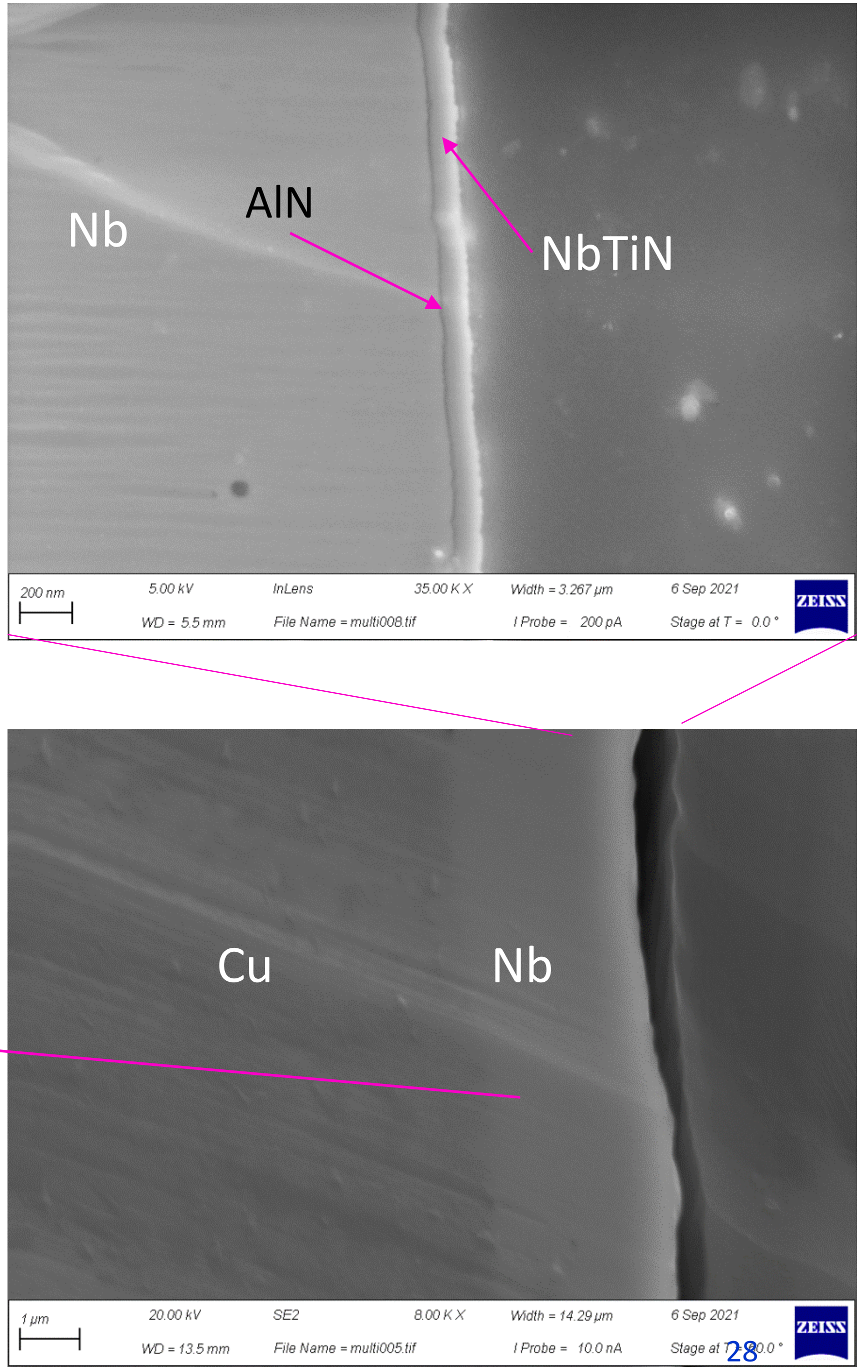


Nb/AlN/NbTiN where the copper has been annealed at 650 °C the top two layer is to thin to be resolved by EBSD.
EBSD is done at University of Liverpool

Examples: Multi-layers

Detailed EBSD of Nb section





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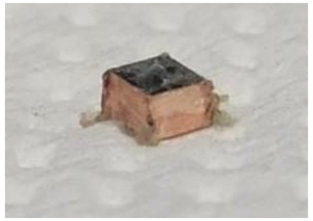
Superconducting Properties Evaluation at IEE

- DC magnetisation measurements using *Vibrating Sample Magnetometer*
- Small planar samples (~ 2 mm x 2 mm – *cutting*)

small sample

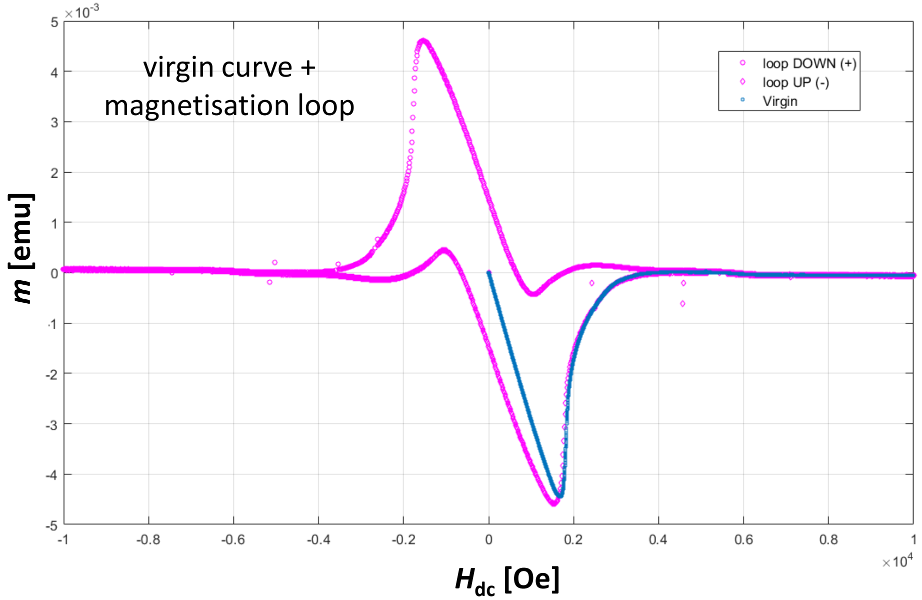


cutting phase (disk saw)

