Growth studies at Northwestern of vapor diffusion samples from Fermilab

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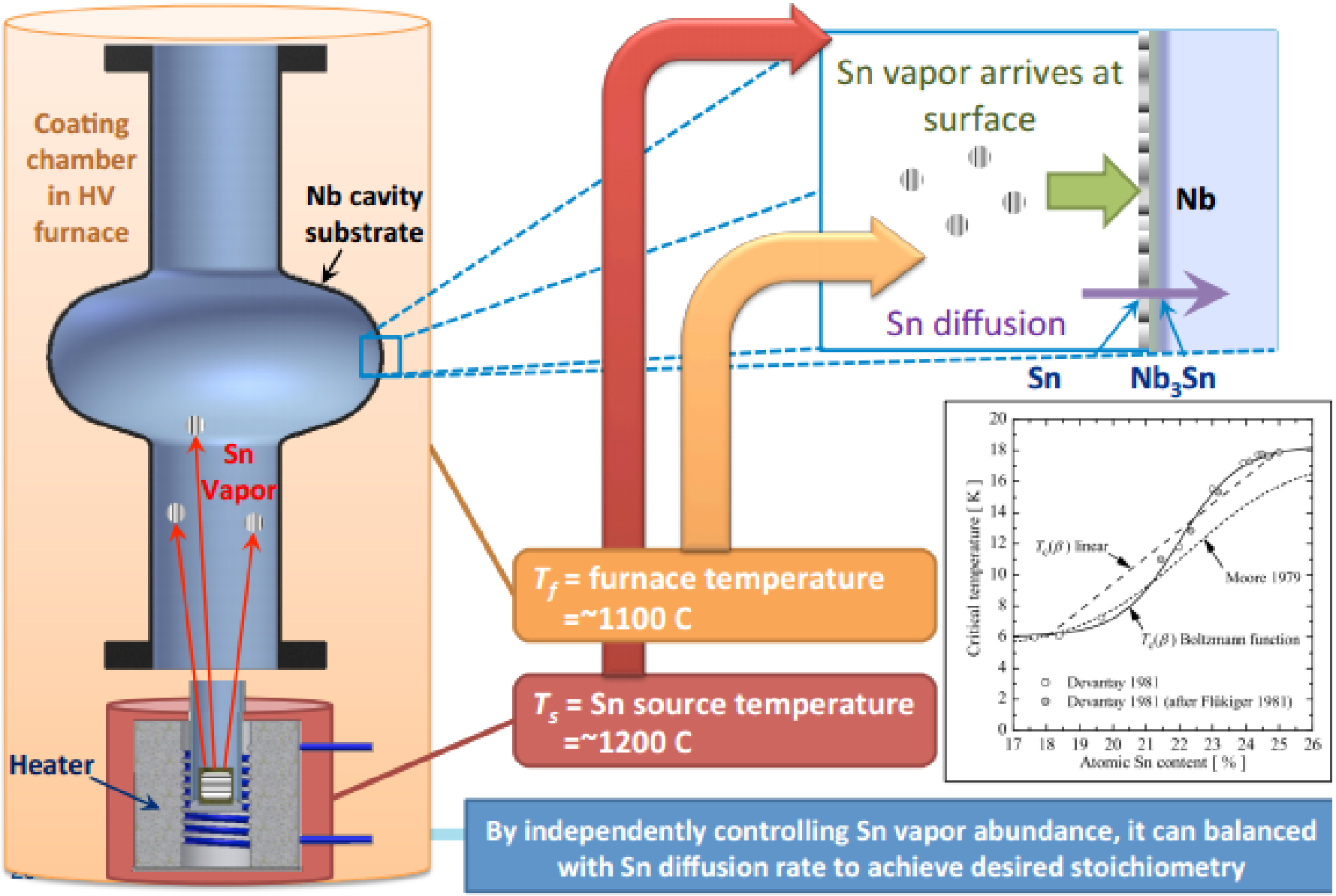
funded by DOE High Energy Physics and Early Career Award (Sam Posen)

Outline

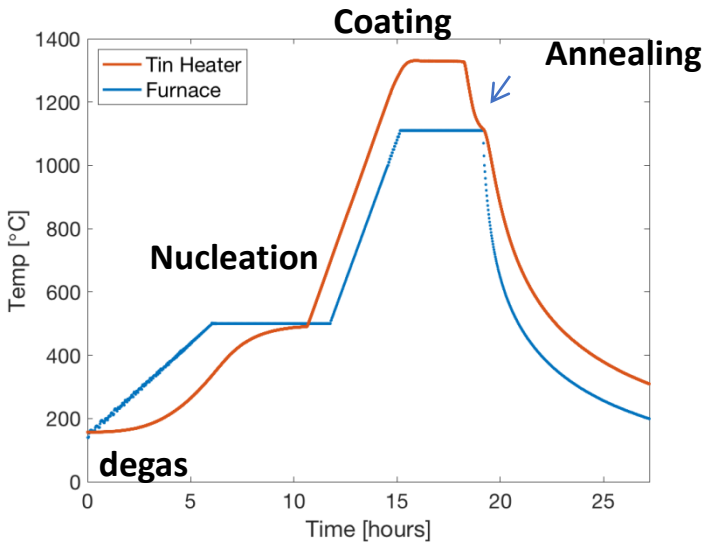
1. Introduction : growth process of Nb_3Sn
2. Detrimental microstructures of Nb_3Sn coatings
    1) Patchy regions
    2) Grain boundaries
    3) Surface roughness & film thickness
3. Conclusion

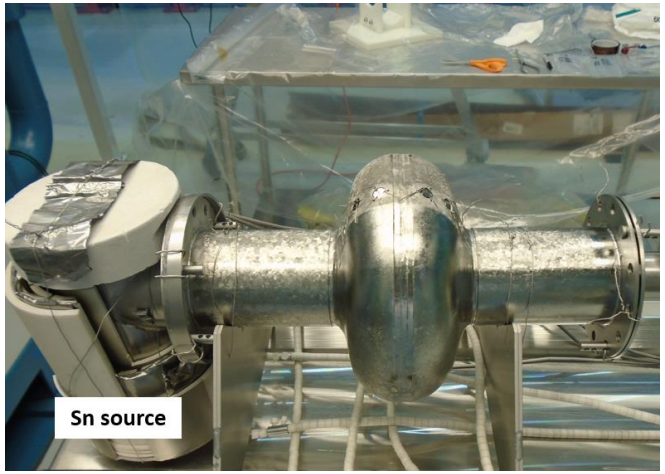
Nb_3Sn coating on Nb : vapor diffusion process



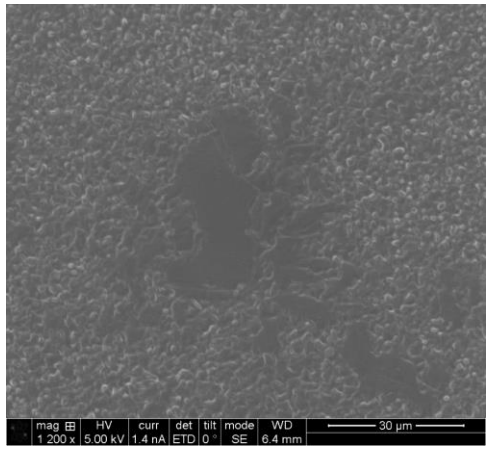
slide from Dr. Sam Posen

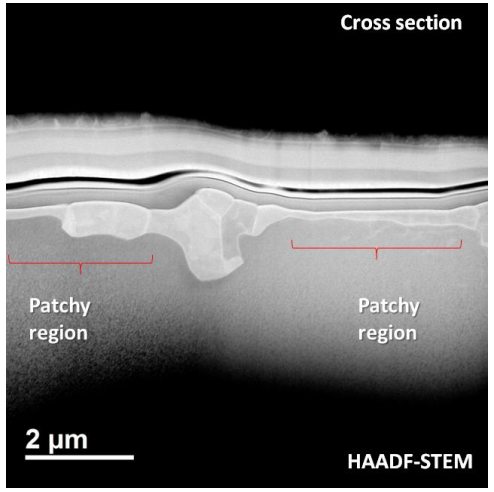
Effect of Sn supply on the growth process of Nb_3Sn coating



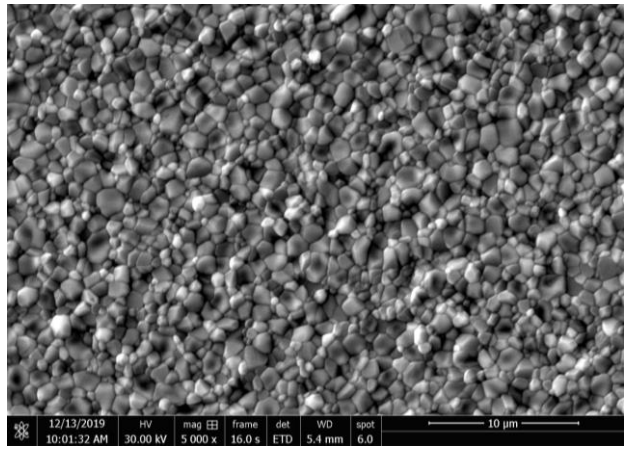


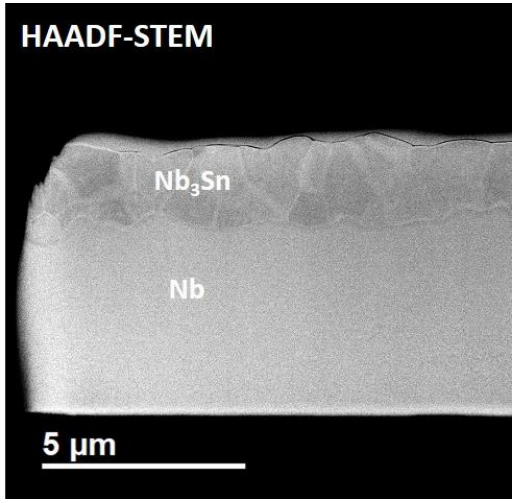
Patchy regions



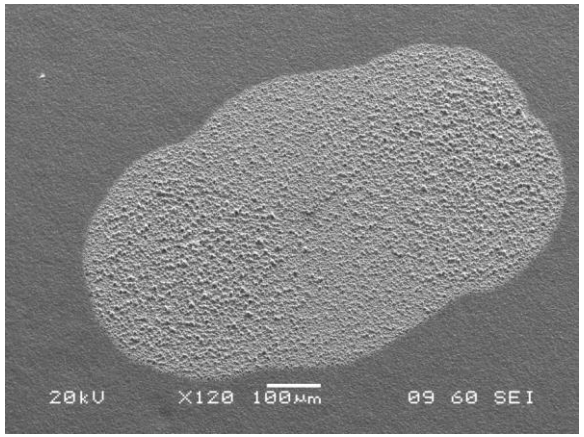


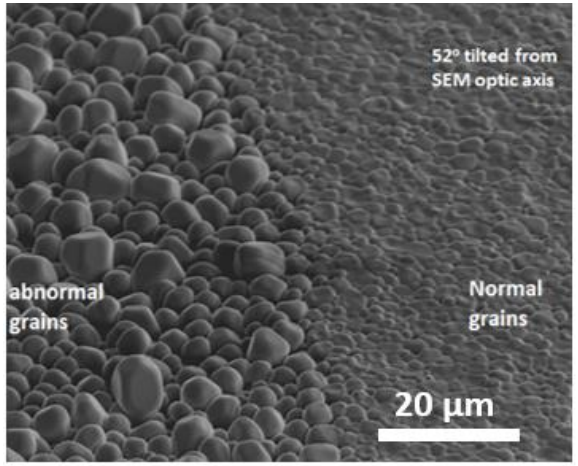
Uniform coatings





Sn spots



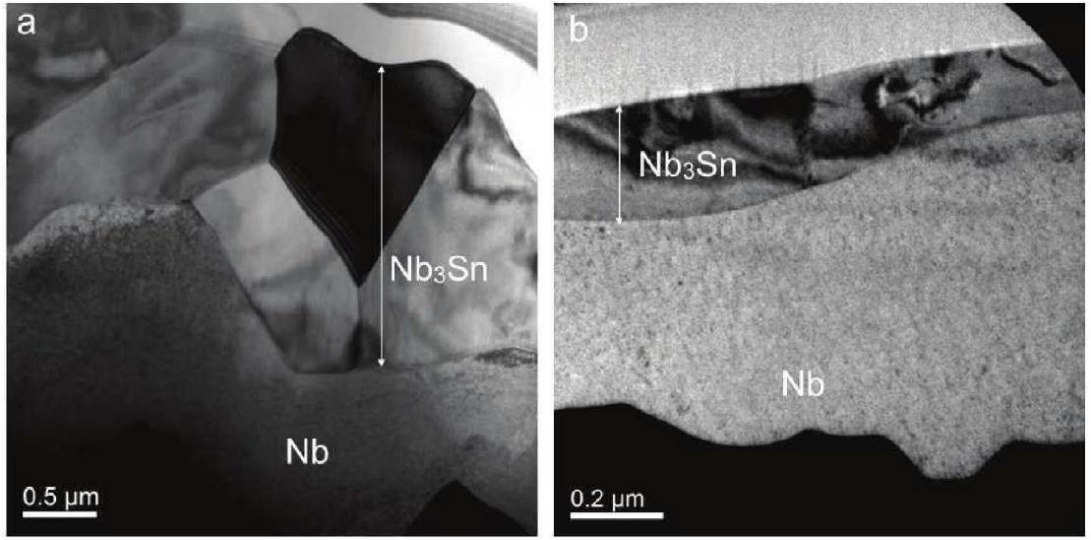


Low → **High**

Sn supply

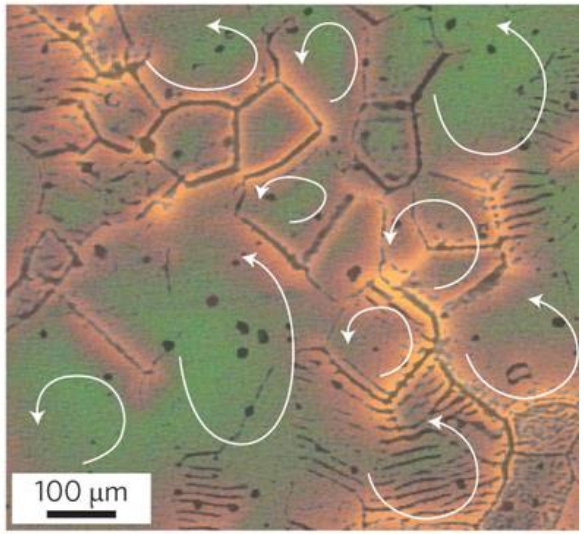
Possible detrimental defects in Nb_3Sn coatings

