Overview Of Recent Progress On Thin Film Technologies

A.-M. Valente-Feliciano
ACKNOWLEDGEMENT

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Recent progress in SRF thin film developments since SRF 2023

- Nb Thin Film Technology
- Beyond Nb – Alternate Materials
  - Nb$_3$Sn
  - NbTiN
  - MgB$_2$
- Beyond Nb – Multilayers
- SRF Thin Film Characterization
- Substrates

Disclaimer:
Non-exhaustive summary
Many more ongoing developments, awaiting results, validation...
Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023

400 MHz Nb/Cu cavities

Best ever produced 400MHz Nb/Cu
FCC 1 cell specs reached
Chemistry: SUBU
EP commissioned and ready for 2024

Next target: FCC 2-cells specs

https://indico.cern.ch/event/1202105/contributions/5391683/attachments/2662106/4612074/HIPIMS_Nb_coatings_FCCweek_2023_carlota_pereira.pptx

G. Rosaz et al.
1.3 GHz Nb/Cu

Samples characterization by local microwave spectroscopy

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.

Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023
1.3 GHz Nb/Cu

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

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

G. Rosaz et al.
1.3 GHz Nb/Cu

Niobium film planarization

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

G. Rosaz et al.
Growth in sequential phases developed for ECR implemented on cavities with HiPIMS

- HiPIMS cavity coating fully resumed after system rebuild
- Deposition ramped up to 1 cavity/week if substrate available
- Build a strong RF measurement program
- Lower frequency cavity deposition (952.6 & 800 MHz, substrates on hand)

A-M Valente-Feliciano et al.

- Lower frequency cavity deposition (952.6 & 800 MHz, substrates on hand)

HiPIMS Nb/Nb 1.3 GHz TE cavity

JLab, 1.3 GHz Nb/Nb TE (ML02.1), -25V/-50V, 150C
**Nb Thin Film Technology**

**Nb/Cu: surface resistance at 4.2 K**

- Target 1
- Target 2

- Variation in $T_c$ due to Cu contamination
- $T_c$ as expected for all samples

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**Bipolar HiPIMS discharge**

Starfire Industries Impulse 2-2
Liverpool University

Ionastics HIPSTER Bipolar
Vilson et. al.*

*https://doi.org/10.1016/j.surfcoat.2021.137407

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**1.3 GHz Cavity deposition system**

**From planar samples to real cavities**

Aim: Flat samples $\rightarrow$ split cavities $\rightarrow$ 1.3 GHz cavities

3 sets of samples:

1. Nb coated planar samples
   - Low power RF test with choke cavity
   - High power RF test with QPR

2. Split cavity deposited with planar magnetron & planar target
   - RF test

3. Split cavity deposited with cylindrical magnetron & tubular target
   - RF test

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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.
Material studies and development of Nb$_3$Sn-coated cavities

U. Pudasaini et al.

5-cells deposition towards implementation in CM

Progress with Nb$_3$Sn C75 Quarter Cryomodule

- One cavity was coated at Fermilab and another at JLab.
- Cavity assembled in the pair and subjected to dips in vacuum because of a leak and assembled again with some degradation in the cavity performance.
- Quarter cryomodule assembly is completed.
- Test scheduled for Jan 2024.
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.

Key to high-performance Nb$_3$Sn film:
1. Temperature uniformity of cavity
2. Sufficient Sn vapor flux
3. Timely removal of residual Sn vapor before cooling

T. Tan et al.
Development of conduction-cooled LHe-free SRF technology

Two cooldown processes crossing 30-15K

Thermal cycle effect of conduction-cooled Nb₃Sn SRF cavity:
1. Uneven temperature distribution in the first slow-cooldown due to the local contact between the copper strip and the cavity,
2. Repeated cooldown has been shown to reduce non-uniformity.

Improvement of RF performance after repeated cooldown process

Frequency vibration caused by GM cryocoolers and suppression:
1. GM cryocoolers can cause frequency vibration of conduction cooled SRF cavity
2. Reasonable damping structure design can significantly reduce the impact of GM cryocoolers

The mechanical vibration of the cavity is obviously suppressed after optimization.

Frequency vibration decreased to ~15Hz after vibrations transmitted to the ground

Frequency vibration~27Hz in the case of vibrations transmitted to the top of module measured by frequency meter

The mechanical vibration of the cavity is obviously suppressed after optimization.

