Overview Of Recent Progress On Thin Film Technologies

A.-M. Valente-Feliciano







Material provided by:

U. Pudasaini (JLab), E. Lechner (Jlab), M. Ge (Jlab), D.R. Beverstock (Jlab/W&M), C. Antoine, T. Proslier, Y. Kalboussi (CEA Saclay), G. Rosaz (CERN), G. Eremeev (FNAL), M. Wenskat (Uni. Hamburg), C. Pira (INFN-LNL), T. Tan (IMP), L. Shpani (Cornell Uni), T. Saeki (KEK), T. Tajima (LANL), S. Sharifuzzaman(ODU), R. Valizadeh (UKRI/STFC), J. Hao (PKU), S. Balachandran (Jlab), S. McNeal (Ultramet), X. Xi (Temple Uni.)







Recent progress in SRF thin film developments since SRF 2023

□ Nb Thin Film Technology

- Beyond Nb Alternate Materials
 - Nb₃Sn
 - NbTiN
 - MgB₂
- Beyond Nb Multilayers
- □ SRF Thin Film Characterization
- Substrates

Disclaimer:

Non-exhaustive summary

Many more ongoing developments, awaiting results, validation...



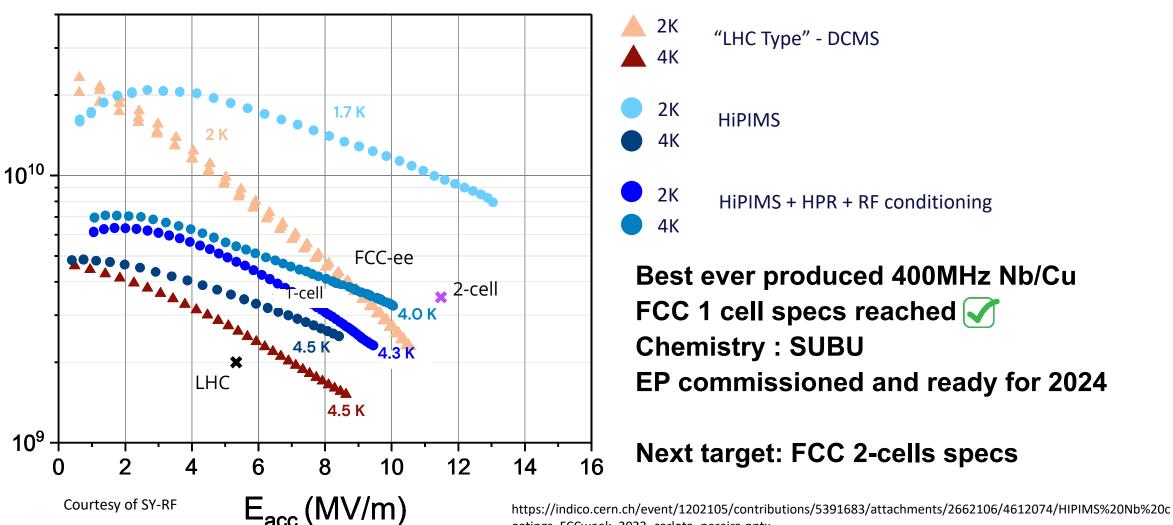


Nb Thin Film Technology



G. Rosaz et al.







oatings FCCweek 2023 carlota pereira.pptx



Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023

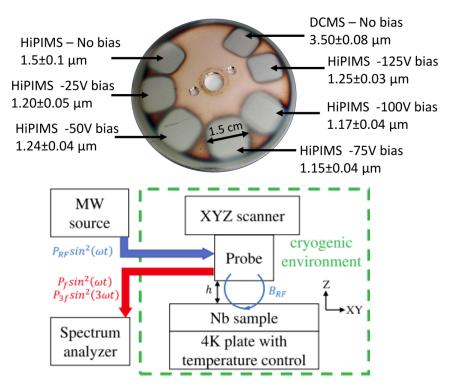


o

Nb Thin Film Technology



1.3 GHz Nb/Cu



Samples characterization by local microwave spectroscopy

https://arxiv.org/abs/2305.07746

bulk Nb region

Sample	$ ho^{defect}$	$h_{penetration}^{defect}$
HiPIMS, 125V bias	low	deep
HiPIMS, 100V bias	low	deep
HiPIMS, 75V bias	low	shallow
HiPIMS, 50V bias	high	shallow
HiPIMS, 25V bias	high	shallow
HiPIMS, no bias	N	/A
DCMS, no bias	N	/A

Optimum coating bias evaluated using local probe technique Surface defects signal through P3f sample response Optimum in agreement with VSM/Squid data

G. Rosaz et al.



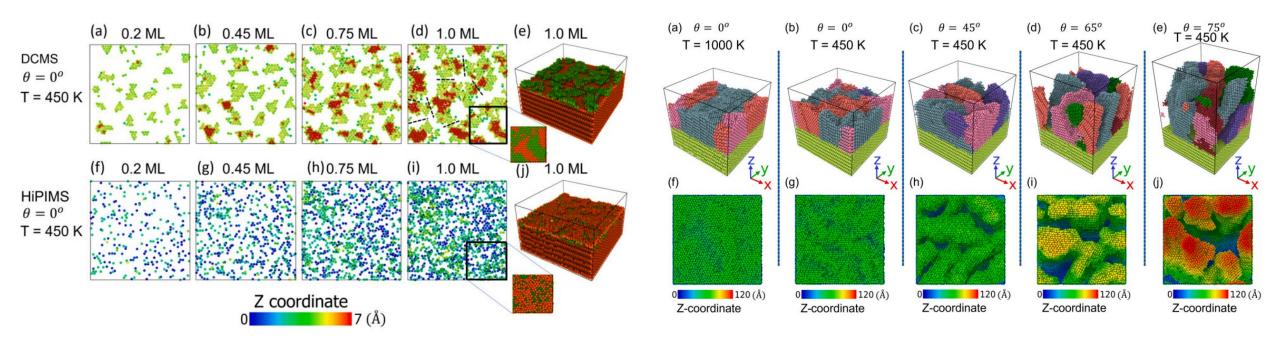


1.3 GHz Nb/Cu



https://doi.org/10.1016/j.surfcoat.2023.130199

Molecular dynamic simulations



Confirmation of Cu presence on top of Nb films – confirmed by XPS analysis Structure of the film studied as function of temperature and incidence angle

Next Step: Study defects formation and annihilation

G. Rosaz et al.



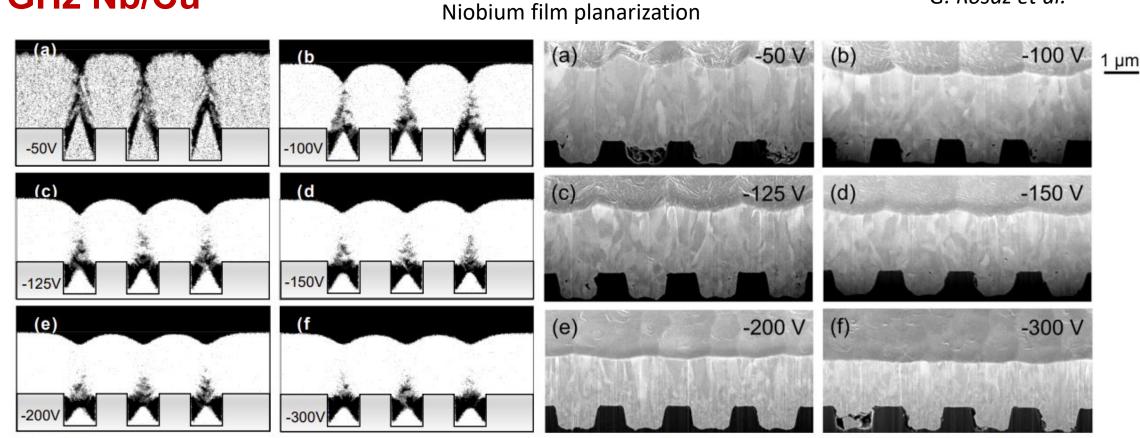


Nb Thin Film Technology



G. Rosaz et al.

1.3 GHz Nb/Cu



Simulations (SRIM, SIMTRA + NASCAM, 70% ionization)

