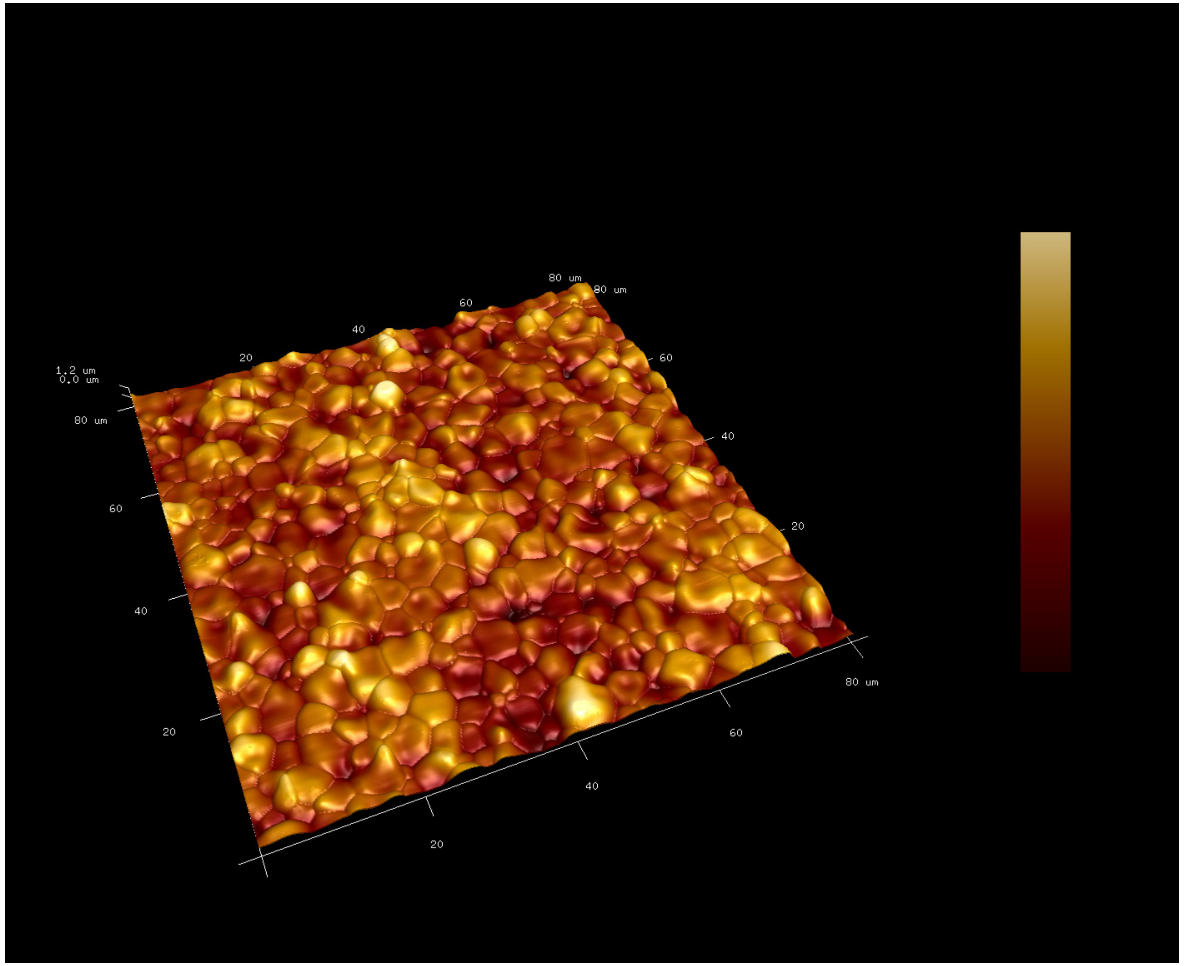
Surface Roughness Reduction and Performance of Nb_3Sn thin-film for SRF Applications



U. Pudasaini, C. E. Reece, E. Lechner,
M. J. Kelley, A.M. Valente-Feliciano, O. Trofimova

Saturday, August 19, 2023

Outline

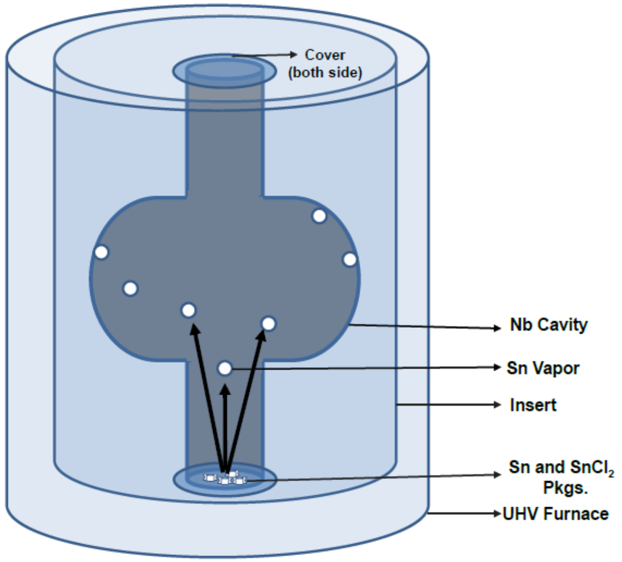
- Nb_3Sn and Sn vapor diffusion technique
- Surface roughness – evolution and characteristics
- Roughness and cavity performance
- Roughness reduction
- Cavity results
- Summary and Outlook

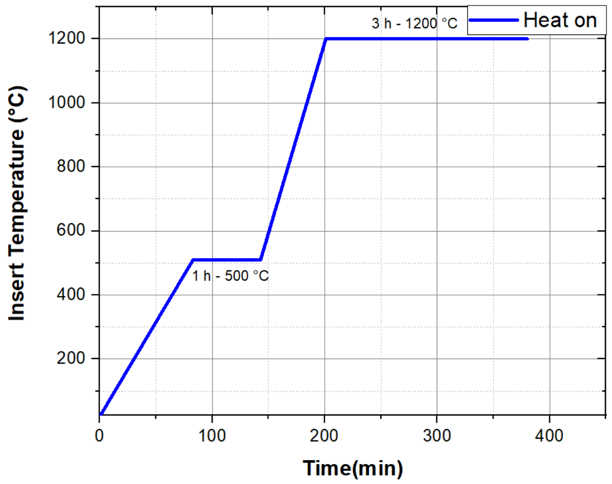
Vapor diffused Nb_3Sn thin film for SRF cavity

- Higher T_c and H_{sh} of Nb_3Sn promise better RF performance (Q_0 and E_{acc}) and/or higher operating temperature (2 K vs 4.3 K)

	Nb	Nb_3Sn
T_c (K)	9.25	**18.3**
H_{sh} (mT)	220	**425**
Q^{BCS} at 2 K	5×10^{10}	**5×10^{14}**
Q^{BCS} at 4 K	5×10^{8}	**5×10^{10}**
E_{acc} (MV/m)	50	100

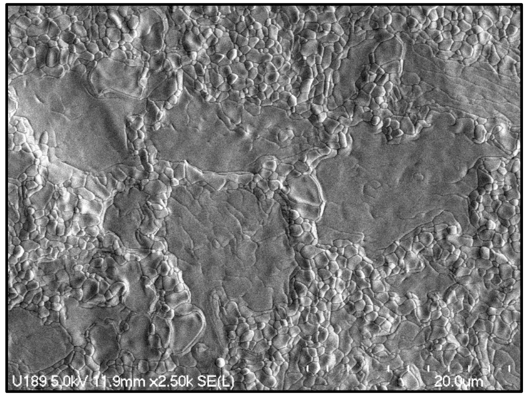
- Vapor diffusion Technique
  - Create Sn vapor, transport it to the interior surface a Nb cavity, and provide suitable temperature environment to grow Nb_3Sn on Nb.

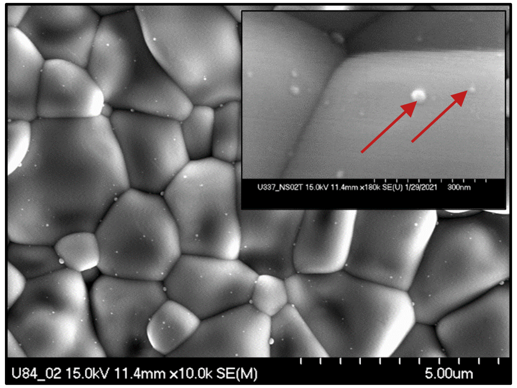




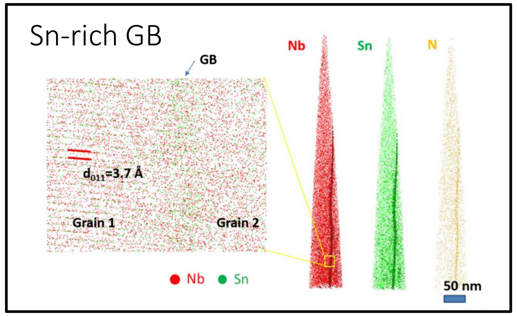
Performance limiting factors

- A few known issues that potentially contribute to performance:
  - Non-uniformity and patchy regions (thin large grains)
  - Accumulation of Sn-residues
  - Incorporated impurities
  - Non-stoichiometric grain boundaries
  - Low-Sn regions
  - **Surface topography (Roughness)**

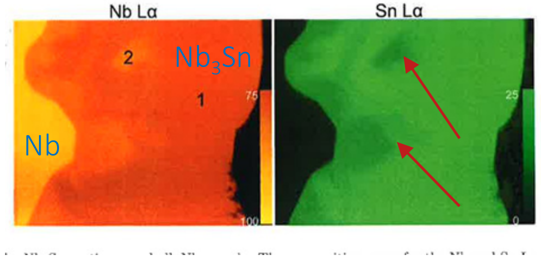




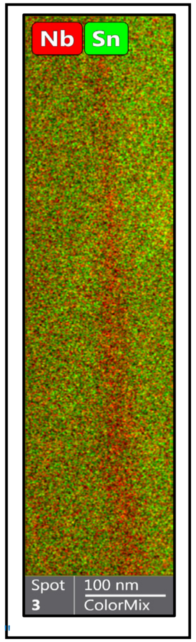
Sn-rich GB



J. Lee, Jaeyel, et al. "Grain-boundary structure and segregation in Nb3Sn coatings on Nb for high-performance superconducting radiofrequency cavity applications." Acta Materialia 188 (2020): 155-165.

