Study of Alternative Materials for Next Generation SRF Cavities at Cornell University

Zeming Sun, Thomas Oseroff, Ryan D. Porter, Katrina Howard, Matthias U. Liepe (CLASSE)

Zhaslan Baraissov, Nathan Sitaraman, Michelle Kelly, James Sethna, Tomas Arias, David Muller (Cornell Physics)

Kevin D. Dobson (Institute of Energy Conversion)

Xiaoyu Deng (UVA)

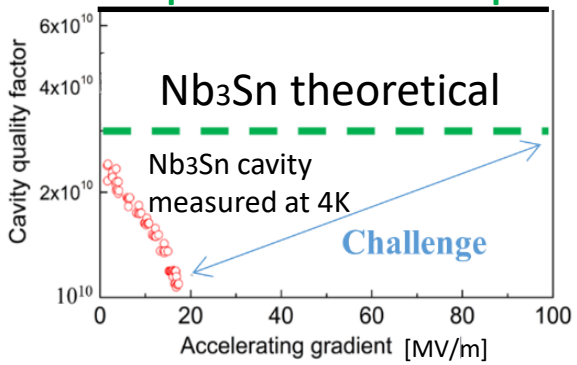
Aine Connolly, Michael O. Thompson (Cornell MSE)

Thin film workshop
03/2021

Overview of alternative materials studied at Cornell

Alternative materials

- Nb_3Sn
  - ✓ Doubled superheating field: ~400 mT vs. Nb: ~200 mT
  - ✓ Increased operation temperature: 2 K to 4 K



- V_3Si
- NbTiN & NbTiN/AlN/Nb SIS structures
- MgB_2

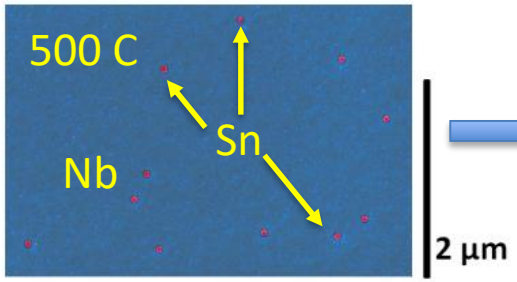
Alternative approaches

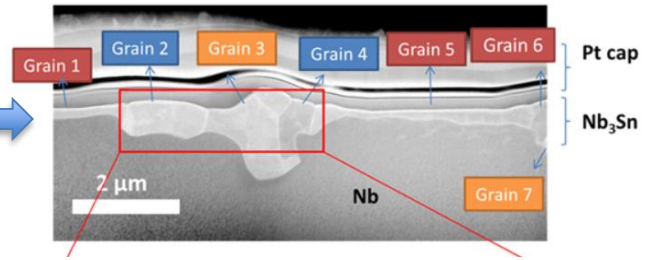
- Vapor diffusion
- Electroplating
- Chemical vapor deposition
- Sputtering
- Plasma-enhanced ALD
- Reactive vapor deposition

Nb_3Sn generation through thermal conversion of electroplated Sn films on Nb substrate

Issues in Nb_3Sn vapor diffusion

- Large surface roughness

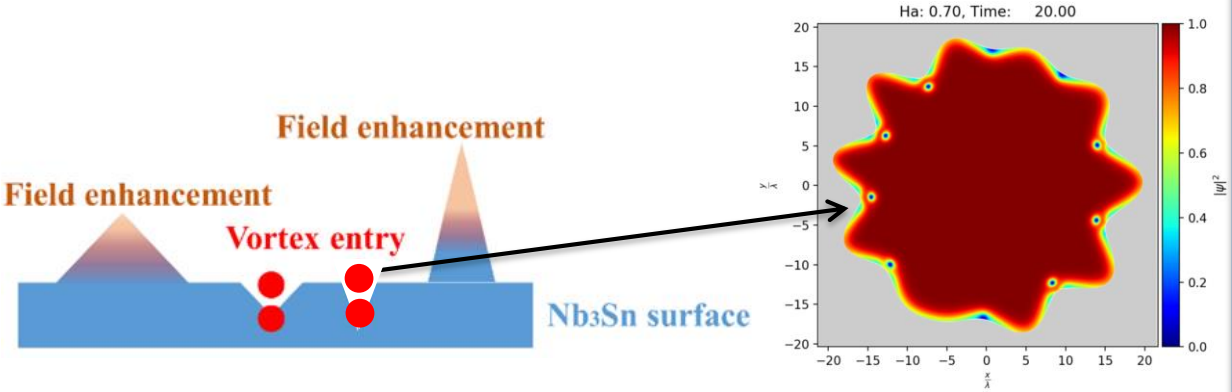




Non-uniform nucleation events
(Ryan Porter, et al., LINAC'19)

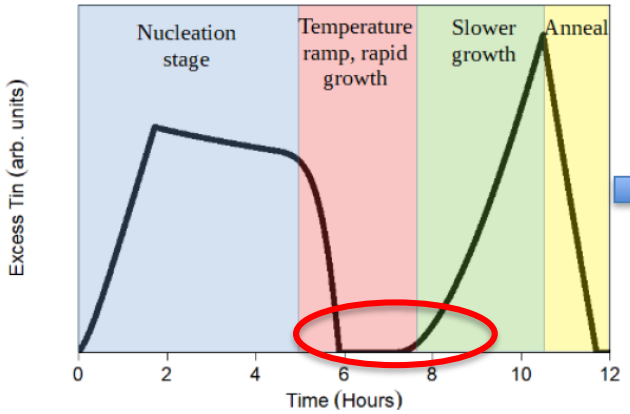
Source of surface roughness: large variation in grains
(Jaeyel Lee, et al., Supercond. Sci. Technol., 2018)

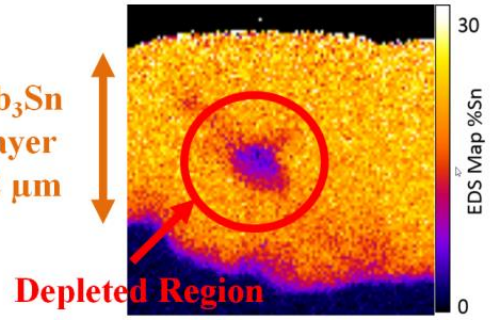
- Impact on the RF performance



Vortex nucleation for rough surface
(Alden Pack, et al., PRB, 2020)

- Sn depletion region/stoichiometry

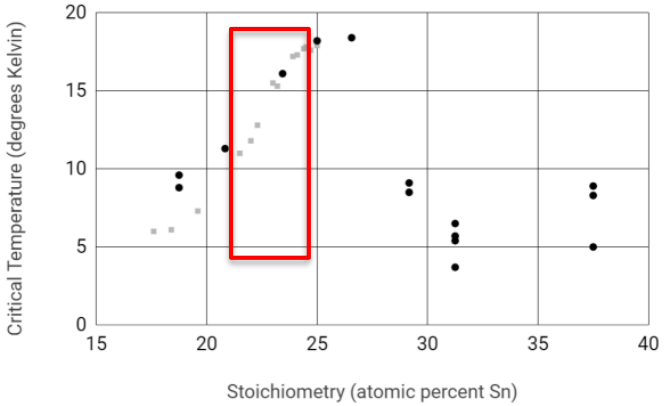




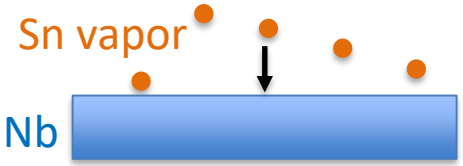
Insufficient Sn supply
(Nathan Sitaraman, et al. arXiv:1912.07576 [cond-mat.supr-con])

(Ryan Porter, et al., LINAC'19)

- Impact on the RF performance



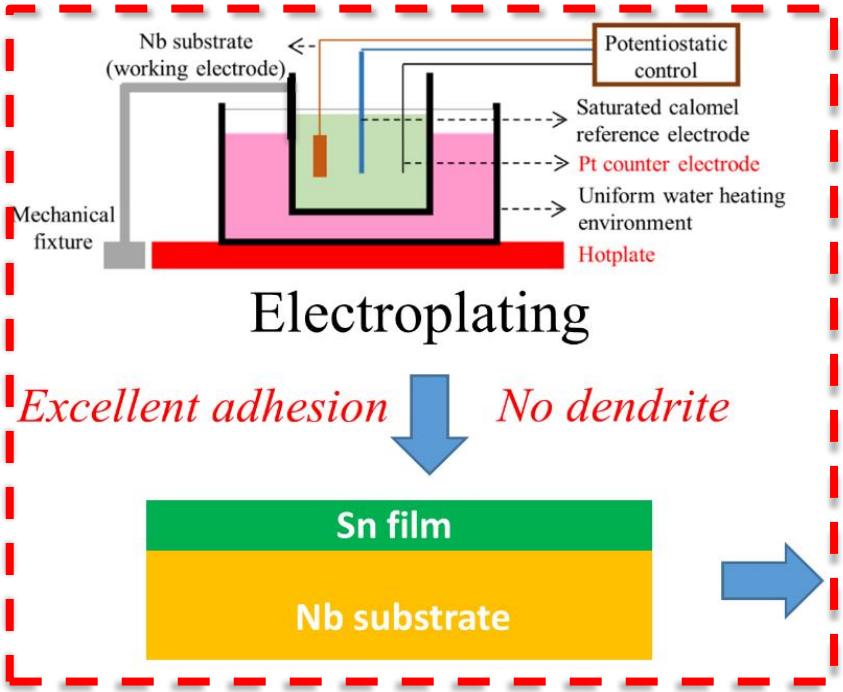
(Nathan Sitaraman, et al. arXiv:1912.07576 [cond-mat.supr-con])