Experimental results, Nb on trenched Si samples Focus Ion Beam (FIB) measurements

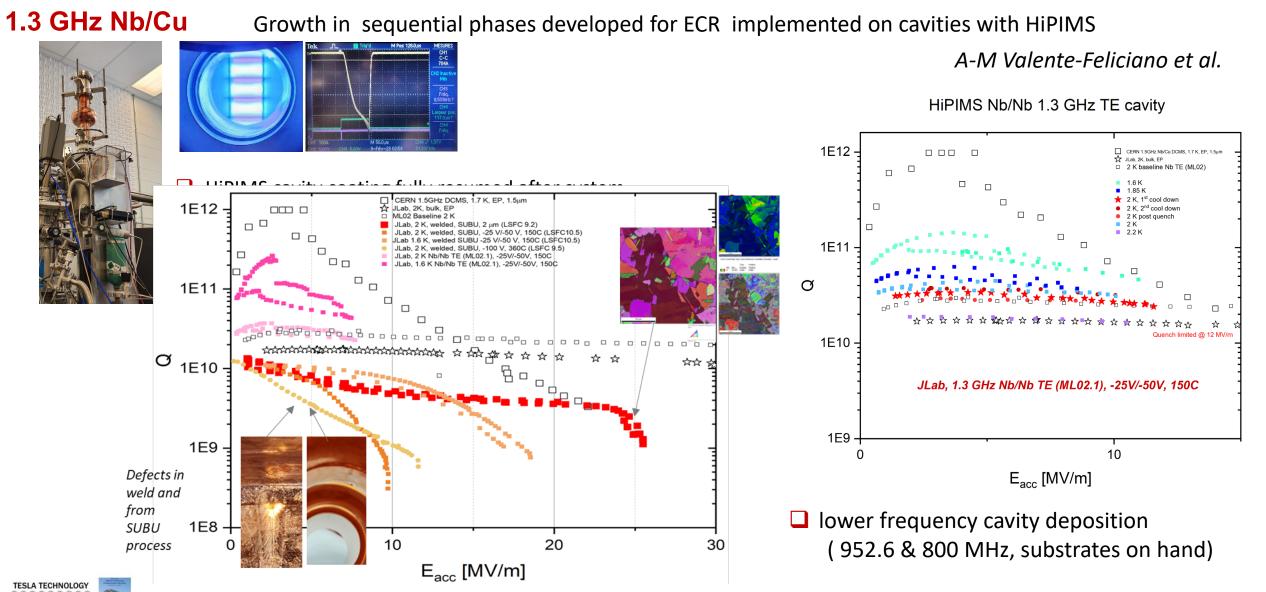
Suppression of substrate-induced defects by ion bombardment To be tested on actual 1.3GHz cavities





Nb Thin Film Technology

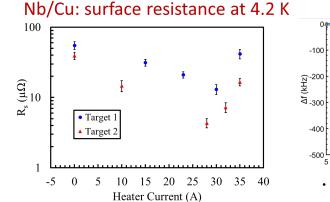


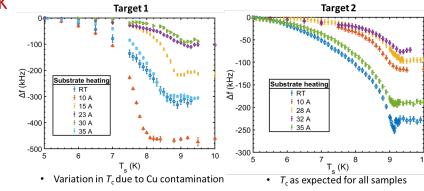


TESLA TECHNOLOGY





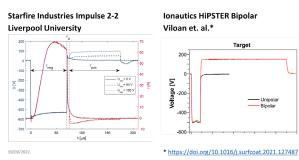




Plasma diagnostic study with Liverpool University



Bipolar HiPIMS discharge



R. Valizadeh et al.

TESLA TECHNOLOGY

COLLABORATION

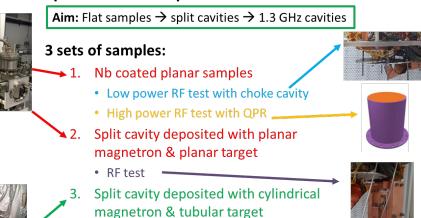
1.3 GHz Cavity deposition system





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.

From planar samples to real cavities



• RF test



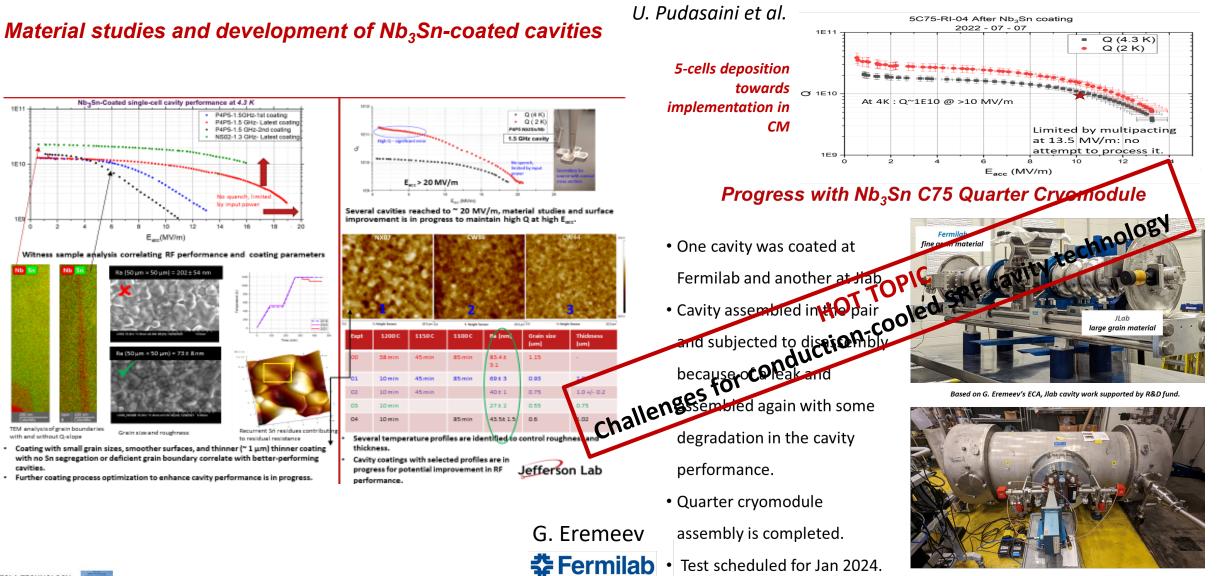




Beyond Nb: Alternate Materials

Nb₃Sn





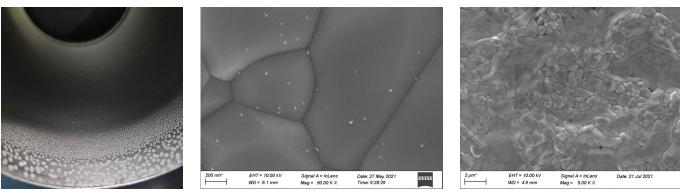


Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023

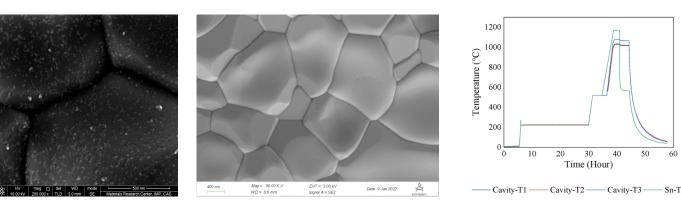
Jefferson Lab

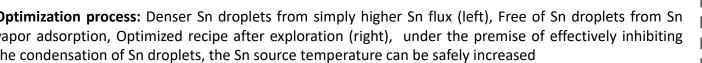
Beyond Nb: Alternate Materials and Multilayer Structures Nb₃Sn