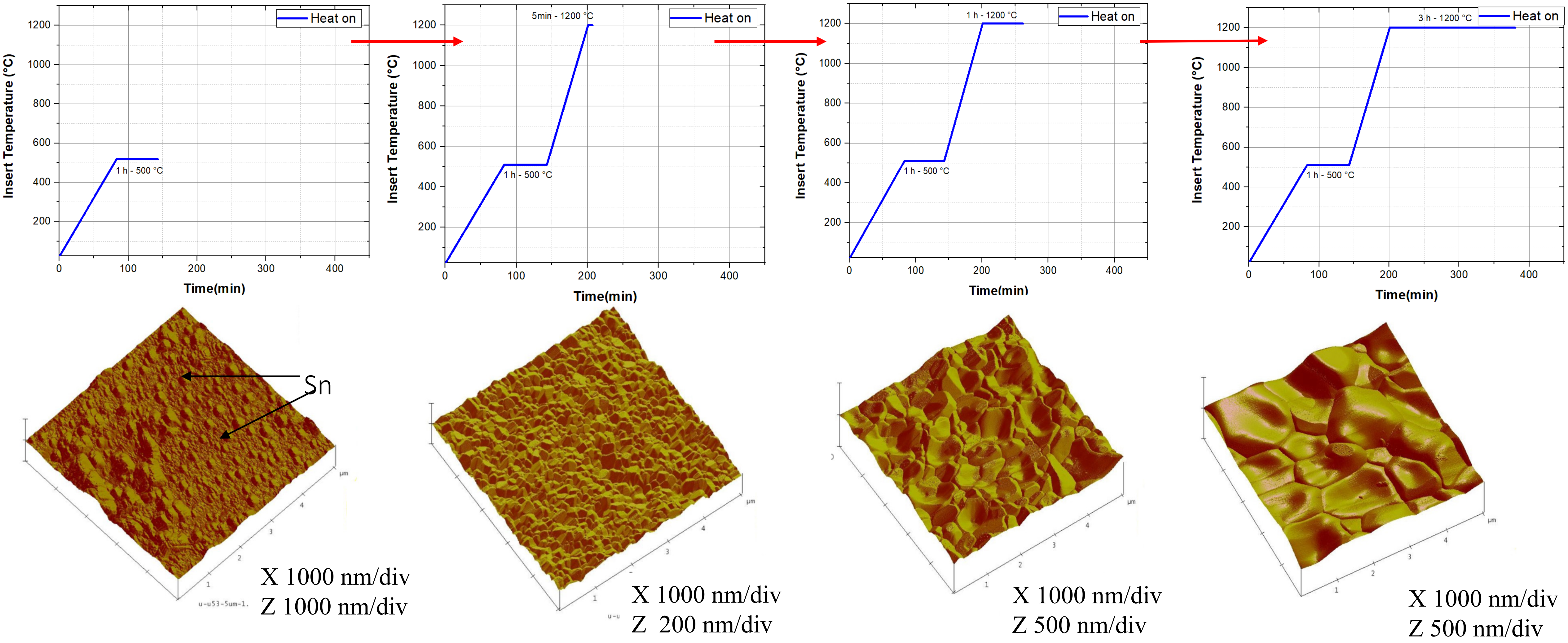


Sn-deficient GB



C. Becker et. al . " Analysis of Nb3Sn surface layers for superconducting radio frequency cavity applications Appl. Phys. Lett. 106, 082602 (2015)
D. Hall "New insights into the limitations on the efficiency and achievable gradients in Nb3Sn SRF cavities" Dissertation. Cornell University 2017.

Roughness evolution during thin film growth



Ra = 54.8 ± 9.4 nm

Ra = 7.3 ± 0.6 nm

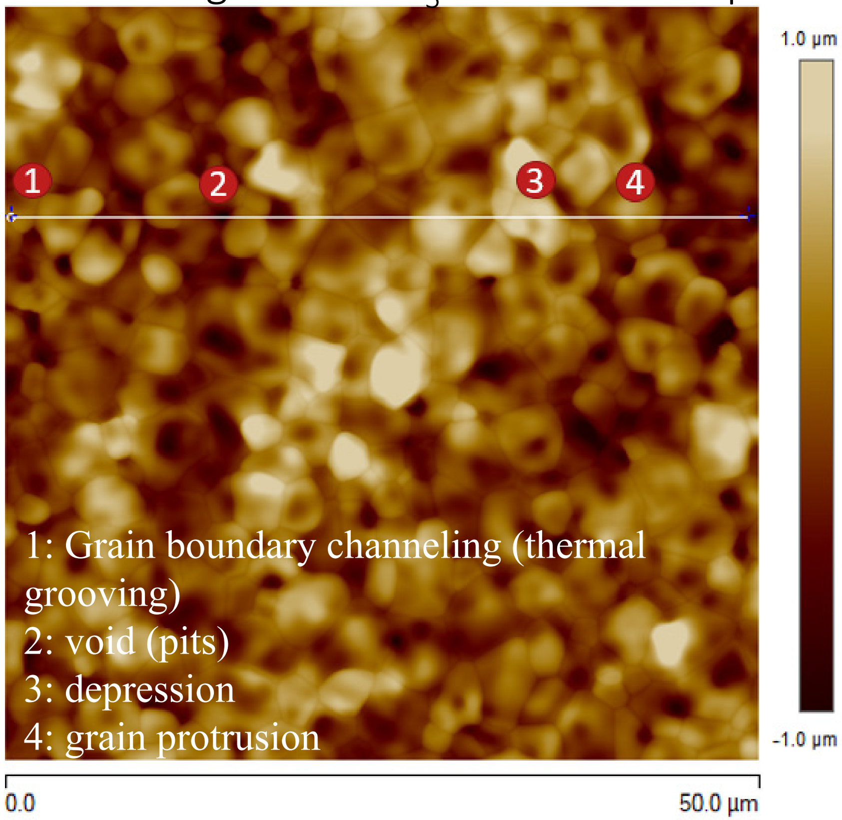
Ra = 23.6 ± 4.6 nm

Ra = 61.8 ± 4.6 nm

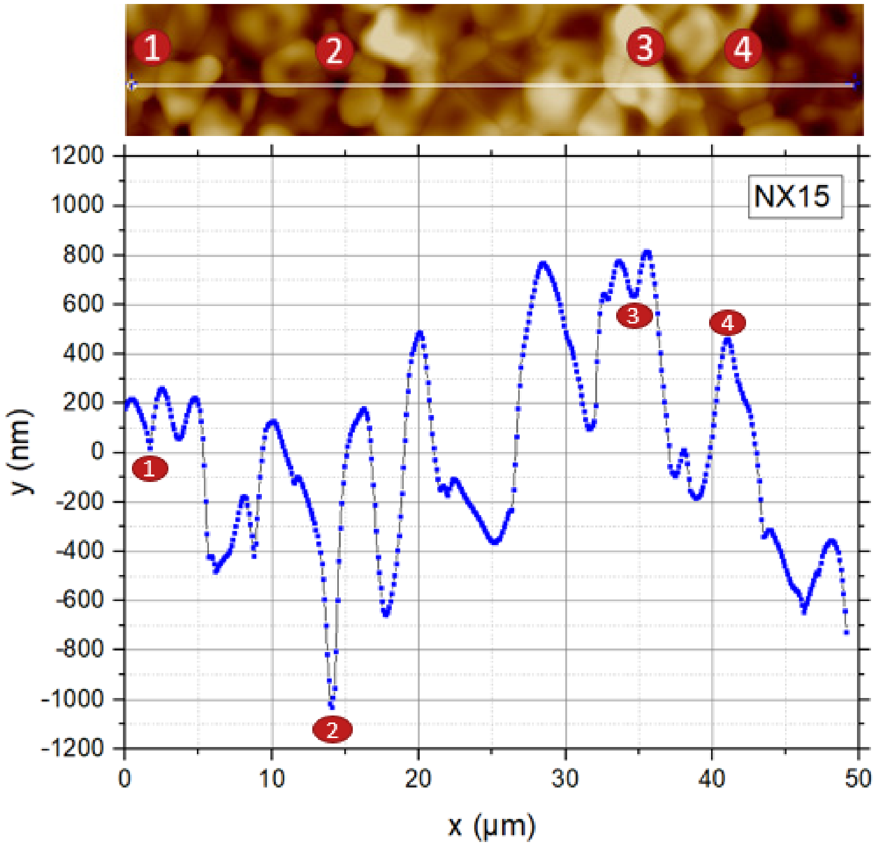
Pudasaini, U., et al. Local composition and topography of Nb3Sn diffusion coatings on niobium. SRF'15, Whistler,Canada

