Advances with Nb_3Sn HiPIMS coatings for SRF cavities

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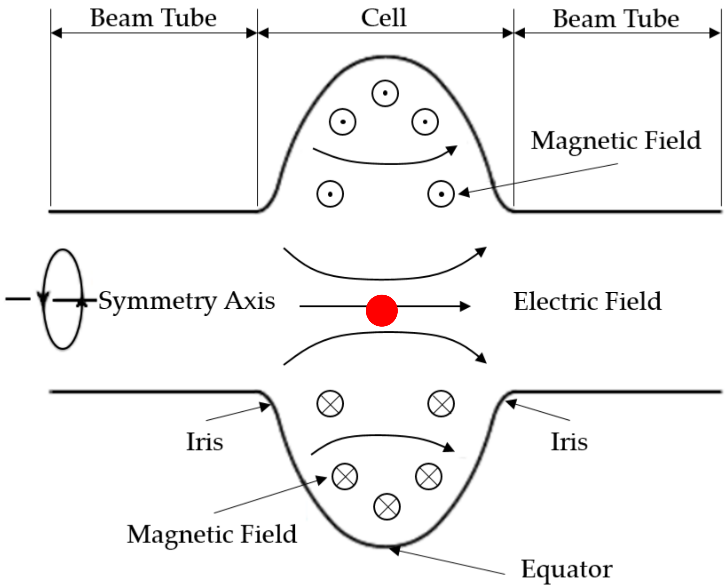
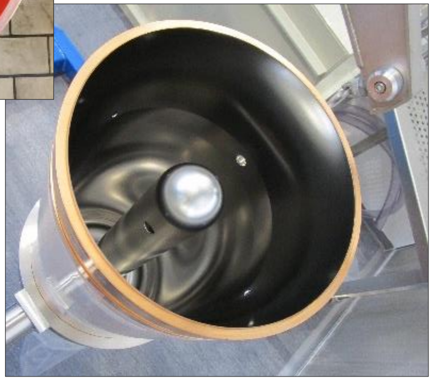
Background

Superconducting Radio Frequency (SRF) Cavities

- Accelerate charged particles

- Bulk Nb vs. **Nb/Cu** → LHC, HIE-ISODLE





Figures of Merit

- Quality factor

$$Q_0 = G/\boldsymbol{R_s} = \frac{\text{stored power}}{\text{dissipated power}}$$

- Surface resistance

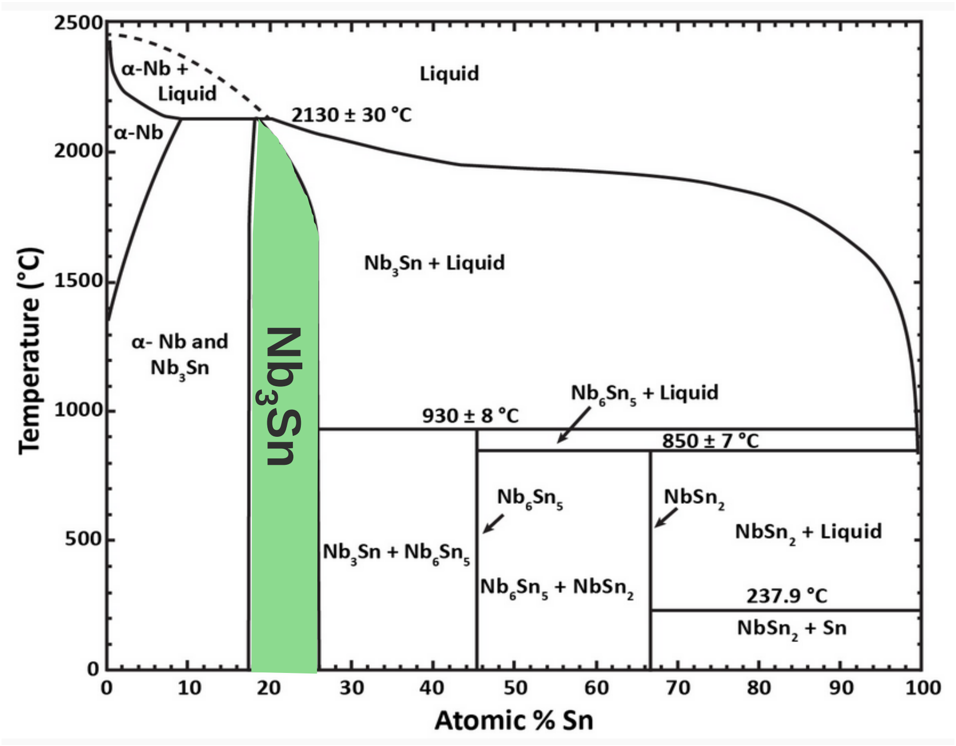
$$\boldsymbol{R_s} = R_{BCS} + R_{res}$$

Motivation

Nb_3Sn

- Q_0 at 4.2 K ~ bulk Nb at 2 K

T_c	Nb ~ 9.2 K
	Nb_3Sn ~ 18.3 K
R_{BCS} @4.2K and 500MHz	Nb ~ 45nΩ
	Nb_3Sn ~ 0.4nΩ



Binary phase diagram of the Nb-Sn system [1]

Challenges

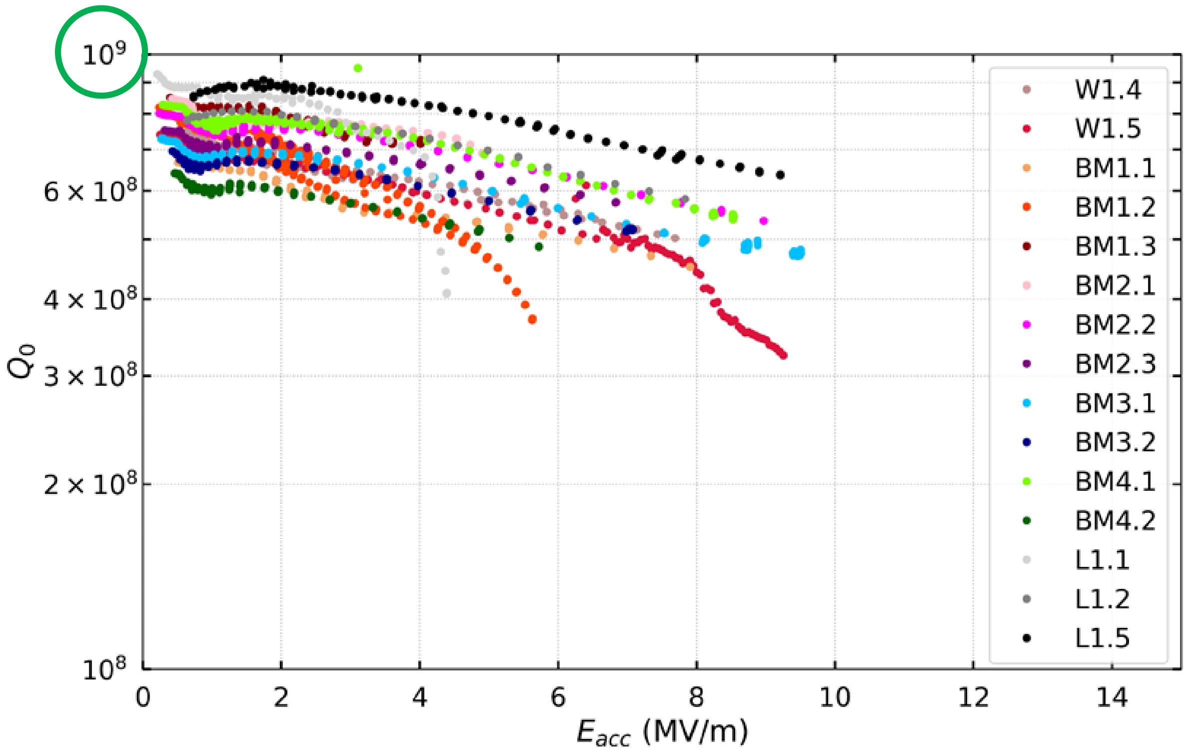
- Superconducting Nb_3Sn phase formation
  - Stoichiometry control (Sn at. % 18 - 26 at. %)
  - High temperature reaction
- Copper substrate influence

[1] J. Charlesworth, I. MacPhail, and P. Madsen, J. Mater. Sci. **5**, 580 (1970).