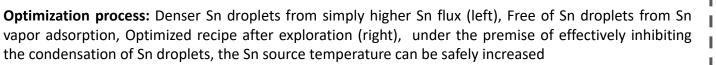
Optimization of Coating Process of Nb₃Sn SRF cavity by vapor diffusion method at IMP

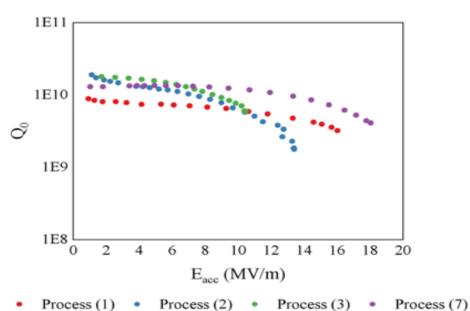


Limiting factors: Millimeter-scale Sn spots (left), Nanometer-scale Sn droplets (middle), Locally extremely thin patchy areas (right), the composition and causes of them were confirmed









Key to high-performance Nb₃Sn film:

- 1. Temperature uniformity of cavity
- 2. Sufficient Sn vapor flux
- 3. Timely removal of residual Sn vapor before cooling

Development and Application of Nb₃Sn Thin Film SRF Cavity at IMP, Jiankui Hao (PKU) – Tues 12/6



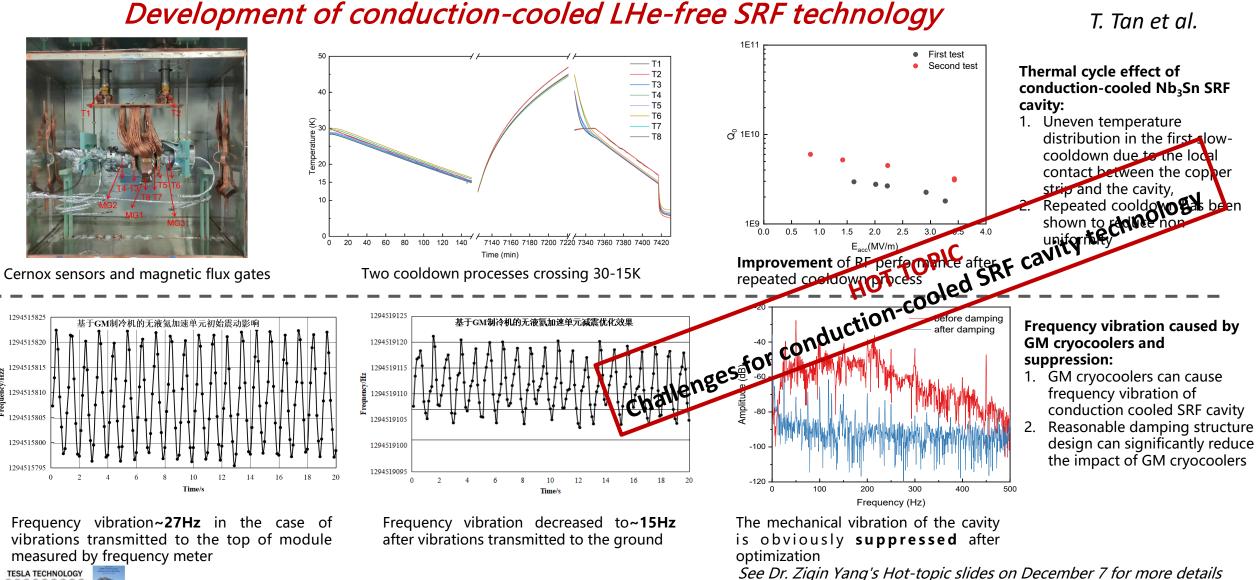
T. Tan et al.

40

Time (Hour)



Beyond Nb: Alternate Materials and Multilayer Structures Nb₃Sn



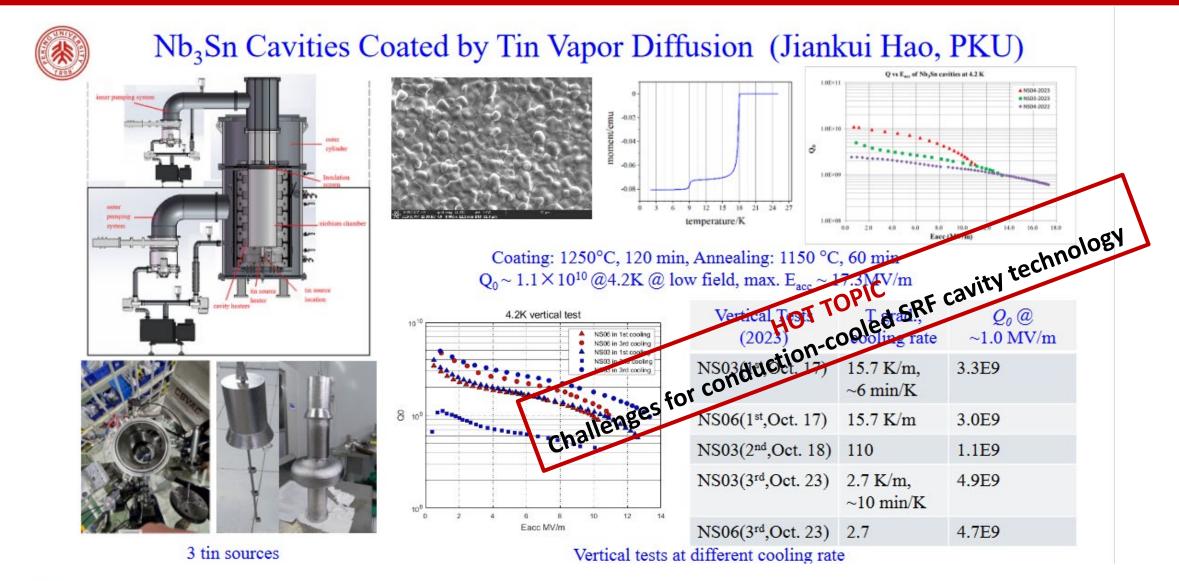




Jefferson Lab

Beyond Nb: Alternate Materials









Electrochemical Synthesis

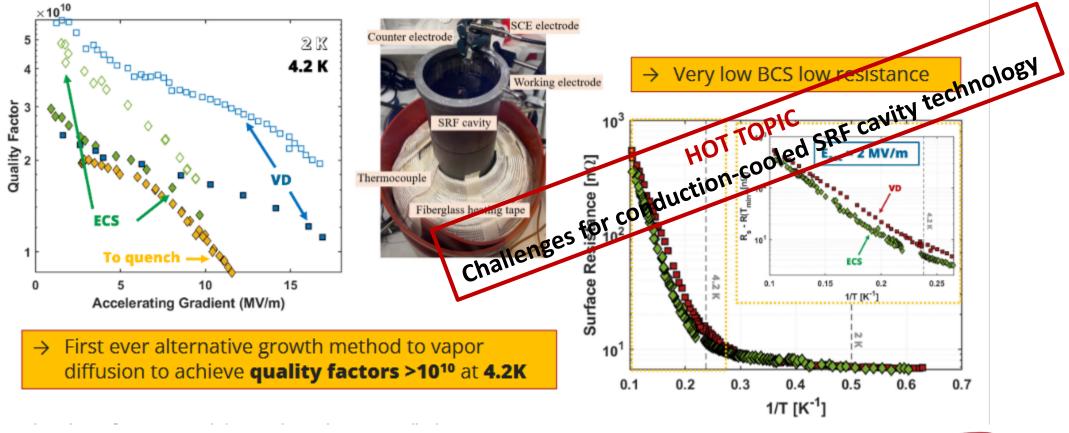
L. Shpani et al.

