thinfilms and NEW IDEAS for SRF

The 9th International Workshop on Thin films and new ideas for Pushing the limits of RF Superconductivity

15-18 March 2021 - Virtual Edition

Cristian Pira

Nb_3Sn films via liquid tin diffusion for SRF application

INFN LN Legnaro
Istituto Nazionale di Fisica Nucleare
Laboratori Nazionali di Legnaro

Work supported by the INFN CSNV experiment TEFEN

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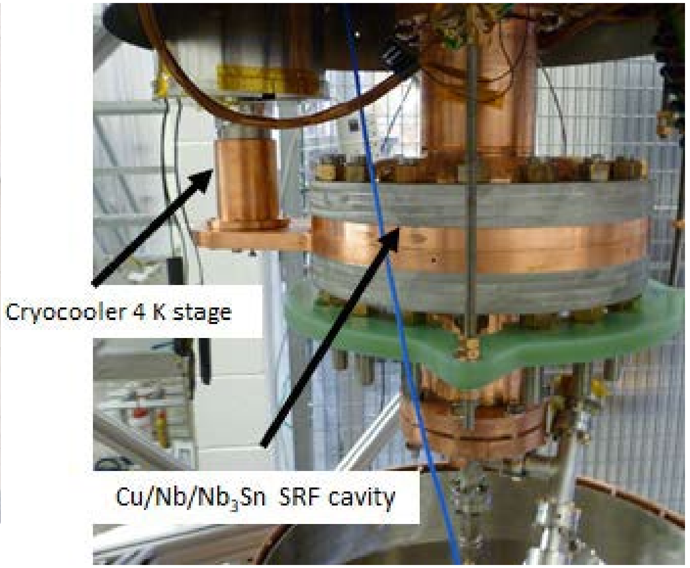
ARIES

Nb_3Sn on Cu - Motivation

High performance of Nb_3Sn **@ 4.2 K** → cooling by **cryocooler**

High thermal conductivity substrate is preferred

Courtesy of G. Ciovati, JLab



Nb_3Sn @ LNL - Motivation

Goal of I.FAST collaboration → Produce a Nb_3Sn coated cavity on Cu

Request → A Nb_3Sn target for cylindrical configuration

Idea → **Single-use Nb_3Sn target by Liquid Tin Diffusion (LTD)**

LTD Motivation

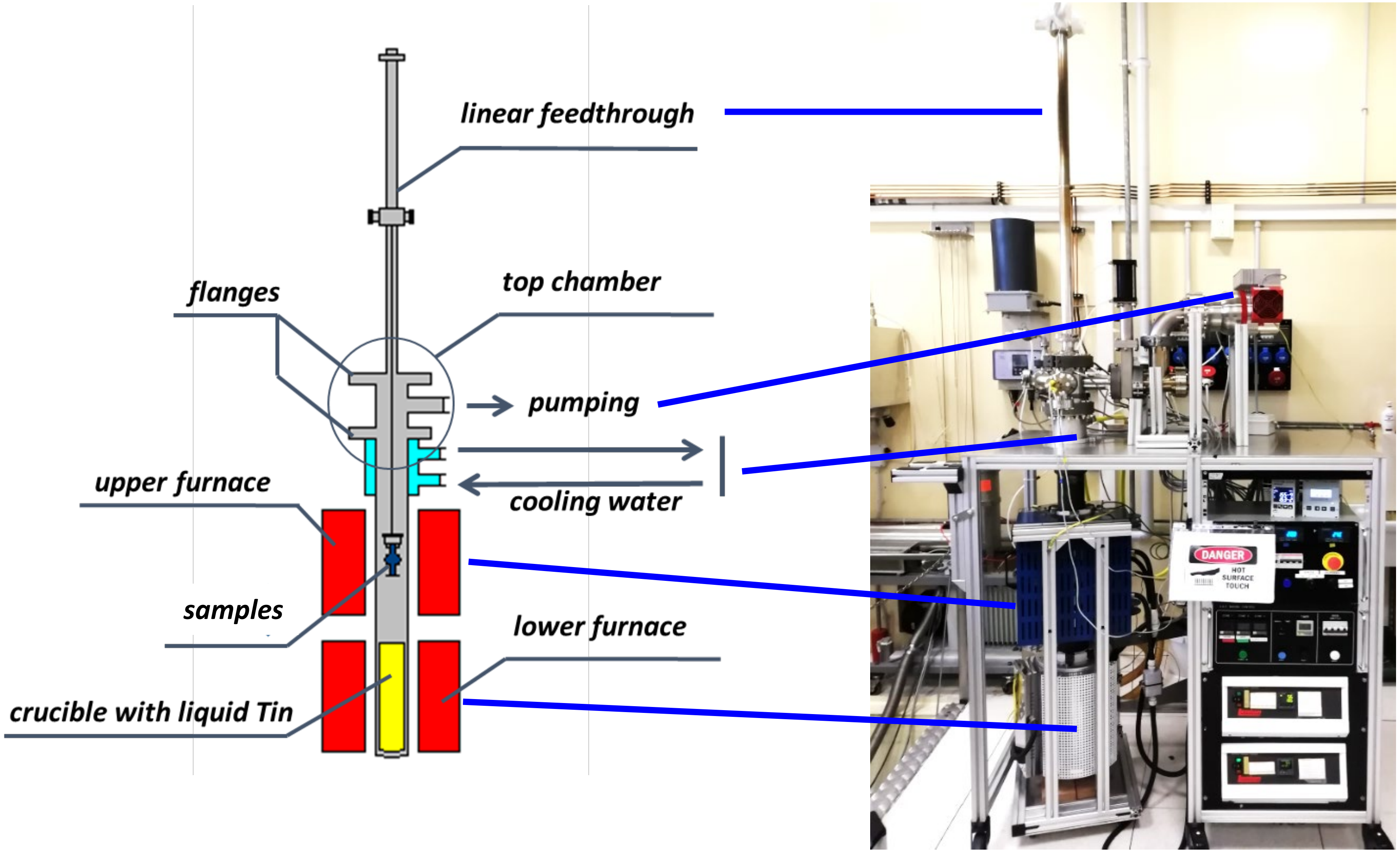
Already explored for SRF @LNL (from 2005 to 2010)

Simple process

Possibility to adopt solutions developed for Tin Vapour Diffusion

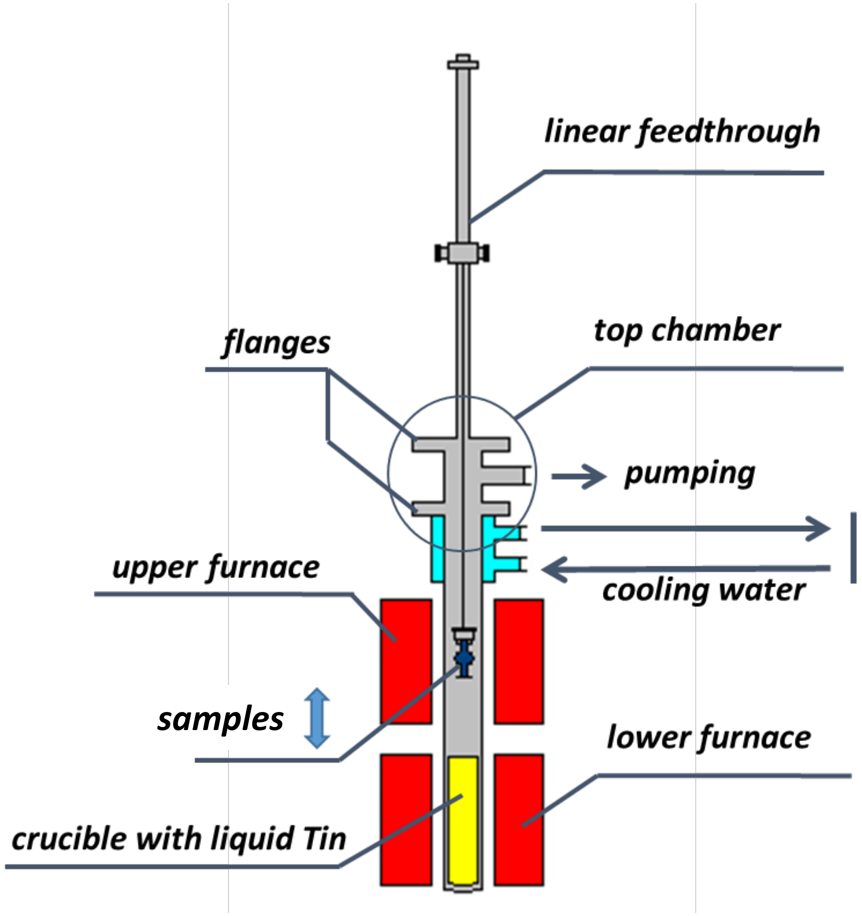
Possibility to **grow thick layer** of Nb_3Sn (> 10 µm)

