

### 2<sup>nd</sup> I.FAST Annual Meeting 18-21 April 2023

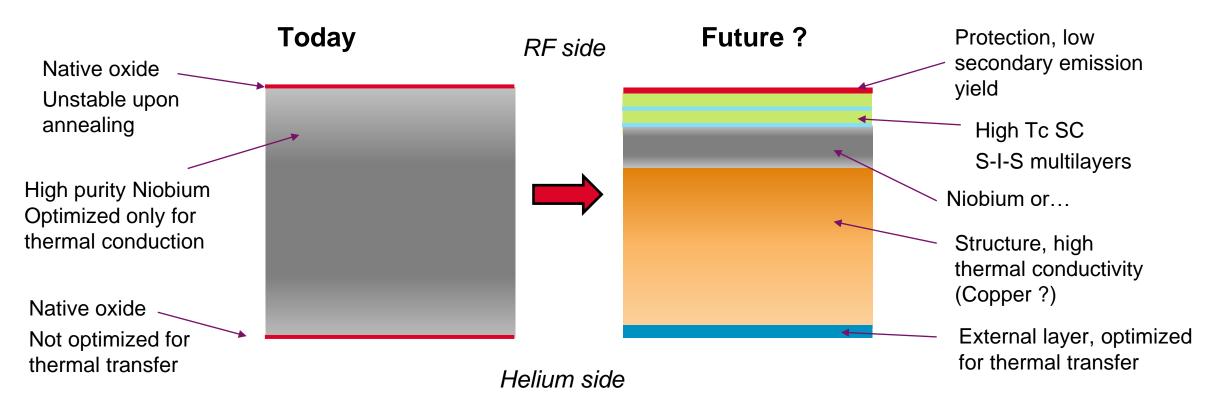
Oleg B. Malyshev (UKRI) / Claire Antoine (CEA) WP9 coordinators





Science and Technology Facilities Council

# **Desired: Tailored material for RF cavities**



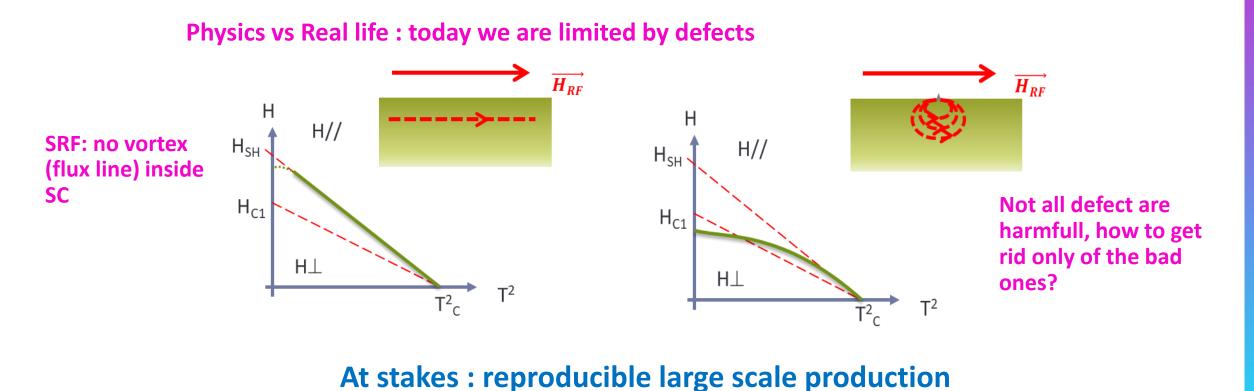
#### At stakes : COST REDUCTION !!!

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- Cooling power (any application ); can we go to cryocooling ?
- High accelerating fields; shorter machines ?

### **Today's technological limitation**

- Lower dissipation (high Q<sub>0</sub>) => goes w. higher T<sub>c</sub>
- Shorter machine (high E<sub>acc</sub>=> goes with higher transition field (H<sub>SH</sub> or H<sub>C1</sub>?)

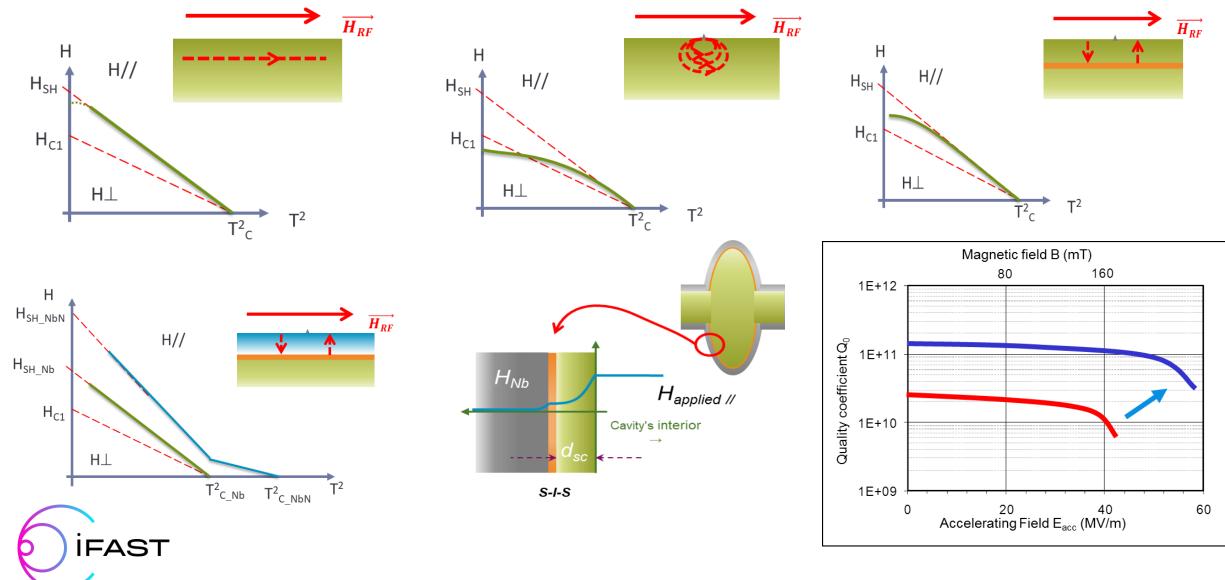


(Accelerators are big machines!)

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# Multilayers (SIS) concept, or how to make theory face reality



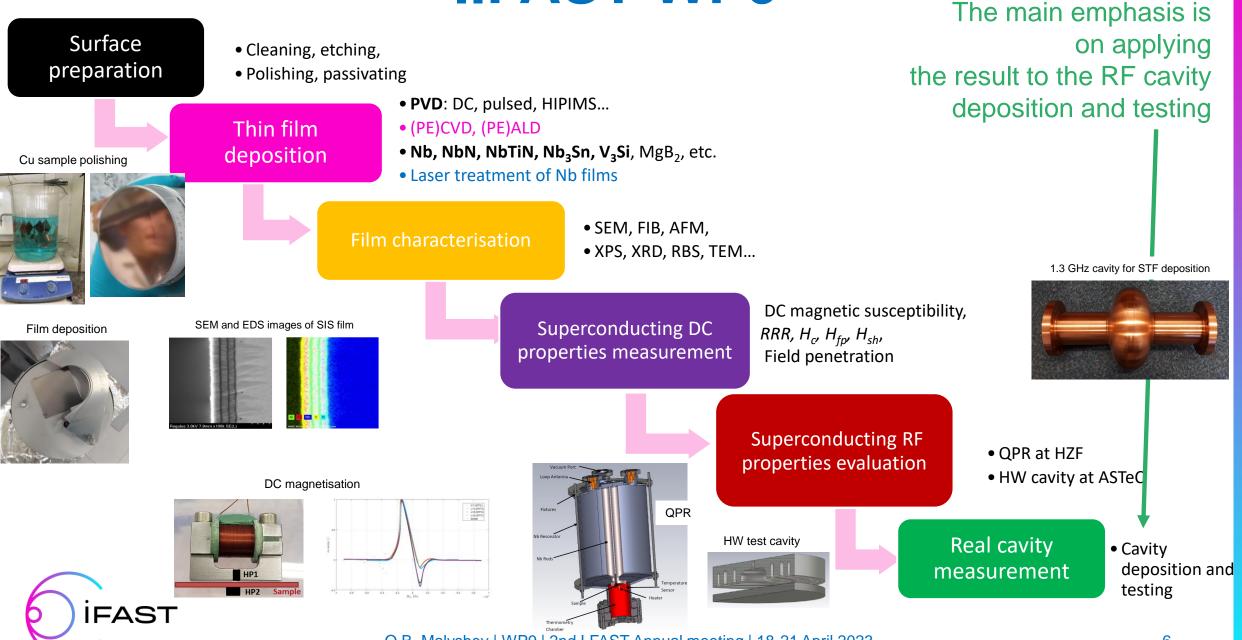
### **Objectives for WP9 Innovative superconducting cavities**

To improve performance and reduce cost of SRF acceleration systems Small community

- We built together a global strategy to be able to produce Superconducting RF (SRF) cavities coated with a superconducting film. Not only IFAST, (informal) WW collaboration
- It includes pursuing the optimisation and the industrialisation:
  - Substrates preparation (Nb, Cu), e.g. PEP, metallographic polishing
    - Pre-and post treatment (laser)
    - The production of seamless copper cavities
  - The optimization deposition techniques: MS, PVD, ALD... to get Nb, NbN, Nb<sub>3</sub>Sn, V<sub>3</sub>Si... thick films (µm) and/or SIS Multilayers (nm)
- Produce and RF test prototypes of SRF cavities at 6 & 1.3 GHz Easier to handle, fabricate, dissect to provide fast feedback
- Produce accelerator type 1.3 GHz cavities (feasibility assessment).