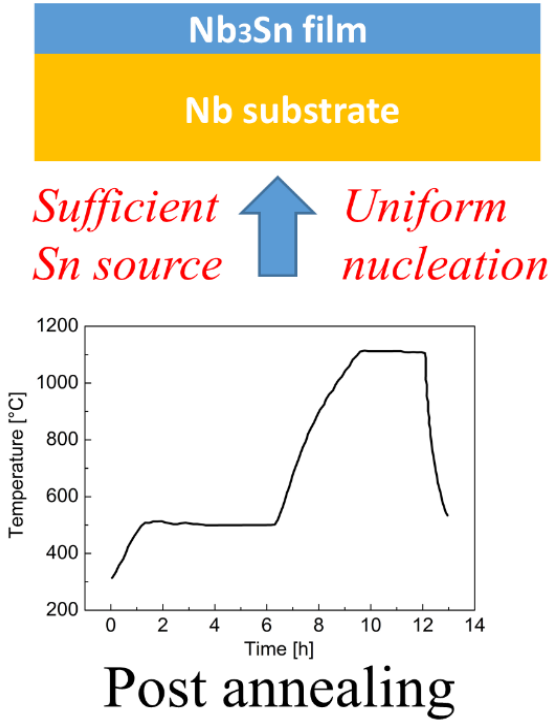


Conventional Sn vapor diffusion method for generating Nb3Sn

Our strategy

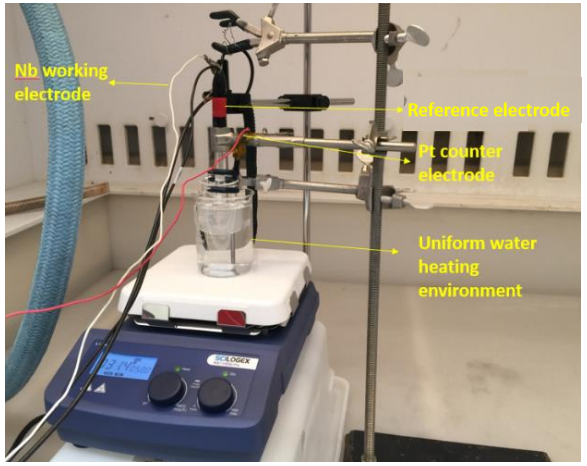
- Pre-deposition of a uniform smooth Sn layer on Nb
- Thermal conversion to Nb_3Sn
- Advantages:
  - ✓ Promote uniform distribution of nucleation to lower the surface roughness
  - ✓ Excess Sn supply to satisfy the kinetic requirement for Nb_3Sn stoichiometry
  - ✓ Possible application of Nb_3Sn on Cu cavity (better heat conduction, low cost)

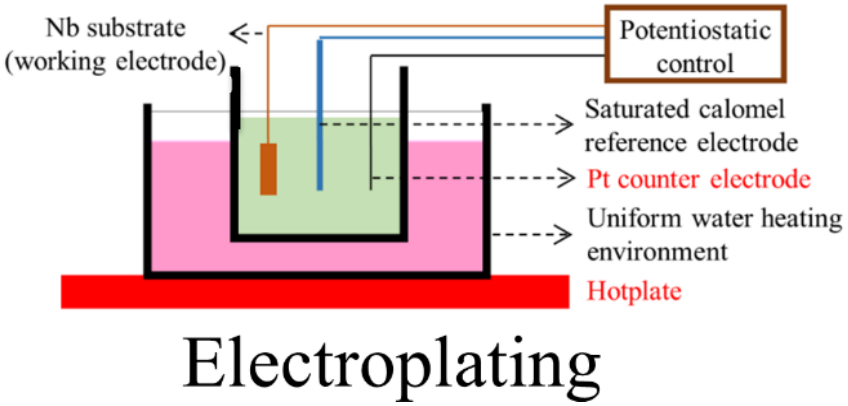




Enable direct Sn electroplating on Nb surface

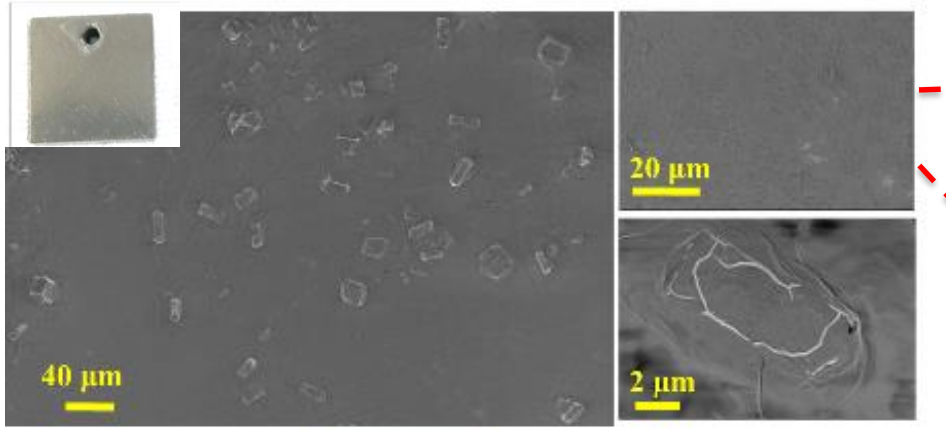
- Challenges
  - ✓ Low lateral surface diffusion
  - ✓ Dendrite formation --- Large initial surface roughness
  - ✓ Poor adhesion at Sn/Nb interface --- Peel-off issue
  - ✓ Control of impurity concentration
- Methods
  - ✓ A three-electrode deposition system
  - ✓ Optimization of temperature, solution chemistry, plating potential, ph-values, and stirring conditions

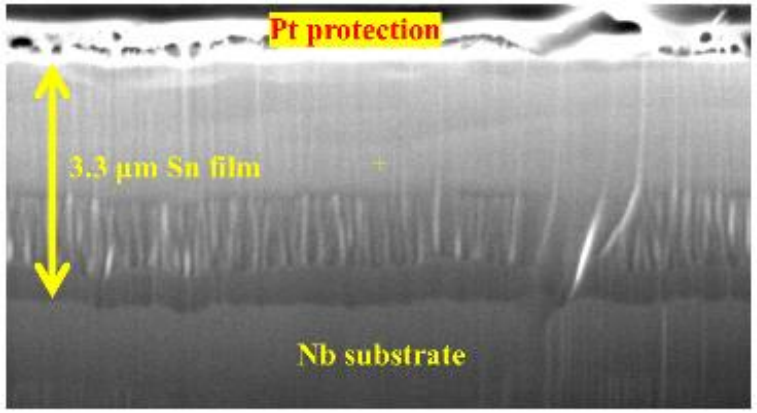




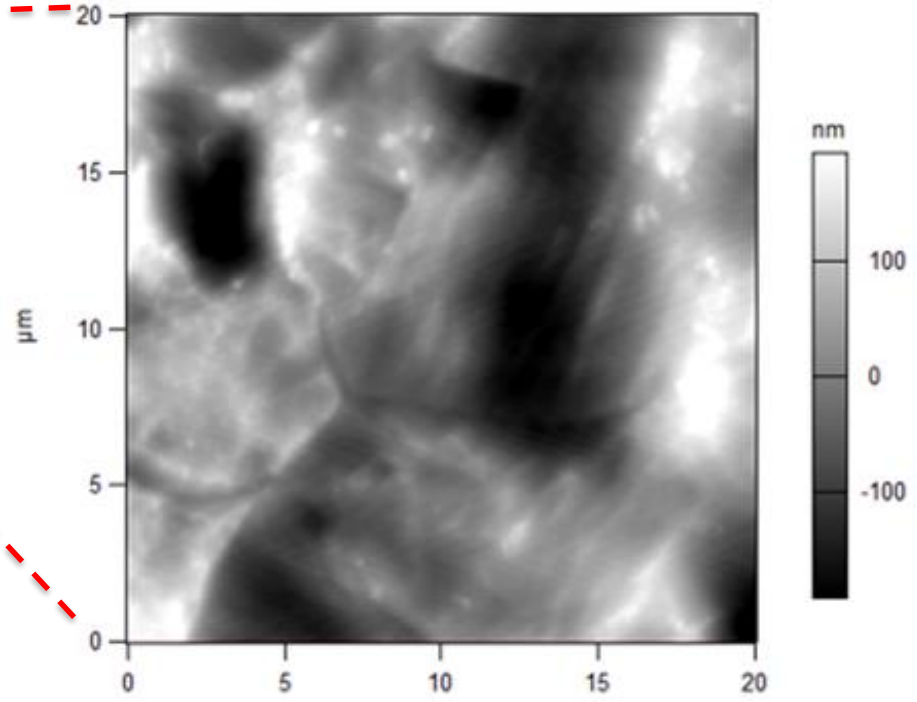
Demonstration of uniform, smooth Sn film on Nb

- Uniform surface morphology (some embedded grains)



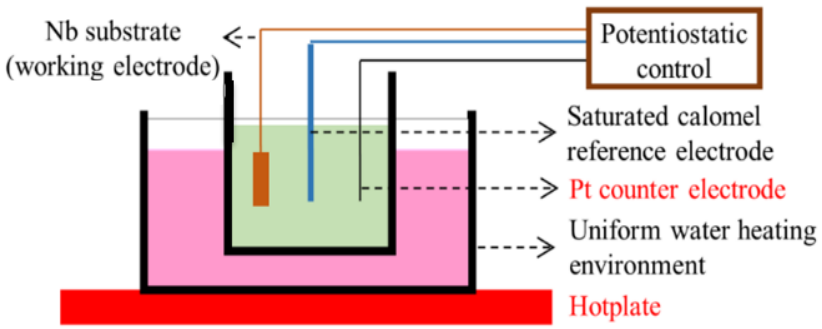


- Low surface roughness on the flat region (Ra= 65 nm, AFM)



- Cross-section SEM shows smooth surface and thickness of ~3 um.

