iFAST
SAMARA
CSN5 INFN experiment

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Cristian **Pira**

On behalf of INFN LNL SRF group

Nb_3Sn on Cu coating by Magnetron Sputtering from Target Synthesized via Liquid Tin Diffusion

10th International Workshop on Thin Films and New Ideas for Pushing the limits of SRF

Jefferson Lab, September 20, 2022

Goal

Realize and test a

Nb_3Sn on Cu

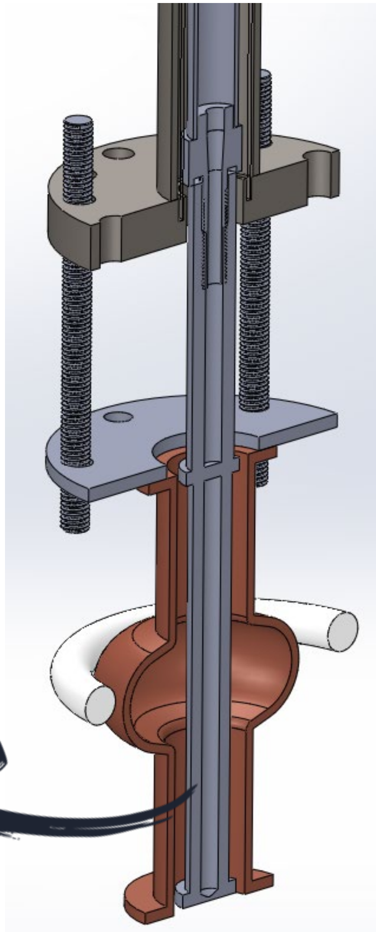
6 GHz elliptical cavity

Nb on Cu 6 GHz cavity

Strategy

Use the **same Magnetron Sputtering configuration** of **Nb on Cu** 6 GHz elliptical cavity

Nb on Cu 6 GHz cavity coating system



1. Replace Nb cylindrical target with a Nb_3Sn one

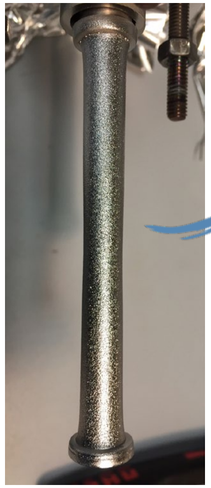
2. Search the best deposition parameters for Nb_3Sn

1. Nb_3Sn target production

Nb_3Sn on Cu
LNL strategy for 6 GHz cavities

Nb target

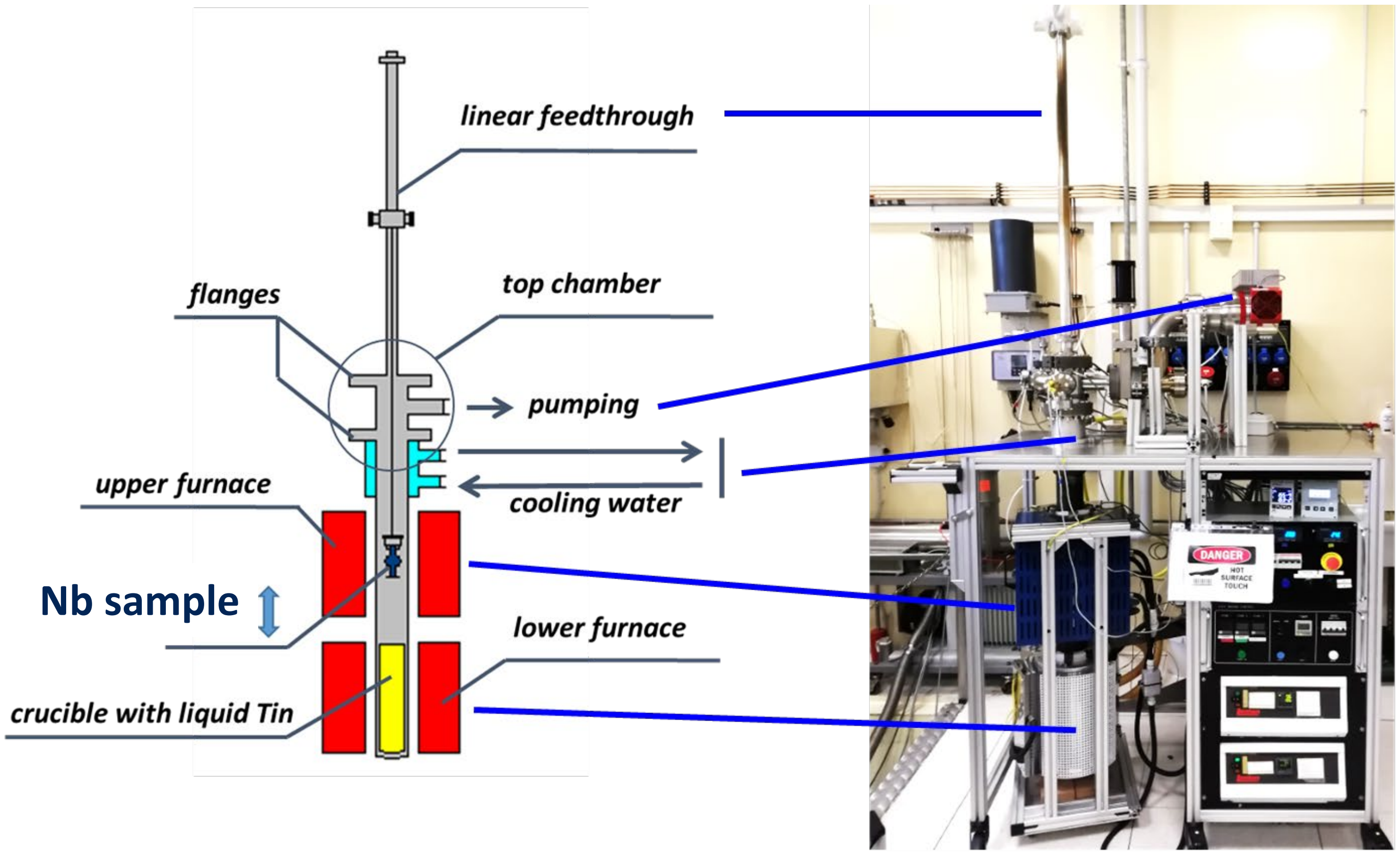
Dipping
in Liquid Tin
+ Annealing



Nb_3Sn on Nb target

Magnetron
Sputtering

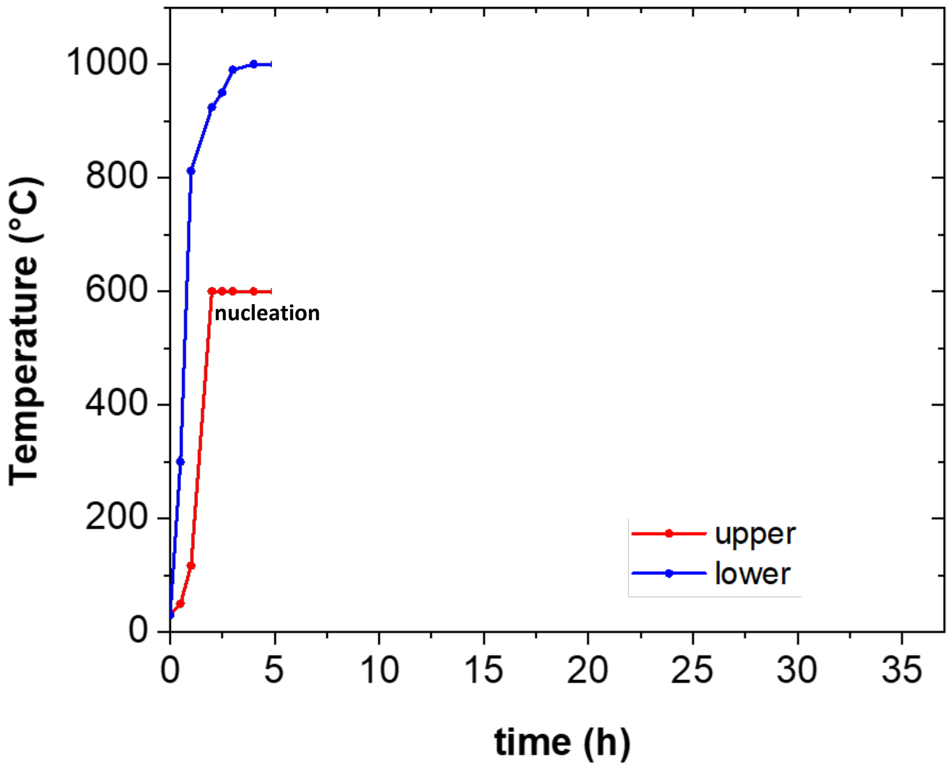
Nb_3Sn target production by Liquid Tin Diffusion



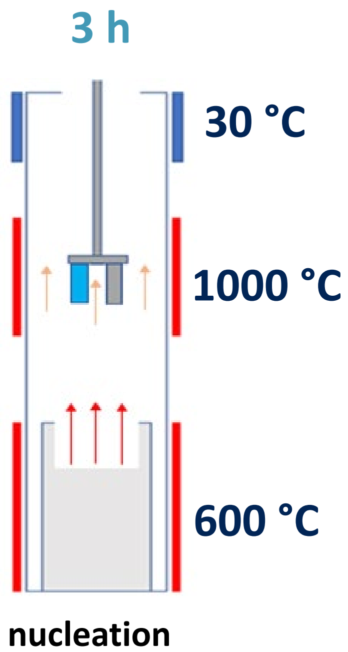
- Similar to **Tin Vapor Diffusion**
- Allows coating of **thick films**

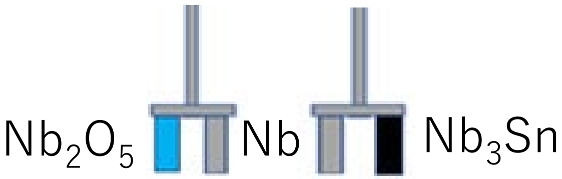
- System designed in **2005**
- **UHV Inconel** chamber
- **Process improved** in 2021