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

Cernox sensors and magnetic flux gates

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### Nb$_3$Sn Cavities Coated by Tin Vapor Diffusion

(Jiankui Hao, PKU)

Coating: 1250°C, 120 min, Annealing: 1150 °C, 60 min

$Q_0 \sim 1.1 \times 10^{10} @ 4.2K @ low\ field, \ max. E_{

<table>
<thead>
<tr>
<th>Vertical Test (2023)</th>
<th>$T_{q_0}$</th>
<th>$Q_0$ @ ~1.0 MV/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>NS03 (1st, Oct. 17)</td>
<td>15.7 K/m, ~6 min/K</td>
<td>3.3E9</td>
</tr>
<tr>
<td>NS06 (1st, Oct. 17)</td>
<td>15.7 K/m</td>
<td>3.0E9</td>
</tr>
<tr>
<td>NS03 (2nd, Oct. 18)</td>
<td>110</td>
<td>1.1E9</td>
</tr>
<tr>
<td>NS03 (3rd, Oct. 23)</td>
<td>2.7 K/m, ~10 min/K</td>
<td>4.9E9</td>
</tr>
<tr>
<td>NS06 (3rd, Oct. 23)</td>
<td>2.7</td>
<td>4.7E9</td>
</tr>
</tbody>
</table>

Challenges for conduction-cooled SRF cavity technology
Electrochemical Synthesis

Achieve a smooth Nb$_3$Sn film with uniform thickness and stoichiometry

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

⇒ smoother Nb$_3$Sn films with little variation in Sn concentration with depth.

→ First ever alternative growth method to vapor diffusion to achieve quality factors $>10^{10}$ at 4.2K

→ Very low BCS low resistance

HOT TOPIC: Challenges for conduction-cooled SRF cavity technology
Beyond Nb: Alternate Materials and Multilayer Structures

CVD Nb₃Sn Thin Film Performance

Shawn R. McNeal, Victor M. Arrieta
Ultramet | Pacoima, California

TESLA Technology Collaboration Meeting (TTC 2023)
Fermilab | Batavia, Illinois | December 5–8, 2023

PROGRESS

CVD Nb₃Sn coating on copper substrate: excellent adhesion

CVD Nb₃Sn coating on CVD niobium interlayer on welded (Niowave) copper cavity substrate

CVD Nb₃Sn on welded copper cavity

HOT TOPIC

Challenges for conduction-cooled SRF cavity technology

SN-4A, seamless copper cavity substrate (BTM, Inc.)

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

Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023
**Beyond Nb: Alternate Materials**

**Nb$_3$Sn Coatings: Target Production**

- **Single target configuration easiest to scale onto elliptical geometry**

- **Nb$_3$Sn cylindrical target are not commercially available**

**LNL Strategy for Nb$_3$Sn cylindrical target production**
(6 GHz cavities)

- **Raw Nb target** → **Dipping in liquid Sn** → **Nb$_3$Sn cylindrical target** → **Nb$_3$Sn Magnetron Sputtering** → **Nb$_3$Sn on Cu cavity**

**Nb$_3$Sn thickness related to dipping time**

**Possible tin content modulation**

**Proof of concept**
Strategy to get the nominal $T_c$

A very thick Nb barrier enhance dramatically $T_c$

$T_c = 17.33 \pm 0.25 \, K$ on Cu + 50 $\mu$m Nb Buffer Layer at $T_{dep} = 600 \, ^{\circ}C$

Beyond Nb: Alternate Materials

Cu + 1 $\mu$m Nb$_3$Sn

Cu + 1 $\mu$m Nb + 1 $\mu$m Nb$_3$Sn

Cu + 50 $\mu$m Nb + 1 $\mu$m Nb$_3$Sn

Beyond Nb: Alternate Materials

Cu + 1 $\mu$m Nb$_3$Sn

Cu + 1 $\mu$m Nb + 1 $\mu$m Nb$_3$Sn

Cu + 50 $\mu$m Nb + 1 $\mu$m Nb$_3$Sn

$T_c = 17.33 \pm 0.25 \, K$ on Cu + 50 $\mu$m Nb Buffer Layer at $T_{dep} = 600 \, ^{\circ}C$
Beyond Nb: Alternate Materials

Nb$_3$Sn coating of a 2.6 GHz Nb SRF cavity using a Cylindrical Magnetron Sputtering System - Sharifuzzaman Shakel, et al.