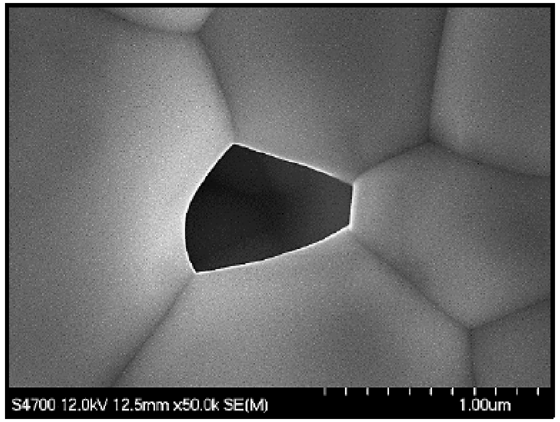
Roughness in vapor diffused Nb_3Sn

AFM image from Nb_3Sn-coated sample

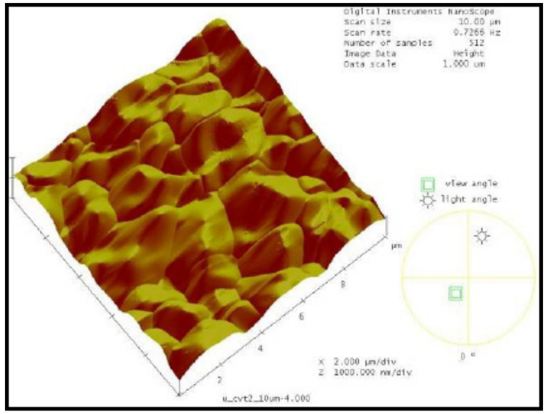


Sectional height profile along the white line





Nb_3Sn Cavity cutout sample



- Magnetic field enhancement?
- "1% of surface analyzed has field enhancement by more than 45%." - R. Porter et. al "Surface Roughness Effect on the Performance of Nb3Sn" LINAC 2016, MSU,MI)

Pudasaini, U., Eremeev, G.V., Reece, C.E., Tuggle, J. and Kelley, M.J., 2020. Analysis of RF losses and material characterization of samples removed from a Nb3Sn-coated superconducting RF cavity. *Superconductor Science and Technology*, *33*(4), p.045012.

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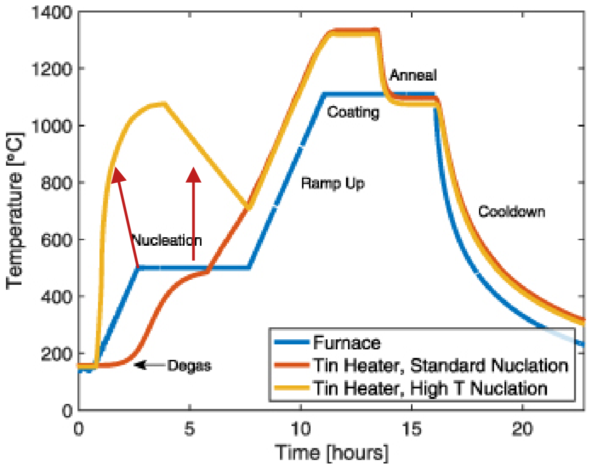
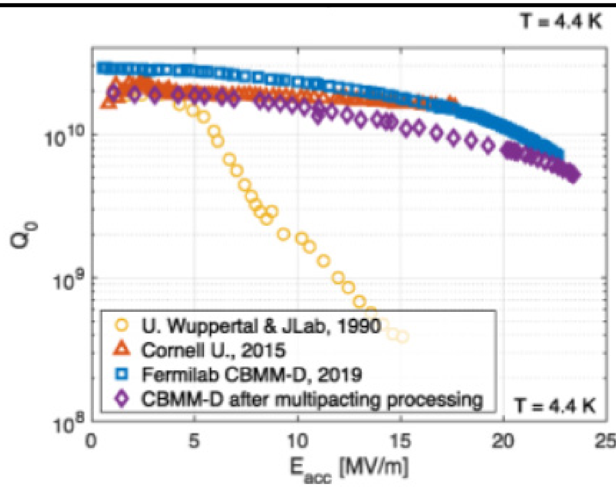
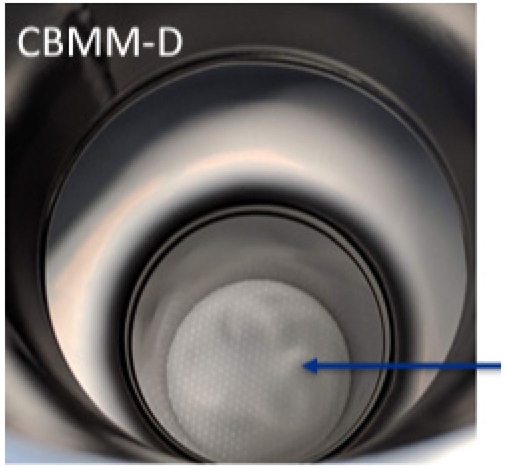
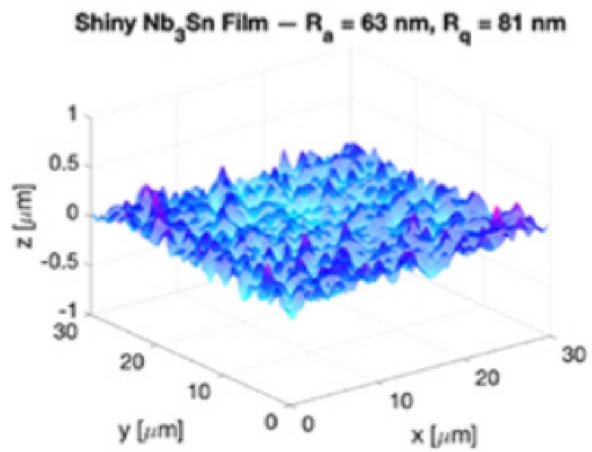
Roughness and Cavity Performance



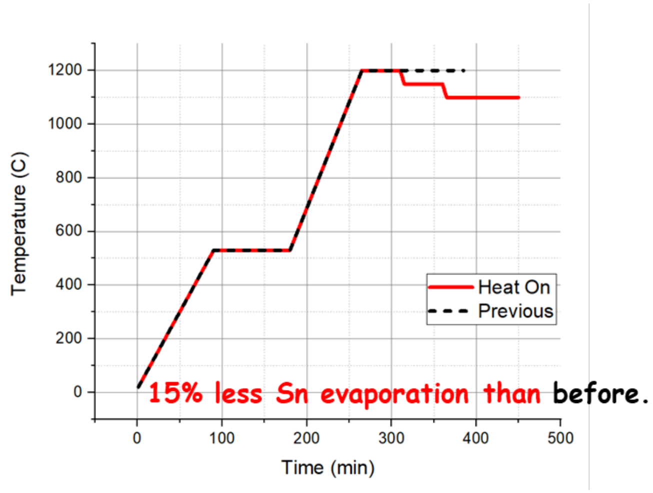






'High-temperature' nucleation

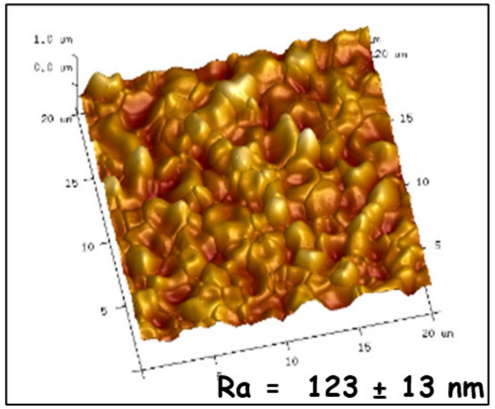
S Posen *et. al.* 2021 *Supercond. Sci. Technol.* **34** 025007

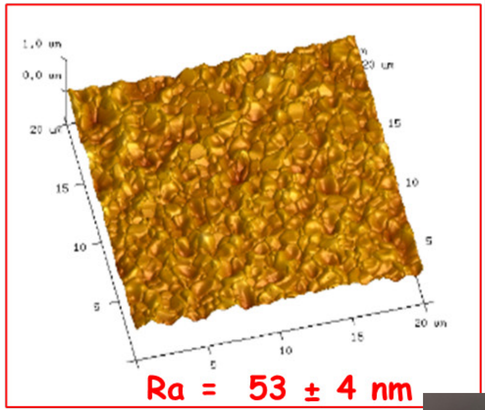


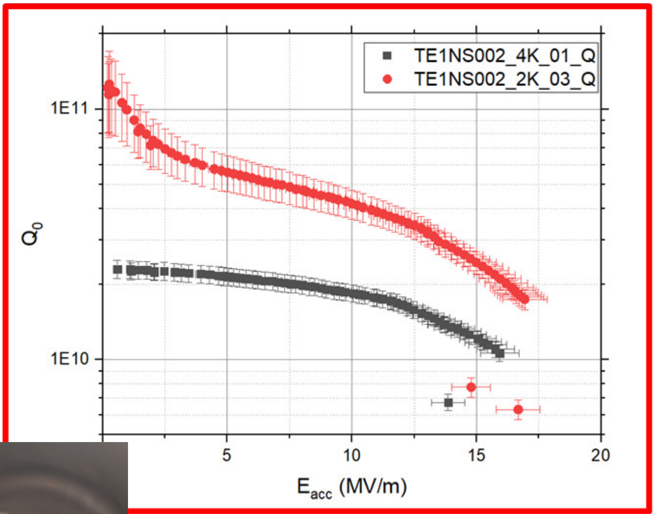
Coating thickness: 1.2± 0.12 µm

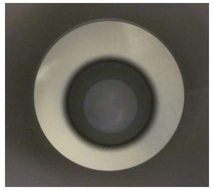
Grain size: 0.96 ± 0.06 µm

20μm × 20 μm AFM Scan









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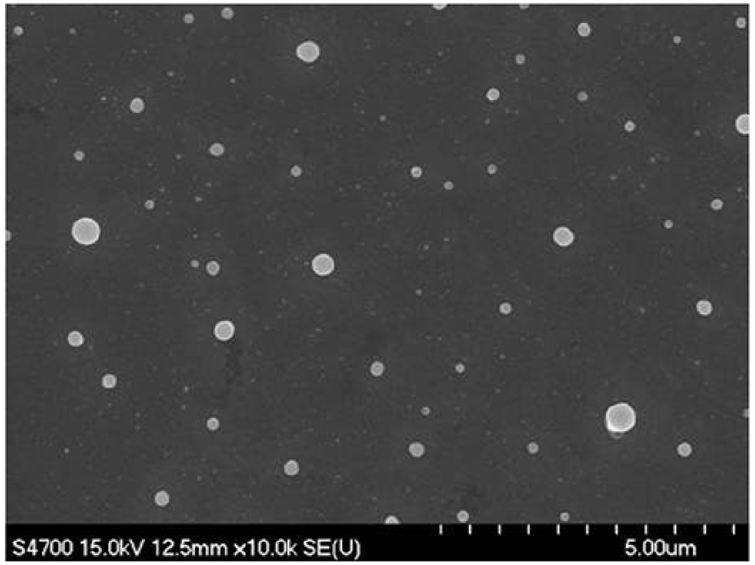
Roughness reduction