Liquid Tin Diffusion Process

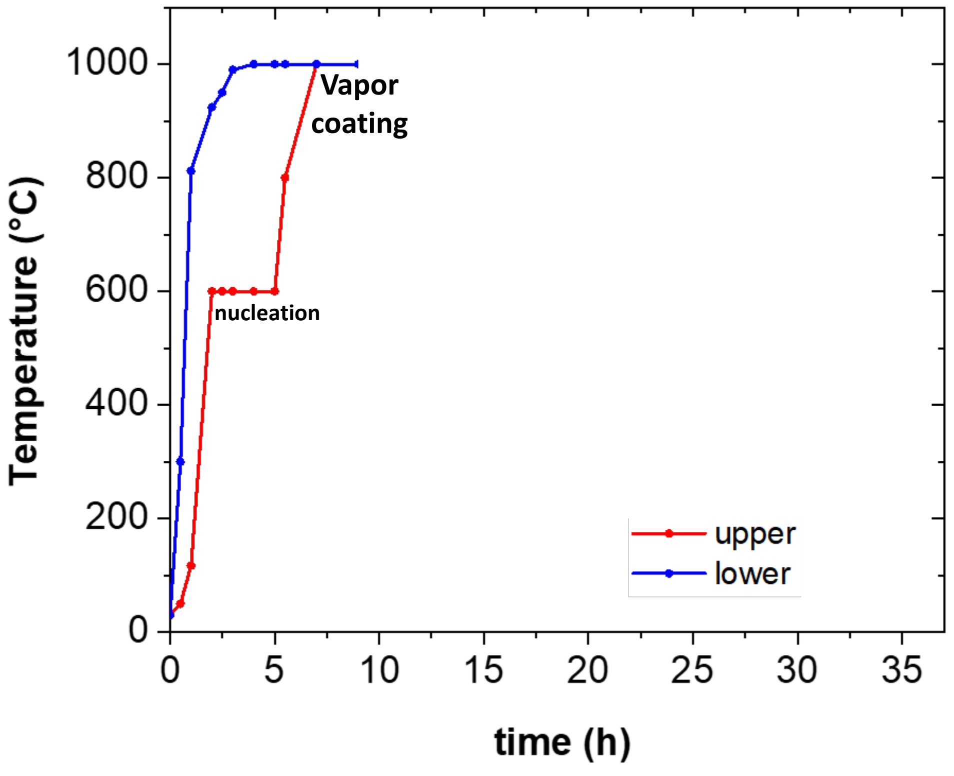


Temperature furnaces profile

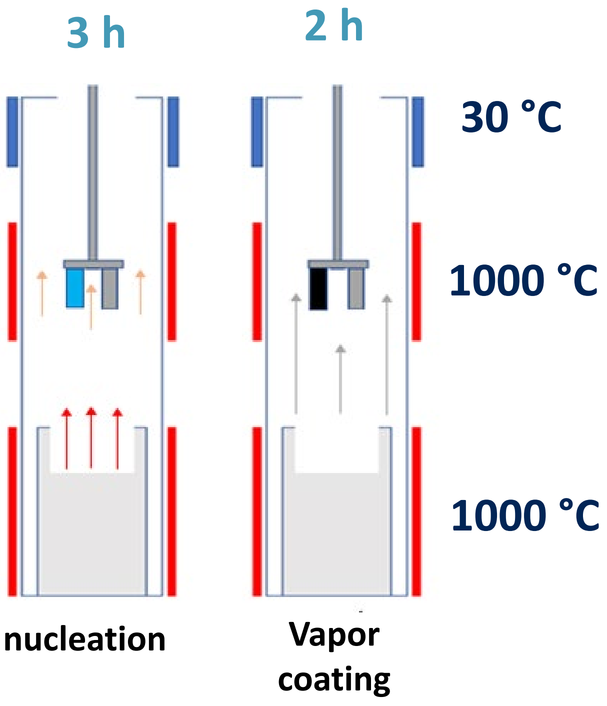


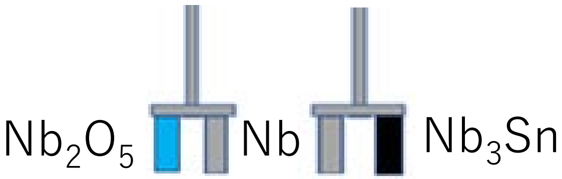


Liquid Tin Diffusion Process

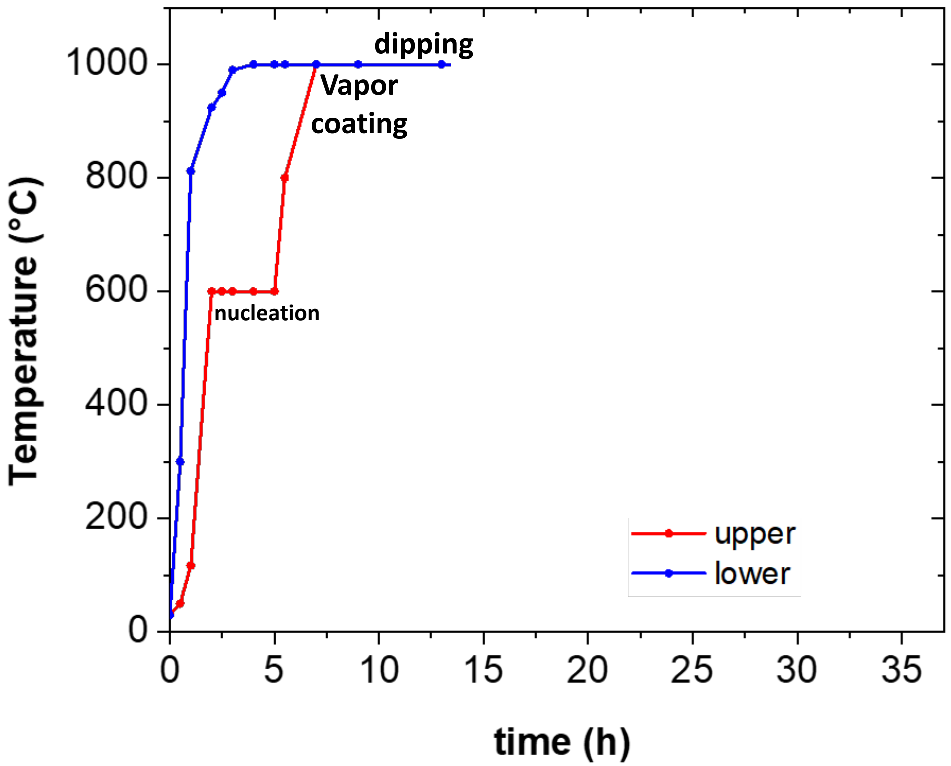


Temperature furnaces profile

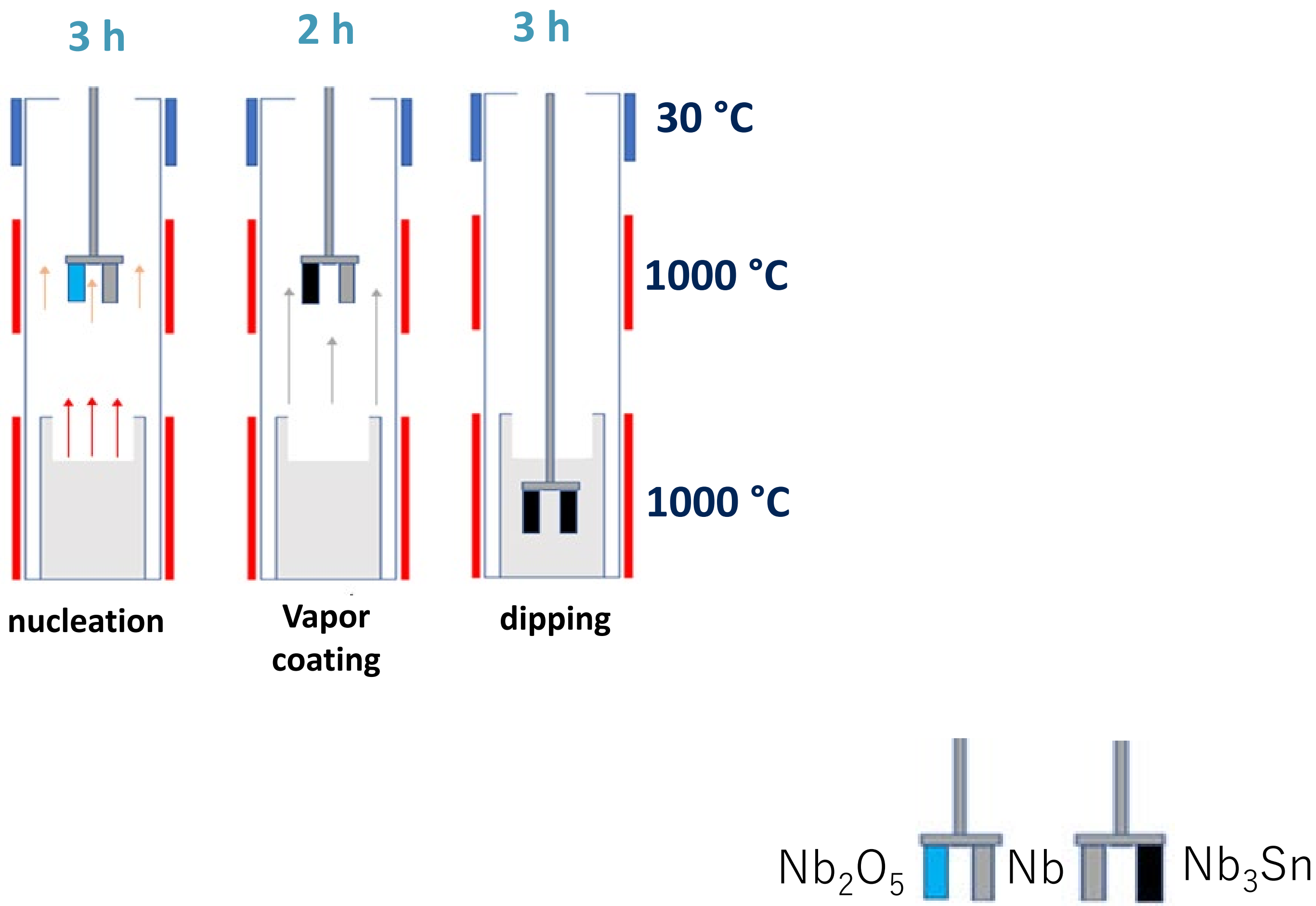




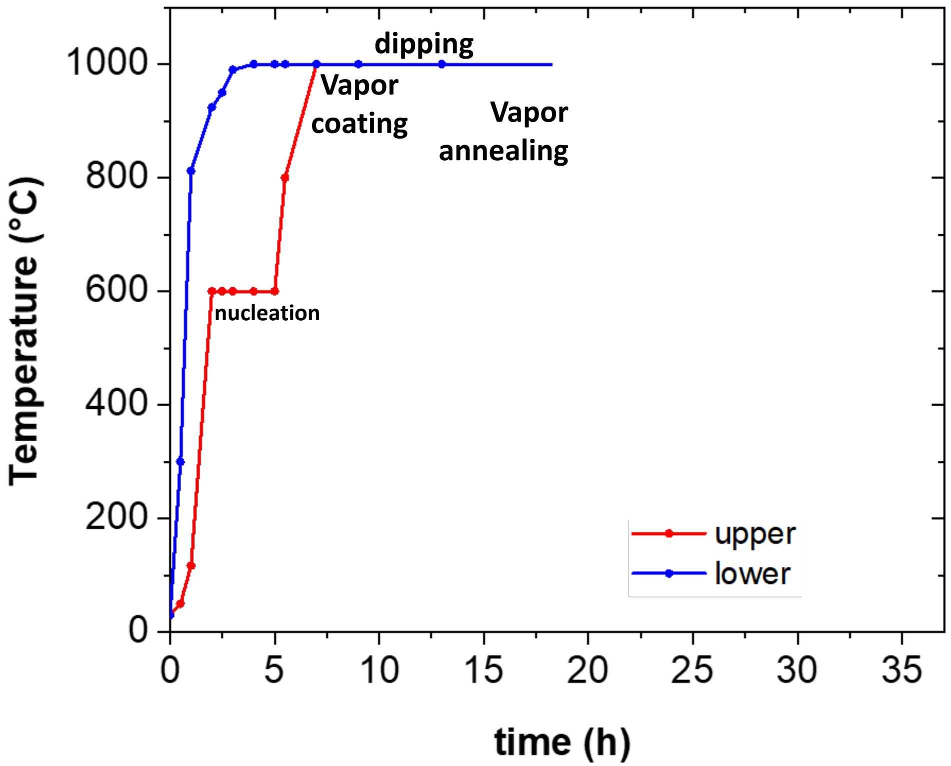
Liquid Tin Diffusion Process



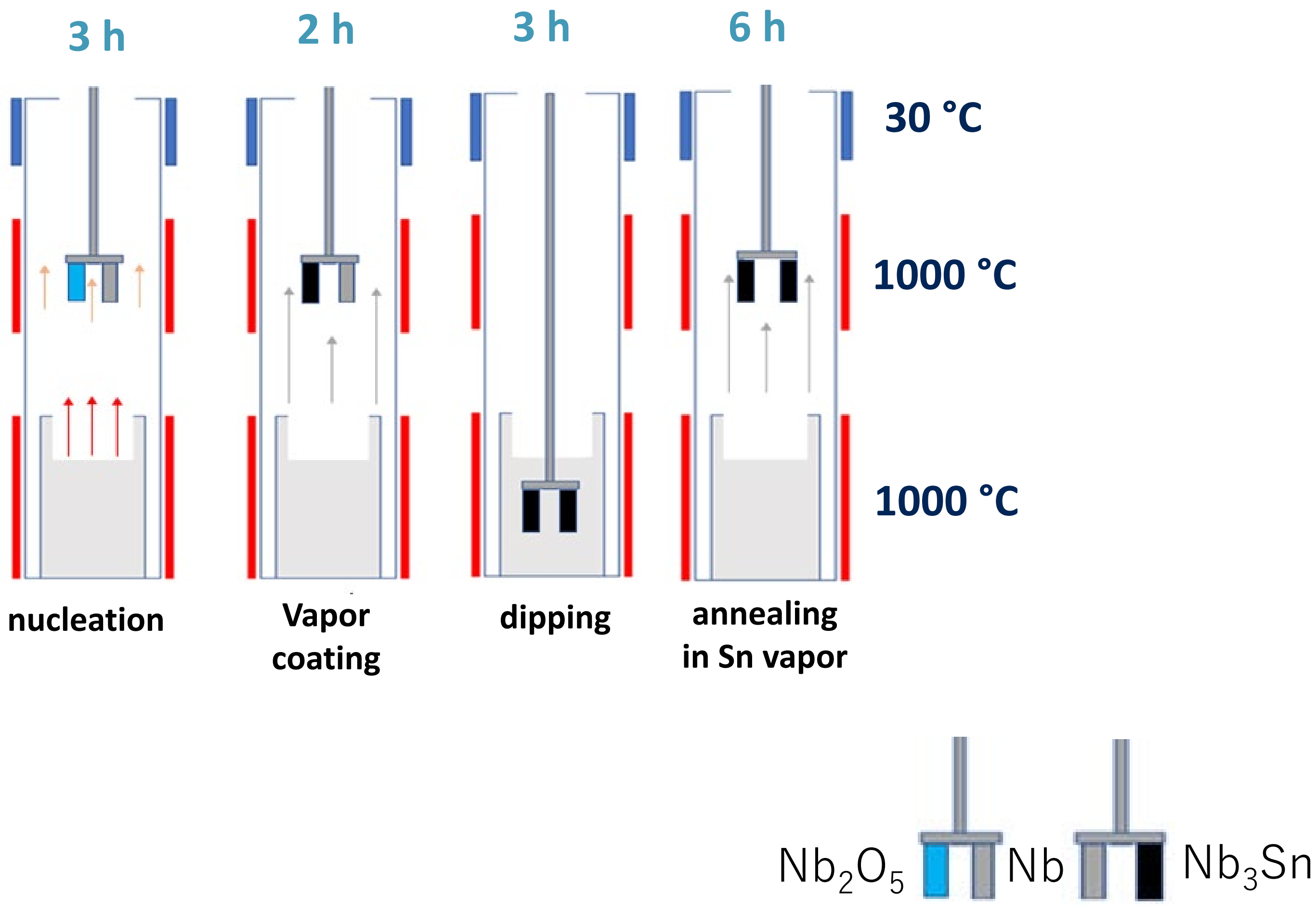
Temperature furnaces profile



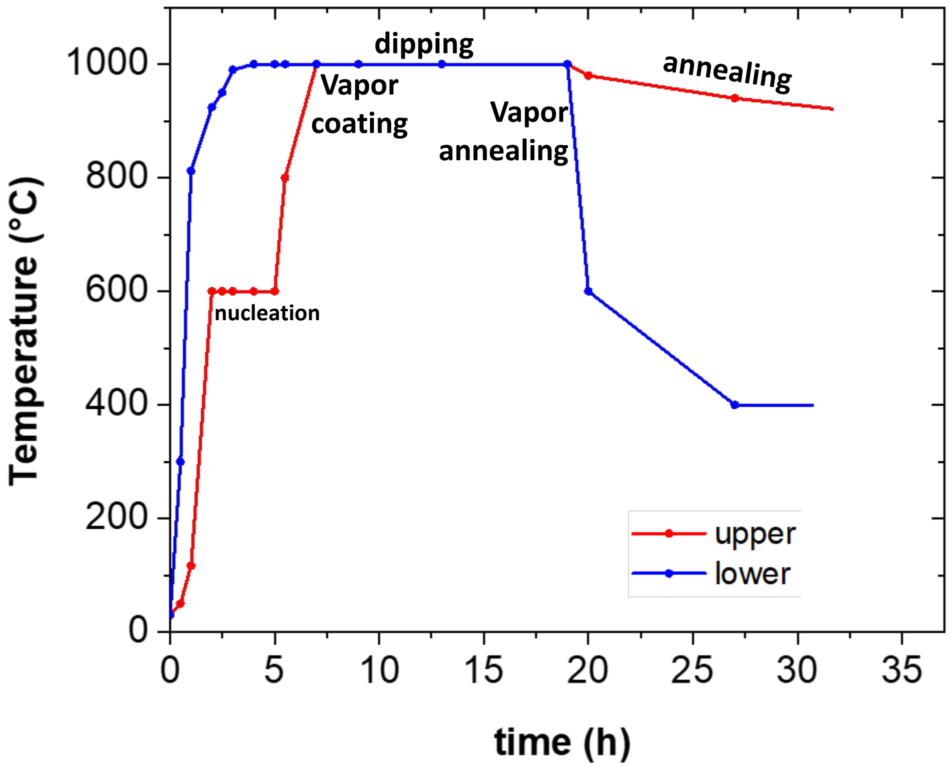
Liquid Tin Diffusion Process



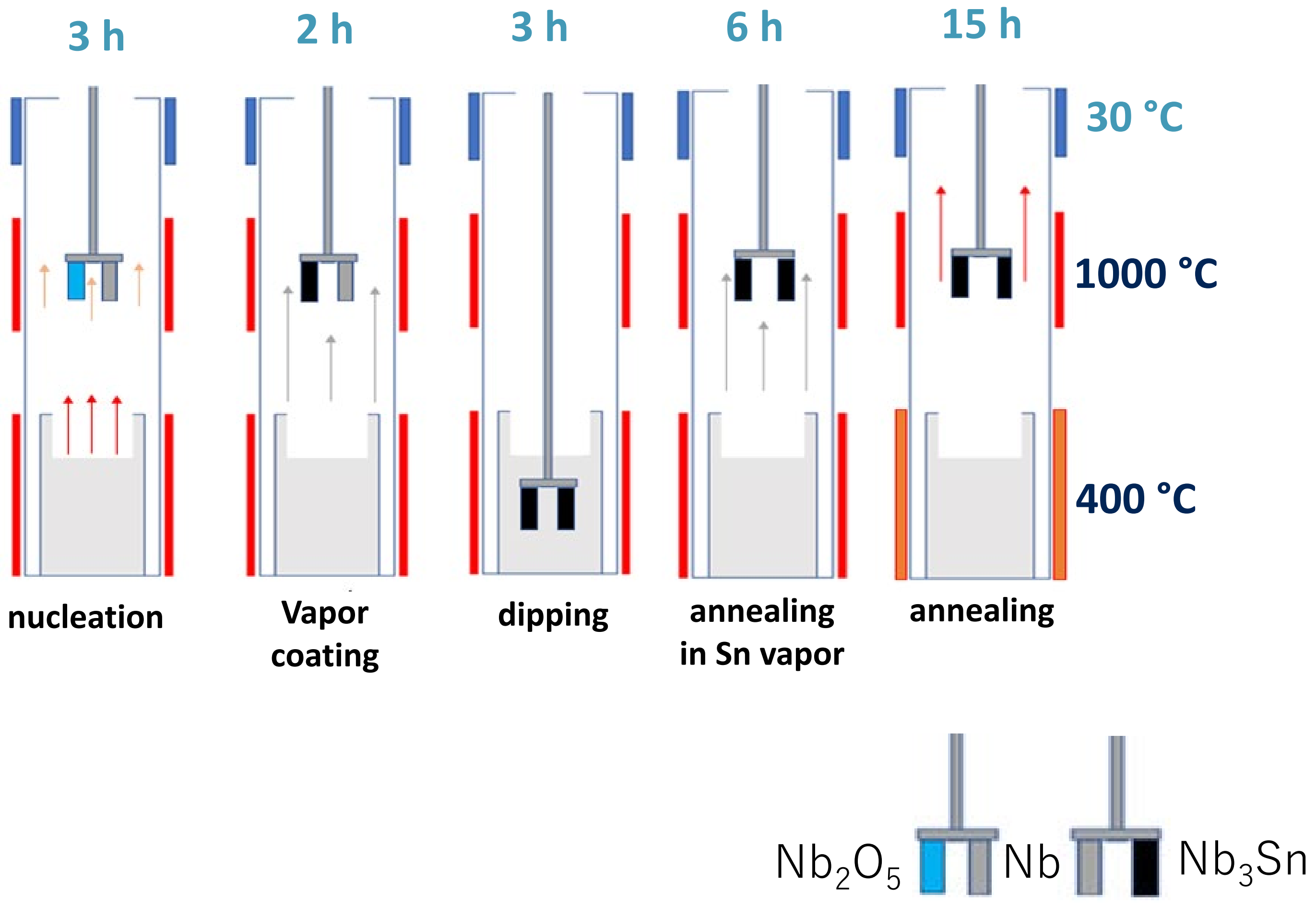
Temperature furnaces profile