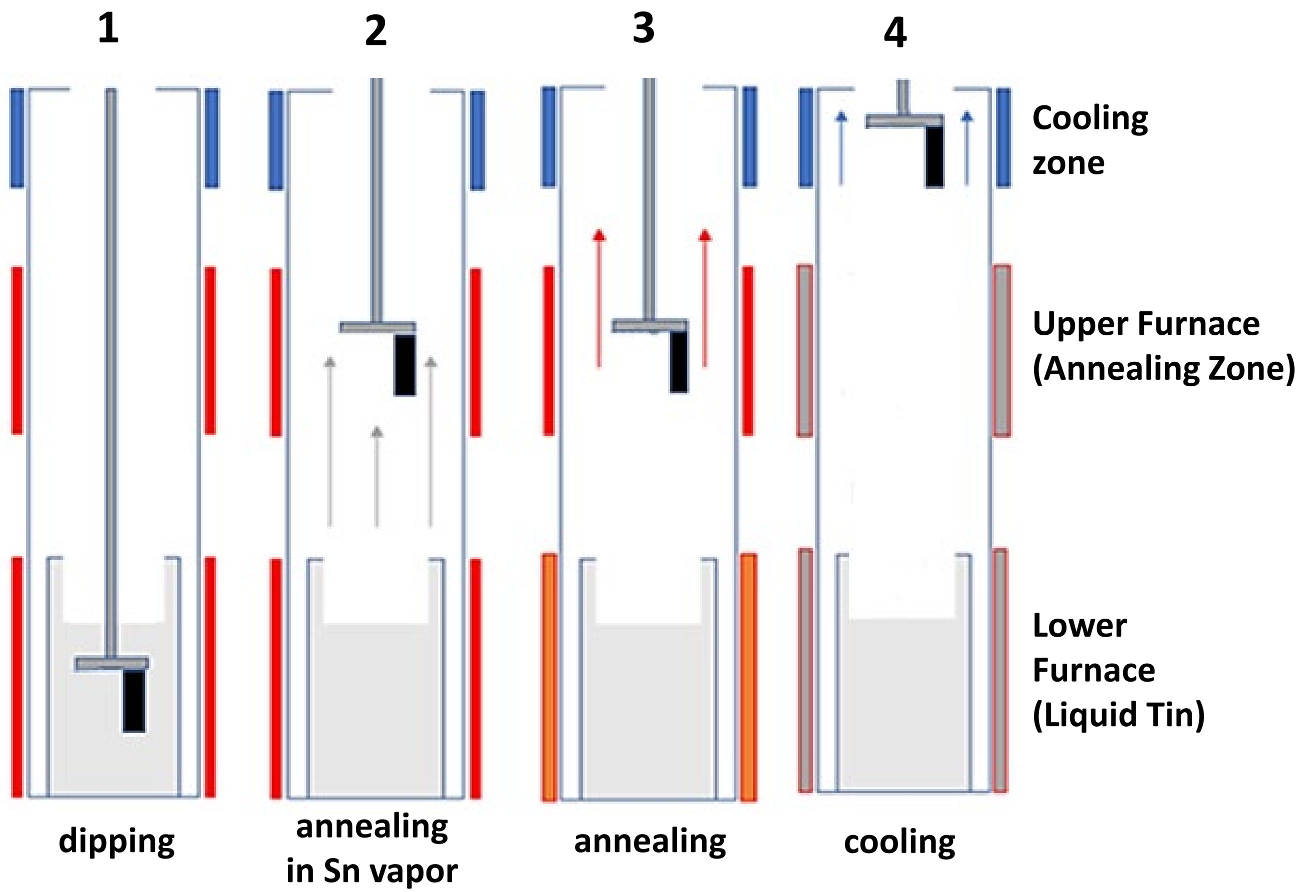
Liquid Tin Diffusion process set-up



Liquid Tin Diffusion process (LNL 2006)

S. M. Deambrosis et al., "A15 superconductors: An alternative to niobium for RF cavities," Physica C, 2006





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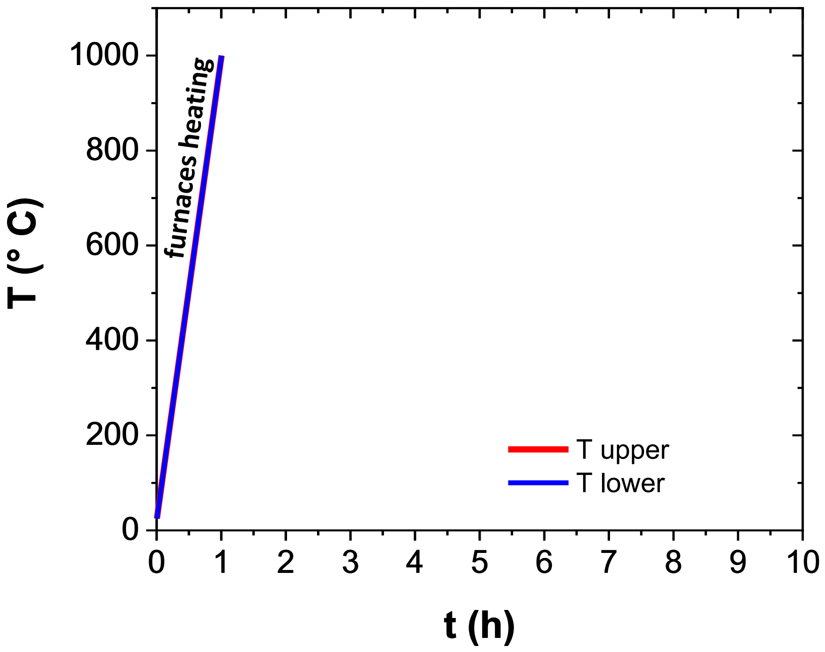
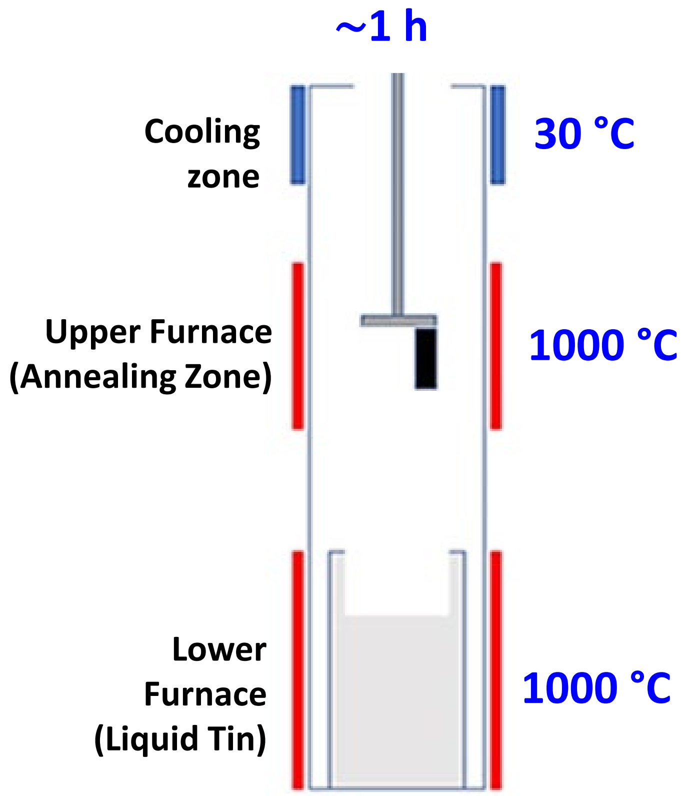