Jefferson Lab

Achieve a smooth Nb₃Sn film with uniform thickness and stoichiometry

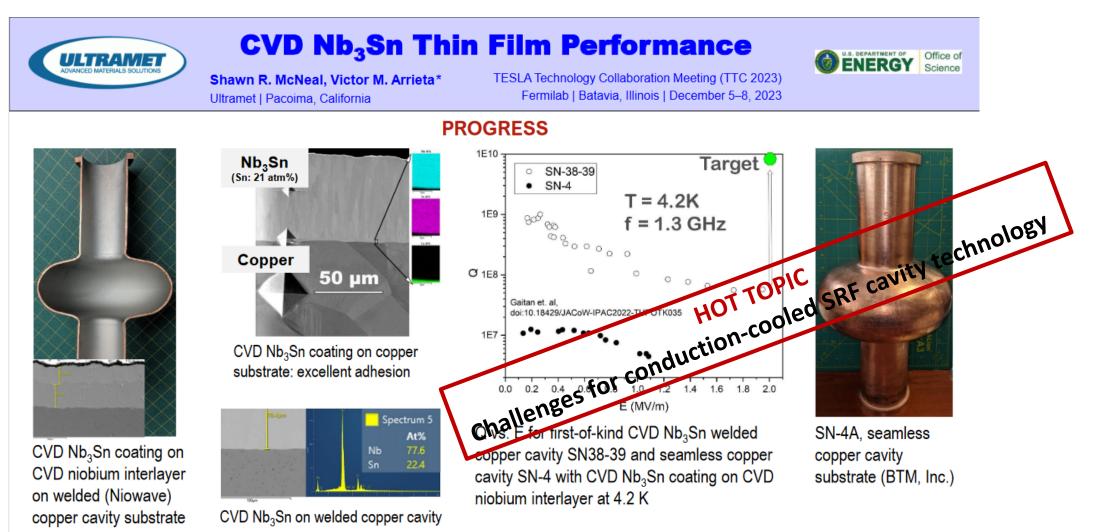
This alternative growth method provides uniform tin nucleation and sufficient Sn supply in critical times

 \Rightarrow smoother Nb₃Sn films with little variation in Sn concentration with depth.





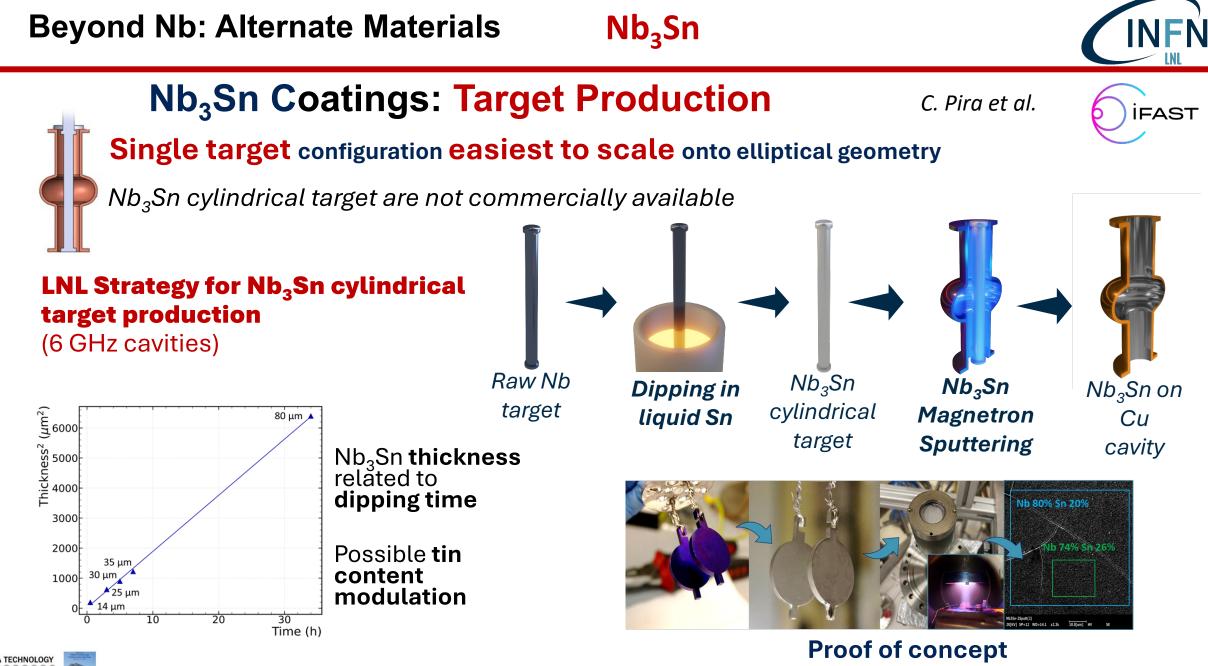
Beyond Nb: Alternate Materials and Multilayer Structures Nb₃Sn



* Shawn.McNeal@ultramet.com Victor.Arietta@ultramet.com



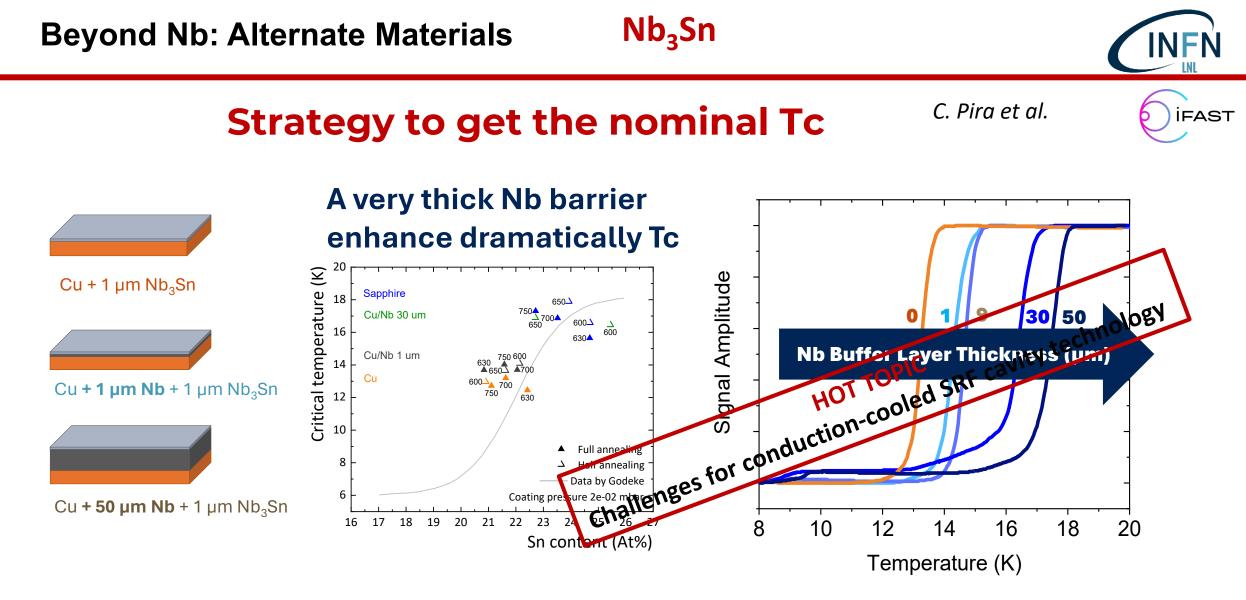






Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023

Jefferson Lab



 $T_c = 17.33 \pm 0.25 \text{ K}$ on Cu + 50 µm Nb Buffer Layer at $T_{dep} = 600 \text{ °C}$