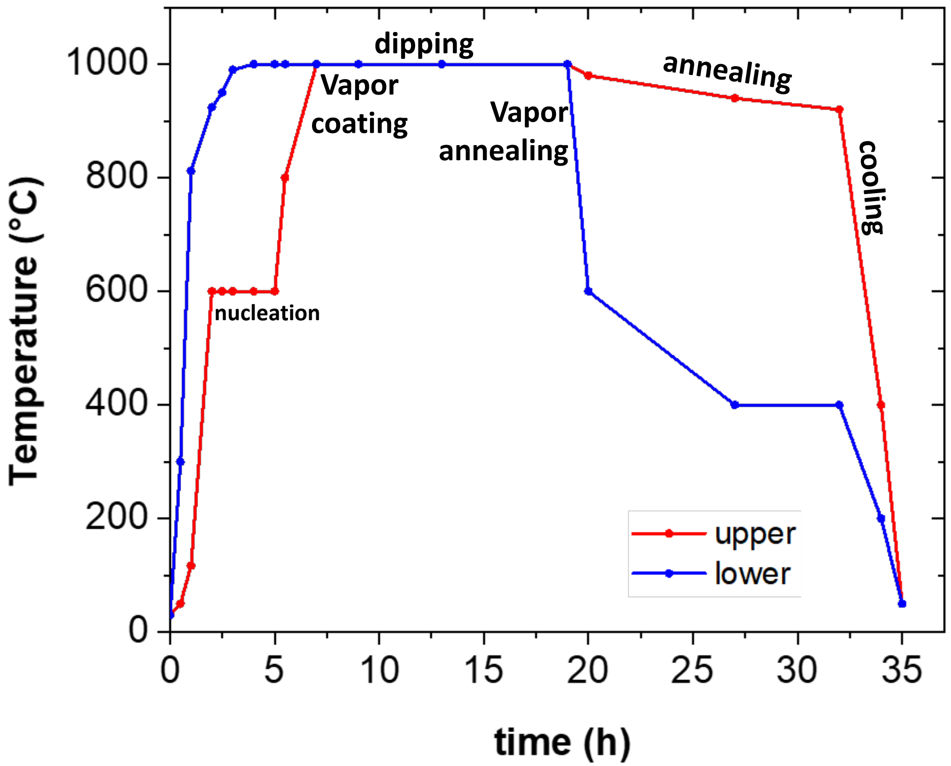
Liquid Tin Diffusion Process



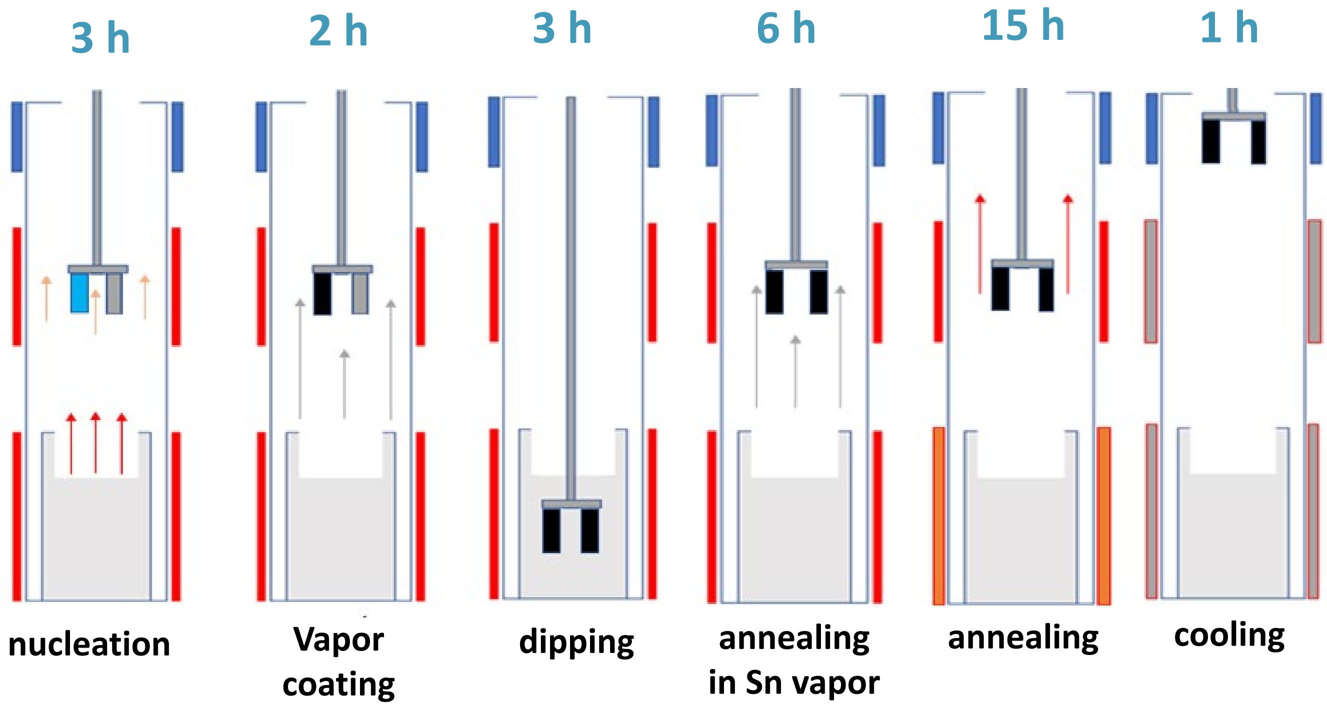
Temperature furnaces profile



Liquid Tin Diffusion Process

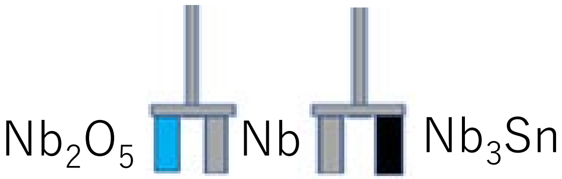


Temperature furnaces profile



Sample temperature

$\Delta T \sim$ 100-150 °C



Dipping time - thickness

Nb_3Sn growing rate by **dipping** is **related to the Sn diffusion** in Nb and can be described by **Fick' laws of diffusion**

$$x^2 = kt$$

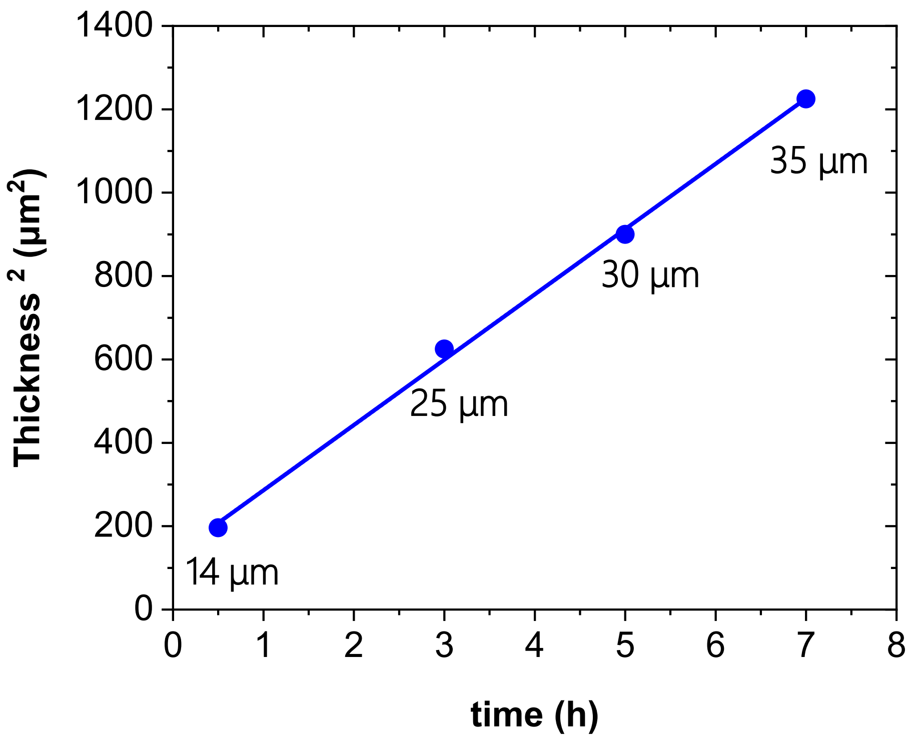
x: Nb_3Sn thickness

t: dipping time

k: a costant that takes in account the diffusion coefficient D

100 µm → ~ 63 hours of dipping!