Temperature furnaces profile



furnace heating

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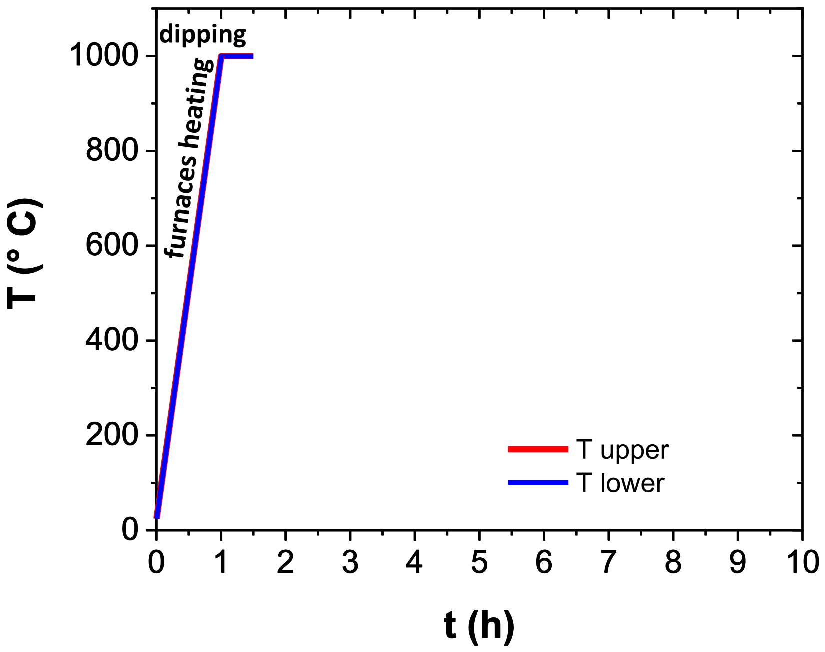

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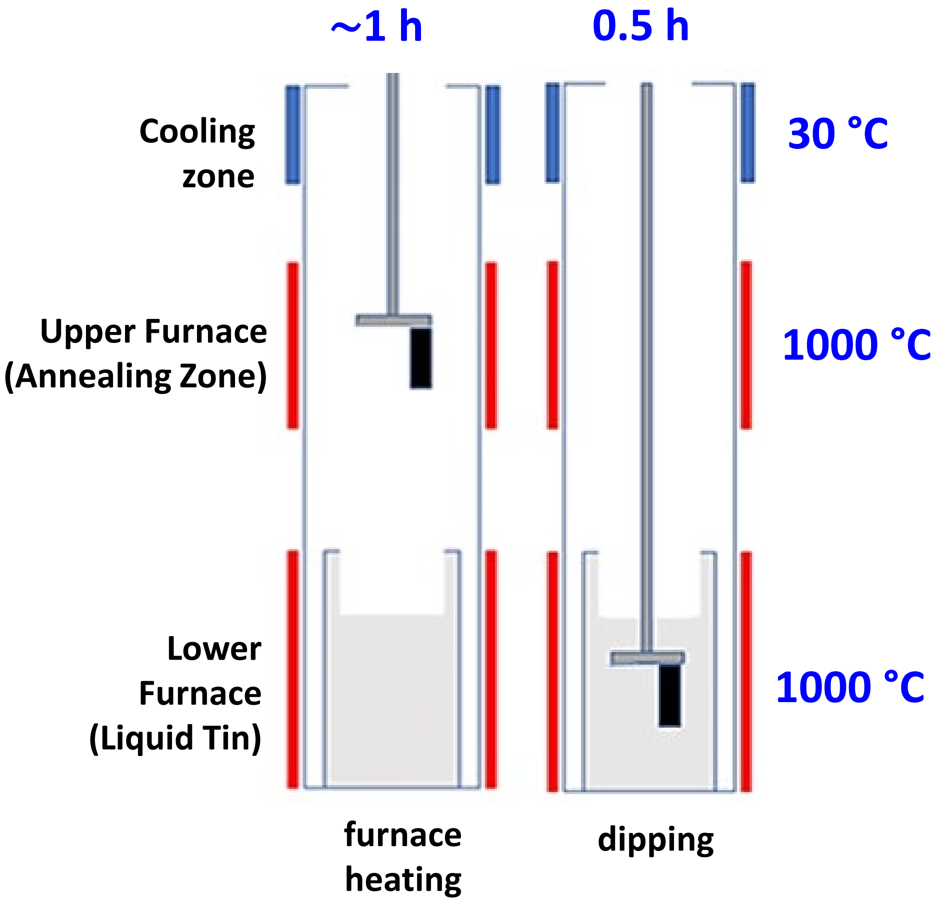

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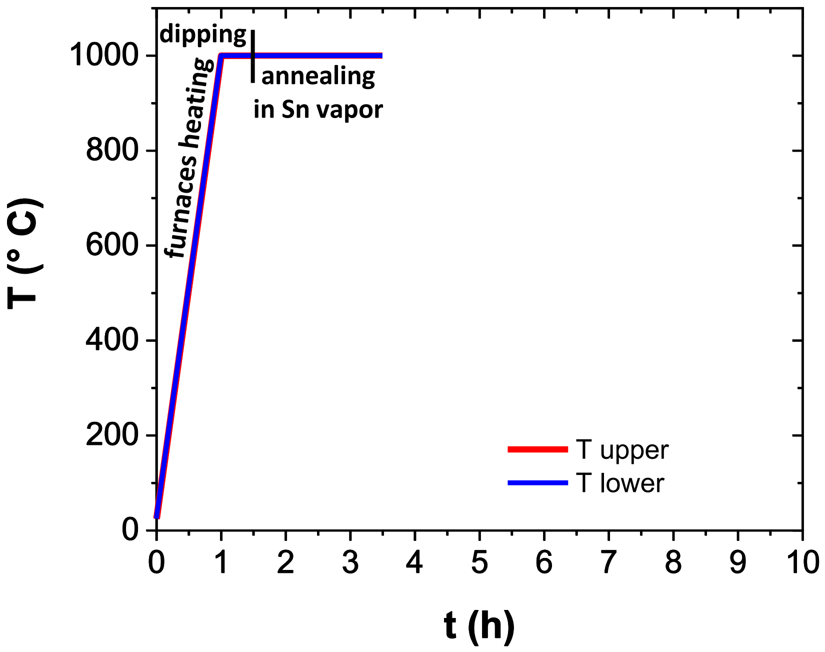

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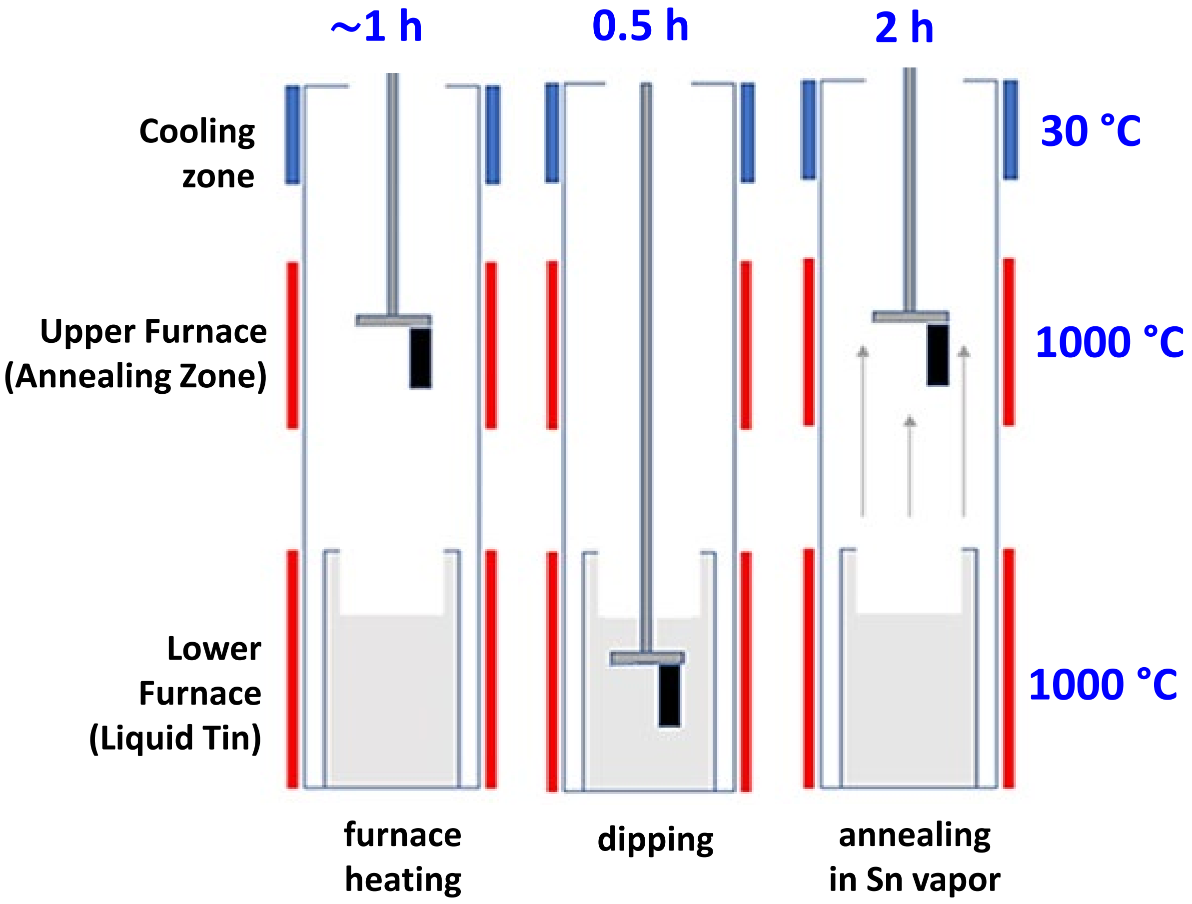