Post annealing of electroplated films



Electroplating

Excellent adhesion *No dendrite*

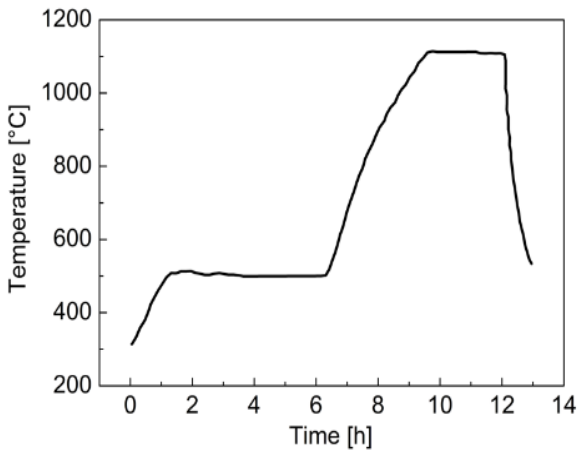
Sn film

Nb substrate

Nb_3Sn film

Nb substrate

Sufficient Sn source *Uniform nucleation*

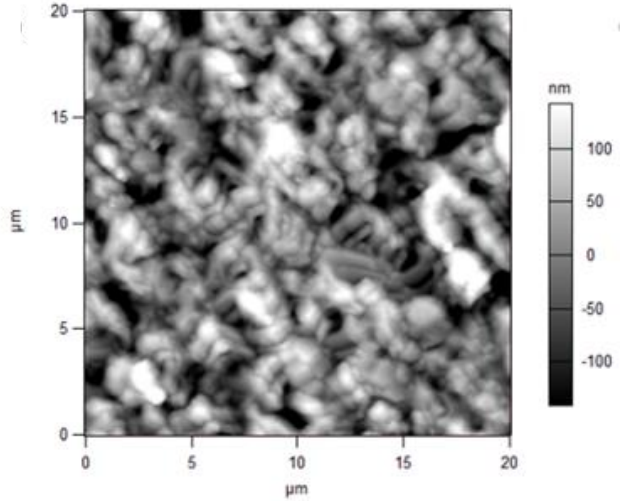


Post annealing

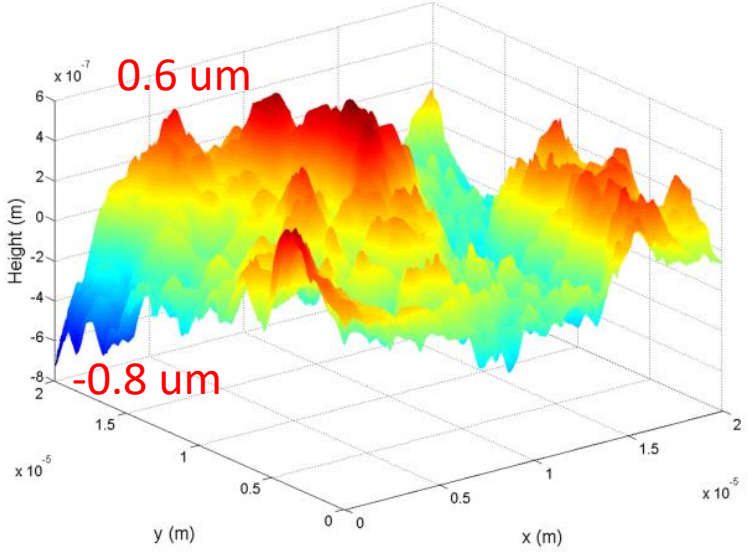
Extremely low surface roughness

- Comparison between vapor diffused sample and plated-Sn converted sample

AFM image of plated-Sn converted Nb3Sn



AFM mapping of vapor diffused Nb3Sn

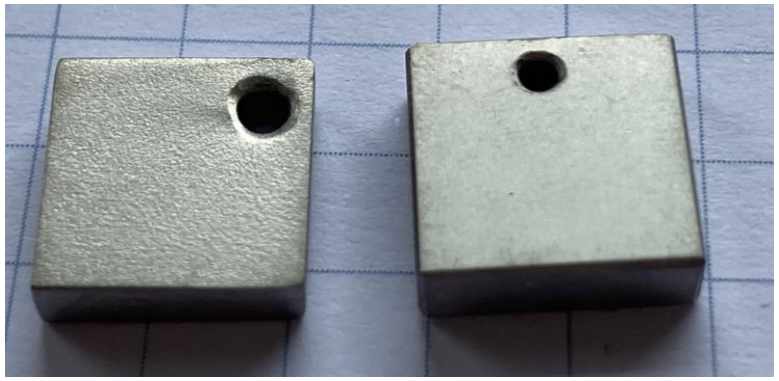


Ryan Porter, et al. LINAC'16

✓ 4 – 5 times reduction in surface roughness

	Ra [nm]	Rq [nm]	Rz [nm]
Starting Nb surface	70	95	2305
Electroplated Sn films	65	80	700
Thermally converted Nb_3Sn films	60	70	650
Vapor diffused Nb_3Sn films	~300	~300	~2000

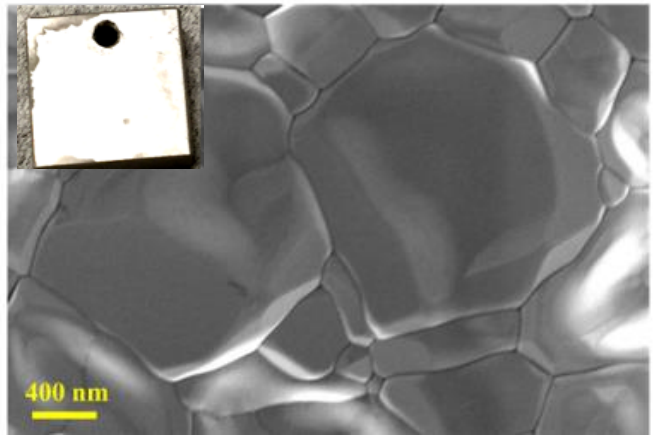
* Recently, Fermi Lab and KEK both report ~100 nm Ra on their new "shiny" coatings

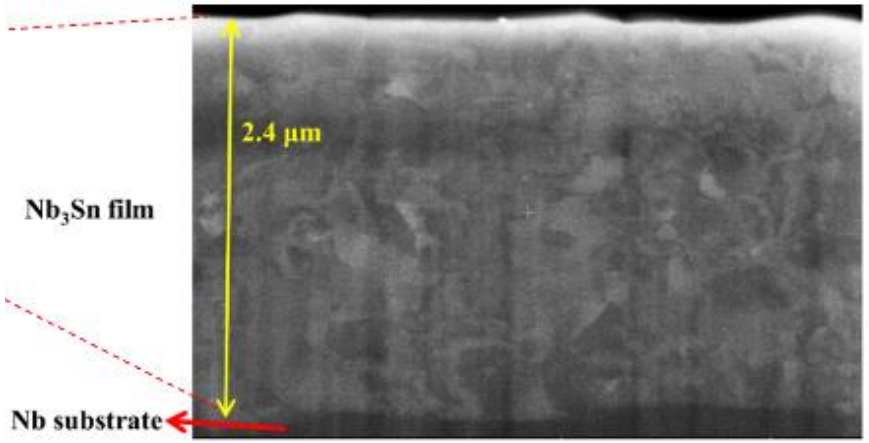


Vapor diffused Nb3Sn Plated-Sn converted Nb3Sn

Stoichiometric Nb_3Sn

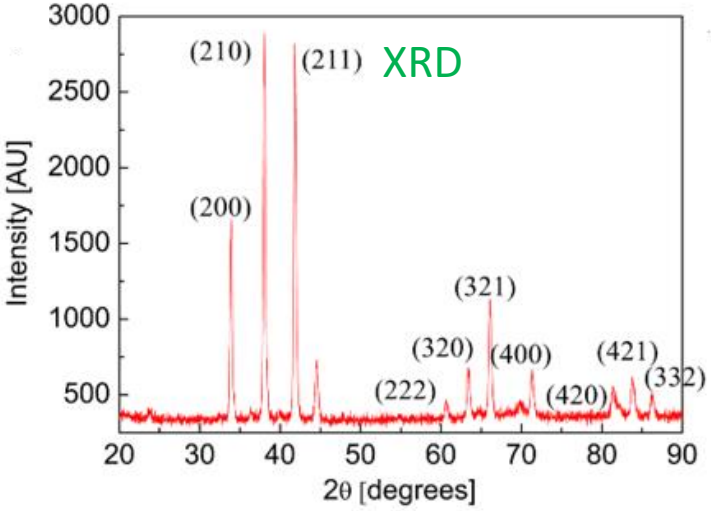
- Uniform film, relative small grain size