State of the art

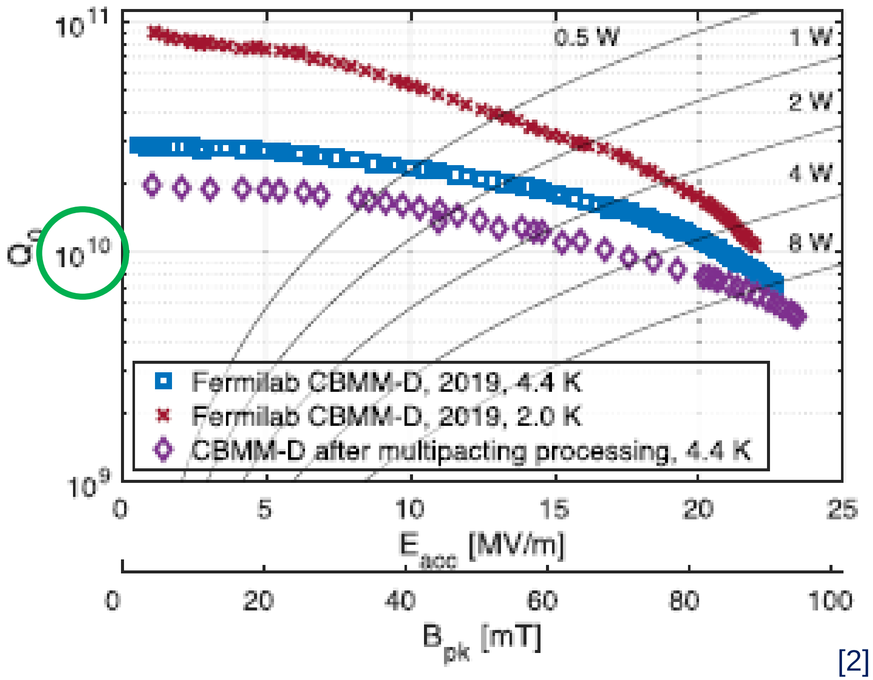
1.3GHz cavities

Nb/Cu @ 4.2K



Courtesy: L. Vega-Cid

Nb_3Sn/Nb @ 4.4K



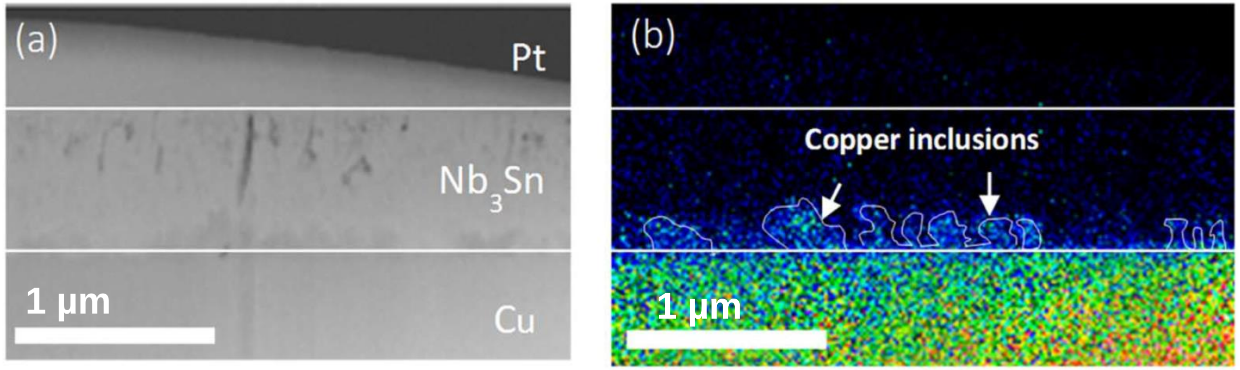
[2]

Order of magnitude improvement in Q_0

[2] S. Posen et al, Supercond. Sci. Technol. **34** (2021)

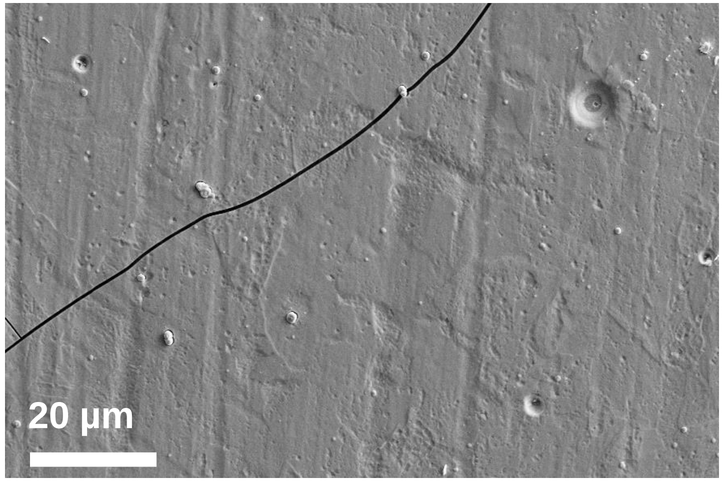
DC MS Nb_3Sn Coatings @ CERN

DC Magnetron Sputtering (DC MS)



- **Formation of A15 phase**
  - Reacted During and After Coating
  - High temperatures required (> 600°C)

- **Cu Diffusion**
  - Barrier interlayer required (Ta or Nb)



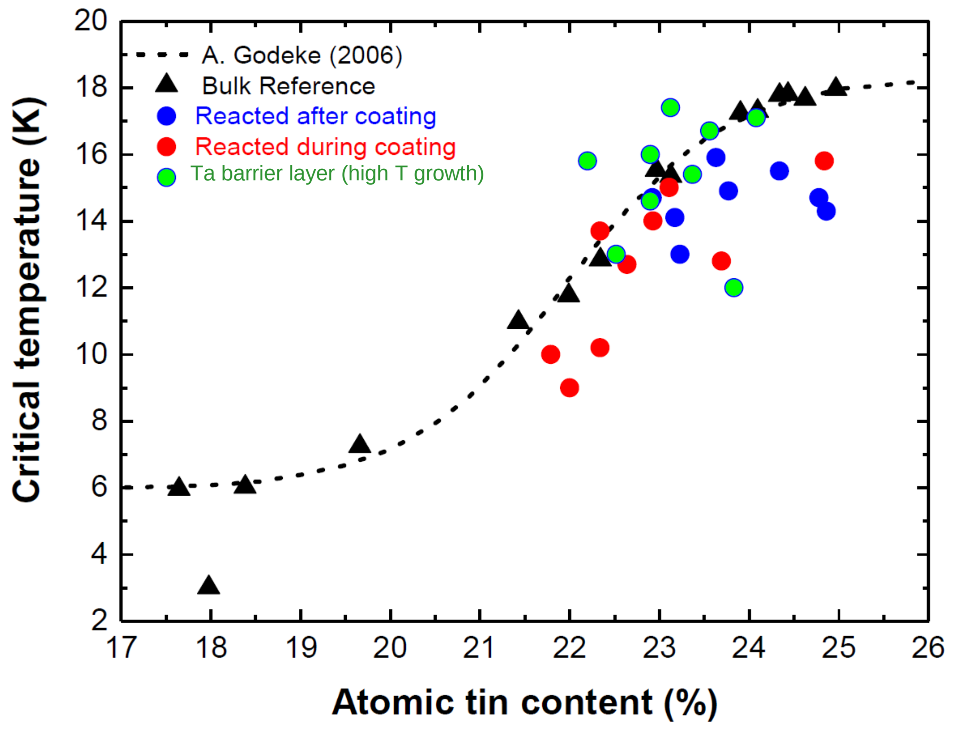
- **Surface cracking**
  - Mitigated with Kr

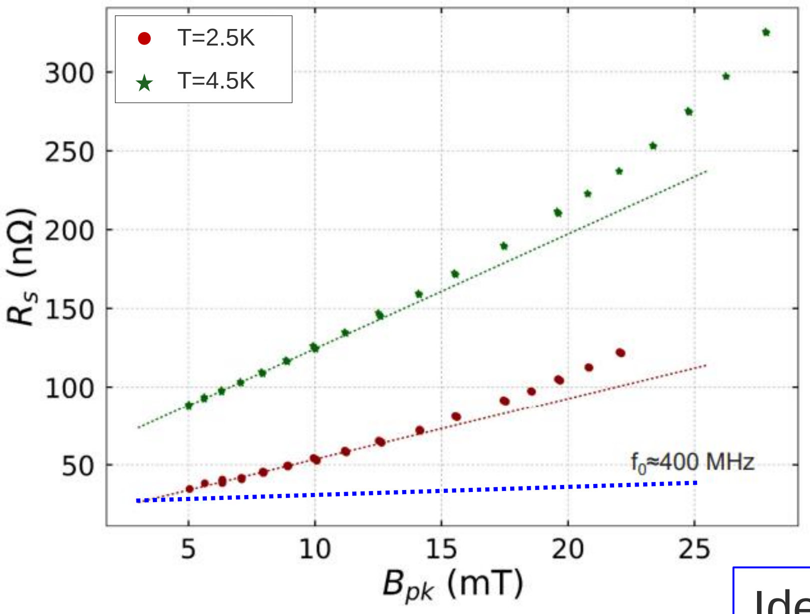
[3] K. Ilyina et al. *Supercond. Sci. Technol.* ***32*** *(2019)*

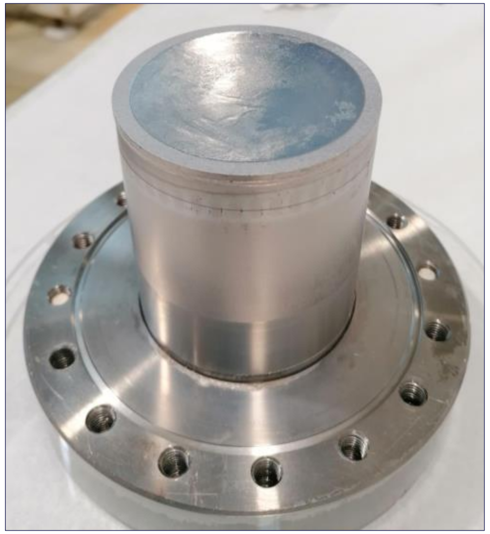
DC MS Nb_3Sn Coatings @ CERN

DC Magnetron Sputtering (DC MS)

- **Impressive T_c**
  - Consistently higher when reacted after coating
  - Sn dependent (increased with Kr)
  - Increased with Ta interlayer







Ideal behaviour

- **Quadrupole Resonator (QPR) Coatings**
  - Reduction of low field R_s
  - Q-slope

Switch to HiPIMS

[3] K. Ilyina et al. *Supercond. Sci. Technol.* ***32*** *(2019)*
[4] M. Arzeo et al. FCC Week 2018