## **I.FAST WP9**



### **WP9 tasks**

Task 9.1:

Coordination and strategy

for innovative superconducting accelerating cavities

Task 9.2:Innovative SC accelerating cavity prototype.1.3 GHz cavity coating and testing



Task 9.3 :

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

6 GHz cavities

#### Task 9.4:

Surface engineering by atomic layer deposition (*ALD*)

#### Task 9.5:

Improvement of mechanical and superconducting properties of RF resonator by *laser radiation*  Task 9.6:

Optimization of flat SRF thin films production procedure **QPR and HWR** 



### **Task 9.1:**

#### Coordination and strategy for innovative SC accelerating cavities WP9 Meetings every 3 months

#### **On scopes:**

done
ongoing
ongoing
ongoing
ongoing
done
ongoing

#### Future:

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- Organising the 2024 Thin Film SRF workshop (scientific committee + local organisation)
  - Will be held @ Paris-Saclay
  - Officially sponsored by IFAST



# Task 9.2:Seamless elliptical copper cavities

#### **GOALS:**

#### Task Leader: Cristian Pira (INFN)





- Move cavity forming process from semiautomatic to fully automatic using CNC machine
- Study annealing temperature effect on formability
- Test reproducibility
- 6 GHz for task 9.3 and 1.3 GHz cavities for prototype

#### Why?

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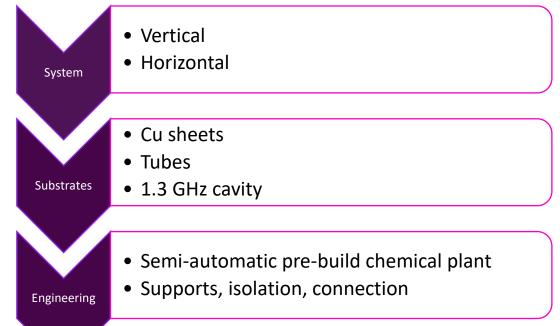
- Need a lot of cavities for destructible tests
- Welding on copper definitively bad for films
- 18 cavities realized 6 GHz
- No intermediate annealing necessary





# Plasma electropolishing (PEP) of 1.3 GHz copper cavity @INFN

Workflow in the framework of a master degree student Roberta Caforio.











# **1.3 GHz cavities coating facility at INFN**

Main chamber parts produced and ready to be assembled





• Features:

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- 'Hybrid' coating system
- Rectangular magnetron & rotating cavity
- Post magnetron configuration with Nb<sub>3</sub>Sn cylindircal target produced via dipping

Courtesy by Alessandro Salmaso, INFN

# **1.3 GHz cavities coating facility at INFN**



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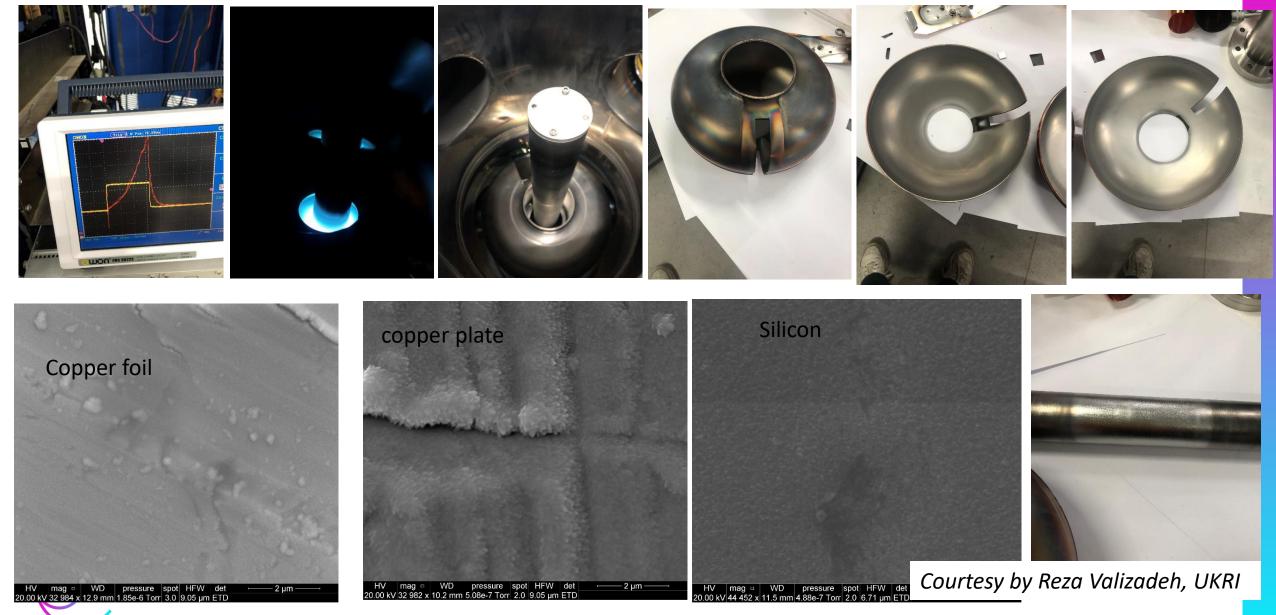
Heating system:

- IR lamps
  - Uniform heat
  - Fragile
  - >800 °C
- Resistive
  - Non-uniform heat
  - Rigit
  - <600 °C

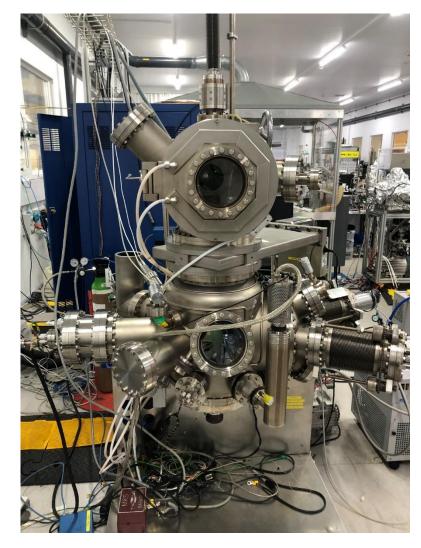
- Rectangular magnetron:
  - Design still ongoing, based on existing project
  - Limiting factor: cut-off diameter (78 mm)
  - Goal for end of 2023: first runs on samples with a mock-up cavity

Courtesy by Alessandro Salmaso, INFN

### HIPIMS dep of Nb on half cell 1.3 GHz cavity RT at UKRI



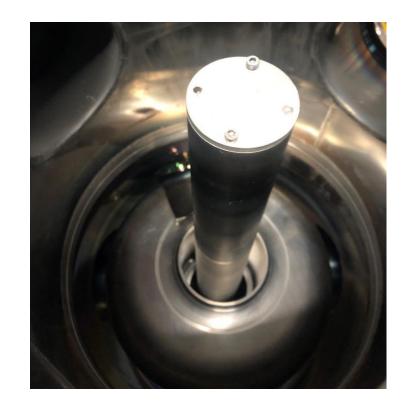
### 1.3 GHz Cavity deposition system at UKRI

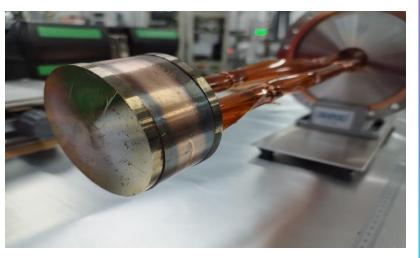


FAST

The system is equipped with load lock chamber, rotating arm that can turn and move up and down, the chamber wall is water cooled, fixed magnetron in the centre.

It will be positioned in an ISO 6 clean room with ISO 4 cabinet for final cavity preparation.

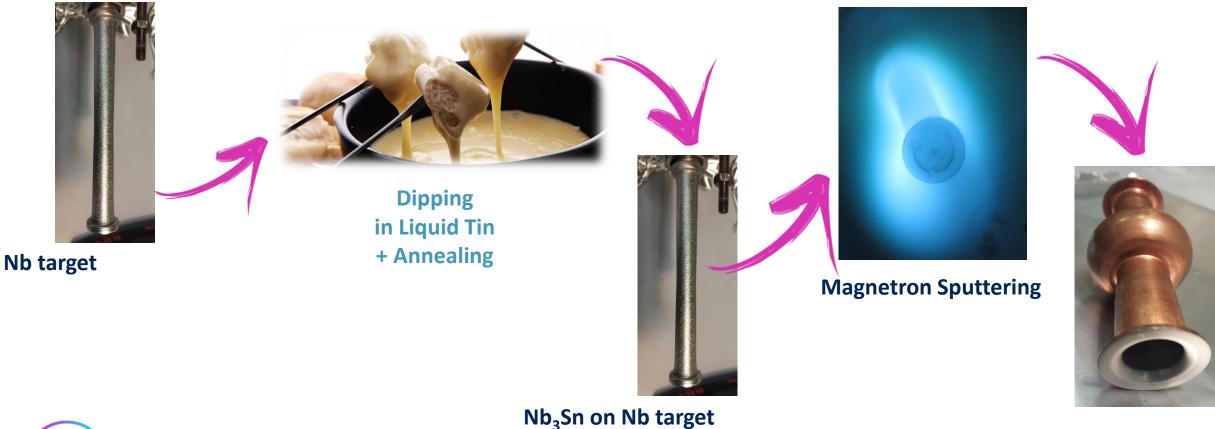




Courtesy by Reza Valizadeh, UKRI

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### Nb<sub>3</sub>Sn Cylindrical target production by tin liquid diffusion (dipping)





