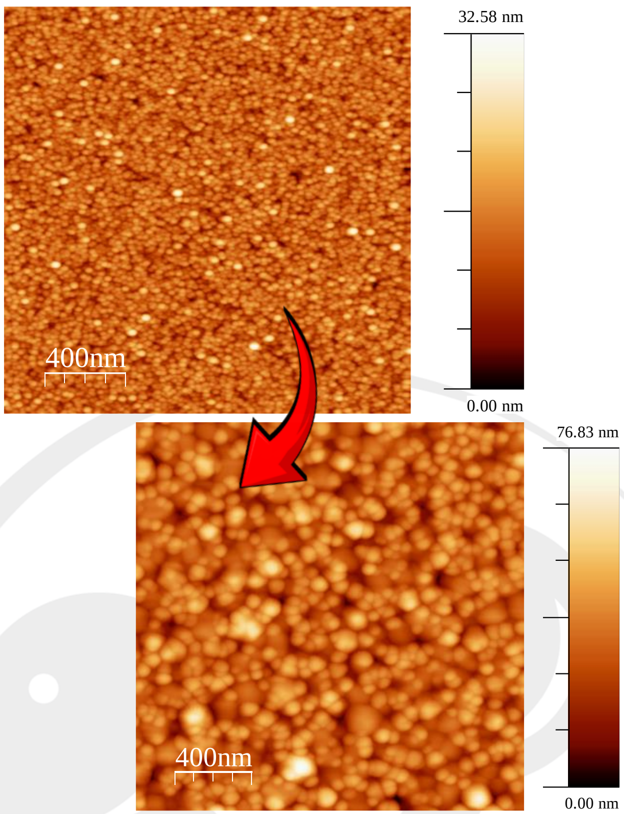
11th International Workshop on Thin Films and New Ideas for Pushing the Limits of RF Superconductivity - TFSRF2024

thinfilms and NEW IDEAS for SRF



Nb_3Sn based single and multilayers

Oleksandr Hryhorenko, Eric Lechner, Uttar Pudasaini, Anne-Marie Valente-Feliciano

MOTIVATION

Development for Nb_3Sn single and multilayers

Why Nb_3Sn?

- T_c ~ 18.3 K (9.25 K for Nb)
- H_{sh} ~ 400 mT (twice of Nb)

The ultimate goal is the development of energetic condensation technique to improve Nb_3Sn film structures

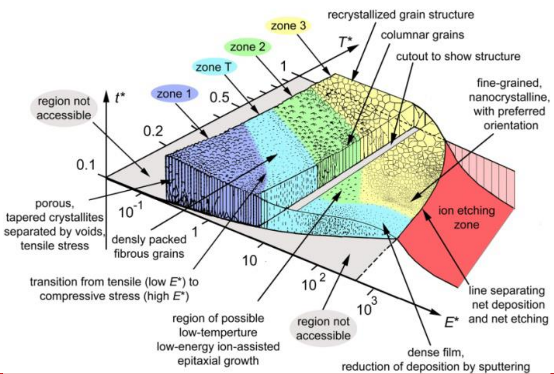
Process development

- Target commissioning
- Film production with a variation of the deposition energy, temperature, and thickness to get a niobium-tin stoichiometry in the range of 18-26 At % within deposited layers
- Multilayers development (SIS structures)
- Use lower annealing temperatures compared to tin vapor diffusion technique (Cu cavities coating)

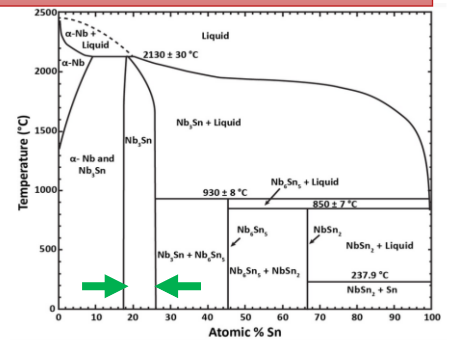
Material control:

- Surface evaluation with EDS, T_c measurement, XRD-GXRD, and topography evaluation to correlate fundamental growth and superconducting performance

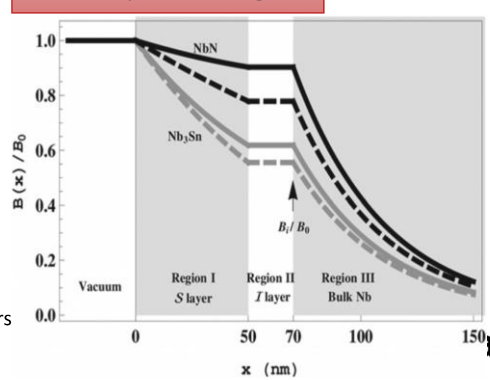
Film structure [1]



Binary phase diagram of the Nb-Sn system [2]



Multilayer coating[3]



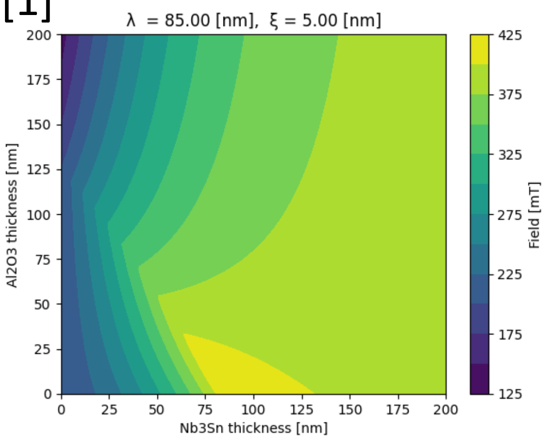
[1] André Anders, A structure zone diagram including plasma-based deposition and ion etching, Thin Solid Films, Volume 518, Issue 15, 2010, Pages 4087-4090, ISSN 0040-6090, https://doi.org/10.1016/j.tsf.2009.10.145.
[2] Charlesworth JP, Macphail I, Madsen PE. Experimental work on the niobium-tin constitution diagram and related studies. Journal of Materials Science. 1970 Jul 1;5(7):580-603.
[3] Takayuki Kubo, Yoshihisa Iwashita, Takayuki Saeki; Radio-frequency electromagnetic field and vortex penetration in multilayered superconductors *Appl. Phys. Lett.* 20 January 2014; 104 (3): 032603. https://doi.org/10.1063/1.4862892

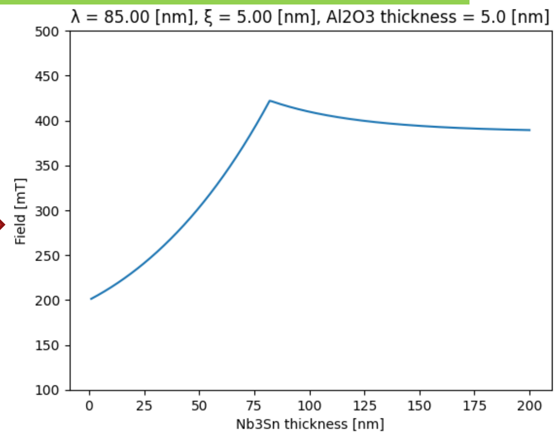
SIS Multilayers based on Nb_3Sn

Optimized contour plots from T. Kubo work [1]. The bulk superconductor is assumed Nb (λ = 40 nm, $B_v^{bulk} = 200\ mT$)

Ideal case

[1]