Bipolar HiPIMS Nb_3Sn/Ta/Cu

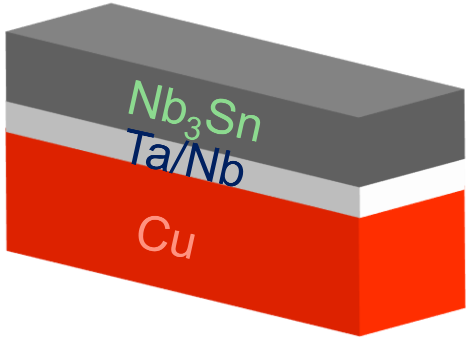
Reacted During Coating

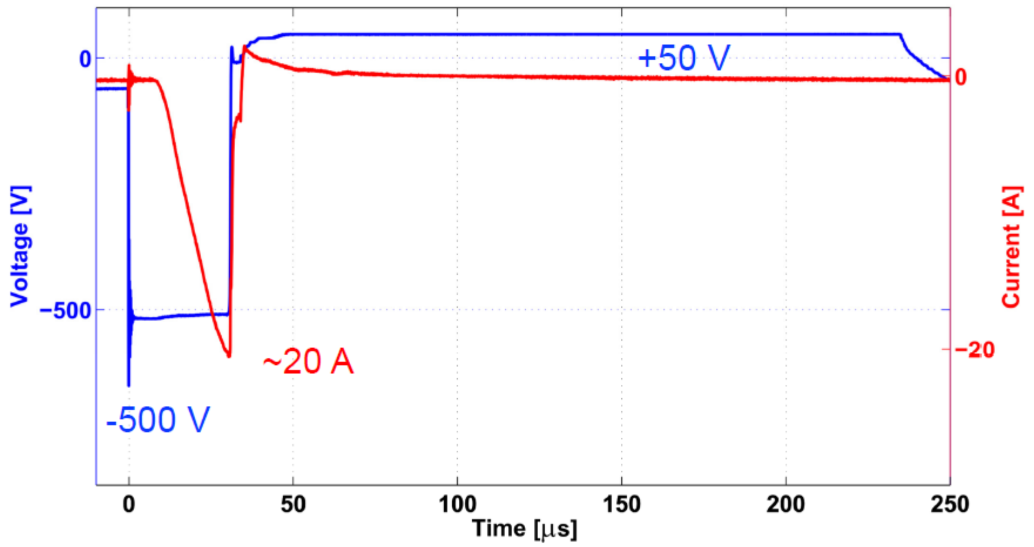
High Power Impulse Magnetron Sputtering (HiPIMS)

- Improved density required for RF performance
- Proven with Nb/Cu

Coating parameters:

- *Gas: Kr*
- T_s: 500 … 750°C
- *P:* $7{\cdot}10^{-4}$ … $5{\cdot}10^{-2}$ *mbar*
- *PP:* 35 … 100 V
- *Post anneal:* 0 … 72 hrs



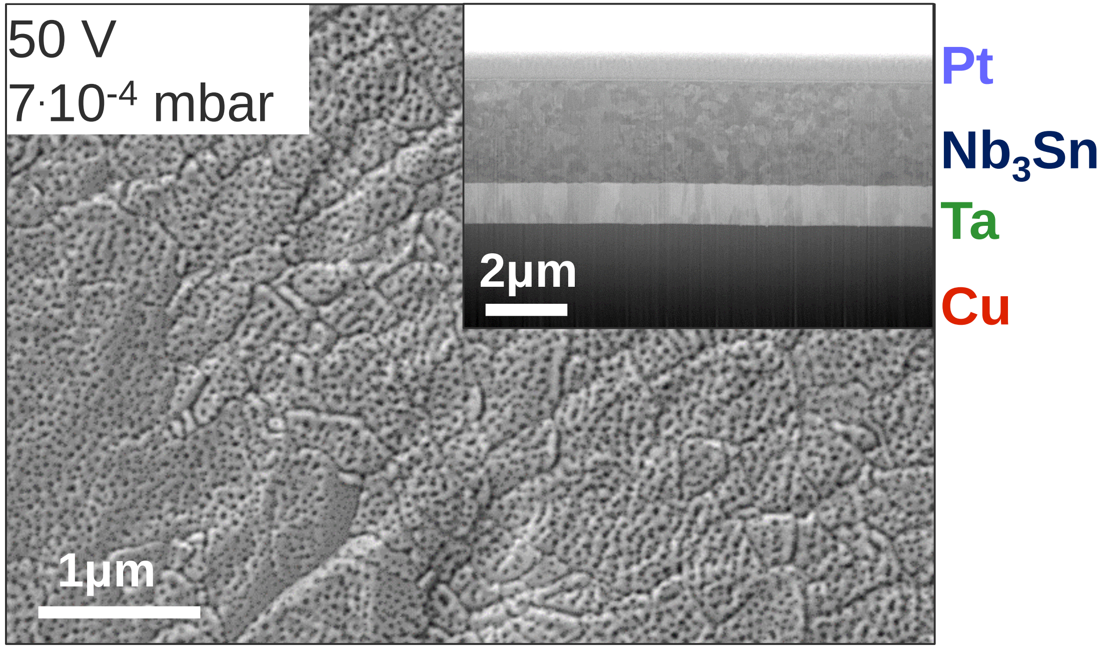


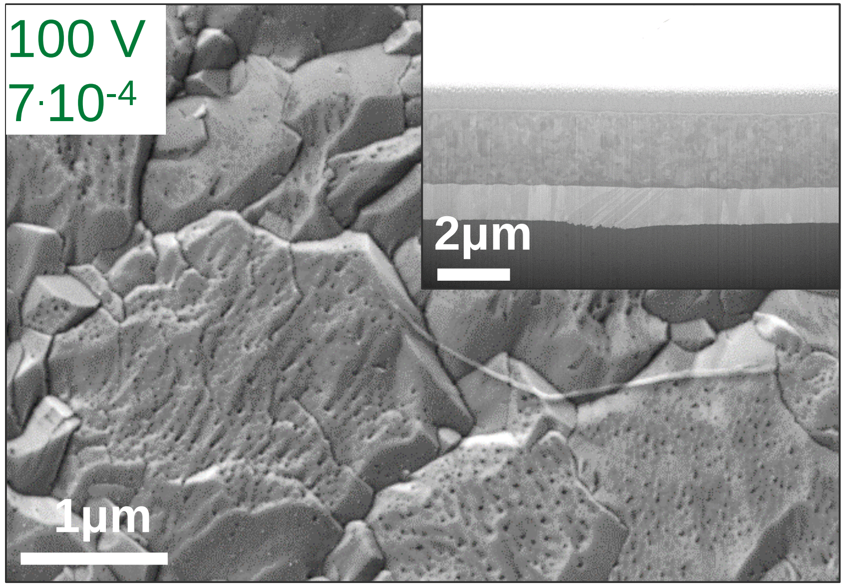
[5]

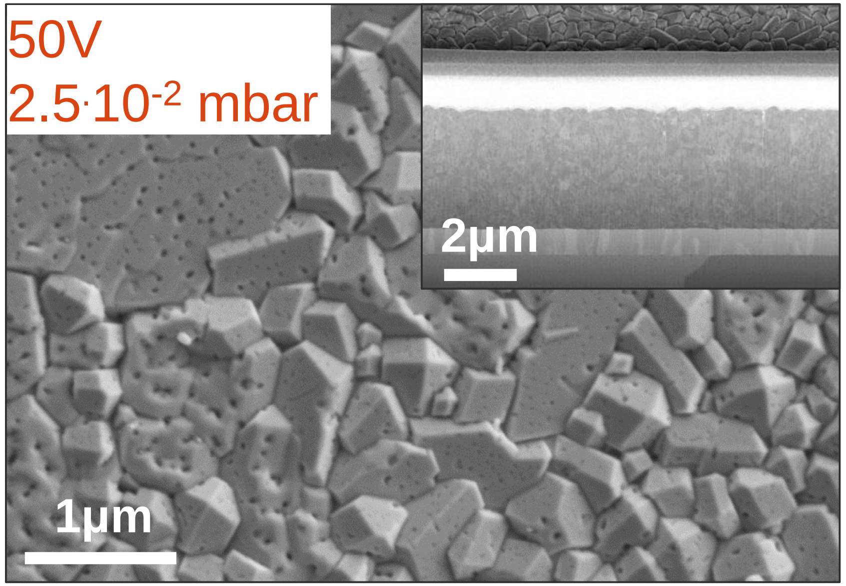
[5] F. Avino *et al* 2019 *Plasma Sources Sci. Technol.* **28** 01LT03

Morphology and Crystallinity

- Correct A15 phase for all samples
- Dense, crack free, porous surface
- No effect due to annealing