# **New System** for Nb<sub>3</sub>Sn Cylindrical target production by tin liquid diffusion (dipping)

New custom vacuum chamber system that contains the Nb chamber and new inductive heating system

- Integral chamber cooling;
- 2 viewports for monitoring;
- Single vacuum pump solution for the entire system
- 3 kW total power

FAST

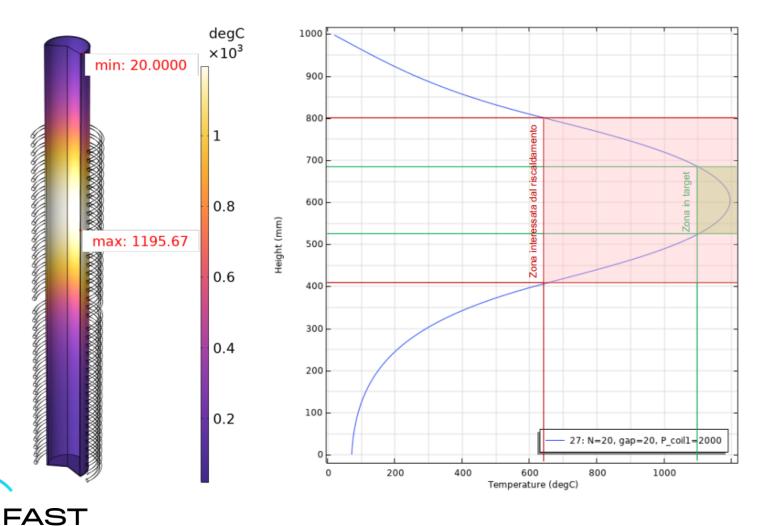
- Process entirely automated and remotely controlled
- More reliable system and more accurate temperature control

System already commissioned and will be delivered by the end of May



### Inductive heating system simulations

#### Single inductor maximum temperature test



Power on Nb	1463 W
Losses	564 W
Yeld	73 %
12 (RMS)	167 A

Single inductor heating meets target temperature of >1000°C

In coparison with the old resistive heating, target temperatures are easily reached even with single inductor heating

Courtesy by Davide Ford, INFN

### Task 9.3 Part 1: Cavity Coating and Evaluation

Task Leader: Reza Valizadeh (UKRI)

Aim:

- Quick deposition, quick testing, low cost (6 GHz)
- Optimization of process parameters with A15 material
- Evaluation of SRF performance by deposition of high T<sub>c</sub> superconductor inside a 6-GHz copper cavity.

### Nb<sub>3</sub>Sn, V<sub>3</sub>Si, NbTiN, NbN, MgB<sub>2</sub>

#### Why?

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- Higher T<sub>c</sub> SC are complex (compound) materials
- Composition needs to be adjusted to get best SRF performance
- Optimised recipes need then to be adapted for complex geometries

### 6 GHz copper cavity at UKRI

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



INFN seamless standard elliptical copper cavity

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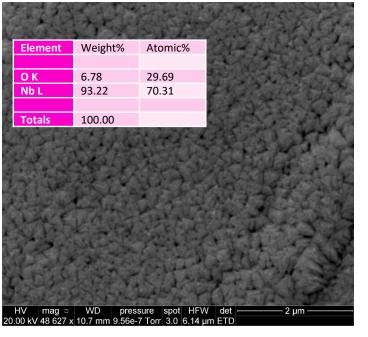


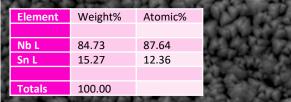
Lancaster University / STFC spilt cavity design

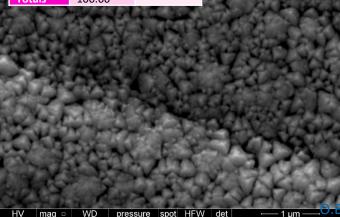
Courtesy by Reza Valizadeh, UKRI



### Nb<sub>3</sub>Sn deposition with Nb tube and Sn wire inside



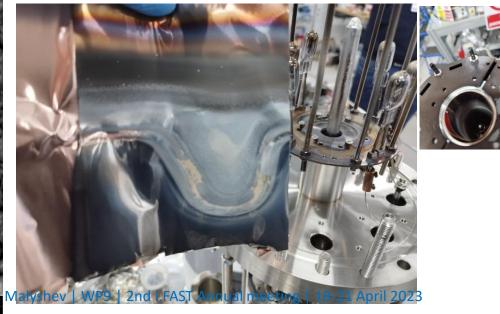


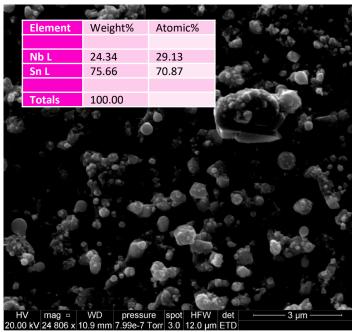


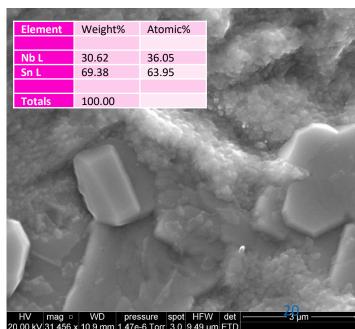
HV mag □ WD pressure spot HFW det -20.00 kV 49 724 x 10.7 mm 9.66e-7 Torr 3.0 6.00 µm ETD



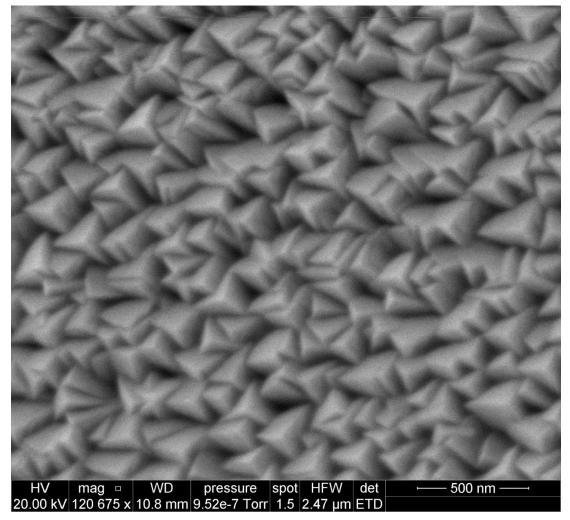
HV mag □ WD pressure spot HFW det \_\_\_\_\_ 20.00 kV 76 145 x 10.9 mm 1.28e-6 Torr 3.0 3.92 μm ETD