- Nucleation enhancement
  - Reduce the distance between nucleation centers ←→ create high-density nucleation sites
  - Nucleation parameter variations – no notable difference in microstructures after the coating
  - Pre-anodization
  - New alternatives
    - High temperature nucleation – need secondary heater (in practice at FNAL)
    - Nucleation enhancement Electrochemical deposition
    - Pre-Sn film deposition
    - Electrochemical thermal deposition (Cornell)
- Tune coating parameters
- Post-coating treatments
  - Mechanical polishing
  - Electrochemical polishing



Nb-surface with typical nucleation

Roughness Reduction
by
Pre-Sn film deposition

Nucleation enhancement with pre-deposition of thin layers of Sn

Sample Preparation

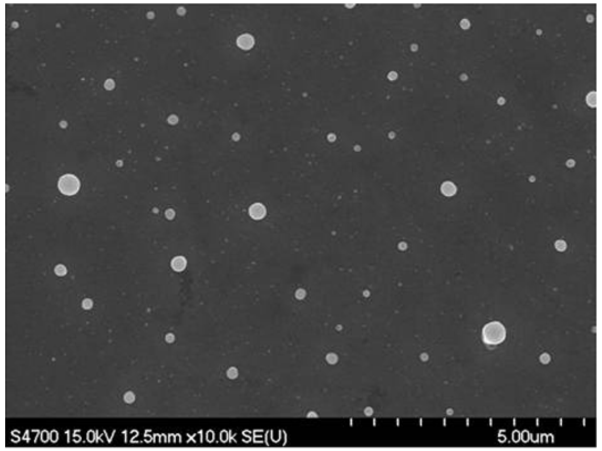
- 10 mm × 10 mm samples cut from the same sheet of material
- Bulk BCP 100 µm + 800°C bake + 20 µm final BCP

10 or 20 nm Sn Deposition

- Magnetron sputtering

Vapor diffusion process

- All samples coated in the same run via vapor diffusion process

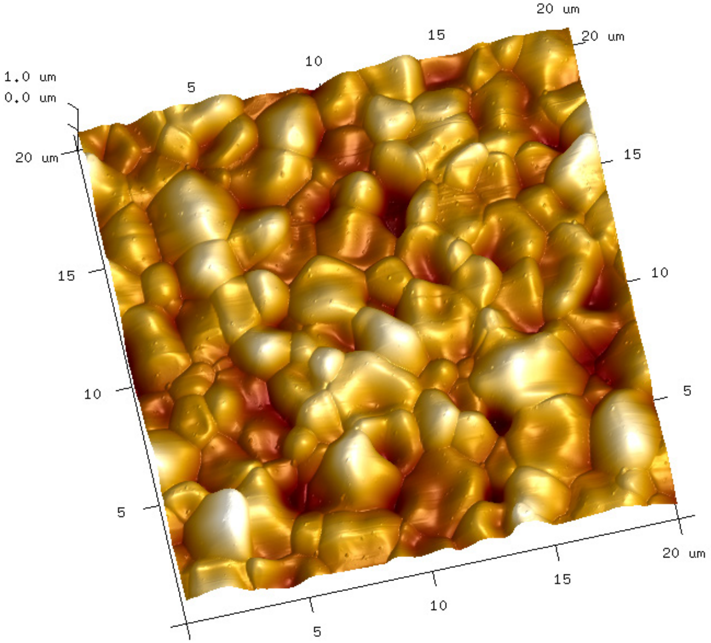


Nb-surface with typical nucleation

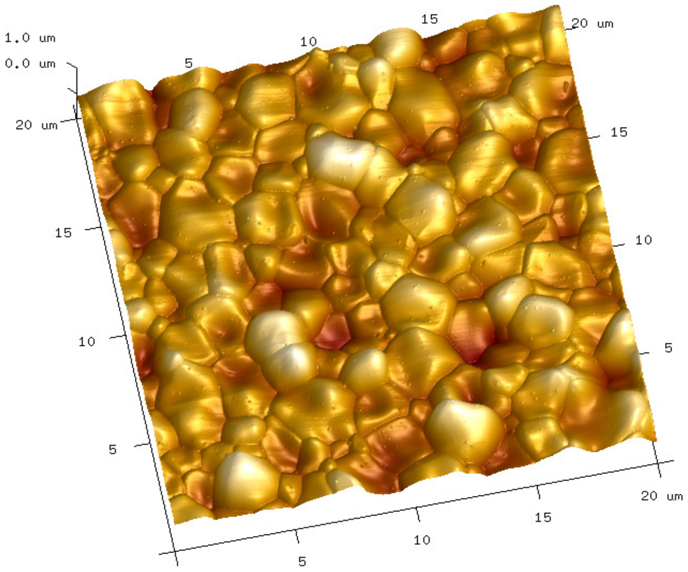
Goal: grow uniform sized dense grains.

Roughness comparison

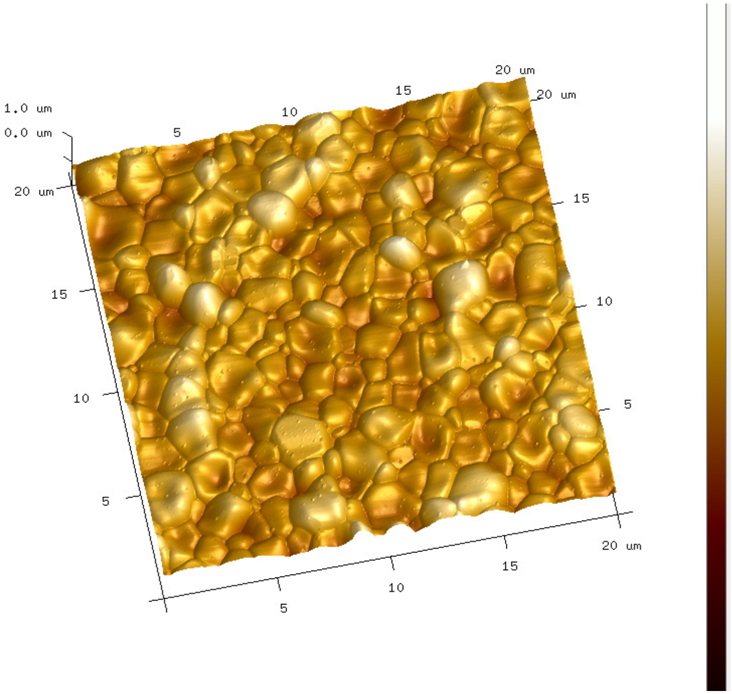
NX11: Nb



X100: 10 nm Sn on Nb



X5: 20 nm Sn on Nb



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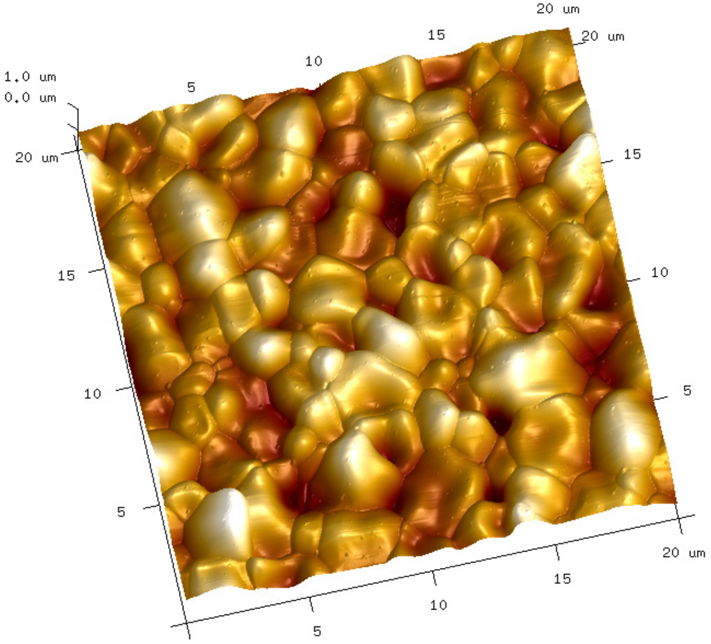
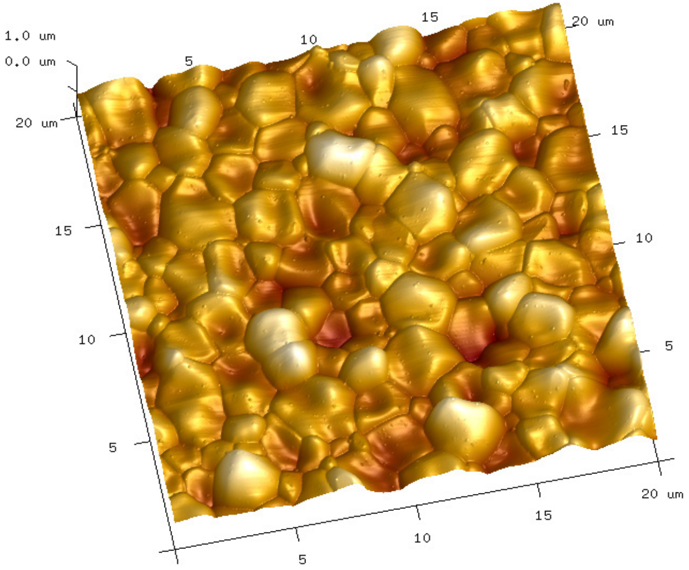
Roughness comparison