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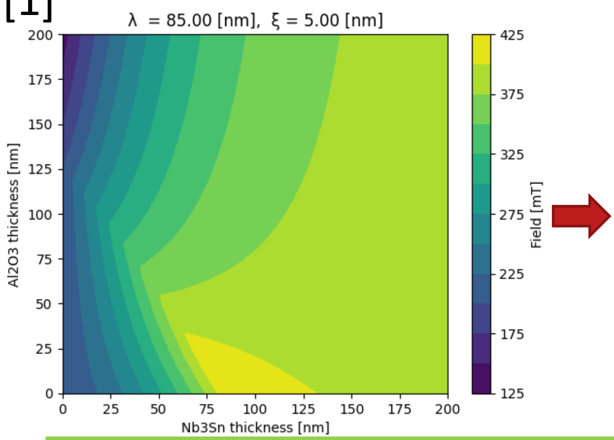
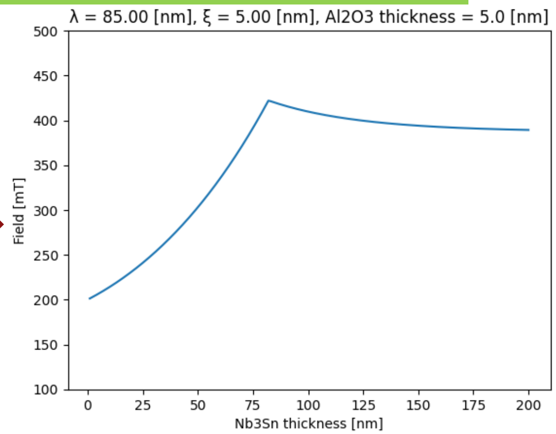
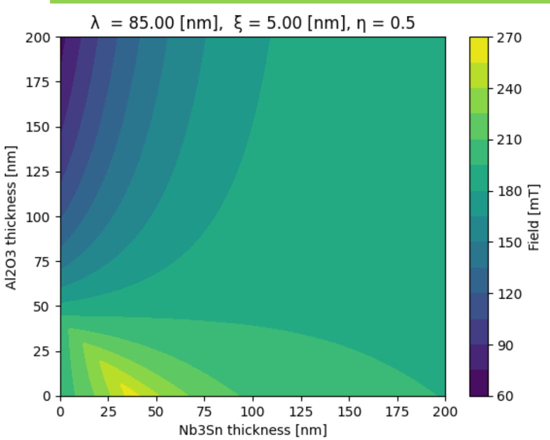
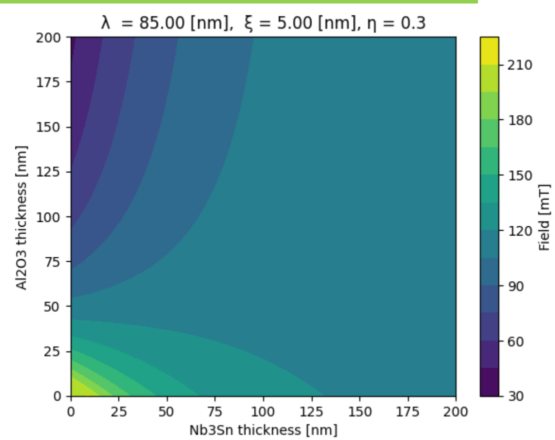




Effect of the defects





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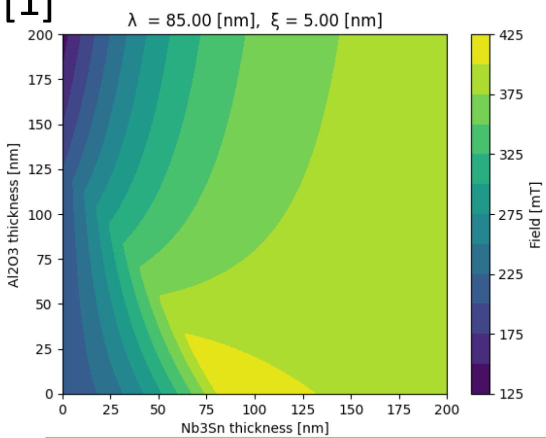
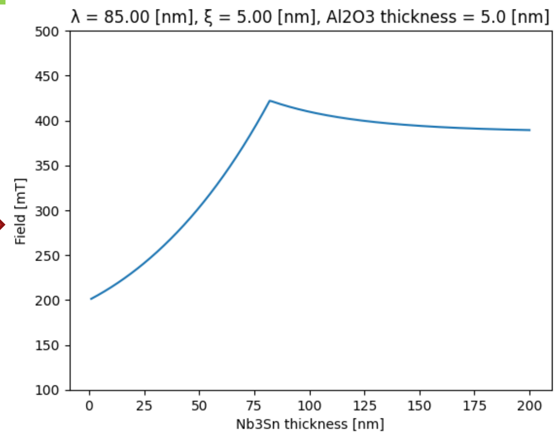
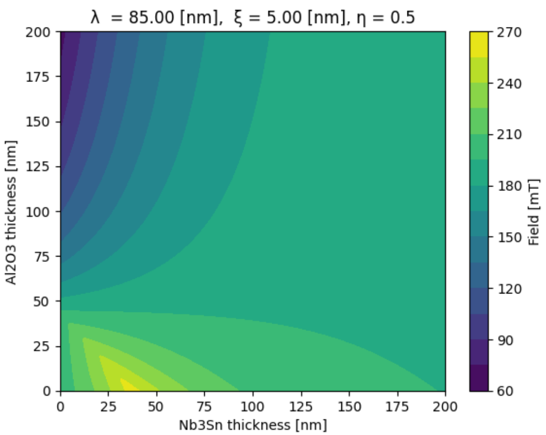
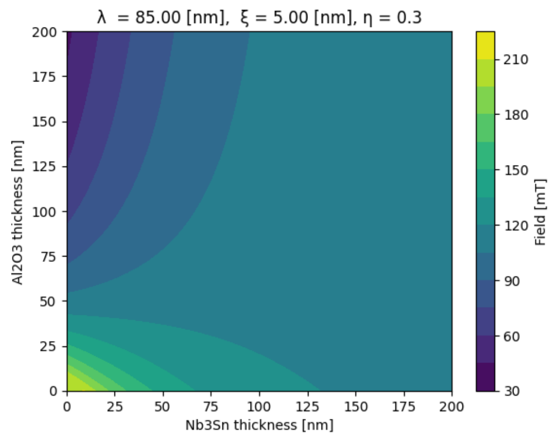




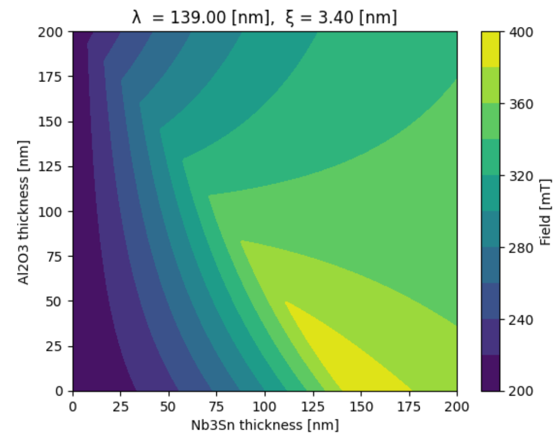
- Nano-scale defects grain boundaries reduce the superheating field at each defect/grain boundary.
- Variation of tin composition results in the reduction of the suppression factor.
- Impurity content changes the landscape of the thicknesses
- The SIS structure thicknesses must be reconsidered based on the effect of defects and impurities.

Effect of the defects





Effect of the impurity content



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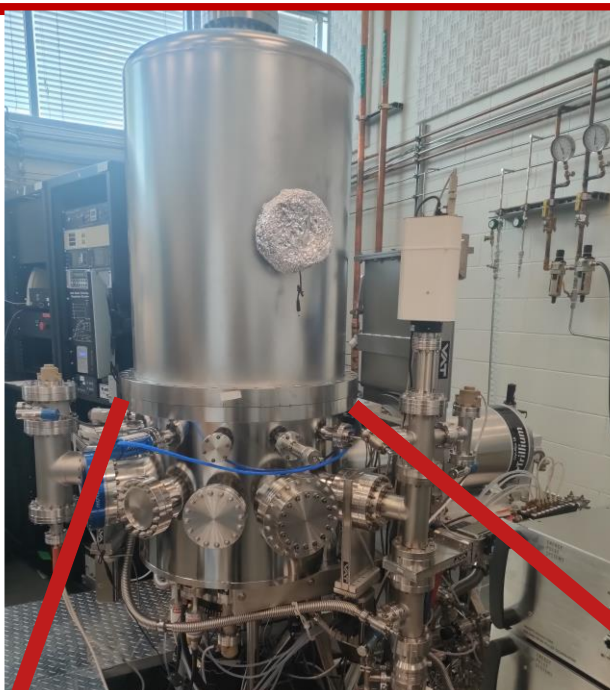
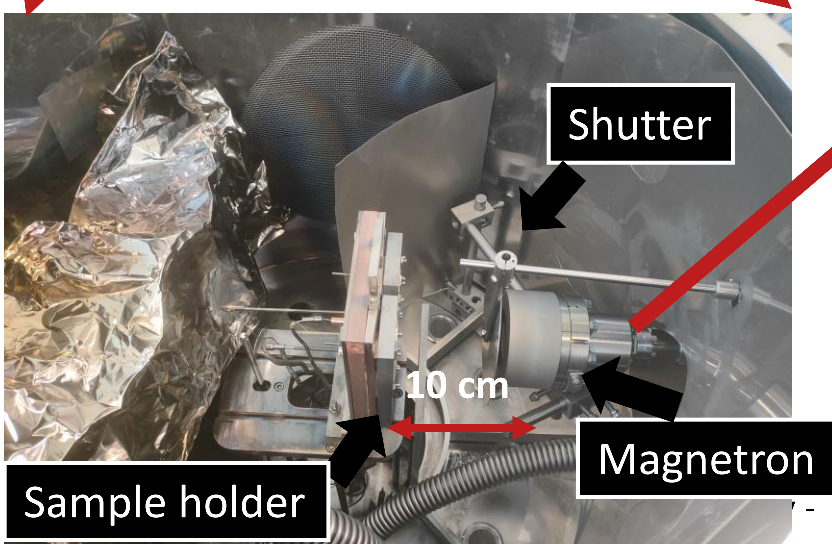
CURRENT DEPOSITION PROCESS

