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

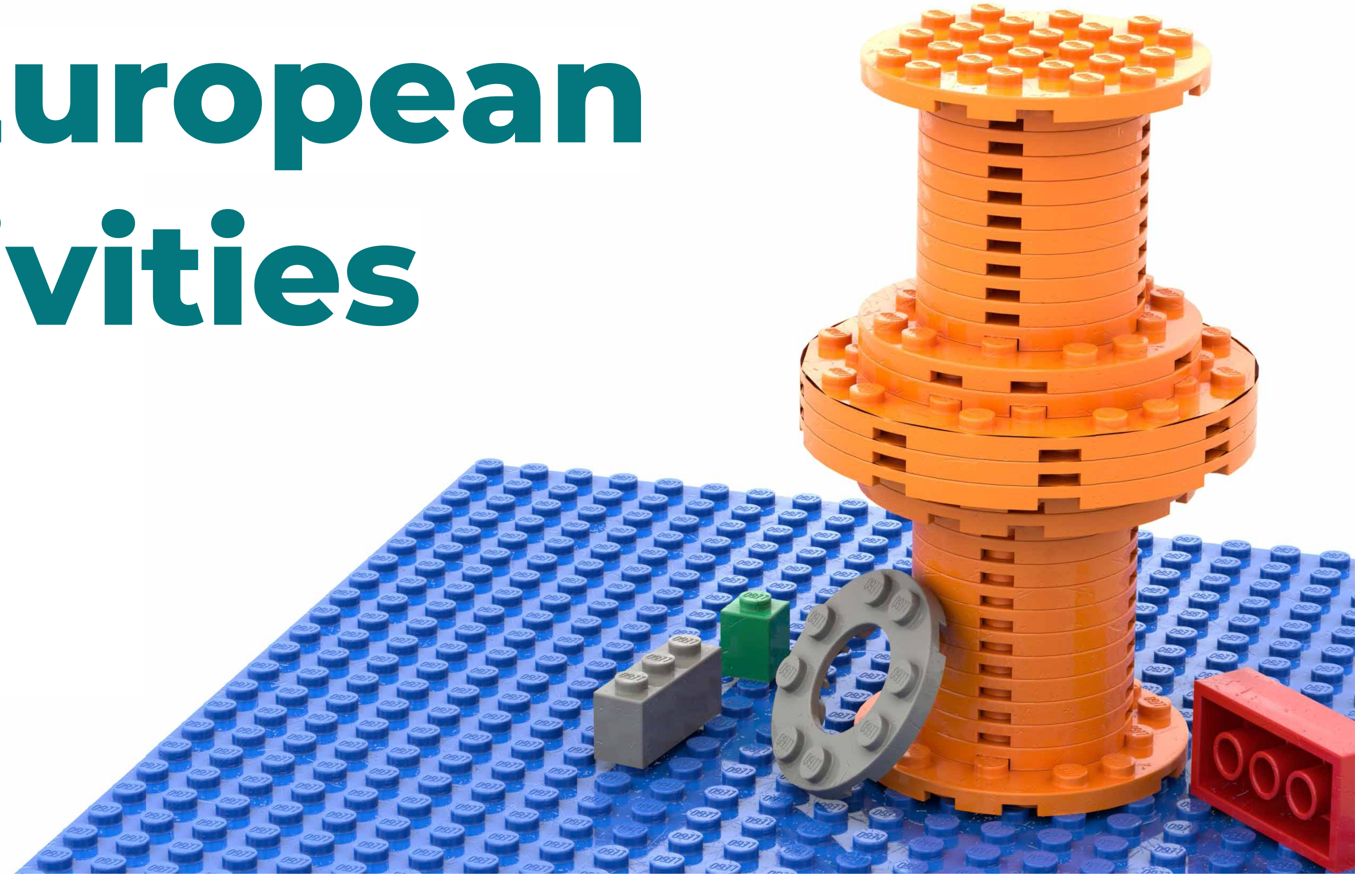
iFAST

INFN LNL

Cristian Pira

On Behalf of I.FAST WP9 Collaboration

Progress in European Thin film activities



SRF 2023 GRAND RAPIDS

28 June 2023

I.FAST WP9 Collaboration

C. Pira, O. Azzolini, R. Caforio, E. Chyhyrynets, D. Fonnesu, D. Ford, V. Garcia, G. Keppel, G. Marconato, A. Salmaso, F. Stivanello (LNL)
M. Bertucci, R. Paparella (LASA)
M. Bonesso, S. Candela, V. Candela, R. Dima, G. Favero, A. Pepato, P. Rebesan, M. Romanato (PD)

C.Z. Antoine, S. Berry, Y. Kalboussi, T. Proslier

D. Longuevergne, O. Hryhorenko,

E. Seiler, R. Ries

S. Keckert, O. Kugeler, J. Knobloch

S. Prucnal, S. Zhou

X. Jiang, T. Staedler, A. Zubtsovskii

R. Berton, D. Piccoli, F. Piccoli, G. Squizzato, F. Telatin

Medvids, A. Mychko, P. Onufrievs

O.B. Malyshev, R. Valizadeh, C. Benjamin, T. Sian, L. Smith,

G. Burt, N. Leicester, S. Marks, D. Seal, D. Turner

W. Bradley, S. Simon

Associated Partners

A. M. Valente Feliciano

Informal Partners

Energy saving is mandatory for the next generation accelerators…

…cryogenics is one of the larger energy cost in modern SRF accelerators

I.FAST Goal (2025)

Realize a prototype of high performance

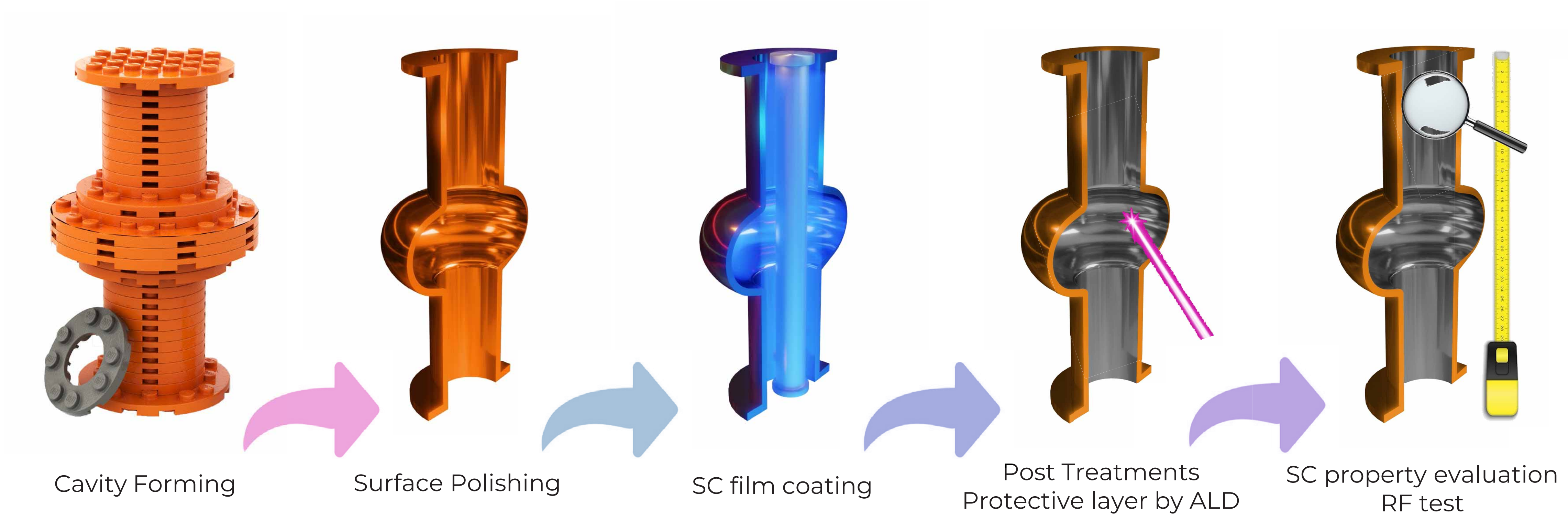
1.3 GHz thin film SRF elliptical cavity

$Q > 1 \cdot 10^{10}$ @ 4.5 K

Multiple challenges:

- A15 are Brittle materials
- Complicate Phase Diagram
- Substrate preparation
- Low melting point substrate
- Interface diffusion
- Target Production
- Necessity of Test RF properties on simple geometry
- …

I.FAST R&D program cover all the production chain



Cavity forming

Seamless Spinning

Forming technology adopted to produce 1.3 GHz and 6 GHz elliptical seamless Cu R&D substrates to all partners

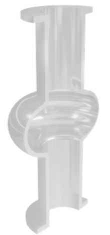
PRIMARY GOAL:

High internal surface quality

OPTIMIZED PRODUCTION PROTOCOL:

- CNC machine
- Reduced Annealing Temperature (400 °C, previous 500 °C)
- New intermediate Deep Drawing Step

Cavity Forming



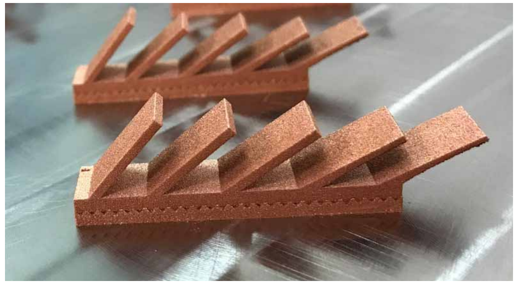
Additive Manufacturing

AM can open to a new way to design RF cavities

GOAL: Prove the possibility to polish properly the surface and get SRF state of the art performances

COPPER

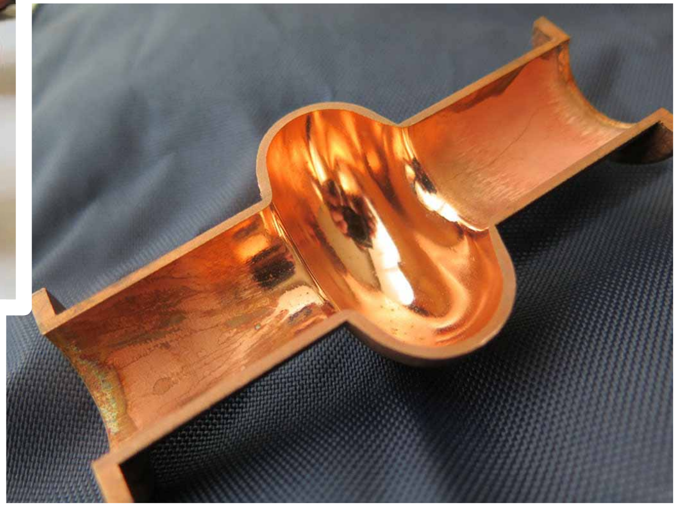
- Density 99.9%
- High quality surface after Vibrotumbling + EP
- Broken after long polishing (>500 μm removed)
- Production of new optimized prototypes ongoing



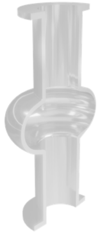
Despite the lower surface quality, Cu can be manufactured with low inclination angles

2 prototypes produced using TRUMPF TruPrint 5000

AM Cavity cutted after VB + EP of the cell



Cavity Forming

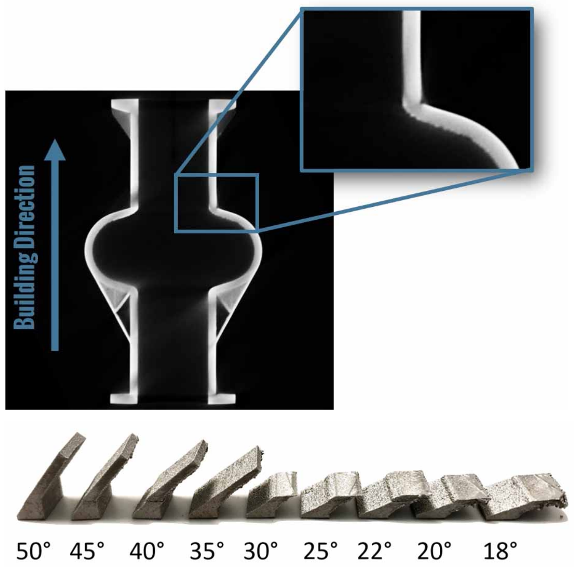


Additive Manufacturing

AM can open to a new way to design RF cavities

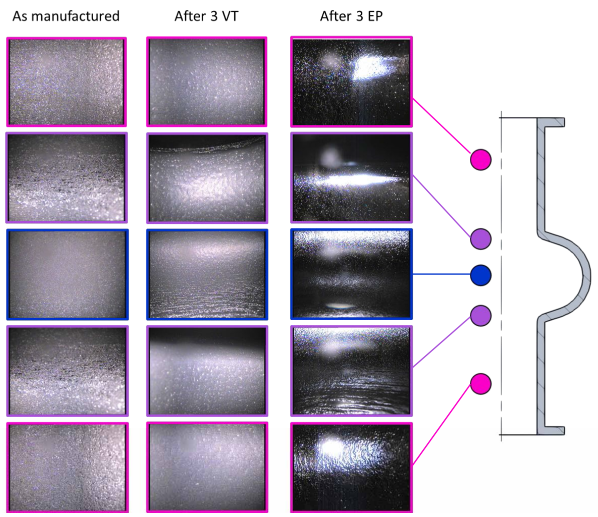
GOAL: Prove the possibility to polish properly the surface and get SRF state of the art performances

NIOBIUM



Down skin optimization

Critical Angle = 35°



Vibrotumbing + EP

Ra = 40 um → 0.4 um

- Tc=9.2 K
- RRR = 10
- No leak at room T
- Ready for cryogenic and RF test

Cavity Forming

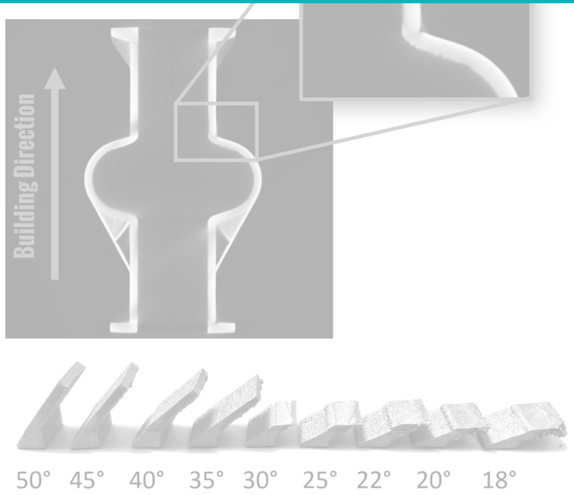
cristian.pira@lnl.infn.it

Additive Manufacturing

AM can open to a new way to design RF cavities

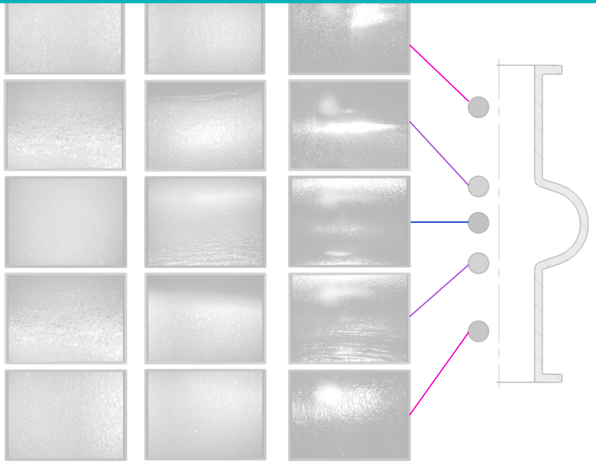
Davide Ford
Poster today: WEPWB119
Additive Manufacturing of Pure Niobium and Copper
Using Laser Powder Bed Fusion for Particle Accelerator Applications