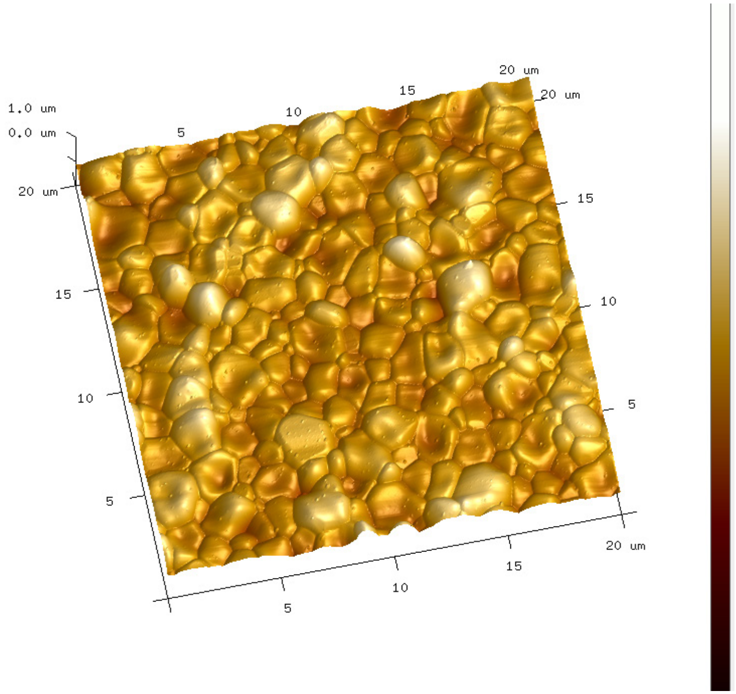
NX11: Nb



X100: 10 nm Sn on Nb



X5: 20 nm Sn on Nb



Samples	Thickness of pre-deposited Sn (nm)	Average grain size (µm)	Average roughness R_a (nm)
NX11	0	1.85 ± 0.14	180 ± 32.4
X100	10	1.52 ± 0.10	125 ± 22.6
X5	20	1.26 ± 0.07	**83 ± 3.1**

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Cornell's Efforts on Mitigating Surface Roughness on Nb_3Sn Coatings

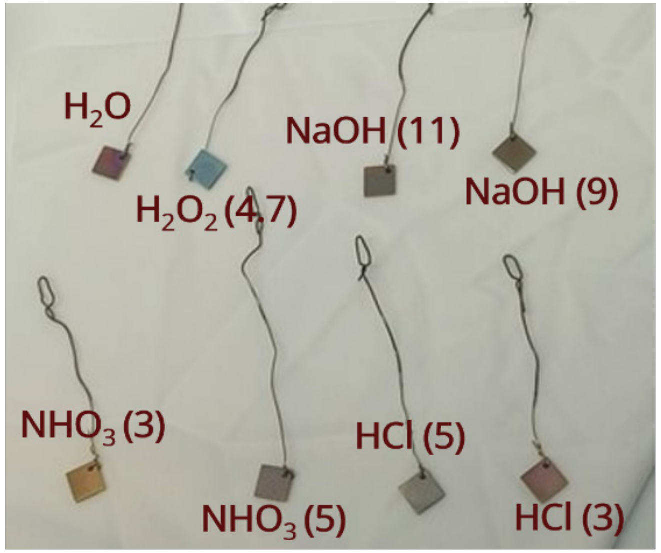
CLASSE
Cornell Laboratory for Accelerator-based ScienceS & Education

Courtesy of L. Shpani and M. Liepe

Thin Film Cavity

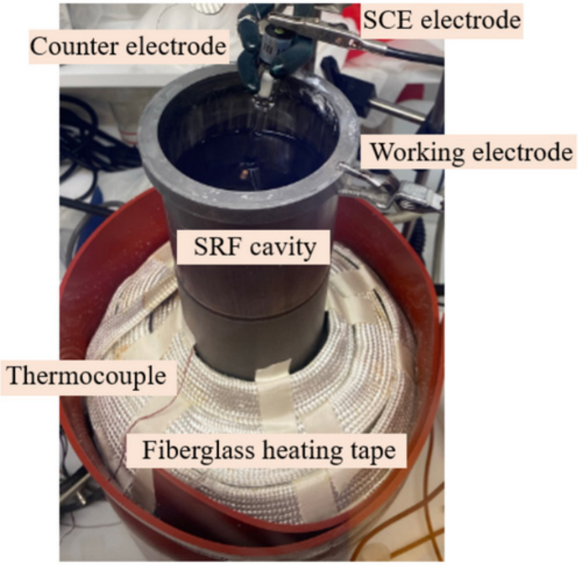
New coating process (inspired by FNAL shiny cavity) which resulted in lower surface roughness.

Nucleation Studies



→ Optimizing the nucleation step of vapor diffusion through surface chemistry treatments to achieve smoother films.
Sample studies have shown that chemical treatments pre-nucleation affect the nucleation of Sn onto Nb.
(SUSPB026)

Electrochemical Deposition

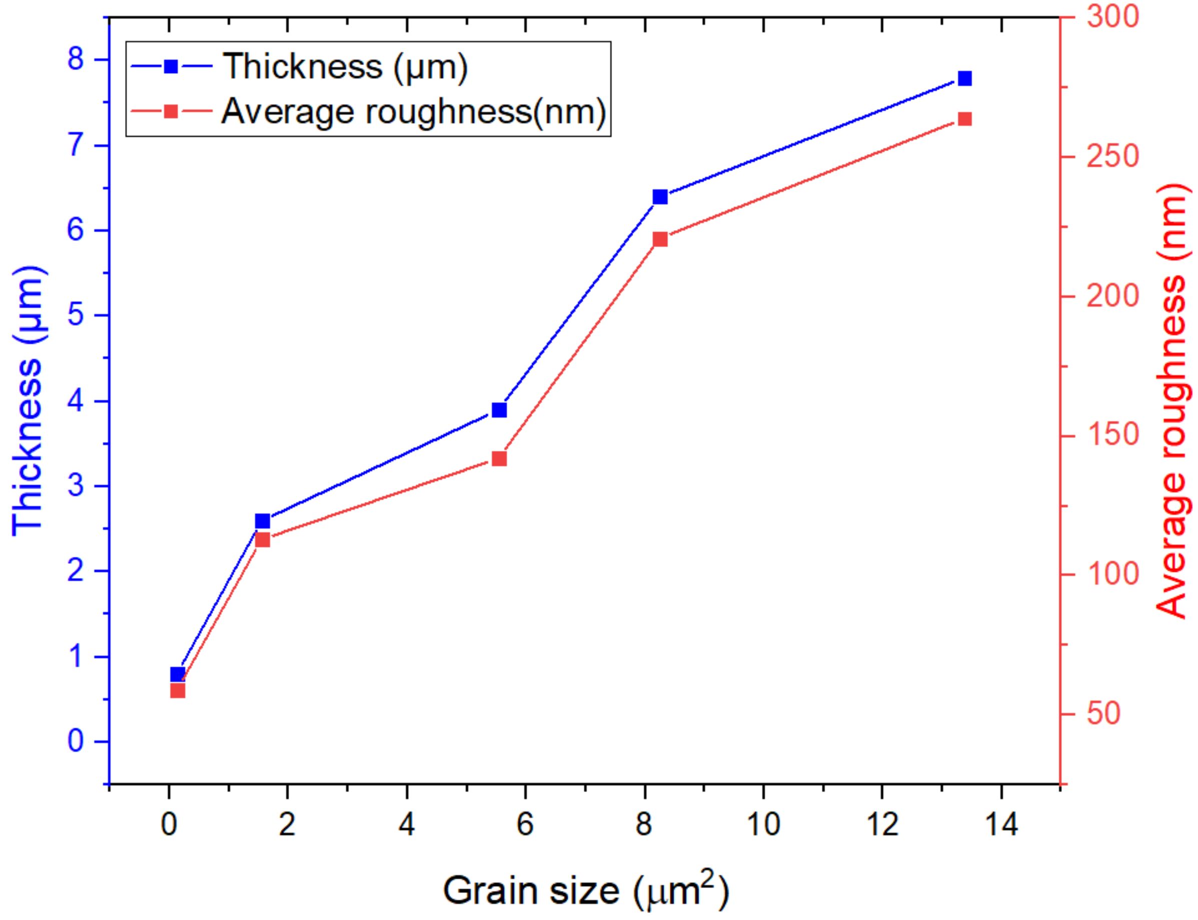


Very promising alternative growth method
→ achieved low surface roughness (~4-5 times lower than vapor diffusion), good stoichiometry, high quality factors and reduced BCS resistance. (TUPTB006) *Sun et al., https://doi.org/10.48550/arXiv.2302.02054*

→ For more details, check out "Development of High-performance Niobium-3 Tin Cavities at Cornell" (WEIAA04)

Roughness Reduction by Tuning Coating Parameters

Grain size, thickness, and Roughness

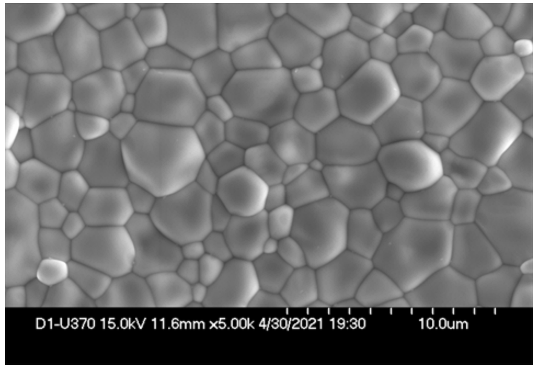


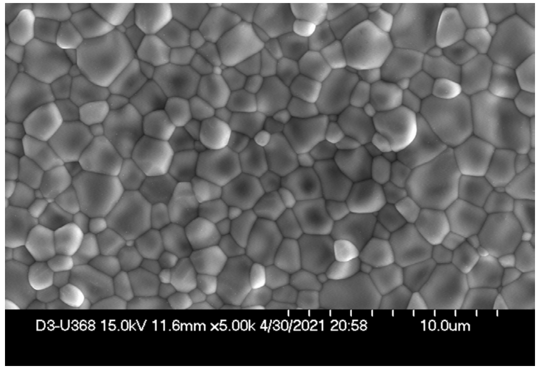
Positive correlation between the Nb_3Sn film growth with increasing grain size and the surface roughness.