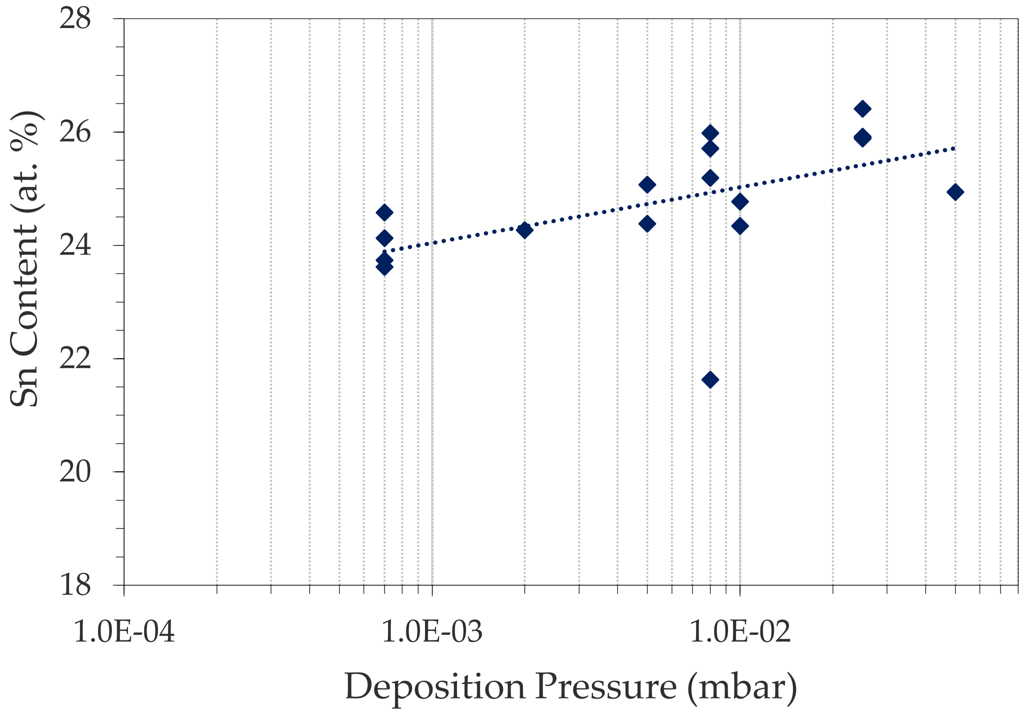




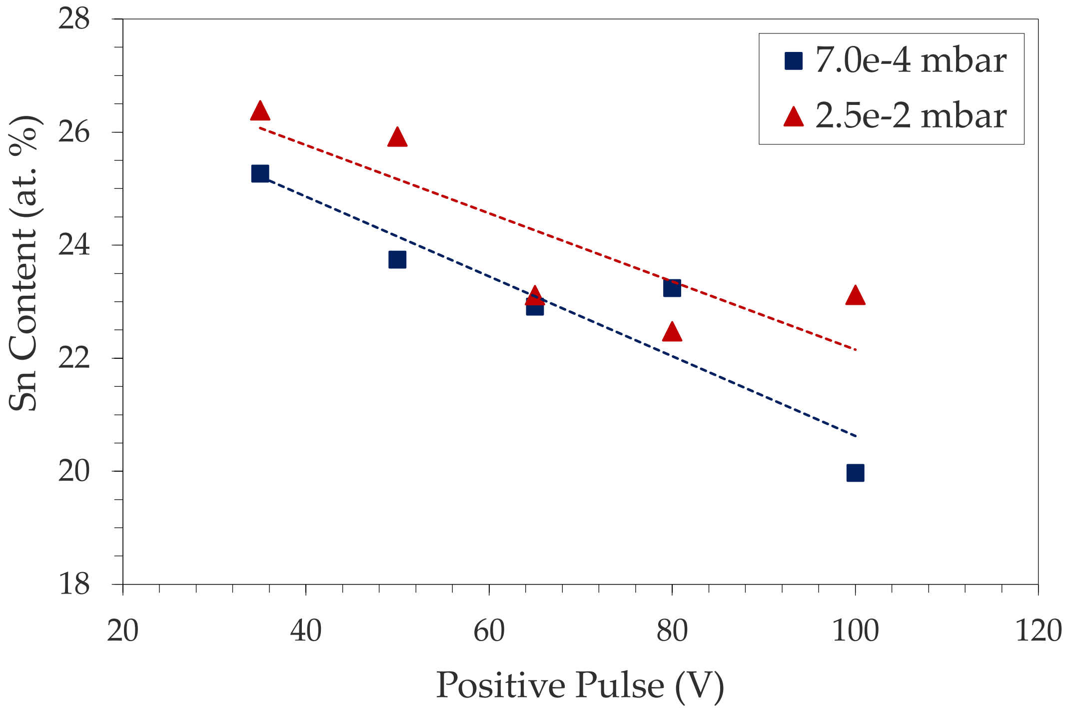


Compositional Analysis

- Sn % = 20 to 26.4 at. % (EDS)
- Coating temperature shows no obvious trend
- Post-coating annealing has no effect on Sn %



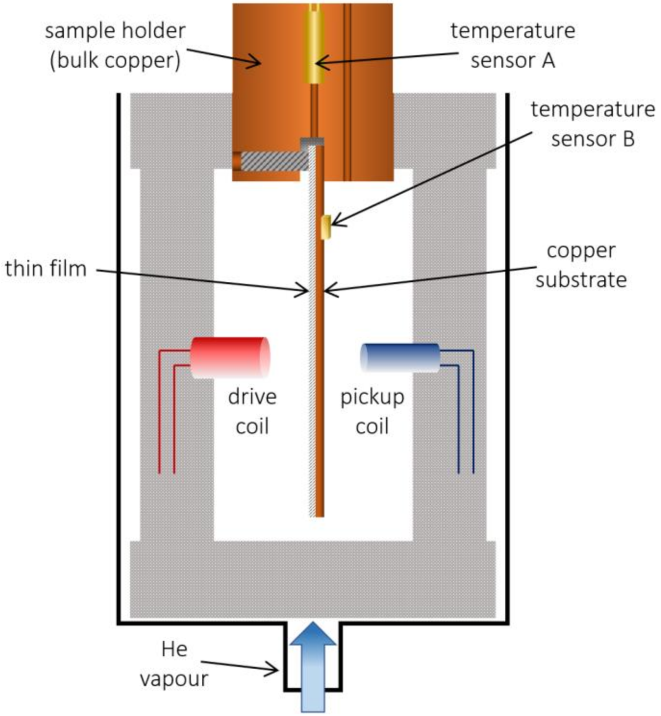
Resputtering or Nb/Sn ion ratio

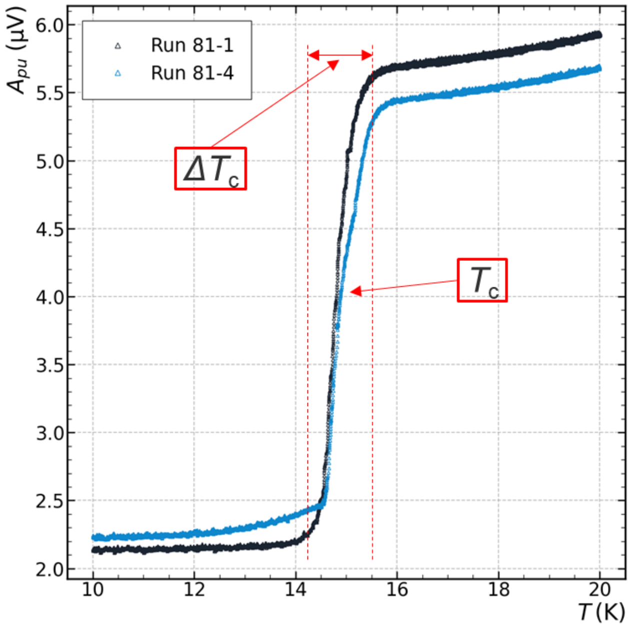


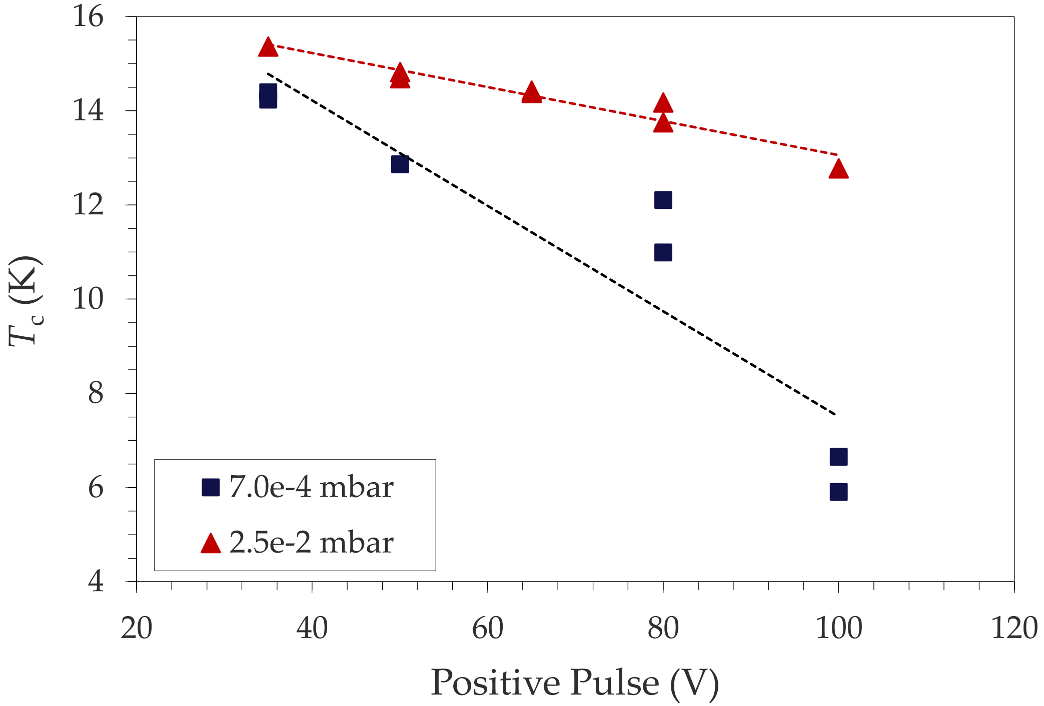
Superconducting Performance

Inductive T_c measurement

- We want high T_c and low ΔT_c







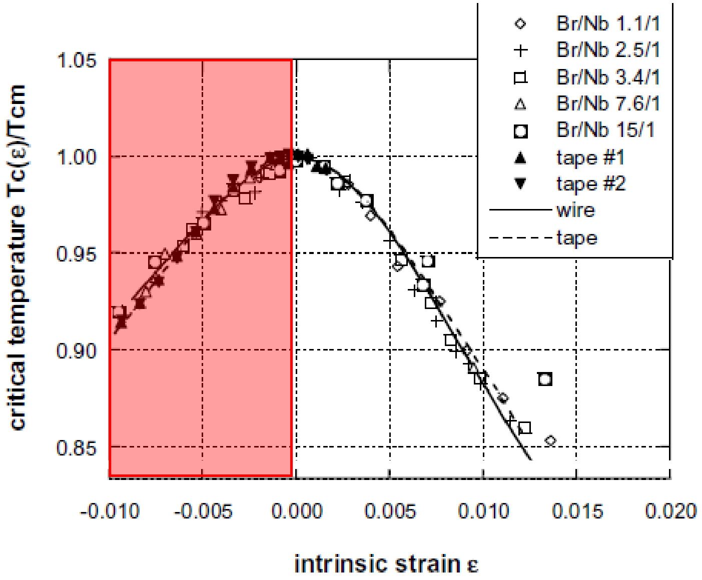
[6] D. Fonnesu - PhD Thesis (to be published)