$\text{Nb}_3\text{Sn}$

$\text{Nb}_3\text{Sn}$ coating using cylindrical stoichiometric target

$\text{Nb}_3\text{Sn}$ has been grown on 4.5" long Nb tube target using Sn vapor diffusion system in Jefferson Lab.

-148 hours of Sn vapor diffusion process produces 20-22 nm of Nb$_3$Sn on the surface of the tube.

$\text{Nb}_3\text{Sn}$ tube target has been used to spatter deposit Nb$_3$Sn film on Sapphire substrate using Cylindrical Magnetron Sputtering System.

$\text{Nb}_3\text{Sn}$ target. Spatter discharged using the bottom magnetron.

30W DC power

10-1017 mA magnetron current

31 mTorr Ar background pressure

50 scm argon flow rate

Composition of as-deposited Nb$_3$Sn films from stoichiometric Nb$_3$Sn tube target

<table>
<thead>
<tr>
<th>Positions</th>
<th>Total Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top beams tube</td>
<td>122 nm</td>
</tr>
<tr>
<td>Bottom beams tube</td>
<td>137 nm</td>
</tr>
<tr>
<td>Emitter</td>
<td>140 nm</td>
</tr>
</tbody>
</table>

$\text{Nb}_3\text{Sn}$ is deposited from the target inside a reactor tube, where the plasma is created.

Sample Position Nb (wt %) Sn (wt %)

| Top beams tube | 74  26 |
| Emitter       | 73  27 |
| Bottom beams tube | 74  26 |

$\text{Nb}_3\text{Sn}$ crystallographic Nb or Sn formation is observed in the XRD pattern of the as-deposited film. Only diffraction peaks from the epitaxial substrate are observed.

As-deposited films will be annealed at 950°C for 3 hours for Nb and Sn to interdiffuse growing Nb$_3$Sn.

1 µm Nb$_3$Sn film will be coated inside 2.6 GHz Nb cavity and annealed. The performance of the coated cavity will be tested.

Overview Of Recent Progress On Thin Film Technologies - TTC FNAL 12/2023
Beyond Nb: Alternate Materials

\( \text{Nb}_3\text{Sn} \)

- KEK introduced the sputtering apparatus, SH-450 (ULVAC inc.).
- SH-450 is capable of \( \text{Nb}_3\text{Sn} \) 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.

- \( \text{Nb}_3\text{Sn} \) coating method can be applied to the inside of 3 GHz cavity.
- \( \text{Nb}/\text{Sn} \) composite cathode is the key.
- Development of the special cathode is ongoing.

T. Saeki, R. Katayama et al.

- 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.
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
Beyond Nb: Alternate Materials

**Nb$_3$Sn**

- 3 DCMS, 1 HiPIMS

- **Aim:** investigate effect of target power/deposition method

- **Substrate preparation:**
  - Diamond turned Cu disks – 10 cm diameter, 3 mm thick
  - Average roughness ~ 2-3 nm

- **Sample preparation:**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>DCMS</th>
<th>HiPIMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate heater current (A)</td>
<td>35 (~ 650 °C)</td>
<td>35 (~ 650 °C)</td>
</tr>
<tr>
<td>Target power (W)</td>
<td>200, 100, 50</td>
<td>100</td>
</tr>
<tr>
<td>Expected thickness (µm)</td>
<td>2.6</td>
<td>2.6</td>
</tr>
</tbody>
</table>

**100 W DC Nb$_3$Sn on 50 and 100 mm diamond turned Cu**

**100 W HIPIMS Nb$_3$Sn on 50 and 100 mm diamond turned Cu**

**50 W DC Nb$_3$Sn on 50 and 100 mm diamond turned Cu**

R. Valizadeh et al.

**Nb$_3$Sn: surface resistance**

T$_c$ ~ 16.5 – 17 K from frequency shift

*BSC from SR/IMP code (Parameters from: A-M Velente-Feliciano, Superconducting RF materials other than bulk niobium: a review)
Beyond Nb: Alternate Materials

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

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<td>35 (~650 °C)</td>
</tr>
<tr>
<td>Target power (W)</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>Expected thickness (μm)</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

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

**NbTiN from Nb Rod and Ti Wire**

After several iteration of changing Ti wire loops composition of Ti$_{10}$Nb$_{35}$ reached

**NbTiN from NbTi Rod**

Composition is fixed Ti$_{10}$Nb$_{37}$

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Other SC Materials beyond Niobium: MgB$_2$

Previous results
- MgB$_2$ was coated on 3.9 GHz mock Cu cavities. Good superconducting property $T_c \approx 36$ K and surface morphology were observed.
- RF test performed shows low Q possibly due to poor connectivity of MgB$_2$ grains caused by incomplete reaction of B with Mg-Cu alloy. Longer deposition should help.