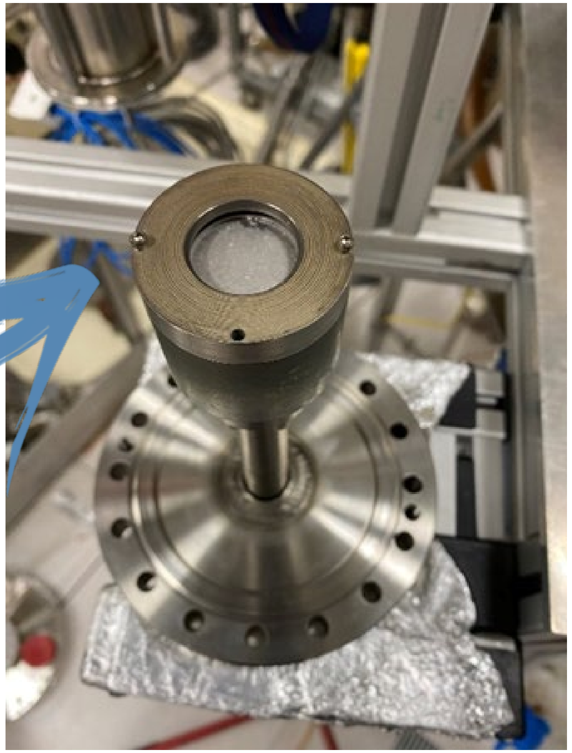
24 hours of dipping → ~ 62 µm

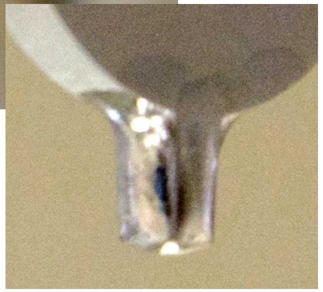


$$x^2(\mu\mathbf{m^2}) = 157 \cdot t(\mathbf{h}) + 130$$

$$R^2 = 0.9983$$

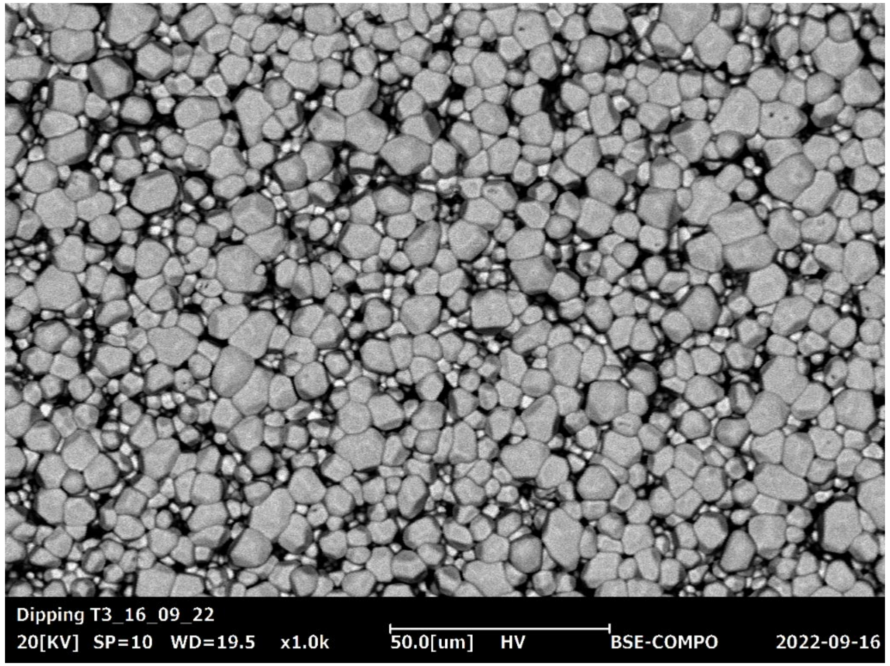
Test with 1" target





Shape optimized to remove Tin excess

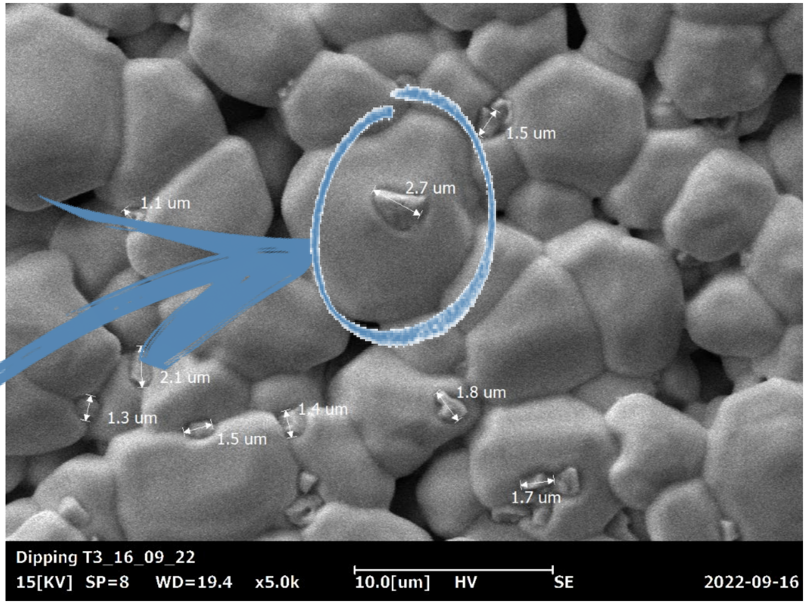
Target Morphology
(SEM + EDS characterization)



Stoichiometric Nb-Sn composition

Nb 75 %

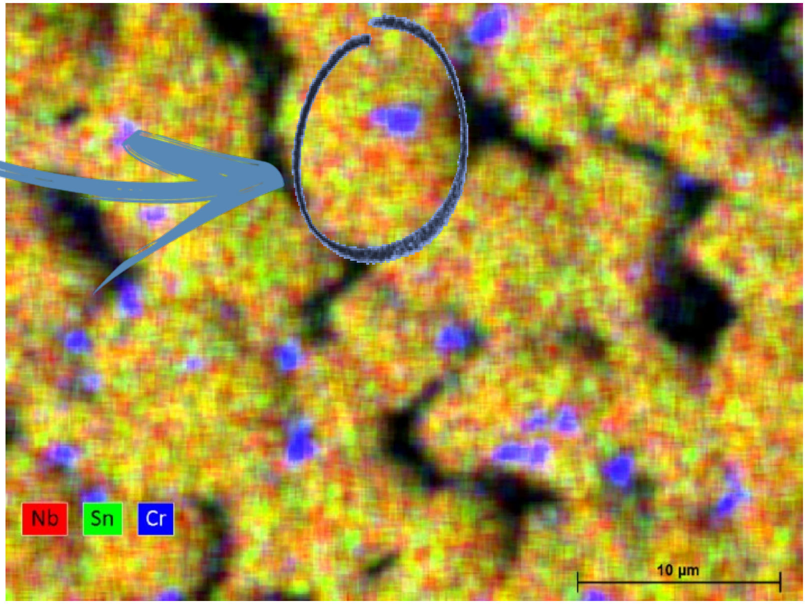
Sn 25 %



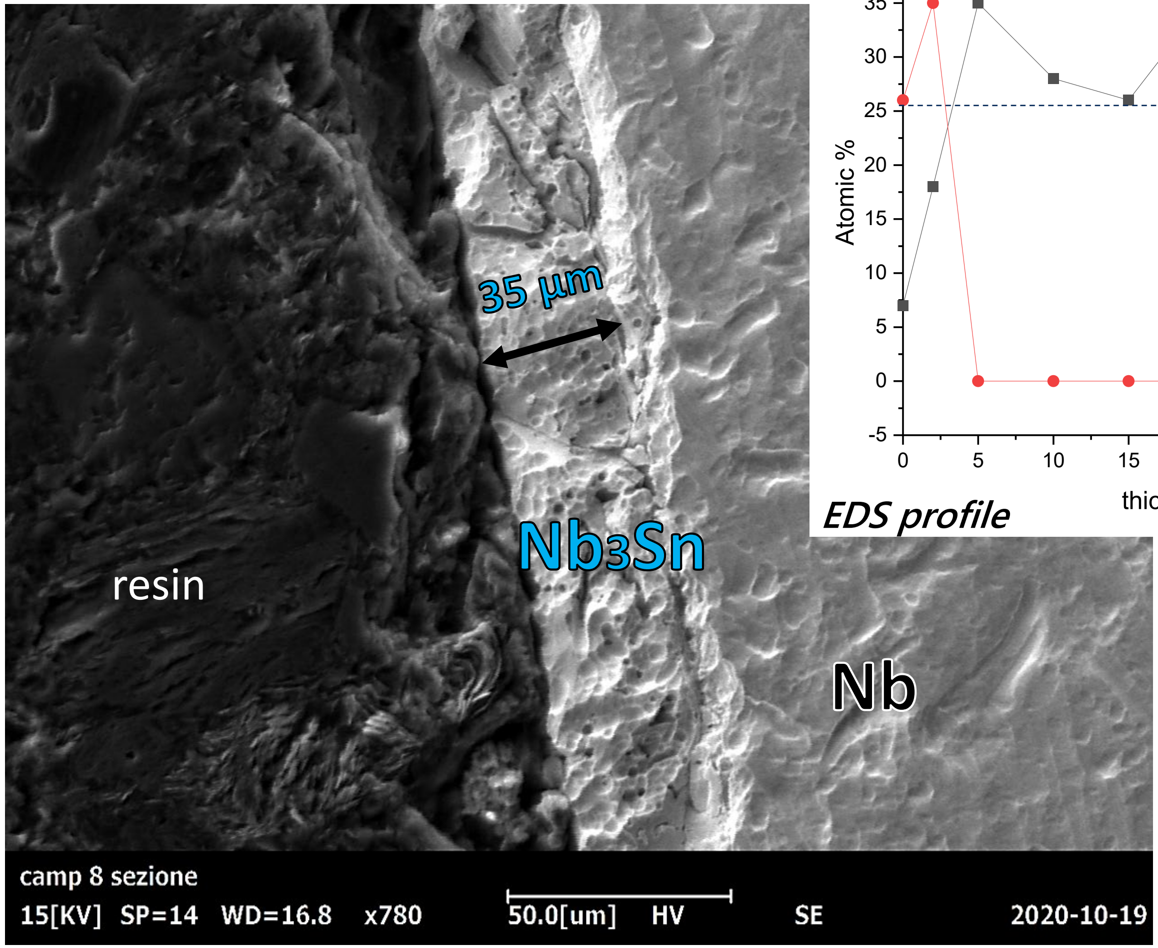
Chromium contamination

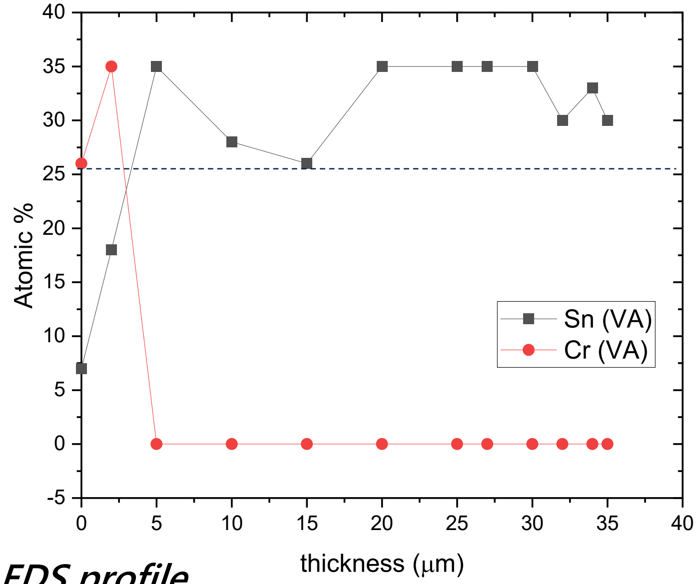
(10-20% on surface)