Superconducting Performance

Nb_3Sn T_c suppression

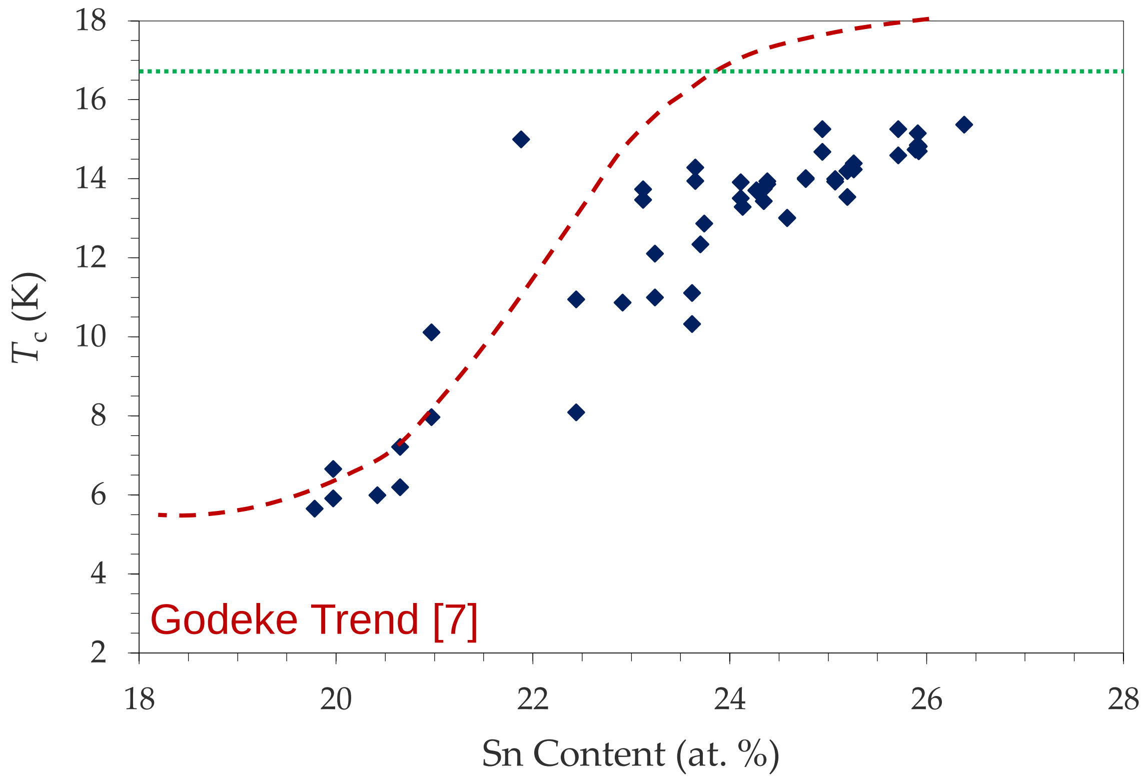
- Sn content in required range
- Residual stress
  - Any stress reduces T_c with Nb_3Sn
  - σ_{Ave} ($2.5 \cdot 10^{-2}$ mbar) = 1.5 Gpa
- Further issues involved

Compressive residual stress



[8]

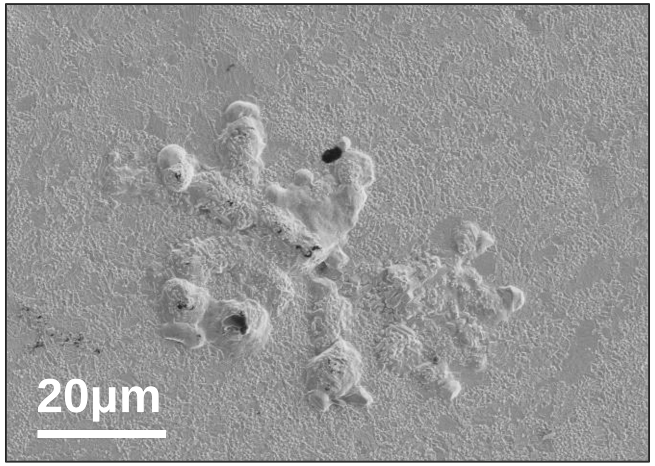
Theoretical Max T_c @ 1.5 GPa

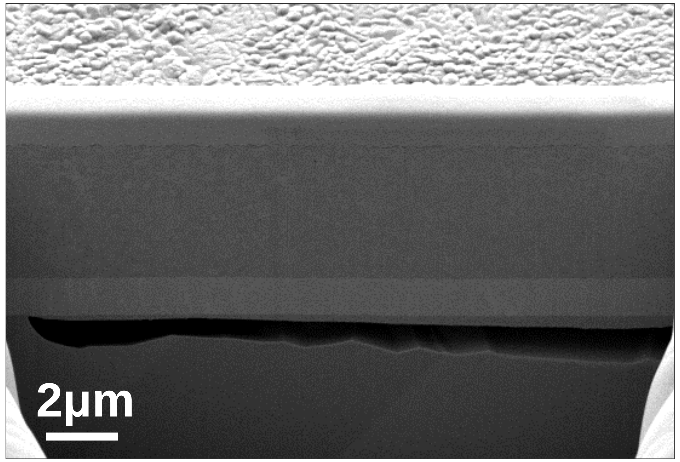


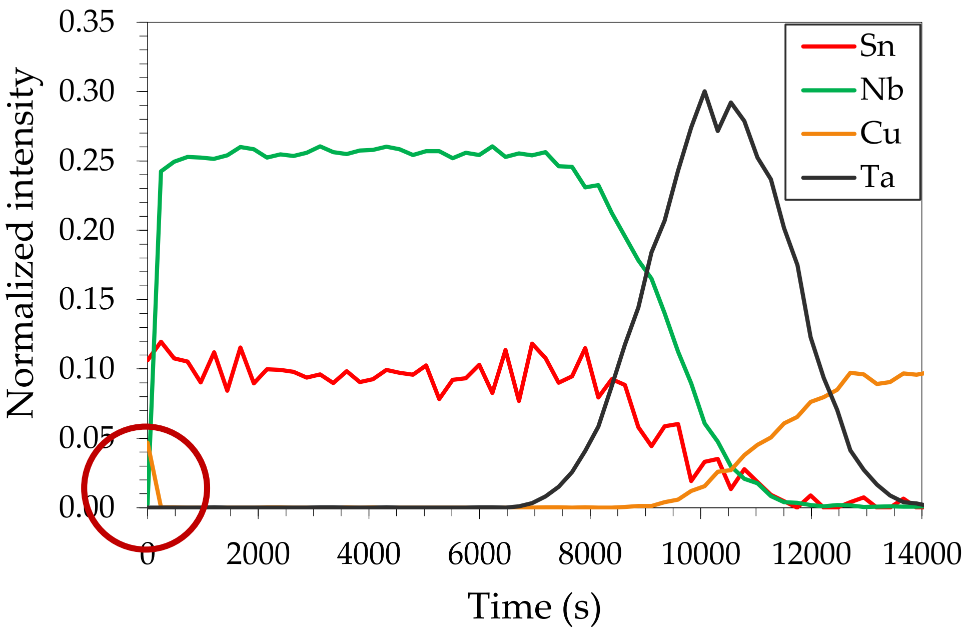
[7] A. Godeke. *Supercond. Sci. Technol.* **19** (2006)
[8] G. De Marzi et al. *J. Phys.: Condens. Matter* **25** (2013)

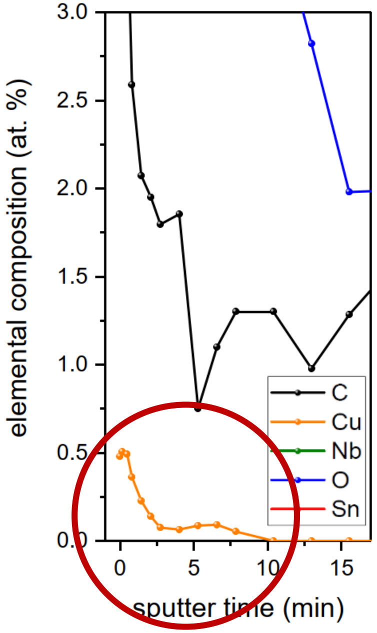
Surface Cu Contamination

- Not always visible in SEM
- Often undetected by EDS – **SIMS / XPS**
- Localised to film surface









Removal of Cu contamination

- Diffusion length
  - Increased Nb_3Sn film thickness

t = 6 µm vs. 3 µm ➡

No

- T_c ~ 16 K (1 K increase)
- Cu surface contamination

- Diffusion dictated by temperature and time
  - Decreased coating temperature

T_{coat} = 500°C ➡

No

- T_c ~ 10 K
- Too low for A15 formation

- Surface Chemistry
  - Ammonium persulfate rinse (15 min) - masked

Cu at. %	Pre Rinse	Post Rinse
Sample 1	3.9	0.6
Sample 2	5.3	1.6

- Still detectable
- How deep?