VSM sample holders

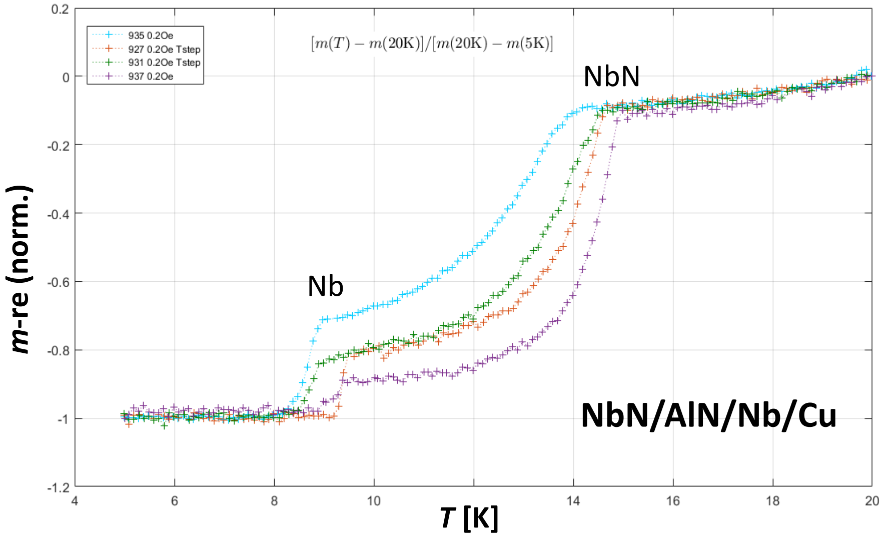
parallel *H*

perpendicular *H*

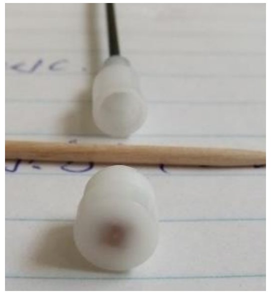


- *AC susceptibility* – temperature scans

Determining T_c of different films in Multilayer and SIS samples

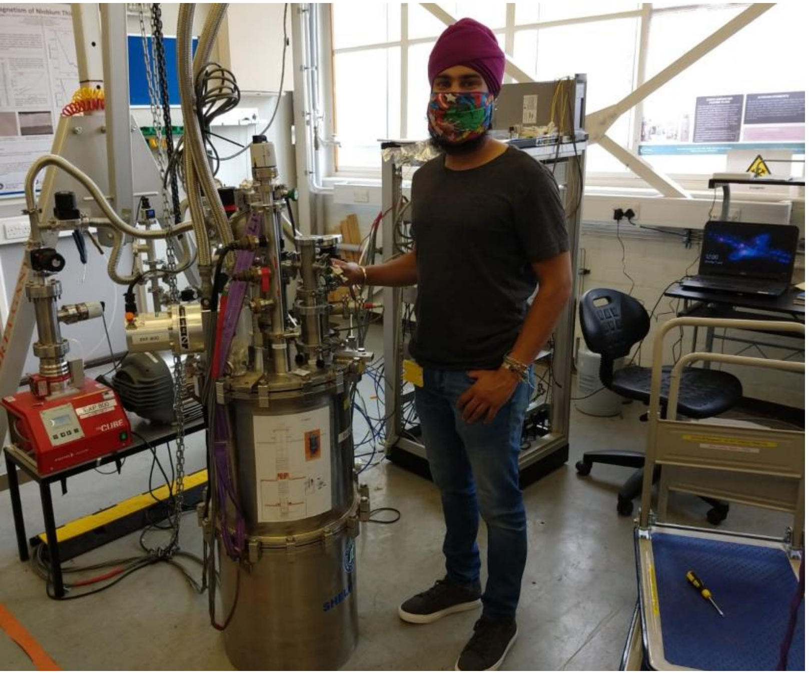


AC susc. sample holder

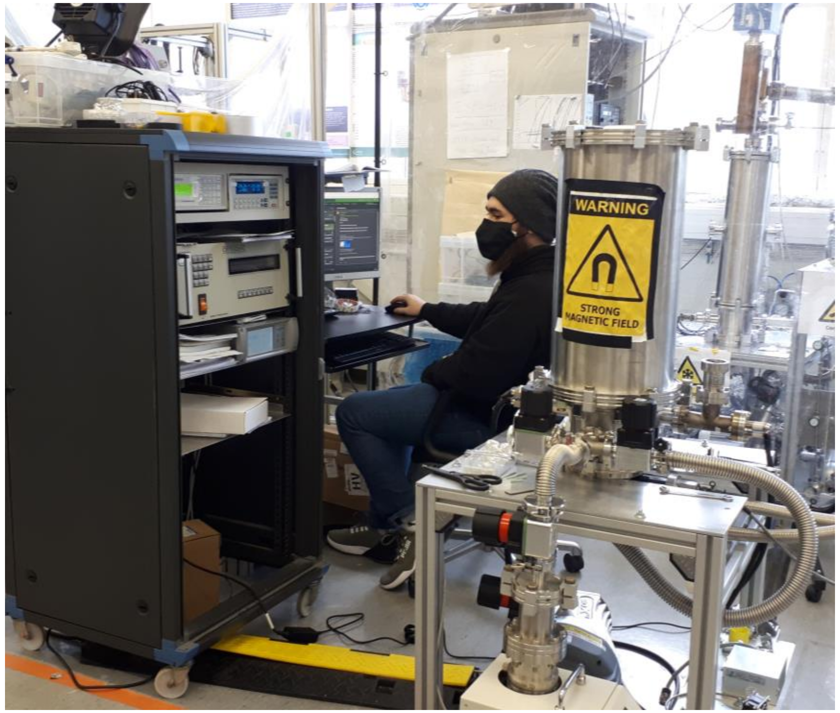


Courtesy of E. Seiler

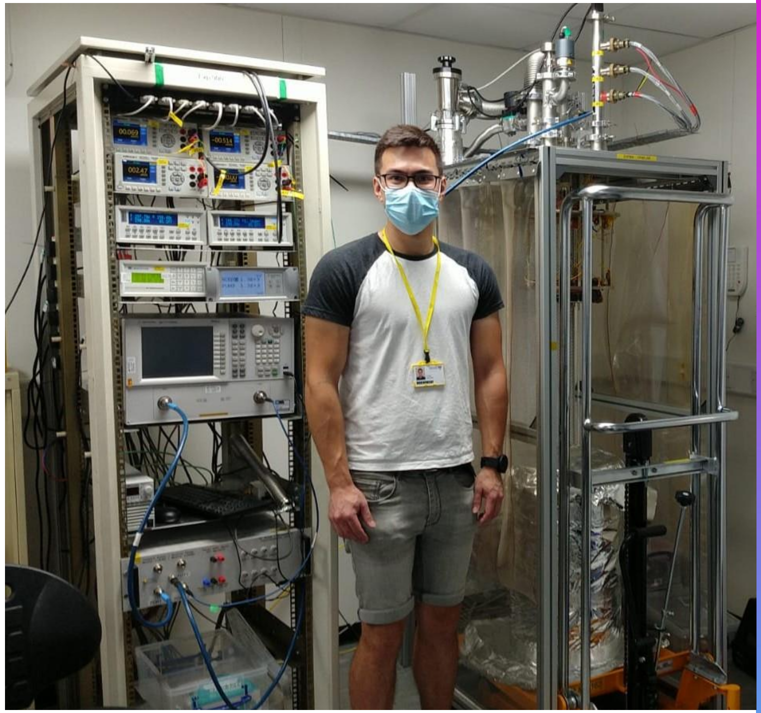
Superconducting Properties Evaluation at UKRI/STFC/CI



EXP800: RRR, Magnetic field penetration
+ 2 other experiments (in-He-gas)



EXP700: Magnetic field penetration facility (in-vacua)



EXP900: R_S measurements with 7.8 GHz cavity

Courtesy of T. Sian

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This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under GA No 101004730.