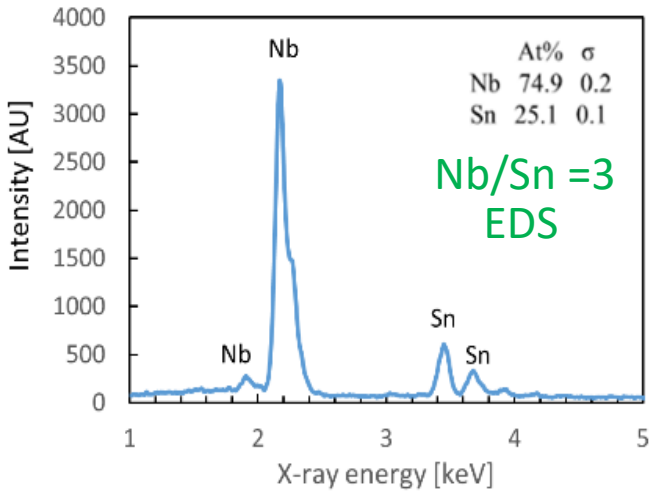


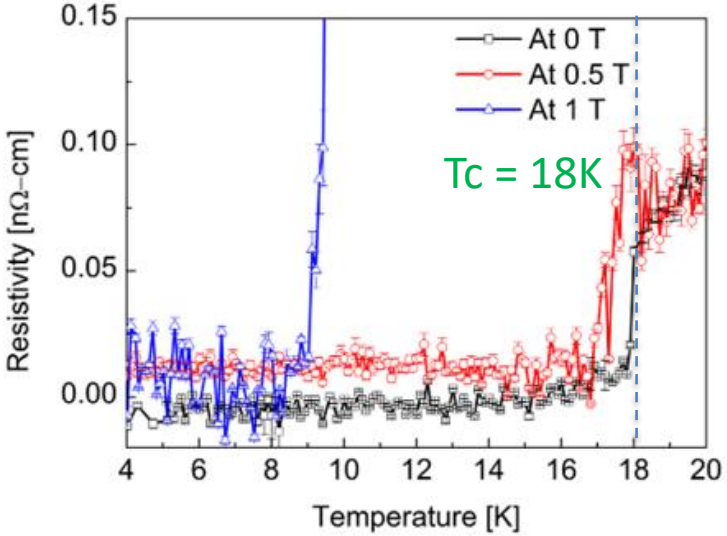


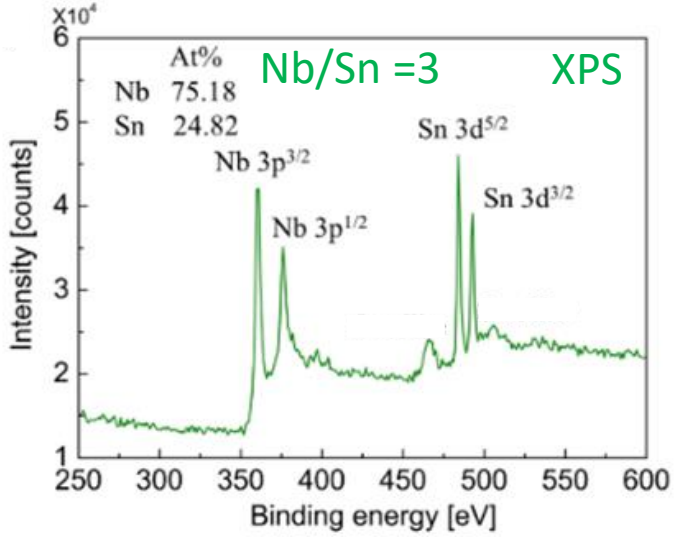
- Cross-section SEM shows smooth surface and 2.4 um thickness

- Stoichiometric Nb_3Sn with Tc of 18 K and low concentration of impurity (O may be a concern)