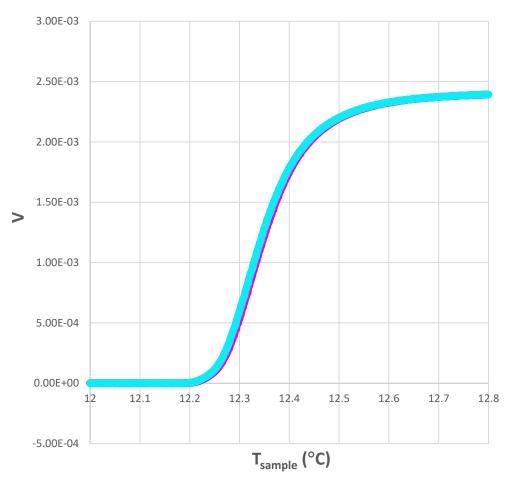


### NbTiN deposition using Nb<sub>37</sub>Ti<sub>63</sub> rod



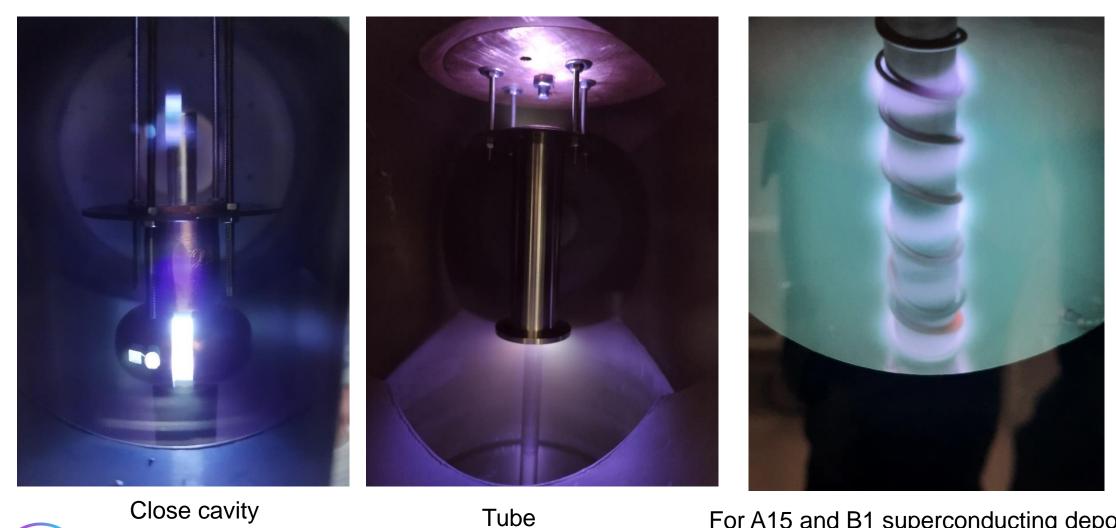
FAST

NbTiN 030423



Courtesy by Reza Valizadeh and Liam Smith, UKRI

### **Nb/NbTiN using permanent magnet magnetron**

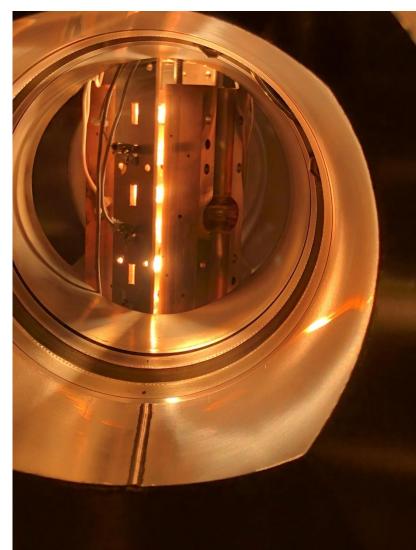


For A15 and B1 superconducting deposition

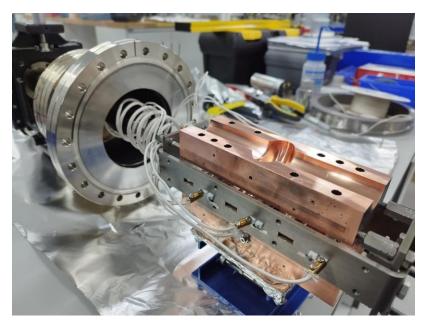
Courtesy by Reza Valizadeh, UKRI

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### **6 GHz split cavity heating stage**



**FAST** 



Without any heat shield the cavity temperature reaches to 600  $^\circ\mathrm{C}$ 





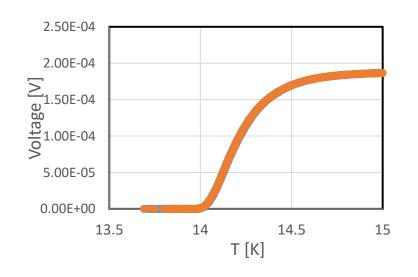
Courtesy by Reza Valizadeh, UKRI

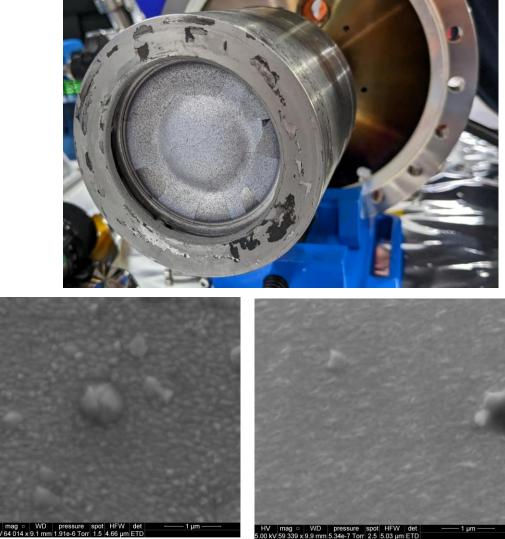
# V<sub>3</sub>Si deposition at UKRI

### Deposition system

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- System is equipped with a single planar magnetron source (V<sub>3</sub>Si alloy target).
- Sample holder capable of heating to 800 °C.
- Kr as the process gas.
- Base pressure 5×10<sup>-9</sup> mbar





#### V<sub>3</sub>Si on Cu morphology

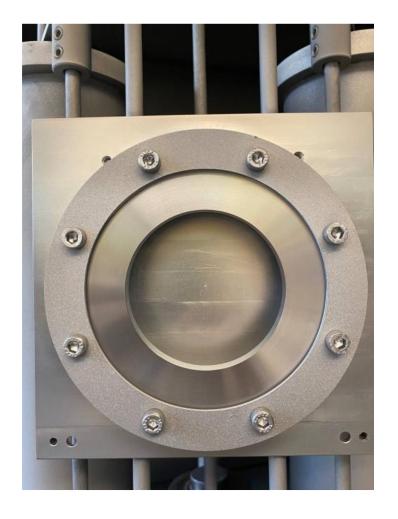
Courtesy by Chris Benjamin and Liam Smith, UKRI

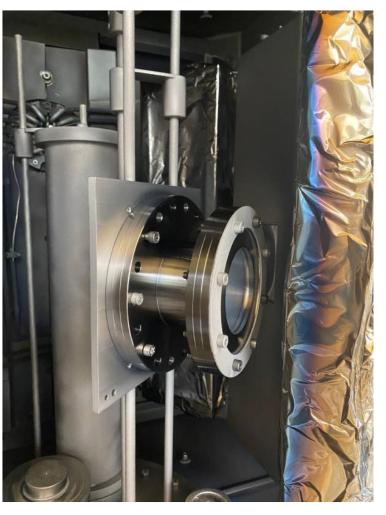
### **Deposition facility at USI**

Various adapters designed and produced

- QPR sample adapter
- Adapter CF125 to CF100

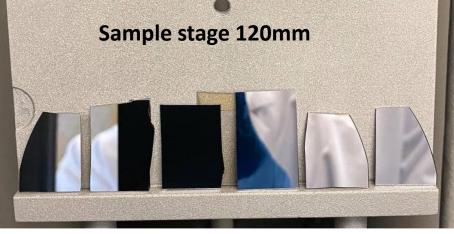
FAST





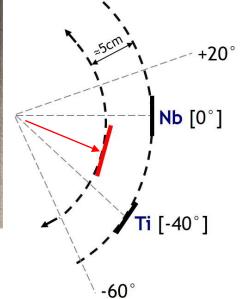
Courtesy by Alexander Zubtsovskii, USI

### **Co-sputtering of NbTiN: rocking angle at USI**



Rocking	angle
-40	0
-45	+5
-50	+10
-55	+15
-60	+20
-65	+25

FAST

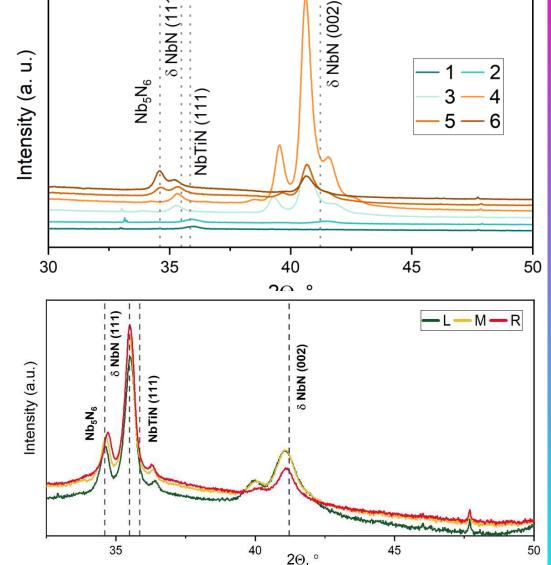


Change the Ti/Nb power ratio:

- Optimum around 500W/300W
- Rocking angle
  - -60° ↔ +20°

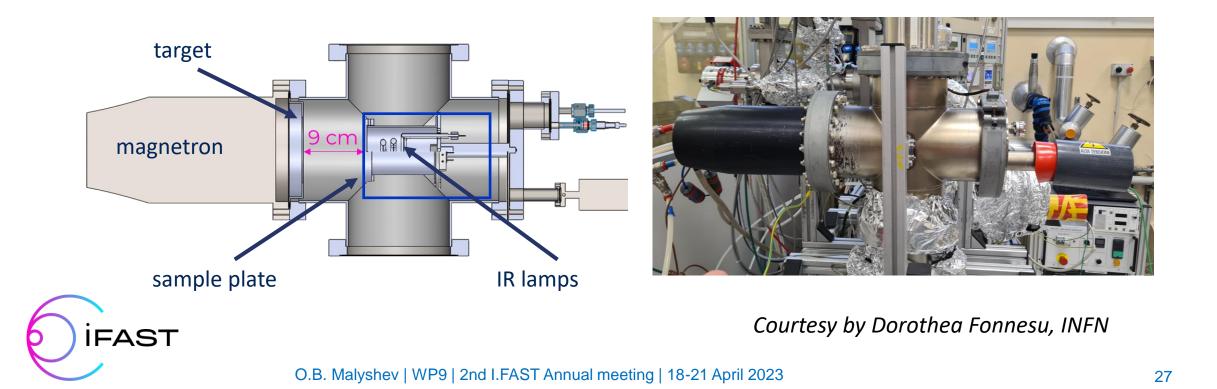
Needs to vary other parameters: p, Ar/N<sub>2</sub>, bias