Task 9.4

Surface engineering by atomic layer deposition (ALD)

Task Leader: Thomas Proslier (CEA)

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ALD-deposited Multilayer to improve the superconducting performances of RF cavities

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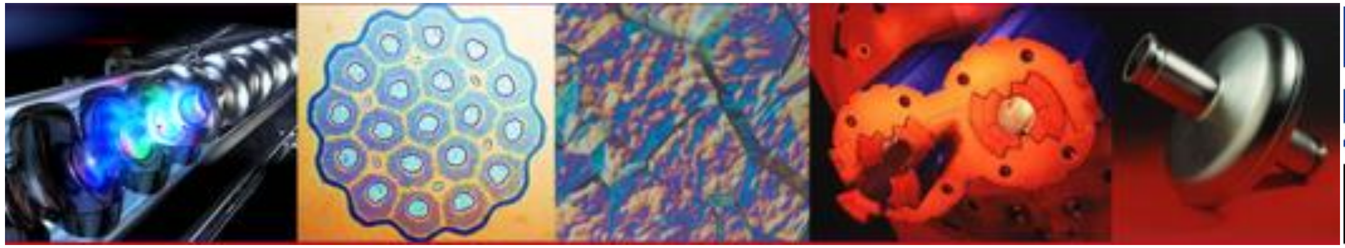
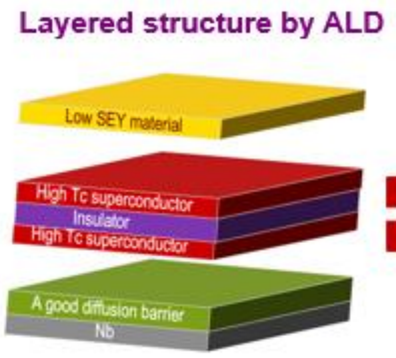
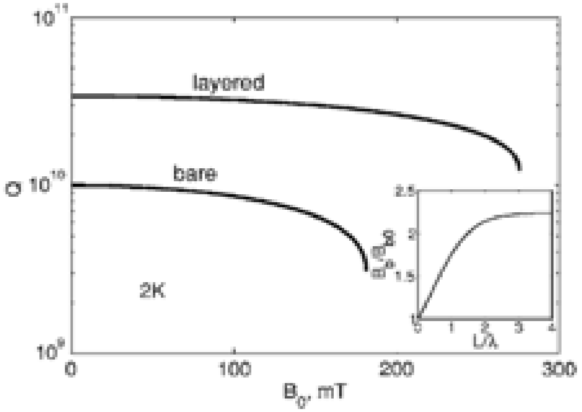
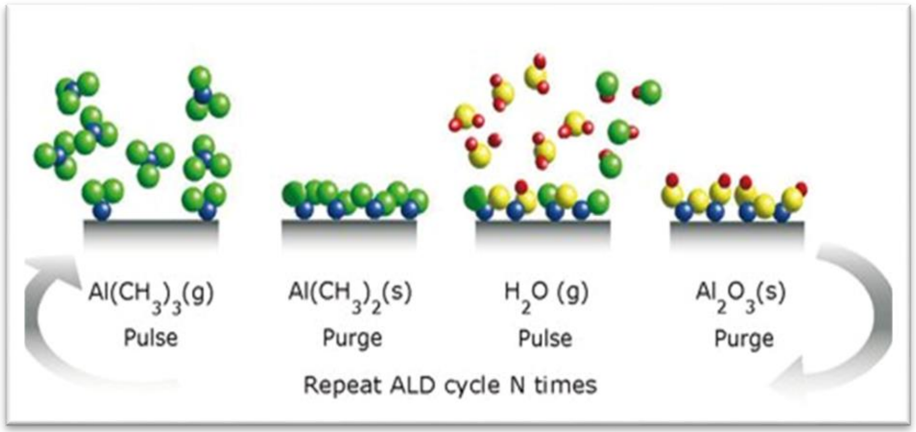






- An original approach proposed by A. Gurevich [1] to improve RF cavities through depositing a superconducting multilayer capable of screening efficiently the magnetic field.
- To optimize this concept, we need as well a thermally stable diffusion barrier as a base and a low SEY material on the outer layer in order to limit multipacting inside the cavities.

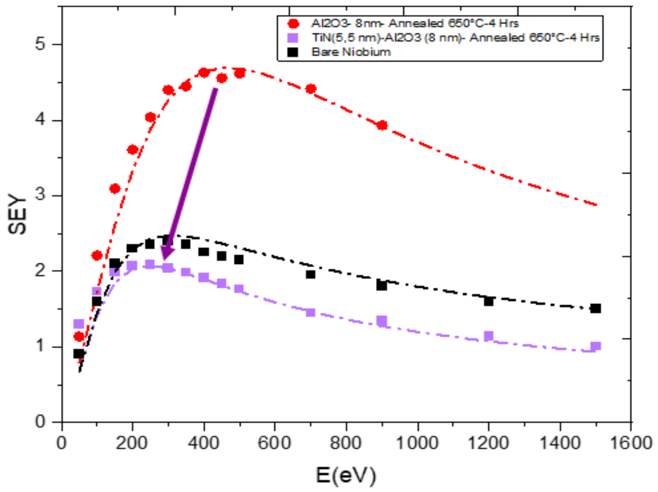


- The multilayer is a stack of nanometric films of high T_c superconductors and insulators.
- To synthesize this structure, we use atomic layer deposition ALD as a deposition technique as it well known to provide high quality Nano-films over large surfaces with complex shapes such as RF cavities.

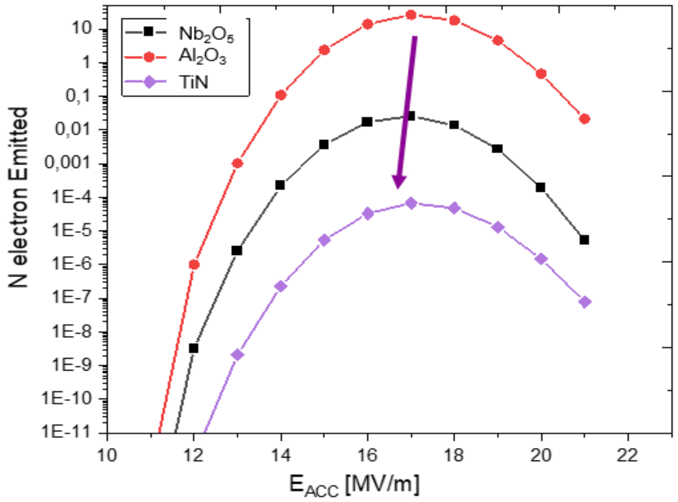
[1] A.Gurevich,Enhancement of RF breakdown field of SC by multilayer coating.Appl. Phys.Lett, 2006.

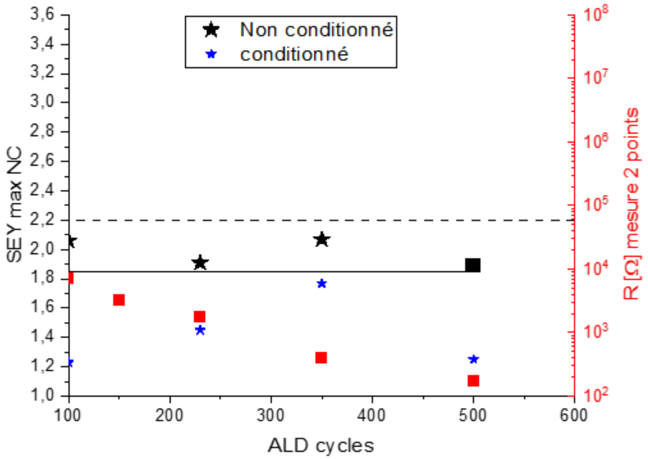
SEY ON ALD-DEPOSITED FILMS

SEY measurment @IJClab



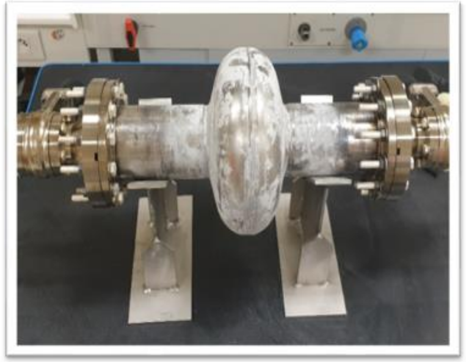
Fishpact simultation
Multipacting in 1,3 GHz cavity