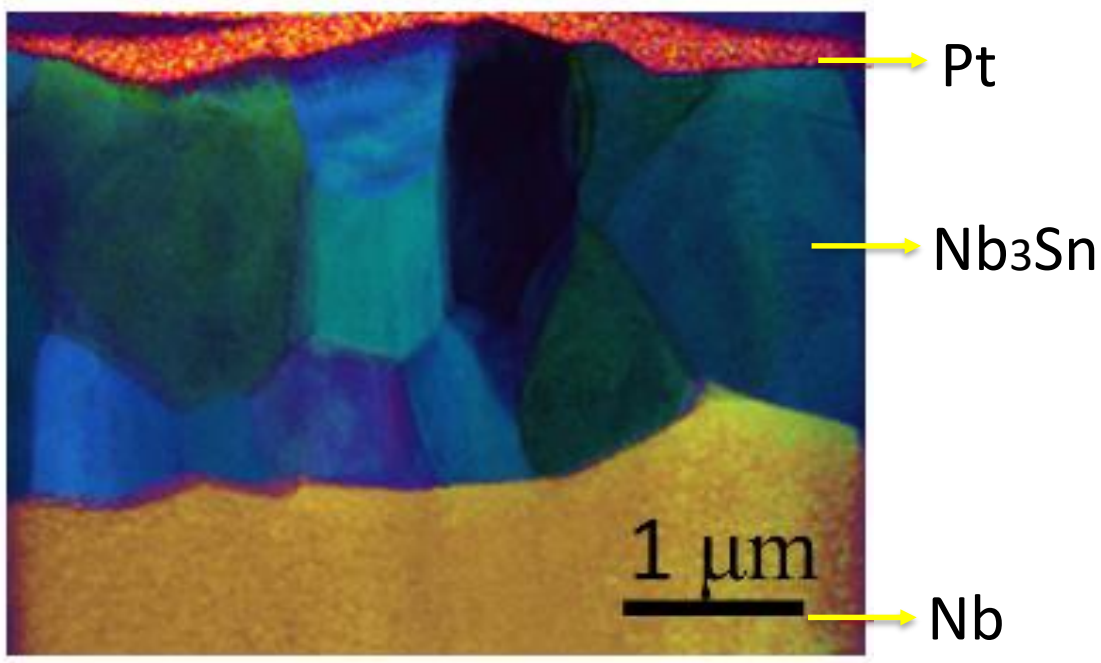






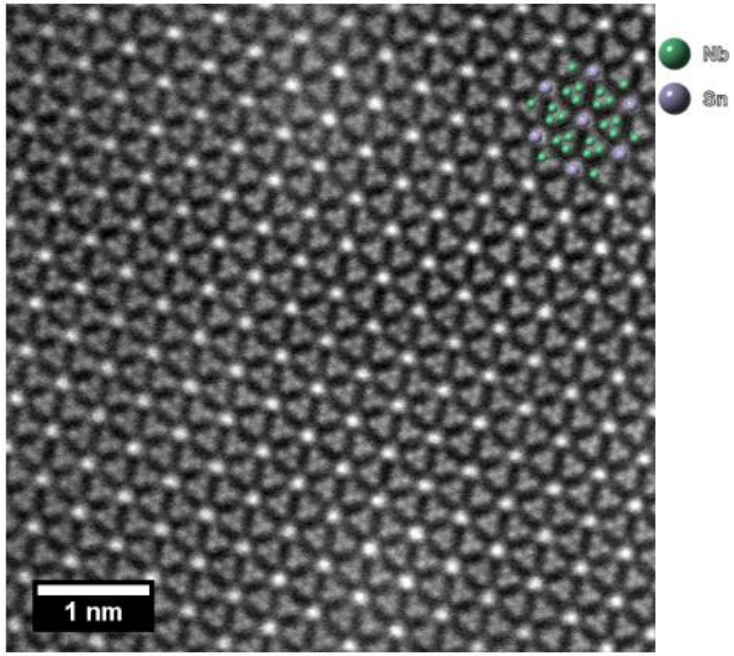
Ongoing: Atomic imaging and analysis

Cross-sectional STEM-EWPC



- Multiple grains in the depth direction

[111] Nb_3Sn



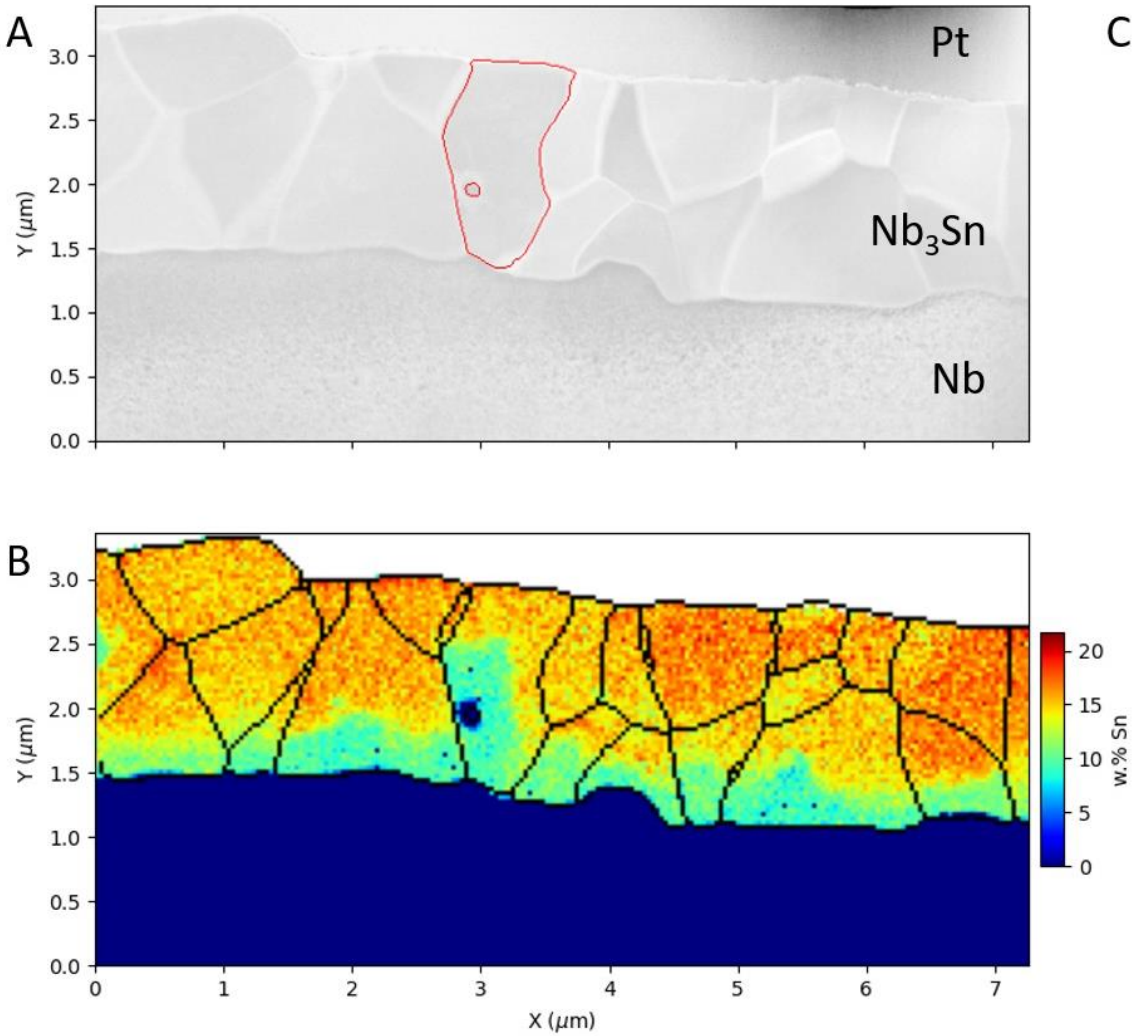
- Sub- angstrom resolution
- Accurate composition and strain mapping

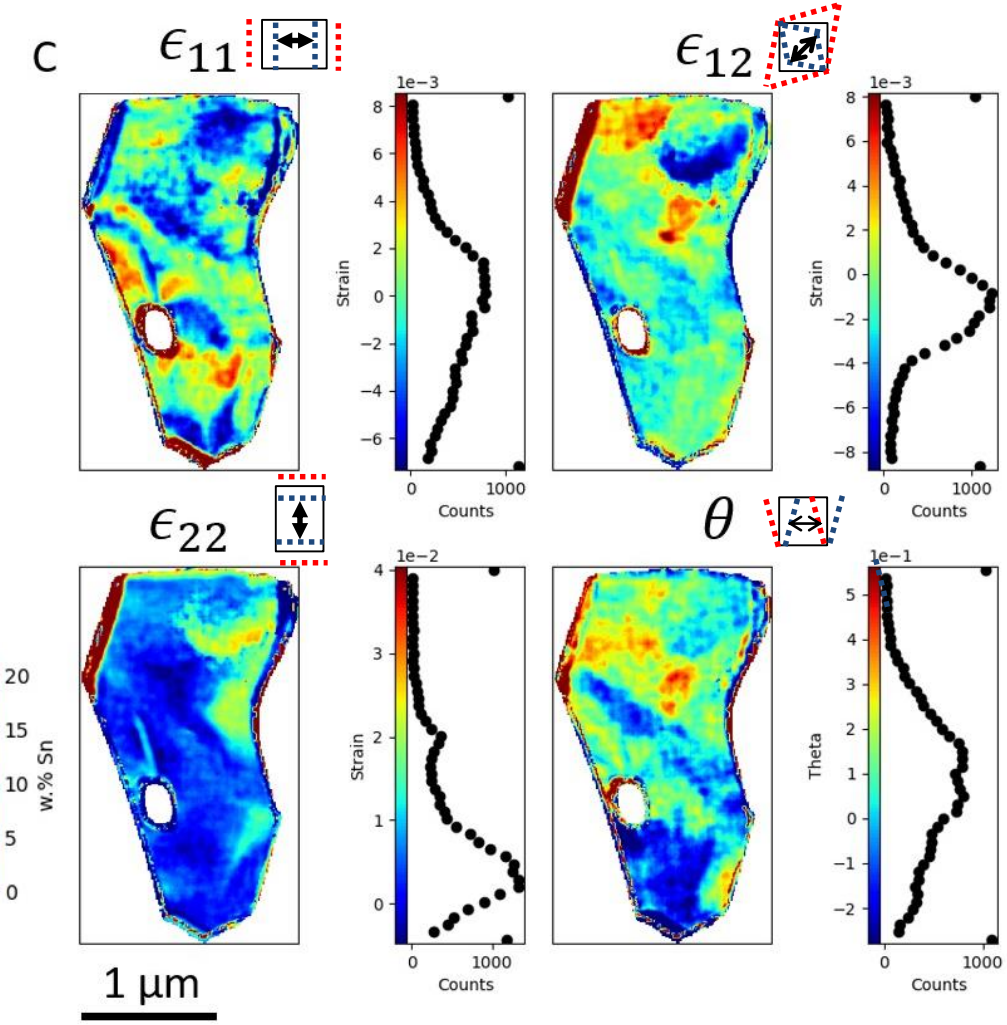
Zhaslan Baraissov

Prof. David A. Muller

Ongoing: Composition and strain mapping

- Plated-Sn converted Nb_3Sn vs. vapor diffused Nb_3Sn
- Preliminary results from vapor diffused Nb_3Sn



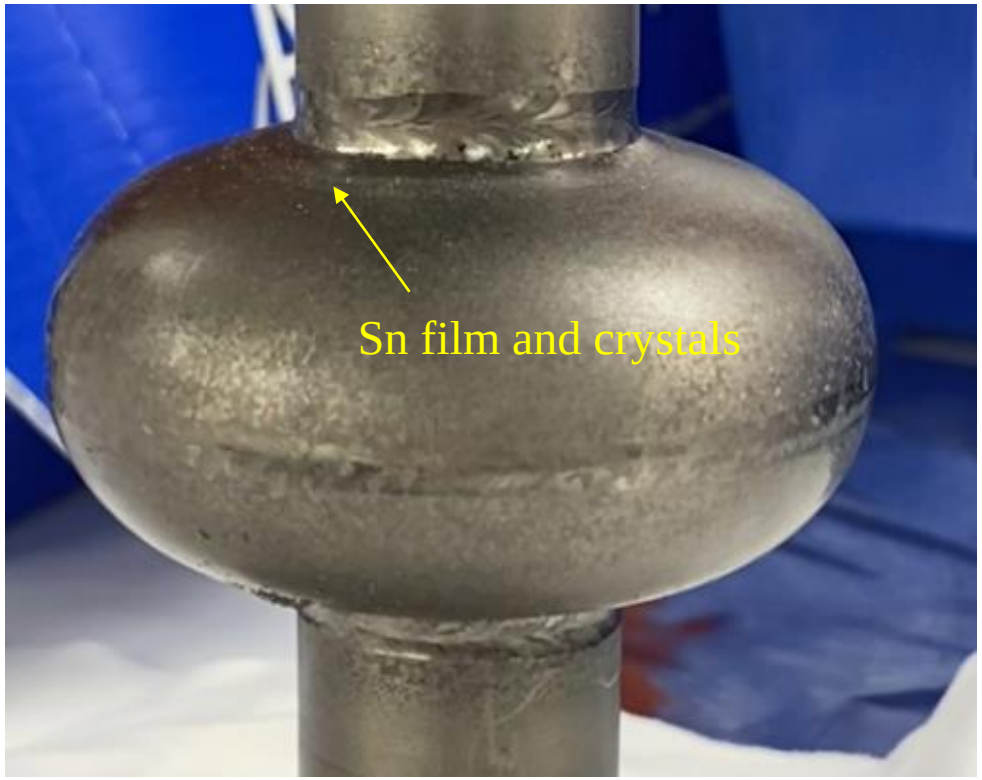


Zhaslan Baraissov

Prof. David A. Muller

Ongoing: scale up to 3.9 GHz cavity

Plating test on outer surface



Plating on inner surface

Before plating

After plating

Sputtered Nb_3Sn and V_3Si

Katrina Howard

Sputtering setup and sample details

- Sputtering deposition system at Cornell Center for Materials Research (CCMR)
  - DC power
  - Bulk Nb_3Sn target
- Sample variables
  - 100 nm Nb_3Sn and V_3Sn films on Nb substrate
    - ✓ Rotating stage at room temperature
  - 2 um Nb_3Sn and V_3Sn films on Nb substrate
    - ✓ Rotating stage at mystery temperature
  - 300 nm Nb_3Sn and V_3Sn films on Cu substrate
    - ✓ Heated stage at 550 C
- Furnace annealing
  - 600 C, 700 C, 800 C, 950 C for 6 h