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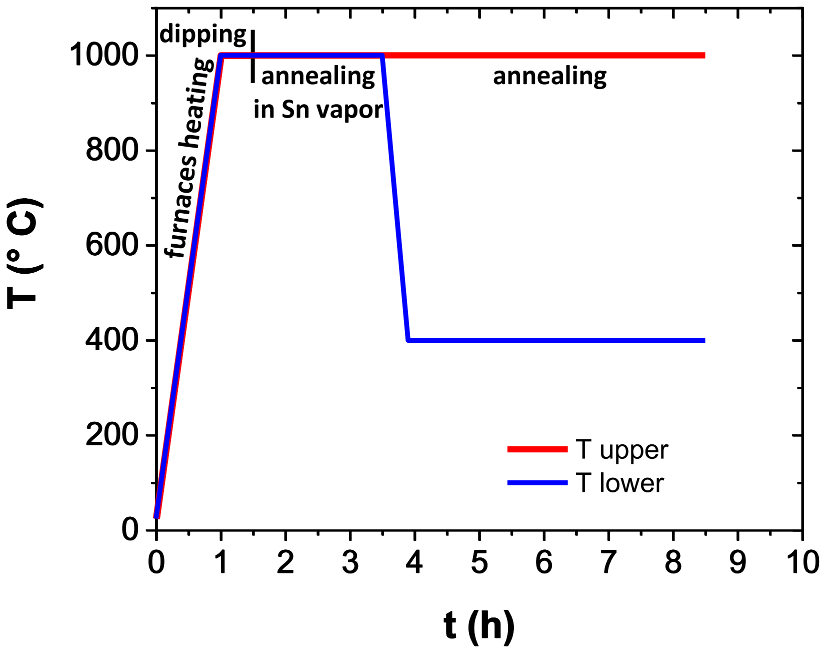

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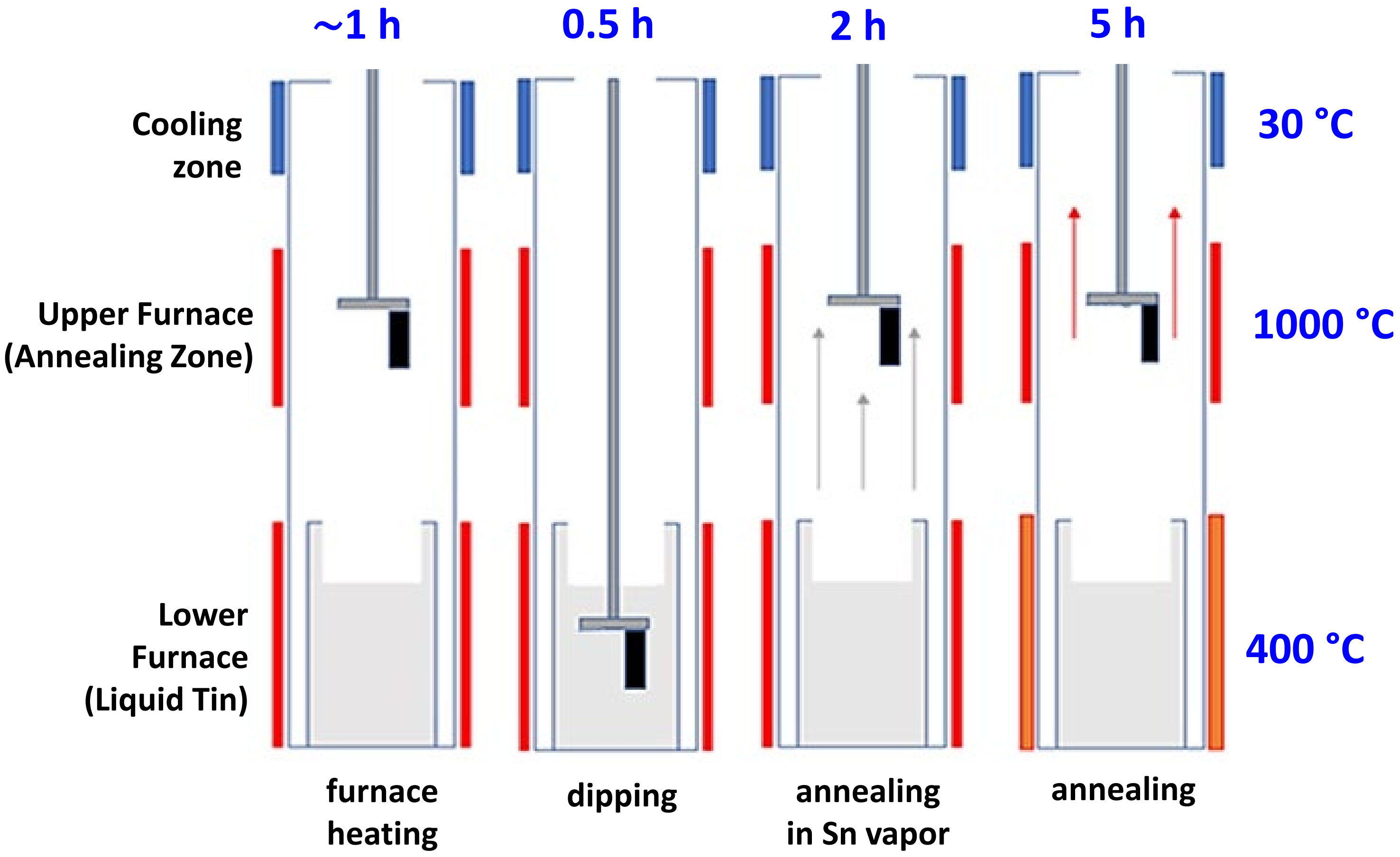