Courtesy by Alexander Zubtsovskii, USI



# Nb<sub>3</sub>Sn samples coated at INFN

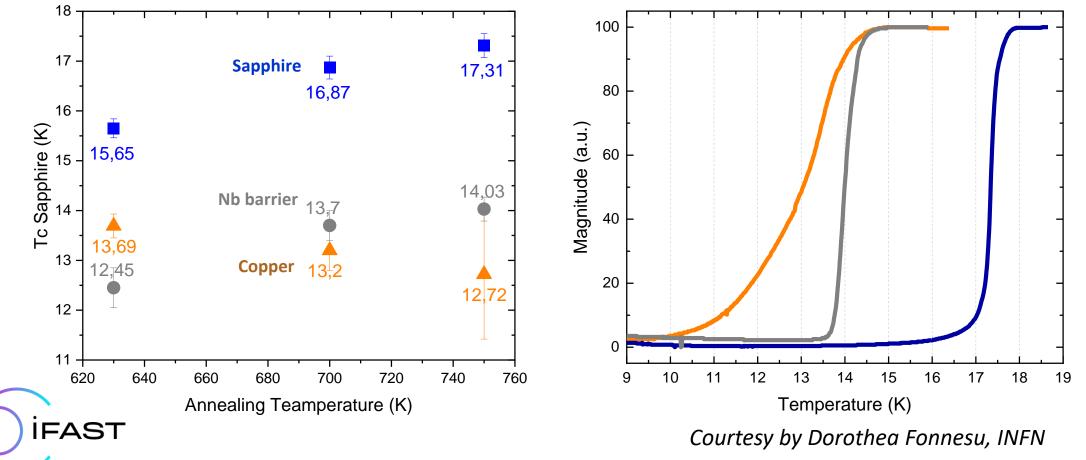
- Commercial Nb<sub>3</sub>Sn stoichiometric planar target (4" diameter)
- System base pressure: 5x10<sup>-9</sup> mbar
- Heated sample holder: up to 950 °C via IR lamps



### Main results for Nb<sub>3</sub>Sn samples coated at INFN

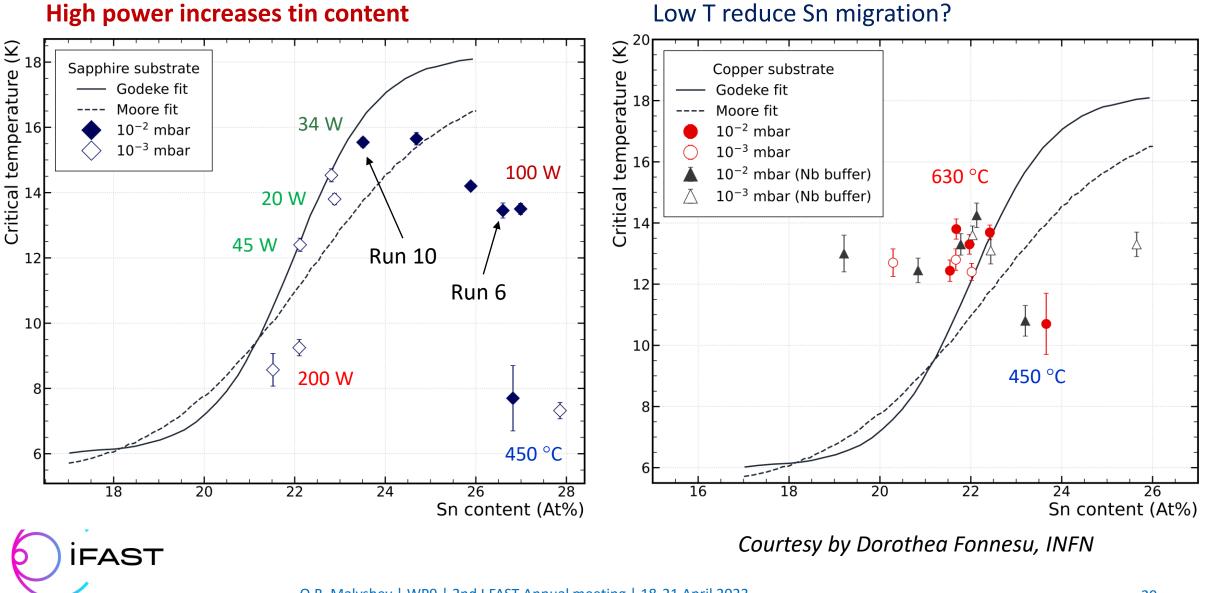
#### Tc vs. Temperature

**Low weakening/melting point of Cu is a limitation** 650 °C can be considered a limit in a Cu cavity Diffusion of Cu into Nb<sub>3</sub>Sn and Sn migration into Cu



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### Main results for Nb<sub>3</sub>Sn samples coated at INFN



# Task 9.3 Part Two: Planar Samples & QPR deposition

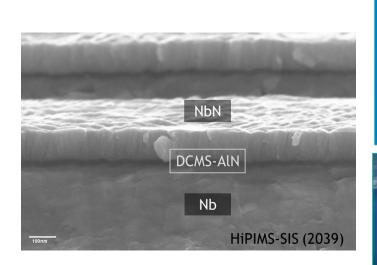
#### Aim:

 Optimise deposition parameters for other high T<sub>c</sub> superconductor and provide sample for other partners for SRF evaluation of the SRF thin Films

#### Why?

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- Optimisation of films still on going
- Alternative surface treatments
- Need to assess RF properties (see Task 9.6)
- Going from flat sample to complex shaped cavities is not straightforward => intermediate step



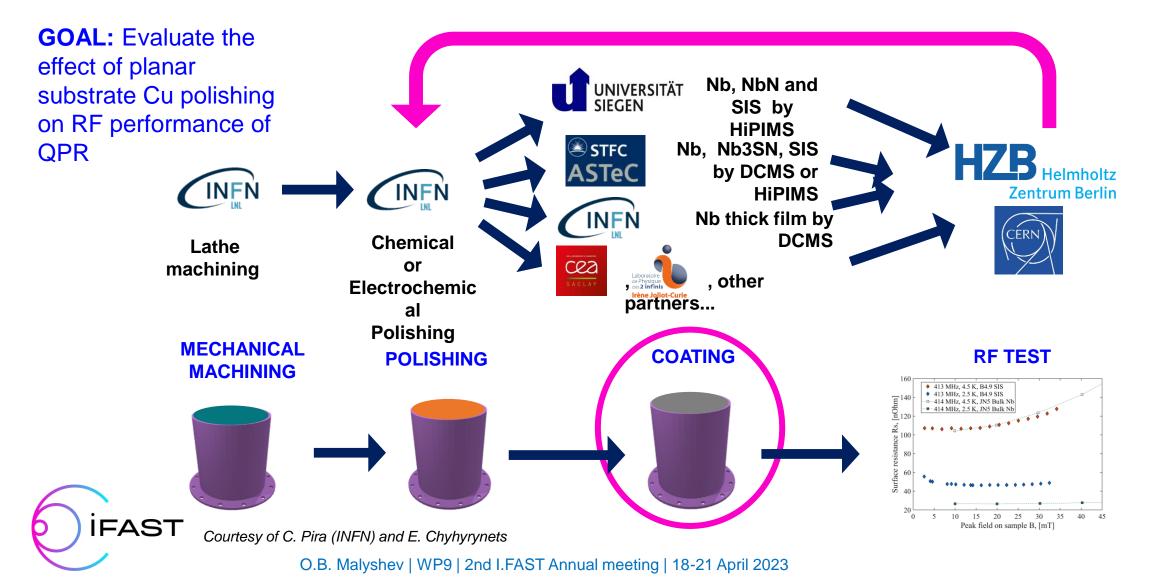
HiPIMS-coated SIS structure on Si



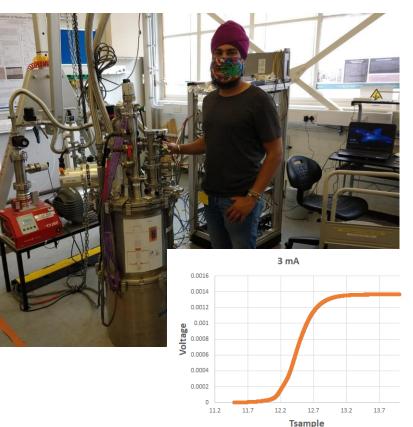


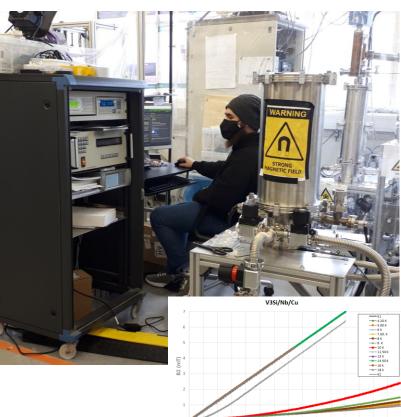


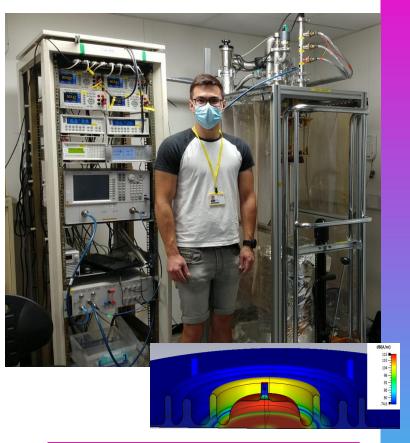
# Task 9.3 Part Two: Planar Samples & QPR deposition