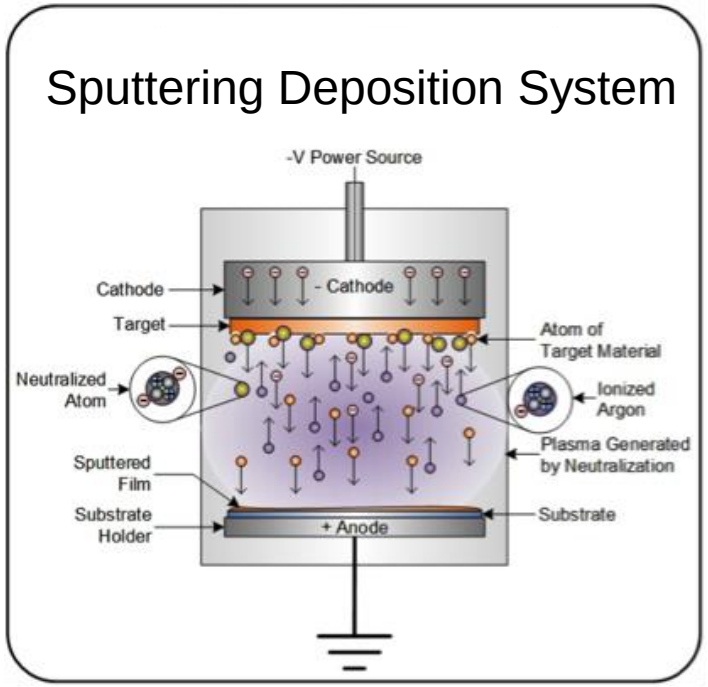


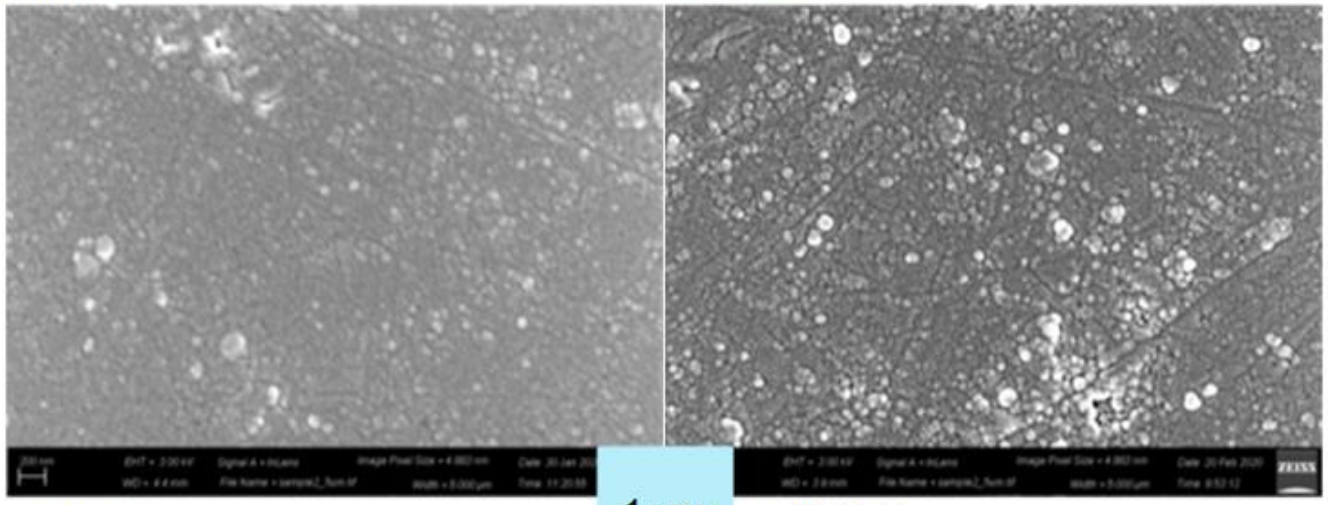
Image source: http://www.semicore.com/what-is-sputtering