1. Thin regions



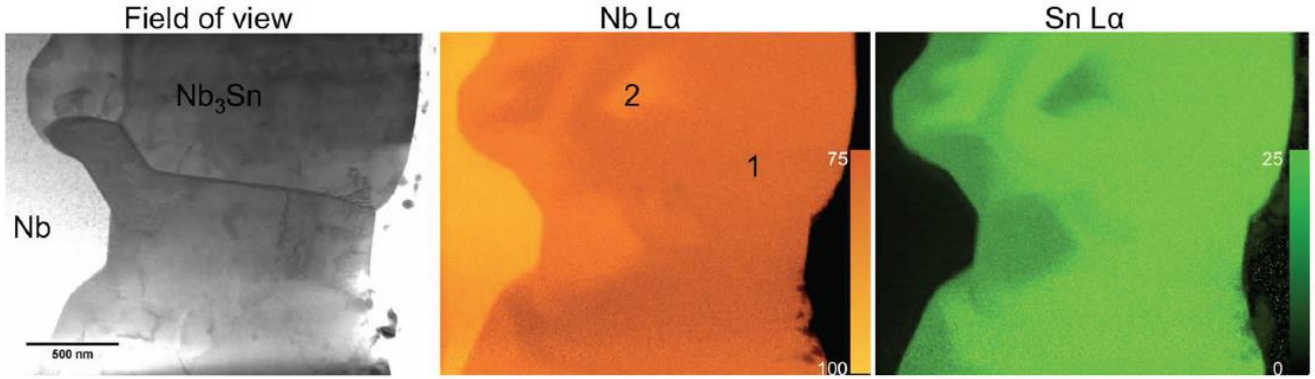
Y Trenikhina et al 2018 Supercond. Sci. Technol. 31 015004

2. Grain boundary



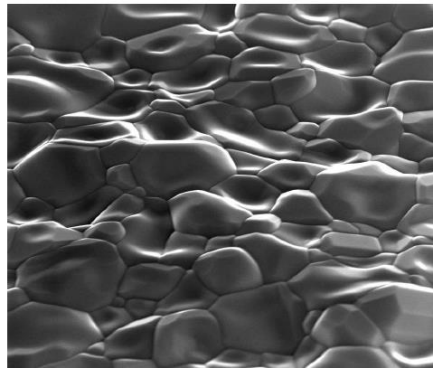
A. Gurevich, Nature Materials 10, 255–259 (2011)

3. Composition variation (Sn-deficient region)



C Becker et al, APL 106, 082602 (2015)

4. Surface roughness



S Posen, PhD thesis (2015)

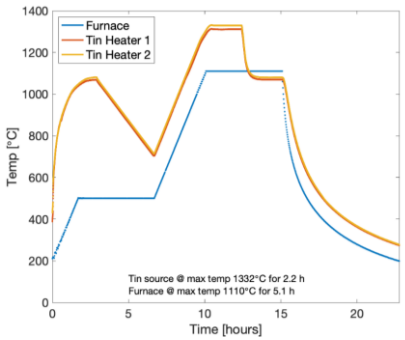
Development of Nb_3Sn SRF cavities

Goals Fabricating high-performance Nb_3Sn SRF cavities
Understanding of microstructures and detrimental defects in Nb_3Sn coatings to the performance

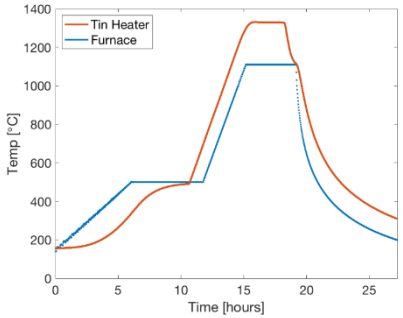
Processing

Coating parameters (Sn source, Temp., etc)

New procedure

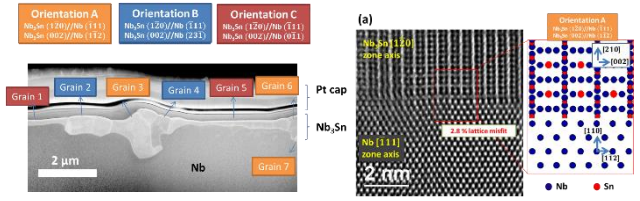


Previous standard procedure

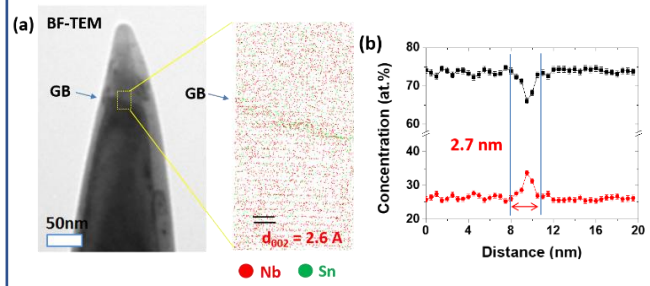


Microstructures

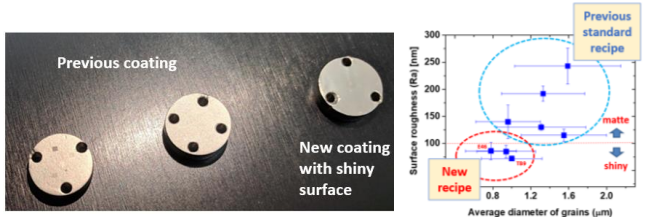
Patchy region



Grain boundary

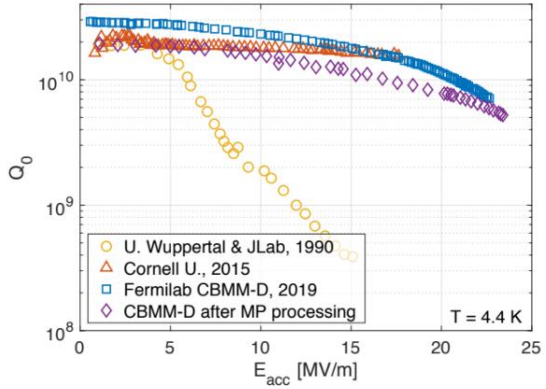


Surface roughness & thickness



Properties

High-performance cavity



NUCAPT
Northwestern University Center for Atom-Probe Tomography

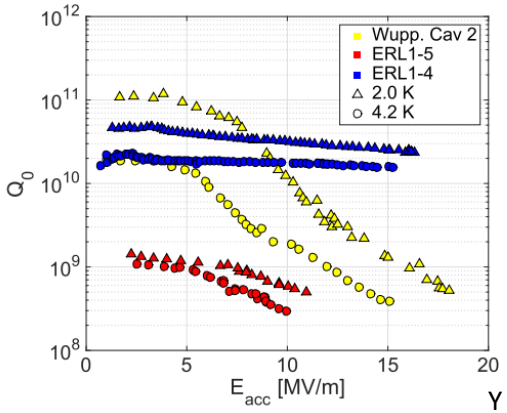
NUANCE
Atomic and Nanoscale Characterization Experimental Center

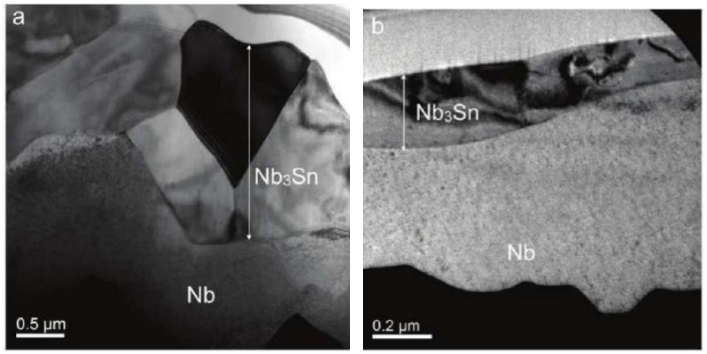
1. Patchy regions

Low Sn flux → Epitaxial growth of Nb_3Sn on Nb

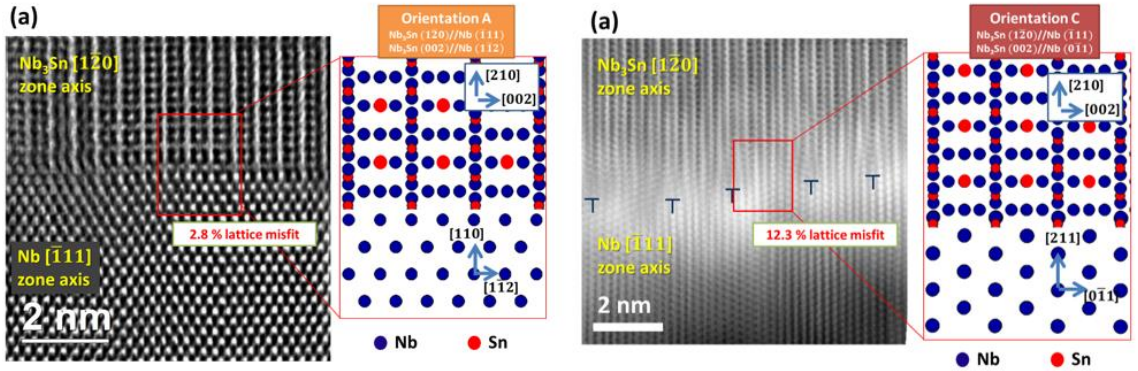
Proper amount of Sn flux is important for uniform coating.

Formation of patchy region



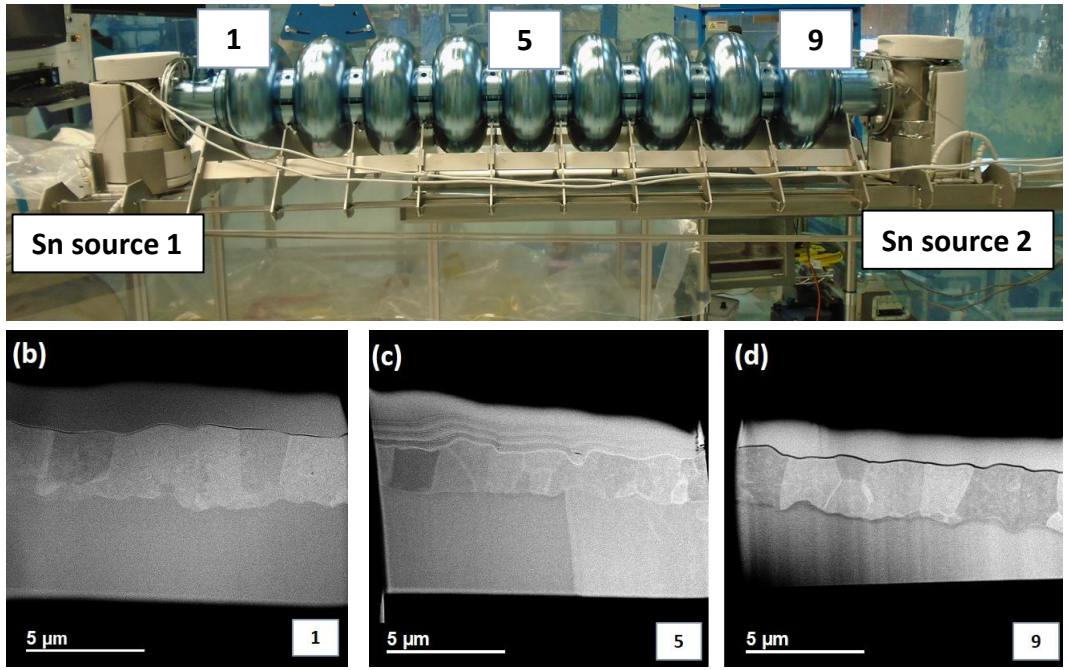


Y Trenikhina et al., Supercond. Sci. Technol. 31 015004 (2018)