### **Superconducting Properties Evaluation at UKRI/STFC/CI**







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

FAST

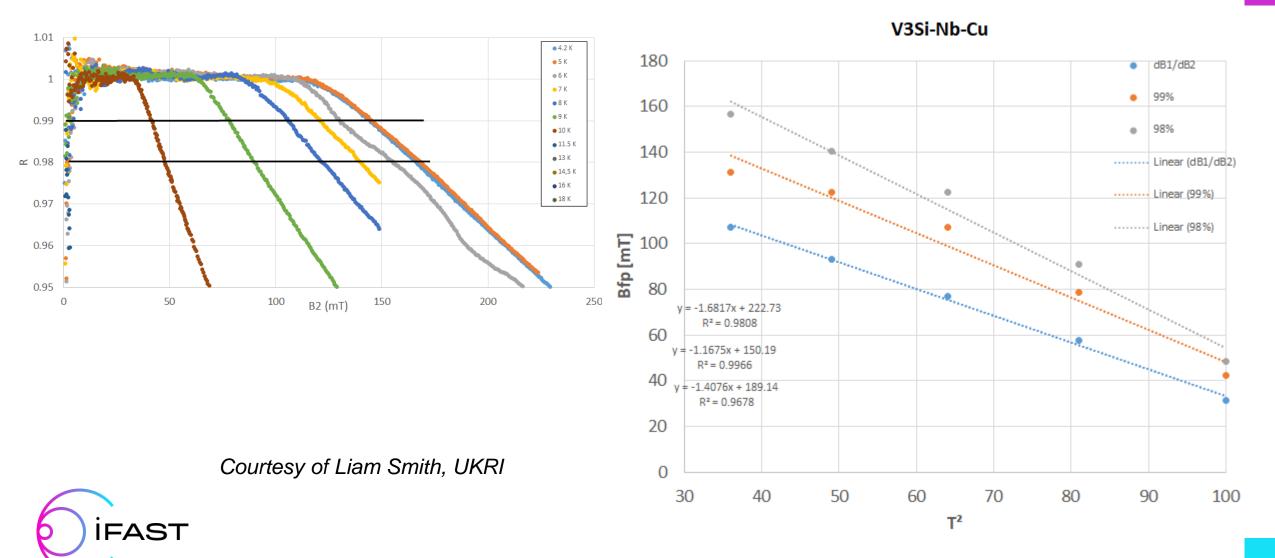
EXP700: Magnetic field penetration facility (in-vacua)

70 80 B1 (mT)

> EXP900: *R*<sub>S</sub> measurements with 7.8 GHz cavity

Courtesy of Taaj Sian, UKRI

### Typical results for magnetic field penetration for V<sub>3</sub>Si/Nb/Cu



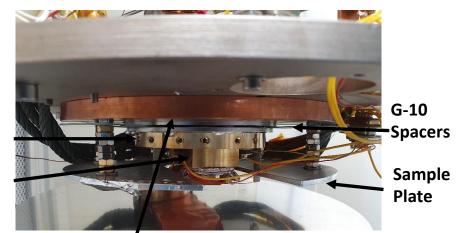
# **The Choke Cavity Facility**

- Two part test cavity in LHe-free cryostat:
  - Bulk Nb choke cavity
  - Planar disk 90 130 mm diameter, 1 - 10 mm thickness
- **RF-DC compensation**  $\rightarrow R_{S}(T, B)$
- VNA measurements  $\rightarrow \Delta f \rightarrow \Delta \lambda$
- Parameters:
  - $f_0 = 7.8 \text{ GHz}$
  - $T_{\text{Sample}} \ge 4 \text{ K}$
  - RF Power up to 1 W (for now!)
  - $B_{\text{sample, pk}} \leq 1 \text{ mT}$

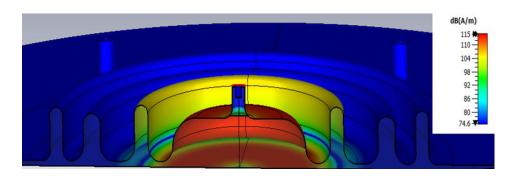


Sample

Sample holder



Cavity



Courtesy by Daniel Seal, Lancaster University

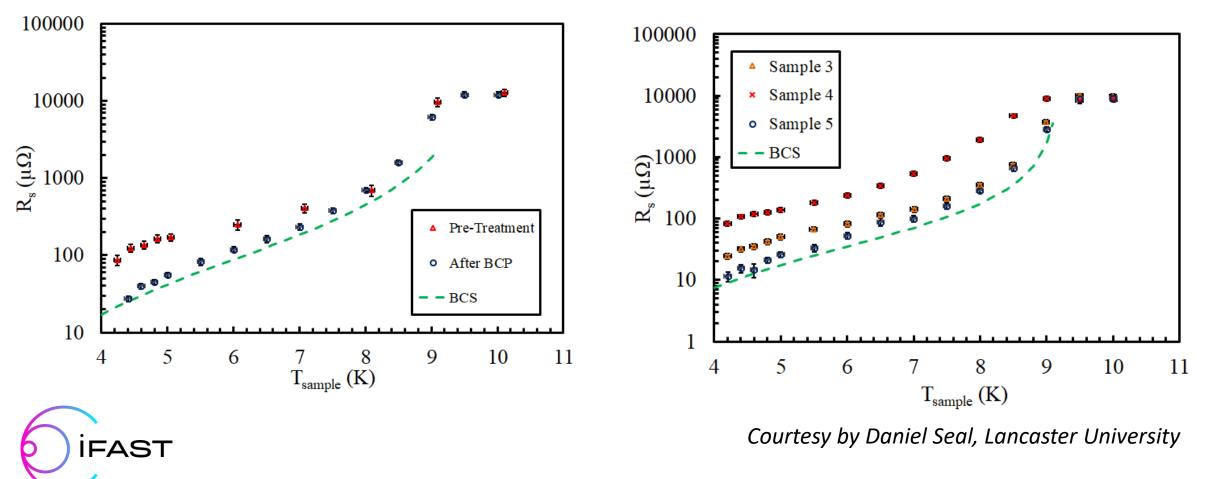
1 sample test R<sub>s</sub>(T)in 2 days



### Results with choked cavity

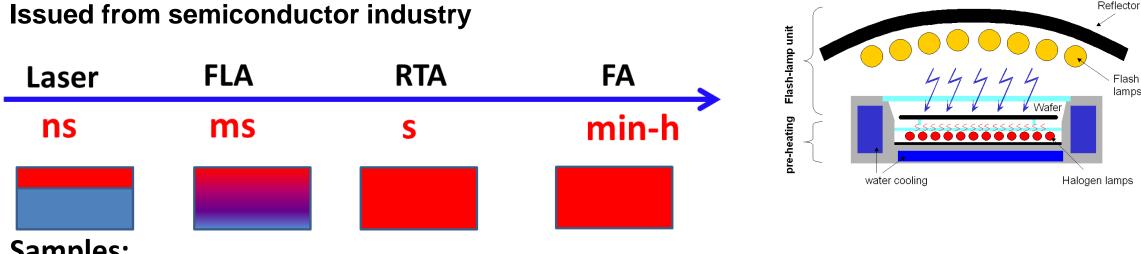
**Bulk Nb**: : RRR = 300, BCP with 60-100  $\mu$ m removed courtesy of E. Chyhyrynets

Nb films: **Sample 3:** 400-450 °C, ~ 3 μm **Sample 4:** RT, ~ 3 μm **Sample 5:** 300-350 °C, ~ 3 μm



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# **FLA processing of Nb-alloys at HZDR**



### Samples:

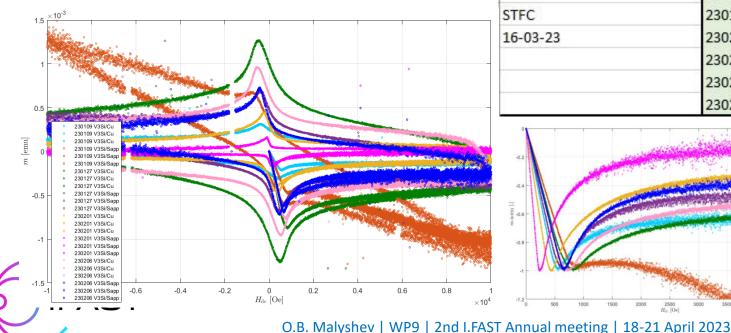
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- Nb, Nb/NbTiN from UKRI: FLA 3.2 ms at 4.0 kV
  - -10 to 40% increase in B<sub>c1</sub>
- Si/AIN/NbTiN from CEA: FLA 3, 6 and 23 ms
  - T<sub>c</sub> increase from 5 to 14.5 K
- Cu/Nb<sub>3</sub>Sn from INFN  $\Rightarrow$ 
  - results to be reported soon

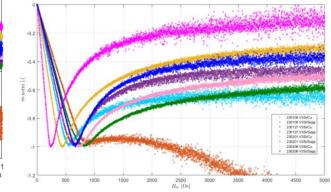
## **Superconducting Properties Evaluation at IEE**

#### Samples from UKRI:

- Nb:
  - laser treated at RTU:
    - $in N_2$
    - in Ar
- V<sub>3</sub>Si films on Cu and Sapphire substrates



	Sample	Ben [Oe] (2% crit. at 4.22 K) Perpend Parallel	Tc [K]
		Perpend Parallel	
Nb / Cu	0_Non_irr	240	9.25
	1_Atm_Laser_max	90	9.15
Arturs RTU	2_Atm_Laser_min	310	9.1
3.2.2023 series	3_Argon_Laser_max		7 ?
	4_Argon_Laser_min	2	7 ?
V3Si	230109_Cu	190	12.8
Substrates: Cu, Sapphire	230109_Sapp	610	14
	230127_Cu	510	13
STFC	230127_Sapp	470	11.5
16-03-23	230201_Cu	330	10.5
	230201_Sapp	200	8.2
	230206_Cu	530	12.5
	230206 Sapp	580	11.5