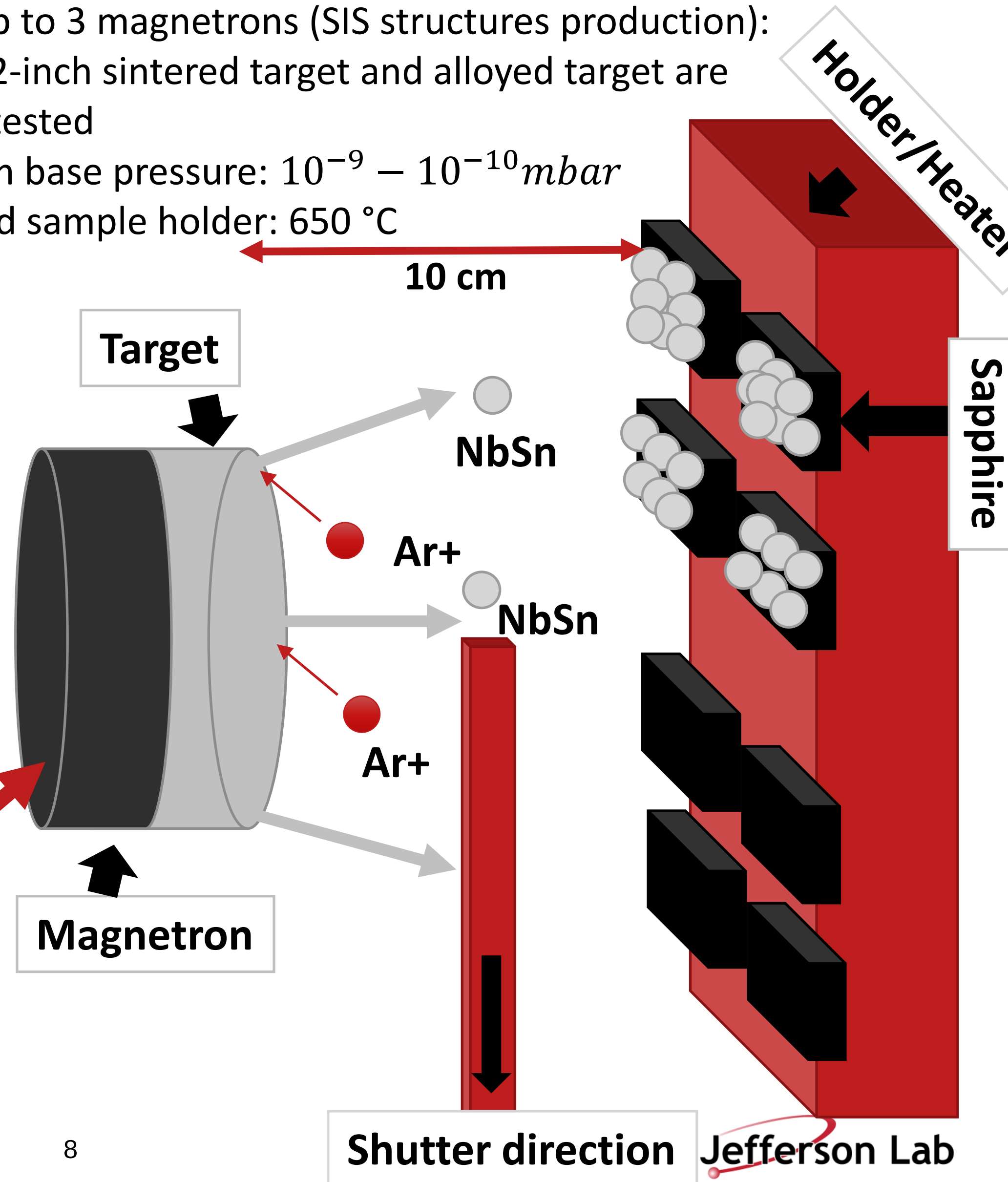
UHV DCMS system at JLAB



Experimental set-up:

- Use up to 3 magnetrons (SIS structures production):
  - 2-inch sintered target and alloyed target are tested
- System base pressure: $10^{-9} - 10^{-10} mbar$
- Heated sample holder: 650 °C