NIOBIUM



Down skin optimization

Critical Angle = 35°



Vibrotumbling + EP

Ra = 40 um → 0.4 um

RRR = 10

No leak at room T

Ready for cryogenic and RF test

Surface polishing

cristian.pira@lnl.infn.it

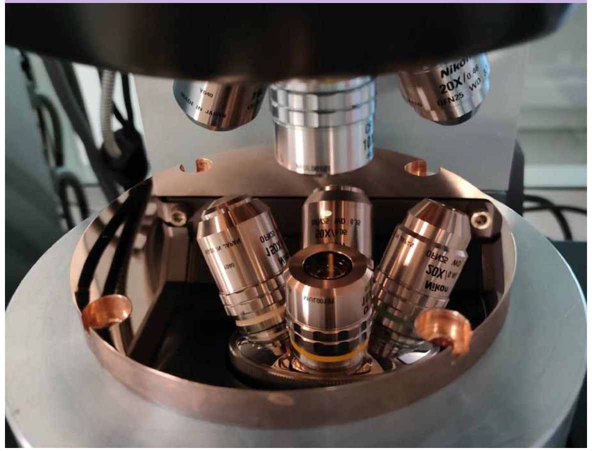
Metallographic Polishing

As received samples

MP polished Nb QPR

MP polished Nb RF disk

MP polished Cu RF disk



Courtesy of Oleksandr Hryhorenko, Claire Antoine and David Longuevergne

Metallographic Polishing

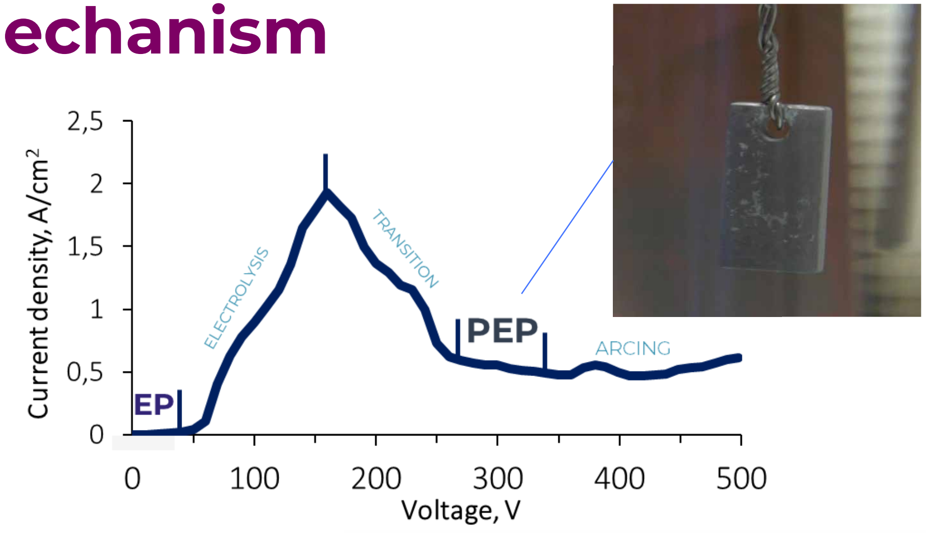
Oleksandr Hryhorenko
Talk today at 12:20: WEIXA06
Recent advances on metallographic polishing for SRF application

Oleksandr Hryhorenko
Poster today: WEPWB050
Exploring innovative pathway of cavity fabrication for SRF application

Courtesy of Oleksandr Hryhorenko, Claire Antoine and David Longuevergne

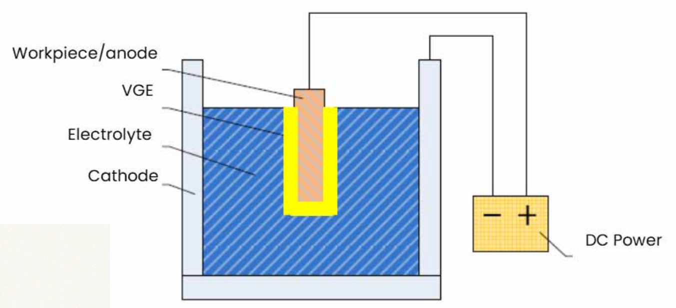
Plasma Electrolytic Polishing PEP

Mechanism

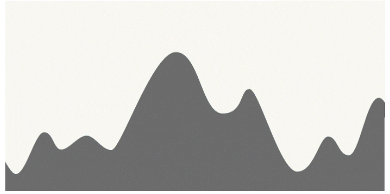


Same EP set-up

Different regime



J. Wang et al., AMR, 2012



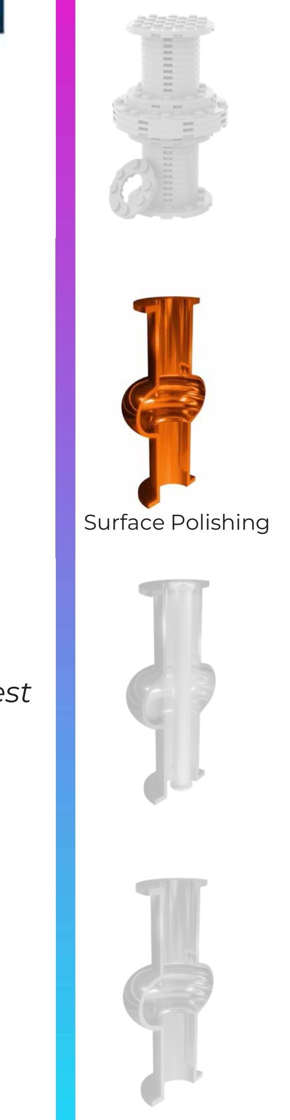
Advantages

“Green” water-based salt solutions! No HF acid!
Nb - NH_4F & NaF, Cu – SUBU5 or $(NH_4)_2HPO_4$ or $K_2P_2O_7$

5-30 times faster than regular EP
Up to 30 µm/min, significant reduction polishing time
From a full day to a couple of hours to treat a cavity!

Lowest roughness 5-50 nm achievable
Efficient polishing! Equal thickness removal yield lowest roughness among traditional polishing (EP, BCP, SUBU)

No preparation of surface is required
PEP can substitute mechanical polishing steps (CBP, grinding, tumbling)

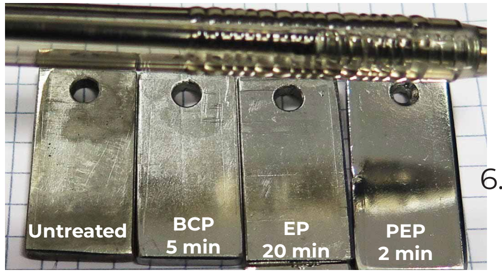


Plasma Electrolytic Polishing PEP Results

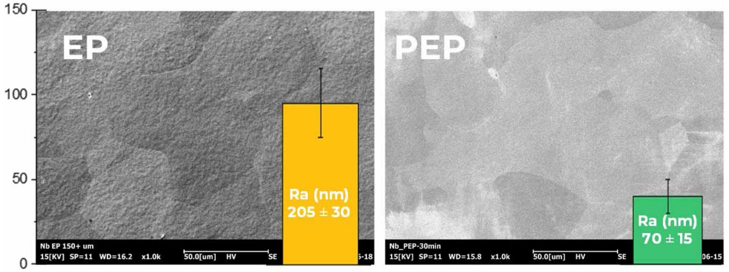
1x Nb 3x Cu

Solution Patentees by INFN

Nb planar samples

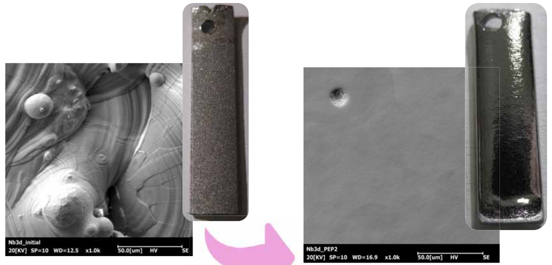


6.5 μm removed



150 μm removed in ~ 5 h

150 μm removed in ~ 40 min



Initial
Ra= 13 μm

PEP 30 min
Ra= 1.5 μm

Additive Manufacturing

Nb QPR polishing optimizaztion on-going

Full Cu QPR ready for coating

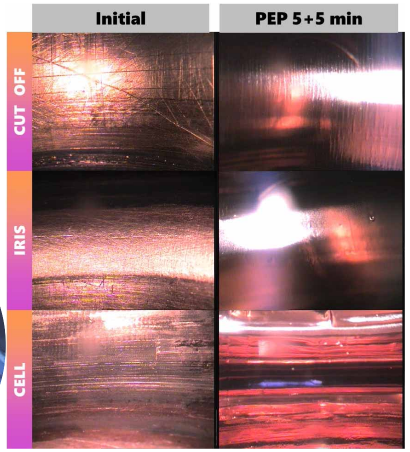
QPR Samples HZB Helmholtz Zentrum Berlin

6 GHz Cu cavity

No internal cathode was used!

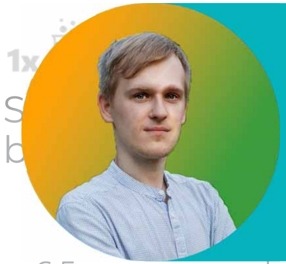
70 μm removed in 10 minutes

30 A (100 cm2 → 1.3 GHz ~ 300 A)



Plasma Electrolytic Polishing PEP
Results

Nb QPR polishing optimizaztion on-going



Eduard Chyhyrynets
Poster on Moday: MOPMB009
Plasma Electrolytic Polishing
Technology Progress Development for Nb and Cu Substrate Preparation

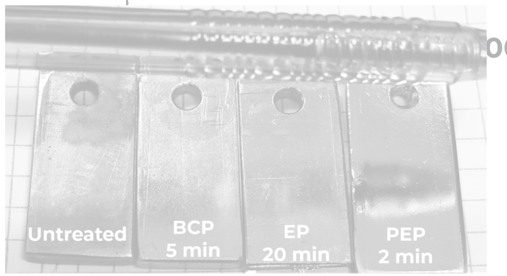
1x
S
b

6.5 µm removed

Ra= 13 µm
Ra= 1.5 µm
1000 X
000 X

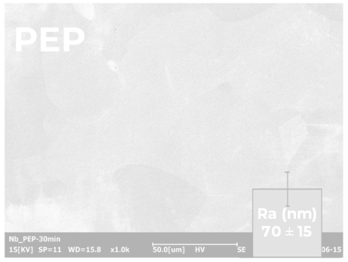
Additive Manufacturing

QPR Samples HZB Helmholtz Zentrum Berlin



Nb planar samples

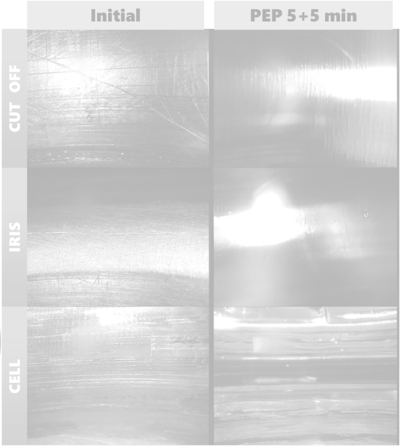
150 µm removed in ~ 5 h



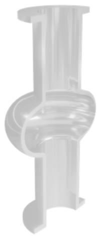
150 µm removed in ~ 40 min

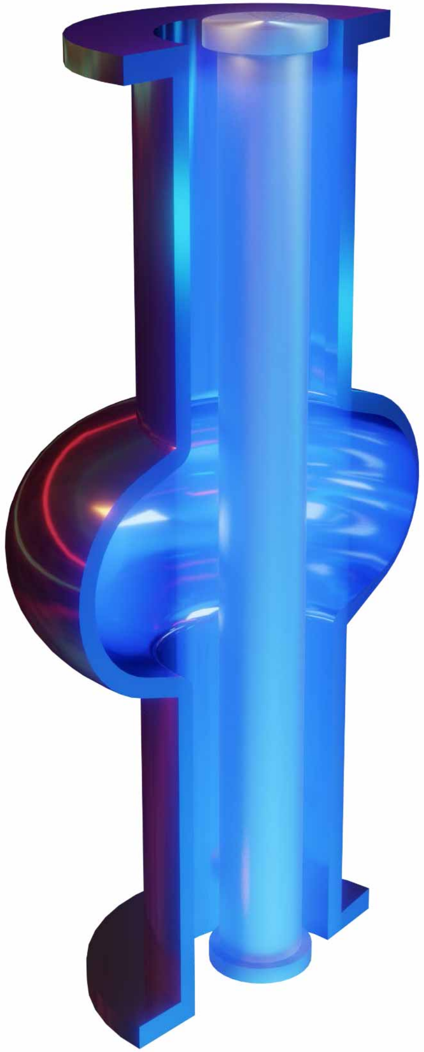
6 GHz Cu cavity

No internal cathode was used
70 µm removed in 10 minutes
30 A (100 cm2 → 1.3 GHz ~ 300 A)




Surface Polishing



SC Coatings

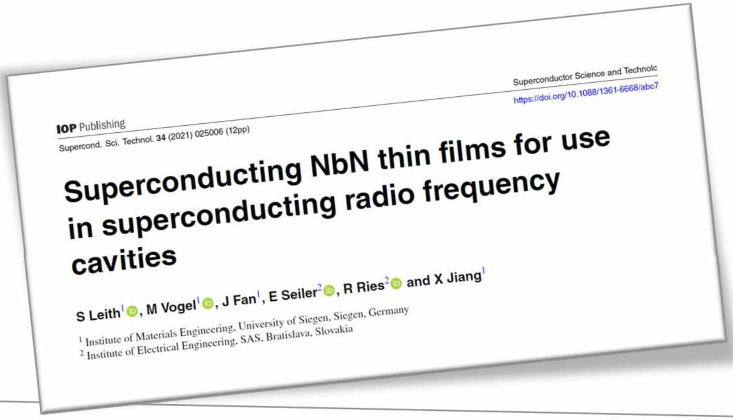
NbTiN SC coatings

UNIVERSITÄT SIEGEN

LOT Lehrstuhl für Oberflächen- und Werkstofftechnologie

From **NbN** to **NbTiN** by HiPIMS

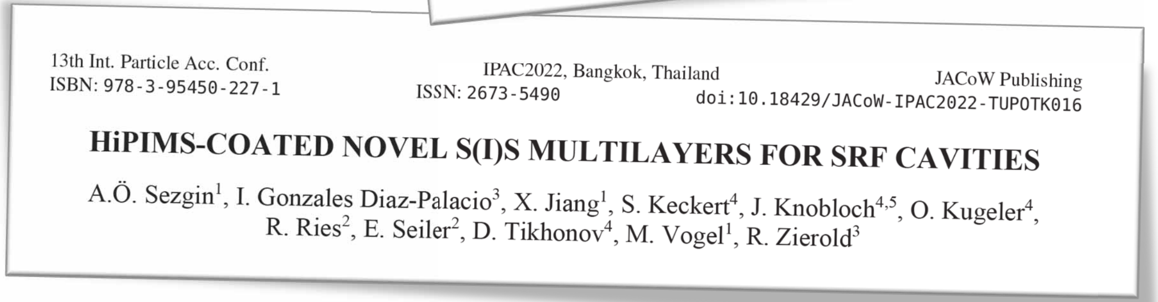
Goal: **SIS structure** by PVD+ALD


IOP Publishing Superconductor Science and Technology
Supercond. Sci. Technol. 34 (2021) 025006 (12pp) https://doi.org/10.1088/1361-6668/abc7