➡

Maybe

- XPS depth profile

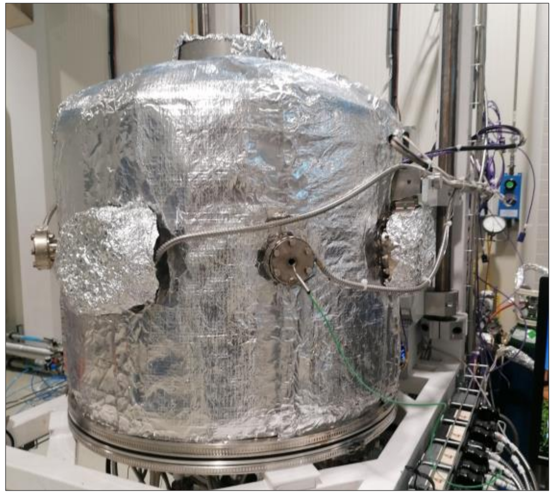
Post coating reaction to optimise A15 phase formation and minimise Cu diffusion

Bipolar HiPIMS Nb_3Sn/Ta/Cu

Reacted After Coating

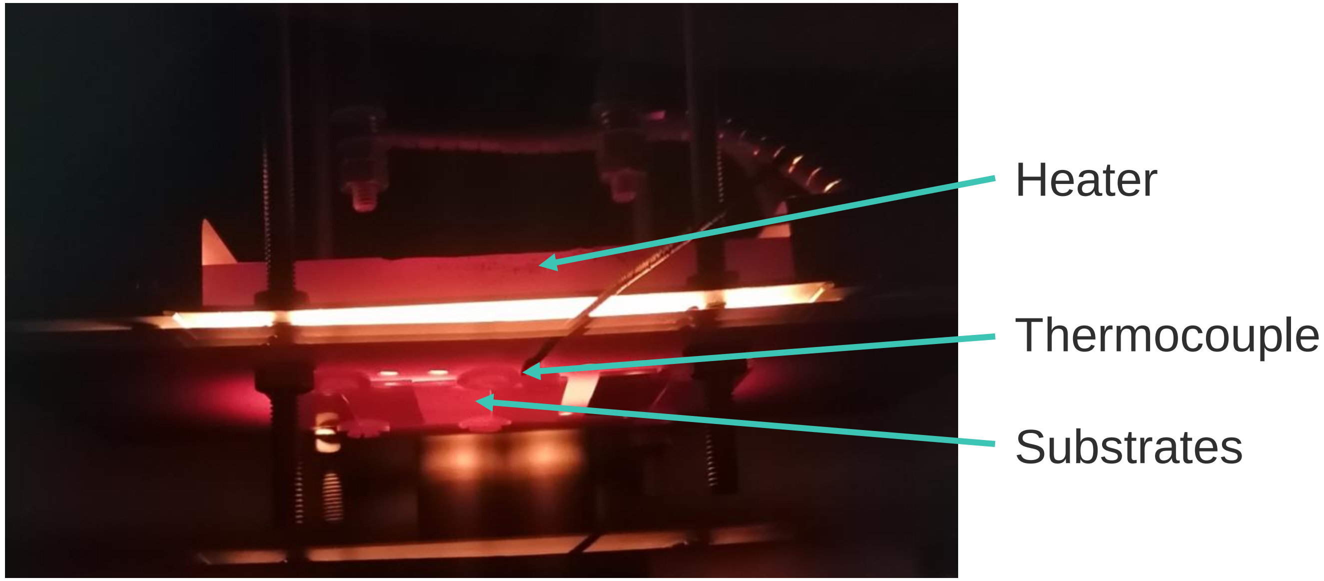
- Low temperature coatings (< 250°C)
  - Plasma self-heating
- High pressure ($2.5 \cdot 10^{-2}$ mbar)
- Low bias (50 V)

Ex-situ (vacuum furnace)



In-situ (coating system)

Annealing pressures	$5 \cdot 10^{-6}$ - $1 \cdot 10^{-7}$ mbar (ex-situ) $2.5 \cdot 10^{-2}$ mbar (Kr) (in-situ)
Annealing temperatures	450 - 750°C (1 - 24 hrs)

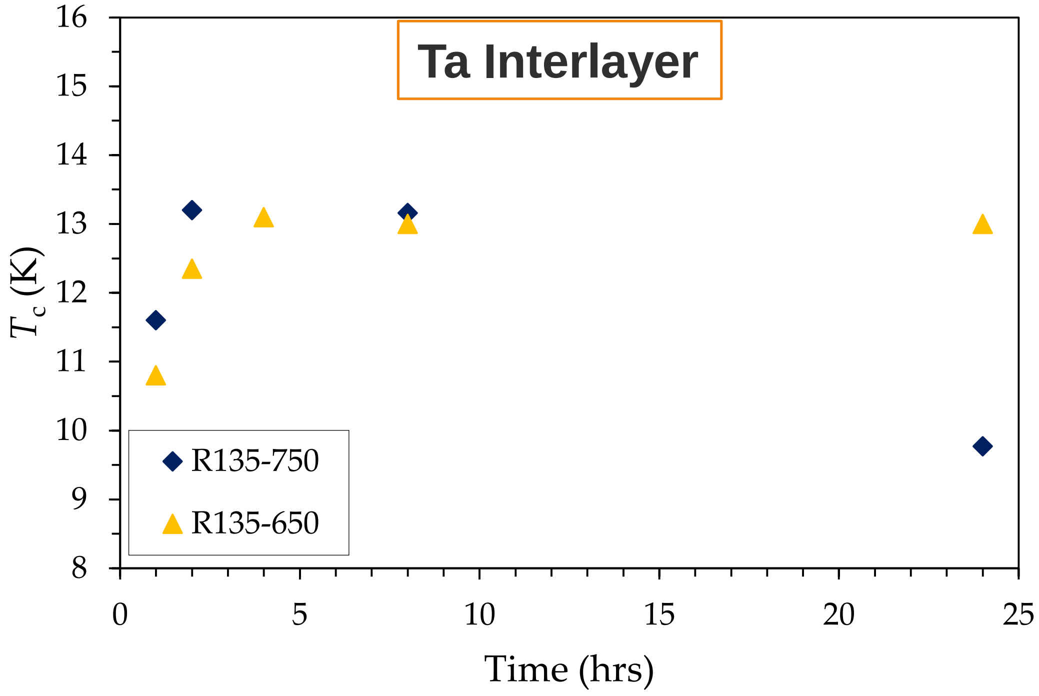


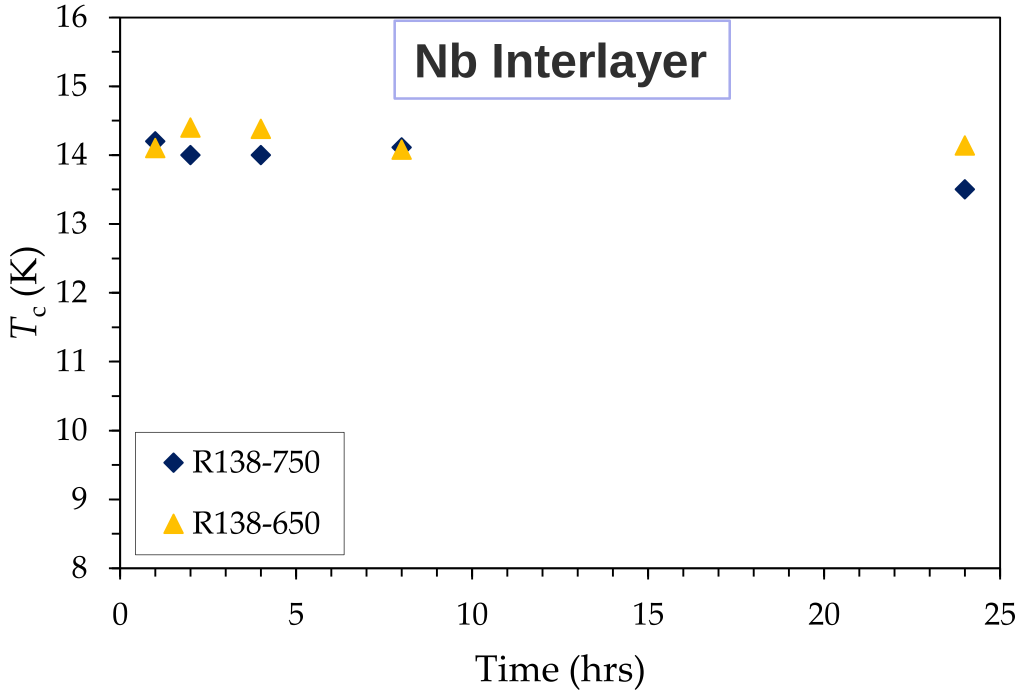
- Ex-situ coatings require air exposure
  - Oxygen leads to smaller crystallites
- In-situ completed in Kr atmosphere

Reacted **After** Coating

Ex-situ

- T_c < 10 K for T_{anneal} = 450/550°C (24 hrs)
    - Focus on 650/750°C
- Discrepancy between Ta and Nb interlayers