J. Lee, DN Seidman, S Posen et al., SuST (2019)

Uniform 9-cell Nb_3Sn cavity coating

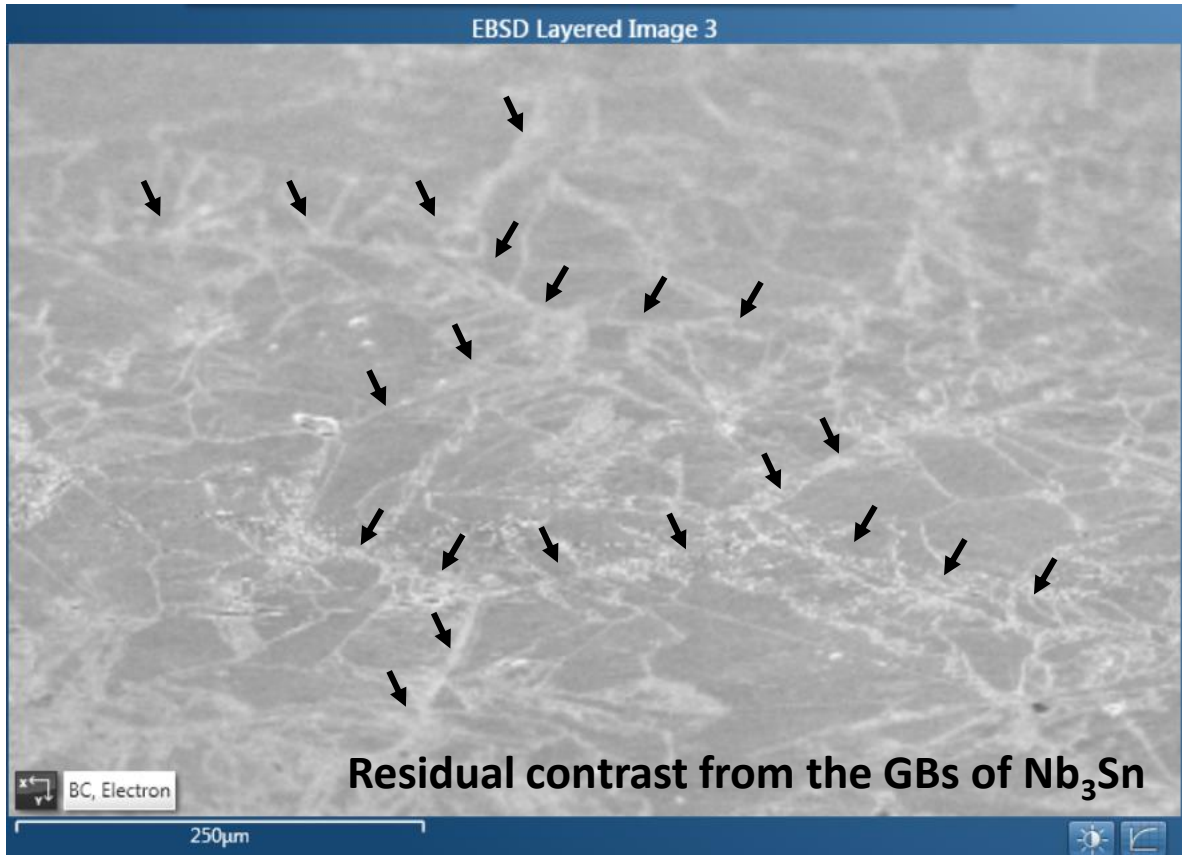


Uniform 1m long 9-cell cavity coating acheived

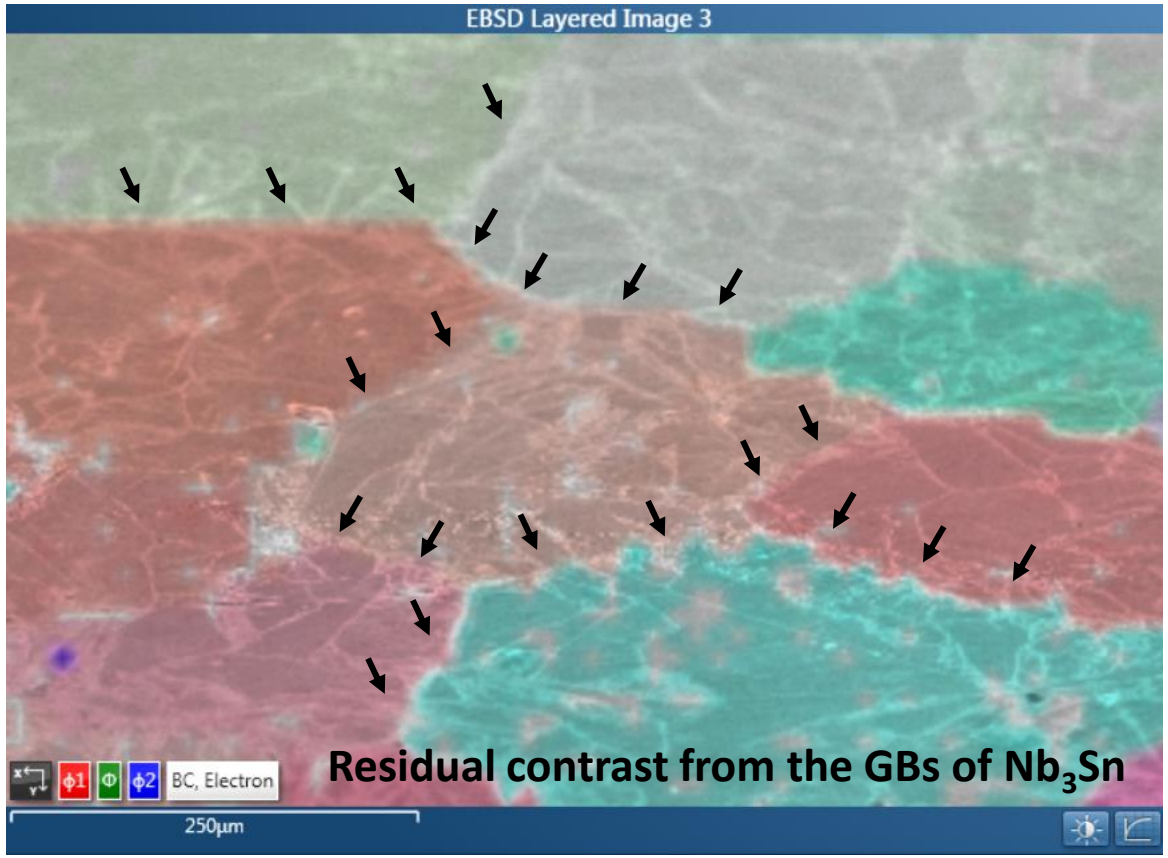
T. Spina et al. SuST (2020)

1. Patchy regions

Low Sn flux → Epitaxial growth of Nb_3Sn on Nb



< SEM image of Nb substrate >



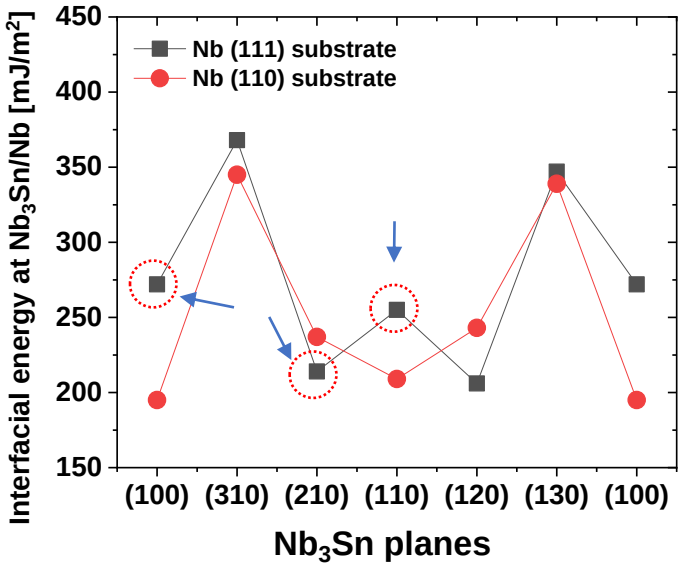
< Overlays of EBSD map and SEM image of Nb substrate >

There is a correlation between GBs of Nb_3Sn coatings and the Nb substrate
→ When Sn flux is low, Nb orientation play an important role in the texture of Nb_3Sn

1. Patchy regions

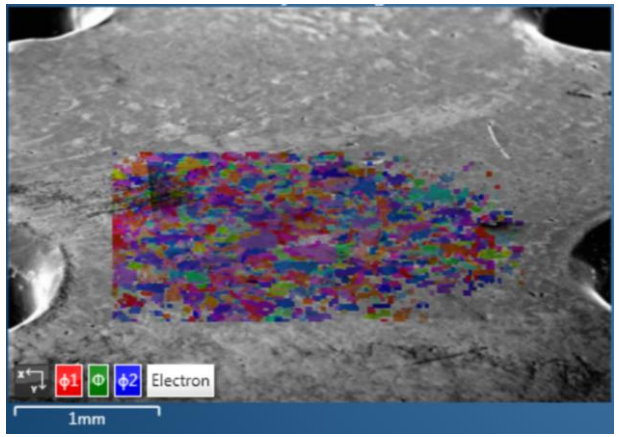
Roles of Nb/Nb_3Sn interfacial energy on textures of Nb_3Sn

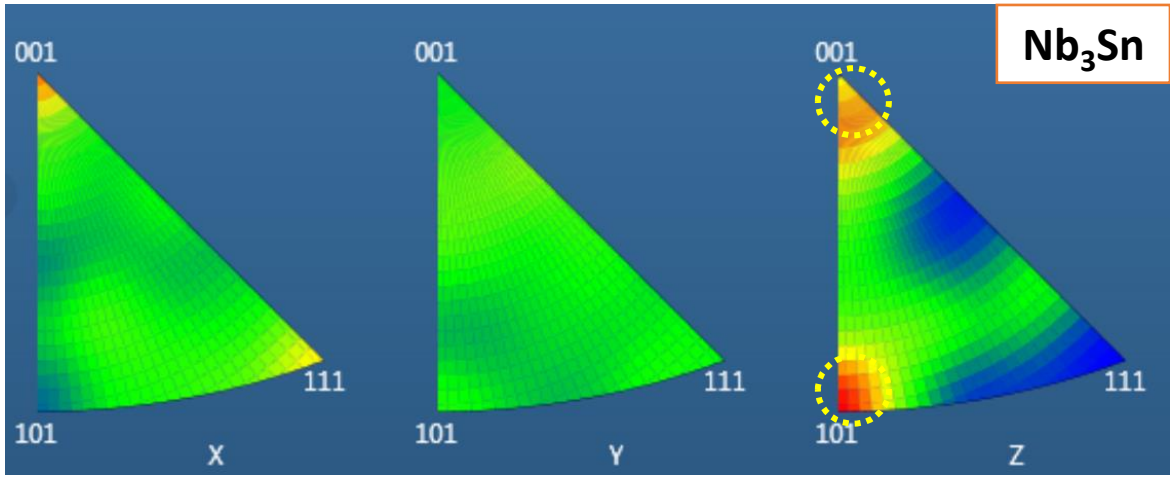
Interfacial energy at Nb/Nb_3Sn using Monte Carlo simulation (by Zugang Mao)



*Modified Embeded Atom Method (MEAM) Nb-Sn potential from Prof. Michael Baskes

EBSD analyses of Nb_3Sn texture on Nb (111) substrate





MC simulation predict favorable texture of (100), (210) and (110) on Nb (111) surface

EBSD analyses also display (001) and (110) texture of Nb_3Sn on Nb (111) substrate

*note that 210 texture is not significant in EBSD, which disagree with simulation

1. Patchy regions

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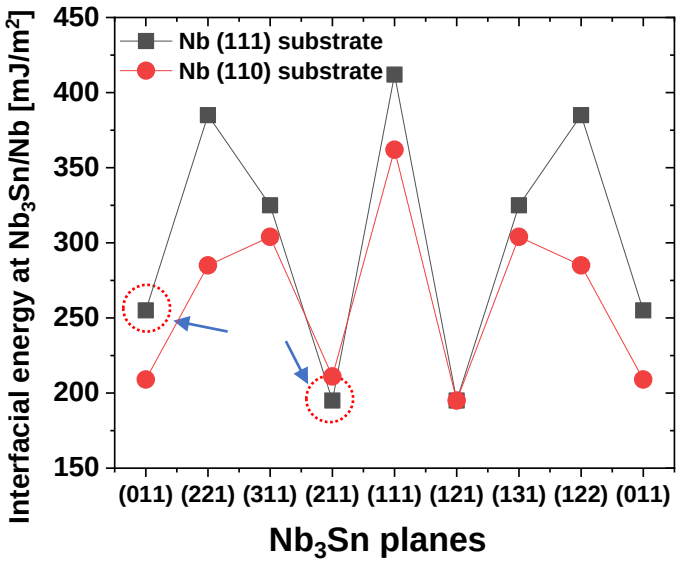

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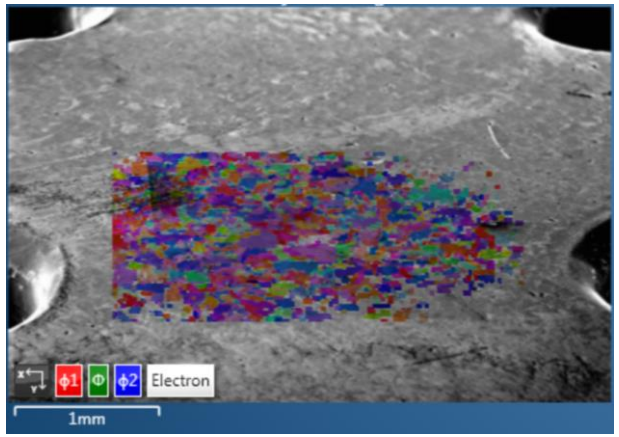
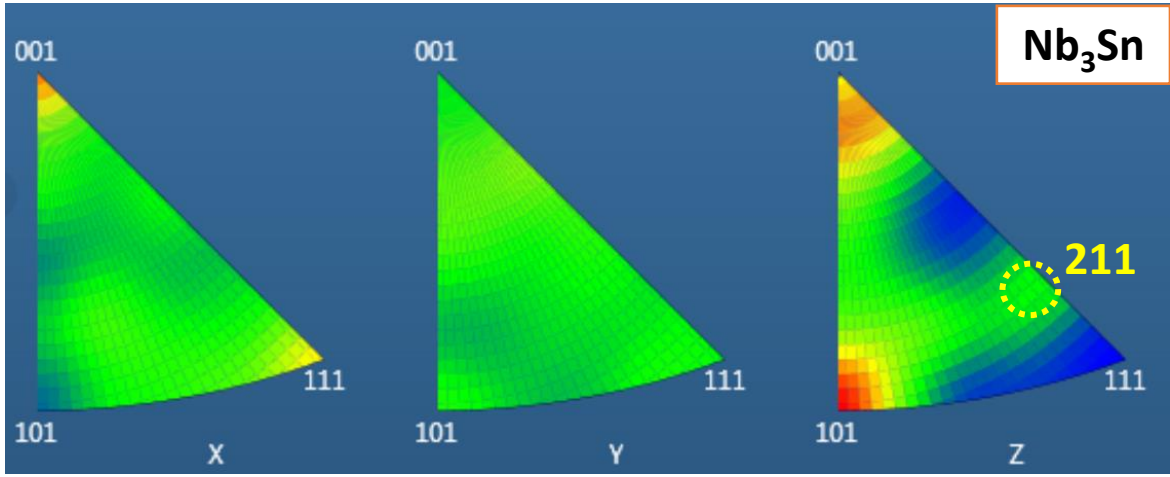
EBSD analyses of Nb_3Sn texture on Nb (111) substrate