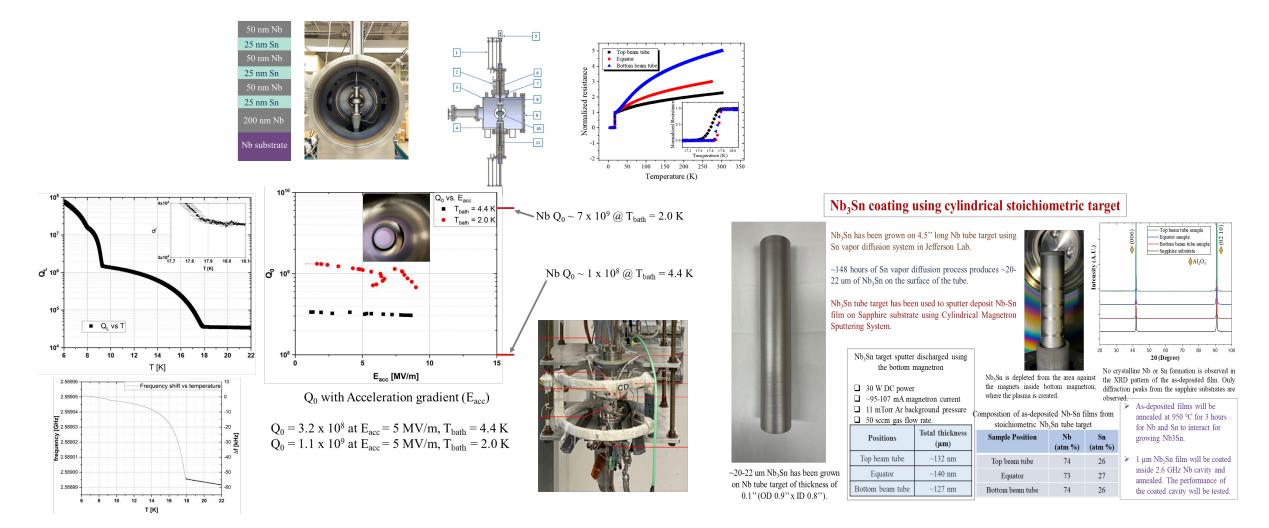


Nb₃Sn coating of a 2.6 GHz Nb SRF cavity using a Cylindrical Magnetron Sputtering System <u>-</u>Sharifuzzaman Shakel, et al.

Nb₃Sn

Frank Batten College of Engineering & Technology

恭 Fermilab





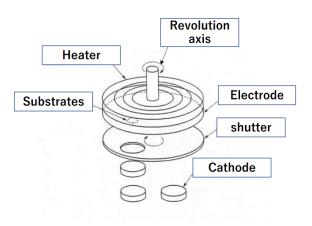
Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023

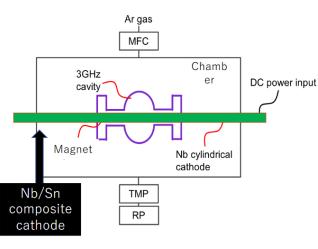


Applied Research Center

Old Dominion Univ

Jefferson Lab



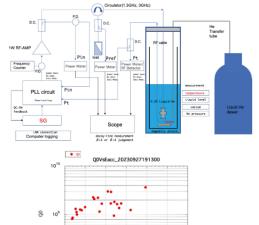


- KEK introduce the sputtering apparatus, SH-450 (ULVAC inc.).
- SH-450 is capable of Nb3Sn coating method almost same as developed by ULVAC-KEK collaboration.
 - In addition, temperature control of substrate is possible.
 - RF sputtering and HIPIMS are also possible only by replacing DC power sources.
- Nb3Sn coating method can be applied to the inside of 3 GHz cavity.
- Nb/Sn composite cathode is the key.
- Development of the special cathode is ongoing.

T. Saeki, R. Katayama et al.

Cavity Test Setup

8 10⁹



15 20 25

Eacc [MV/m]

• This year, we prepared VT setup for 3 GHz cavity at KEK STF for evaluation of the cavity performance with S'IS structure.

- We performed the first VT of a 3 GHz cavity made of a pure bulk Nb on Sep 27.
 - Treatment
 - BCP and 120 °C bake for 48 h
 - No anneal (we missed)
- Problem
 - RF feedback system was unstable if Eacc is greater than 20 MV/m.
- We are developing new RF feedback system designed to be work stably.



Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023



SRF multi-layer thin film R&D at KEK, Ryo Katayama , Wed 12/06

Beyond Nb: Alternate Materials

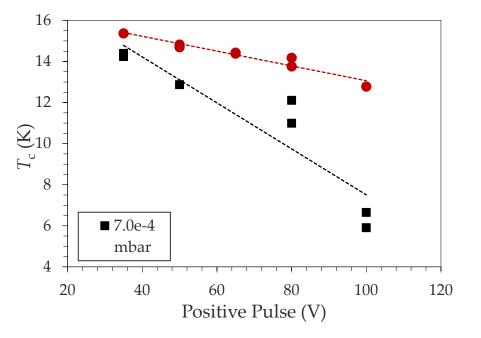


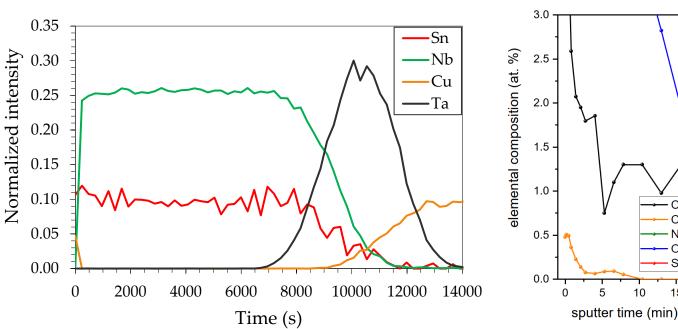
A15/Cu

https://indico.jlab.org/event/535/contributions/10694/attachments/8476/12127/Recent%20advances%20with%20bipolar%20HiPIMSdeposited%20Nb3Sn%20films%20on%20Cu S.%20Leith.pptx

Choice to move to Bi-polar HiPIMS

- Q-slope mitigation proven on Nb/Cu
- Detrimental to long range order parameter (bombardment energy)





Sn composition: OK

T_c: still lower than the theoretical value Cu surface contamination is a key issue

Recent RF measurements are very encouraging Communication under preparation



Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023



5

10



G. Rosaz et al.

Сι

Nb

15

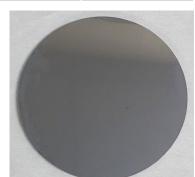


- 3 DCMS, 1 HiPIMS
- **Aim:** investigate effect of target power/deposition method
- Substrate preparation: ٠
 - Diamond turned Cu disks 10 cm diameter, 3 mm thick
 - Average roughness ~ 2-3 nm
- Sample preparation:

TESLA TECHNOLOGY

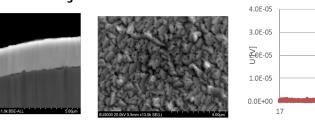
COLLABORATION

Parameter	DCMS	HiPIMS
Substrate heater current (A)	35 (~ 650 °C)	35 (~ 650 °C)
Target power (W)	200, 100, 50	100
Expected thickness (µm)	2.6	2.6



100 W DC Nb₃Sn on 50 and 100 mm diamond turned Cu

Nb₃Sn



100 W HIPIMS Nb₃Sn on 50 and 100 mm diamond turned Cu

176

0.00003

0.00002 > 0.00001

-0.00001

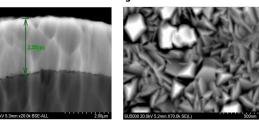
1761

18

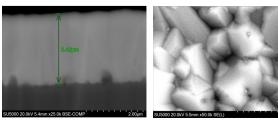
3.0E-05

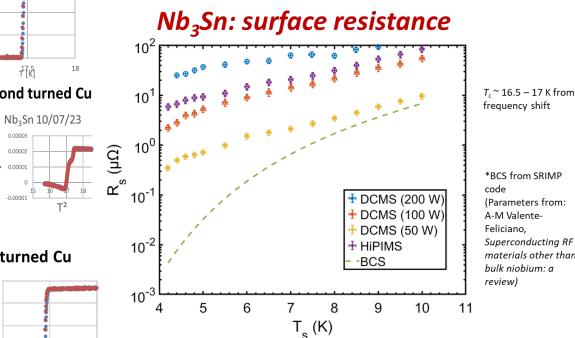
<u>⊋.0E-05</u>

1.0E-05 D.0E+00 17



50 W DC Nb₃Sn on 50 and 100 mm diamond turned Cu





R. Valizadeh et al.

*BCS from SRIMP code (Parameters from: A-M Valente-Feliciano, Superconducting RF materials other than

bulk niobium: a review)