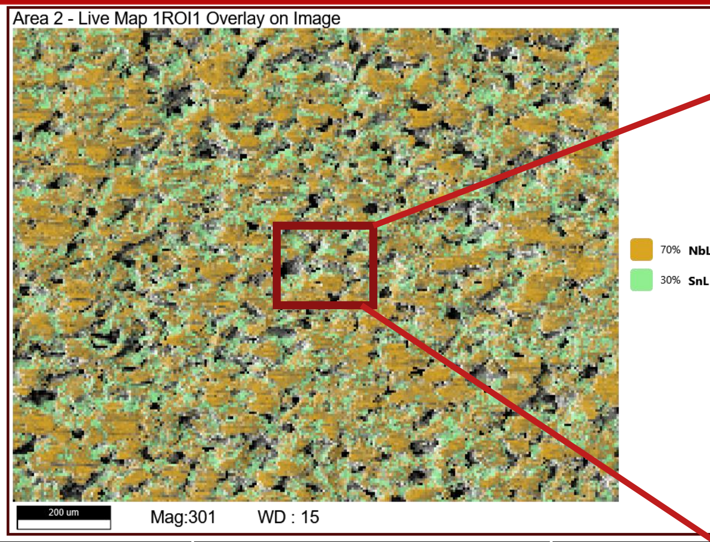


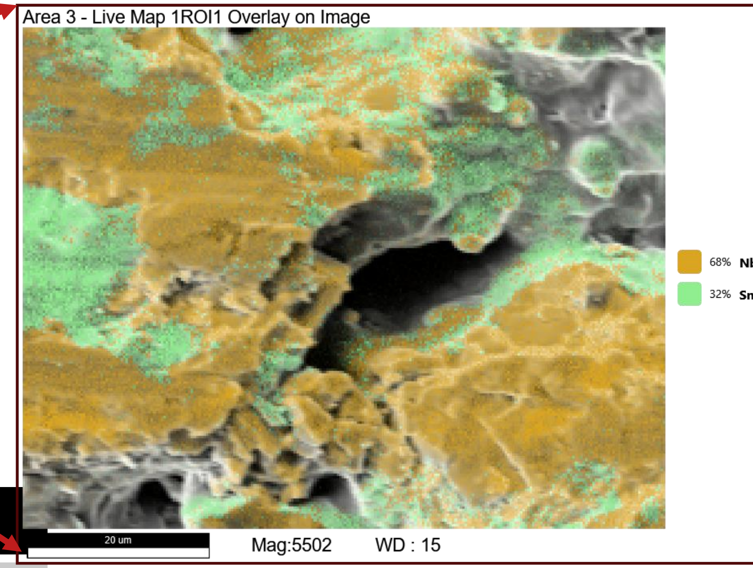


SINTERED TARGET

Sputtering parameters

2” sintered NbSn target /(Sn= 33.80 ± 0.65 (At%)), Commercial, Purity 99.95 %

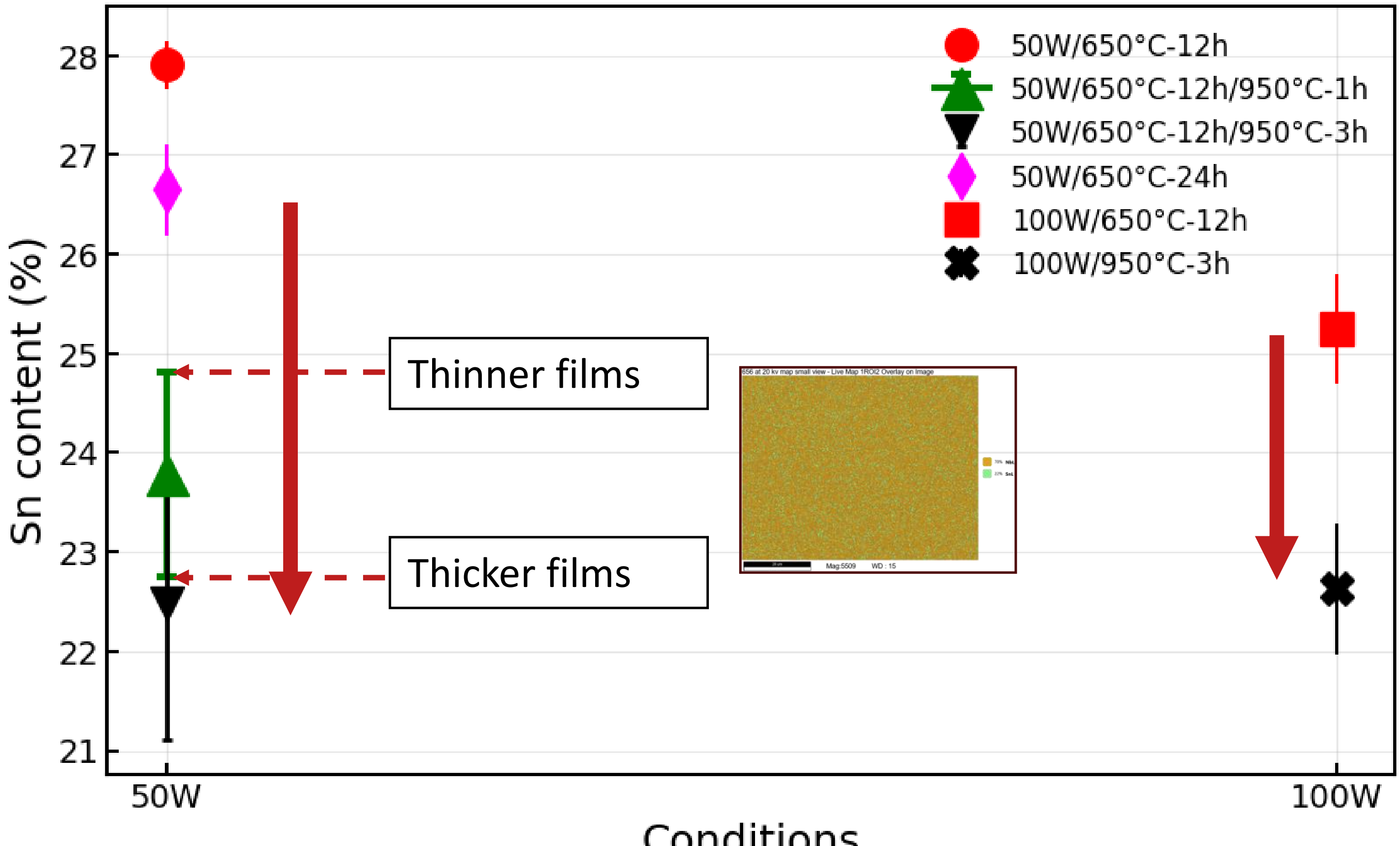




Parameters/Conditions	50 W	100 W
Substrate heated	650 °C for 12 h	650 °C for 12 h
Base pressure	$<10^{-9} mbar$	$<10^{-9} mbar$
Discharge gas	Ar	Ar
Pressure	2x10^{-3} $mbar$	2x10^{-3} $mbar$
Target-substrate distance	10 cm	10 cm
Current/Voltage	0.15A/330V	0.29A/343V
Deposition rate	~1.5 nm/min	~5 nm/min
Annealing, in-situ	650 °C for 12-24 h	650 °C for 12h