Superconducting NbN thin films for use in superconducting radio frequency cavities

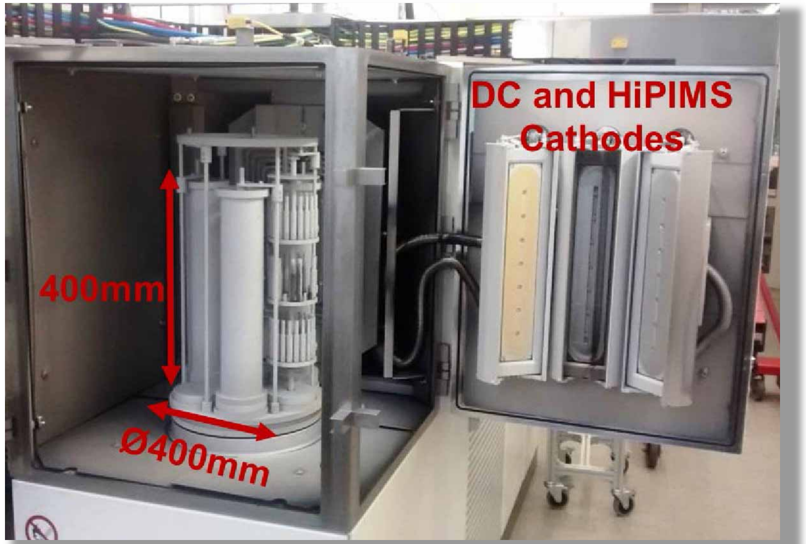
S Leith[1], M Vogel[1], J Fan[1], E Seiler[2], R Ries[2] and X Jiang[1]

[1] Institute of Materials Engineering, University of Siegen, Siegen, Germany
[2] Institute of Electrical Engineering, SAS, Bratislava, Slovakia


13th Int. Particle Acc. Conf. IPAC2022, Bangkok, Thailand JACoW Publishing
ISBN: 978-3-95450-227-1 ISSN: 2673-5490 doi:10.18429/JACoW-IPAC2022-TUPOTK016

HiPIMS-COATED NOVEL S(I)S MULTILAYERS FOR SRF CAVITIES

A.Ö. Sezgin[1], I. Gonzales Diaz-Palacio[3], X. Jiang[1], S. Keckert[4], J. Knobloch[4,5], O. Kugeler[4], R. Ries[2], E. Seiler[2], D. Tikhonov[4], M. Vogel[1], R. Zierold[3]



Courtesy of A. Zubtsovskii

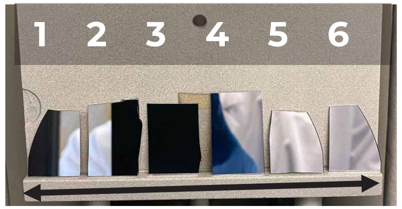
NbTiN SC coatings

Universität Siegen — LOT Lehrstuhl für Oberflächen- und Werkstofftechnologie

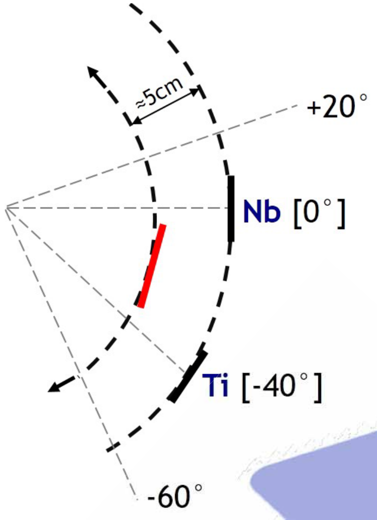
PARAMETER OPTIMIZATION

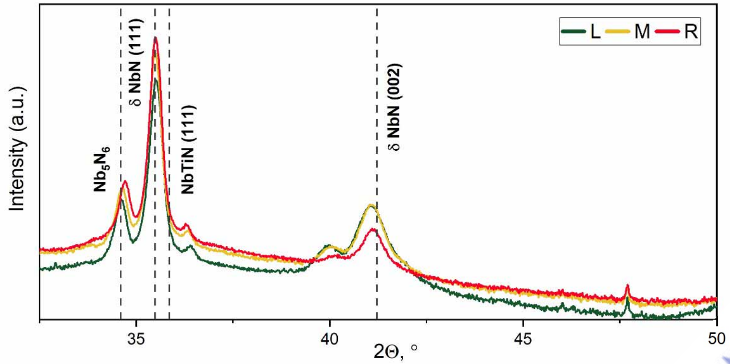
Defined the **optimal rocking angle** in Nb-Ti co-sputtering

Rocking angle	
-40	0
-45	+5
-55	+15
-60	+20
-65	+25
-70	+30



Sample stage 120mm

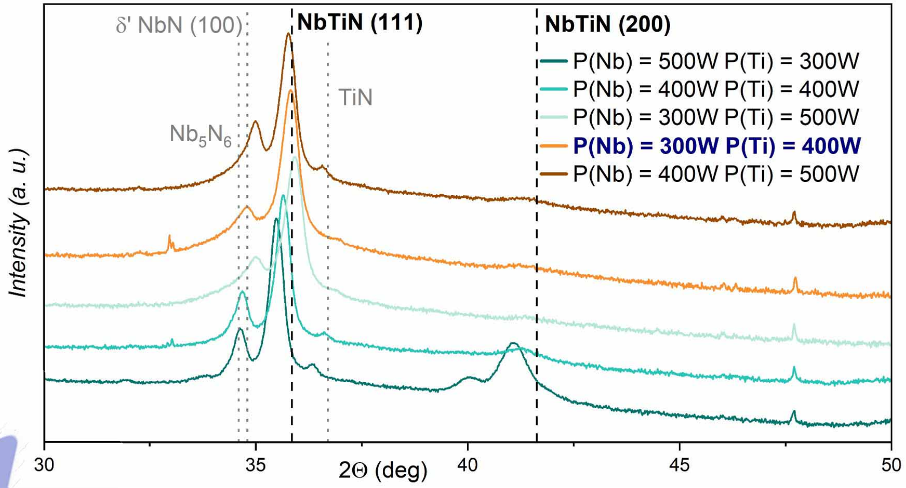




Optimal rocking angle, but wrong phase...

Defined the **optimal Cathode Power:**

P(Nb) = 300W P(Ti) = 400W



Right NbTiN phase obtained

Other parameters (P, Ar/N2 ratio, bias V, ...) must be explored to reduce spurius phases

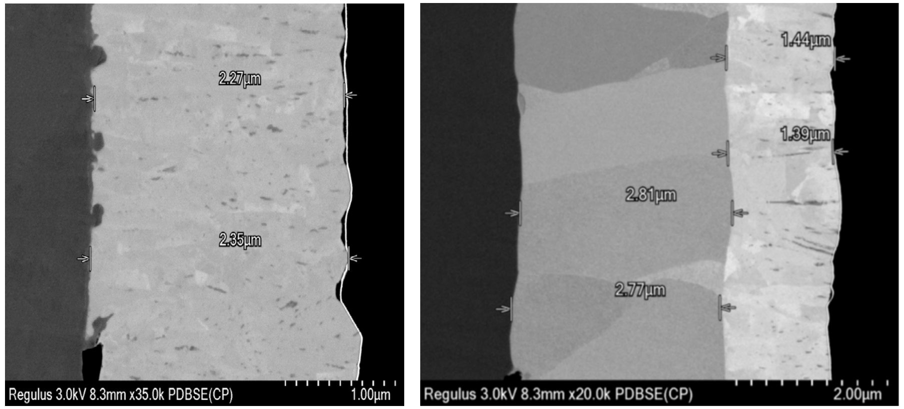
Courtesy of A. Zubtsovskii

SC coatings

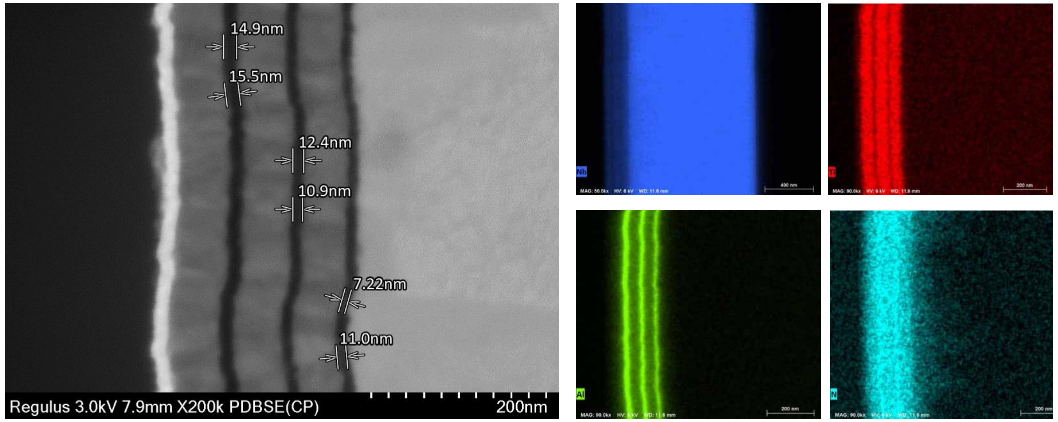
cristian.pira@lnl.infn.it INFN LNL

Nb_3Sn, V_3Si, NbTiN SC coatings

Science and Technology Facilities Council

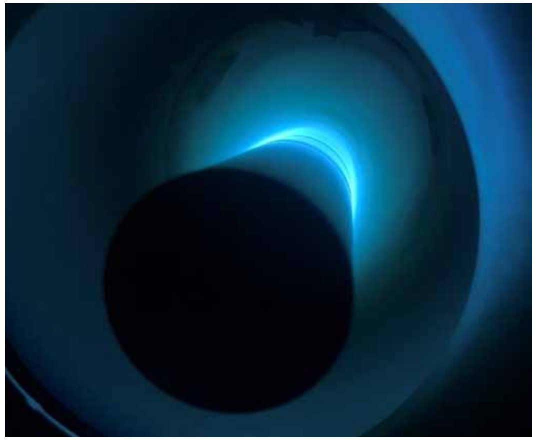


Nb_3Sn on Cu and Nb_3Sn on Cu with Nb barrier layer

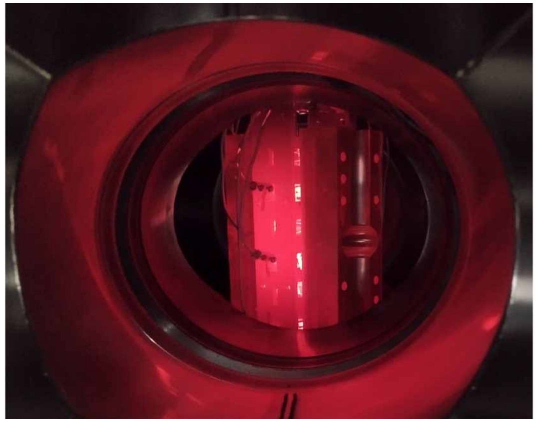


SIS multilayer structure (Nb/AlN/Nb_3Sn) deposited on Ta

A QPR sample during and after the Nb depoosition



Development of 6 GHz and 1.3 GHz cavity coating system



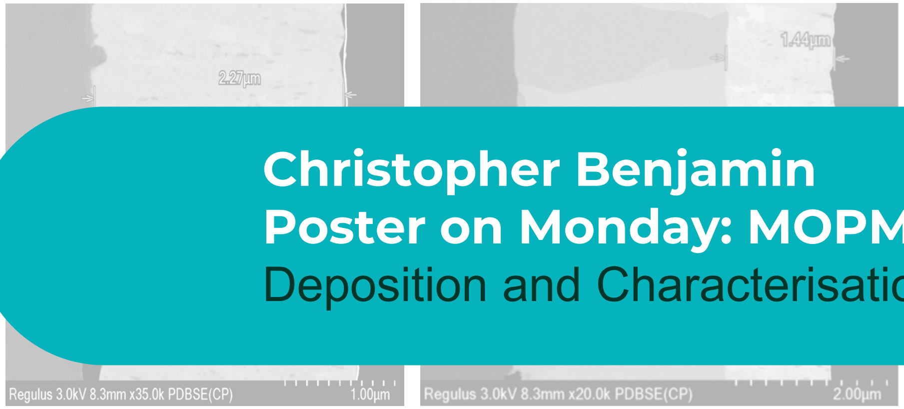
Coating of 6 GHz split cavity

Courtesy of R. Valizadeh

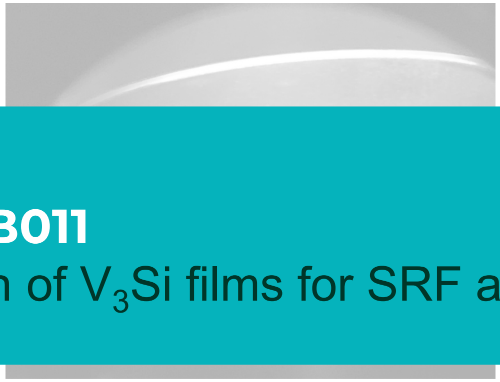
SC coatings

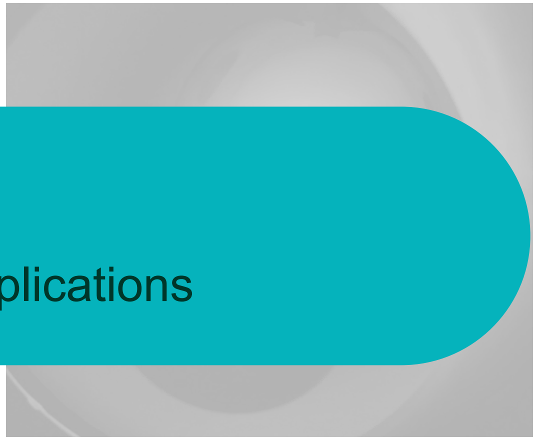
Nb_3Sn, V_3Si, NbTiN SC coatings

Science and Technology Facilities Council



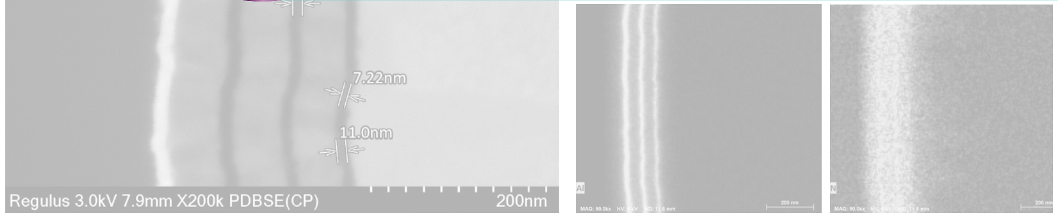
Nb_3Sn on Cu and Nb3Sn on Cu with Nb barrier layer