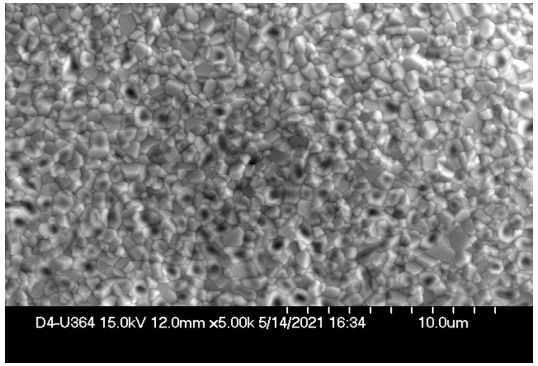
Tuning Sn-flux

Fixed coating surface area & other coating parameters identical

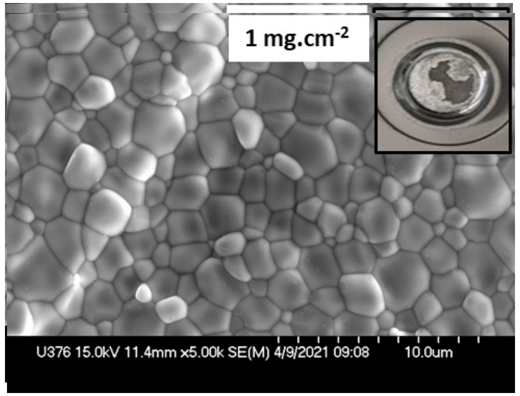
Diameter (inch)	Cross-sectional area (cm^2)	Sn use (g)	Grain size (µm)	Average Roughness Ra (nm)
1.0	5.07	0.81	1.63 ± 0.02	126 ± 1.0
0.5	1.27	0.33	1.21 ± 0.06	95.8± 3.4
0.25	0.32	0.10	0.56 ± 0.02	28.2± 3.3

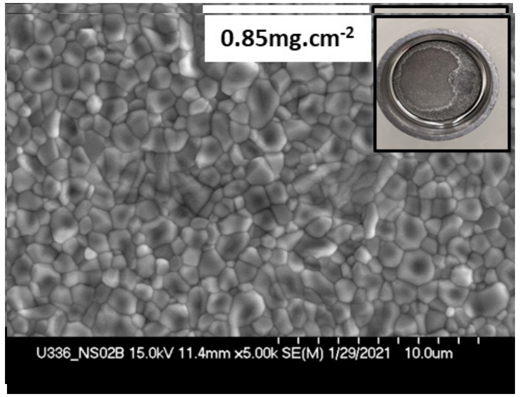






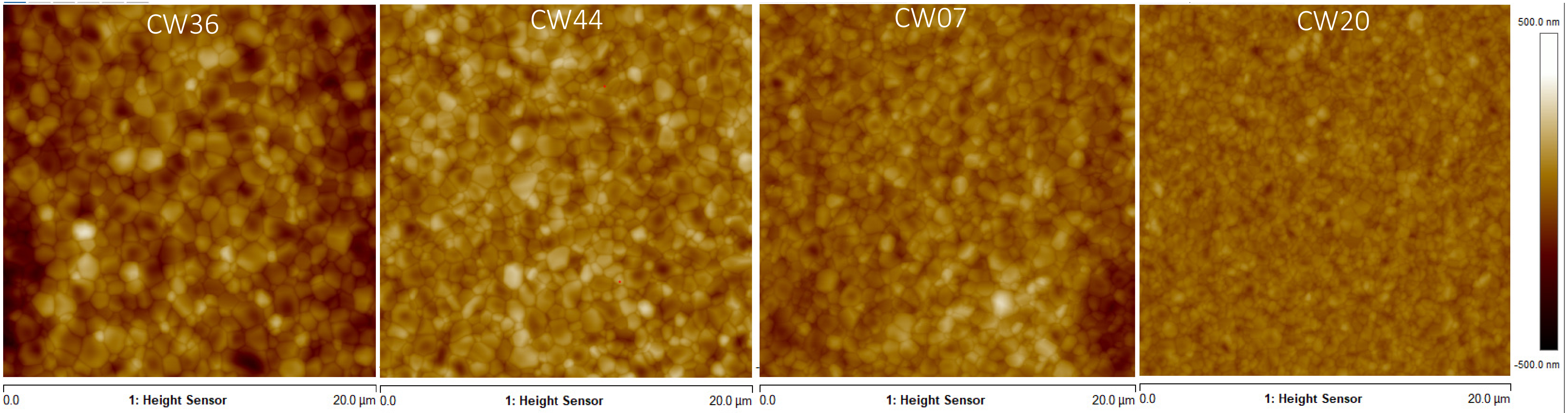
Fixed Sn-source and other coating parameters identical



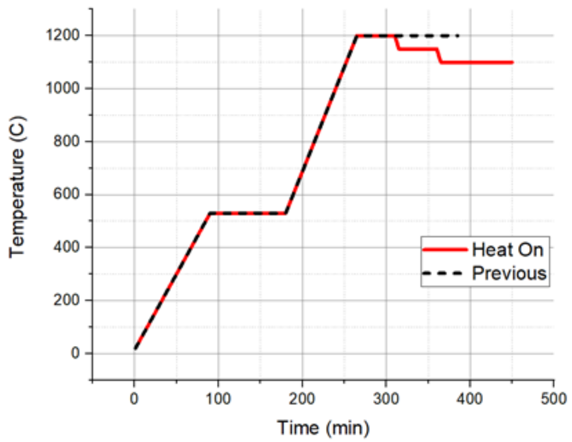


U. Pudasaini, C. E. Reece, and J. K. Tiskumara, "Managing Sn-Supply to Tune Surface Characteristics of Vapor-Diffusion Coating of Nb_3Sn", in ***Proc. SRF'21***, East Lansing, MI, USA

Variation of temperature profile during the coating stage



Expt	1200 °C	1150 °C	1100 °C	Sample	R_a (nm)	Grain size (µm)	Thickness (µm)
01	10 min	45 min	85 min	CW36	69 ± 3	0.93	-
02	10 min	45 min		CW44	40 ± 1	0.75	1.0 +/- 0.2
03	10 min			CW20	27 ± 2	0.55	0.75
04	10 min		85 min	CW07	43.5± 1.5	0.6	1.02



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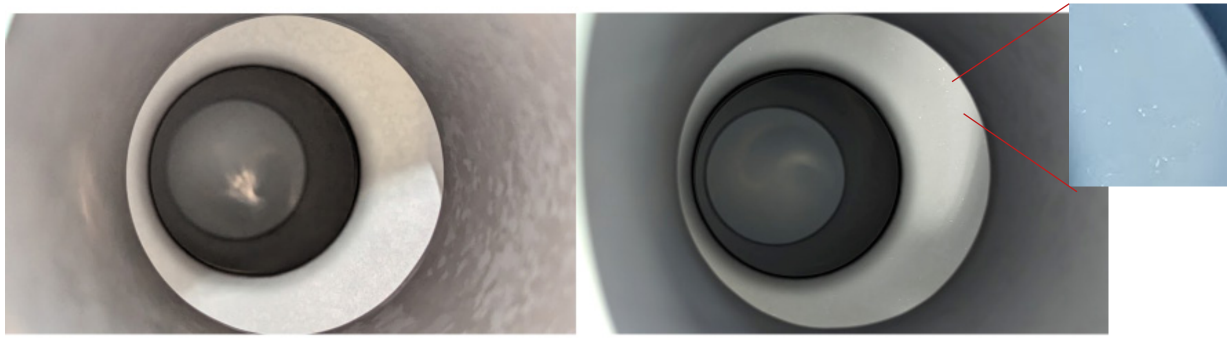
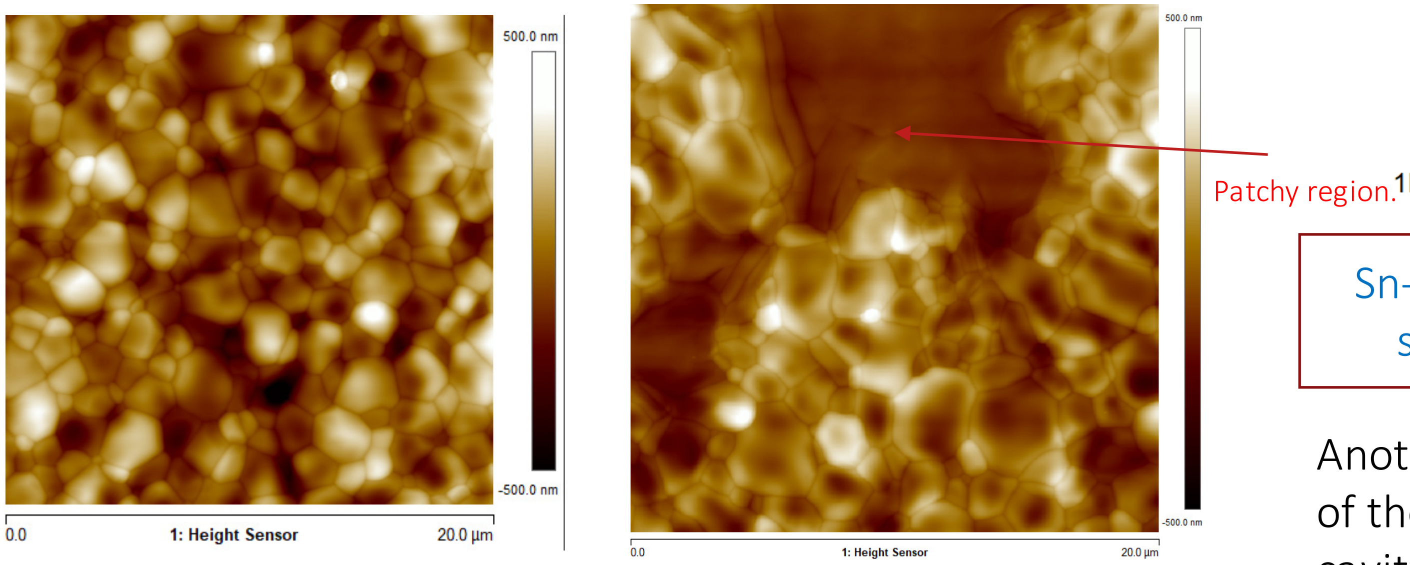
Cavity coating