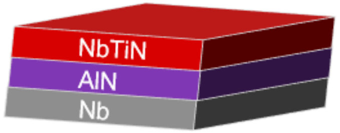
Picture of the inside of a Nb cavity coated with TiN-Al_2O_3

Picture of the outside of a Nb cavity coated with TiN-Al_2O_3



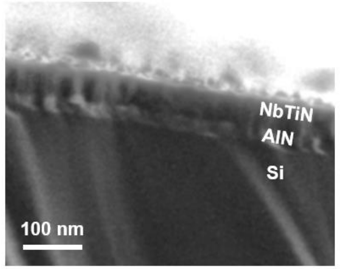
- Mitigation of SEY by TiN deposited by ALD down to very thin layers ~ 6 Å
- Applicable to coupleurs and cavities (outside deteriorated by air exposed high temperature)

BILAYER AlN-NbTiN

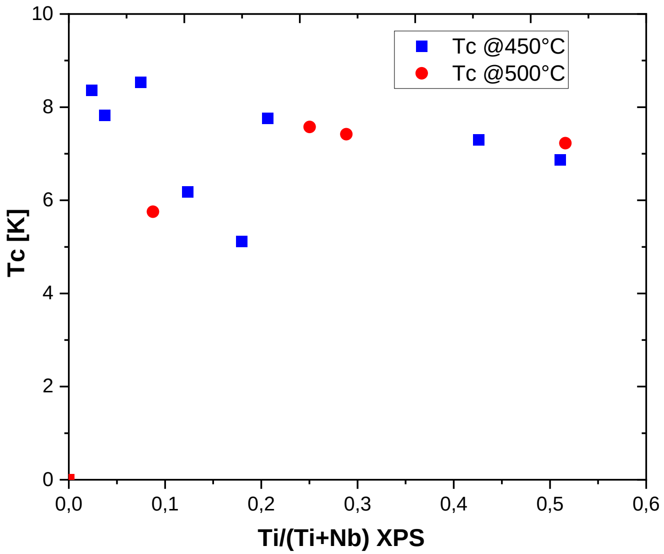


- Motivation: NbTiN has good superconducting performance (T_c = 17 K).
- Chemistry: Combination of $NbCl_5$/ NH_3 and $TiCl_4$/NH_3 cycles:

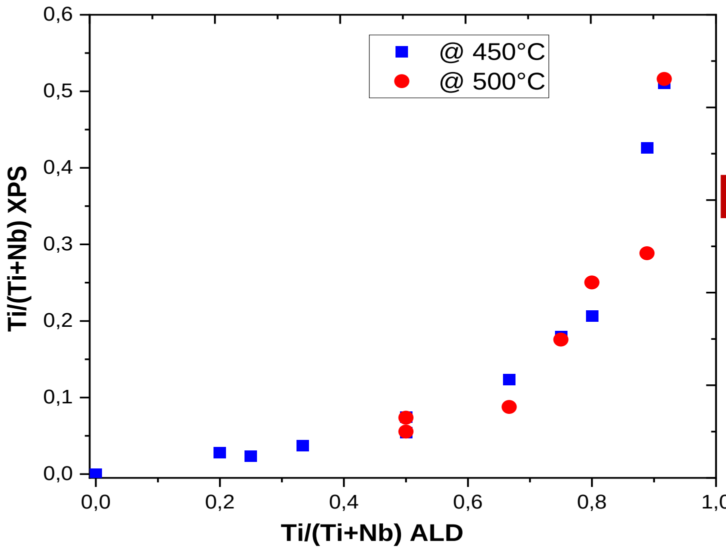
n ($TiCl_4$ + NH_3) + m ($NbCl_5$ +NH_3)



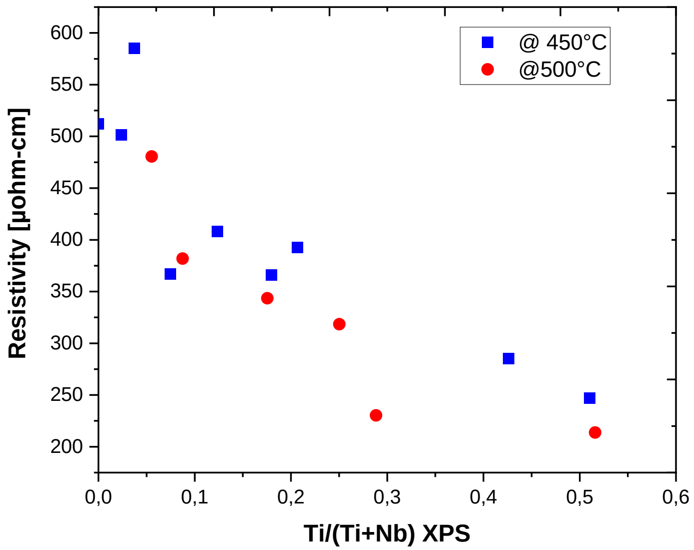
Critical temperature



NbTiN Chemical composition



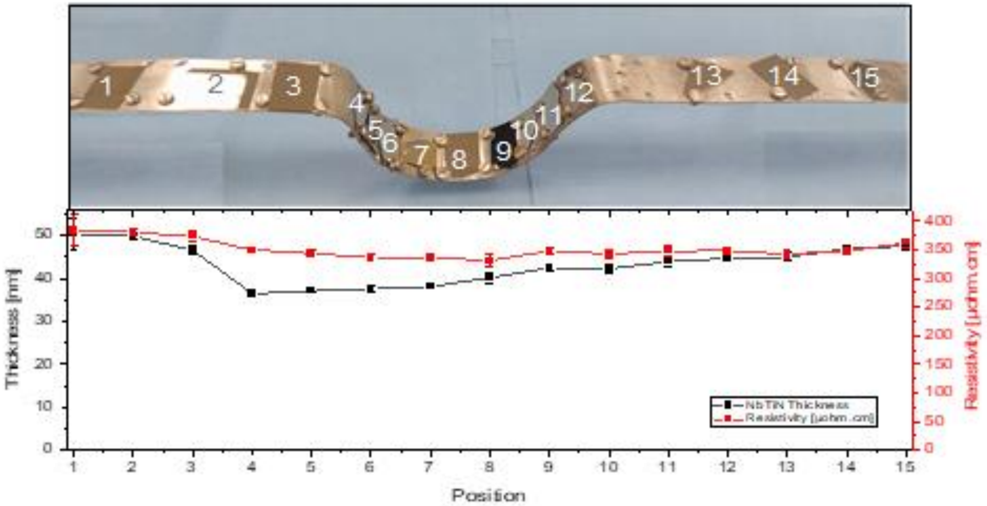
Resistivity



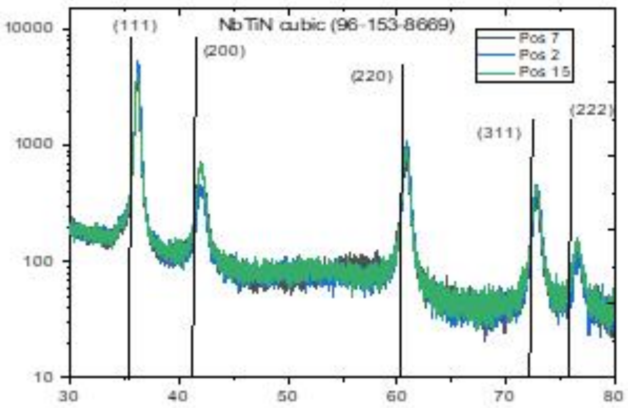
- Composition control $Nb_xTi_{1-x}N$ from x=0 to x=1 and reproducible at 450°C and 500°C
- 45 nm films T_c and resistivities have to be improved: new chemistry on new ALD system

AlN-NbTiN ON COPPER 1.3 GHz CAVITY

Test 2 : purge 20s



GIXRD diffraction patterns on different samples



- Optimized deposition parameters
- Homogeneous deposition (thickness, T_c and structure) over 1.3 GHz test cavity.