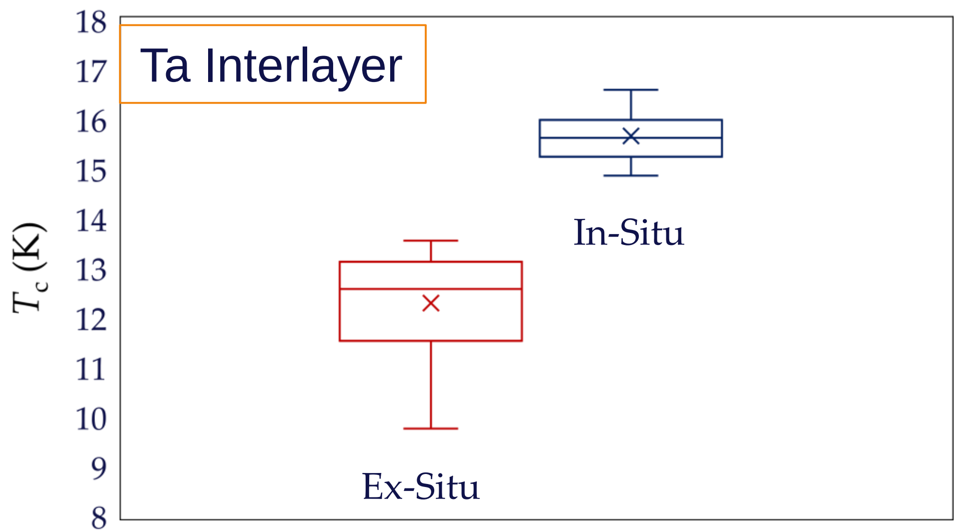
Reacted **After** Coating

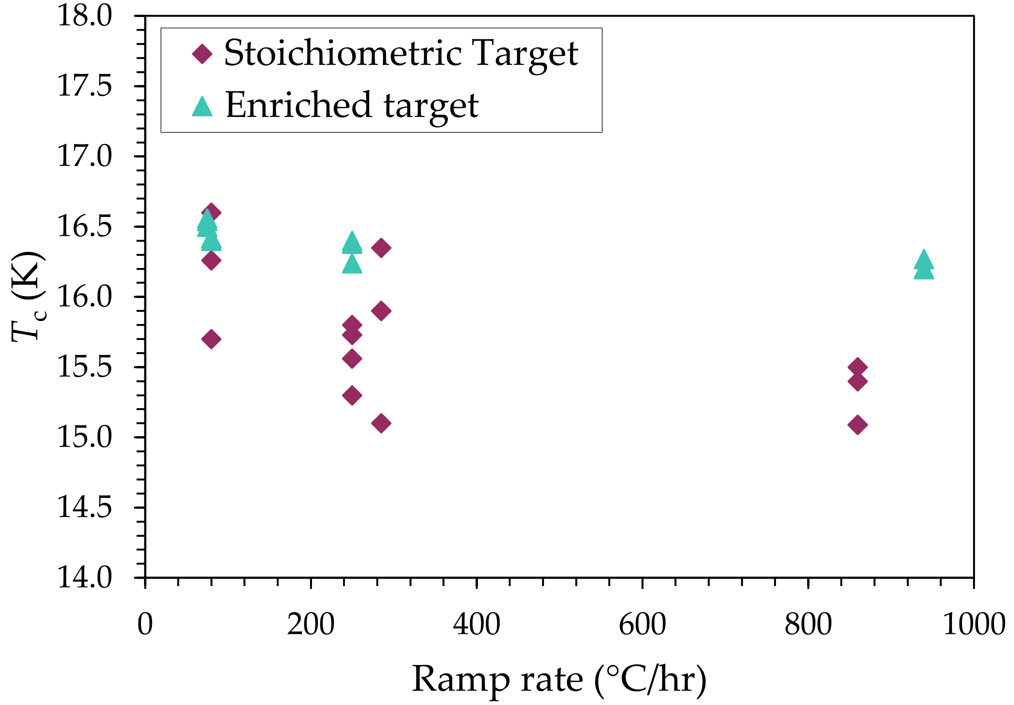
In-situ

- Higher T_c than ex-situ (Stoichiometric target)
  - Highest T_c = 16.6 K
  - Effect of air exposure?
  - Sn P_v = 1.91·10^{-7} mbar

- Ramp rate dependency
  - Similar to Nb_3Sn magnets

- Two targets (Ta interlayer initially)
  - Stoichiometric (25 % Sn)
  - Enriched (27% Sn)
  - Enriched target reduces T_c spread



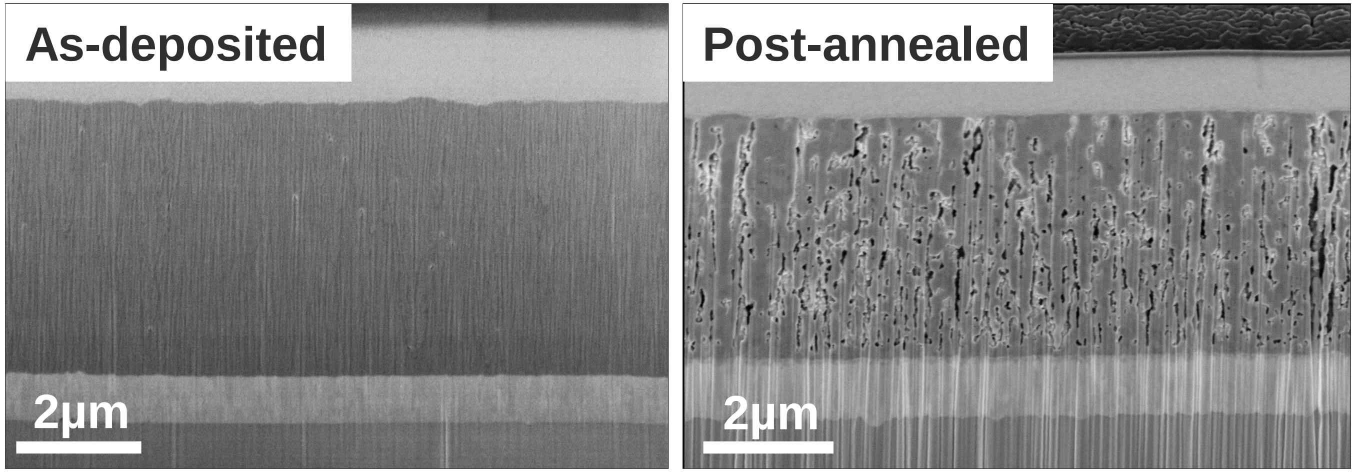


Reacted After Coating

In-situ

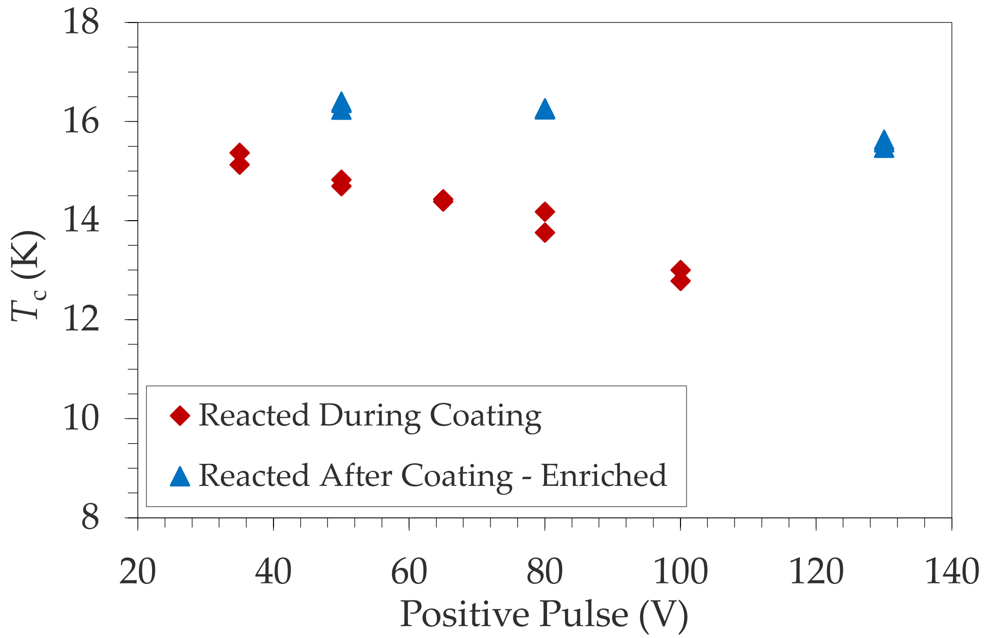
- Porous film post-anneal with both targets
- Increased Cu surface contamination with enriched target
- Instances of delamination

Stoichiometric Target



Solutions?

- Increase positive pulse
  - Reduced decrease in T_c
  - Cu contamination still high
- Ongoing
  - Decrease annealing temp/time
  - Deposition parameter investigation



Summary

- Reacted After Coating shows consistently higher T_c
  - Similar to DC MS observations
  - T_c pushed to 16.6 K
  - Enriched target provides better film performance

- Surface Cu contamination still an issue
  - Effect on RF still outstanding

Future Work

- Process optimisation required
  - Deposition parameters to be explored to densify film
  - Further enriched target (30%) to be attempted

- Deposition of further QPR samples required
  - Before and after ammonium persulfate

Thank you for your attention

Questions?