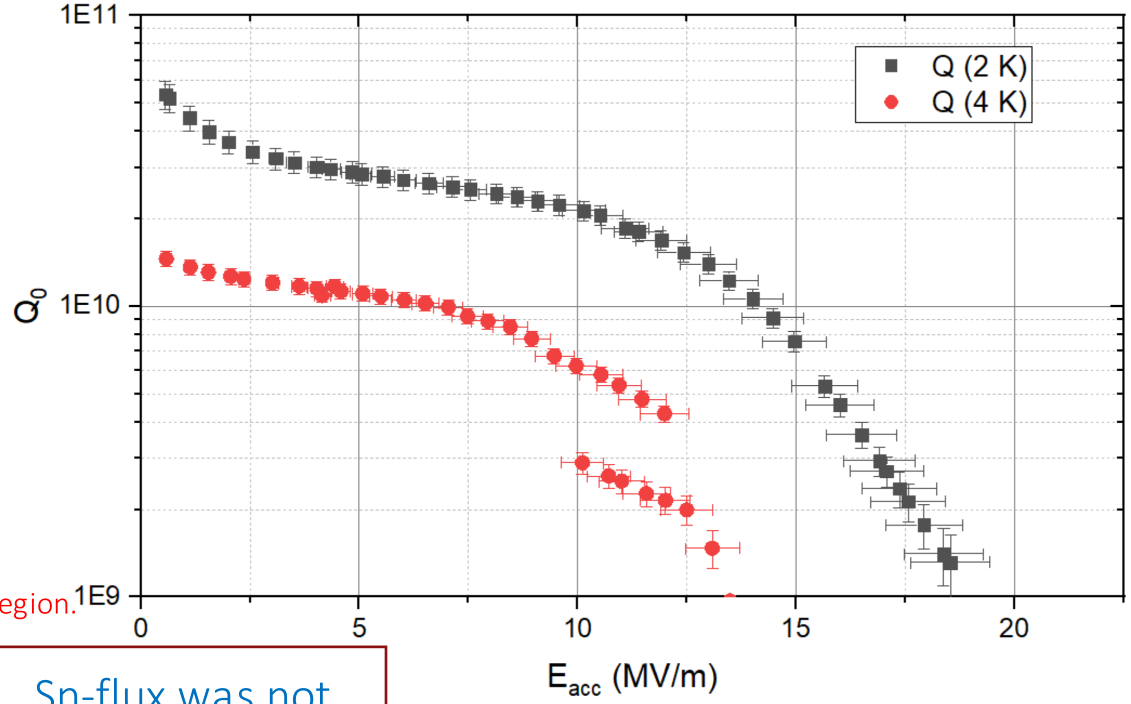
Expt	1200 °C	1150 °C	1100 °C	Sample	R_a (nm)	Grain size (µm)	Thickness (µm)
01	10 min	45 min	85 min	CW36	69 ± 3	0.93	-

Visual inspection





The roughness measured from these samples was 80±10 nm



Sn-flux was not sufficient.?

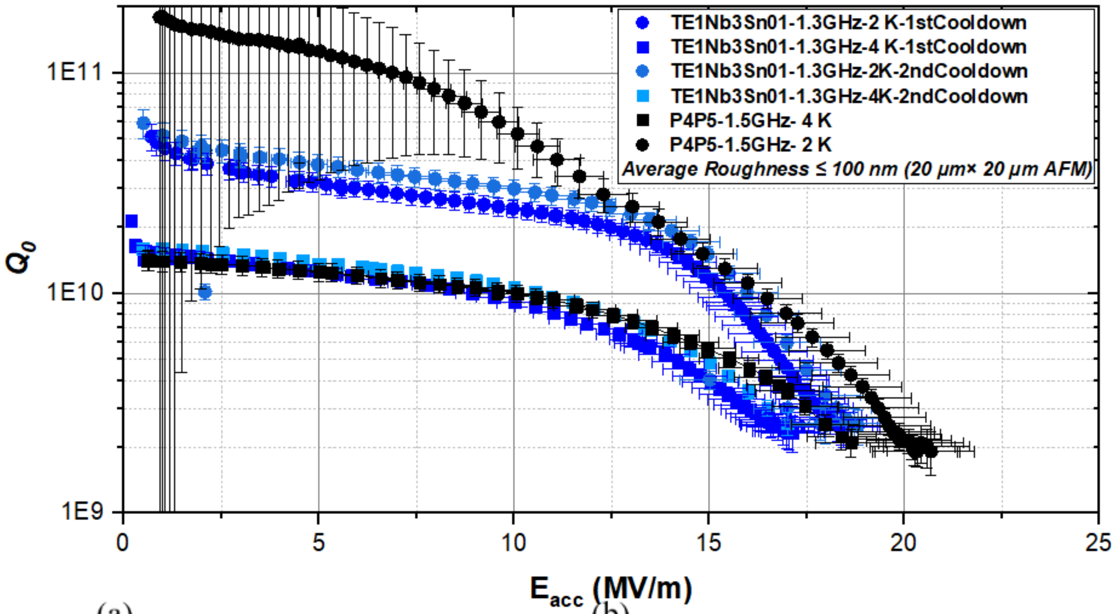
Test was limited by input power.

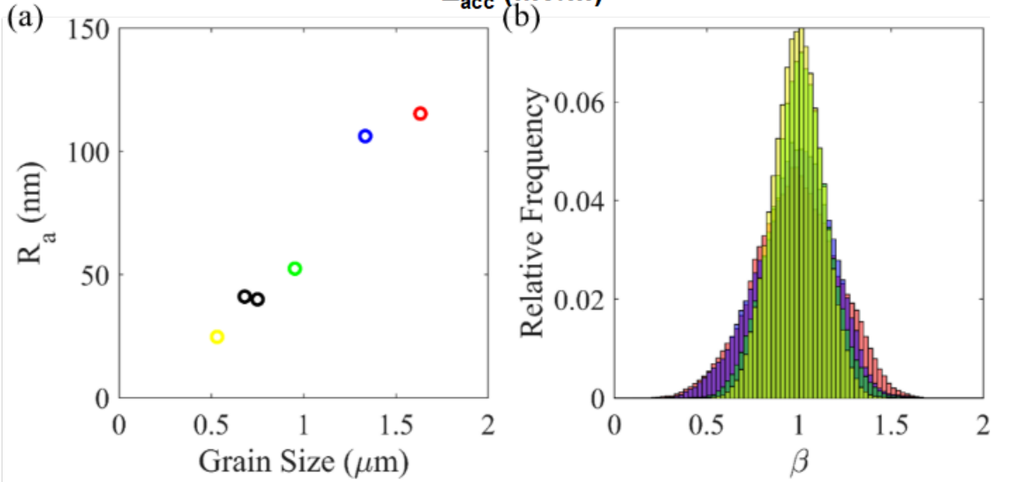
Another cavity coated with slight modification of the coating process is ready to test. Another cavity is ready to coat.

Cavity Performance

Cavity	Low field Q at 4.3 K	E_{acc} with Q $>10^{10}$ at 4.3 K	Max E_{acc} (MV/m)	Witness sample Ra (nm)
RDT7	3×10^{10}	15.5	15.5	57 ± 4
TEN1S002	2.5×10^{10}	16.5	17.5	68 ± 3
P4P5#	1.3×10^{10}	10	20.7*	98 ± 4
→ TE1NS001	1.6×10^{10}	11	18.9	87 ± 4
→ TE1NS001	2.3×10^{10}	9.5	16	102 ± 16
→ TE1NS001	1.7×10^{10}	9	16.5	154 ± 10
TE1NbSn01	1.3×10^{10}	7.5	19.2*	77 ± 1
TE1NbSn02	1.4 × 10	8	18.8*	80± 10
#P4P5 – 1.5 GHz cavity		* Limited by input power		

- Cavities typically attained higher gradients with lower surface roughness, even with patchy regions or Sn residues in a few instances.
- The spread of MFE factors increases with roughness or grain size.
- The improvement in gradient reach is likely to be the result of thinner Nb_3Sn coating (poor superconductor) and roughness reduction both.