Christopher Benjamin
Poster on Monday: MOPMB011
Deposition and Characterisation of V_3Si films for SRF applications

Daniel .J. Seal
Poster on Monday: MOPMB062
Optimisation Of Niobium Thin Film Deposition Parameters For SRF Cavities



SIS multilayer structure (Nb/AlN/Nb_3Sn) deposited on Ta

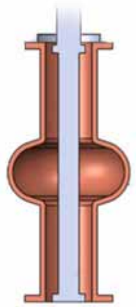
A QPR sample during and after the Nb depoosition

Coating of 6 GHz split cavity

Courtesy of R. Valizadeh

iFAST

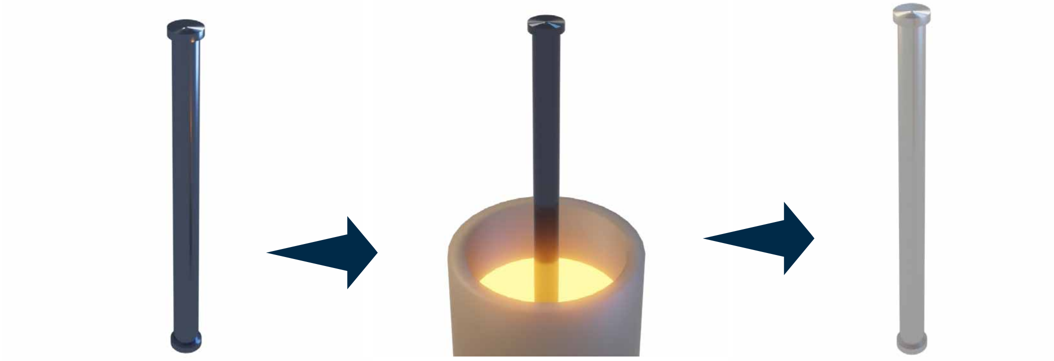
Nb_3Sn coatings



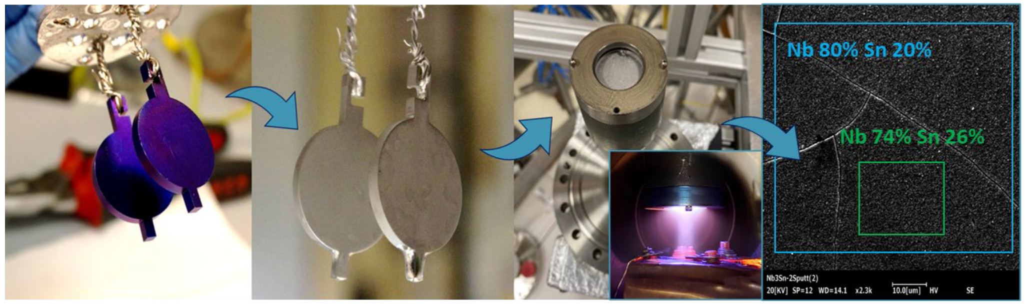
Single target configuration easiest to scale onto elliptical geometry

Nb_3Sn cylindrical target are not commercially available

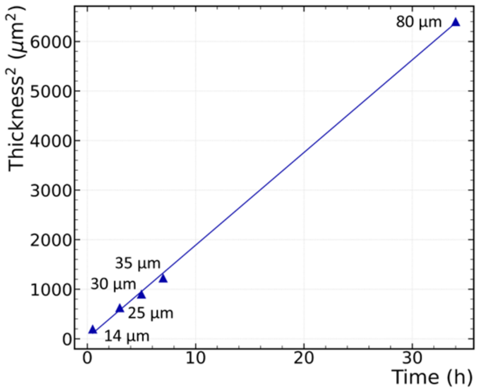
LNL Strategy for Nb_3Sn cylindrical target production



Raw Nb target → *Dipping in liquid Sn* → *Nb_3Sn cylindrical target*



Proof of concept



Nb_3Sn **thickness** related to **dipping time**

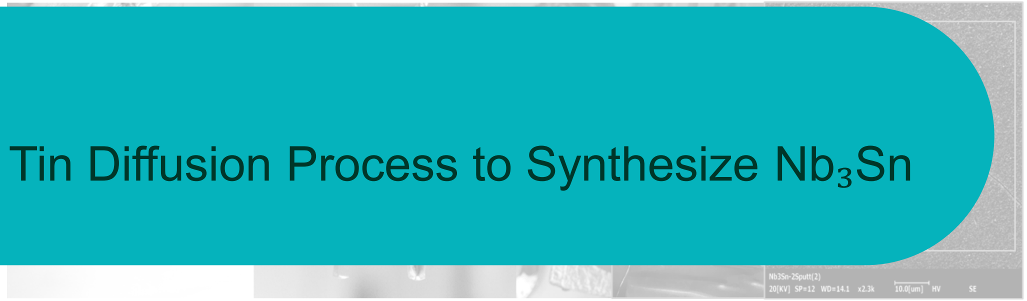
Possible **tin content modulation**

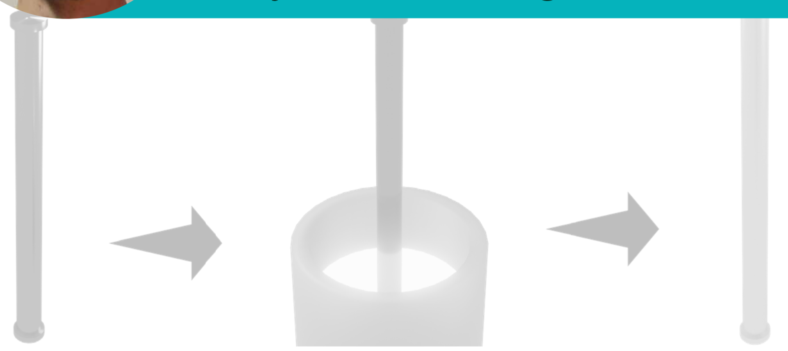
SC coatings

Nb_3Sn coatings

Nb_3Sn cylindrical target are not commercially available

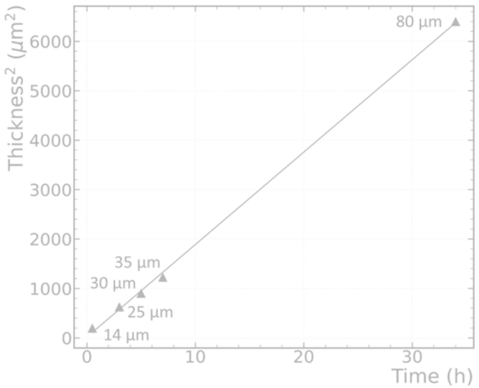
Davide Ford
Poster today: WEPWB118
Study and Improvements of Liquid Tin Diffusion Process to Synthesize Nb_3Sn Cylindrical Targets





Raw Nb target → *Dipping in liquid Sn* → *Nb_3Sn cylindrical target*

Proof of concept



Nb_3Sn **thickness** related to **dipping time**

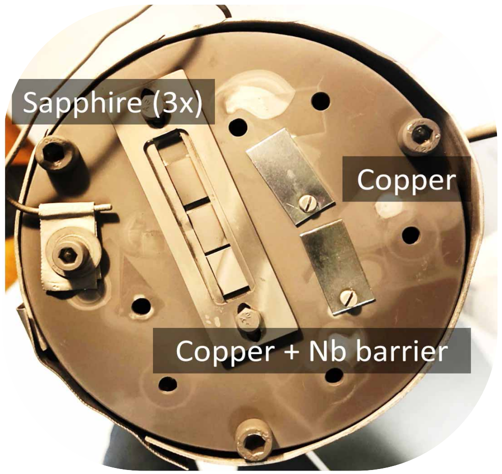
Possible **tin content modulation**

SC coatings

Nb_3Sn coatings

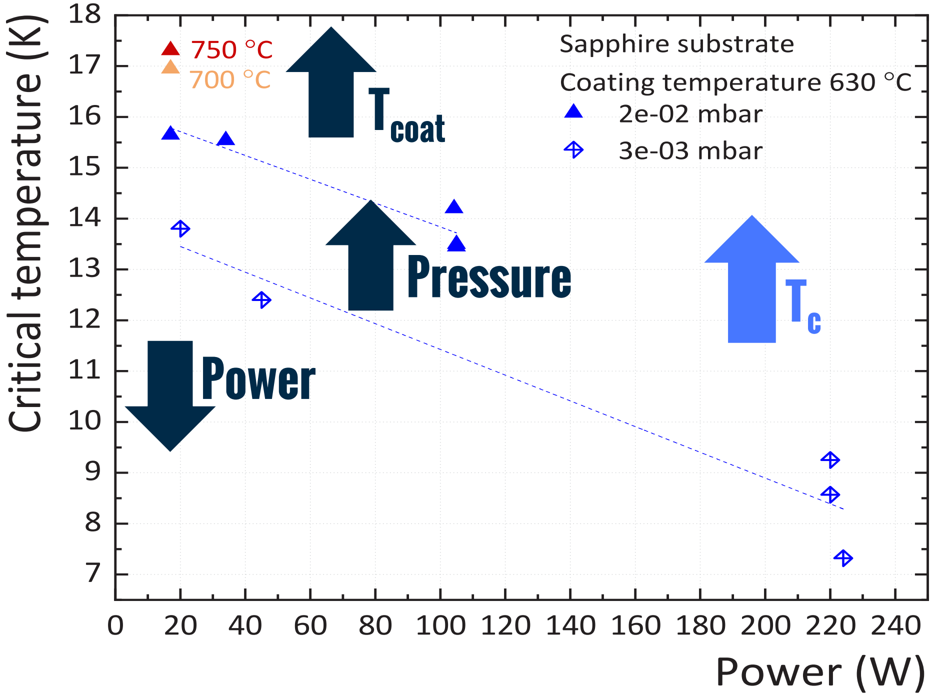
Sputtering parameter optimization

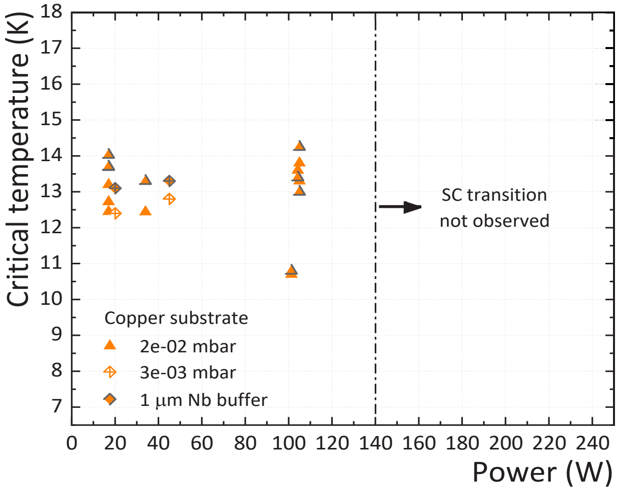
Nb_3Sn deposited via DCMS from 4" planar **stoichiometric target** in Ar atmosphere → **Single target configuration easiest to scale onto elliptical geometry**