MC simulation also predict favorable texture of (211) on Nb (111) surface

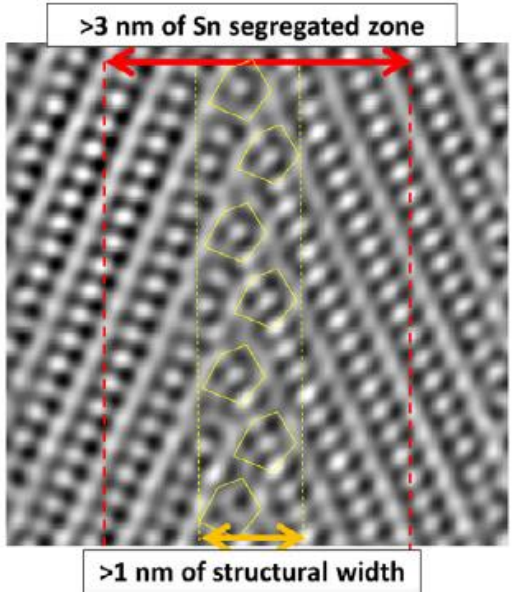
→

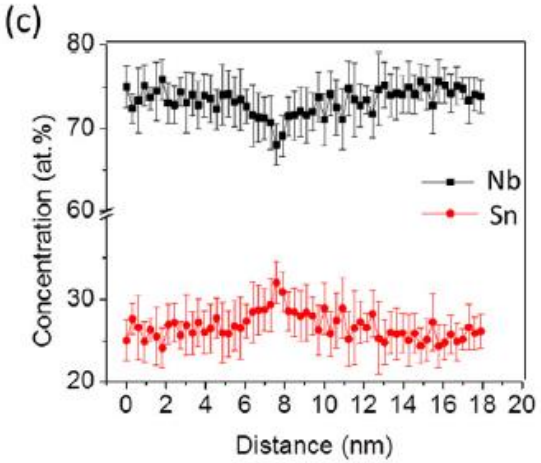
EBSD analyses also display (211) texture of Nb_3Sn on Nb (111) substrate

2. Grain boundaries (GBs) of Nb_3Sn and Q-factor of SRF cavities

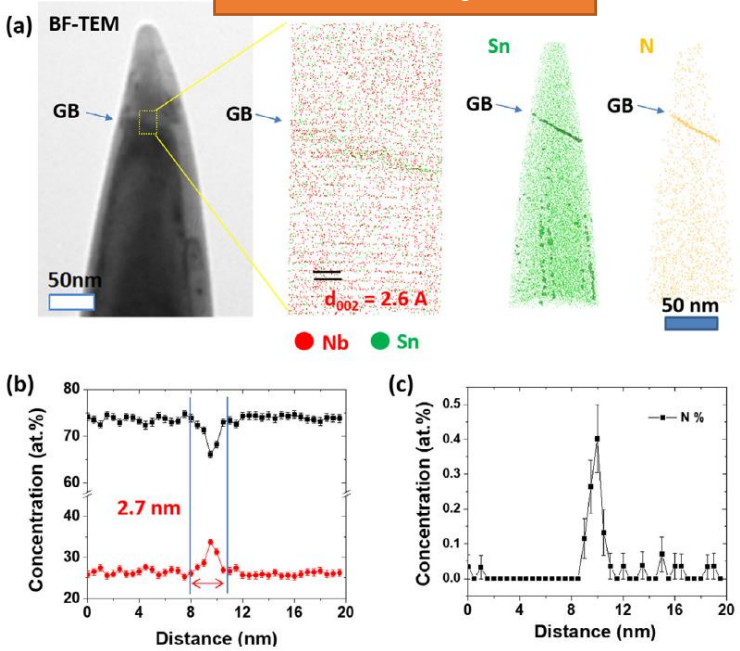
1. *How defects at GBs look like compared to the coherence length of Nb_3Sn (~3nm) ?*
2. *What cause the compositional variation in GBs?*
3. *How we control?*
4. *How is the correlation between GBs and the performance of cavities?*

HR-TEM analysis





APT analysis



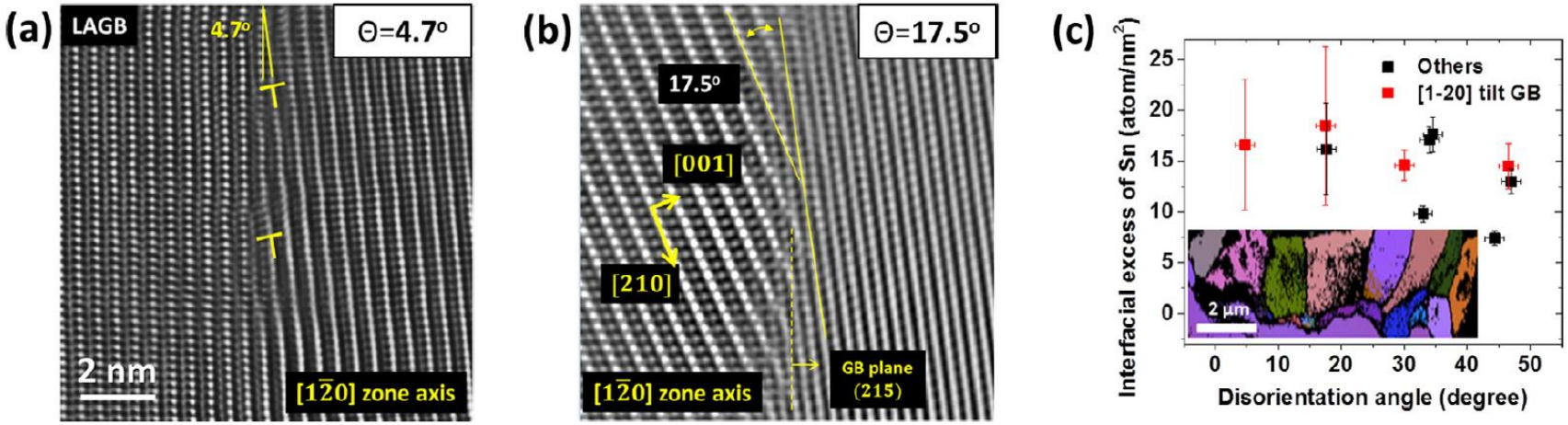
J Lee, DN Seidman, S Posen et al *et al, Acta Materialia (2020)*

2. Grain boundaries (GBs) of Nb_3Sn and Q-factor of SRF cavities

1. How defects at GBs look like compared to the coherence length of Nb_3Sn ?
2. **What cause the compositional variation in GBs?**
3. How we control?
4. How is the correlation between GBs and the performance of cavities?

Thermodynamic origin: GB structure

Sn segregation vs disorientation



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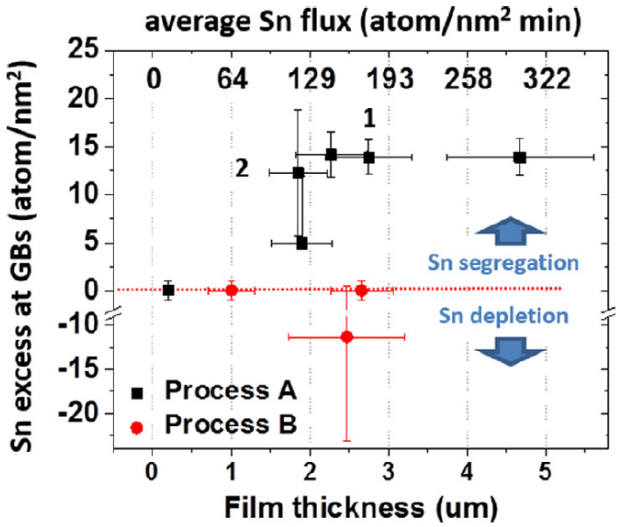
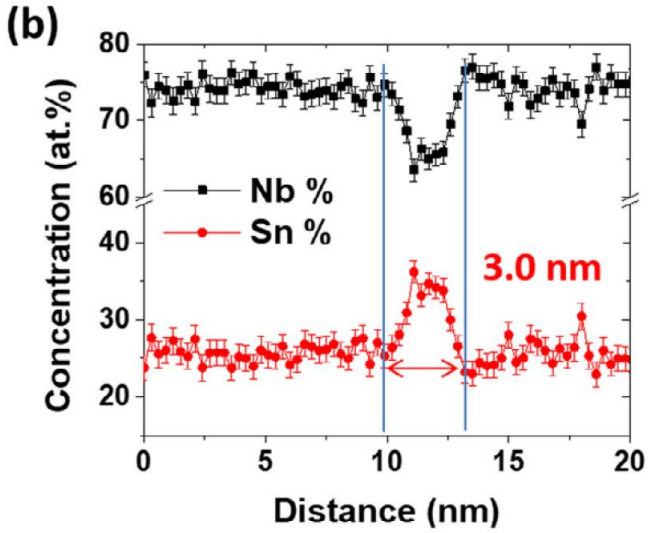
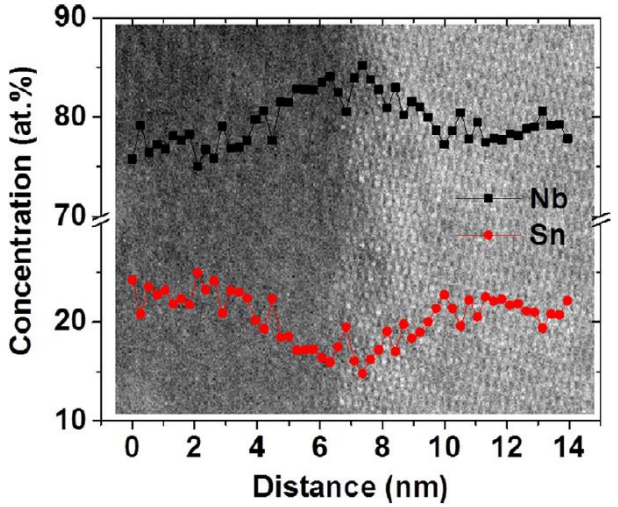
Kinetic origin: GB diffusion of Sn and Nb



As-coated



Anealing at 1300C for 3hrs



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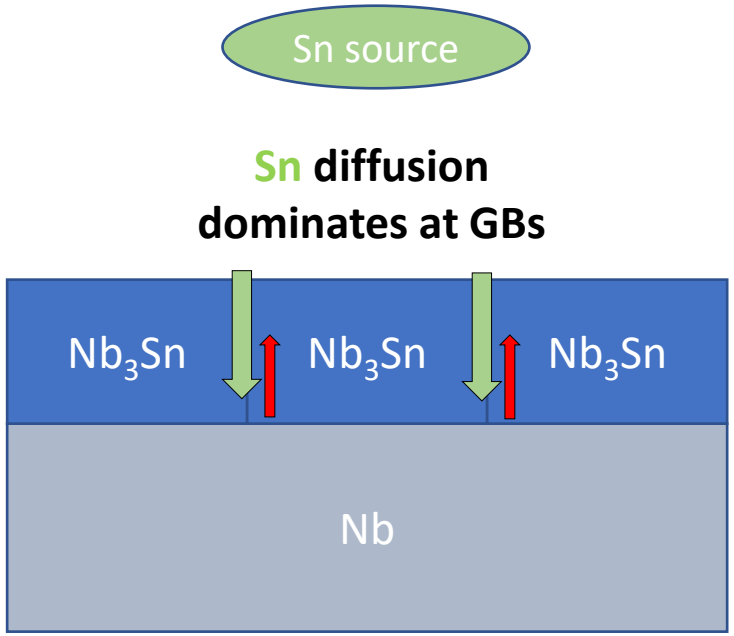
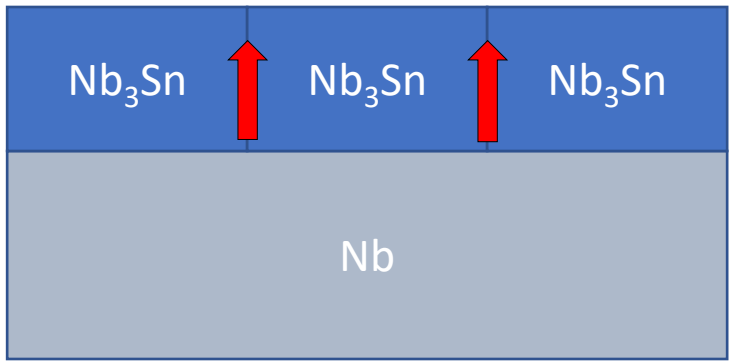
During coating process