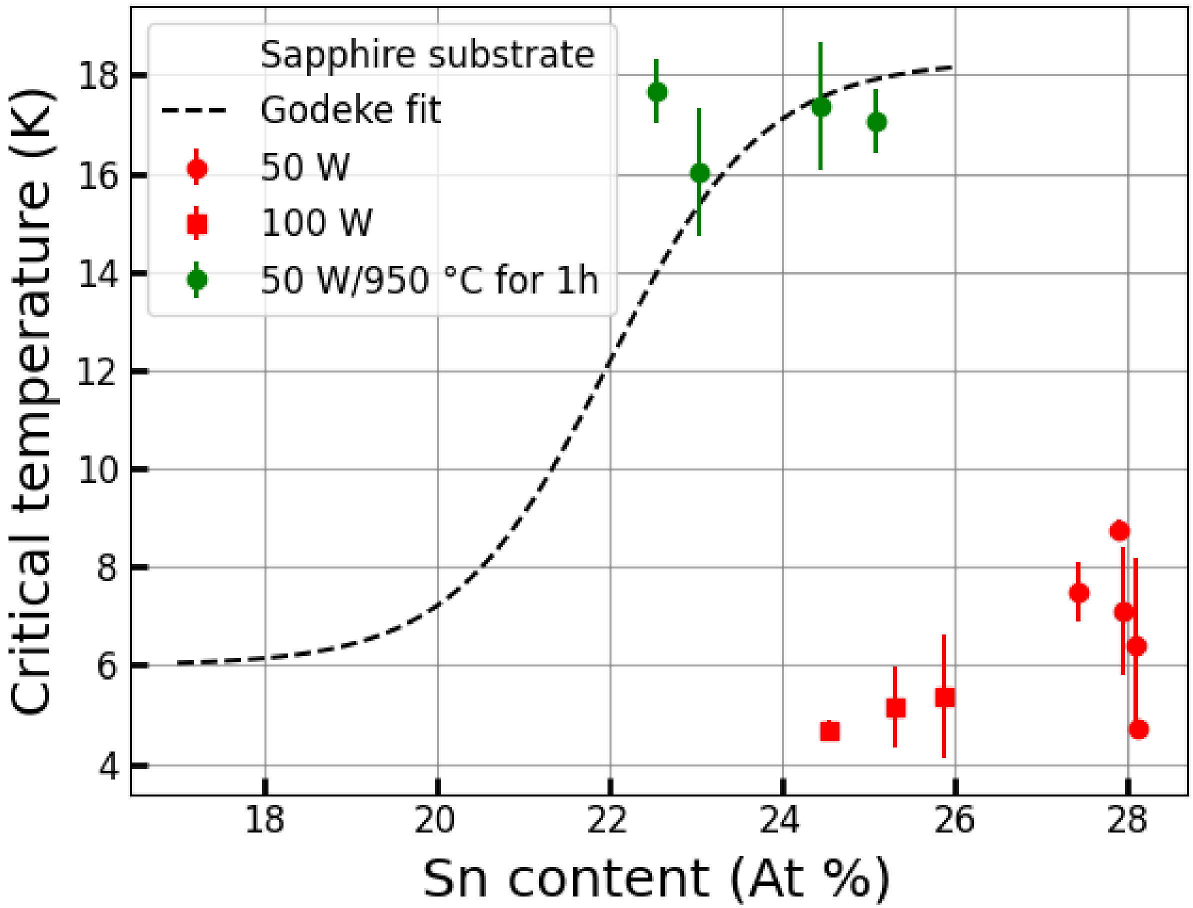
STOICHOMETRY CONTROL

Stoichiometry control



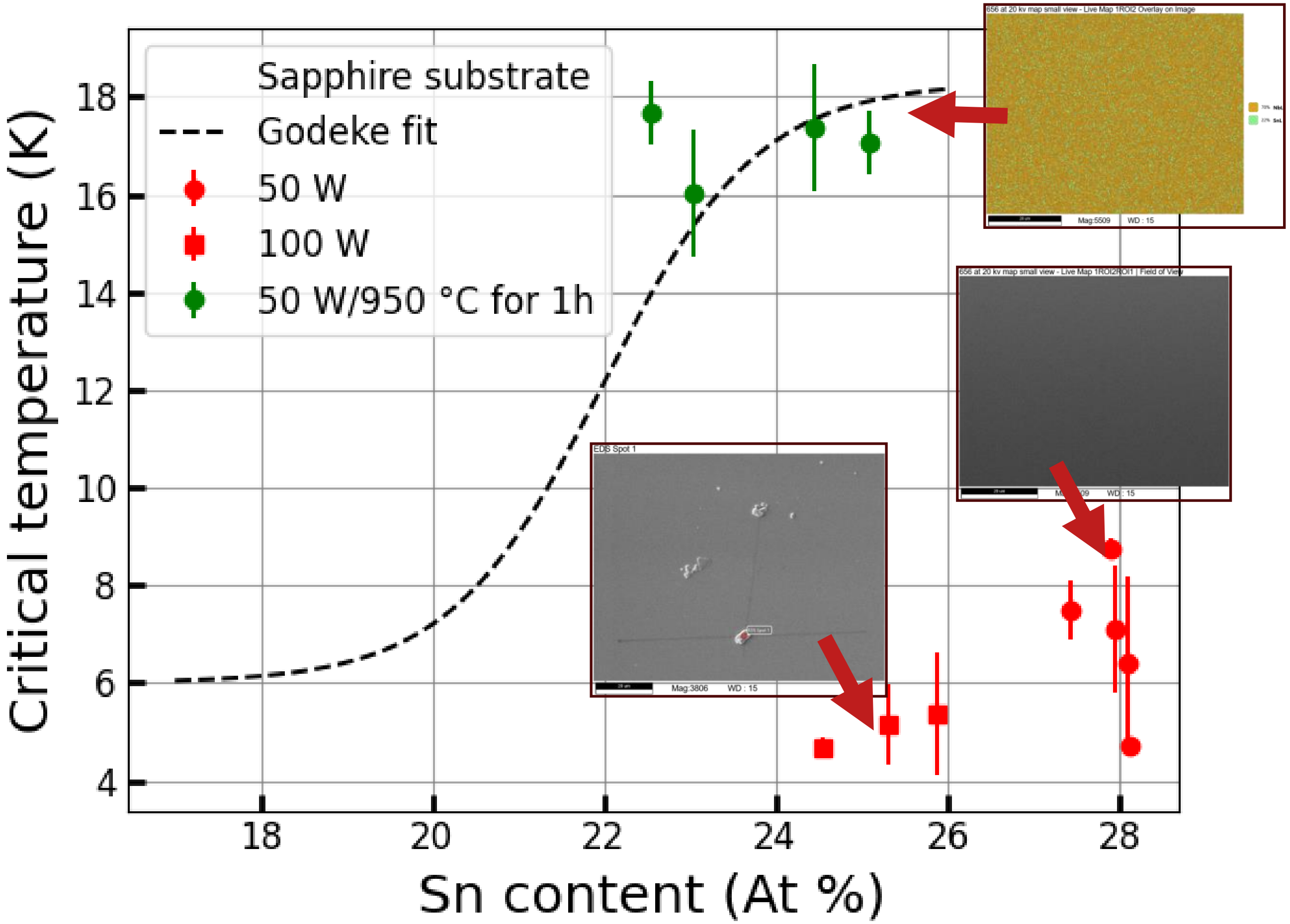
- **Optimization** of annealing parameters can help adjust tin content to the desired values.
- Sn content can be tailored by varying the film thickness: thicker film result in lower Sn (explored range from the few 10s of nm up to several microns)
- Increasing target power leads to reduced tin content

Critical temperature vs Tin content



- Power increase leads to reducing the tin content at the same annealing conditions, but also reduces Tc
- Additional annealing at 950 °C for 1h resulted in the tin content drop, also improves Tc (17.7K).

Critical temperature vs Tin content



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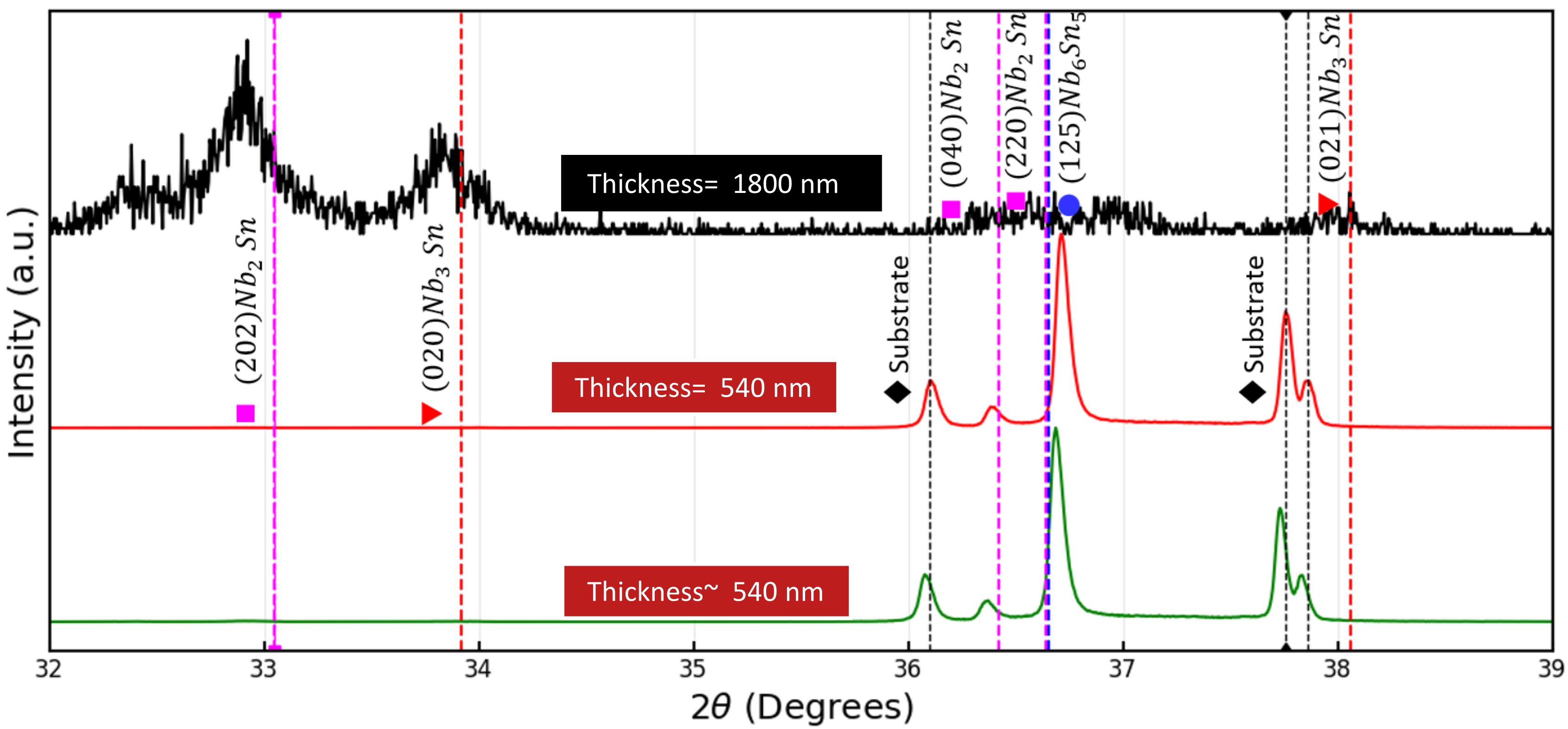
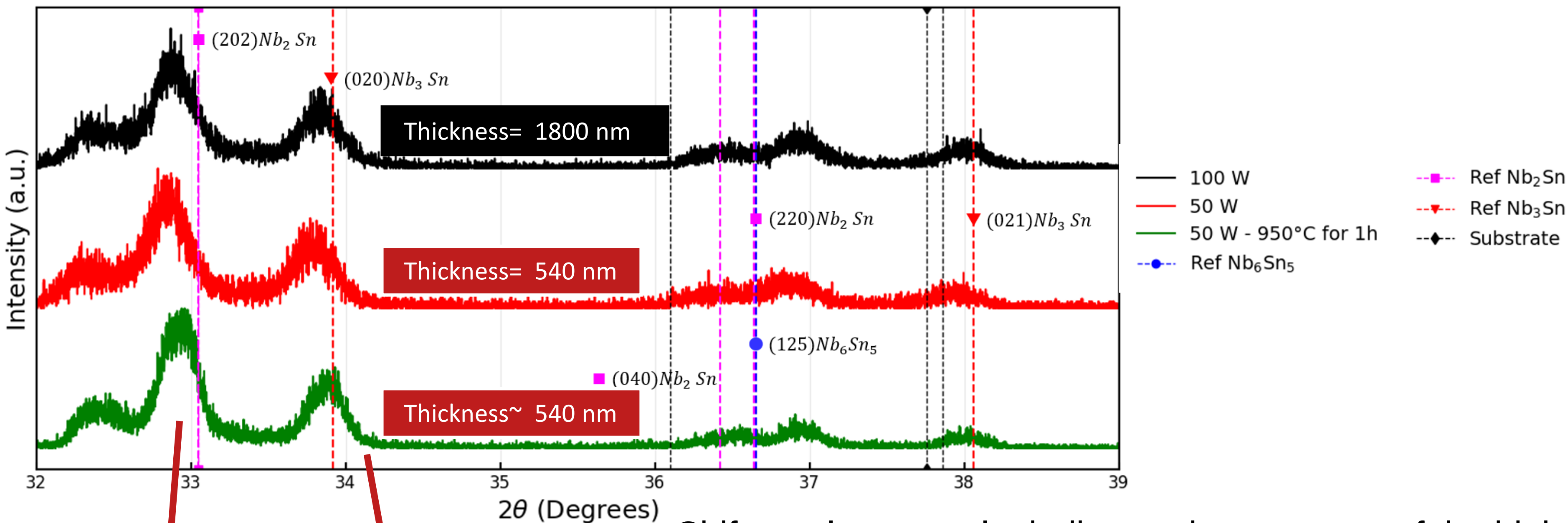
CRYSTALLINITY CONTROL