CAVITY ALD OVEN

- <u>Home made ALD system:</u>

9 precursor lines:

- 4 solids
- 2 liquids
- 2 gazes
- 1 High temperature
- Labview control program

- <u>Oven:</u>

- Retort: L =55 ; Ø = 50 cm
- 650°C (900°C + N_2)
- Vacuum: < 2.10^{-6} mbar (650°C)
- Handle 1,3 and 0,7 GHz cavities
- Some Mutlicellular possible

- Oven installed and tested + ALD system under commissioning.
- Cavity adaptors and custom growth chamber ordered.

CONCLUSION

- ✓ We manage to deposit uniformly a thin film of Alumina and reduce drastically niobium native oxides.
- ✓ RF test shows a slight improvement of the Q_0 under low and medium Fields.
- ✓ TiN film is promising to reduce multipacting inside RF cavities.
- ✓ Growth of superconducting NbTiN by ALD with homogeneous composition and thickness control over large surface areas.
- ✓ Oven received and installed. ALD system almost completed.

Future Goals will be:

- ❖ Optimization of NbTiN process to improve superconducting properties.
- ❖ Test the NbTiN-AlN structure on Niobium RF cavities in the ALD oven system.

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Task 9.5

Improvement of mechanical and superconducting properties of RF resonator by laser radiation

Task Leader: Arturs Medvids (RTU)

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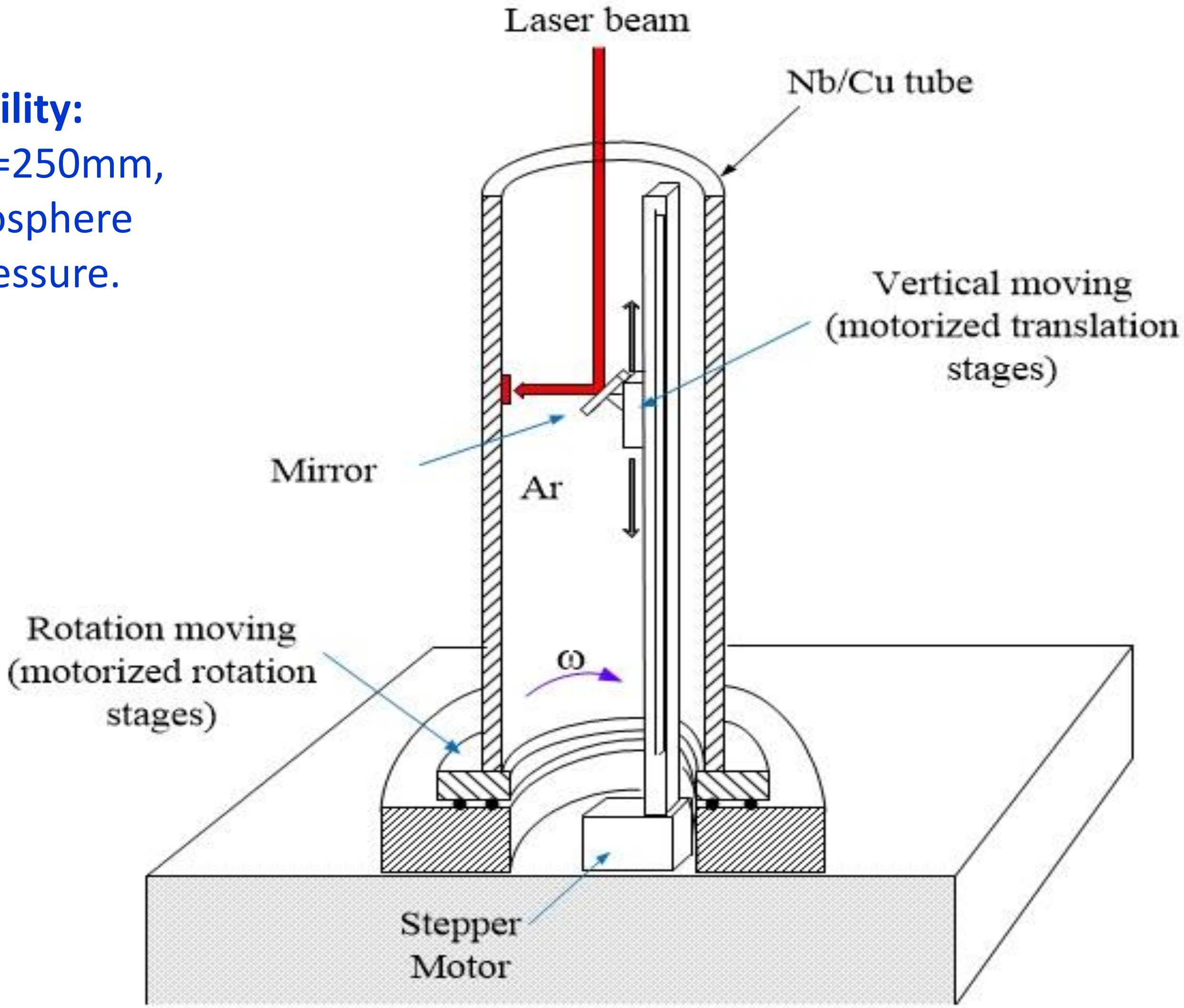
Cross-section of the laser facility for irradiation inner surface of RF cavity