NbTiN

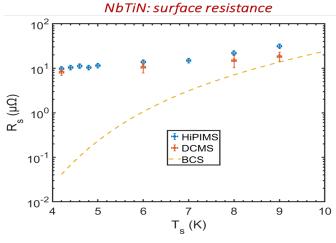


Synthesis Of NbTiN As An Alternative Thin Film For SRF Cavity – R. Valizadeh



Parameter	DCMS	HiPIMS
Substrate heater current (A)	35 (~ 650 °C)	35 (~ 650 °C)
Target power (W)	300	300
Expected thickness (µm)	0.8	0.8

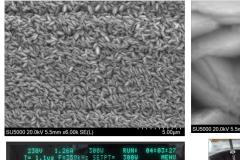




*BCS from SRIMP code (Parameters from: A-M Valente-Feliciano, Superconducting RF materials other than bulk niobium: a review)

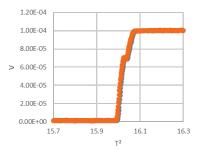
NbTiN from NbTi Rod

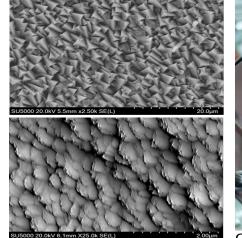
NbTiN from Nb Rod and Ti Wire

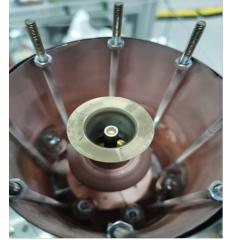


After several iteration of changing Ti wire loops composition of Ti_{0.5}Nb_{0.5} reached









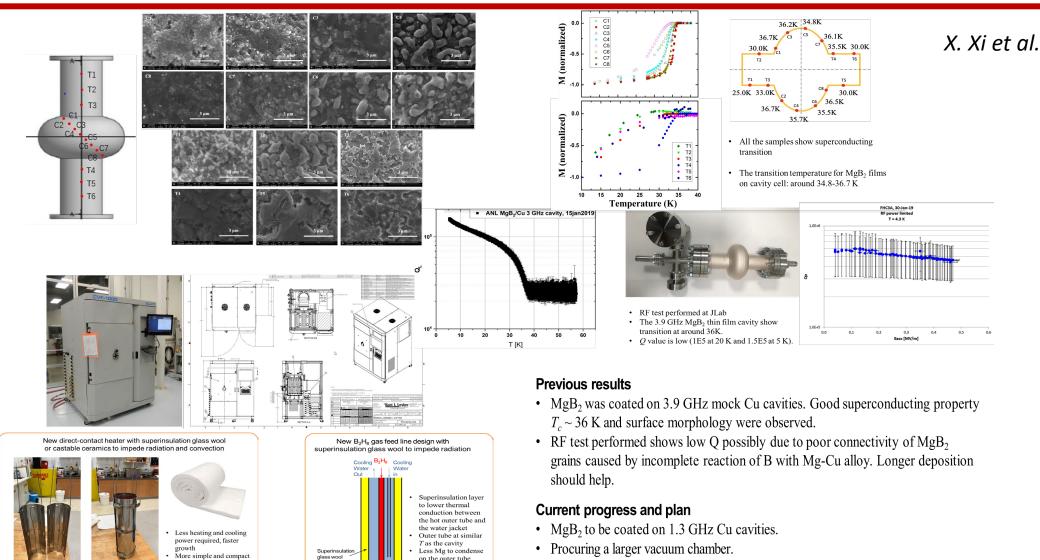
Composition is fixed Ti₆₃Nb₃₇





Other SC Materials beyond Niobium: MgB₂





- Cavity heater design is changed to direct-contact heating with superinsulation.
- Superinsulation replaces vacuum jacket to the B₂H₆ gas feeding line.

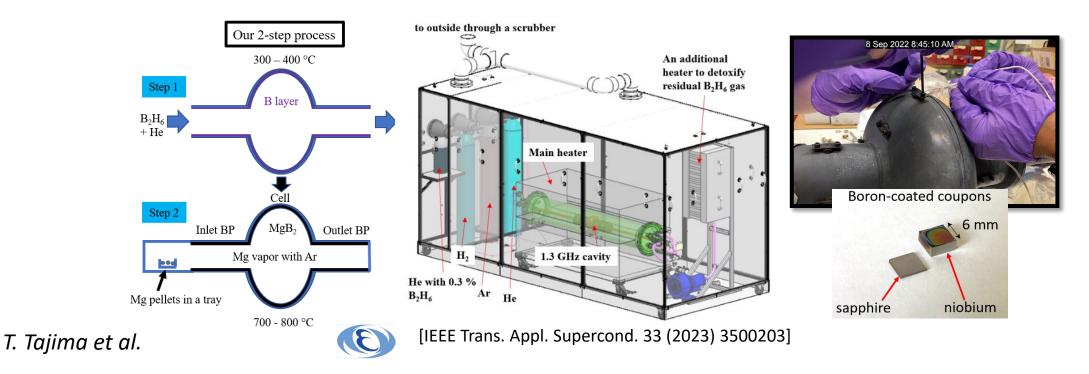






LANL MgB₂ coating status [U.S. – Japan Science and Technology Cooperation Program]

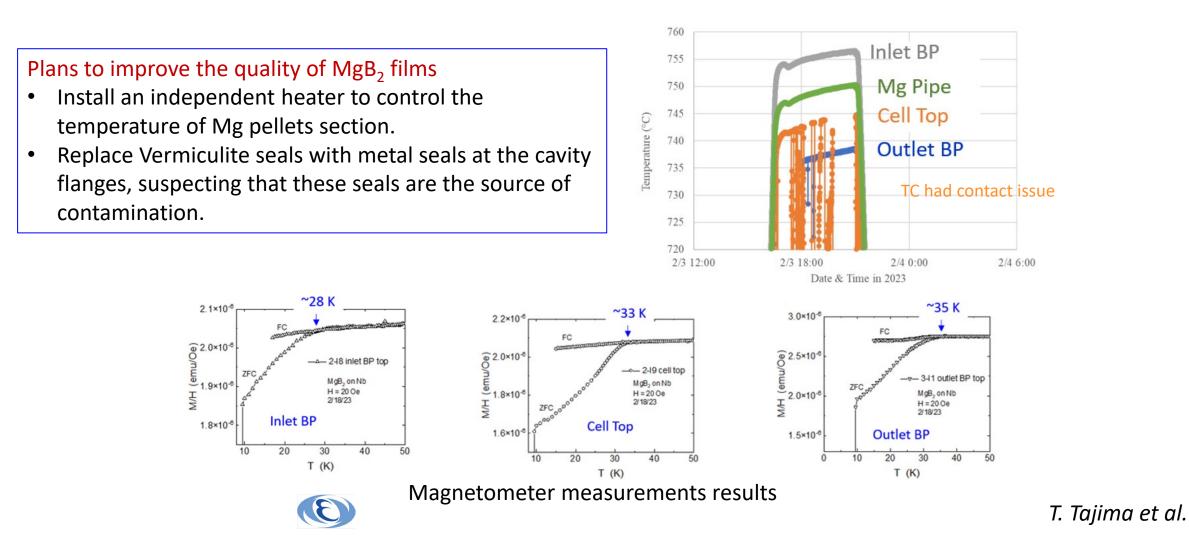
- Cavity coating booth with ventilation constructed using LANL LDRD funding
- B coating system plumbing, installation of flow and gas detectors, and interlocks underway
- Using old B-coated coupons, B-Mg reaction tests were conducted and confirmed SC transitions, but not high quality with lower T_c and broader transition as shown on the next slide