During annealing



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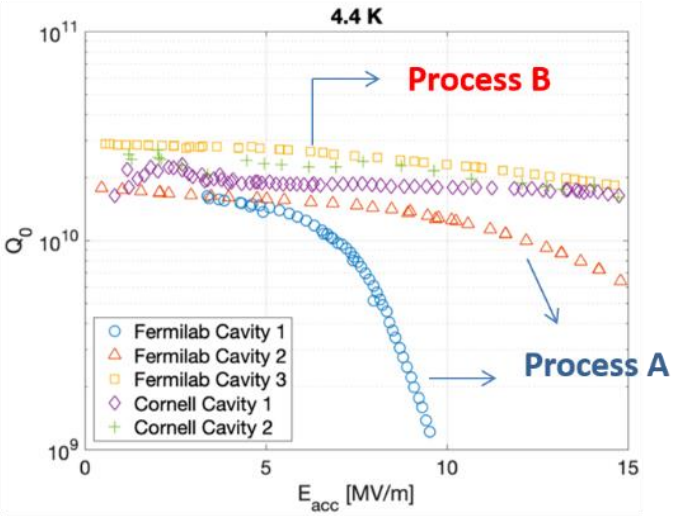
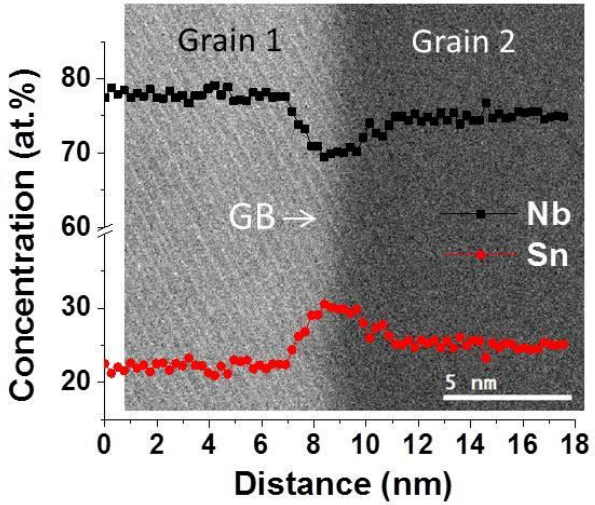
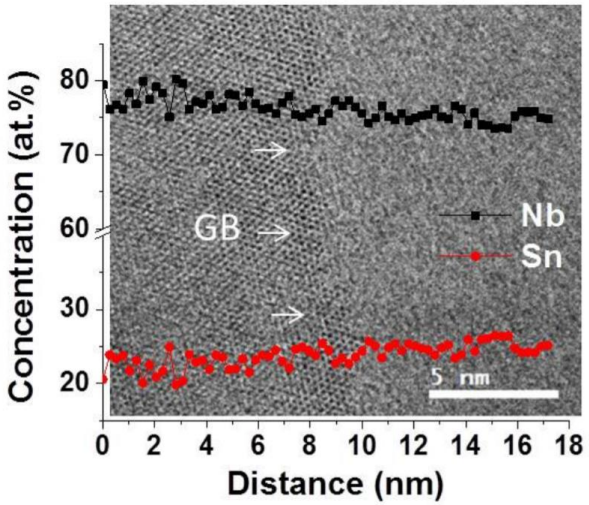


Process A



Process B



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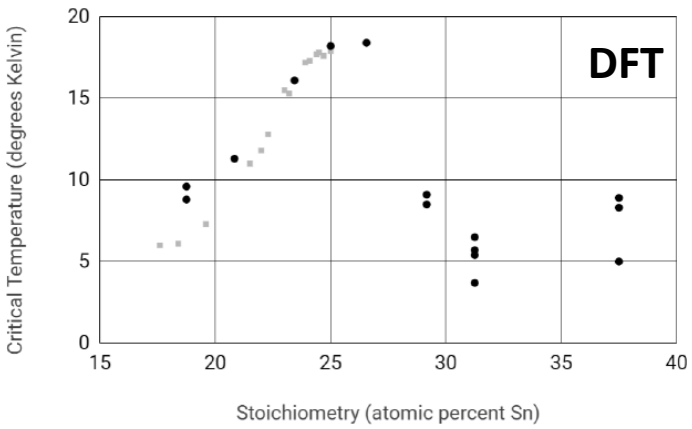
4. ***How is the correlation between GBs and the performance of cavities?***

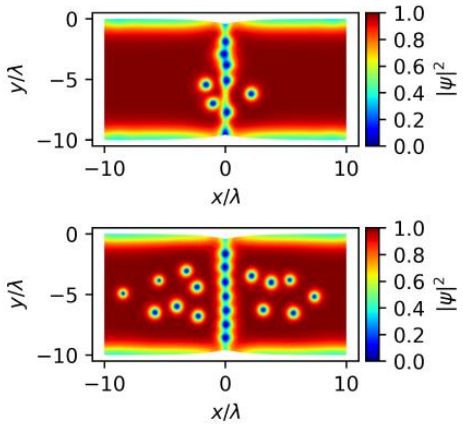
Theoretical approach to understand possible flux penetration into Sn-segregated GBs of Nb_3Sn

T_c vs Sn concentration (N. Sitaranaman)

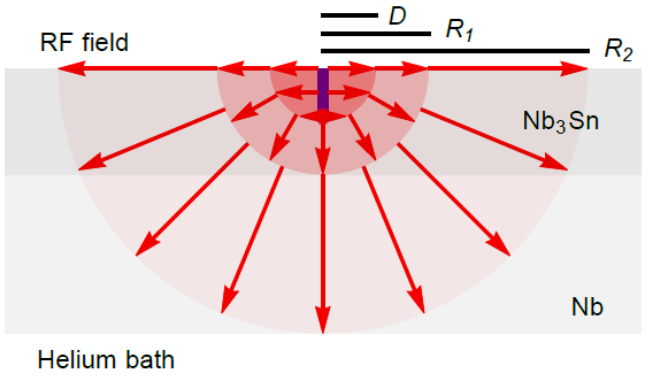


Vortice penetration into GB (J. Carlson)

Ginzburg-Landau model



Estimation of Heat dissipation (D. Liarte)



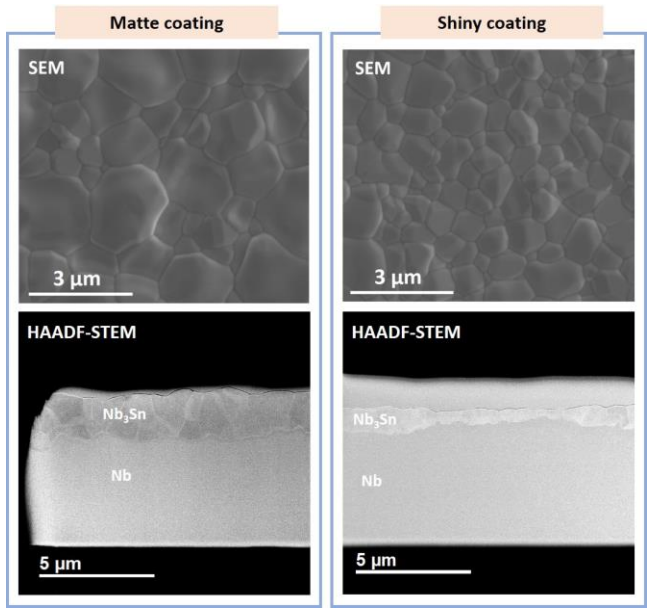
J. Carlson et al, arXiv 2003.03362 (2020)

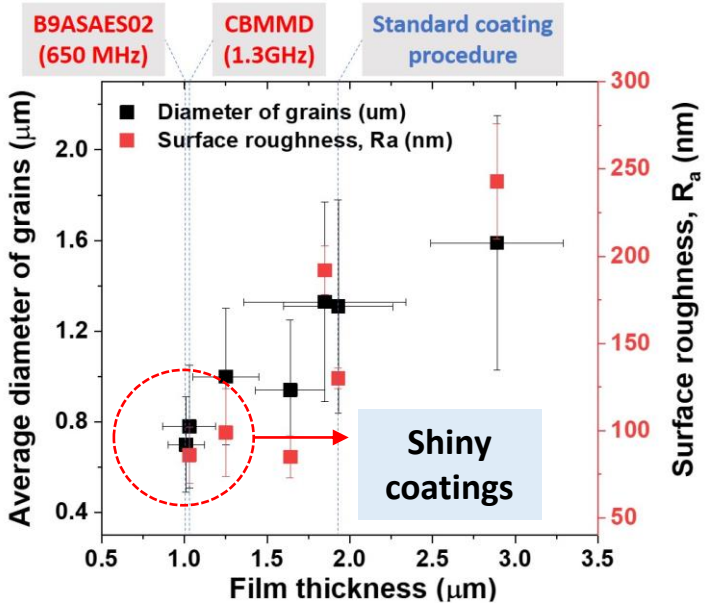
3. Surface roughness & thickness for high-performance Nb_3Sn SRF cavities

New shiny coatings improve the performance of cavity significantly.

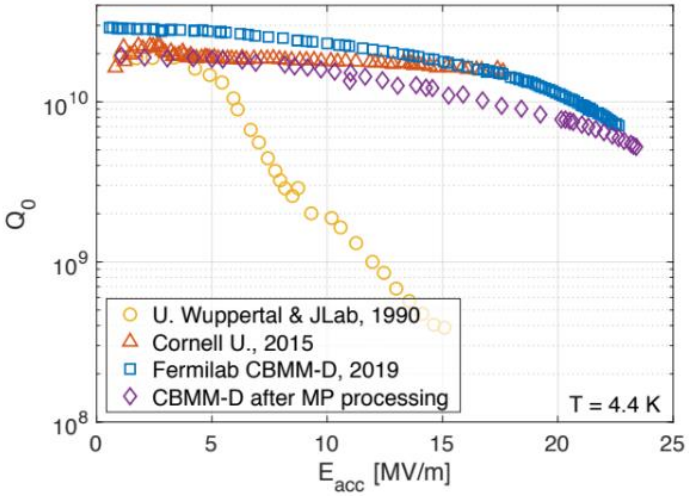
(i) Surface roughness
(ii) Thin thickness

Microstructure of shiny coatings





Improved cavity performance



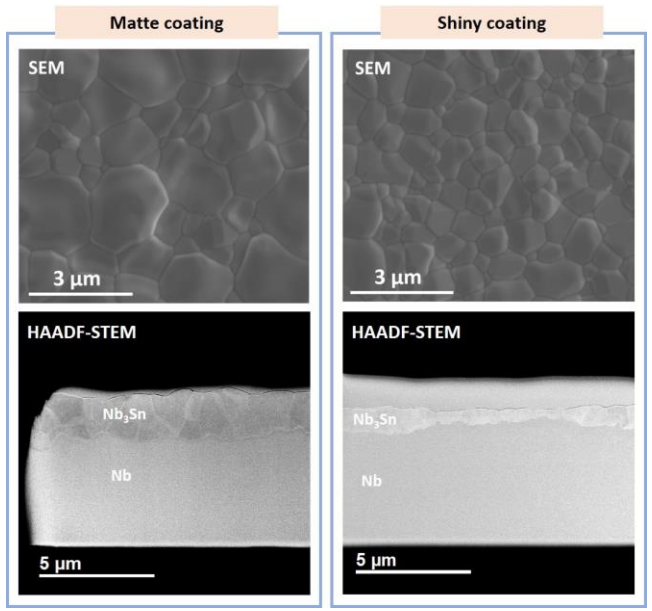
S. Posen et al., SuST accepted

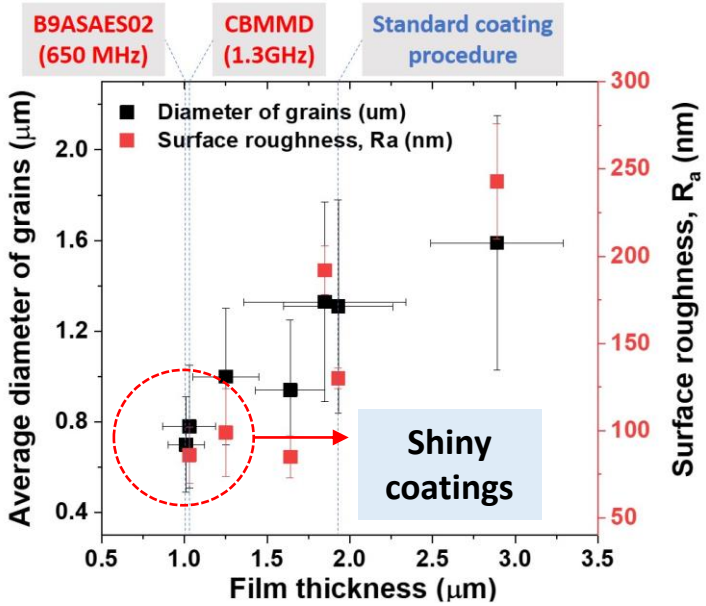
3. Surface roughness & thickness for high-performance Nb_3Sn SRF cavities

New shiny coatings improve the performance of cavity significantly.