Thermal characteristics and recrystallization

100 nm Nb_3Sn on Nb (room temp) SEM

initial

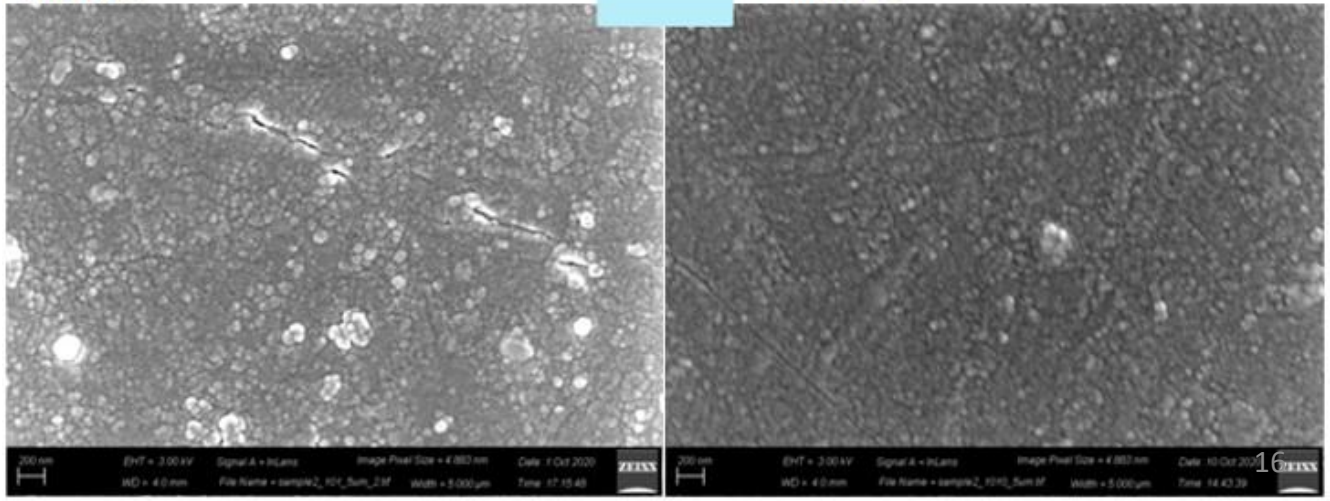
600 C



1 um

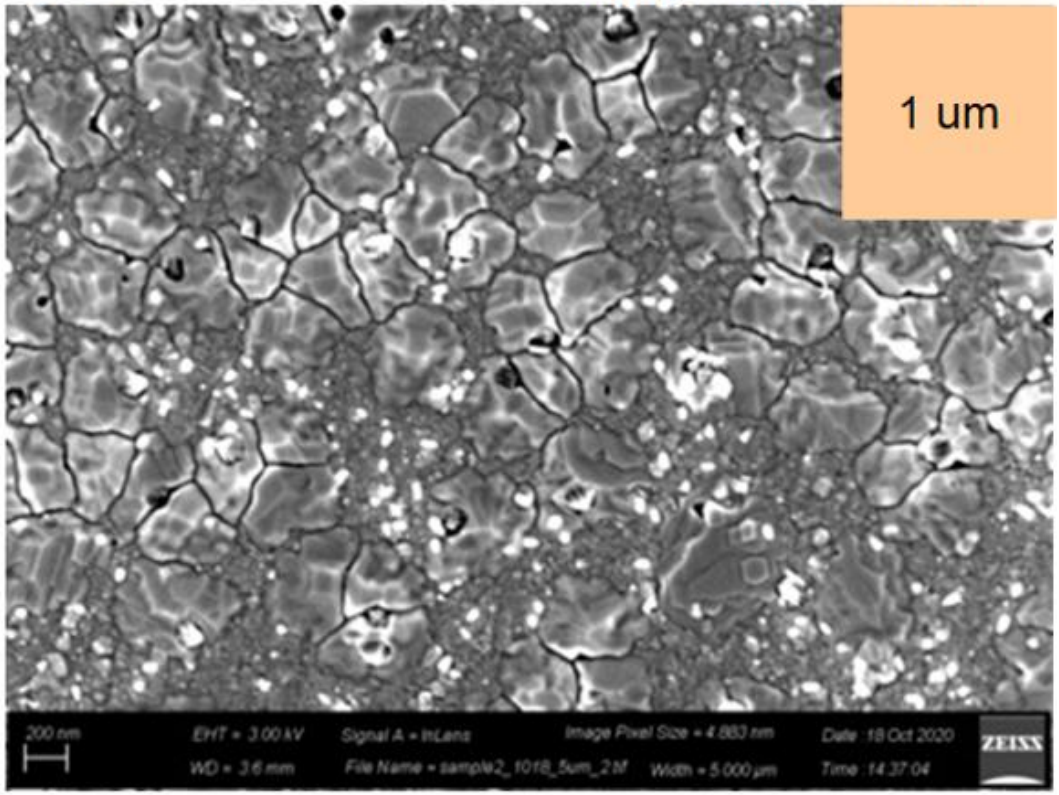
700 C

800 C



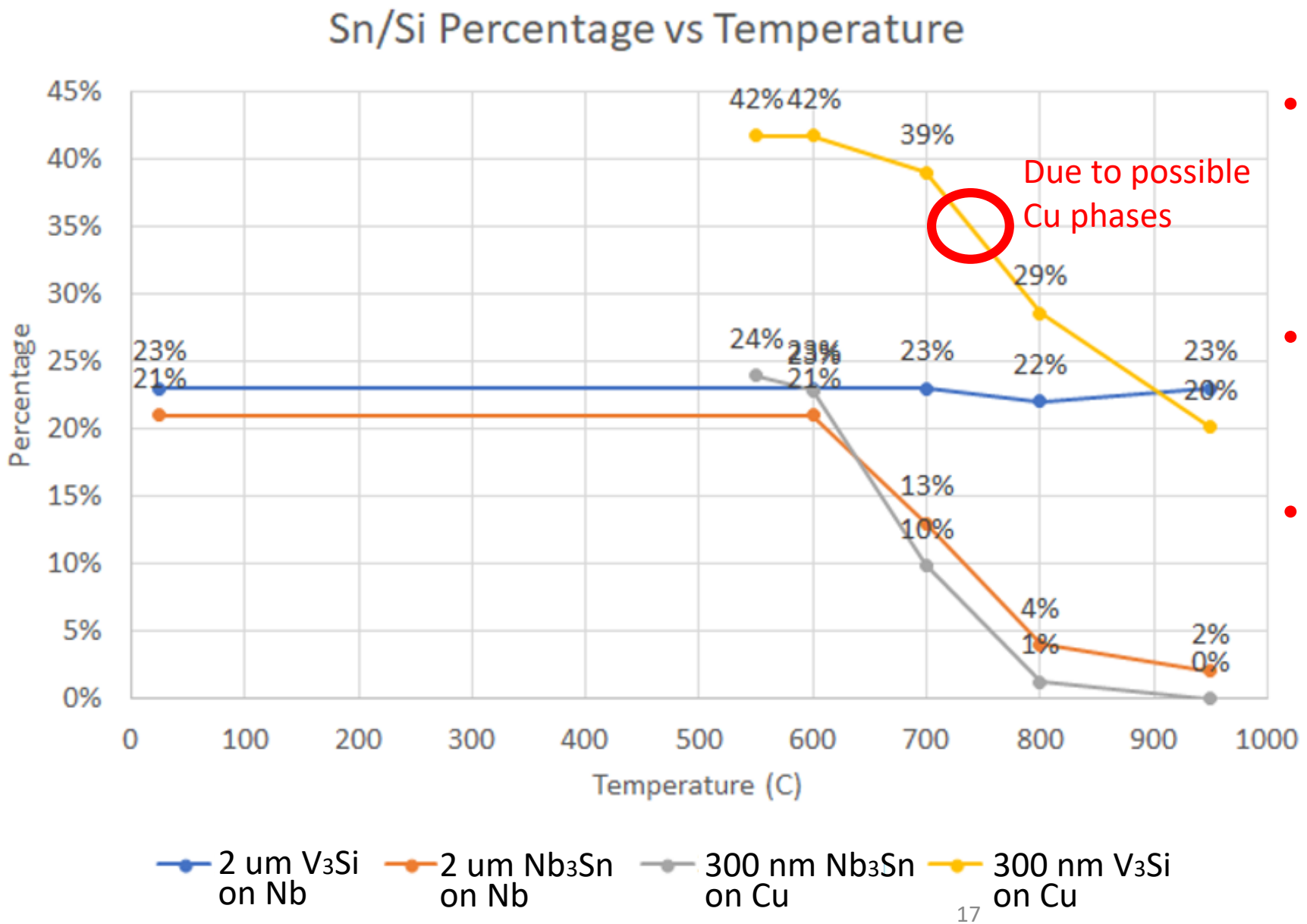
950C

images are 5 um wide



- Observe recrystallization at 950 C with larger grain size at ~300 nm

Loss of Sn in Nb_3Sn or Si in V_3Si during annealing



- Relative percentage
  - ✓ Sn vs. Nb%
  - ✓ Si vs. V%
- Observe significant Sn/Si loss expect 2 um V_3Si samples
- 300 nm sample results were calibrated according to electron penetration depth and film thickness

Plasma-enhanced atomic layer deposited NbTiN/Nb vs. NbTiN/AlN/Nb SIS structure

Plasma-enhanced atomic layer deposition

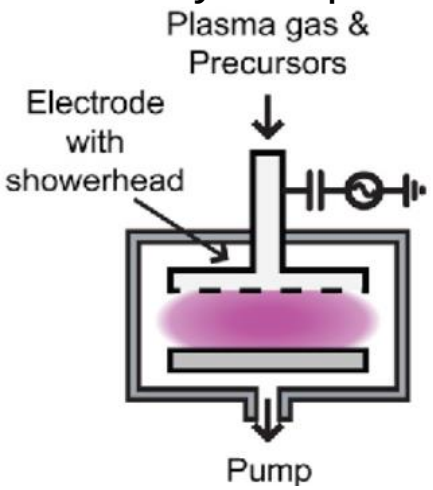
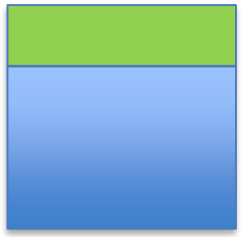
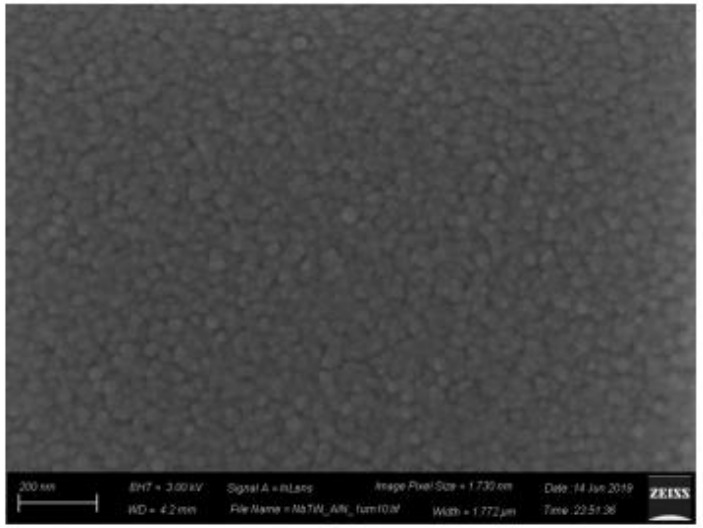


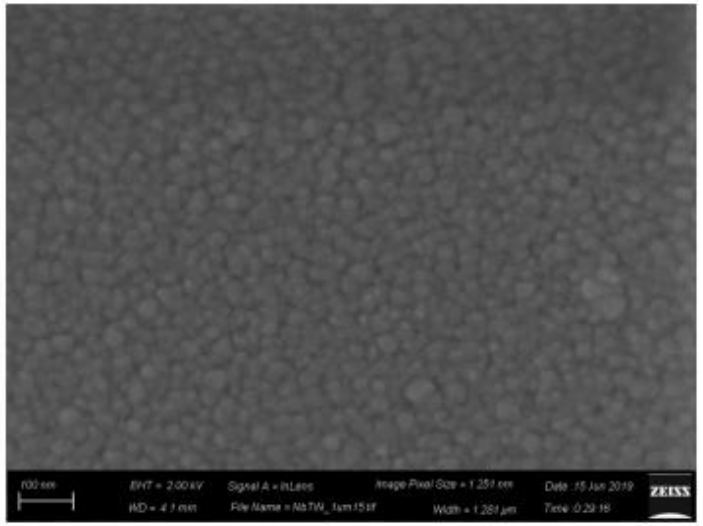
Image source: H.H. Profijt, et al., J. Vac. Sci. Technol. A 29, 050801 (2011)

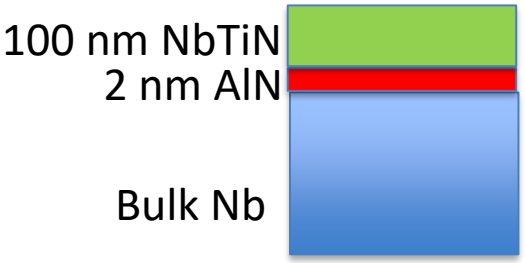
Sample information and surface resistance

