Overview Of Recent Progress On Thin Film Technologies

A.-M. Valente-Feliciano







Material provided by:

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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.







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oatings FCCweek 2023 carlota pereira.pptx



Nb Thin Film Technology



1.3 GHz Nb/Cu



Samples characterization by local microwave spectroscopy

https://arxiv.org/abs/2305.07746



Sample	ρ^{aefect}	$h_{penetration}^{ueffect}$
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





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Plasma diagnostic study with Liverpool University



Bipolar HiPIMS discharge



R. Valizadeh et al.

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







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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



Optimization process: Denser Sn droplets from simply higher Sn flux (left), Free of Sn droplets from Sn vapor adsorption, Optimized recipe after exploration (right), under the premise of effectively inhibiting the condensation of Sn droplets, the Sn source temperature can be safely increased



Process (2) • Process (3) Process (1) Process (7) ٠ ٠

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



See Dr. Ziqin Yang's Hot-topic slides on December 7 for more details



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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.





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COLLABORATION

Beyond Nb: Alternate Materials and Multilayer Structures Nb₃Sn



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Beyond Nb: Alternate Materials









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

娄 Fermilab





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Applied Research Center

Old Dominion Univ

Jefferson Lab





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Jefferson Lab



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









- 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





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

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

- 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.



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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

Nb₃Sn

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



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5

10

sputter time (min)





Сι

Nb

15

Science and Technology **Facilities** Council

- 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:

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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

0.00003

0.00002 > 0.00001

-0.00001

1761

18

3.0E-05

₹.0E-05

1.0E-05 D.0E+00 17



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





 $T_{s}(K)$

R. Valizadeh et al.



 $T_c \simeq 16.5 - 17$ K from

frequency shift



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 $\rm Ti_{0.5}Nb_{0.5}$ reached









Composition is fixed Ti₆₃Nb₃₇





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







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



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















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.



Further Characterization

Flux Trapping

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



Flux expulsion studies with CERN show new phenomena called *"ratcheting"* on SIS samples.

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!



M. Wenskat et al.

RF Measurements

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

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





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Thin film R&D @ CEA

- 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.

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

- ✓ 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.

90

100

rson Lab



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 -

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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



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