Current progress and plan
- MgB$_2$ to be coated on 1.3 GHz Cu cavities.
- Procuring a larger vacuum chamber.
- Cavity heater design is changed to direct-contact heating with superinsulation.
- Superinsulation replaces vacuum jacket to the B$_2$H$_6$ gas feeding line.
Other SC Materials beyond Niobium: MgB$_2$

LANL MgB$_2$ 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

Other SC Materials beyond Niobium: MgB$_2$

Plans to improve the quality of MgB$_2$ films
- Install an independent heater to control the temperature of Mg pellets section.
- Replace Vermiculite seals with metal seals at the cavity flanges, suspecting that these seals are the source of contamination.

Magnetometer measurements results

T. Tajima et al.
Beyond Nb: Multilayer Structures

**NbTiN/AiN**

Measurement on NbTiN SIS structures on 1" & 2" Nb substrates

- Deposited SIS structures fit the theoretical model
- 3rd harmonic measurements show field enhancement
  up to 20-40% compared to bare bulk Nb
- Effect most sensitive to coherence length

**Re-HiPIMS**

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

**Implementation on QPR samples & elliptical cavities for RF evaluation**

**Development of Nb3Sn Based SIS started**

A-M Valente-Feliciano et al.
Beyond Nb: Multilayer Structures

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SIS by ALD

Thermal ALD

Several coated cavities by ALD of Al₂O₃.

No deterioration of performances.

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

Plasma-enhanced ALD

PEALD of AlN/NbTiN multilayers.

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

Cavities

Thermal ALD

Several coated cavities by ALD of Al₂O₃.

No deterioration of performances.

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

Plasma-enhanced ALD

PEALD of AlN/NbTiN multilayers.

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

Post-deposition annealing achieves NbTiN $T_c \sim 16$ K

SEY NbTiN as literature

SEY of Al₂O₃ is 3.5

Samples


Deneke, K. unpublished


Deneke, K. unpublished
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.

Beyond Nb: Multilayer Structures

Thermal budget reduction of coating SRF cavities

Simulation of ALD Process

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

M. Wenskat et al.

[Deu, G. et al., Chemical & Fluid Simulations on Cavity Coating – to be submitted]
Further Characterization

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.
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.

Home built Cavity deposition set up

- High Q studies for Qubits and accelerators
- High Gradient for accelerators increased penetration field.

- 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.

Beyond Nb: Multilayer Structures

SIS by ALD
Advanced Substrate Preparation

**Plasma Electrolytic Polishing (PEP)**

**Mechanism**

- Same EP set-up - Different regime

**Advantages**

- Green: Diluted water solutions, environmentally friendly
- Fast: The fastest non-destructive polishing
- Efficient: Equal thickness removal yield lowest roughness among competitors
- Versatile: Less sensitive to the cathode shape! AM compatible
Plasma Electrolytic Polishing (PEP)

Results

150 μm removed in ~ 5 h
150 μm removed in ~ 40 min

Ra ~ 8 nm!

Solution Patented by INFN

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

6 GHz Cu cavity

Plasma Electrolytic Polishing, Cristian Pira (INFN LNL), Tues 12/5
Substrates & Film Characterization

Thin film R&D @ CEA


- 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: Nb$_3$Sn/Cu (CERN) and Nb/Ta resonators (USA).

Cryocooled 3D printed Cu cavity

Multipacting mitigation by ALD of TiN

Tunneling spectroscopy of Nb3Sn/Cu
Summary

- **Nb Thin Film Technology**
  - Results on cavities at different frequencies
  - Demonstrations of Nb/Cu Q-slope mitigation

- **Beyond Nb: Alternate Materials**
  - Progress with Nb$_3$Sn by vapor diffusion towards cryomodules and conduction cooled cavities
  - Further development of alternate Nb$_3$Sn 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**
  - $\mu$-SR, $\beta$-NMR, PCT, QPR, flux expulsion …

- **Superconducting TF applications beyond SRF keep expanding (devices, sensors quantum …)**
  - Nb/Al$_2$O$_3$ 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