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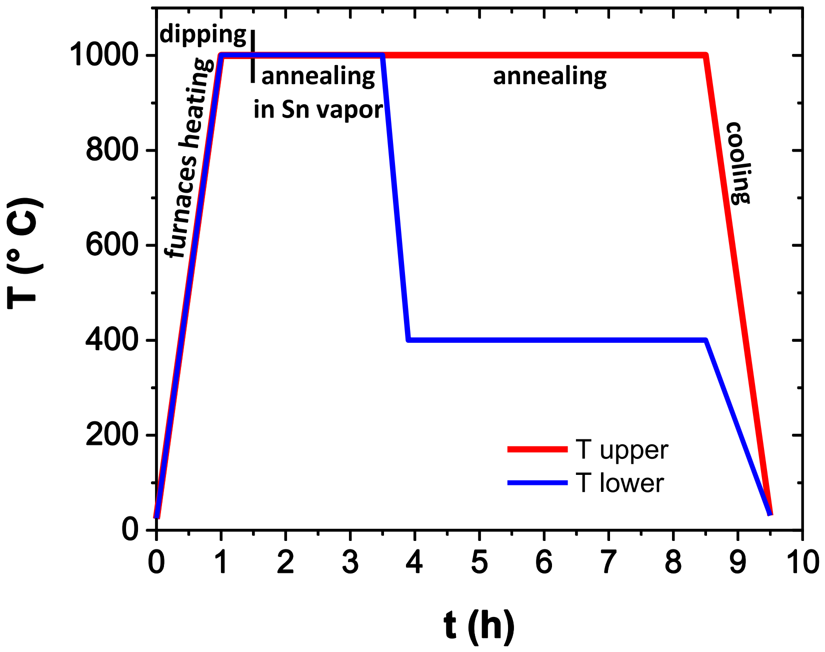
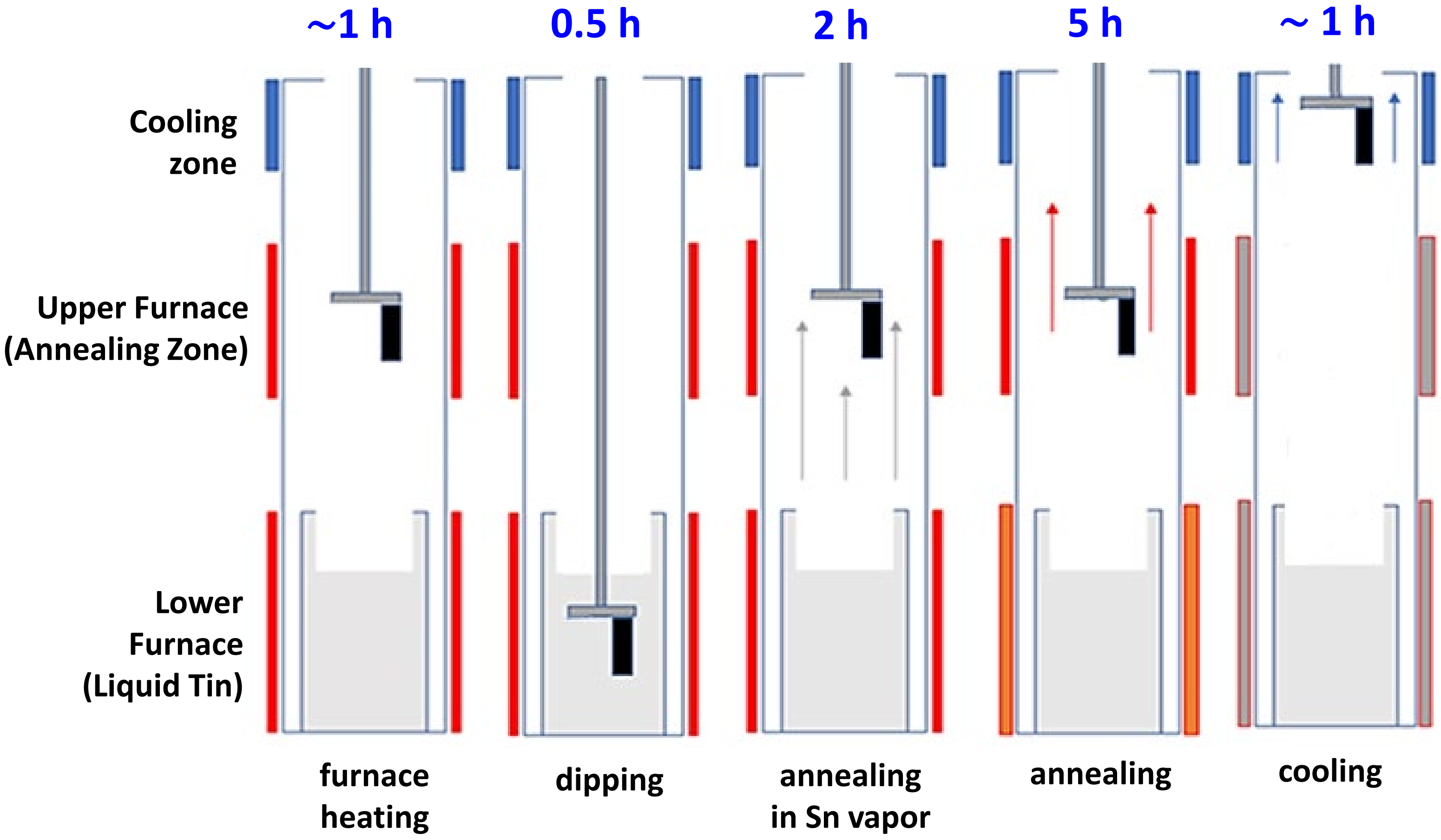
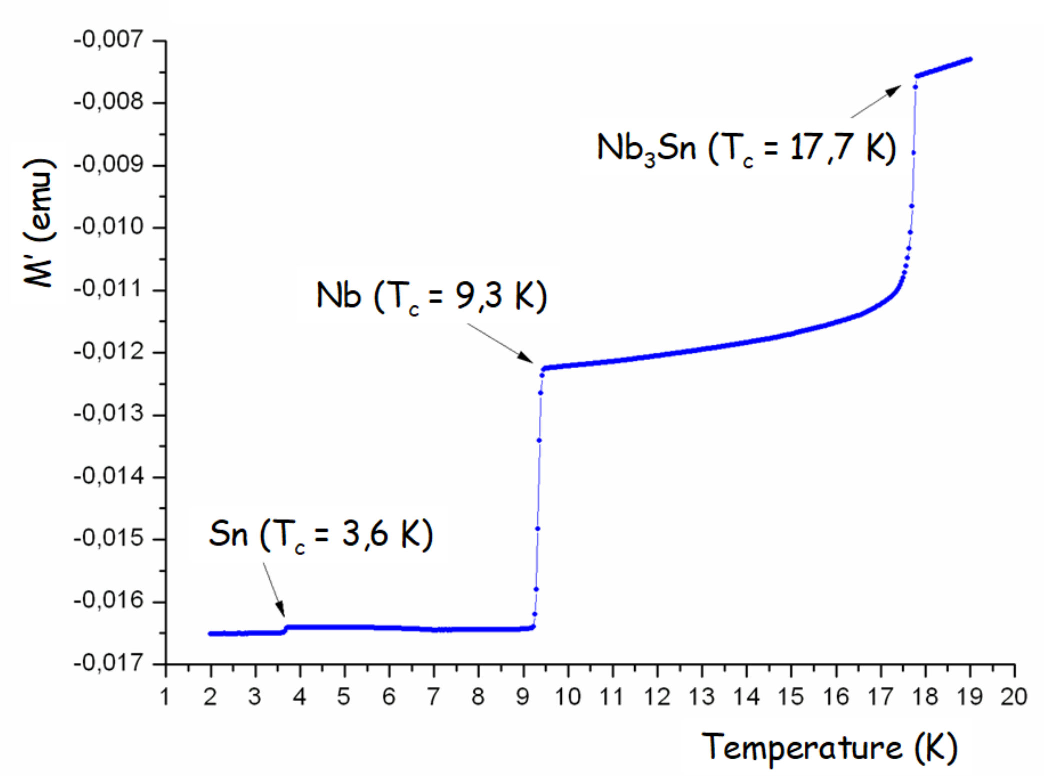