- Samples obtained from Veeco-CNT



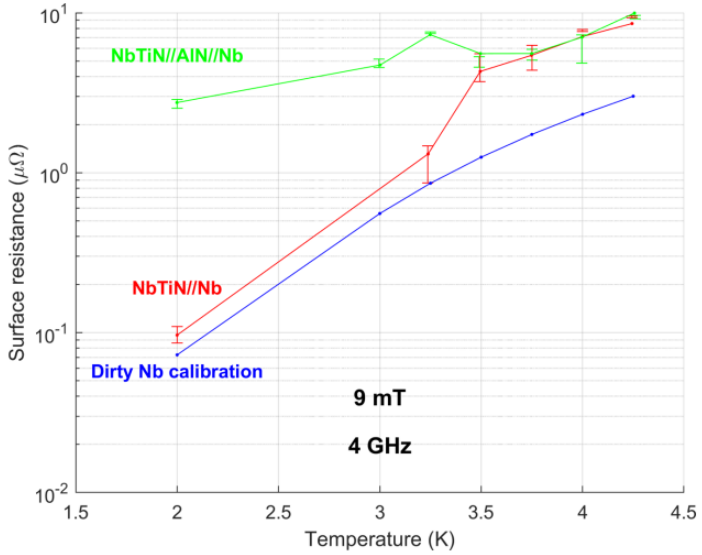




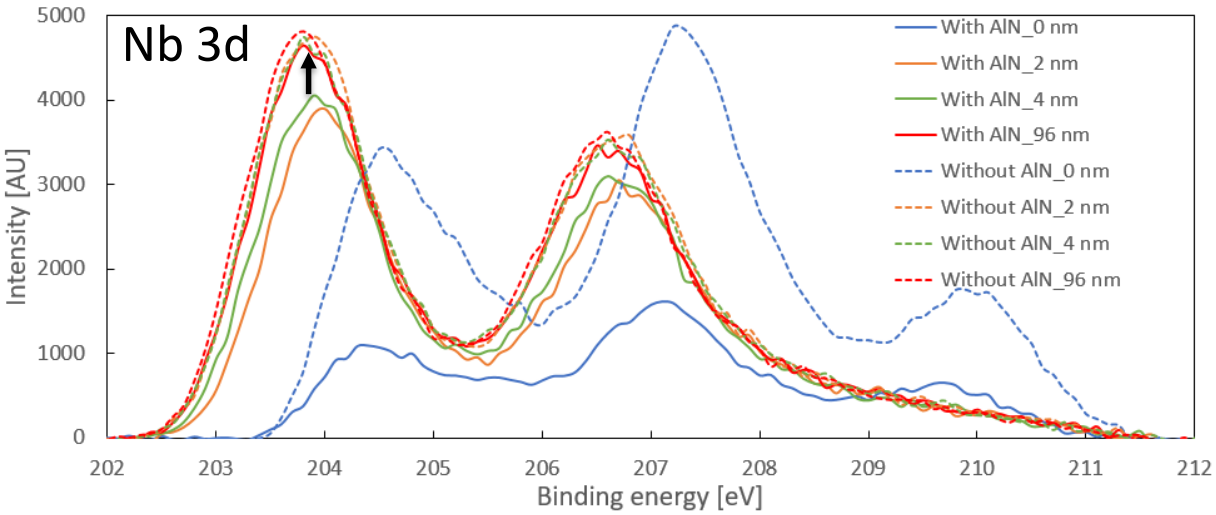


- Surface resistance
  - Measured by Cornell sample host
  - Observed the different behaviors between two structures

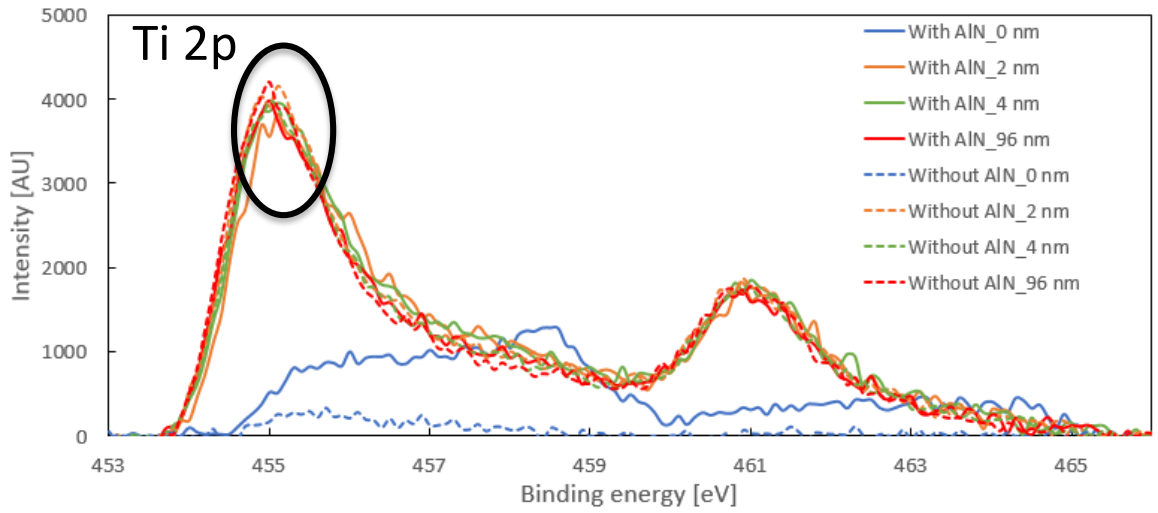
"Upgrades to the Cornell sample host cavity", Thomas Oseroff, THU 9 am EST

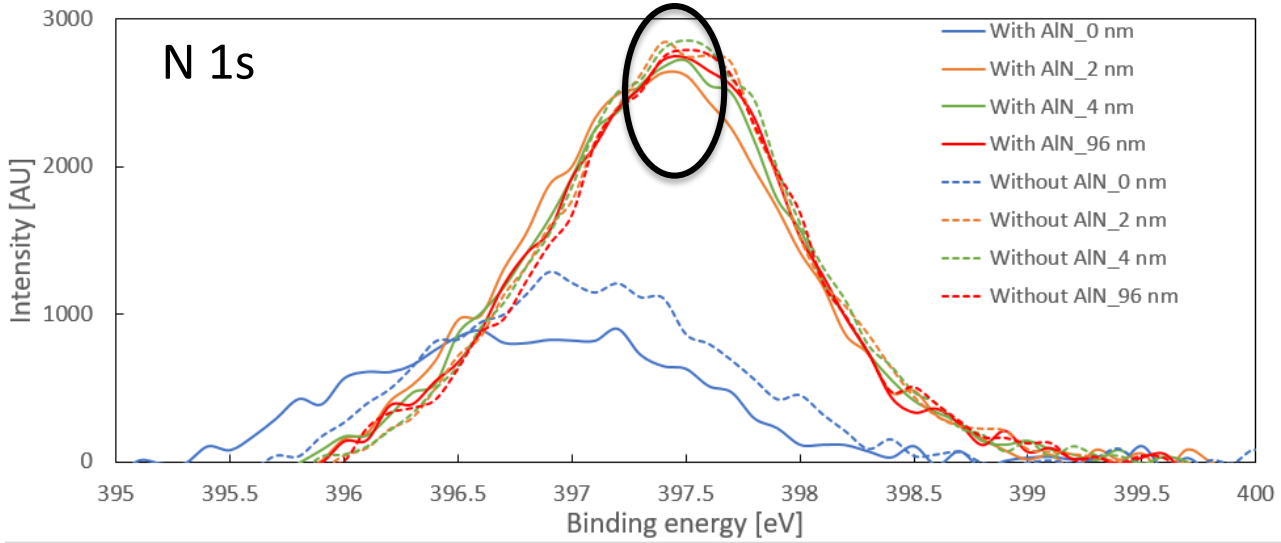


XPS surface profiling



- X-ray photoelectron spectroscopy
- Relative peak intensity determines element concentration
- Observe a Nb concentration gradient as a function of depth
- Ti and N concentrations are constant





Conclusions

- Provided overview of alternative materials study at Cornell
- Demonstrated stoichiometric Nb_3Sn with low surface roughness via Sn electrochemical deposition and thermal conversion
- Achieved recrystallization via annealing of sputtered Nb_3Sn and V_3Si
- Observed Nb deficiency as a function of depth in NbTiN/AlN/Nb SIS structure

Acknowledgements

- This work is supported by Center of Bright Beams from the National Science Foundation under Grant No. PHY-1549132.
- This work made use of the Cornell Center for Materials Research Shared Facilities which are supported through the NSF MRSEC program (DMR-1719875).
- This work was performed in part at the Cornell NanoScale Facility, a member of the National Nanotechnology Coordinated Infrastructure (NNCI), which is supported by the National Science Foundation (Grant NNCI-2025233).
- Thank M. Sowa (Veeco-CNT) for providing the SIS samples.

Thank you for your attention!