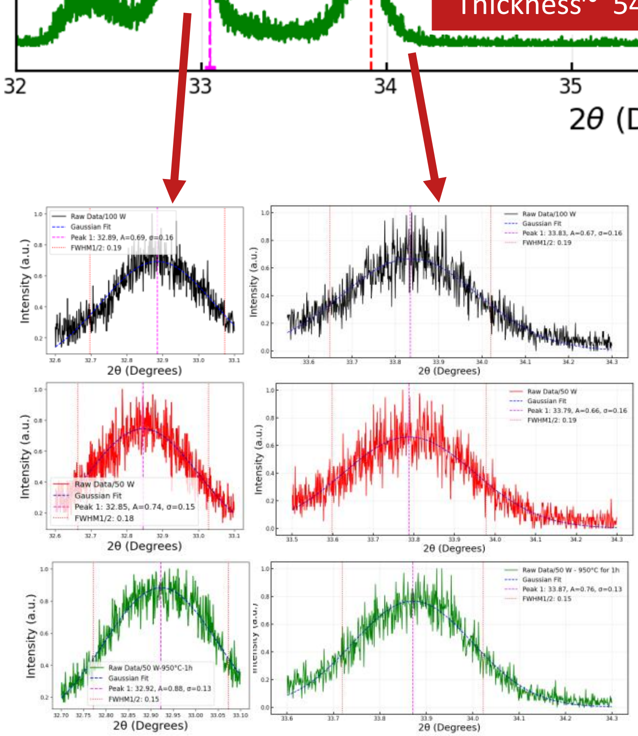
High angle XRD



- Films on sapphire with all three conditions showed Sn rich particles (different phases are presented).
- Thinner films barely show the presence of Nb_3Sn phase as the dominated phase is Nb_6Sn_5=> grazing incidence XRD is required

GXRD (X-ray source fixed at 10 degree)



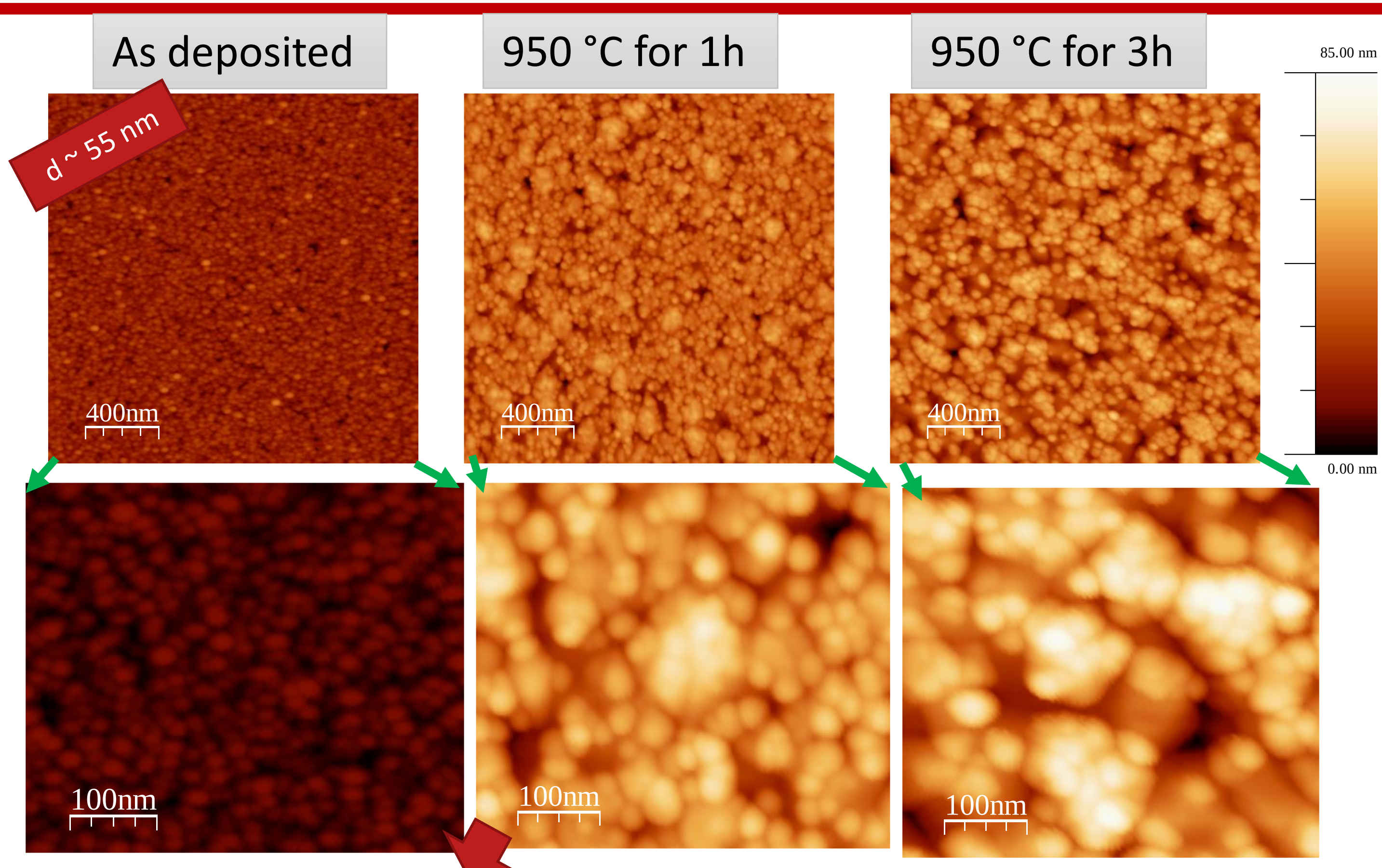


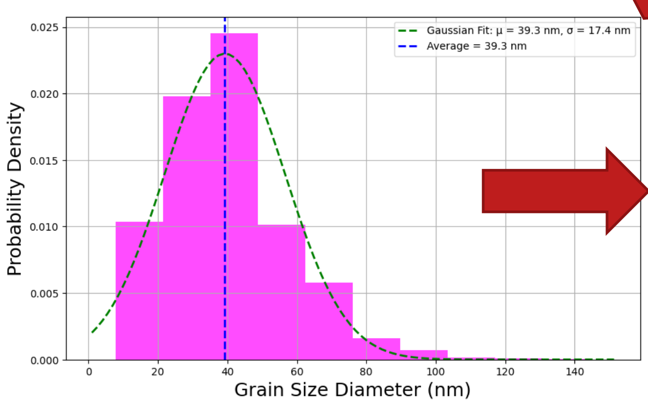
- Shift to a lower angle, indicates the presence of the high dislocations.
- Annealing leads to surface recovery and crystalline growth
- Peak broadening suggests non-linear strain, likely caused by inclusions/tin reached in the material.