Temperature furnaces profile

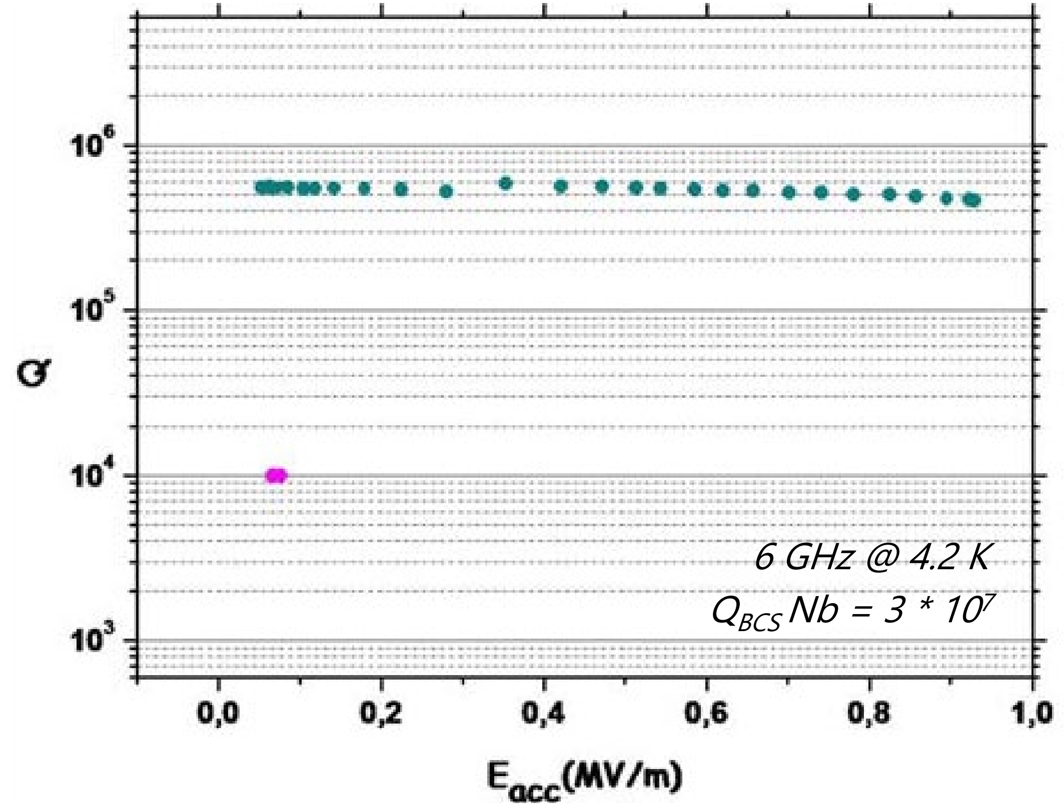


Results (LNL 2006)

S. M. Deambrosis et al., "A15 superconductors: An alternative to niobium for RF cavities," Physica C, 2006
S. M. Deambrosis, "SRF2009



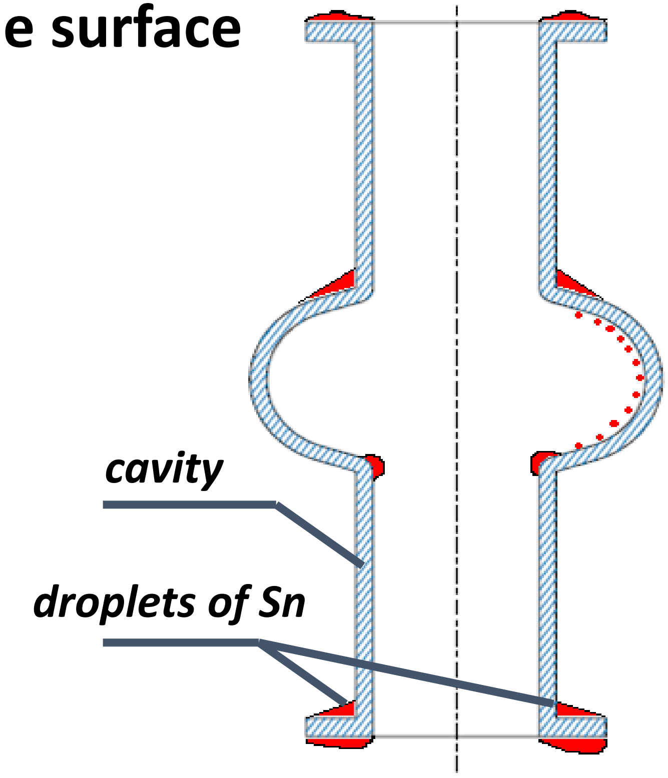
Good performance on planar samples

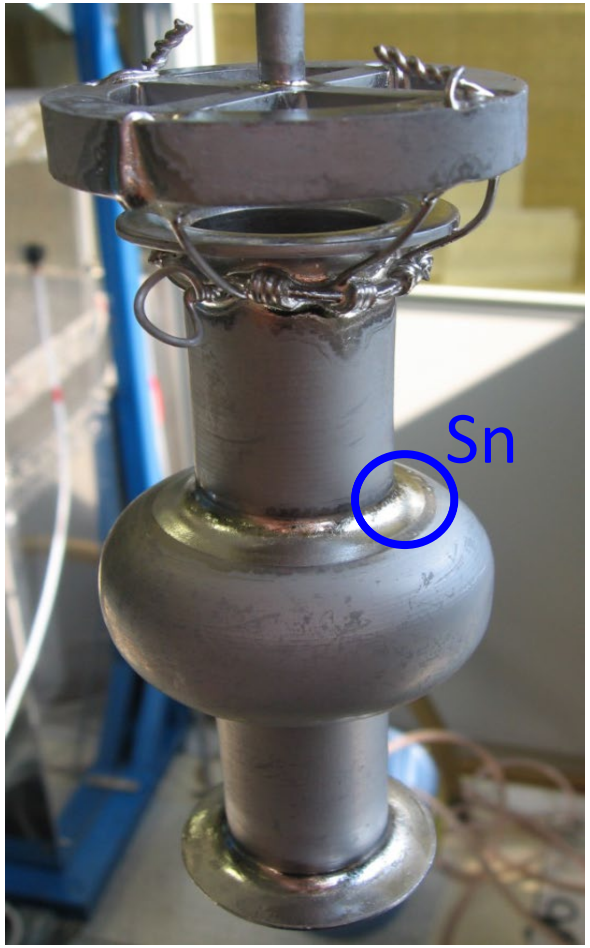


Poor performance on 6 GHz cavity

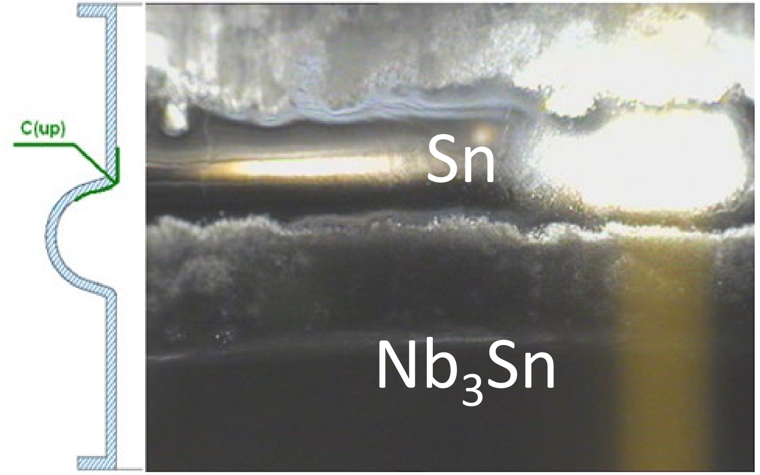
Results (LNL 2006)