In the same run different substrates are coated in the same conditions

Tc clear trends visible on Sapphire



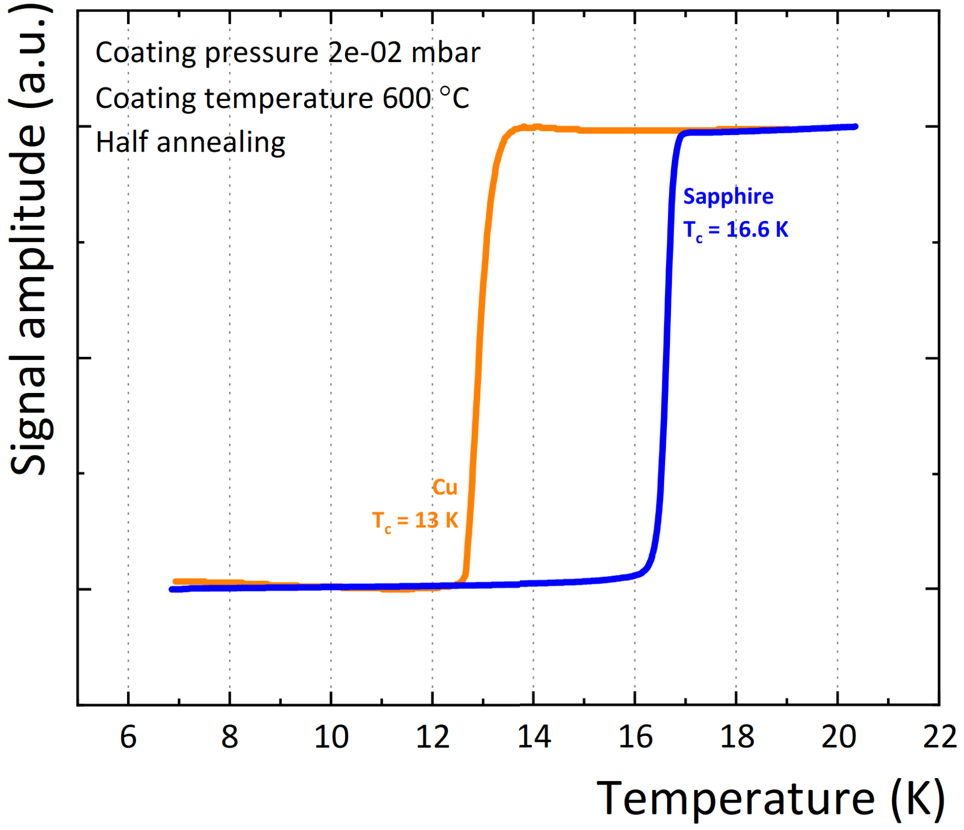


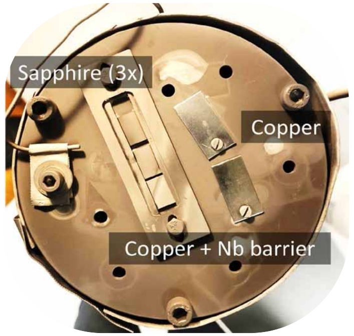
Nb_3Sn Tc stucks at 14 K on Cu

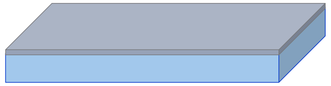
Interface effect

Nb_3Sn coatings

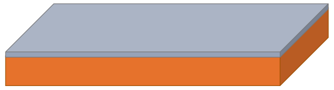
Sputtering parameter optimization







Sapphire + 1 μ Nb_3Sn



Cu + 1 μ Nb_3Sn

SC coatings

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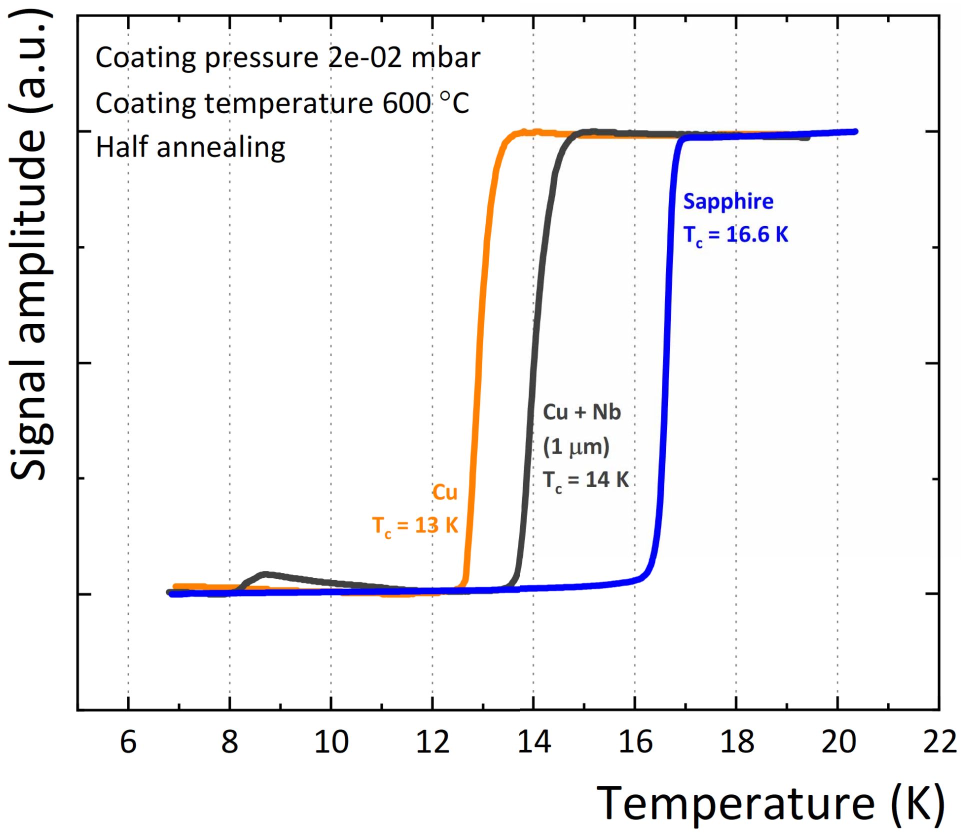
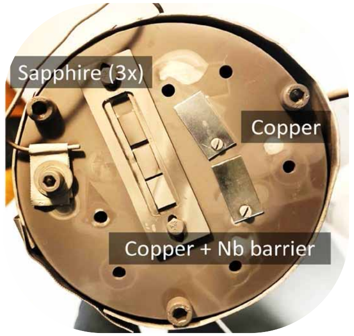
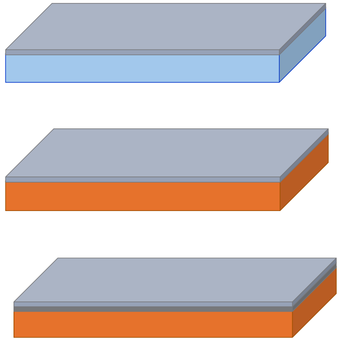
Nb_3Sn coatings

Sputtering parameter optimization







Sapphire + 1 µ Nb_3Sn

Cu + 1 µ Nb_3Sn

Cu **+ 1 µ Nb** + 1 µ Nb_3Sn

SC coatings

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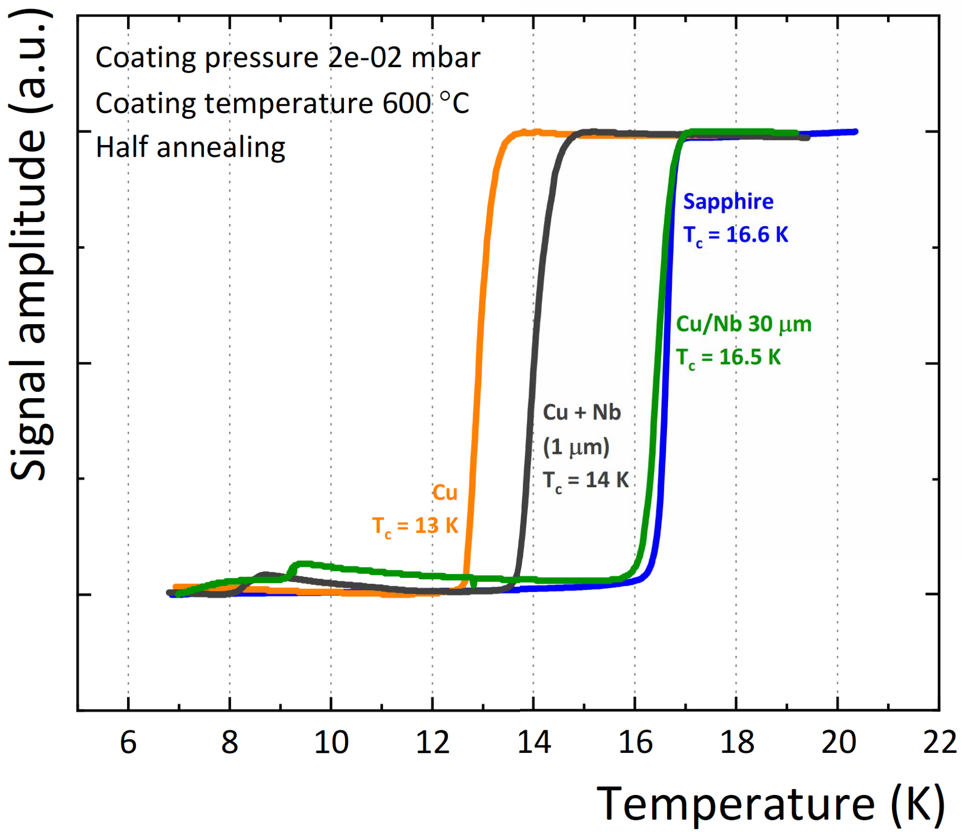
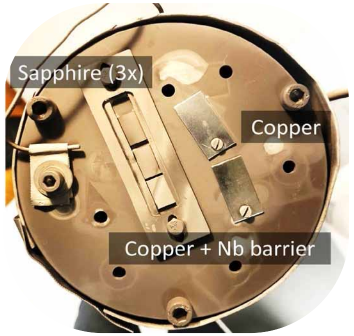
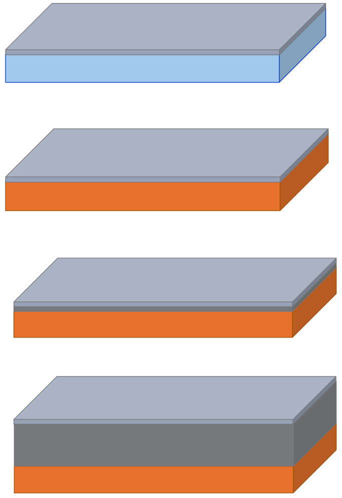
Nb_3Sn coatings

Sputtering parameter optimization







Sapphire + 1 μm Nb_3Sn

Cu + 1 μm Nb_3Sn

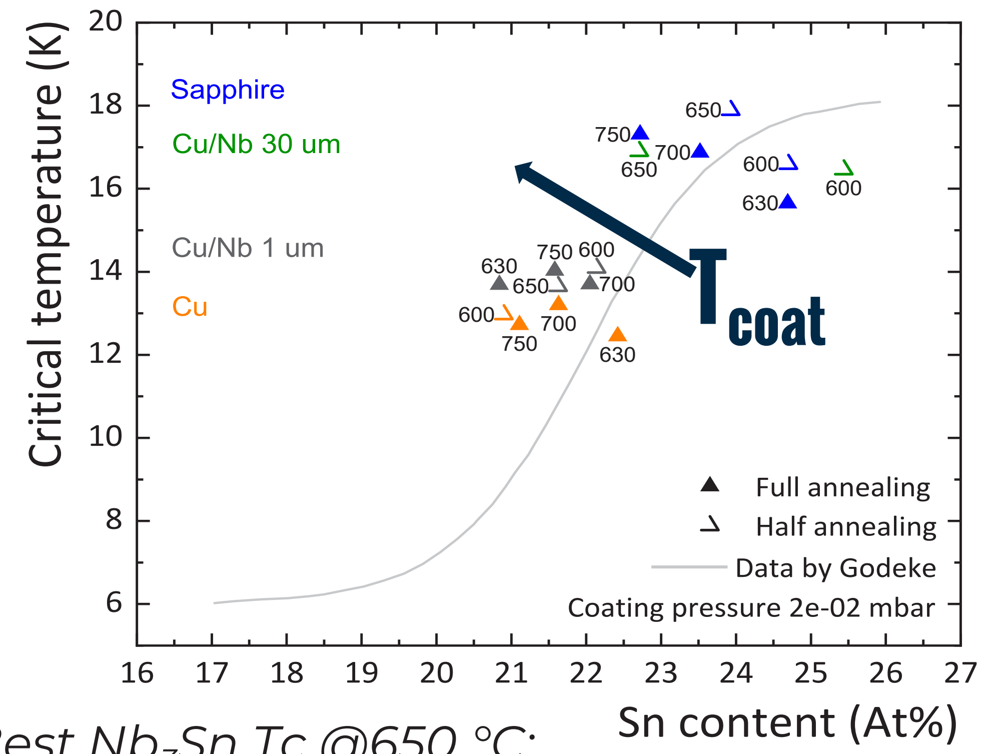
Cu **+ 1 μm Nb** + 1 μm Nb_3Sn

Cu **+ 30 μm Nb** + 1 μm Nb_3Sn

A very thick Nb barrier layer can prevent diffusion at the interface

Nb_3Sn coatings

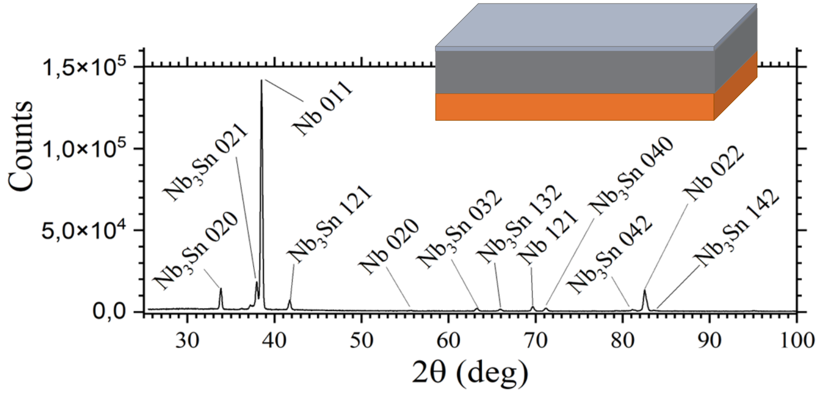
Sputtering parameter optimization



Best Nb_3Sn Tc @650 °C:

- *18 K on Sapphire*
- *17 K on Cu + barrier*

Cu + 30 μ Nb + 1 μ Nb_3Sn



Only Nb_3Sn phase present

Ready to coat a QPR for RF test

Room for further improvements:

- Barrier layer thickness, other materials, …
- Annealing time, Temperature, SC film thickness, …

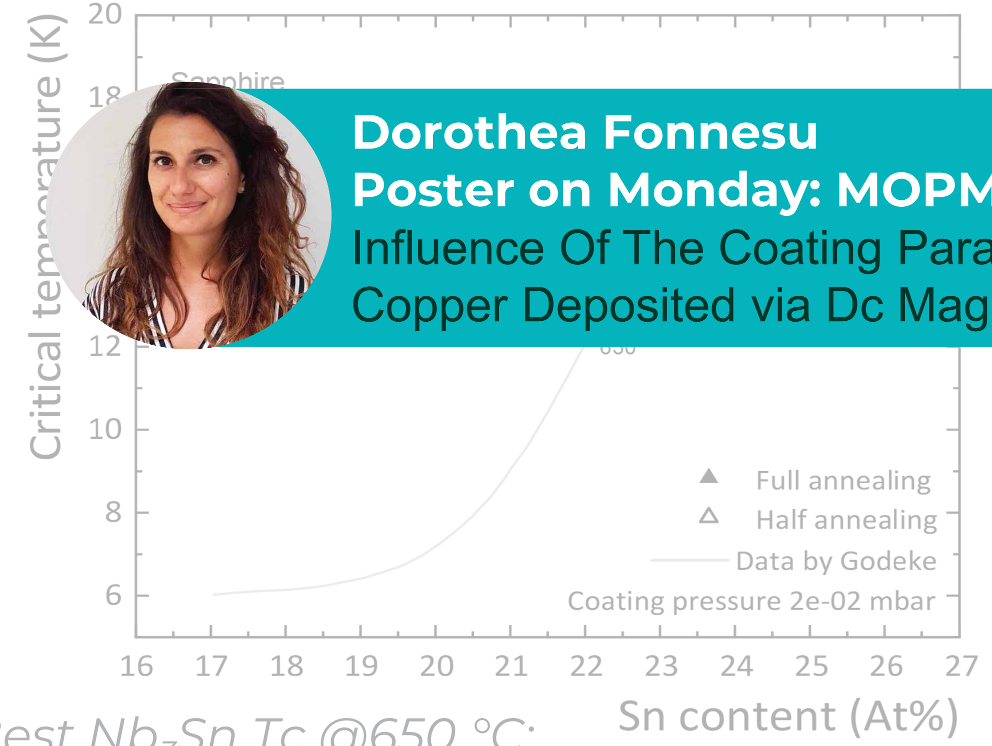
- **Alternative ways to prevent diffusion**

SC coatings

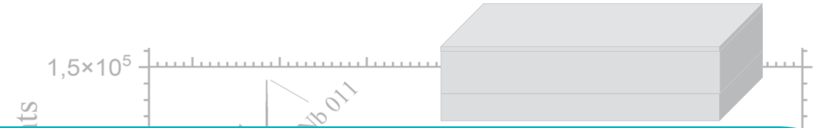
Nb_3Sn coatings

INFN LNL

Sputtering parameter optimization



Cu + 30 μ Nb + 1 μ Nb_3Sn



Dorothea Fonnesu
Poster on Monday: MOPMB013
Influence Of The Coating Parameters on the Tc of Nb_3Sn Thin Films on Copper Deposited via Dc Magnetron Sputtering