Conditions	FWHM/Peak position (deg), Nb_2Sn	FWHM/Peak position (deg), Nb_3Sn
100 W	0.19/32.89	0.19/33.83
50 W	0.18/32.85	0.19/33.79
50W-950C-1h	0.15/32.92	0.15/33.87

IMPORTANCE OF SURFACE ROUGHESS CONTROL

Nb_3Sn Films – Influence of annealing on topography

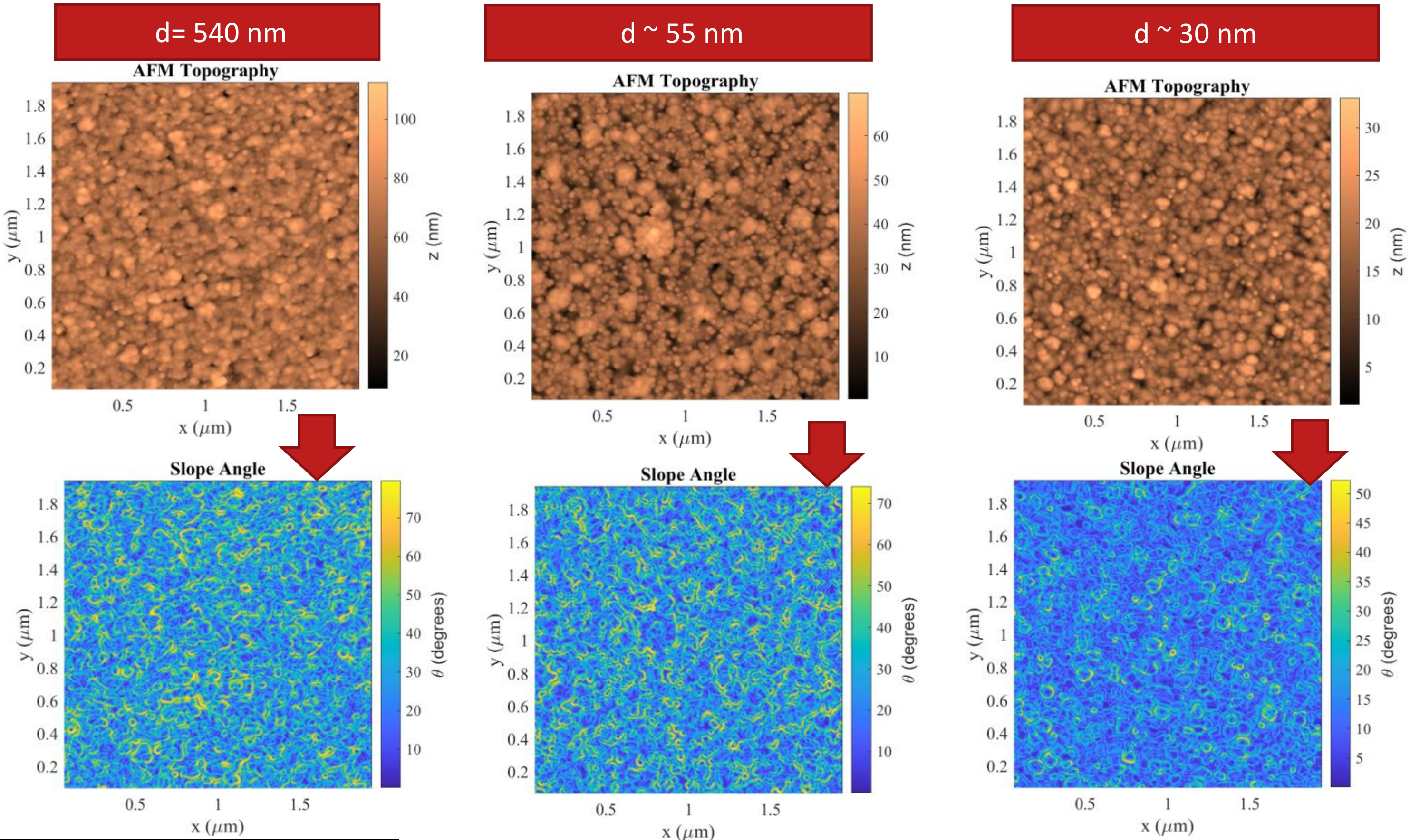




- Cluster formations were observed after annealing.
- Nanovoids/porosity were also present in the films
- Average grain size of the DCMS Nb3Sn after annealing ~40 nm

(Typical grain size of the tin vapor diffused Nb3Sn ~ 1-3 µm)

Qualitative analysis of the suppression factor



Courtesy of Eric Lechner (JLAB)

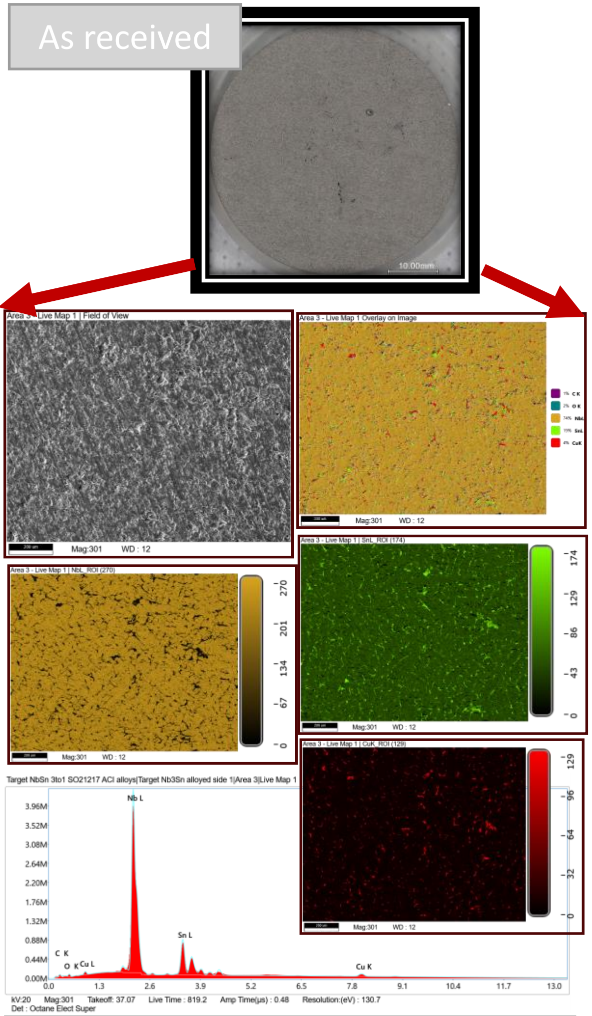
Preliminary characterization of the suppression factor:

- Key parameters influencing the superheating suppression factor include slope angle, groove depth, penetration depth, and coherence length. Notably, high slope angles were observed even in thin films. As film thickness increases, the frequency and magnitude of high slope angles also rise, which in turn leads to a reduction in the overall suppression factor.