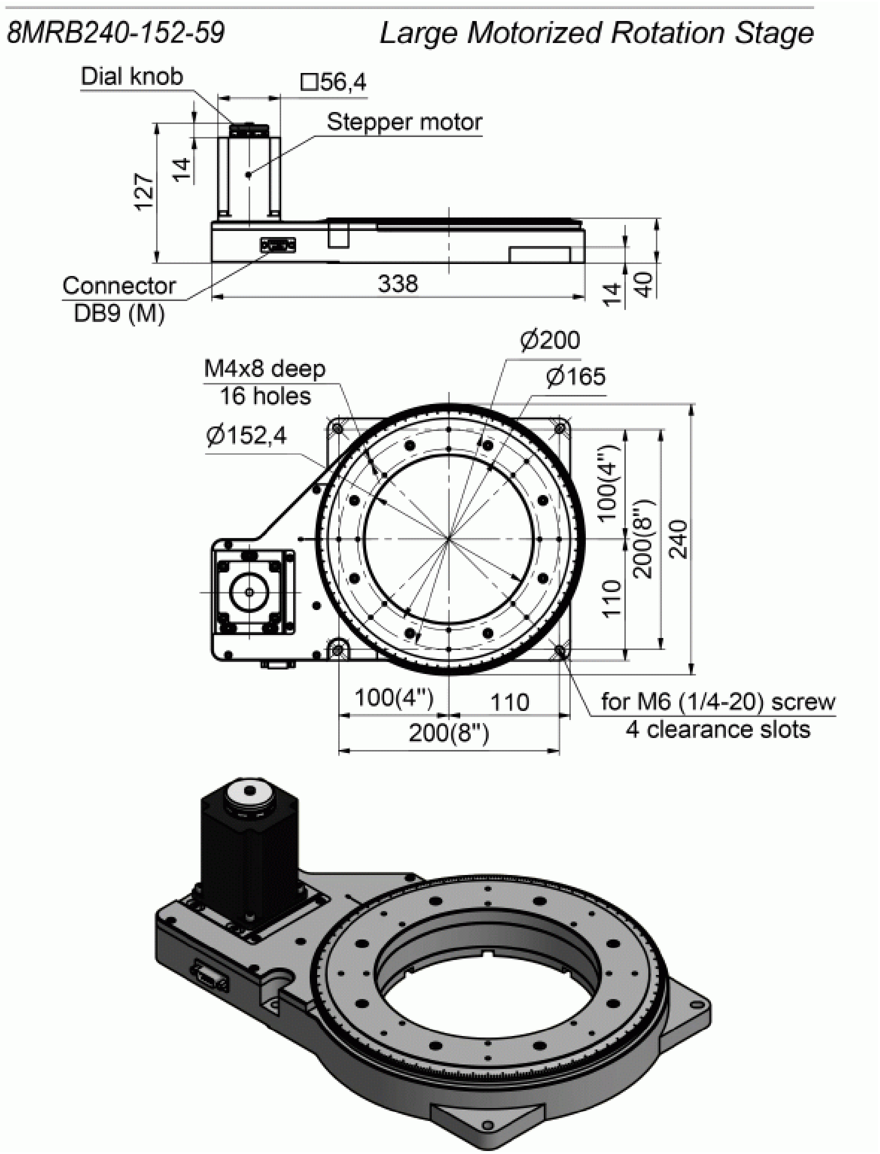
Laser facility:
L=450mm, D=250mm,
Ar gas atmosphere
1.5 atm pressure.



Now we are making a vacuum chamber with an optical window.
And we wait for the results of the purchase competition of two motors for the chamber: stepper motor and motorized rotation stage.

8MRB240-152-59 - Large Motorized Rotation Stage

This rotator is a perfect example of our personal approach with clients and custom design flexibility. The device was designed in cooperation with microchip manufacturing company for operations with silicon wafers.



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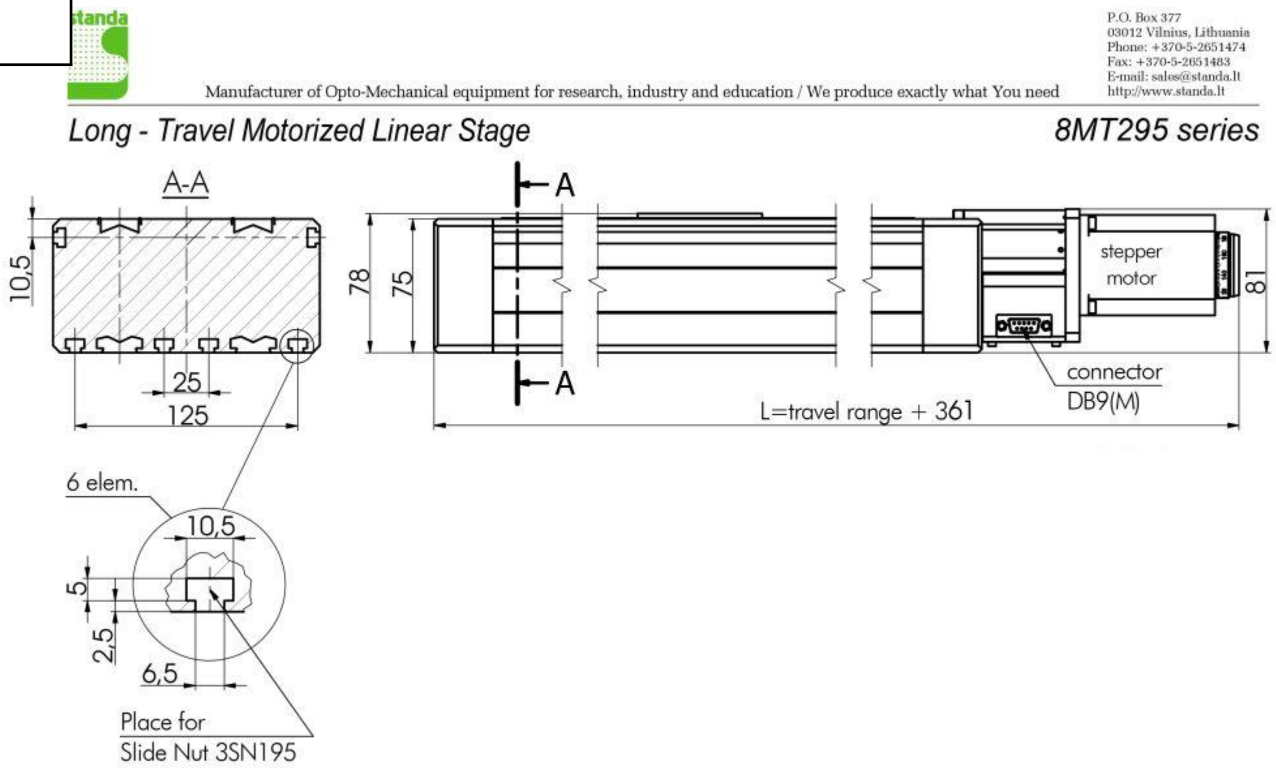
8MT295 - Long-Travel Motorized Linear Stages



Long Travel Motorized Linear Stage 8MT295 series stages are designed to provide high-speed movement. Standard motors allow moving loads up to 60 kg. Load capacity can be increased using more powerful motors. This stage provides moderate resolution and accuracy. 8MT295 series stages are supplied equipped with **3P295** platform, **3BP295** bases plates (2 pc) and appropriate amount of **3SN195** inserts. Resolution and speed of 8MT295 series stages can be varied choosing appropriate ball screw pitch. Several standard options are available and should be specified upon ordering.

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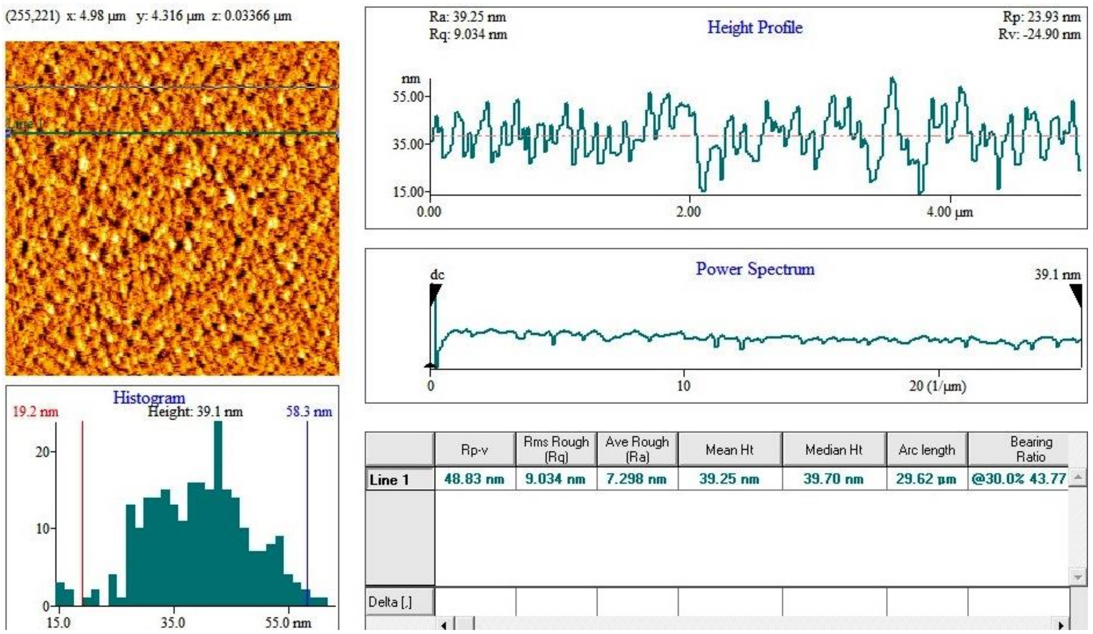
Laser parameter optimization on Nb coated copper samples