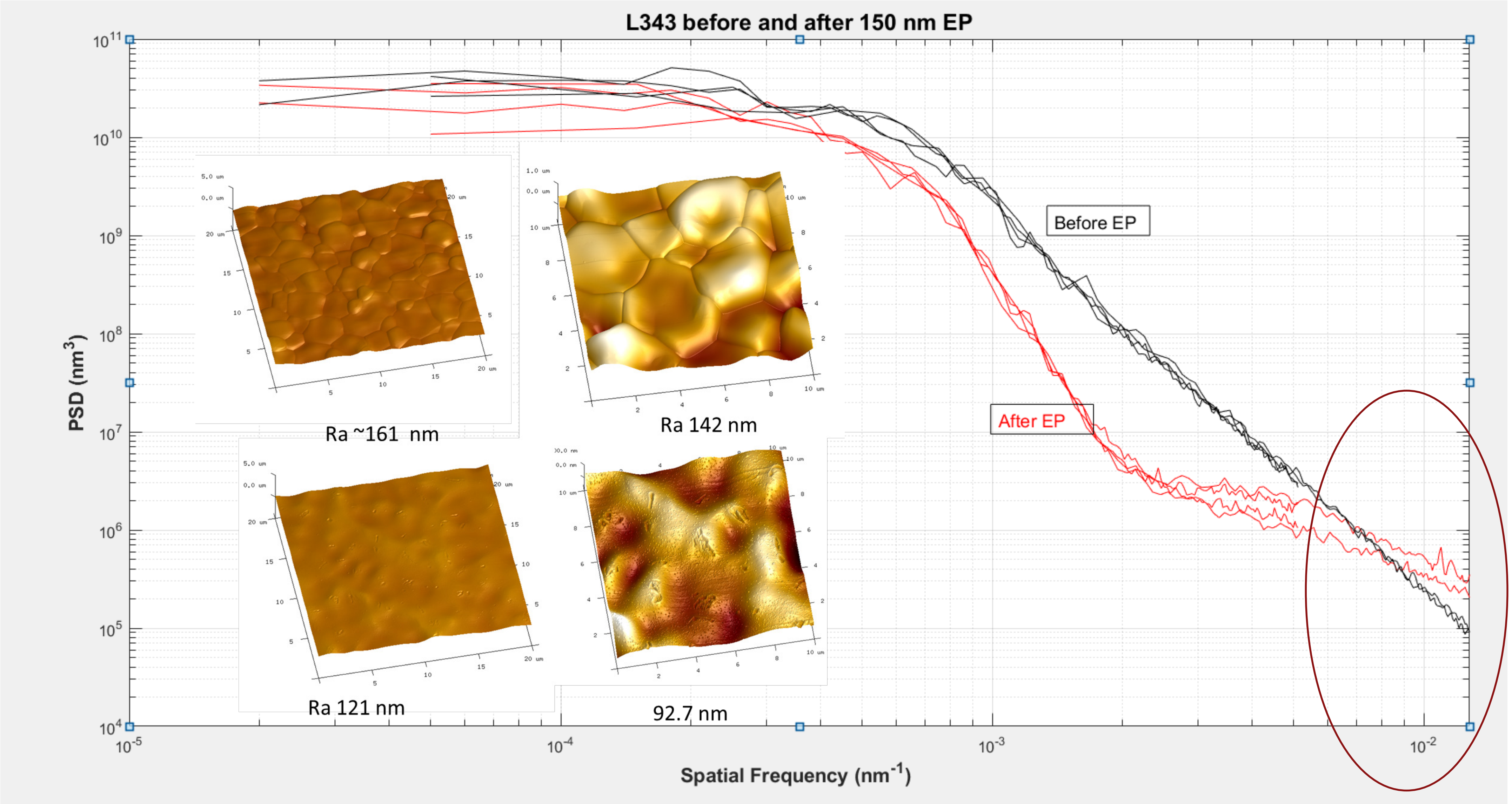




Measurement of surface roughness, R_a, as a function of grain size. (b) Relative frequency of the magnetic field enhancement factor calculated from AFM topographies using a perfect electrical conductor model. The colors shared between (a) and (b) indicate measurements derived from the same AFM topography

Post-coating treatments
for
Roughness Reduction

Electropolishing

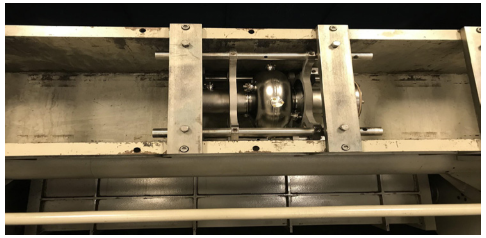


- 9 V resulting in current density ~ 20 mA/cm^2 at 11°C.
- Electrolyte 1:10 ratio of 49% HF to 98% H_2SO_4.

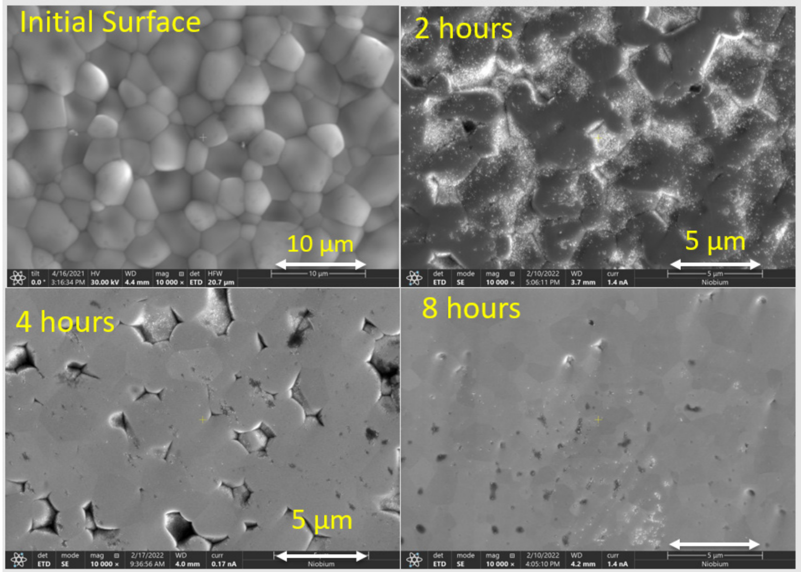
Roughness Reduction - Fermilab

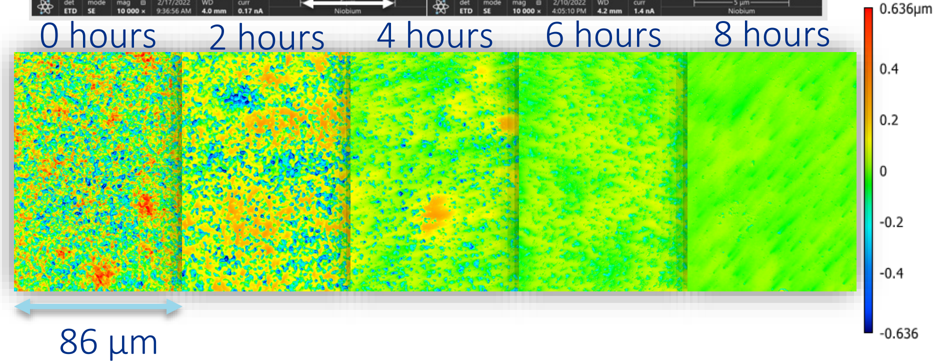
- The Centrifugal Barrel Polishing method (CBP) used commonly to smooth Nb cavities has been adapted to smooth Nb_3Sn coated cavities

- Nanoparticle abrasive suspension (40 nm silicate or 50nm alumina particles) plus (0.25'' wooden ball or 0.5'' felt cube)



- 120 RPM in the barrel polishing machine

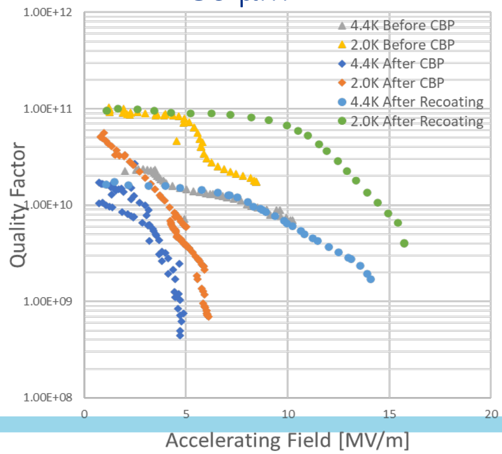




Before Coating

After Coating

After Polishing



Cavity performance was improved after CBP followed by a short recoating procedure

Courtesy of E. Viklund and S. Posen

TUPTB065

Fermilab

Summary and Outlook

- Micro-roughness of Nb_3Sn should be managed to improve RF performance.
- Pre-deposition of Sn layer promising to mitigate roughness
  - creation of high-density nucleation centers

    ↪ plan to coat 2.45/2.6 GHz cavity
- Thicker coating ←→ increased grain size → higher surface roughness

  ↪ vary coating parameters like time & temperature & Sn-flux etc.
- 1st coating of single-cell w/ new parameters

  → asymmetric coating with patchy regions and shiny features

  → limited to 19 MV/m
- Reduced roughness typically yields higher gradients!
- EP produces smoother Nb_3Sn surface, but more studies needed to understand the surface defect.
- Significant efforts are in progress in different laboratories aiming at smoother Nb_3Sn thin film.

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