(i) Surface roughness
(ii) Thin thickness

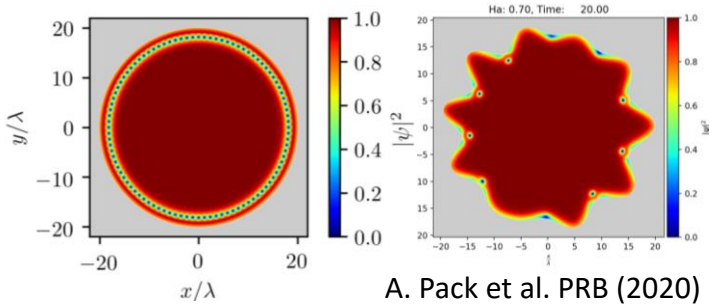
Microstructure of shiny coatings





Improved cavity performance

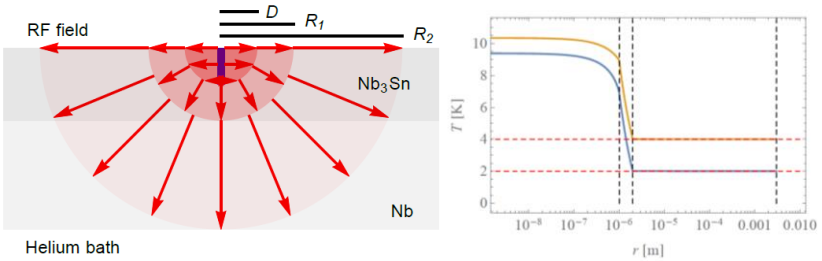
1. Surface roughness vs Vortex nucleation



A. Pack et al. PRB (2020)

2. Improved thermal conducting property

Done by D. Liarte



J. Carlson, arXiv 2020

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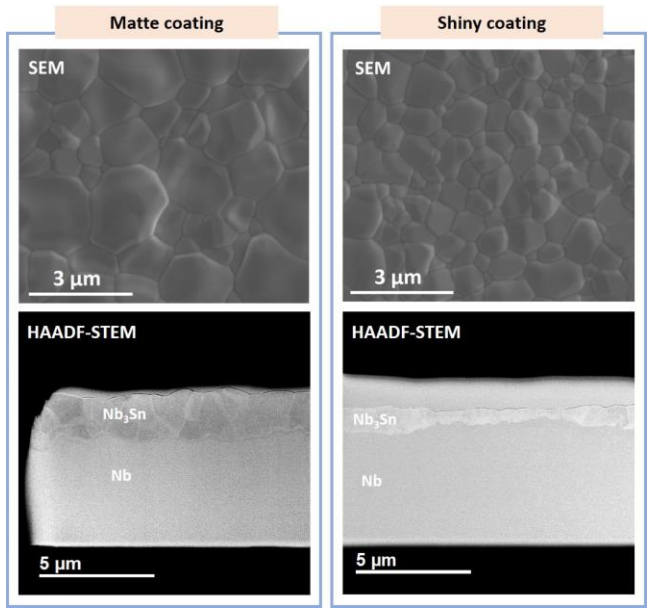
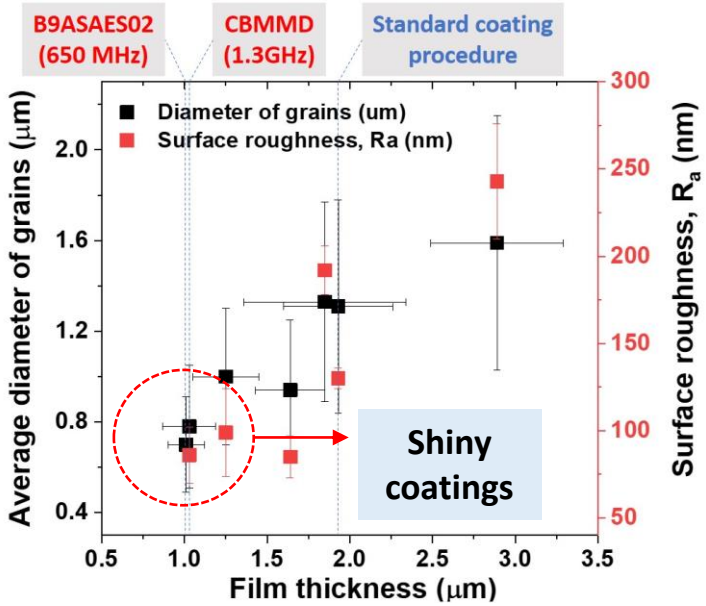
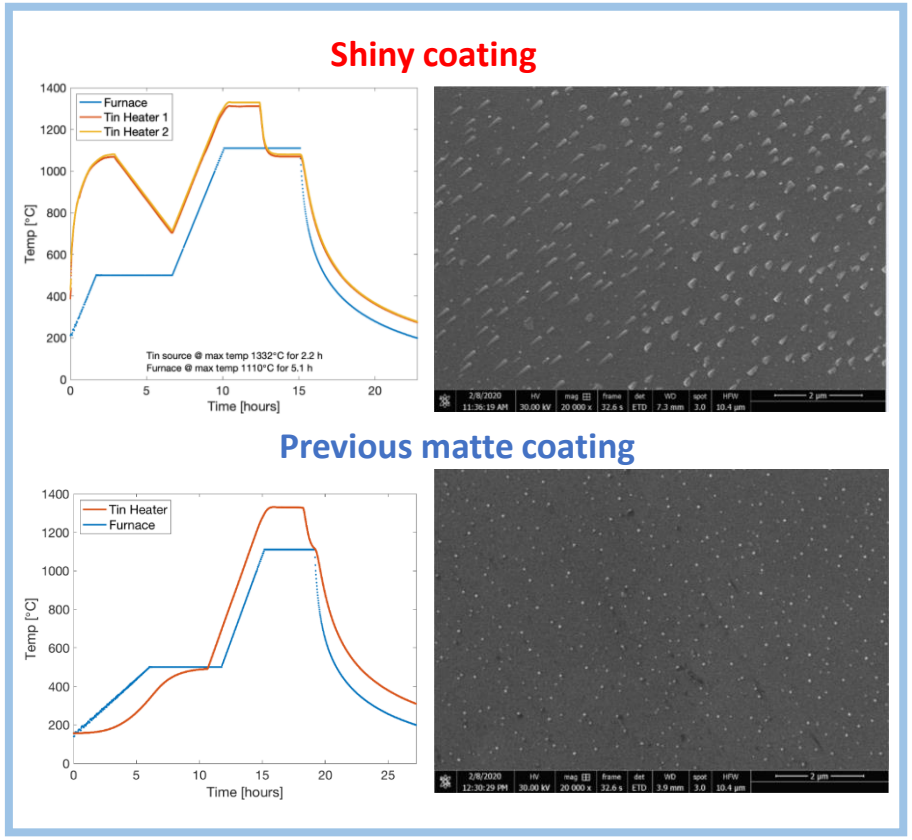


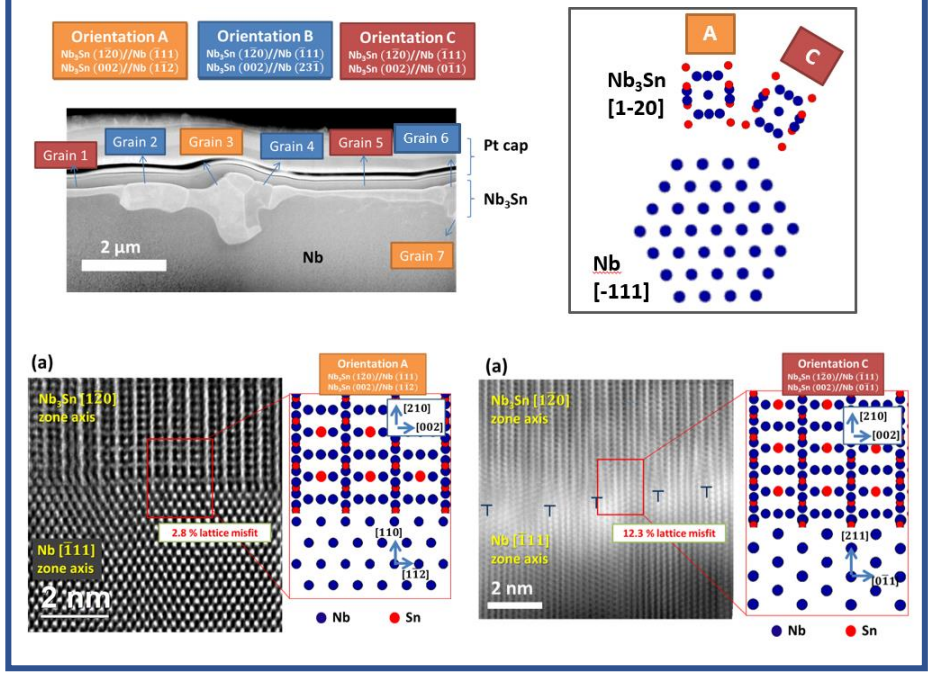




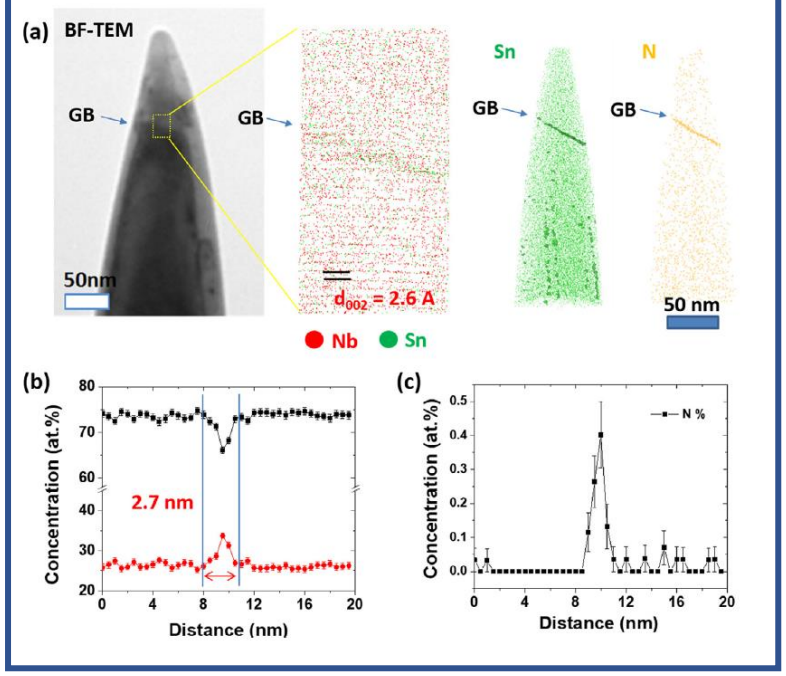
Summary

1. Sn supply play important roles in the microstructure of Nb_3Sn coatings on Nb during vapor diffusion process
2. Microstructural control of Nb_3Sn coatings can lead to the improvement of Nb_3Sn SRF cavity performance

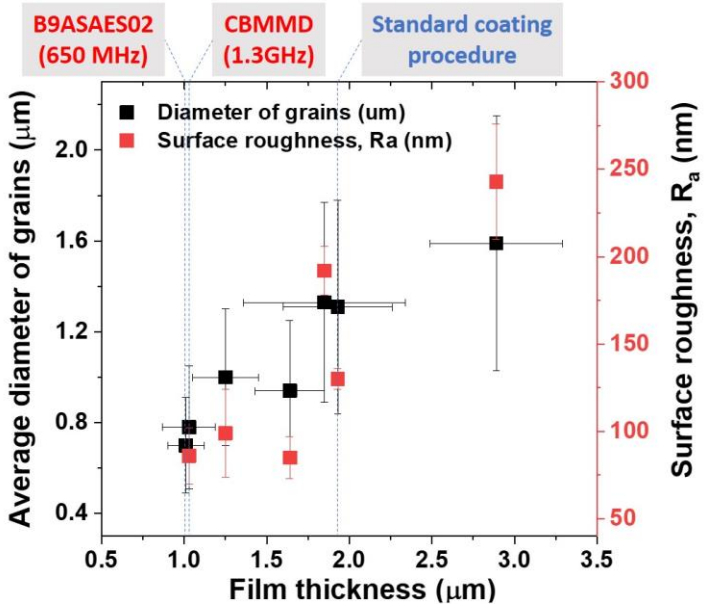
Patchy regions



Grain boundaries



Surface roughness & film thickness



Acknowledgement

Co-advised by Prof. David N Seidman (Northwestern) and Dr. Sam Posen (Fermilab)

Funded by DOE Early Career Award (Dr. Sam Posen)

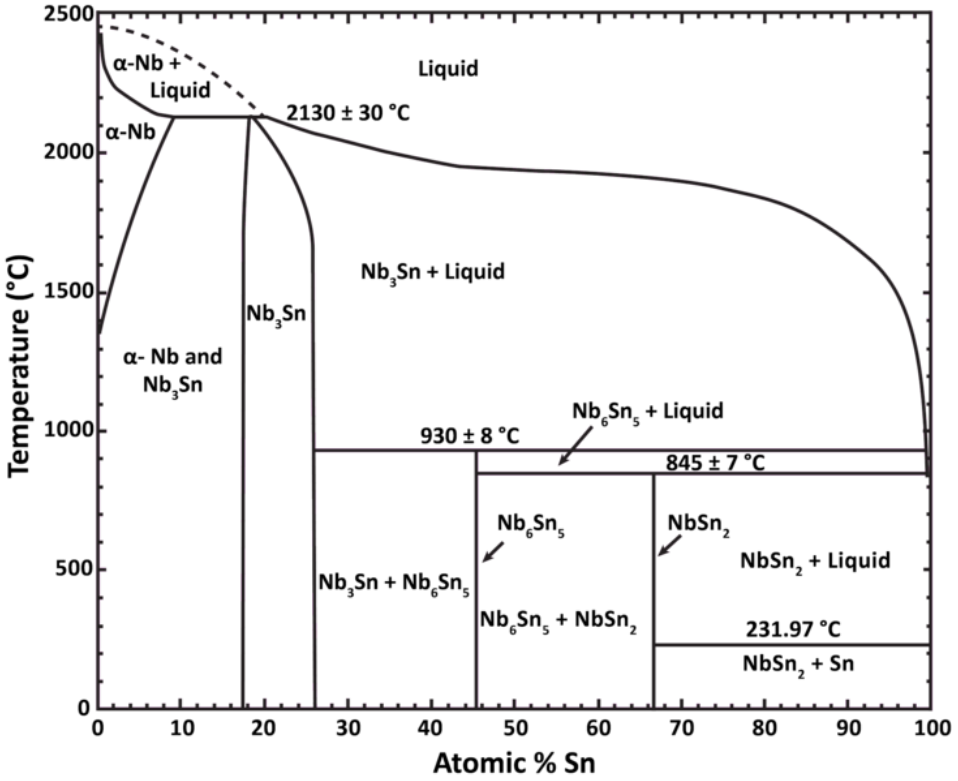
Thanks to

- ***Nb_3Sn cavity : Brad Tennis, Dr. Tiziana Spina***
- ***Electron Microscopy : Drs. Kai He (TEM) and Amir Farkoosh (EBSD)***
- ***Atom Probe Tomography (APT) : Drs. Dieter Isheim and Sung-Il Baik***
- ***Simulation : Dr. Zugang Mao, Prof. Michael Baskes***
- ***Collaborations: Profs. James Sethna, Thomas Arias, Matthias Liepe's groups (Cornell), and Mark Transtrom's group (BYU)***