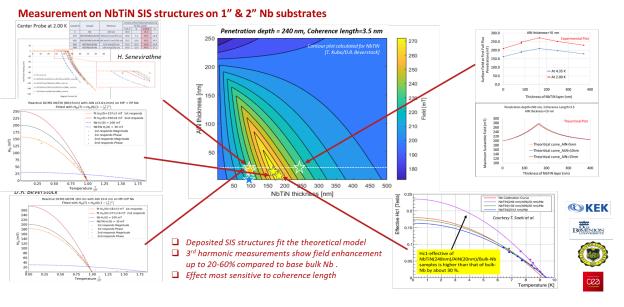


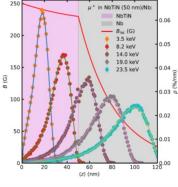


Beyond Nb: Multilayer Structures



NbTiN/AIN







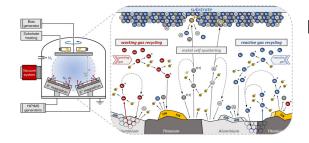
- \Box µSR measurements demonstrate the requirement of the dielectric layer in the SIS model
- □ High quality SIS structures for thicknesses all the way down to the nm level (Stack of 32 bilayers NbTiN/AIN/NbTiN/MgO is fully crystalline



TESLA TECHNOLOGY COLLABORATION



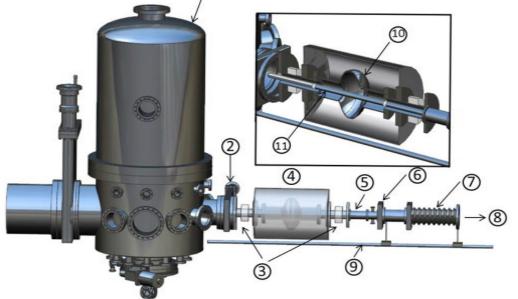
Re-HiPIMS



A-M Valente-Feliciano et al.

Refine deposition process for denser, more relaxed material in thin layers

Implementation on QPR samples & elliptical cavities for **RF@valuation**



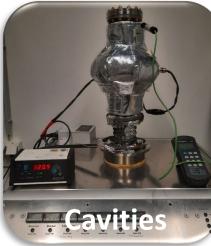
Development of Nb₃Sn Based SIS started



SIS by ALD



All under one roof: Coating & Testing - Samples & Cavities

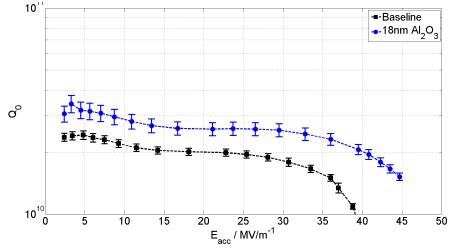


Thermal ALD

Several coated cavities by ALD of Al_2O_3 .

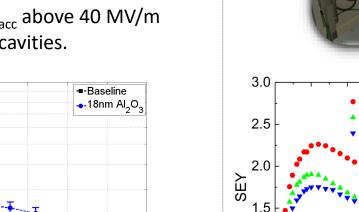
No deterioration of performances.

Maintaining E_{acc} above 40 MV/m for 2 out of 2 cavities.



[Wenskat, M. et al., Supercond. Sci. Technol. 36 015010 (2023)]





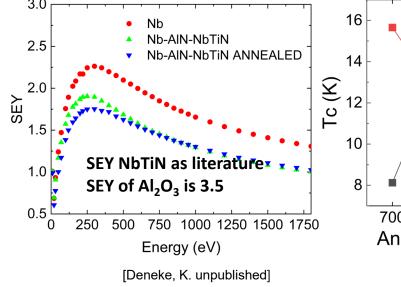


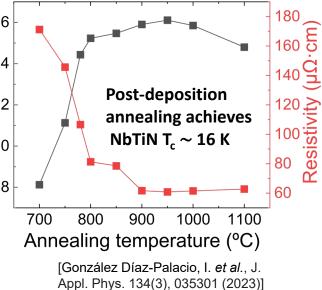
M. Wenskat et al.

Plasma-enhanced ALD

PEALD of AIN/NbTiN multilayers.

Characterization methods: ETO, VSM, SEM/TEM, SEY, XPS, etc.





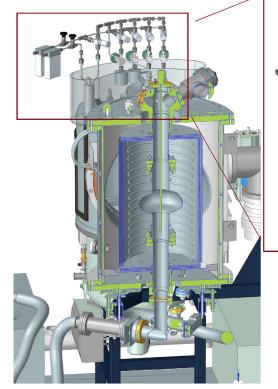
Jefferson Lab

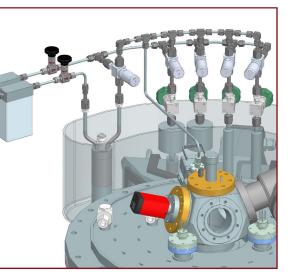
SIS by ALD

UHH

Merge SIS sample results and cavity coating

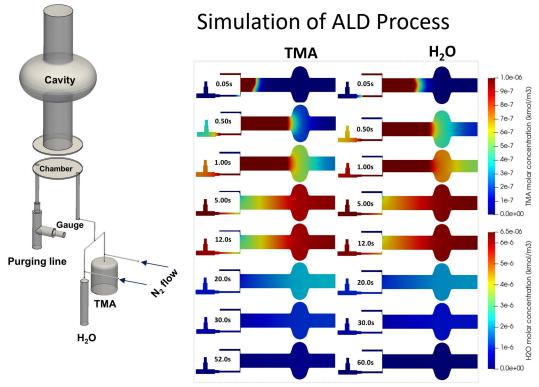
Plasma-Enhanced – Atomic Layer Deposition setup for single-cell cavities





- Design completed.
- In the stage of parts fabrication and purchasing.
- □ Commissioning spring 2024.
- □ First coatings planned for summer 2024.

Thermal budget reduction of coating SRF cavities



- Significant time reduction and process optimization while maintaining quality.
- Excellent agreement of experiment and simulations of fluid dynamics and chemical reactions.

[Deyu, G. et al., Chemical&Fluid Simulations on CavityCoating - to be submitted]



M. Wenskat et al.



Flux Trapping



[Turner, D. et al., MOPMB003, SRF2023]

Flux expulsion studies with CERN show Drastic **increase of expulsion efficiency** by continuous expulsion for each heat pulse even with **constant dT/dx**.

Thermal Resistance

[Saribal, C. et al., MOPMB017, SRF2023]

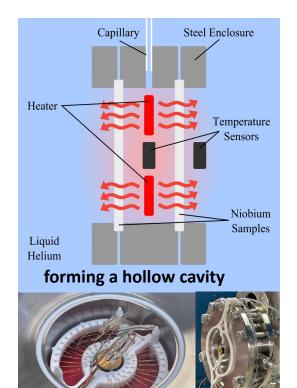
Thermal resistance of Nb-AlN-NbTiN shows no increase!

RF Measurements

[Monroy-Villa, R. et al., THCAA02, SRF2023]

QPR is now ready and 5 new samples for R&D are currently getting fabricated.