Ni and Fe are also present



Target cross section





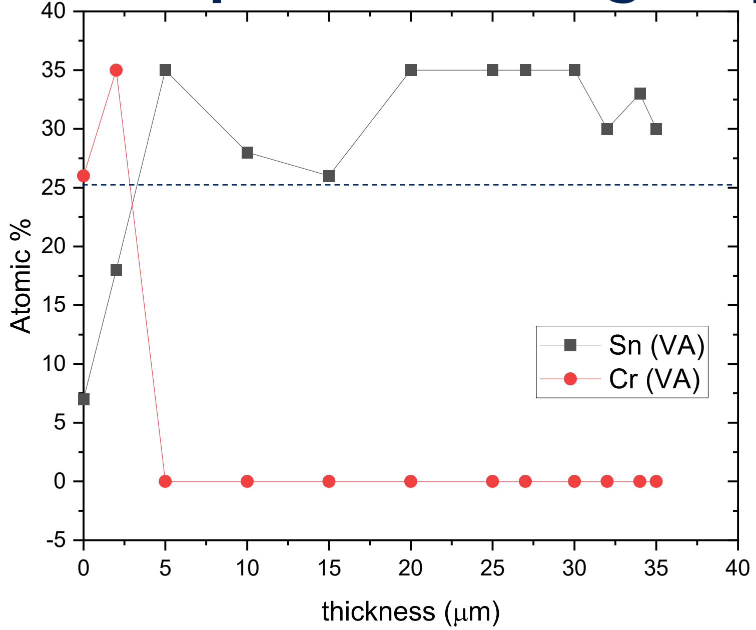
EDS profile

- **Contamination is superficial**
- **No contamination in Sn crucible**

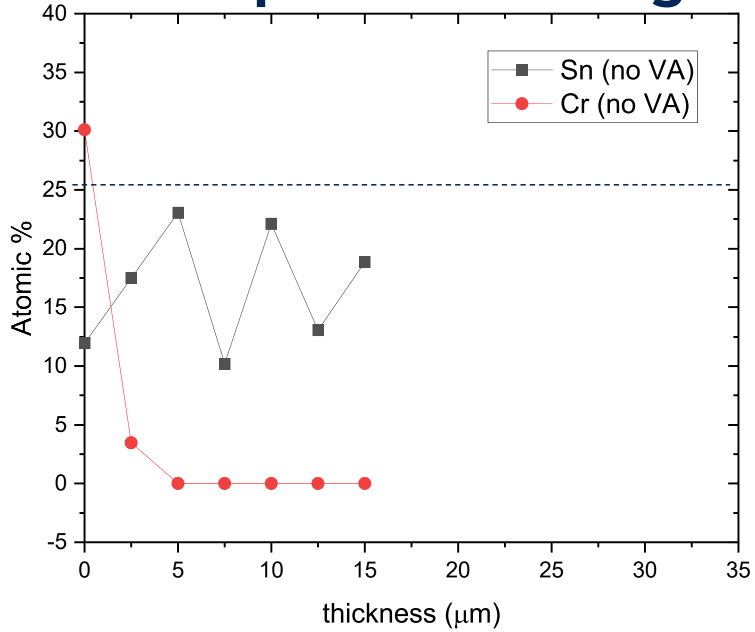
Main candidate for Cr contamination:
Vacuum chamber made by **Inconel Alloy**

Tin content modulation

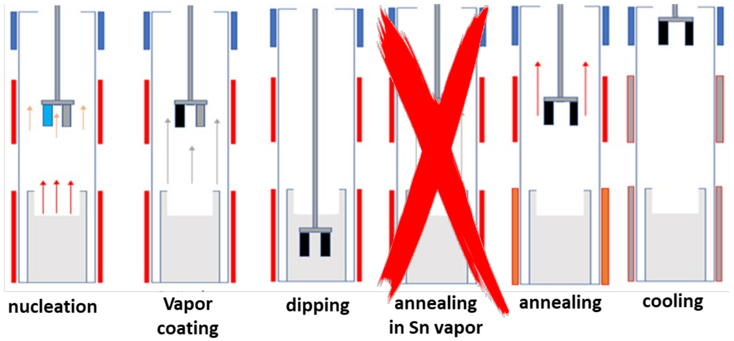
With Vapour Annealing Step



Without Vapour Annealing Step



Vapour annealing step can be used to modulate Tin content



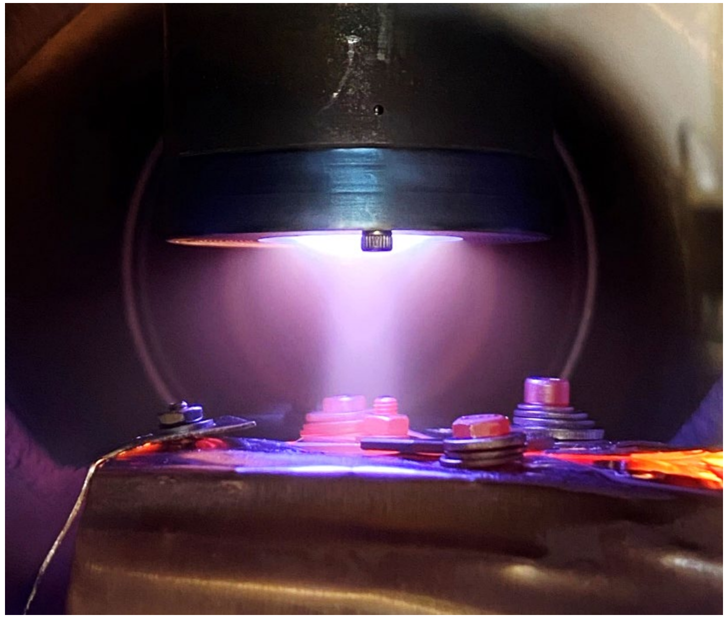
First sputtering test on Quartz

- **1" target** prepared by **Dipping** (30 microns thick)

- Coating on **quartz** samples by MS

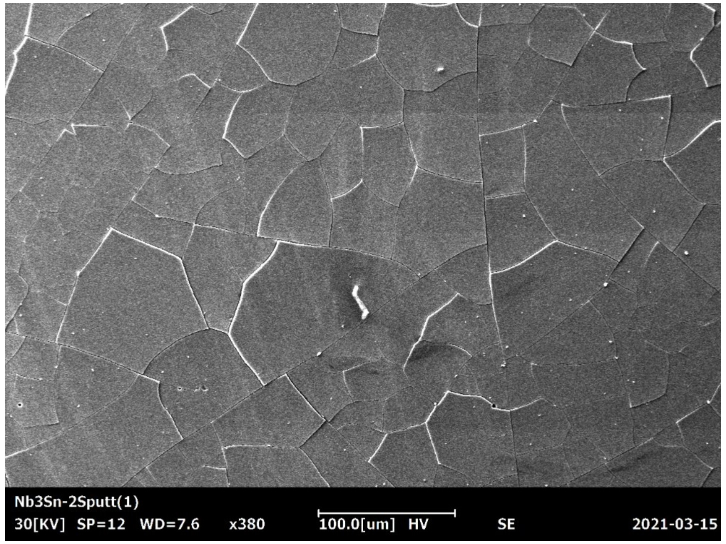
  I = 0.1 A (5 mA/cm^2)
  t = 30 min
  T = 750 °C

- Process stopped when V started to decrease

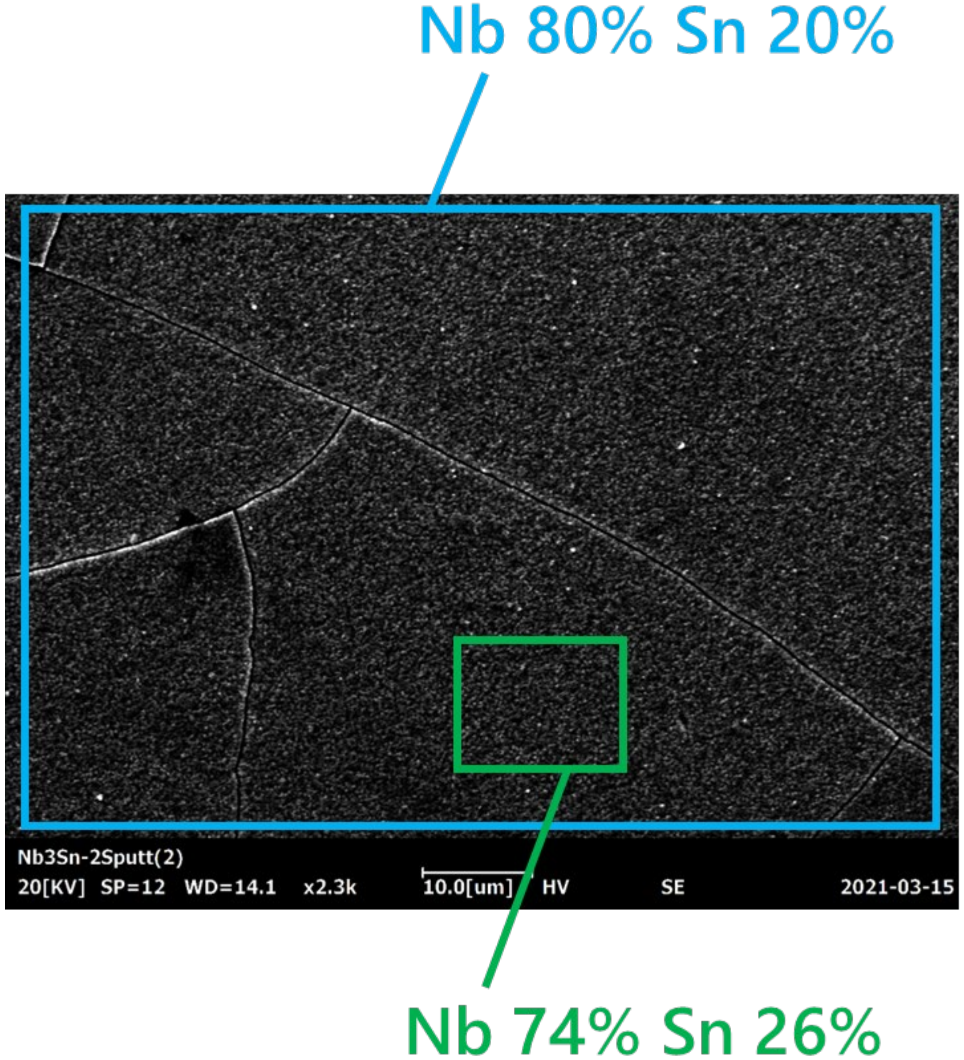


Zanierato et al,
SRF Proceedings 2021

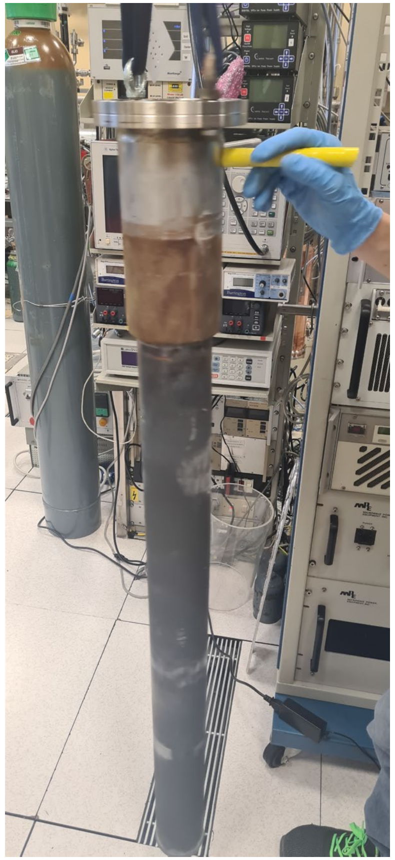
First sputtering test on Quartz



- **Good composition**
- **No Cr** visible by EDS
- **Cracks due to Quartz substrate**
- **No Tc measured**
- **2 New targets ready for new tests**



Solution to Cr contamination



Inconel chamber replaced

by a **Nb chamber**

produced at Zanon

New problems have come…

Nb is not stable as Inconel at high T in air

We tested several solutions without success:

- Nb anodization
- High T paints
- Allumina by HVOF

After the first test
(30 min @ 1000 °C)

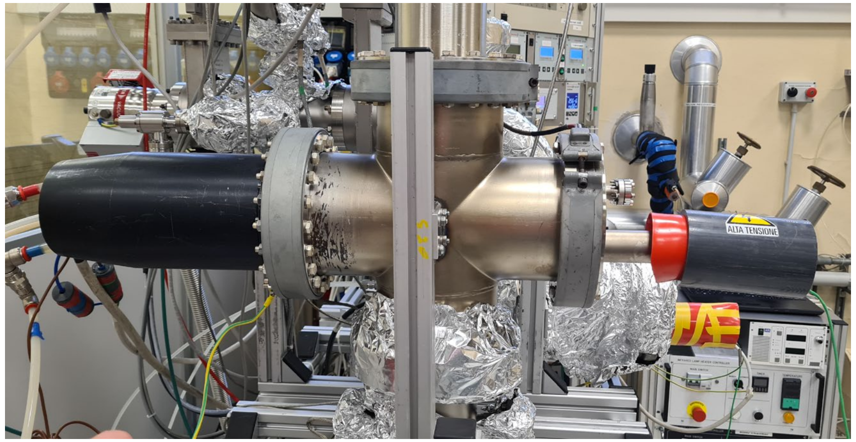
a **very thick film of Nb_2O_5**
growth on the external surface