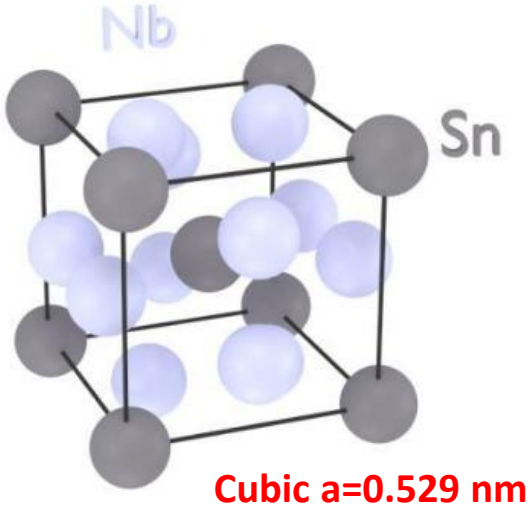
Thank you

Supplementary slides

Nb_3Sn: structure, composition, and properties



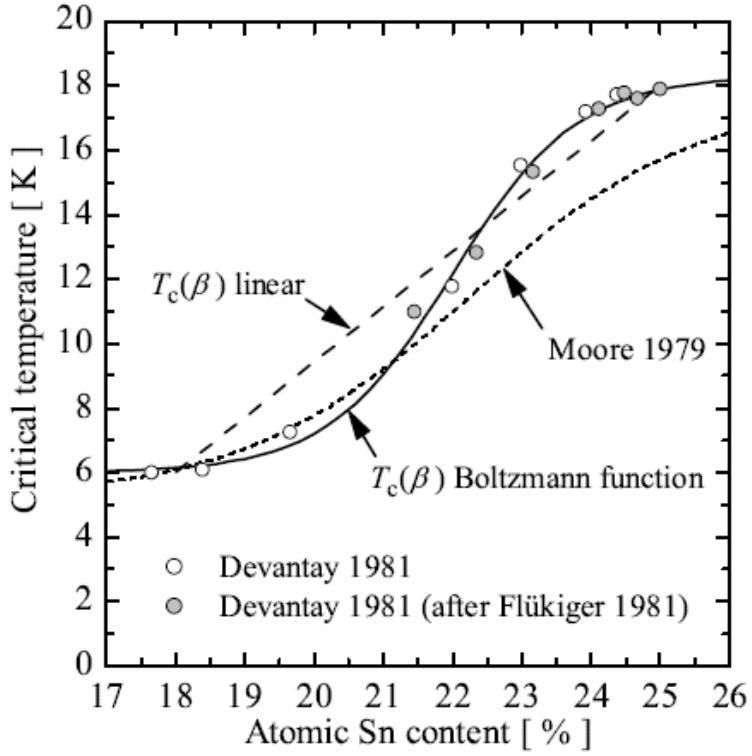
Wide range of composition: 17-28 at.% Sn



Cubic a=0.529 nm

	Formation energy
Nb antisite	0.268 eV
Sn antisite	0.637 eV

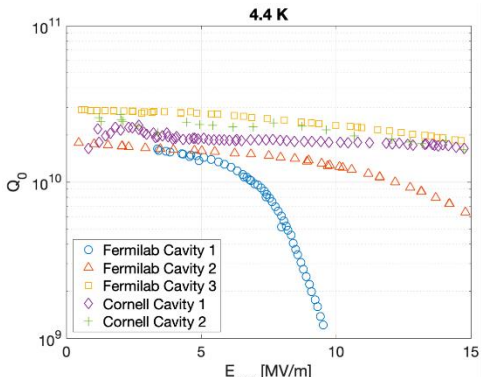
** DFT calculation by Z Mao*

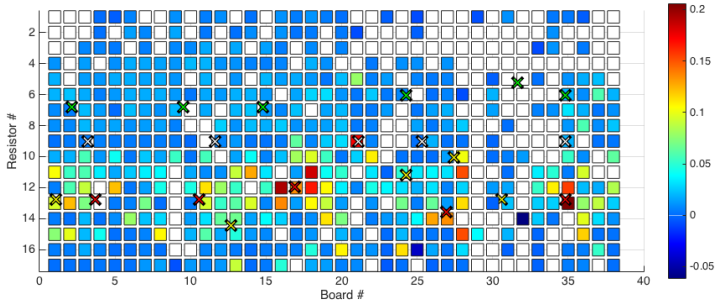


Nb_3Sn research at Fermilab-NU

Nb_3Sn cavity coatings and test

- **Fabrication of Nb_3Sn cavities via Sn-vapor diffusion**
- **SRF cavity performance test**



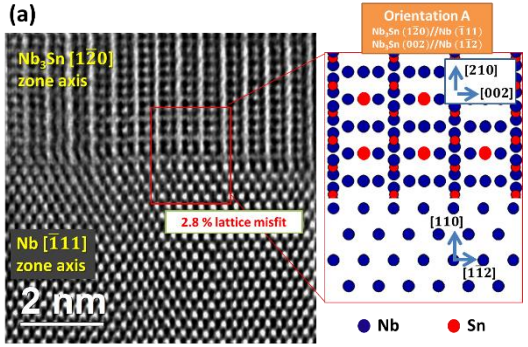


Fermilab

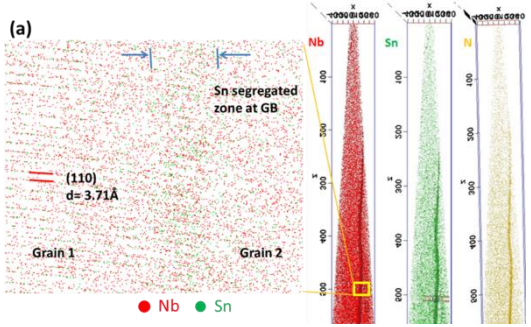
Materials research of Nb_3Sn

- **Atomic-scale analyses of Nb_3Sn coatings on Nb utilizing advanced microscopy**
- Transmission electron microscopy (TEM)
- Atom probe tomography (APT)
- Electron backscatter diffraction (EBSD)
- X-ray photoelectron spectroscopy (XPS)

NUANCE
Atomic and Nanoscale Characterization Experimental Center



NUCAPT
Northwestern University Center for Atom-Probe Tomography

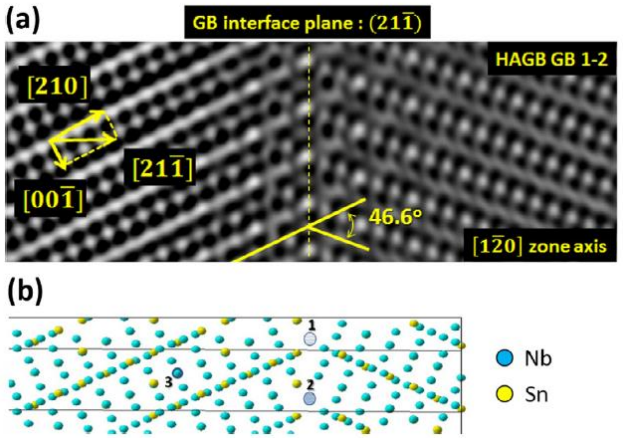


2. Grain boundaries (GBs) of Nb_3Sn and Q-factor of SRF cavities

1. How GBs look like compared to the coherence length of Nb_3Sn ?
2. **What cause the compositional variation in GBs?**
3. How we control?
4. How is the correlation between GBs and the performance of cavities?

Thermodynamic origin: (i) defect formation energy; (ii) GB structure

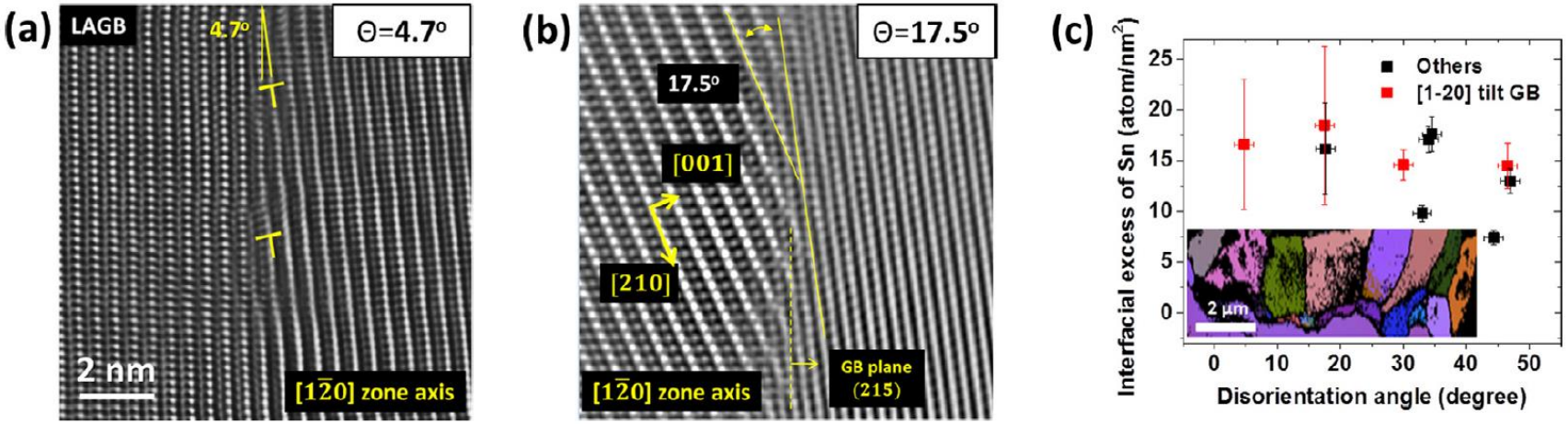
DFT calculation



	Formation energy
Nb antisite	0.268 eV
Sn antisite	0.637 eV

Courtesy of Z. Mao (NU)

Sn segregation vs disorientation

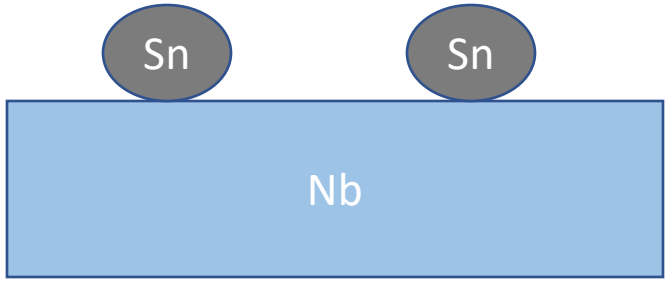


*J. Lee, Z. Mao, K. He, Z. Sung, S. Baik, T. Spina, D.L. Hall, M. Liepe, D.N. Seidman, S. Posen, Acta Materialia **188**, 155 (2020)*

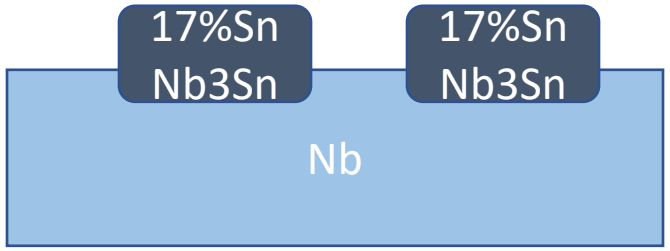
Proposed hypothesis of the formation of Sn-deficient regions in Nb_3Sn

Steps analyzed by TEM

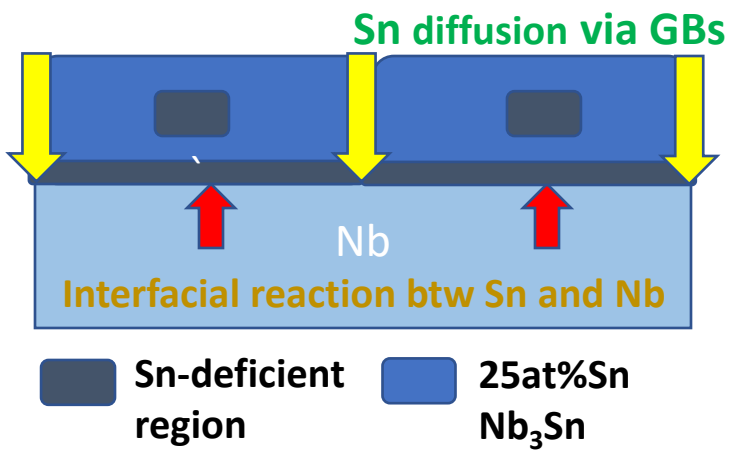
1. Formation of nucleation site



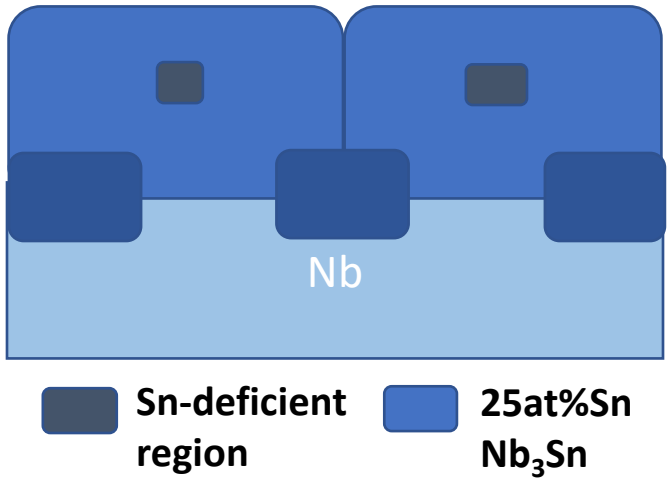
2. Nucleation of Nb3Sn



3. Grain growth of Nb3Sn



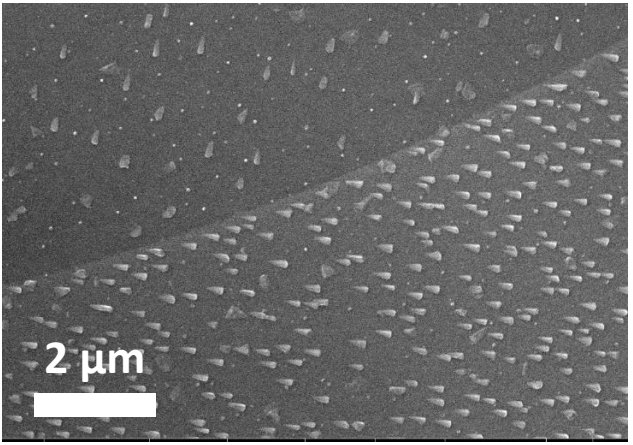
4. Completion of Nb3Sn coating



1. Sn-deficient regions might be formed during the early stage of Nb_3Sn formation.
2. Slow Sn-diffusion results in some of Sn-deficient left after the coating process