Big thanks to:

C.P.A. Carlos, G. Rosaz, P. Garritty, M. Watkins, M. Himmerlich, M. Taborelli, S. Forel (TE-VSC)

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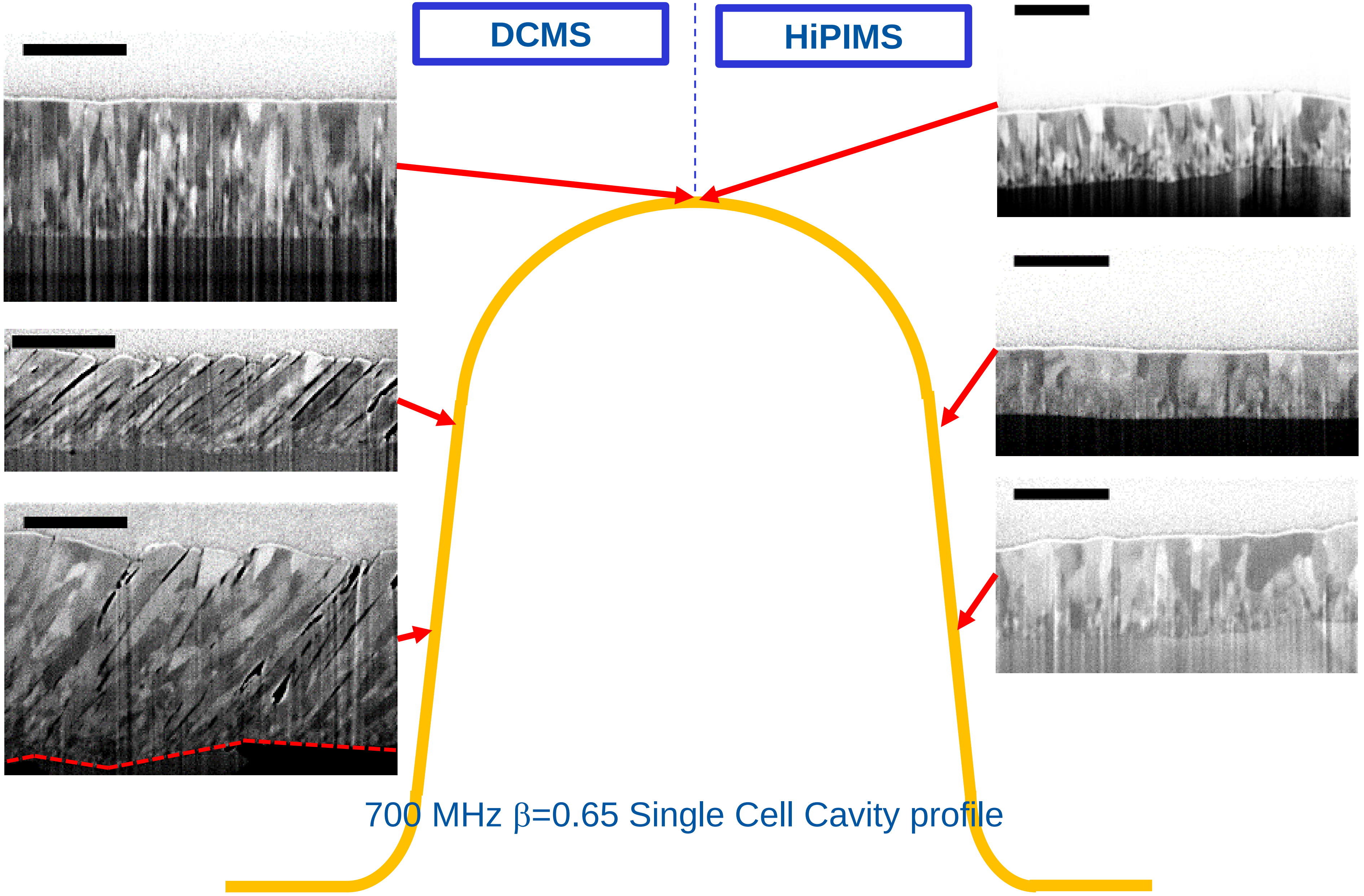
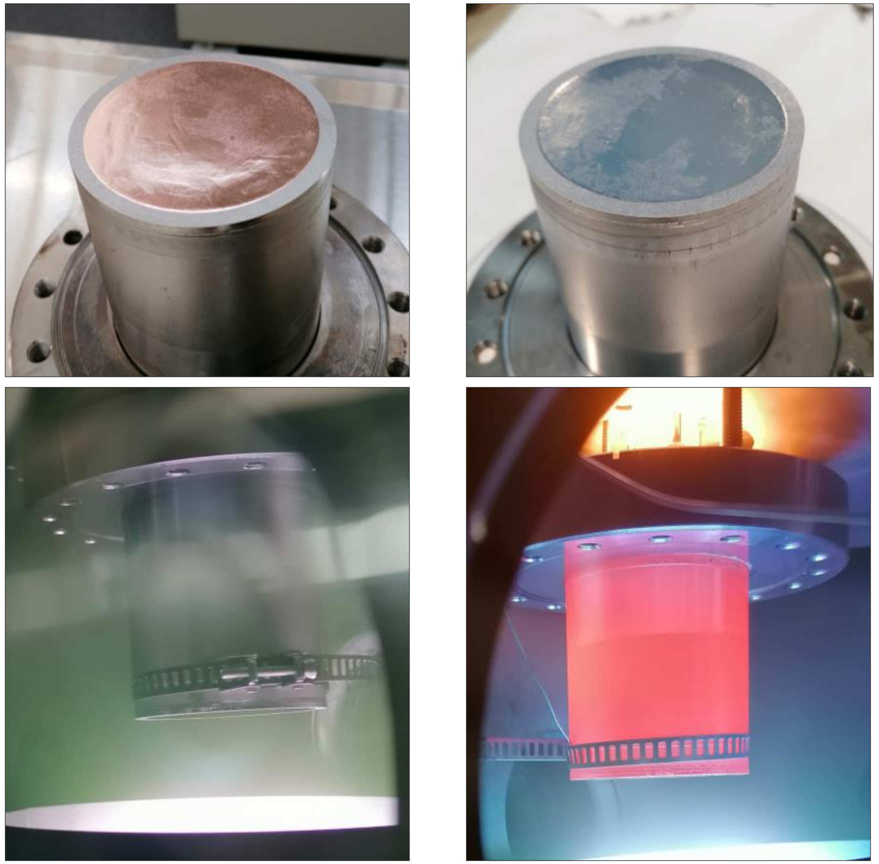
L. Vega-Cid, A. Bianchi, G. Pechaud, W. Venturini-Delsolaro (SY-RF)

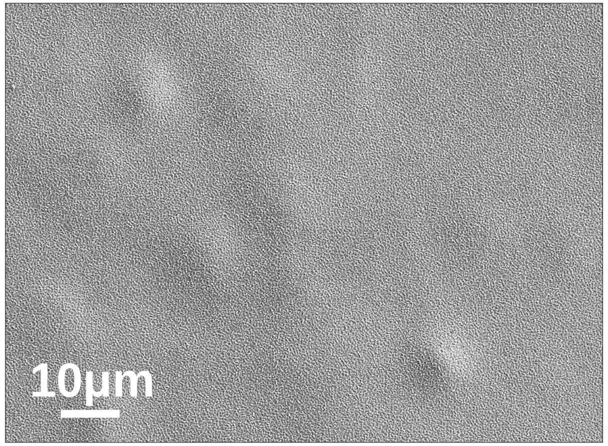
Back up slides

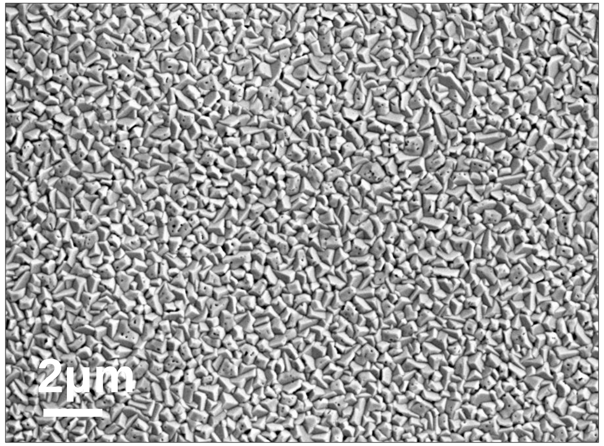

DCMS
HiPIMS
700 MHz β=0.65 Single Cell Cavity profile

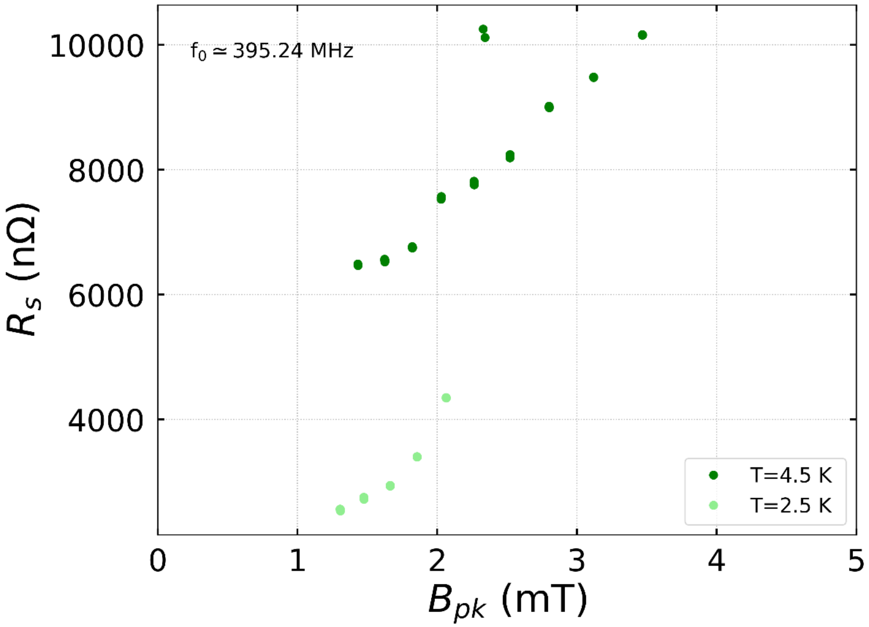
QPR Coatings

QPR Coating for RF measurements
- Sn % = 26.08 ± 0.21 %
- T_c ~ 16 K









Courtesy: L. Vega-Cid