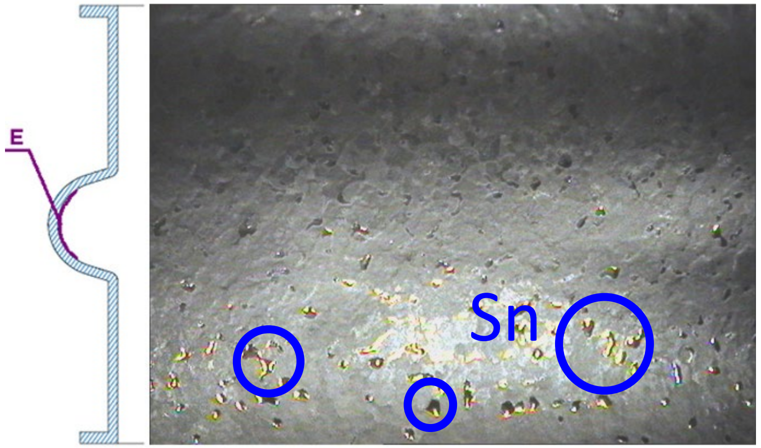
Drops of
unreacted Tin
on the surface





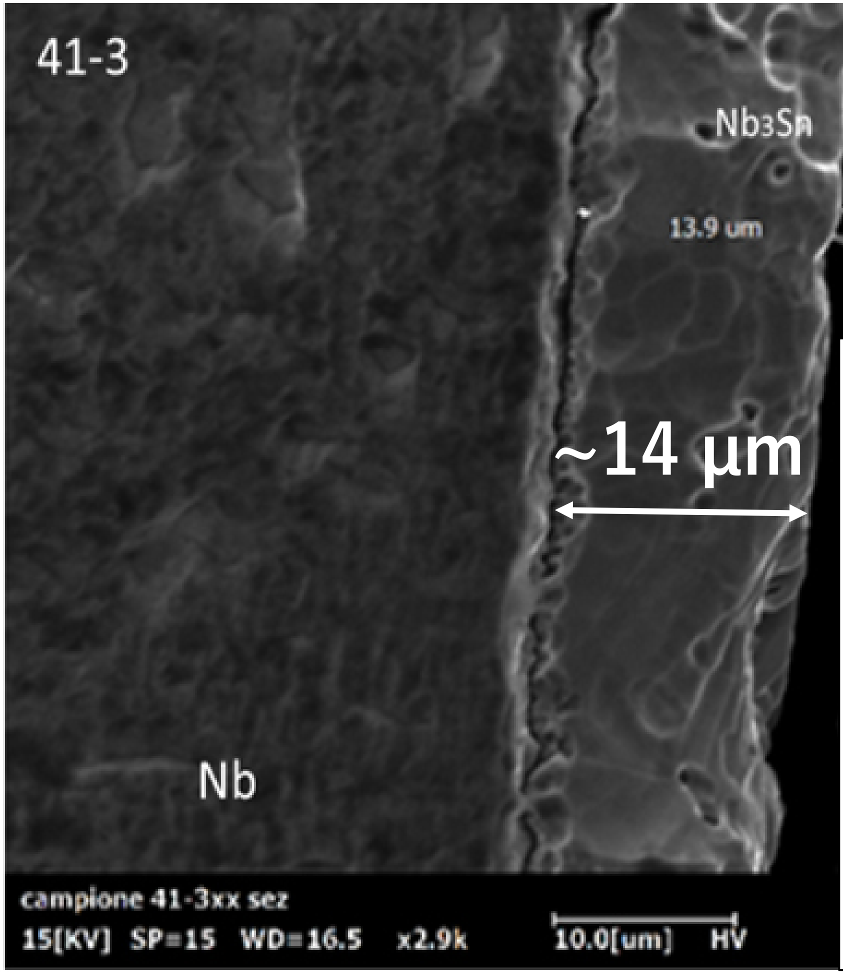
External surface

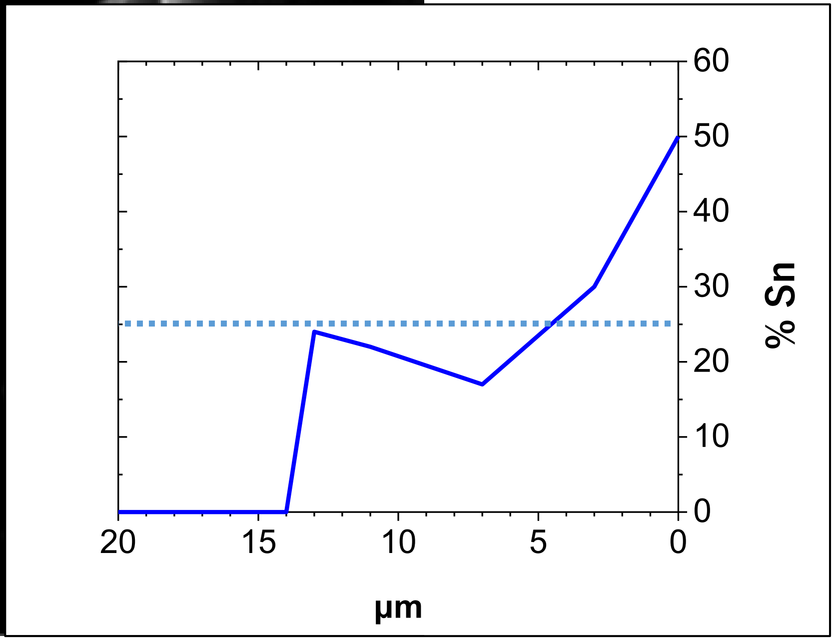




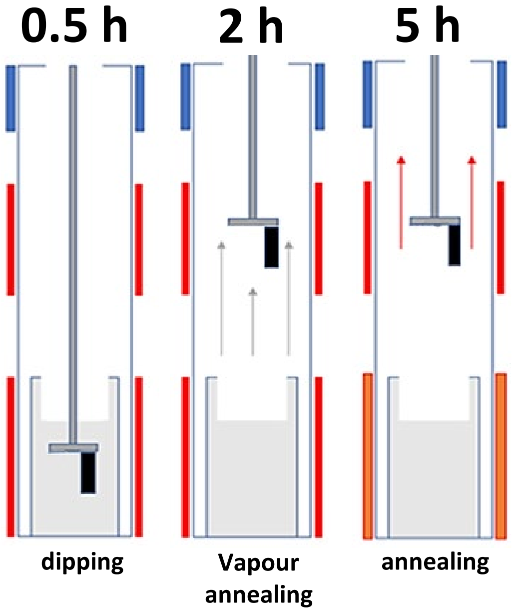
Internal surface

Coating thickness (old process, 2006)