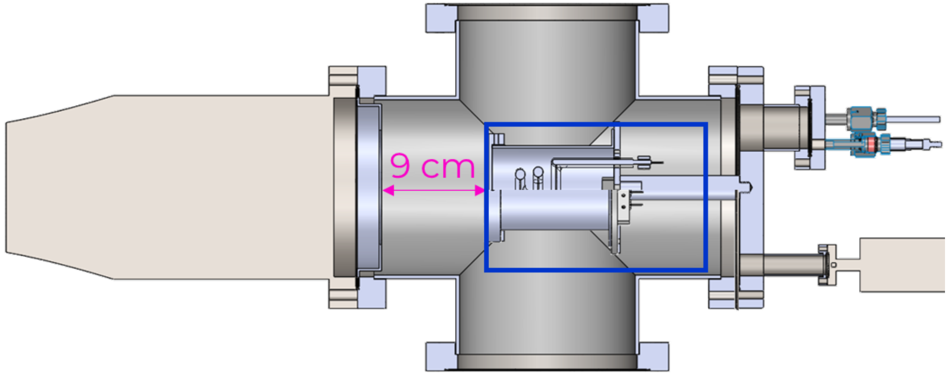
We are redesigning the system with inductive heating and a double chamber to avoid oxidation of Nb chamber

2. Magnetron sputtering parameters optimization

INFN

Experimental Set-up





- Single 4” Nb_3Sn stoichiometric target (commercial)
- P < $5*10^{-9}$ mbar
- I=0,25 A ($3mA/cm^2$)
- V= 300-400 V

Parameters investigated

- **Deposition and annealing Temperature**

  (550, 600, 650, 700, 750 C)

- **Annealig time**

  (0, 24, 48 hours)

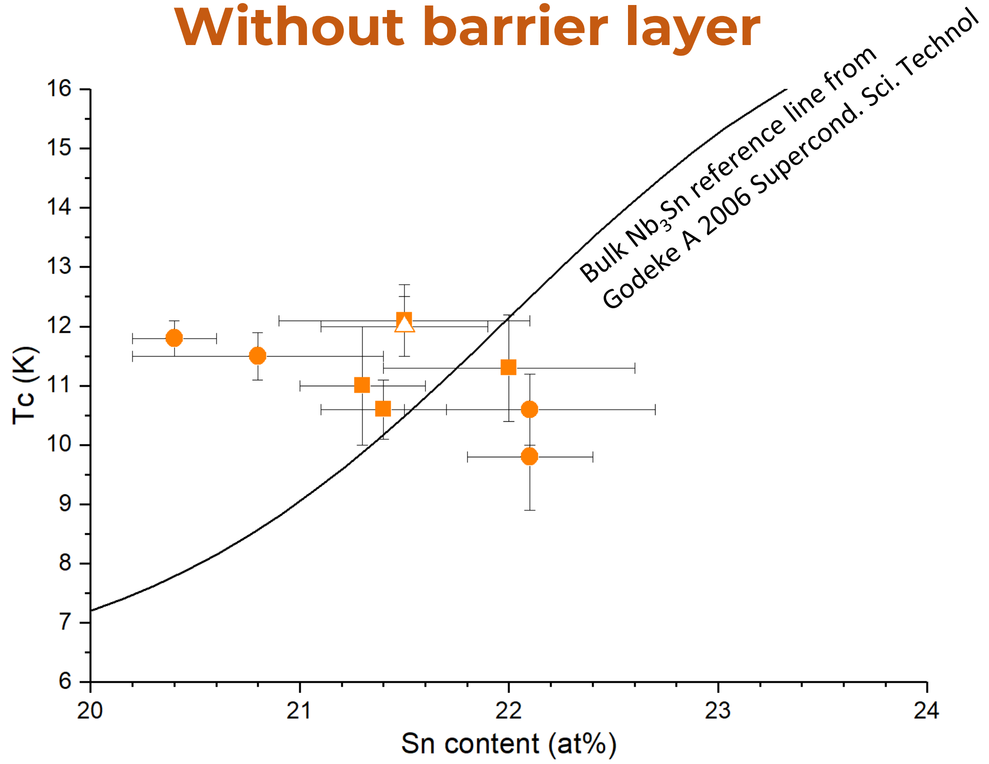
- **Ar Pressure**

  ($3*10^{-3}$, $7*10^{-3}$, $2*10^{-2}$ mbar)

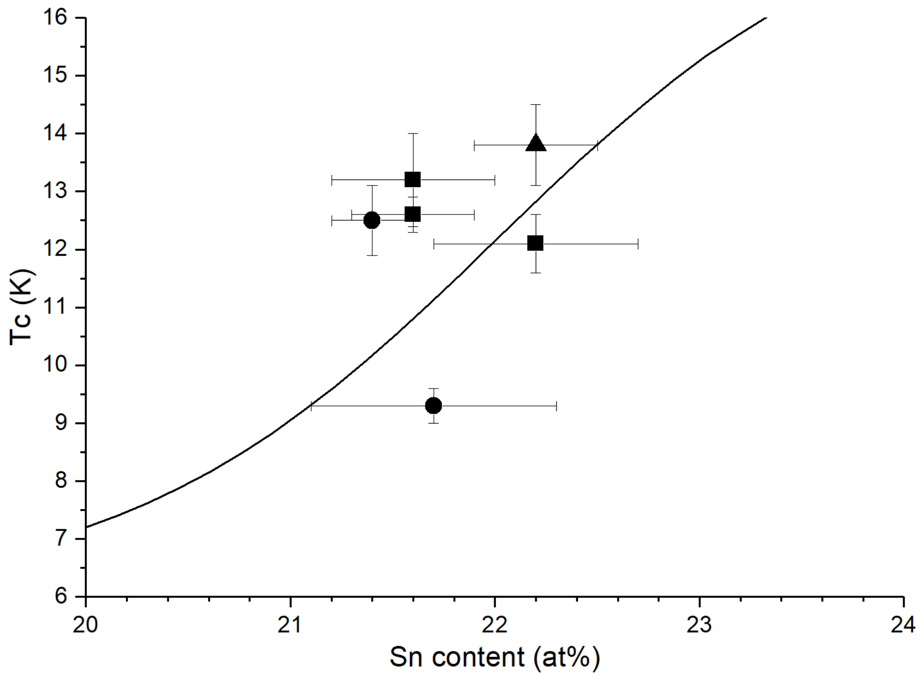
Effect of Nb barrier layer
(0, 0.5, 1.0 micron)