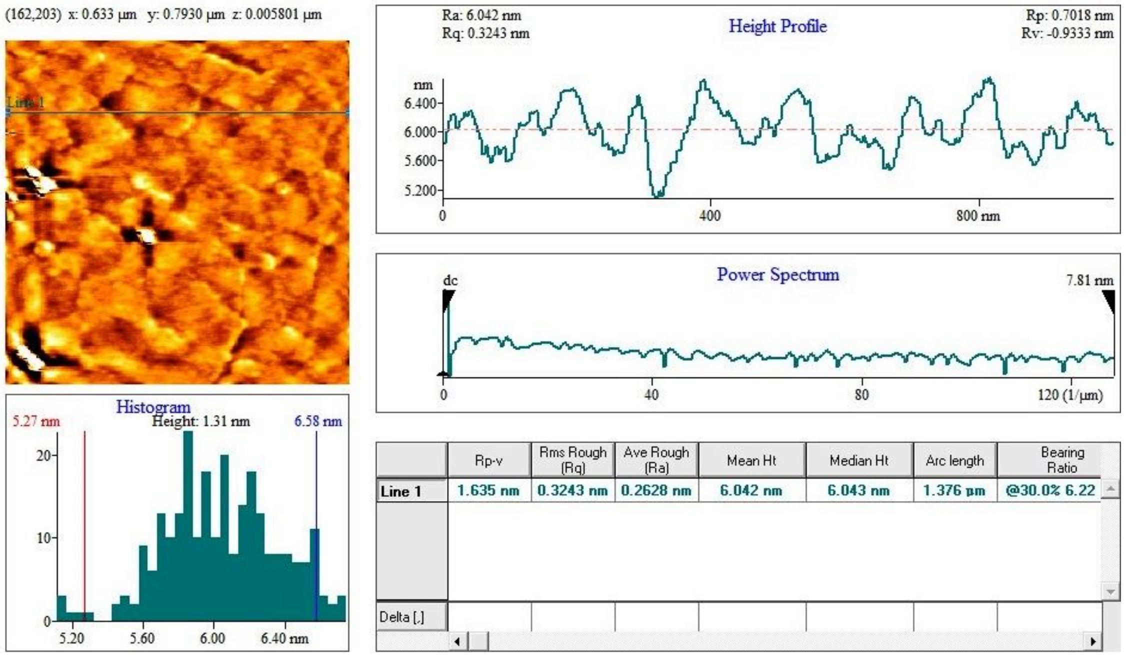
Six Nb coated samples 35 mm x 53 mm has been laser treated to smooth the Nb surface

Example:

before laser treatment



after laser treatment



- Reduction in R_a by a factor 1.5 to **5**
- Next steps:
  - Magnetic field penetration measurement to see how it has affected superconducting properties
  - Laser treatment of a sample for RF test at 7.8 GHz

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Task 9.6

Optimization of flat SRF thin films production procedure

Task Leader: Oliver Kugeler (HZB)

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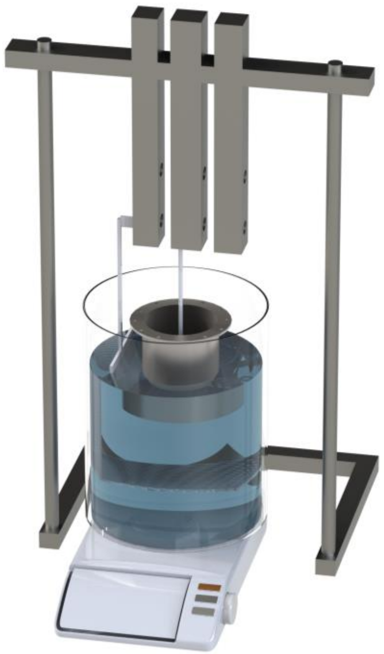
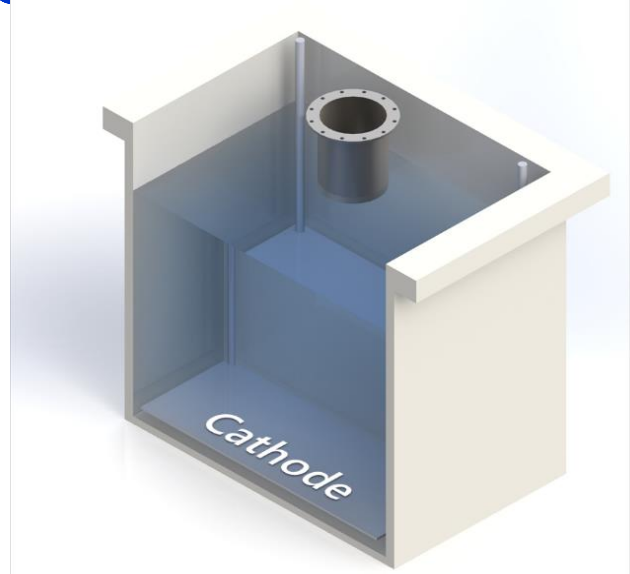
Status WP9 Task 9.6

- MS42 achieved and report submitted
  “ARIES samples prepared for renewed SC film deposition”
- Two QPR Nb bulk samples prepared at INFN Legnaro and CEA Paris for subsequent film deposition
- Baseline measurements performed with QPR at HZB
- Breakthrough side effect of QPR activities within I.FAST:
  - ✓ Significant improvement of measurement accuracy (< 4 nW)
  - ✓ Publication submitted to Review of Scientific Instruments (preview at http://arxiv.org/abs/2110.07236)

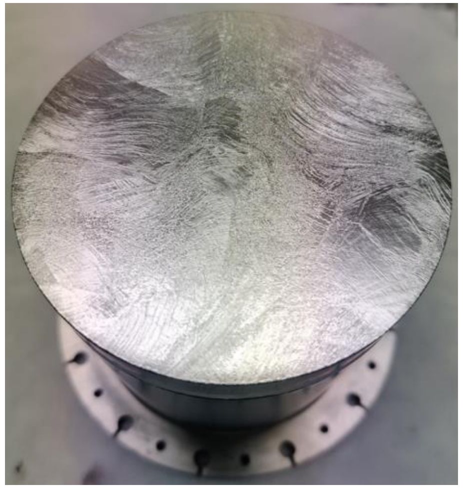
Plasma electropolishing on QPR samples at INFN