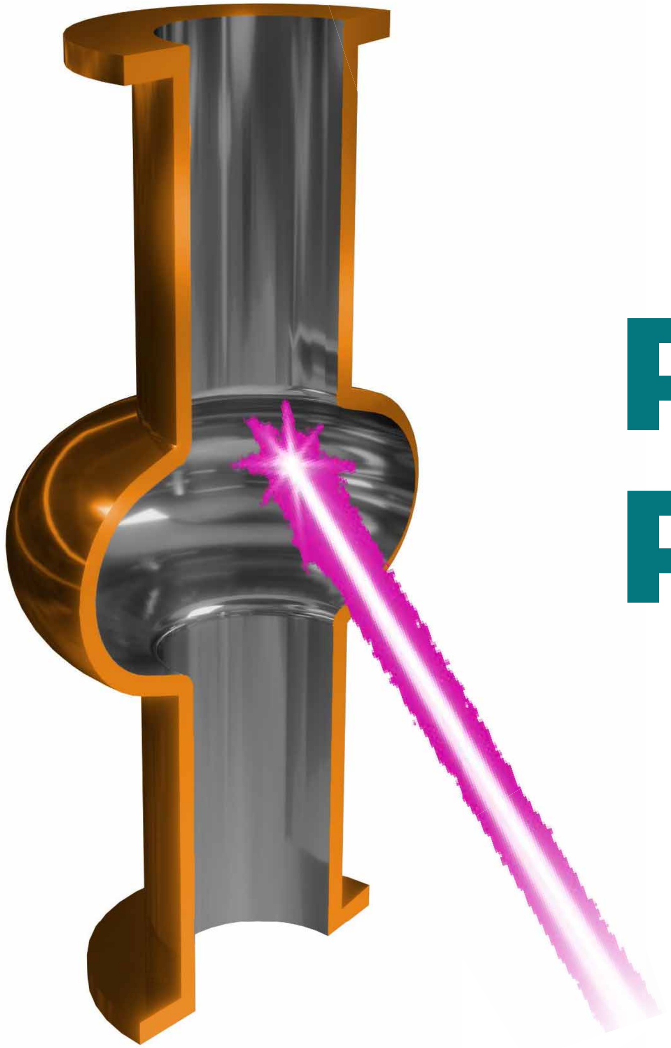
Only Nb_3Sn phase present

Ready to coat a QPR for RF test

Best Nb_3Sn Tc @650 °C:
- *18 K on Sapphire*
- *17 K on Cu + barrier*

Room for further improvements:
- Barrier layer thickness, other materials, …
- Annealing time, Temperature, SC film thickness, …

- **Alternative ways to prevent diffusion**



Post Processing

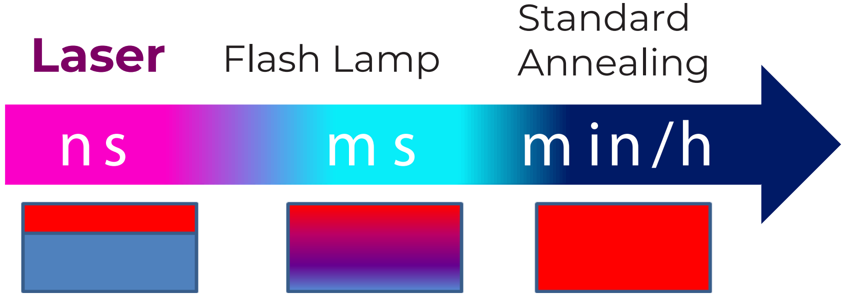
cristian.pira@lnl.infn.it

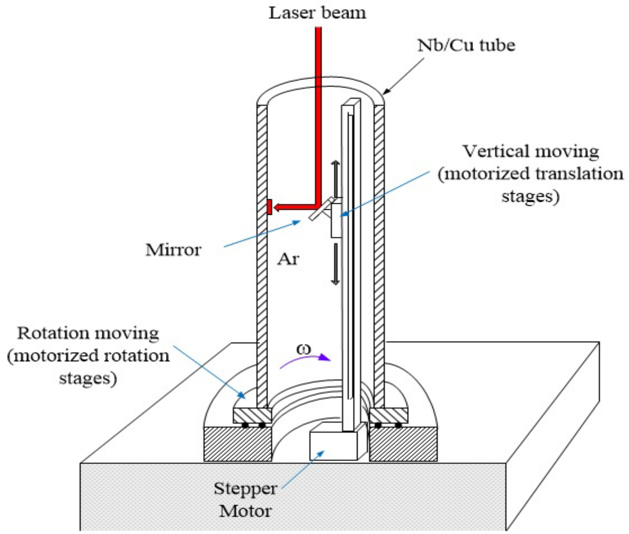
Laser Annealing

RĪGAS TEHNISKĀ UNIVERSITĀTE

Idea:

Anneal the coating without affecting the copper substrate
to avoid diffusion at the interface





Scaled to cylindrical geometry

Crystal size ↑ ~ 20%
Adhesion Nb/Cu ↑ ~36%
Roughness divided by >~2

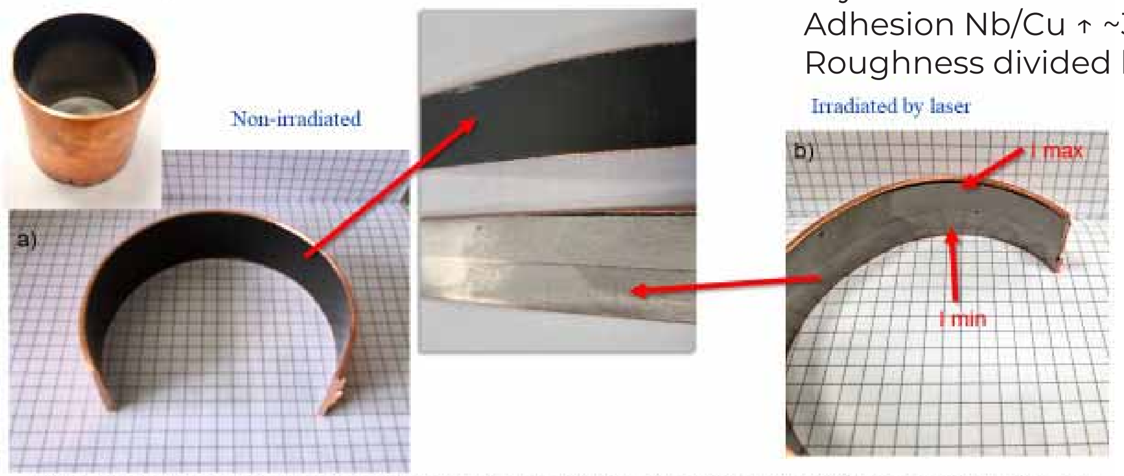
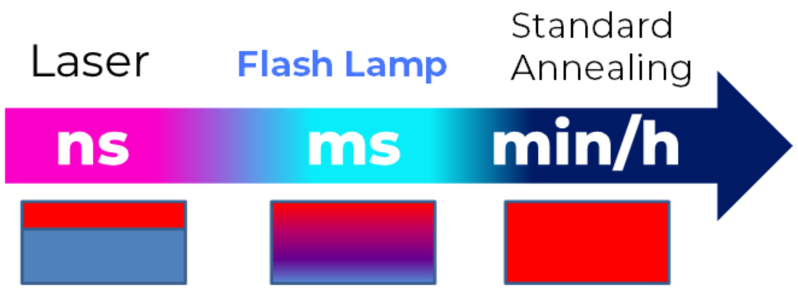


Fig. Samples formed from cylindrical copper tube with Nb film: (a) non-irradiated; (b) irradiated in Ar chamber by ns laser radiation.

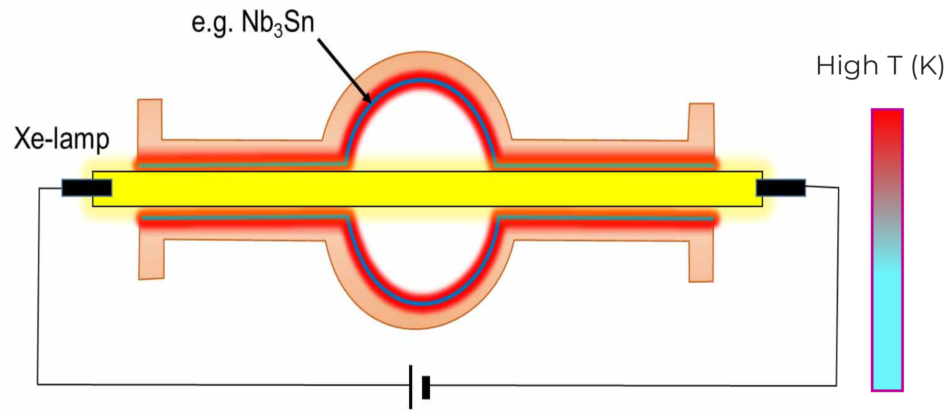
Courtesy of A. Medvids

ms-Flash Lamp Annealing
ms-FLA



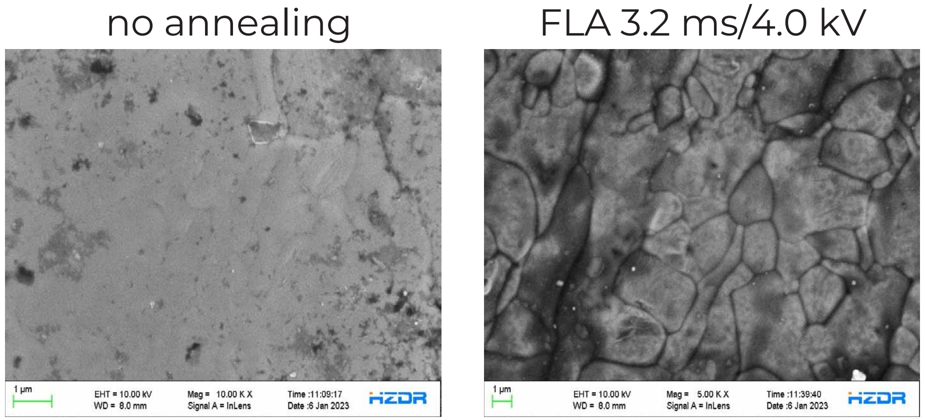
Only the coating layer is heated (even to 1000 ℃) while the rest of the cavity remains cold

Improve coating crystallinity but too short to induce the diffusion of Cu



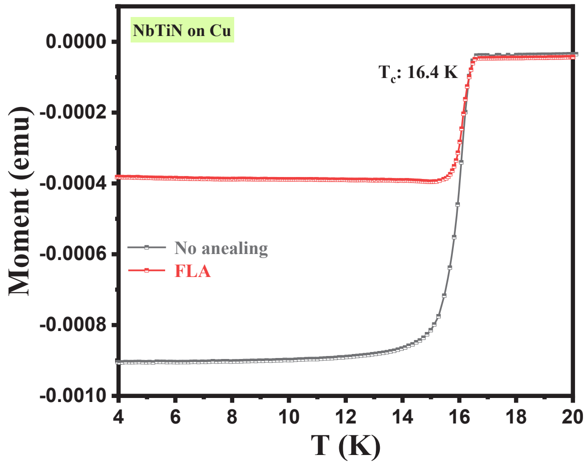
HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF

First tests on NbTiN (Nb_3Sn ongoing)



Crystalline grain size increased

Tc remain stable



Courtesy of S. Prucnal and S. Zhou

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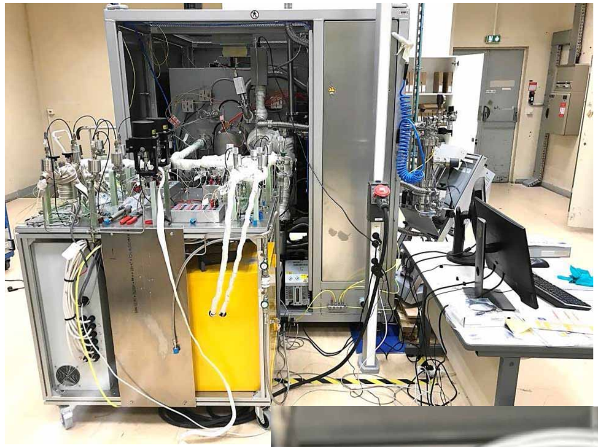
Atomic Layer Deposition (ALD)

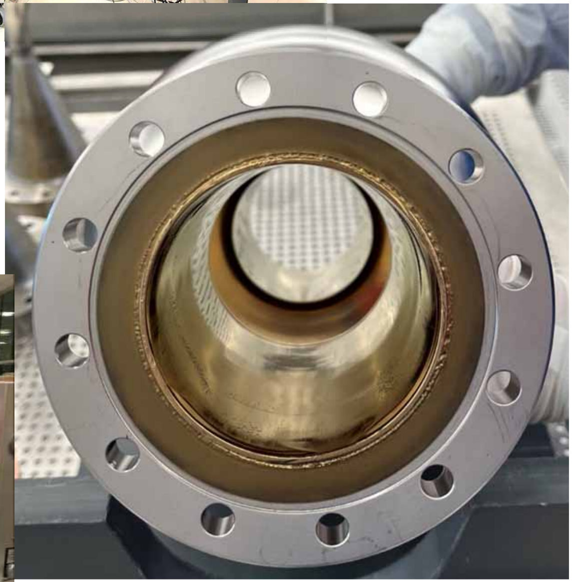
Goal

Deposition of functionalized layers :

- Low secondary yield cap layer (↓ multipacting)
- SIS multilayers
- Dielectric surface engineering and doping



New ALD system @CEA
Already coated several 1,3 Ghz cavities
Compatible w. 700 MHz cavities

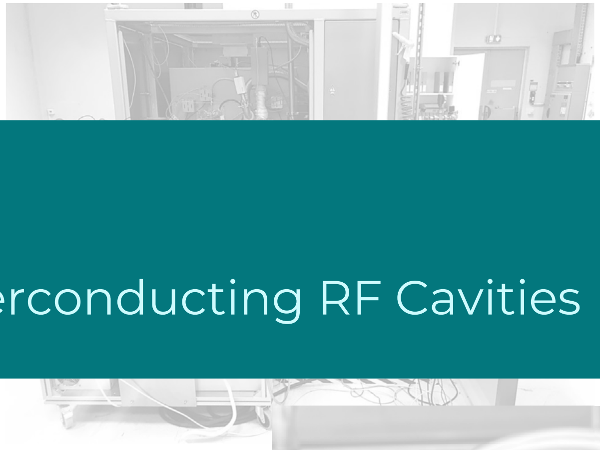


AlN-NbTiN coated cavity

Courtesy of Y. Kalboussi, C. Antoine and T. Proslier

Post Processing

Atomic Layer Deposition (ALD)



Yasmine Kalboussi
Talk today at 10:00: WEIBA01
Surface Engineering by ALD for Superconducting RF Cavities

- Low secondary yield cap layer (↓ multipacting)
- SIS multilayers
- Dielectric surface engineering and doping

New ALD system @CEA
Already coated several 1,3 Ghz cavities
Compatible w. 700 MHz cavities

AlN-NbTiN coated cavity

Courtesy of Y. Kalboussi, C. Antoine and T. Proslier

Post Processing

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SC Properties Evaluation

cristian.pira@lnl.infn.it

DC/AC Superconducting Properties Evaluation

INSTITUTE OF ELECTRICAL ENGINEERING SAS

DC magnetisation measurements

Vibrating Sample Magnetometer

Small planar samples (~ 2x2 mm – *cutting*)