M. Wenskat et al.

QPR Setup for RF Measurements

UH







SIS by ALD

Y. Kalboussi, B. Delatte, C. Antoine, A. Four, F. Miserque, Y. Zheng, D. Hrabovsky, T. Junginger,



son Lab

Thin film R&D @ CEA

N.Lochet, D.Bafia, L.Grasselino, T. Proslier New cooling techniques: 3D printing of 3.9 GHz

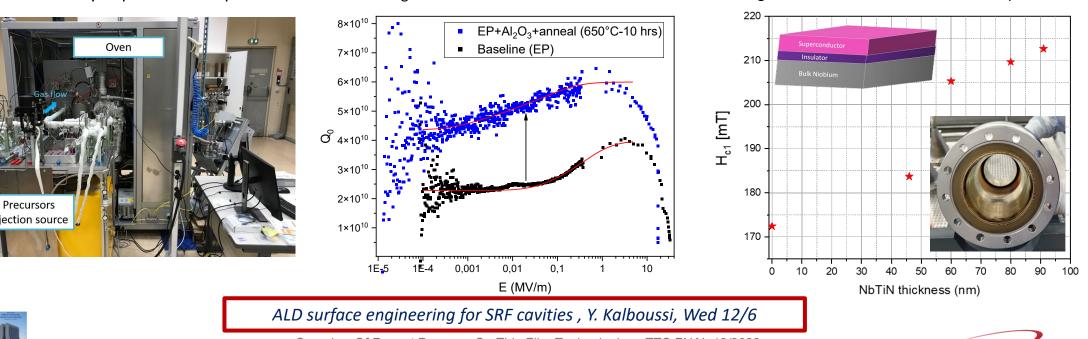
- cavities with closed loop cryocooler.
- Mitigating multipacting in SRF cavities by ALD and thermal treatments.
- Superconducting characterization of cavities and Qubits by tunneling spectroscopy.

✓ Increased Q at low field for 3D superconducting resonators 1.3 GHz.

✓ Increased penetration field on samples by 24%. First depositions of multilayers in 1.3 GHz cavities.

✓ N doped cavity by ALD of NbN. Optimization underway. First depositions of multilayers in 1.3 GHz cavities.

High Gradient for accelerators increased penetration field.



High Q studies for Qubits and accelerators

Home built Cavity deposition set up



COLLABORATION

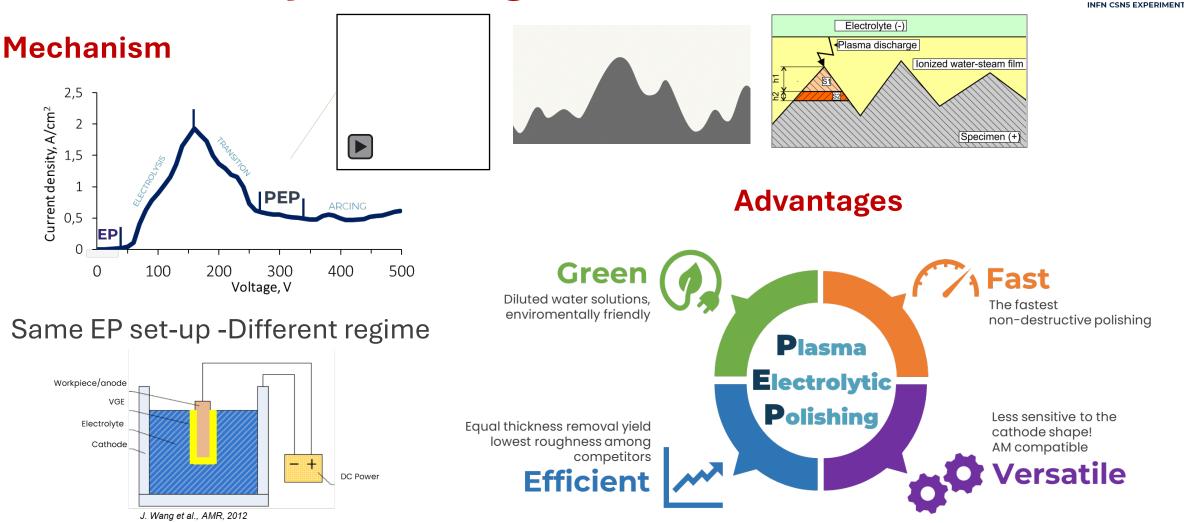
Advanced Substrate Preparation



S

MARA

Plasma Electrolytic Polishing PEP







Advanced Substrate Preparation

Photocathode

150

100 -

50 -

TESLA TECHNOLOGY

COLLABORATION





Plasma Electrolytic Polishing, Cristian Pira (INFN LNL), Tues 12/5



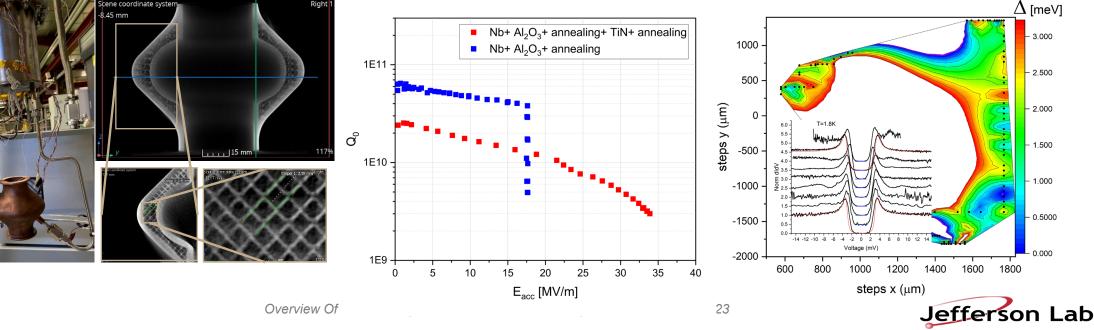
Thin film R&D @ CEA

Y. Kalboussi, B. Delatte, C. Antoine, A. Four, F. Miserque, Y. Zheng, D. Hrabovsky, T. Junginger, N.Lochet, D.Bafia, L.Grasselino, T. Proslier

- Engineering superconducting surface for high Q operation by Atomic layer deposition (ALD) and thermal treatments.
- Engineering superconducting surface for high gradient operation by ALD and thermal treatments: Doping without chemistry and multilayers.

- ✓ 3D printed 3.9 GHz cavity cryocooled to 4.2K. Successful power dissipation studies.
- ✓ Successful multipacting mitigation in 1.3 GHz cavities by TiN deposition.
- ✓ First samples measured: Nb₃Sn/Cu (CERN) and Nb/Ta resonators (USA).

Tunneling spectroscopy of Nb3Sn/Cu



Multipacting mitigation by ALD of TiN

Cryocooled 3D printed Cu cavity





Summary

Nb Thin Film Technology

- Results on cavities at different frequencies
- Demonstrations of Nb/Cu Q-slope mitigation

Beyond Nb: Alternate Materials

- □ Progress with Nb₃Sn by vapor diffusion towards cryomodules and conduction cooled cavities
- □ Further development of alternate Nb₃Sn coating techniques HiPIMS, sputtering, CVD...

Beyond Nb: Multilayer Structures

- □ Further development on samples for characterization and RF measurement (QPR)
- Development of concept from samples to cavities

Advance substrate fabrication & preparation

SRF Film Characterization

- \square µ-SR, β-NMR, PCT, QPR, flux expulsion ...
- Superconducting TF applications beyond SRF keep expanding (devices, sensors quantum ...)
 - □ Nb/Al₂O₃ films for qubits
 - NbTiN Films for Superconducting Digital Logic
 - □ Film based cavities for Axion research (NbTi cavity, INFN, C. Pira)





Save the date

11th International Workshop on Thin Films and New Ideas for Pushing the Limits of RF Superconductivity



International Organizing Committee

C. Antoine (CEA Saclay, France)A.- M. Valente-Feliciano (Jefferson Lab, USA)C. Pira (INFN LNL, Italy)A. Gurevich (Old Dominion University, USA)

W. Venturini (CERN, Switzerland)

R. Valizadeh (STFC, UK)

T. Saeki (KEK, Japan)



Will be held in 16-20 September 2024

In Paris Area, France Hosted by CEA Saclay, Sponsored by iFAST Program