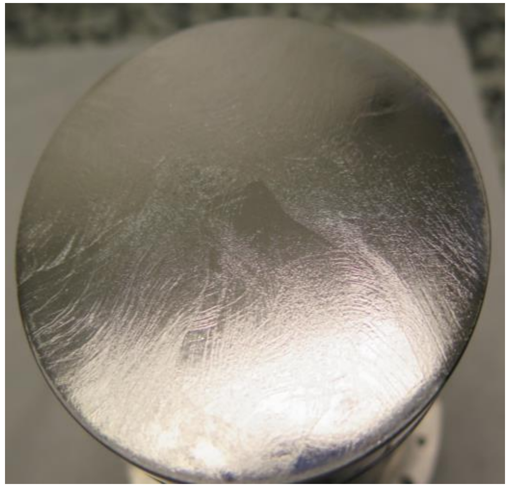
Old system



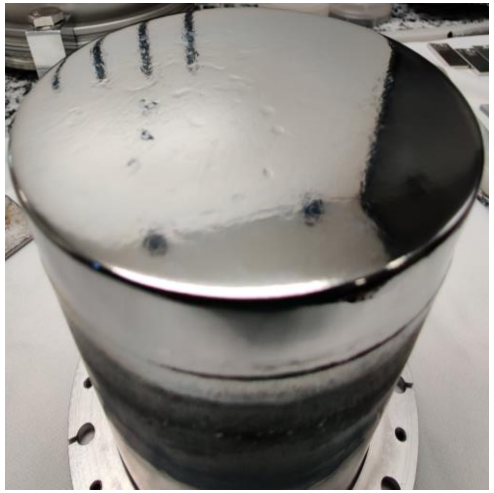
Upgraded system
- Larger volume
- More stable temperature
- More stable current



Initial Nb QPR sample



Surface improvement after 10 min



100-µm removal in 60 min.
Mirror finish

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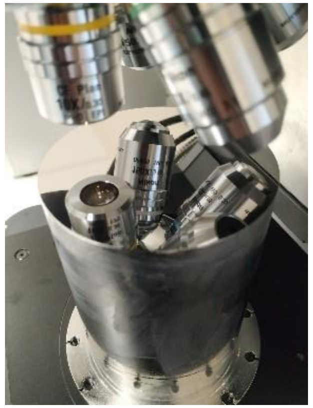
Metallographic polishing at CEA/IJCLab

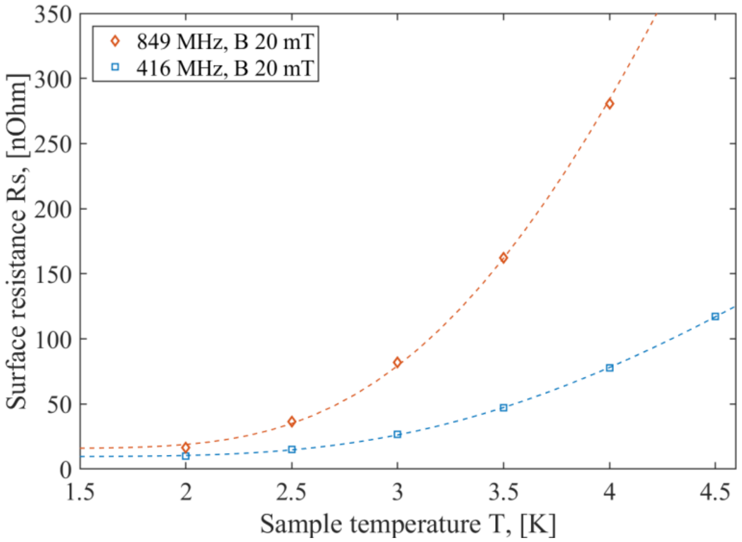
Investigating metallographic polishing in collaboration with CEA to evaluate possibility of omitting electrochemistry step in the cavity surface preparation recipe

QPR sample milled from ASTM0 large grain material
S_a = 7 µm
Poor RF performance, In contaminations



Mirror polishing at IJCLab
S_a = 0.5 µm

After EP 150 µm + annealed at 900°C under vacuum + EP 20 µm at CEA
S_a = 1 µm



Great RF performance of baseline sample (9 Ohm residual resistance at 416 MHz)

Pictures courtesy Oleksandr Hryhorenko (CEA IJCLab)

Omit this step and re-evaluate performance

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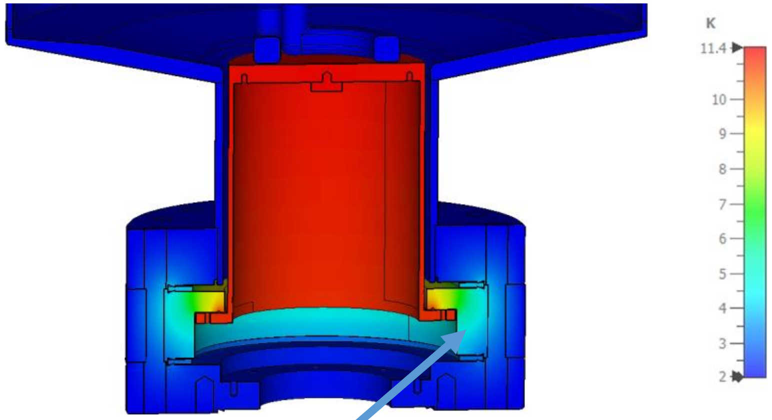
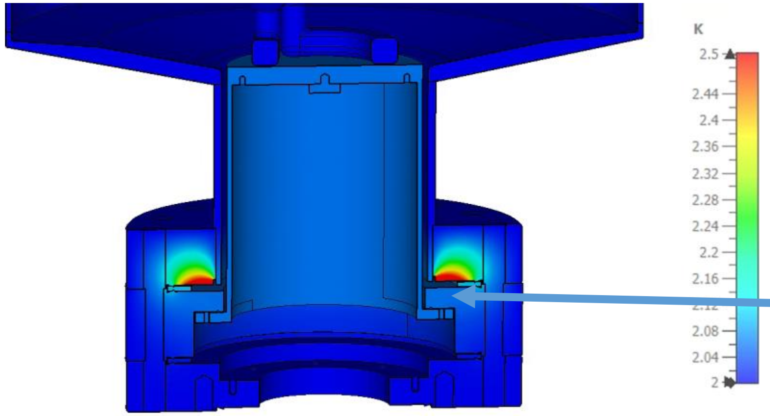
QPR performance improvement

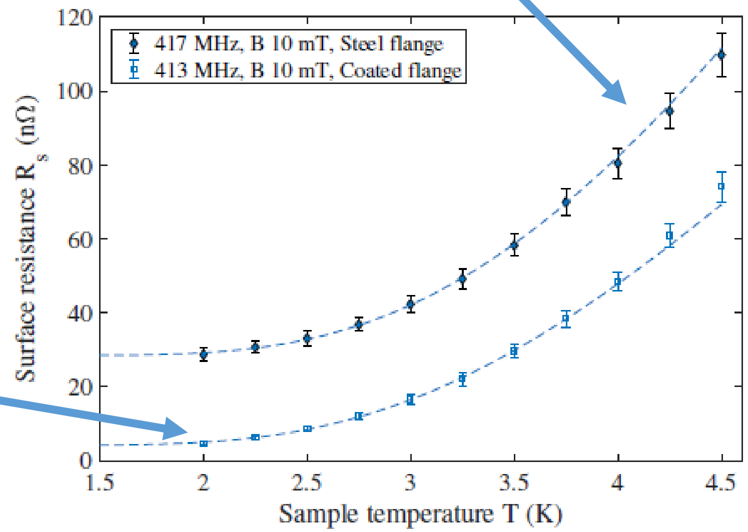
- Forensic analysis of coaxial gap of QPR
- Dipole mode propagates to adapter flange causing RF heating in sample holder
- Result: Systematic error
- Solution: Coating adapter flange with Nb layer at Uni Siegen
- Accuracy significantly improved



Steel adapter flange



Nb coated adapter flange



Summary

- WP9 team is working in a full power
- Significant progress demonstrated with each Task

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Thanks for your attention!

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