ALLOYED TARGET

Alloyed target

As received

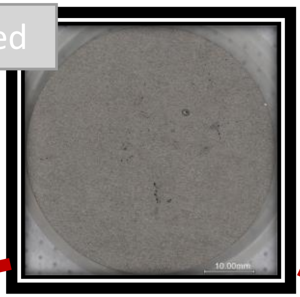


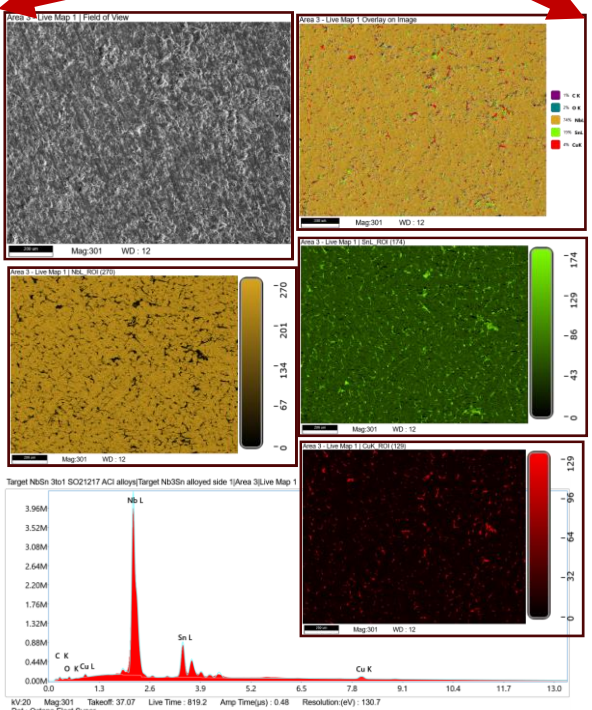
Cu particles are observed on target

Jefferson Lab

Alloyed target

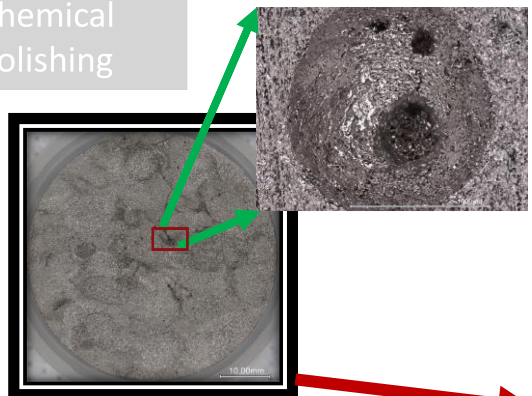
As received

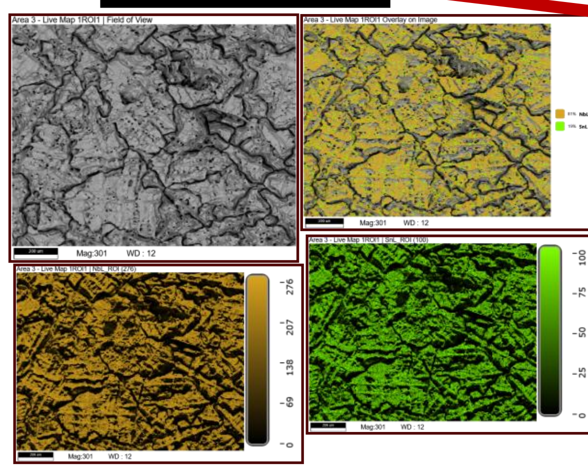




Cu particles are observed on target

Chemical polishing

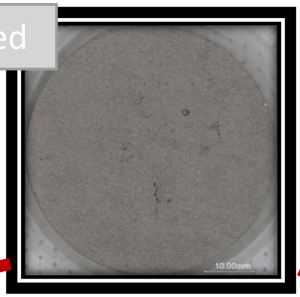


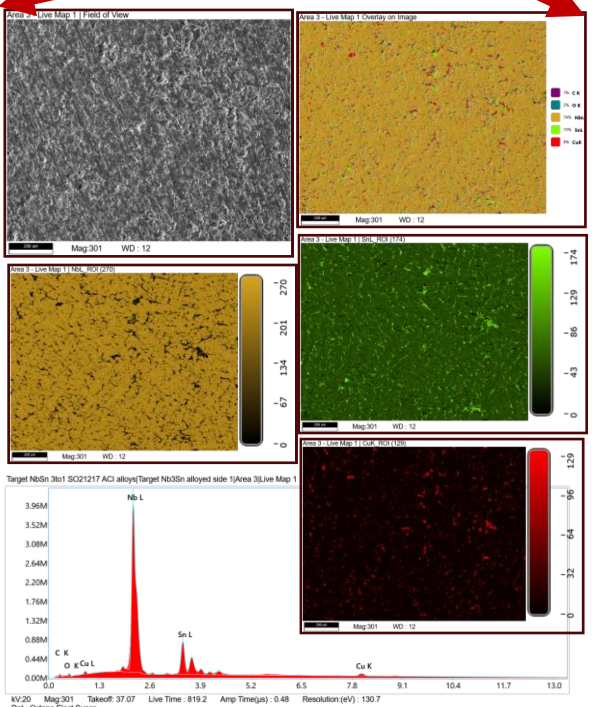


Clean Nb3Sn surface. No contamination

Alloyed target

As received





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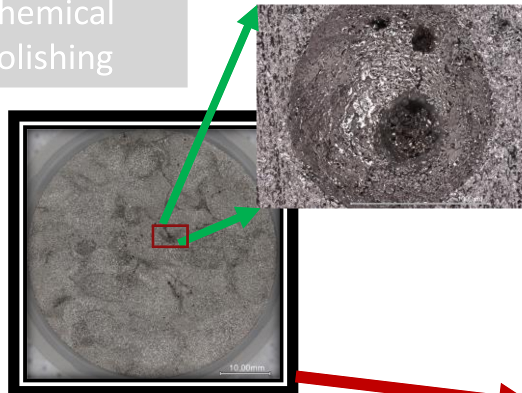
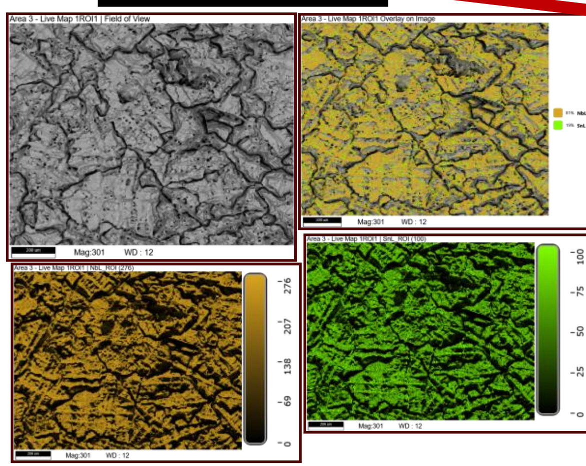
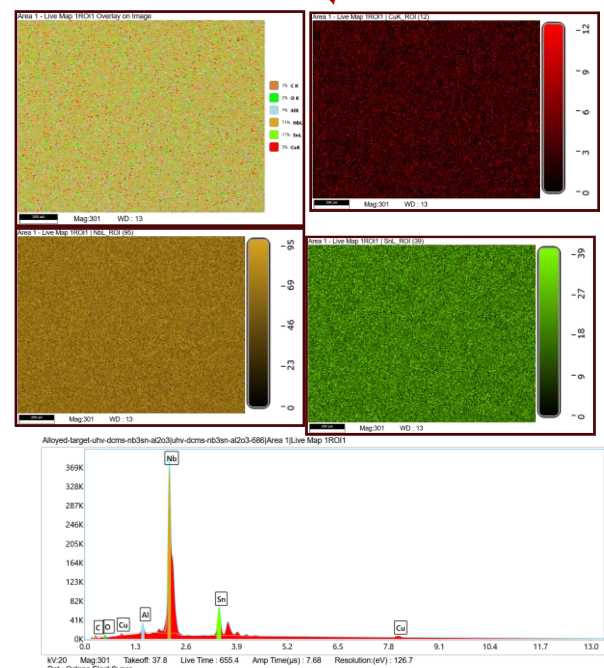




Clean Nb3Sn surface. No contamination

Deposited films



Cu pollution is reappeared

Summary

Commercial Alloyed Target:

•The commercial alloyed target failed to meet expectations (Cu contamination).

•**Commercial sintered Target:**

- Achieved a **critical temperature 17.7±0.7K** on sapphire after annealing at 950°C for 1 hour.
- Different annealing conditions were explored but remain unevaluated due to cryoplant refurbishment.
- The presence of different phases might be linked to unreacted sintered target

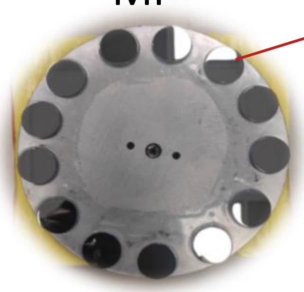
Next Steps:

•Evaluate a new alloyed target to achieve better Tc on sapphire.

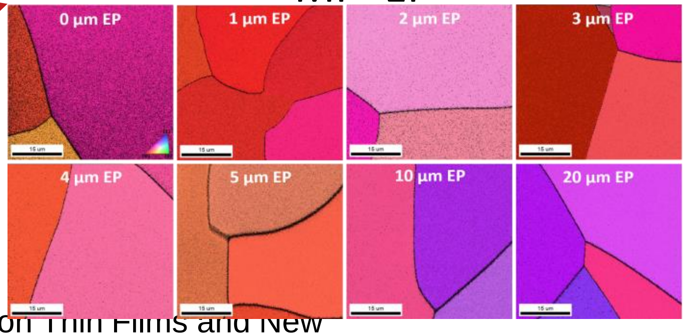
•To deposit Nb_3Sn on the high quality substrates (Nb and Cu).

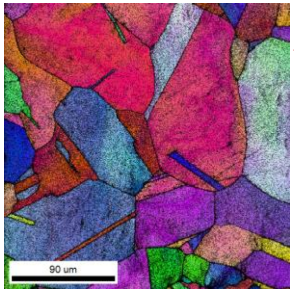
•Explore energetic condensation (HiPIMS)

MP



MP+EP





MP