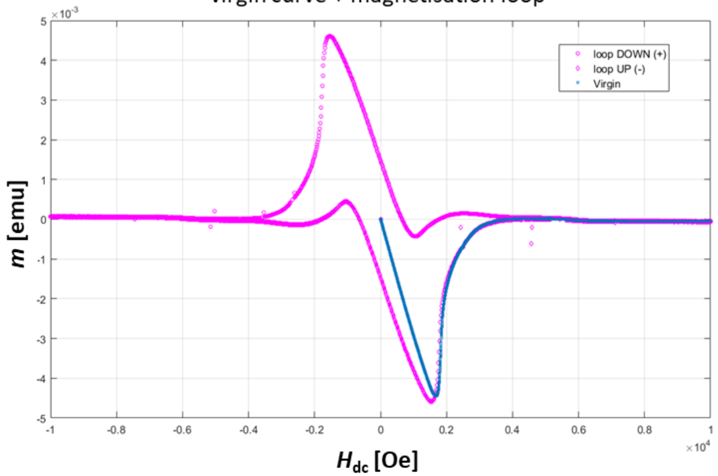
perpendicular *H*

VSM sample holders

parallel *H*

small sample

virgin curve + magnetisation loop

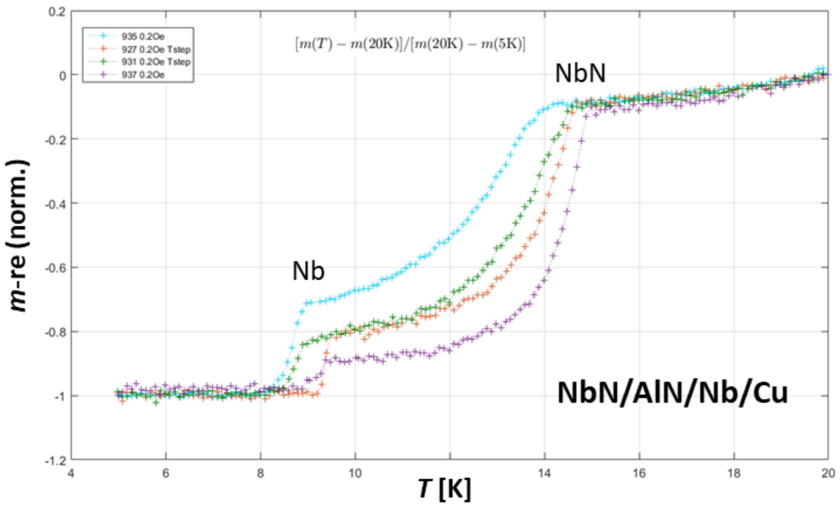


cutting phase (disk saw)

AC magnetisation measurements

Susceptibility – temperature scans

Determining Tc's of different films in Multilayer and SIS samples



AC susceptibility sample holders

Courtesy of E. Seiler

Post Processing

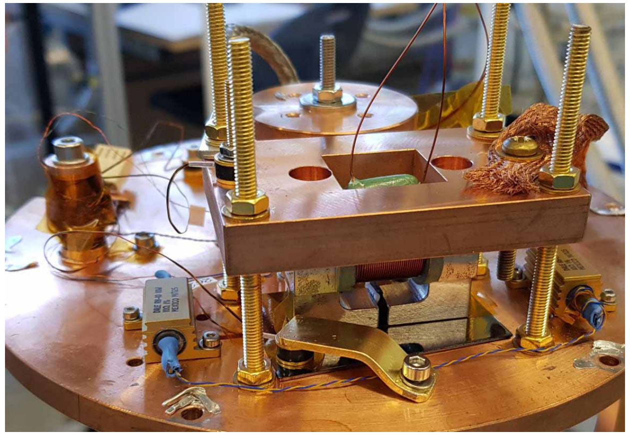
Magnetic Field Penetration

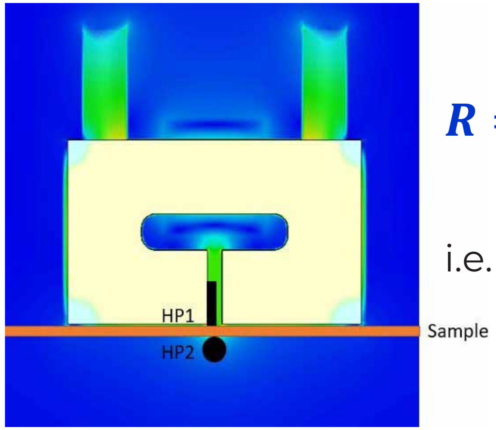
Science and Technology Facilities Council

Thin Film samples can be compared in conditions similar to ones in the cavity

- *DC magnetic field parallel to the surface*
- *Magnetic field applied from one side of the sample (similar to an SRF cavity)*

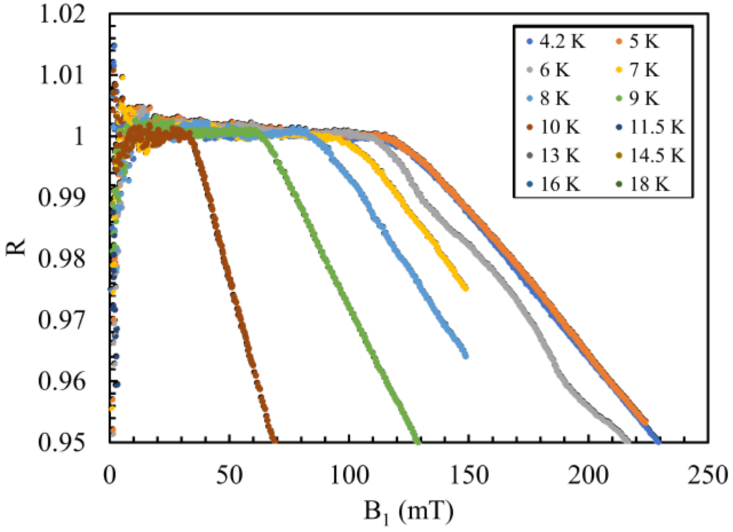
Applied and penetrated field measured by Hall probe sensors





$$R = 1 - \frac{B_2}{B_1}$$

i.e. SC is in Meissner state when $R = 1$



Courtesy of L. Smith

SC properties evaluation

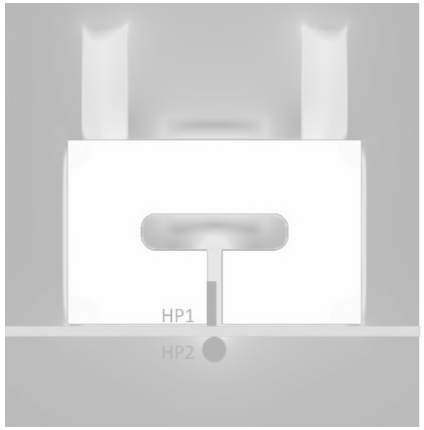
Magnetic Field Penetration

Science and Technology Facilities Council

Thin Film samples can be compared in conditions si

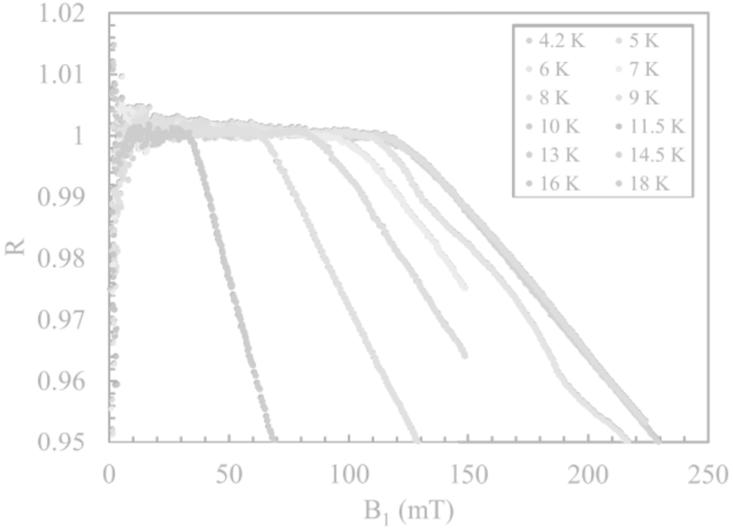
Liam G. P. Smith
Poster on Monday: MOPMB012
Investigation, Using Nb Foils to Characterise the Optimal Dimensions of Samples Measured by the Magnetic Field Penetration Facility

Applied and penetrated field measured by Hall probe sensors



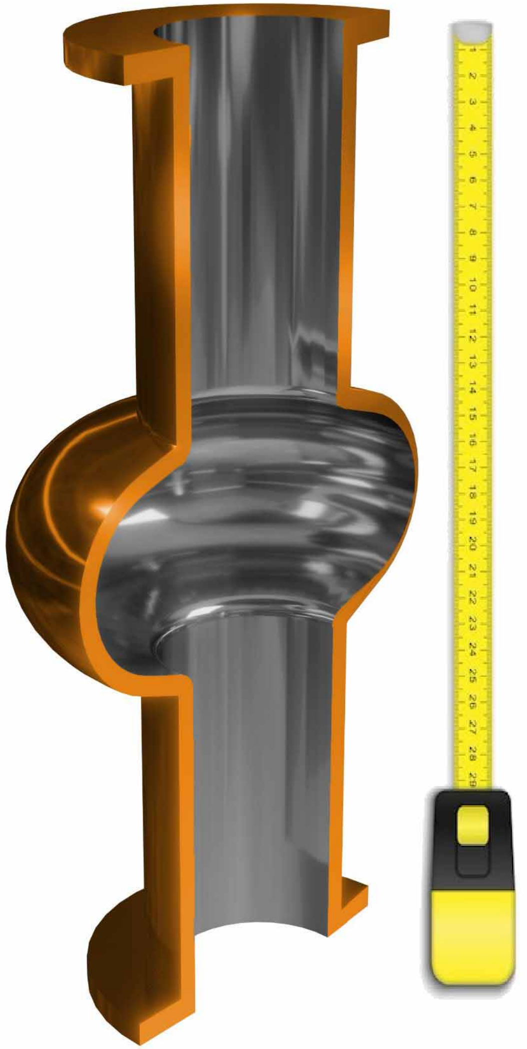
$$R = 1 - \frac{B_2}{B_1}$$

i.e. SC is in Meissner state when $R = 1$



Courtesy of L. Smith

SC properties evaluation



RF Measurements

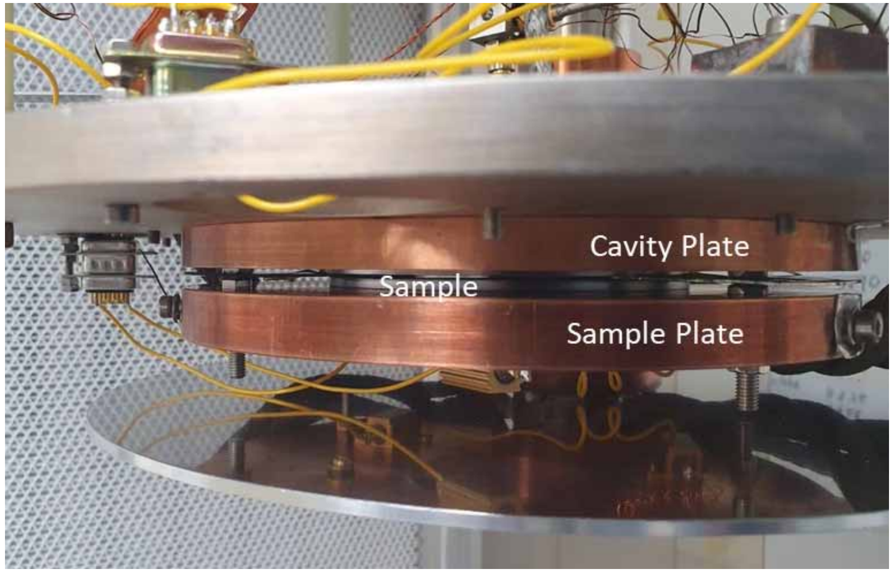
cristian.pira@lnl.infn.it

Sample RF test with 7.8 GHz Choke cavity

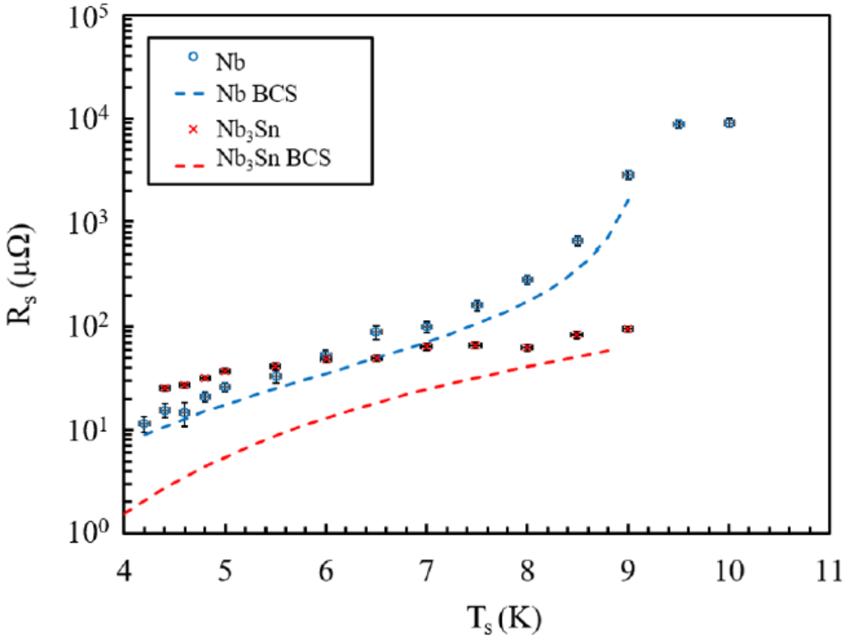
Operation with a closed-cycle refrigerator: T_{cavity} = 4.0 K and T_{sample} = 4.0 K

Low power (≤1.0 W) measurements with an emphasis on fast turn-around time (~2 days sample)

Flat Sample – a disk diam. 90 - 130 mm



An example of $R_s(T_s)$ measurements for Nb and Nb_3Sn TF planar samples with the choked cavity



Courtesy of D. Seal

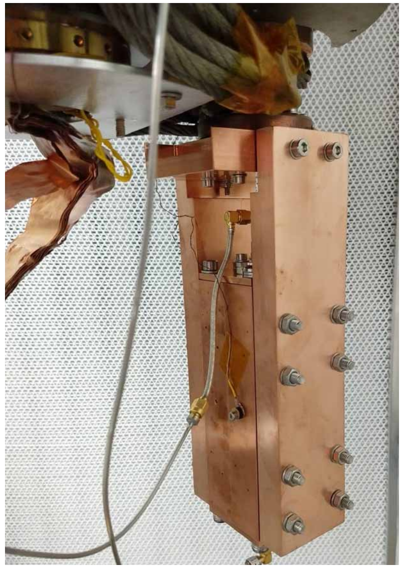
cristian.pira@lnl.infn.it

6 GHz split cavity

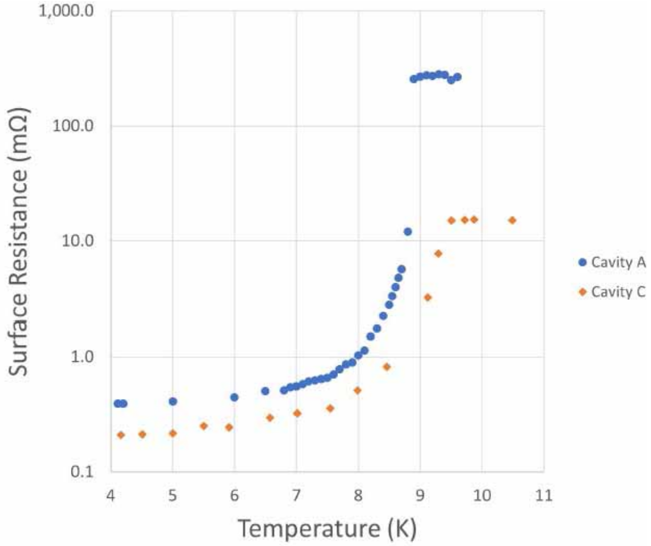
Science and Technology Facilities Council