Tc VS Sn content

Without barrier layer



With 1 micron Nb barrier layer



All samples present low Sn content

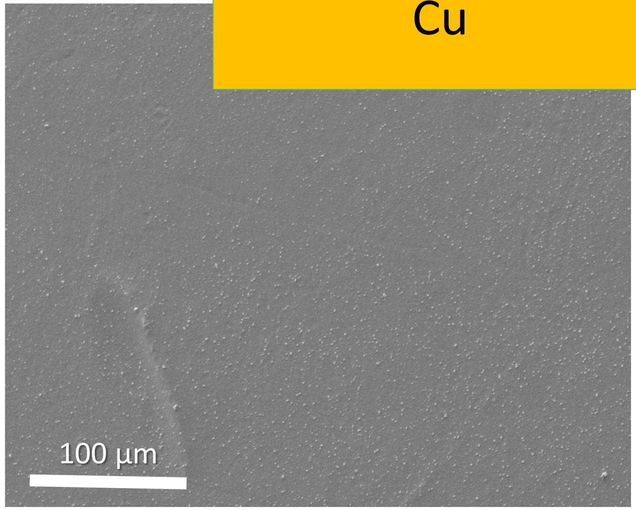
Spread distribution of Sn content

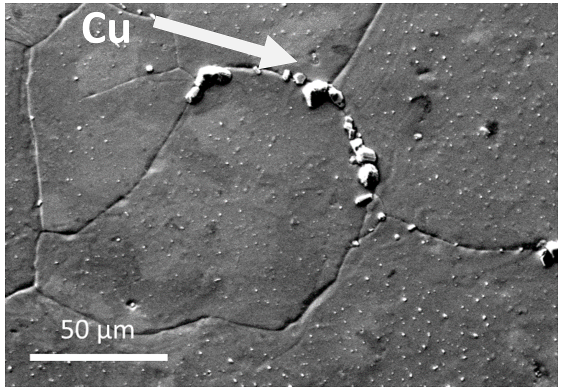
Nb barrier layer increase Tc

and stabylize Sn content

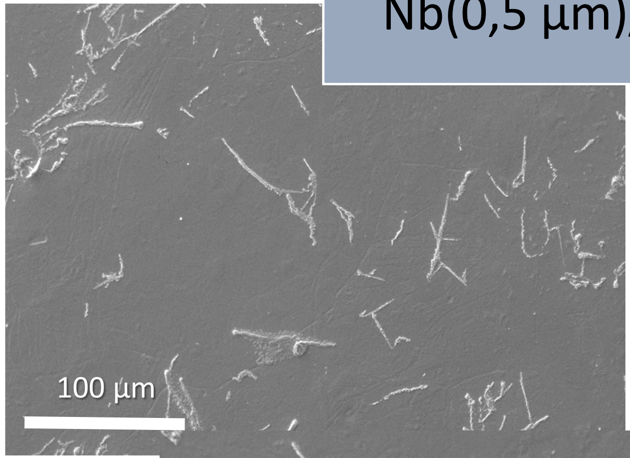
PVD Nb_3Sn Film morphology

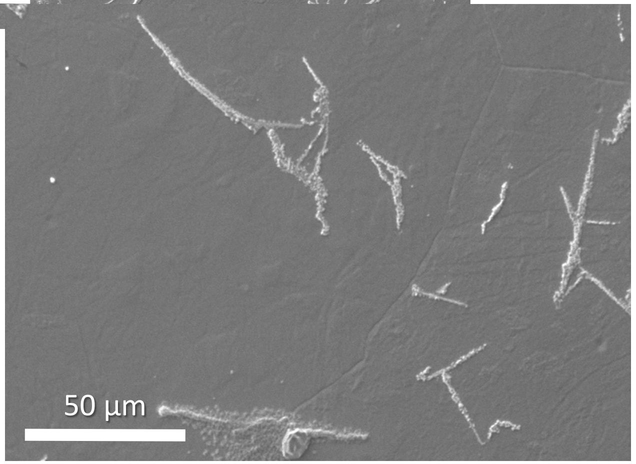
Cu



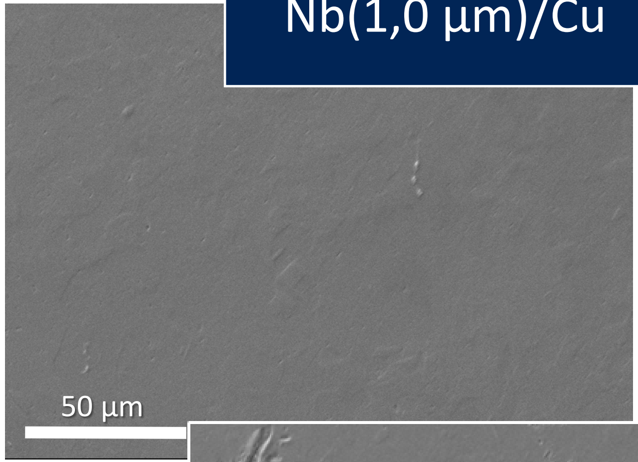


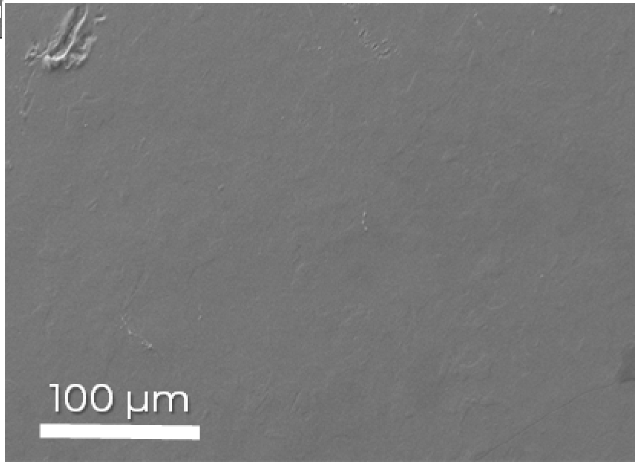
Nb(0,5 µm)/Cu



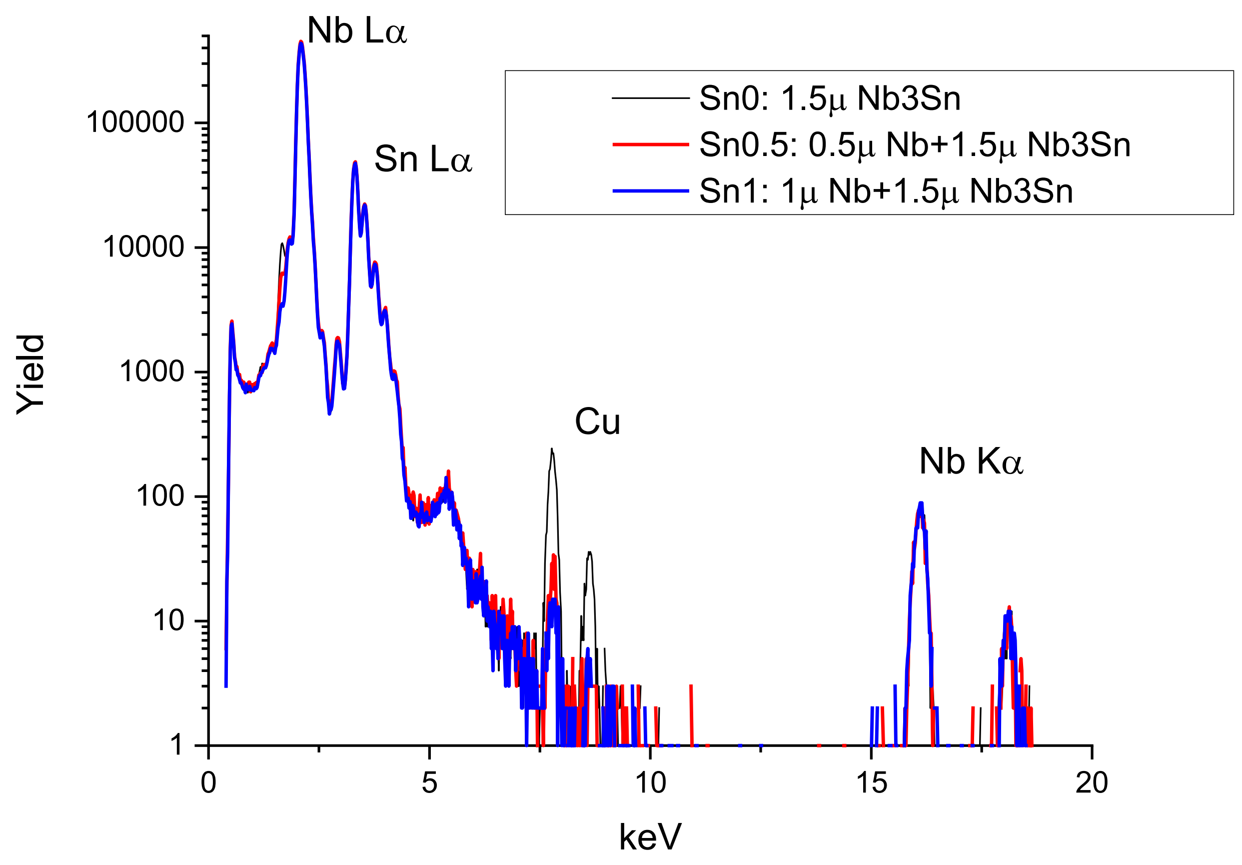


Nb(1,0 µm)/Cu



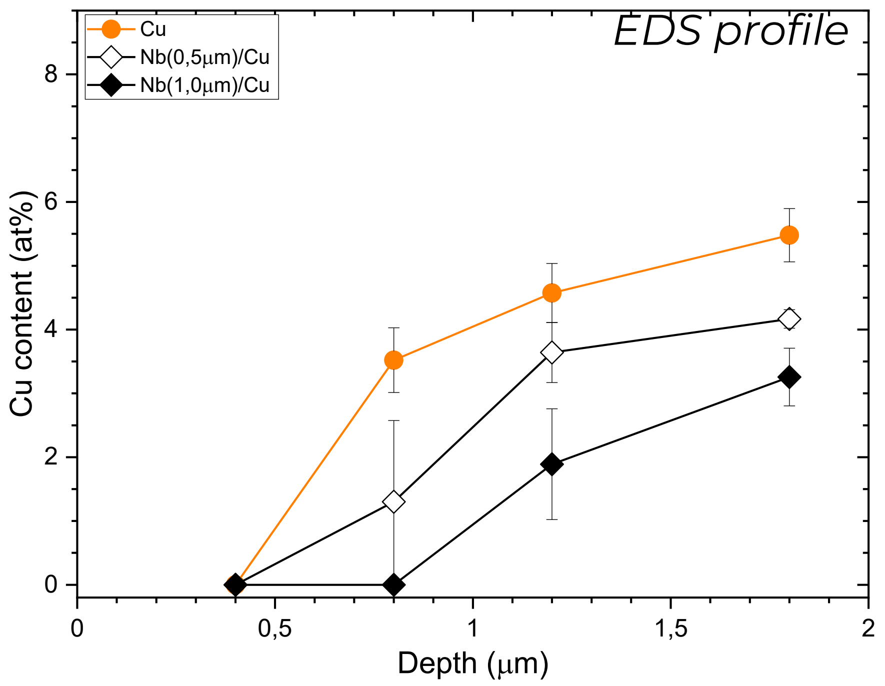


Cu diffusion into Nb_3Sn?



PIXE Analysis courtesy of S. Prucnal and S. Zhou, HZDR

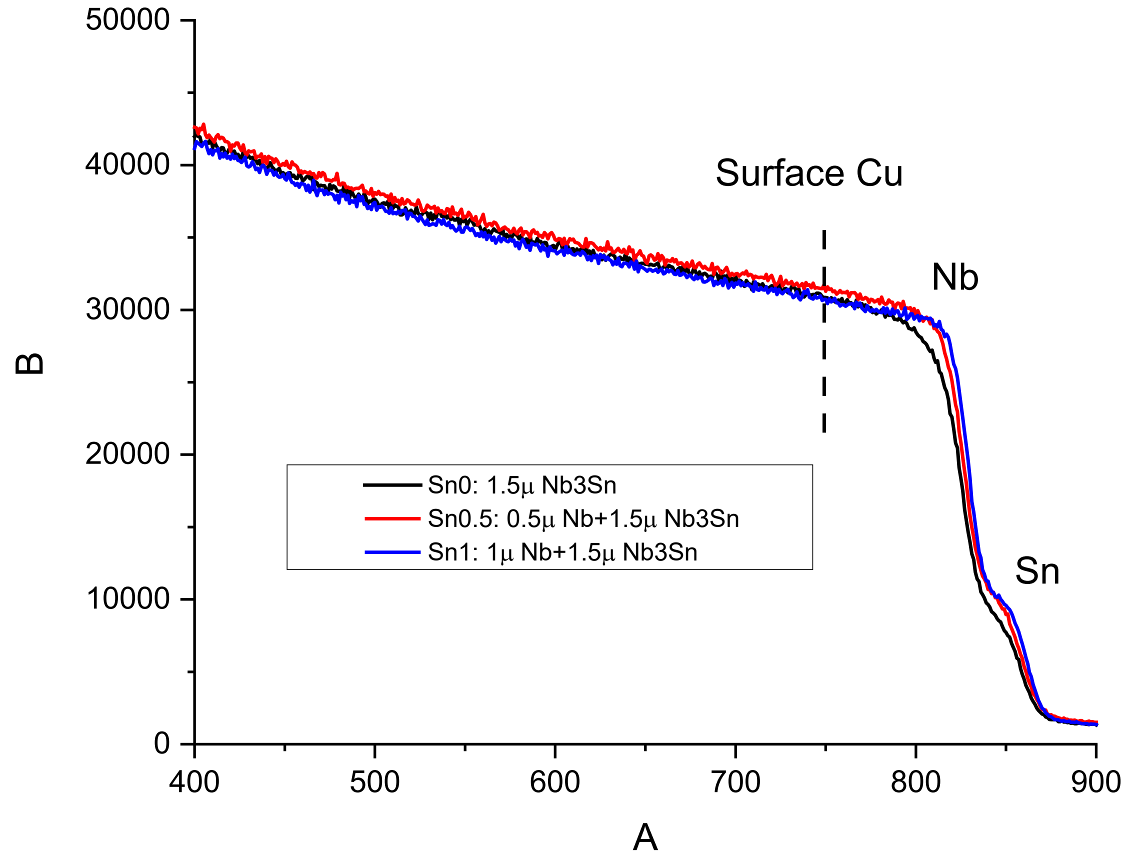
T=730 C, P=7E-3 mbar, Annealing t=24h



Nb barrier layer reduce Cu signal both in EDS and PIXE spectra

Can be an effect of the total coating thickness…

Cu diffusion into Nb_3Sn?



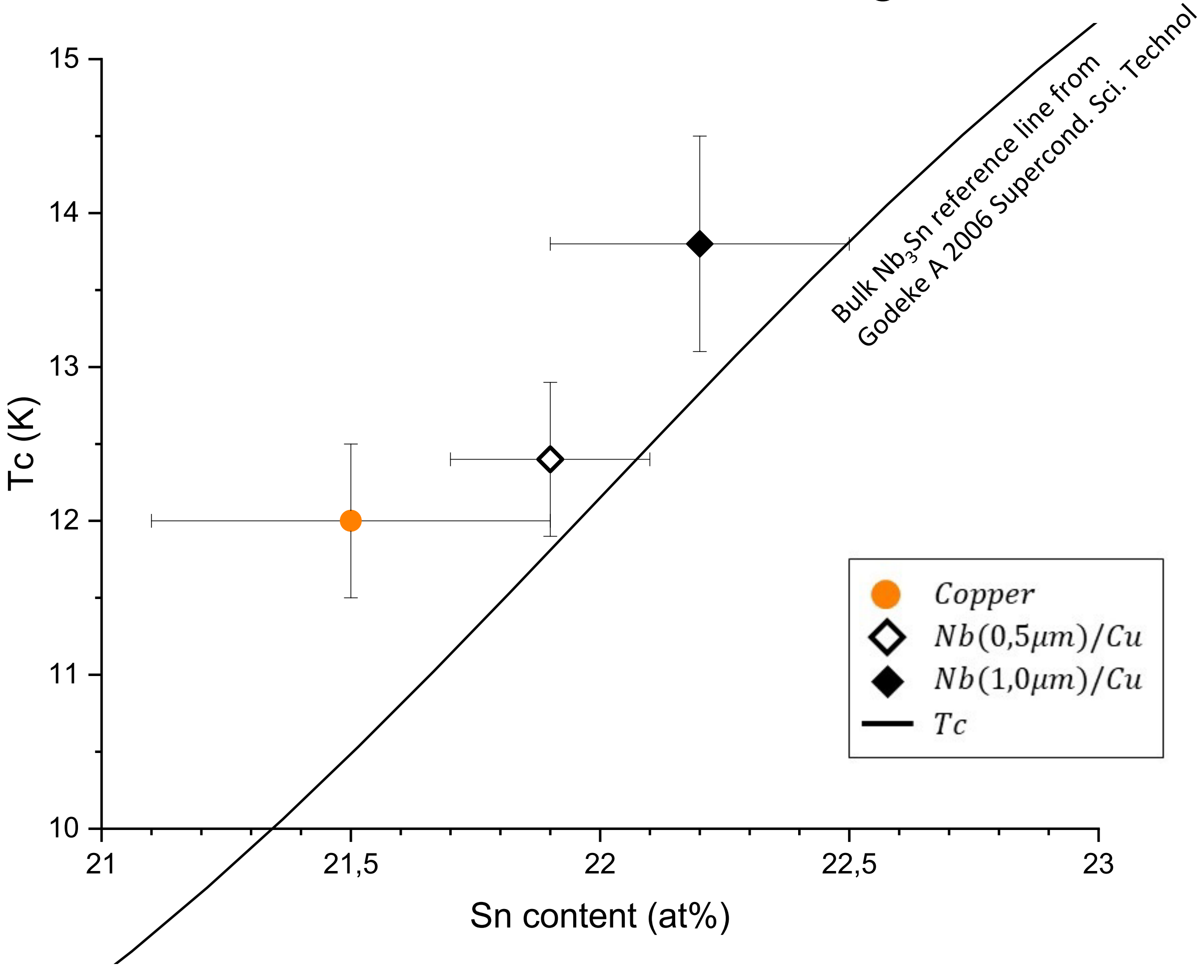
RBS profile instead **do not show Cu**

(RBS investigation depth ~ 0.5-1 micron)