Courtesy of Eugen Seiler, IEE

# Task 9.4:Surface engineering by atomic layer deposition (ALD) Task Leader: Thomas Proslier(CEA)

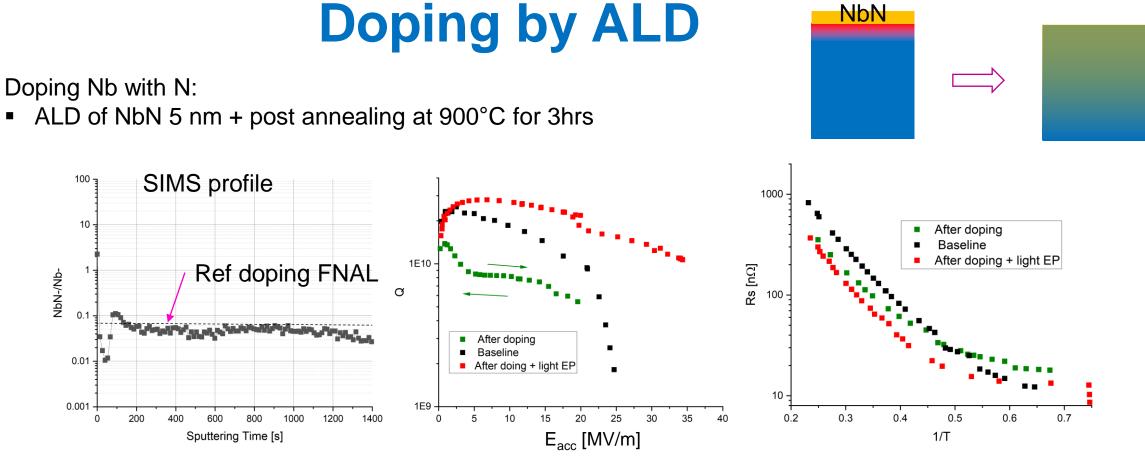
Aim:

- Deposition of functionalized layers :
  - Low secondary yield cap layer (
     multipacting)
  - SIS multilayers
  - Dielectric surface engineering and doping
- Development of a 1.3 GHz deposition set-up

Why?

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- ALD = highly conformational => adapted to complex shapes
- Chemical technique => wide range of compounds manageable in the same deposition set-up
- Can be used to upgrade Bulk Nb cavities



- N Doping profile by SIMS similar to reference recipe from FNAL.
  - Better quality factor at 4,2K but reduced performance at 2K as compare to baseline.
  - Possibility to tune surface dopant and oxide protective layer by trying other ALD layers: TiN etc...

FAST

-2

-6

-8

-10

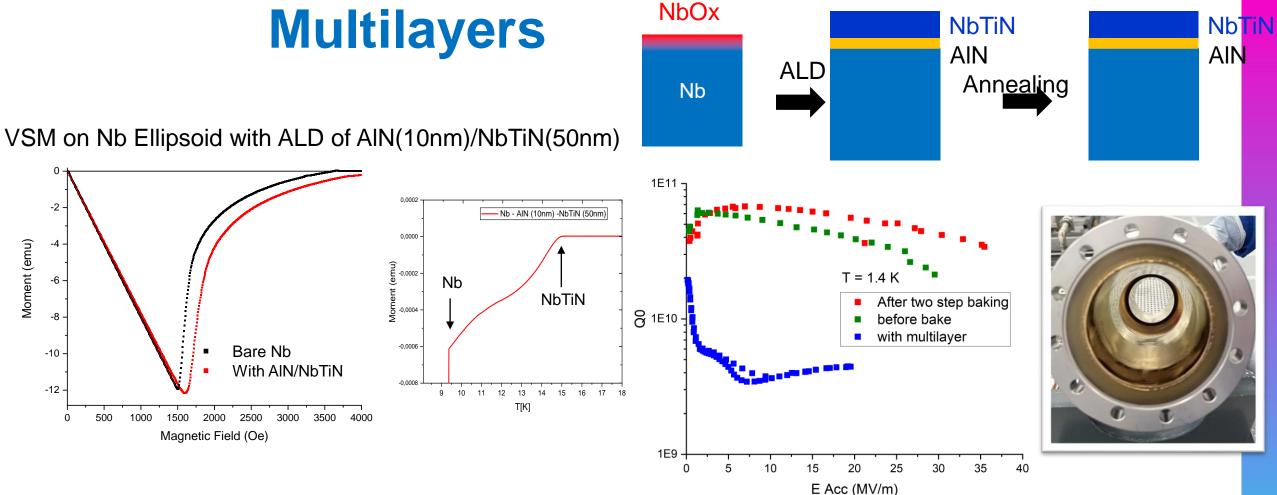
-12

0

1000

500

Moment (emu)



- Increased penetration field with AIN/NbTiN multilayer on Nb.
- First deposition on a cavity coating successfull (but unexpected delamination in the beamtubes during the post annealing).
- Recovery of good performances after EP and HPR.
- Work in progress: currently limited HR ressources in clean rooms and LHe for RF test. FAST

# Task 9.4 results summary



 Atomic Layer deposition strength is its scalable capability: from bench to industrial scale (coupons to cavities and much more high aspect ratio and large surface objects).

#### **Achievement within IFAST Task 9.4:**

- Control and reduction by ALD of the secondary electron yield and the resulting multipacting mitigation.
- Control and optimisation of the superconducting multilayer properties by ALD (Tc =15 K).
- Homogeneous deposition and multilayer properties on Nb and Cu 1.3 GHz cavities.
- Compatible with cavity surface treatments (stability after HPR, thermal treatments...)
- New doping approach and dielectric layer engineering by ALD.



# Task 9.5: Improvement of mechanical and superconducting properties of RF resonator by laser radiation

Task Leader: Arturs Medvids (RTU)

#### Aim:

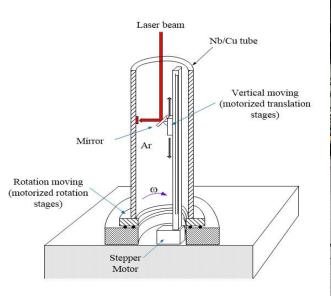
- Pre-and Post-surface Laser treatment
  - On copper, to improve film deposition
  - On deposited films, to improve their crystalline quality

Why?

- Smoothing of surfaces
- Recrystallization
- Decreasing porosities



## The laser facility for irradiation inner surface of RF cavity



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

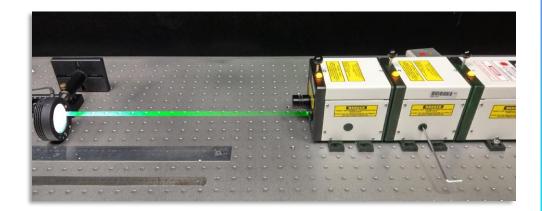




Scanning system for cylindrical copper tubes Nd:YAG laser.

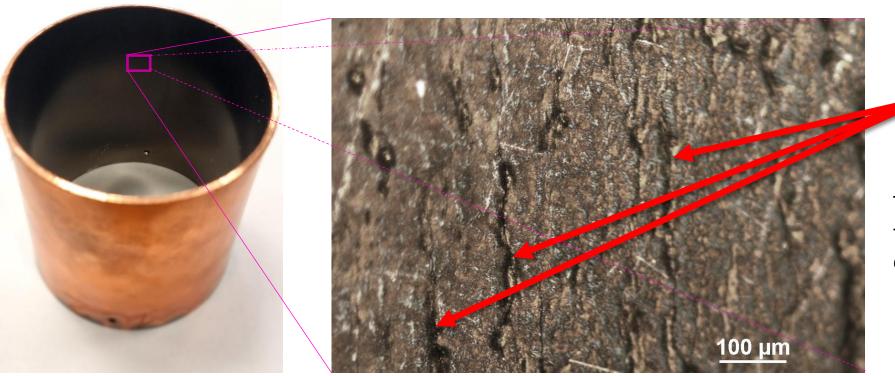
#### Nanosecond Nd:YAG laser,

- wavelength 1064 nm,
- pulse duration 6 ns,
- repetition rate of 10 Hz
- beam diameter of 0.5 mm.
- Scanning of the laser beam was performed normally to the surface with a speed of 1.2 mm/s and hatch of 0.4 mm.
- The irradiation of the samples was carried out at room temperature in Ar chamber to prevent oxidation.



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## Applying laser on Nb coated tube Optical microscope image



Cracks

The samples were sent back to UKRI for determination of reasons of cracks.

Nb coated OFHC copper tube

Optical microscope image of the surface of a cylindrical copper tube with a thin film of niobium (Nb) before laser processing.



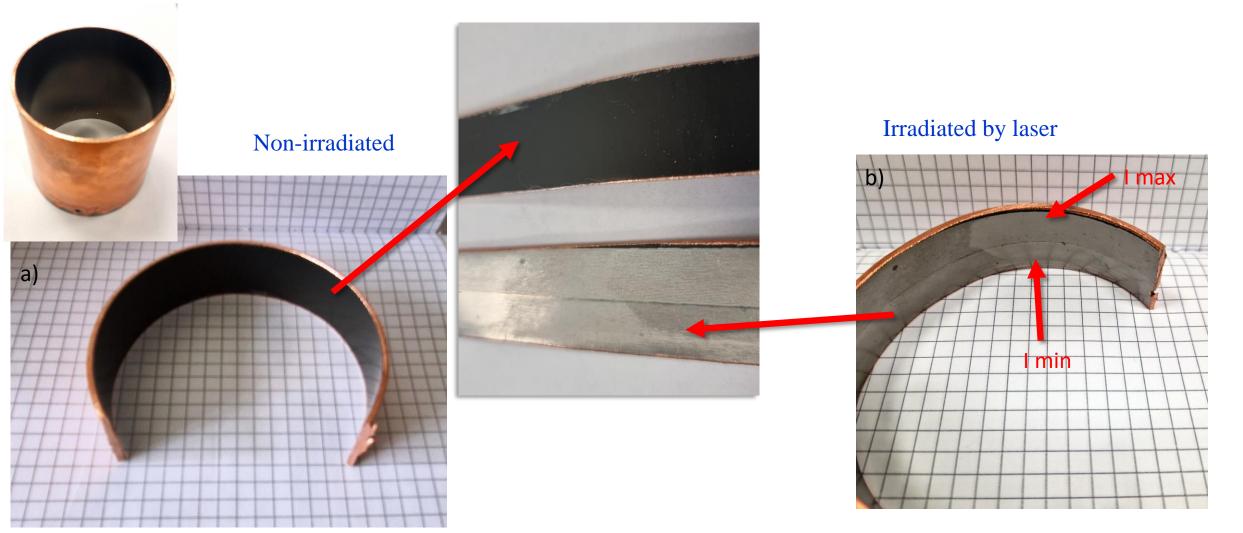


Fig.5. Samples formed from cylindrical copper tube with Nb film: (a) non-irradiated; (b) irradiated in Ar chamber by ns laser radiation.