- An idea suggested by G. Burt (CI)
- The cavity cut is along the electric field lines, i.e. electric current is not crossing the cut
- Easy to coat with either conventional planar magnetron or in tubular geometry used for RF cavities
- Easy to inspect
- Two 6 GHz cavities were Nb coated and tested at 4.2 K ≤ T ≤ 11 K

Nb thin films coated split cavity



Split cavity mounted on the cold head for cryogenic measurements



Surface resistance R_s as a function of temperature for Nb thin films coated split cavity

Courtesy of N. Leicester, T. Sian, and J. Conlon

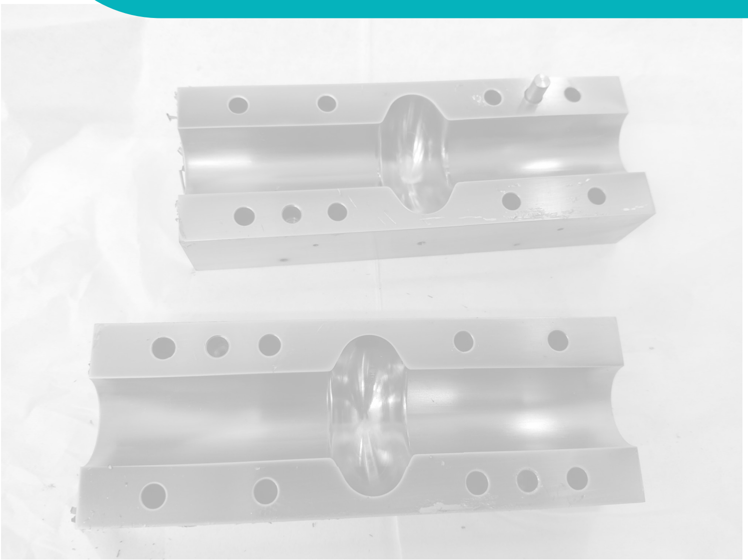
RF measurements

6 GHz split cavity

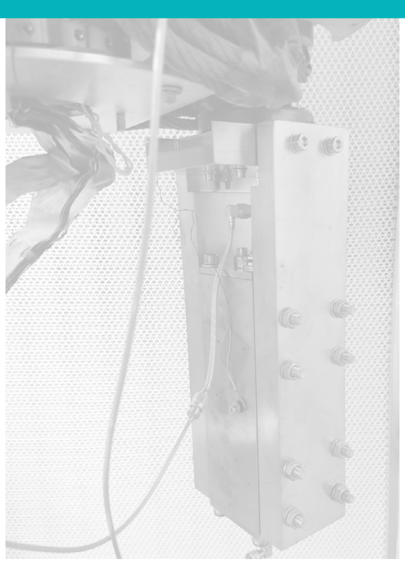
Science and Technology Facilities Council

- An idea suggested by G. Burt (CI)
- The cavity cut is along the electric field lines, i.e. electric current is not crossing the cut
- Ea

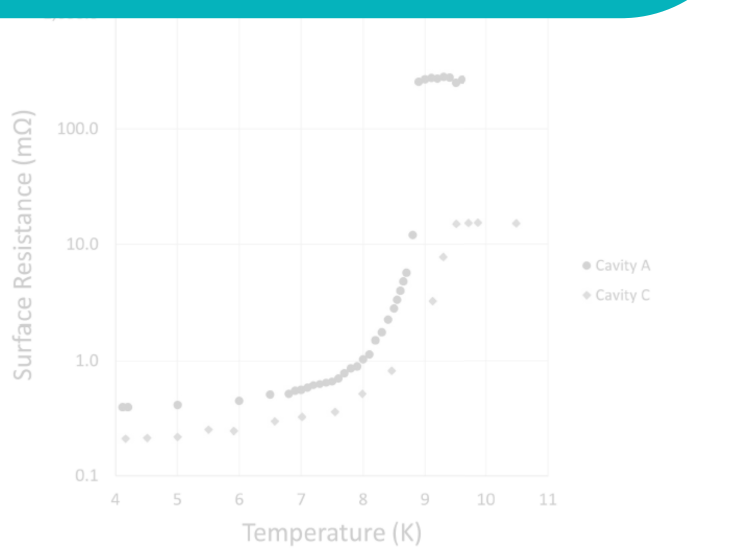
Nathan Leicester
Poster on Monday: MOPMB001
Development and Testing of Split 6 GHz cavities with Niobium Coatings



Nb thin films coated split cavity



Split cavity mounted on the cold head for cryogenic measurements

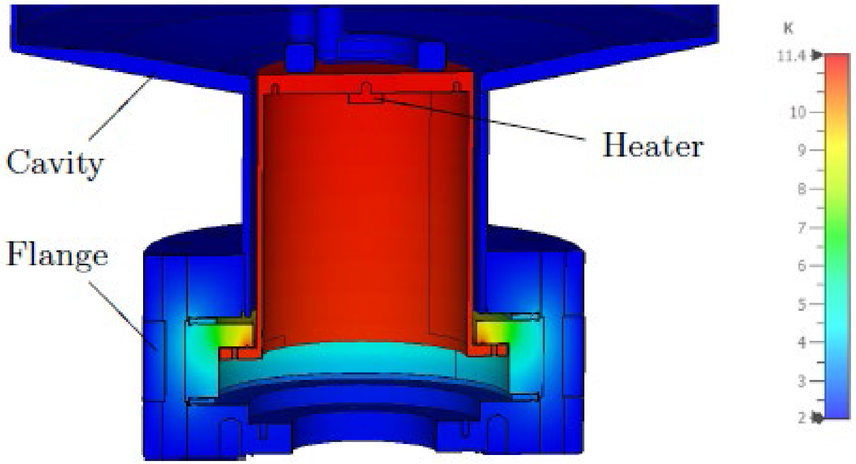


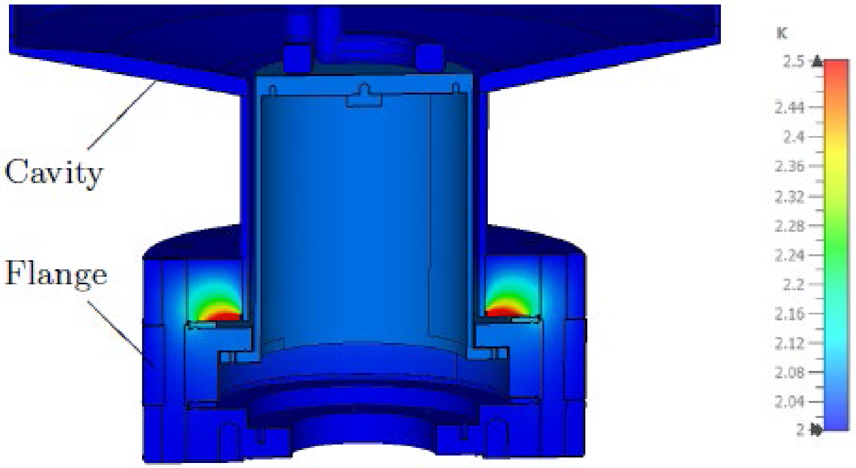
Surface resistance R_s as a function of temperature for Nb thin films coated split cavity

Courtesy of N. Leicester, T. Sian, and J. Conlon

RF measurements

QuadruPole Resonator





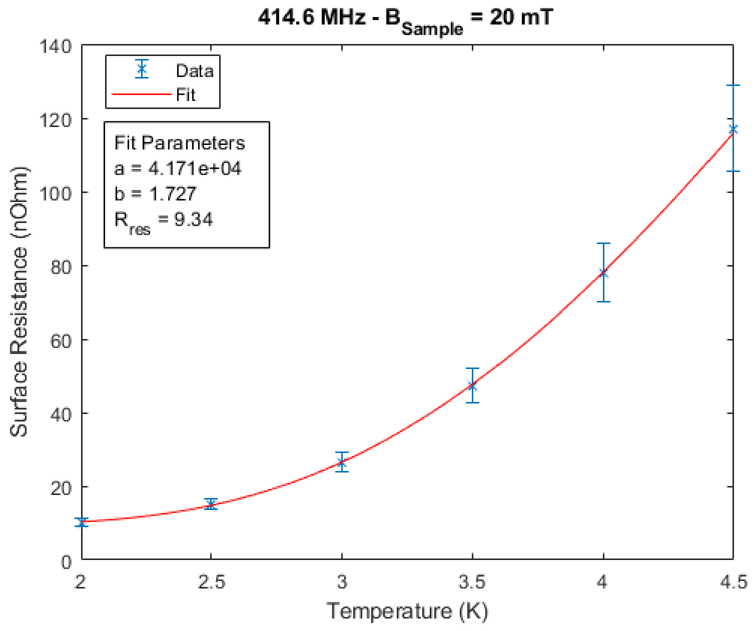
RF losses on nc adapter flange lead to **unwanted heating of the sample** compromising the measured R_res value – resulting in a **systematic error of 12 nOhm**

A SC coating of the adapter flange eliminates the RF heating of the sample

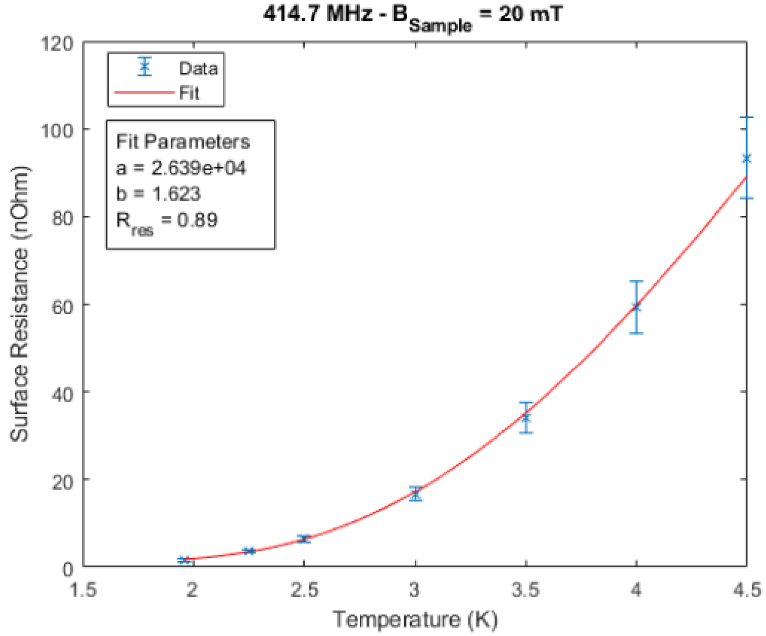
*Keckert, S., et al. (2021). "Mitigation of parasitic losses in the quadrupole resonator enabling direct measurements of low residual resistances of SRF samples." AIP Advances **11(125326).***

Courtesy of O. Kugert and S. Keckert

QuadruPole Resonator



State-of-the-art chemically polished Nb sample



Same sample after optimized metallographic polishing
R_{res} < 1nOhm directly measured with QPR

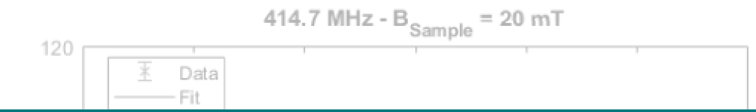
- **Metallographic polishing produce high quality baseline samples for thin-film investigations**
- **QPR is now able to measure R_res with same accuracy and precision as an SRF cavity**

Courtesy of O. Kugert and S. Keckert

RF measurements

QuadruPole Resonator

HZB Helmholtz Zentrum Berlin



Oleksandr Hryhorenko
Talk today at 12:20: WEIXA06
Recent advances on metallographic polishing for SRF application

Oleksandr Hryhorenko
Poster today: WEPWB050
Exploring innovative pathway of cavity fabrication for SRF application

State-of-the-art chemically polished Nb sample

Same sample after optimized metallographic polishing
R_{res} < 1nOhm directly measured with QPR

Metallographic polishing produce high quality baseline samples for thin-film investigations

QPR is now able to measure R_res with same accuracy and precision as an SRF cavity

Courtesy of O. Kugert and S. Keckert

RF measurements

1.3 GHz cavity testing

- Facilities ready at
  CEA, HZB, INFN LASA
  INFN LNL for 6 GHz cavities

- Assembling in process at STFC

Cold-test inserts @ INFN LASA

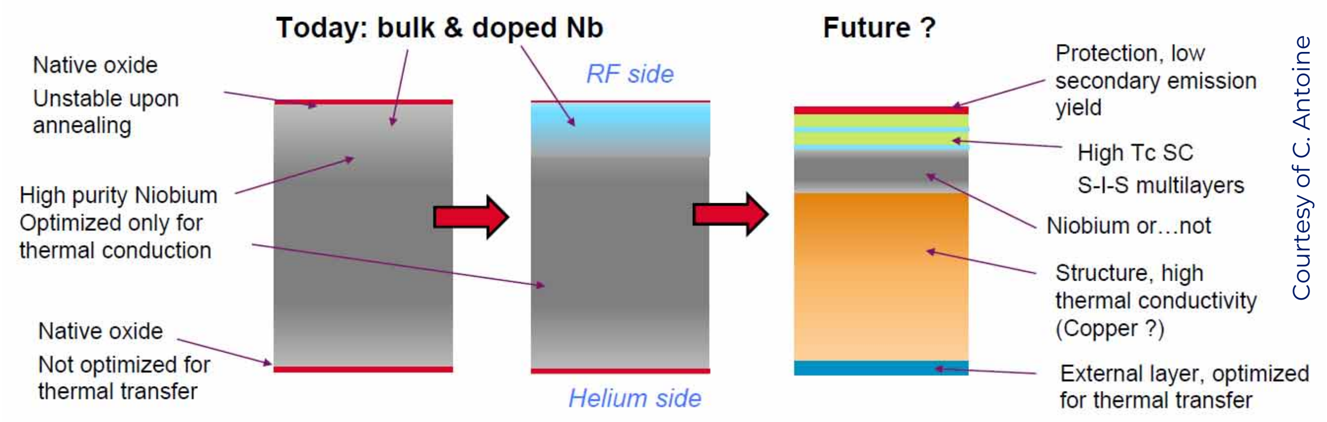
New 1.3 GHz insert @ STFC

Courtesy of O. Malyshev

Conclusion

- **Great advancements** on planar samples
- First **RF results** on QPR expected by the **end of the year**
- The **project is on the track** to produce the first 1.3 GHz prototype in 2025

Thin film SRF presents **multiple challenges** that can be **won collaborating** among the different Laboratories



Our Vision of the future for SRF

INFN
LNL

cristian.**pira**@lnl.infn.it

Thank You for Your Attention!