Uniformity of nucleation site: New recipe

#1



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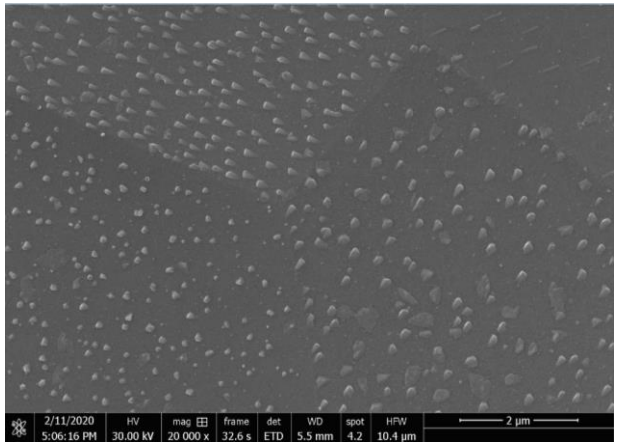

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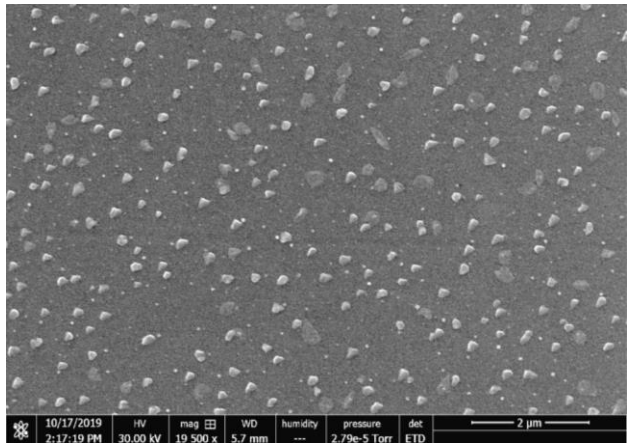

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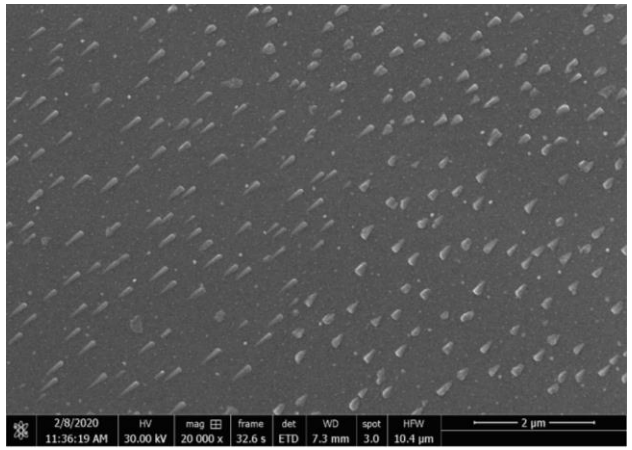

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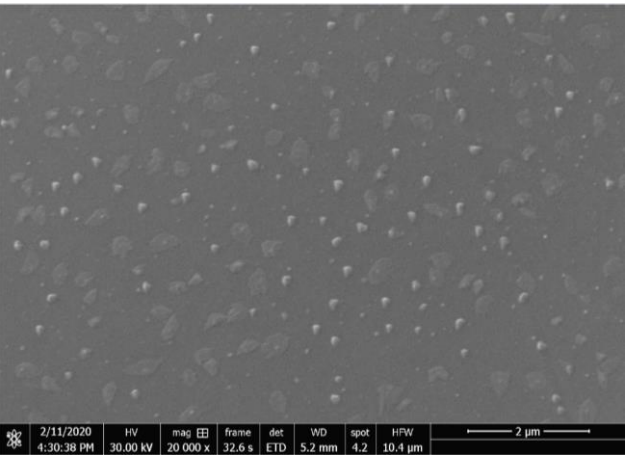

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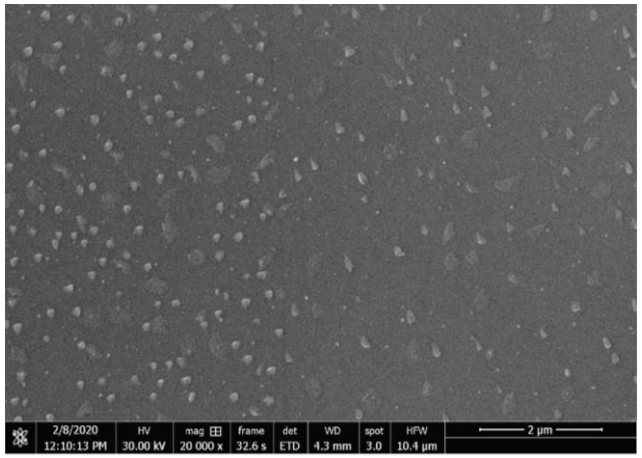

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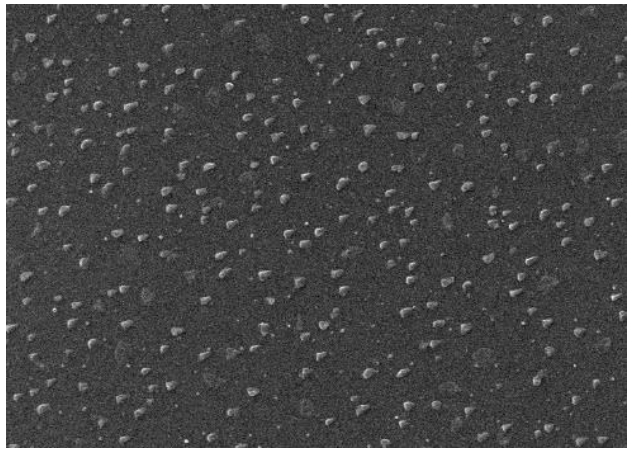

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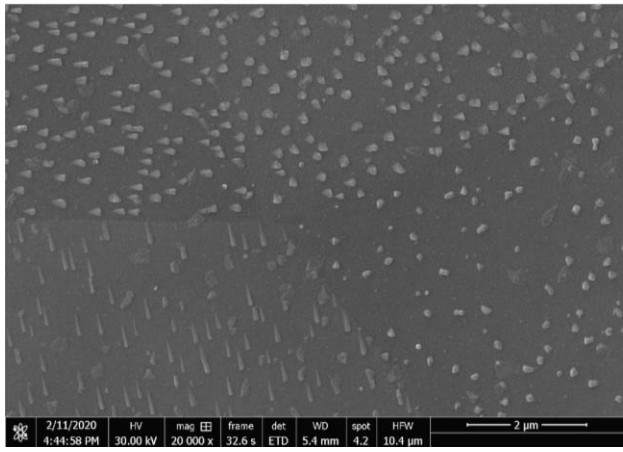
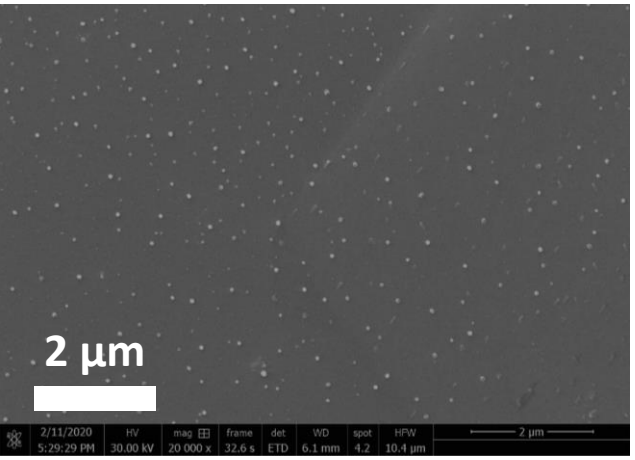


Fairly good uniformity of size and number density of nucleation sites in HT nucleation samples

Uniformity of nucleation site: Previous standard recipe

#1



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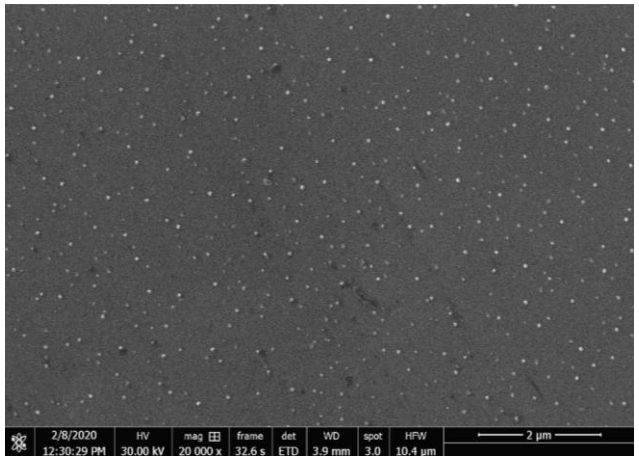

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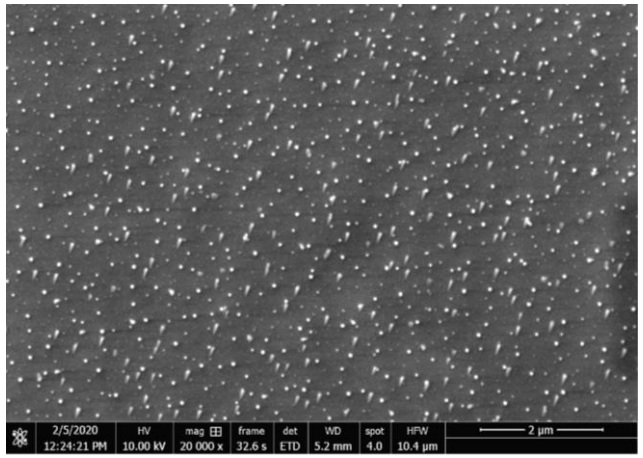

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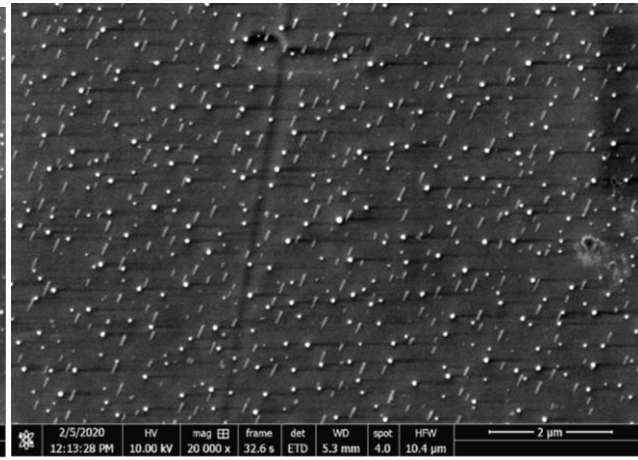

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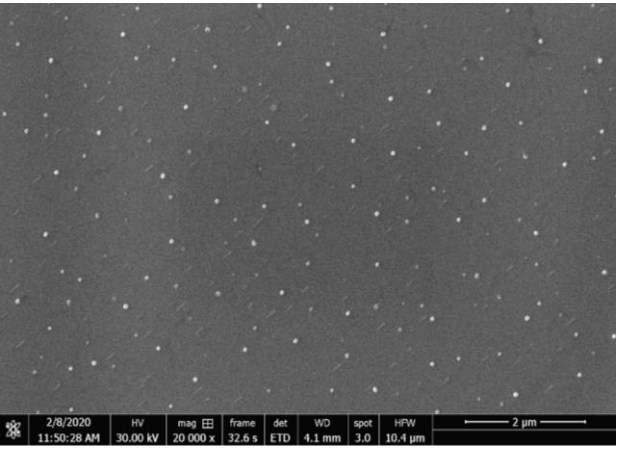

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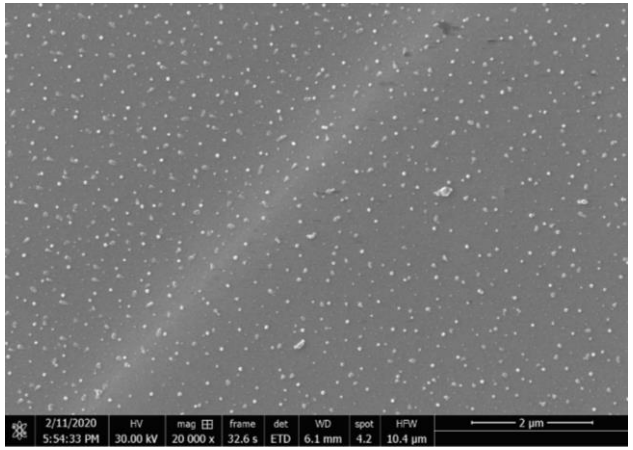

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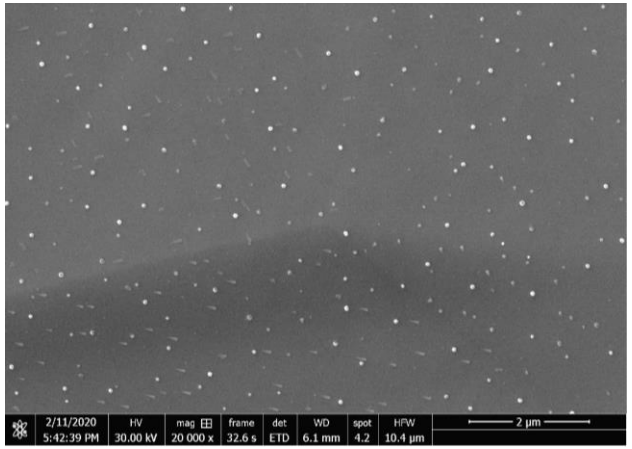

Some variations in uniformity of size and number density of nucleation sites in standard recipe samples