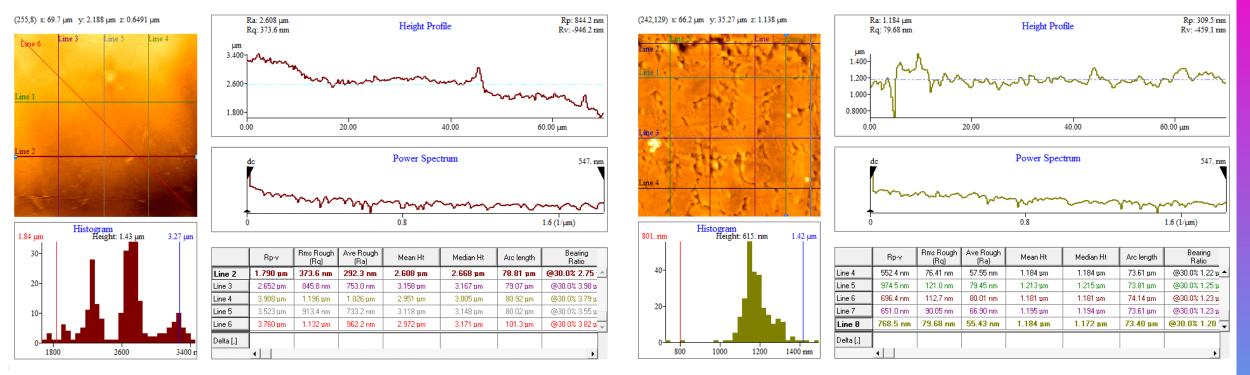
**References:** 

1. C. K. Gupta, A. K. Suri, S Gupta, K Gupta (1994), *Extractive Metallurgy of Niobium*, CRC Press, <u>ISBN 0-8493-6071-4</u> <u>0.B. Malyshev | WP9 | 2nd I.FAST Annual meeting | 18-21 April 2023</u> 45

#### Non irradiated surface texture parameters

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#### **Irradiated surface texture parameters**



- 1. The cylindrical copper tubes with Nb were effectively scanned using laser radiation in an Ar chamber.
- 2. X-ray diffraction (XRD) analysis showed that there were no traces of niobium oxide present in either the non-irradiated or irradiated samples treated with the Nd:YAG laser.
- 3. The irradiated samples' surface roughness (Ra) decreased by more than ten times compared to the nonirradiated samples.
- 4. The number of cracks on the irradiated samples increased, they became smaller in size after laser processing.

# **Task 9.6:**Optimization of flat SRF thin films production procedure

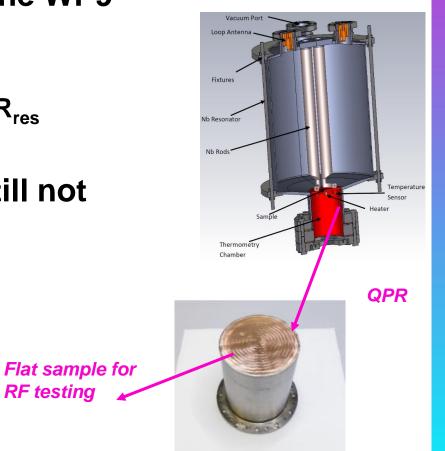
Task Leader: Oliver Kugeler (HZB)

#### Aim:

- RF testing of the films developped throughout the WP9
  - Sample is small enough for easy handling
  - Smaple is flat (one problem less in the way)
  - 3 ≠ frequencies available : large exploration (R<sub>BCS</sub> vs R<sub>res</sub>

### Why?

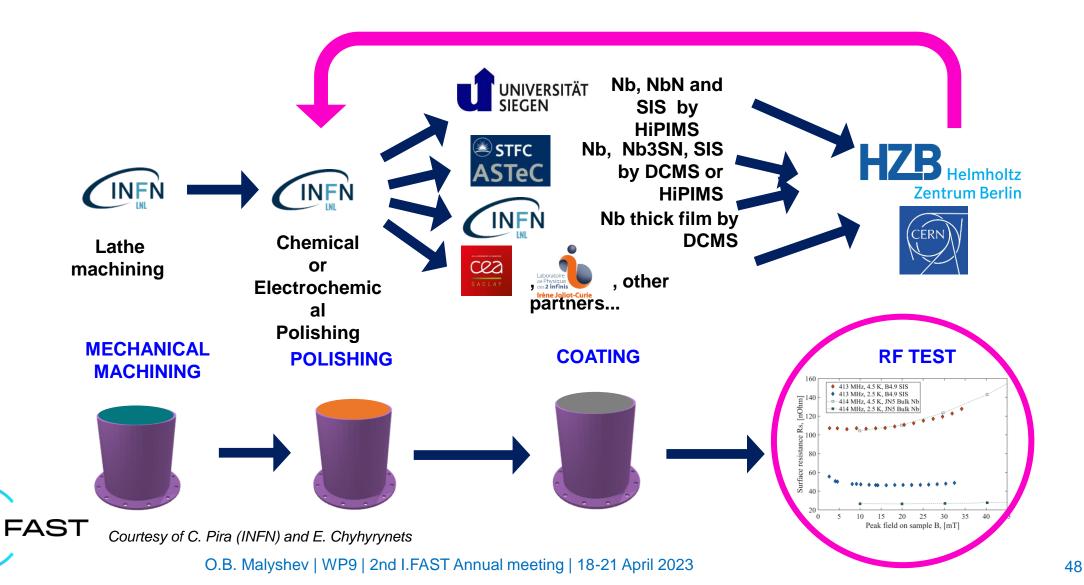
 Material characterization, even advanced are still not predictive of future RF behavior



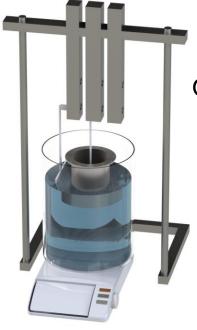
**RF** testing



# Task 9.6:Optimization of flat SRF thin films production procedure

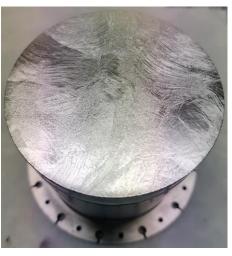


## Plasma electropolishing on QPR samples @ INFN



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Old system



Initial Nb QPR sample

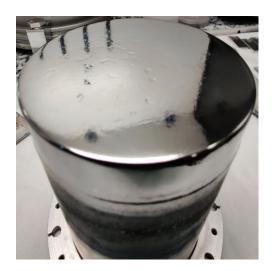




Surface improvement after 10 min

Upgraded system

- Larger volume
- More stable temperature
- More stable current



100-µm removal in 60 min. Mirror finish

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# **Polishing QPR samples @ INFN**

#### Cleaning

1. PEP disk + 3 cm 2. Bulk QPR SUBU



#### Courtesy of Eduard Chyhyrynets, INFN

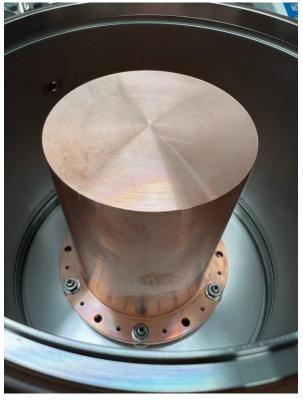
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A1 Nb

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## **RF-performance measurements of films with QPR**

- Decision to use bulk copper QPR sample instead of brazed Cu/Nb samples as used in ARIES
- First Copper Sample C1 manufactured at HZB and shipped to INFN Legnaro for cleaning.
- Next step will be: Nb coating at Siegen University (for minimisation of unwanted sample RF-heating) and subsequent coating with SIS structure.



Courtesy of Oliver Kugeler, HZB



## **Bulk Nb QPR samples – scheduled for future**

One Nb bulk QPR sample was cleaned at Saclay and is currently scheduled for baseline testing in the QPR in CW 20.

This sample will then be shipped to Daresbury for coating with suitable SIS structure and subsequent RF characterisation with QPR at HZB

Two more Nb samples are currently scheduled for baseline surface preparation at Saclay for further processing (tbd)

One Nb bulk sample was baseline-tested in the QPR at HZB and then shipped back to Saclay for coating with an insulator layer by ALD. The impact of the passivation coating on the baseline performance will be measured in the QPR at HZB



Courtesy of Oliver Kugeler, HZB

## **CONCLUSIONs**

Things are going according to IFAST WP9 plan
4 milestones already achieved.



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## Thanks for your attention!



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