RBS Analysis courtesy of S. Prucnal and S. Zhou, HZDR

Nb barrier **effect**

T=650 C, P=2E-2 mbar, Annealing t=24h

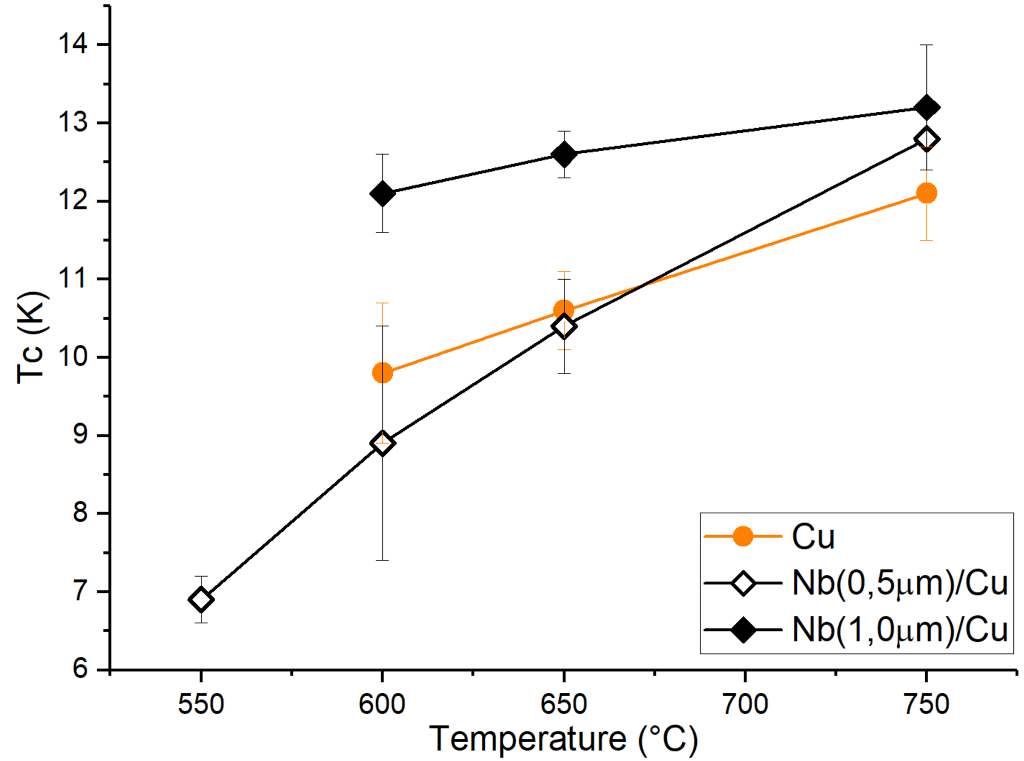


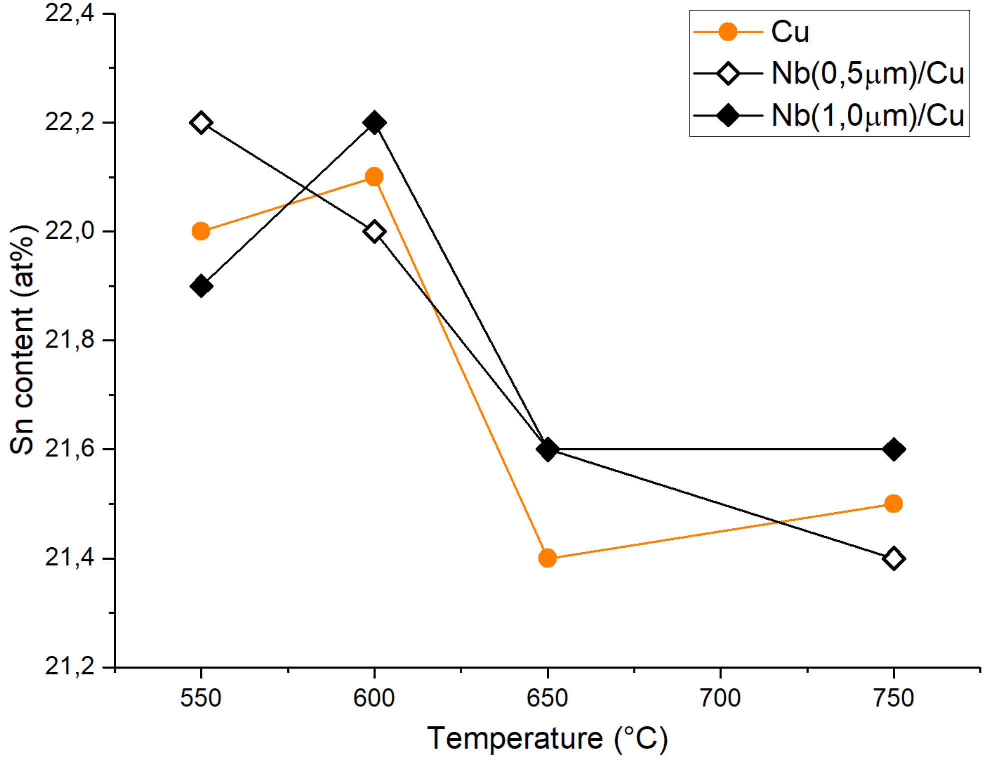
The **Nb barrier layer** seams **reduce Sn migration to Cu**

A cross section is mandatory for better understanding

Temperature effect

P=3E-3 mbar, Annealing t=24h





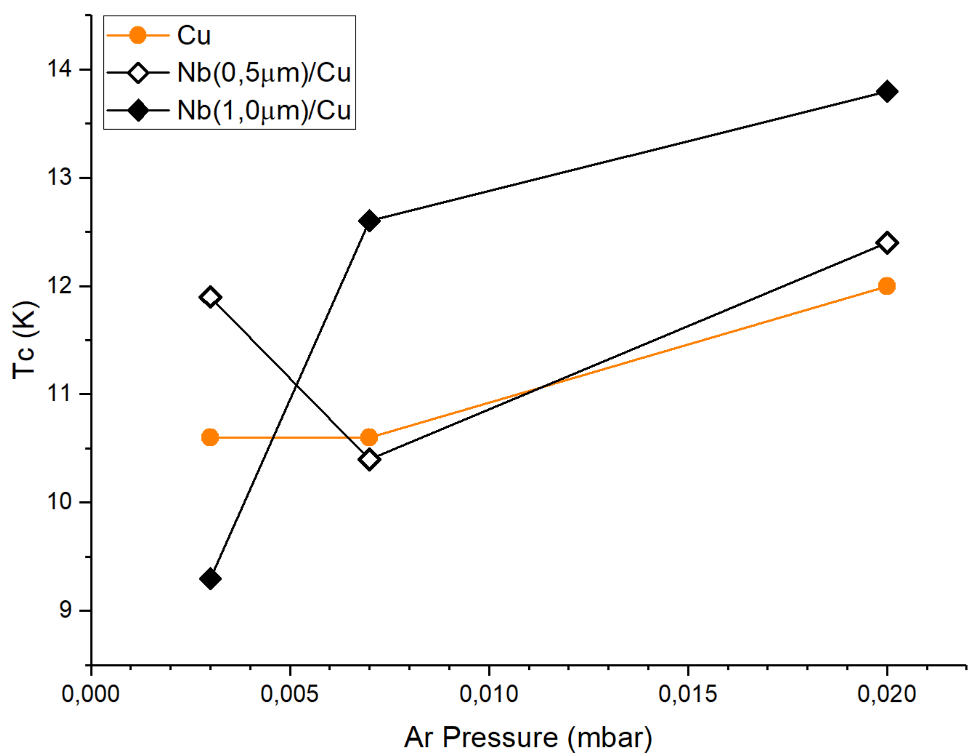
↑ Temperature ↑ Long-range crystallographic order ↓ Sn content

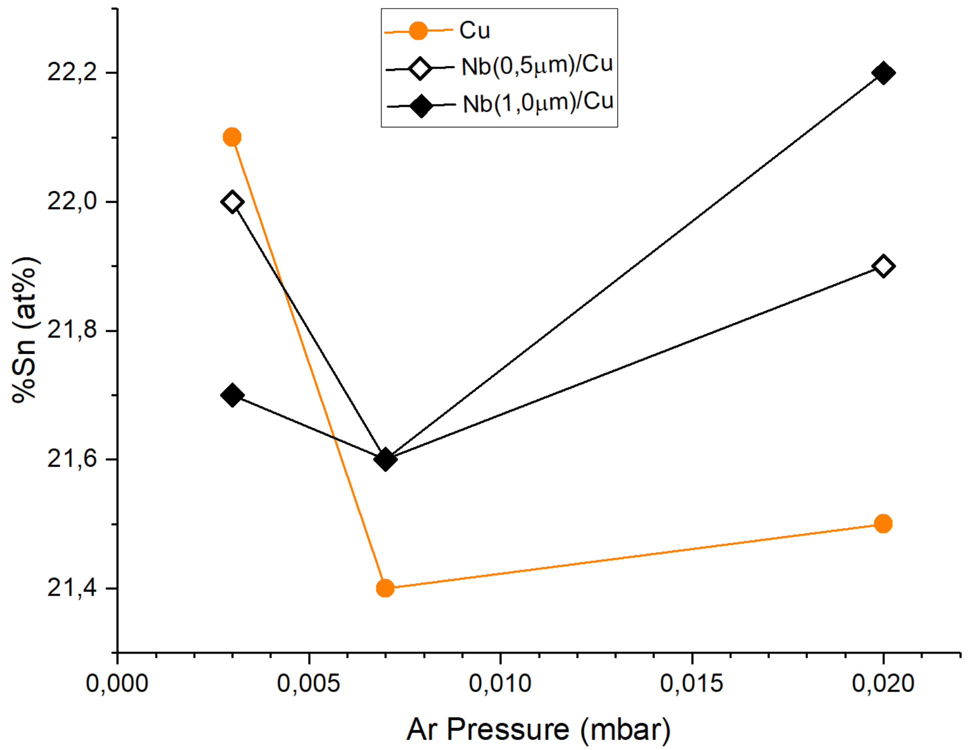
P effect

Pressure

T_c

T=650 C, Annealing t=24h





Conclusions

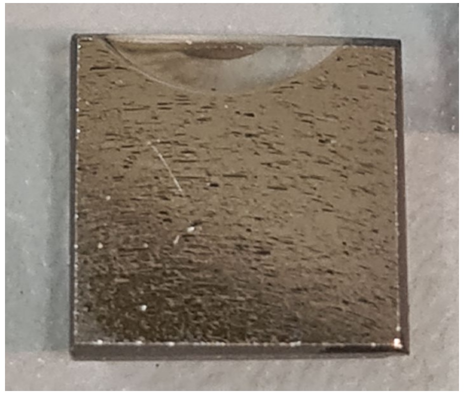
- Proved the possibility of realizing targets via LTD
- Easy modulation of target Tin content
- Nb barrier layer improve PVD film quality

Two main aspect to optimize:

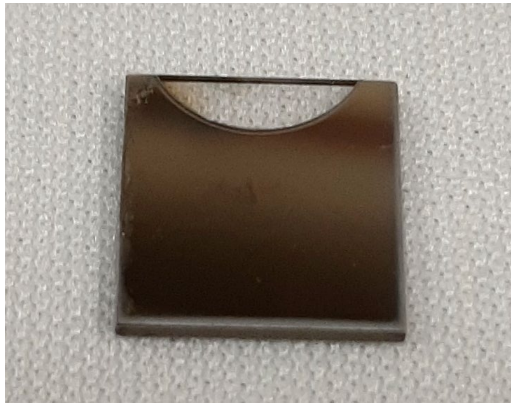
→ **Chromium poisoning**

→ **Increase Sn content of sputtered coatings**

Take home message

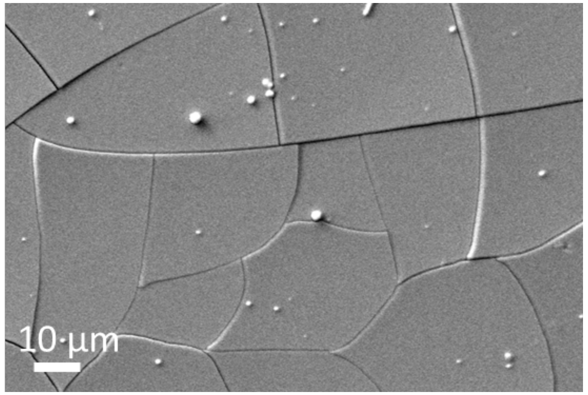


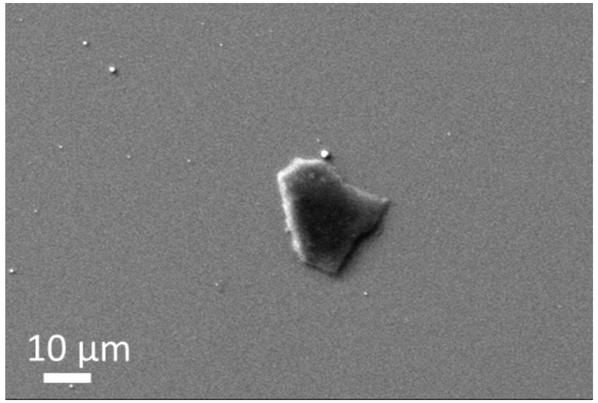
Quartz

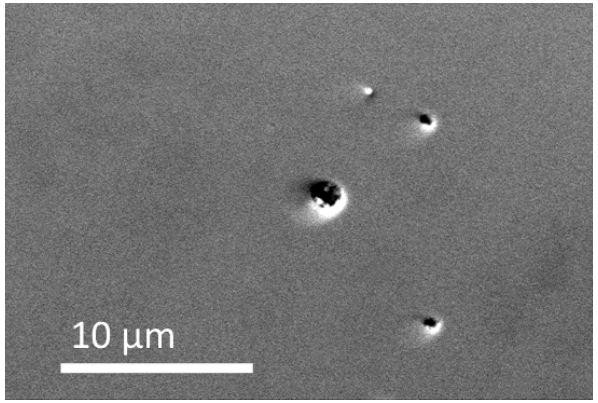


Sapphire

Copper







Do not use quartz for temperatures above 550 C!

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Special thanks to
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Thanks for your attention

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