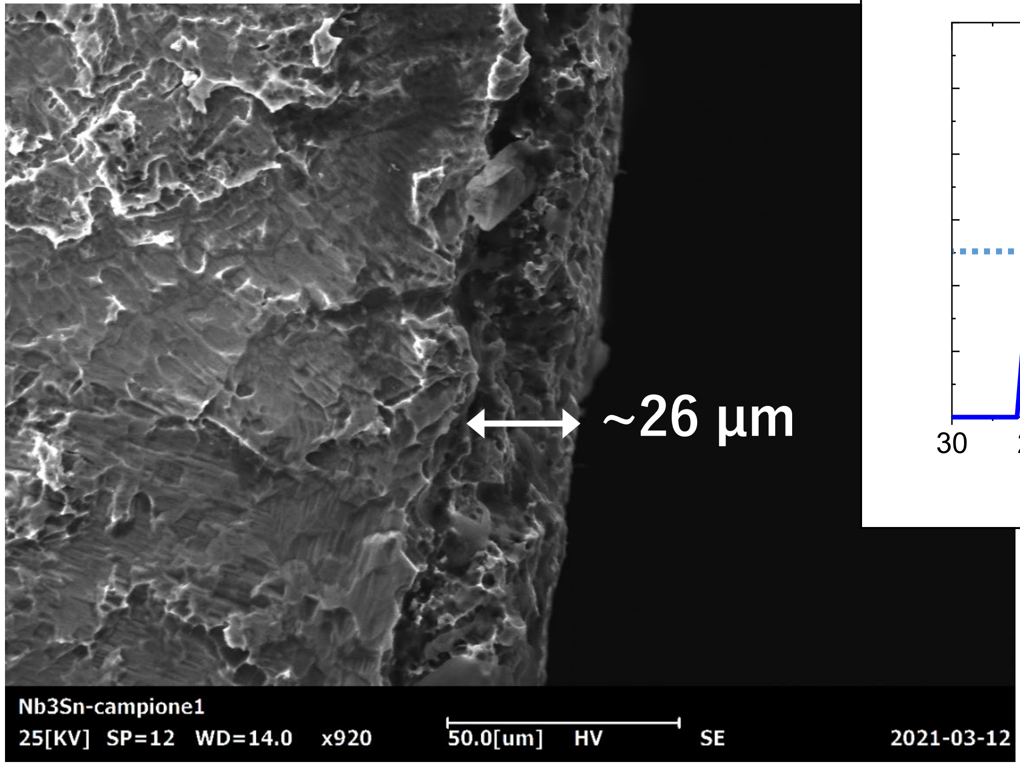


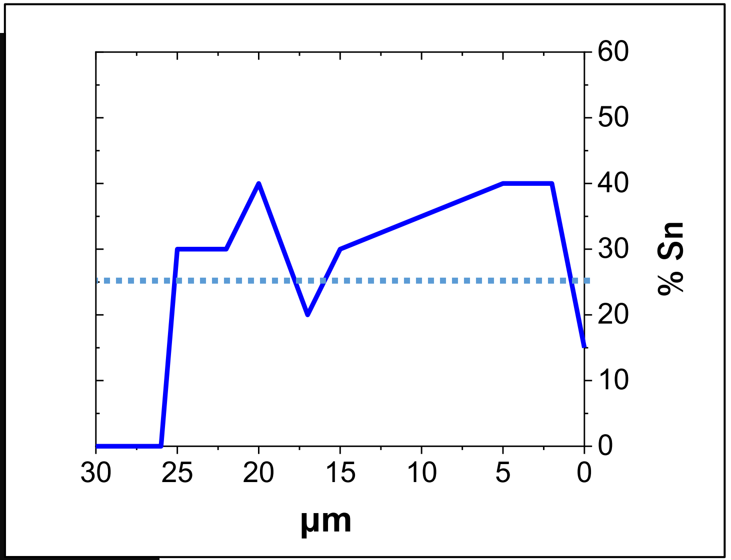


Sn concentration profile by EDS

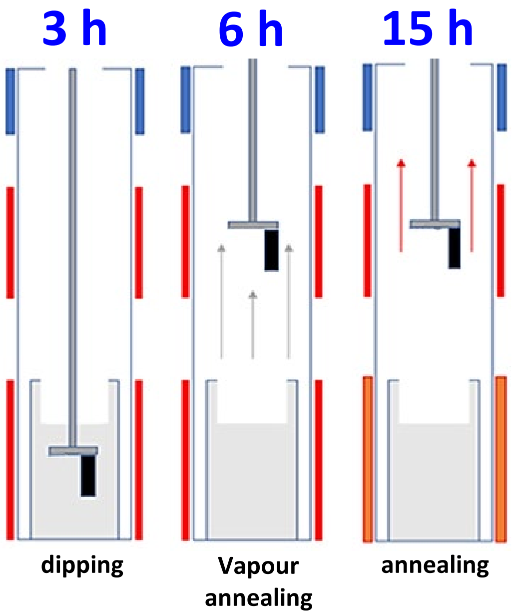


Increasing coating thickness (old process, 2021)

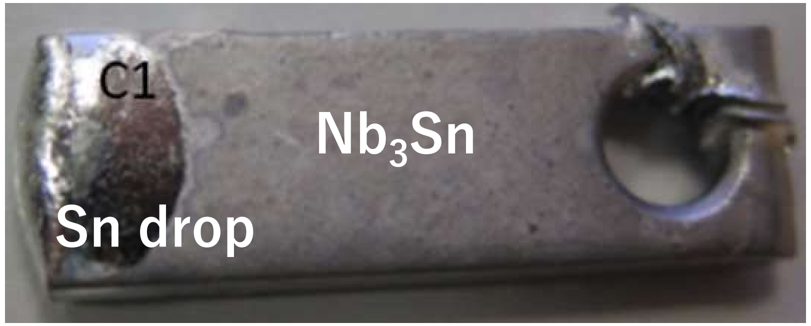




Sn concentration profile by EDS

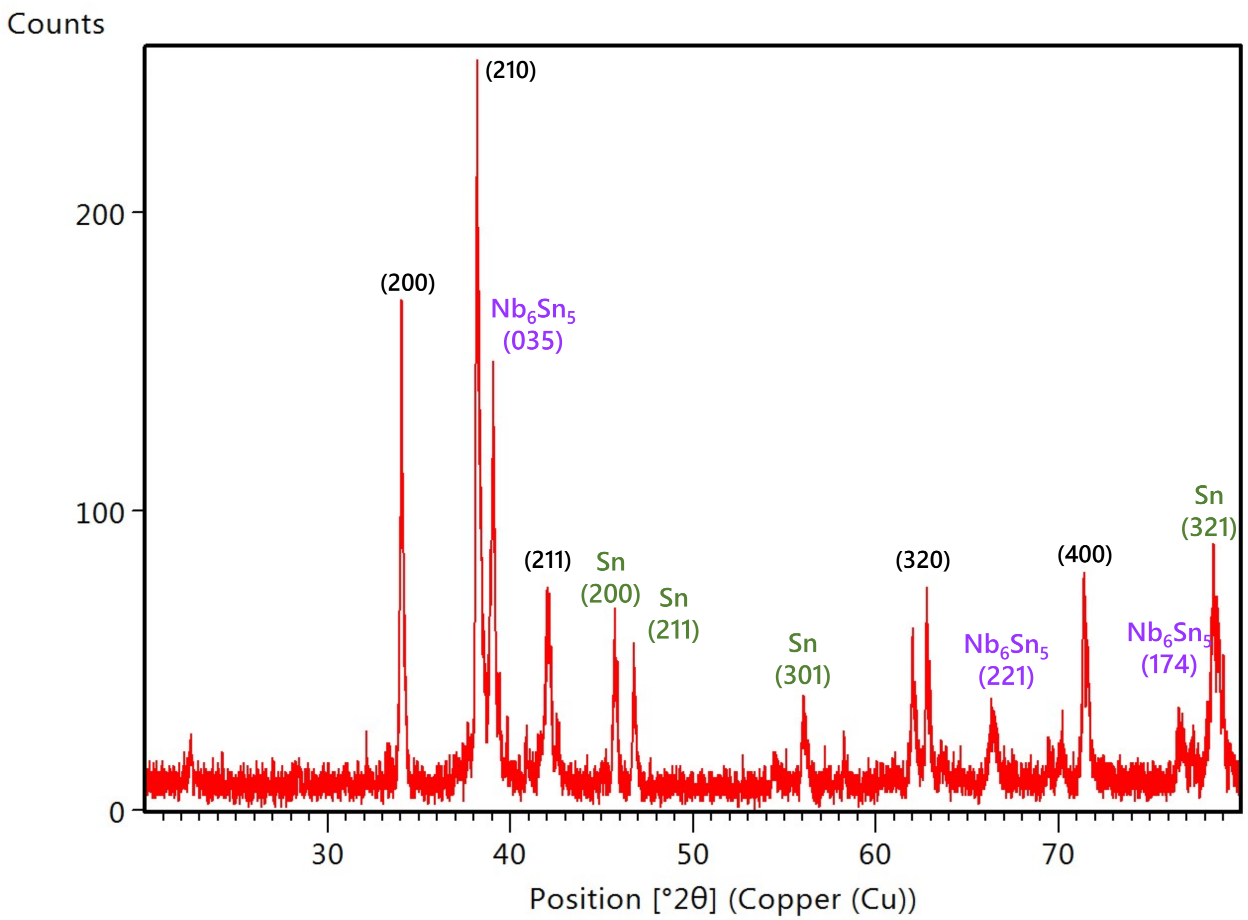


Process time increased



26 µm thick!!! → Thickness can be modulated

XRD (old process, 2021)



Presence of **unreacted Sn** and **Nb_6Sn_5** spurious phase

Morphology (old process, 2021)

Same process, different substrate

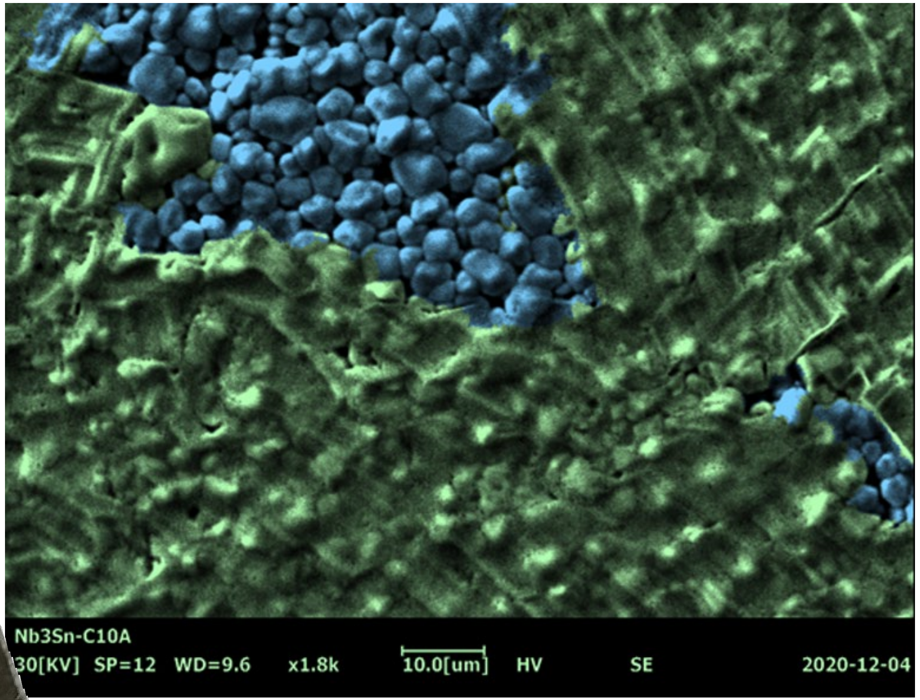
Nb substrate (BCP)

Nb_2O_5 substrate (Nb BCP anodized)

Morphology (old process, 2021)

Same process, different substrate

Sn 64%   Nb 36%
Sn 26%   Nb 74%

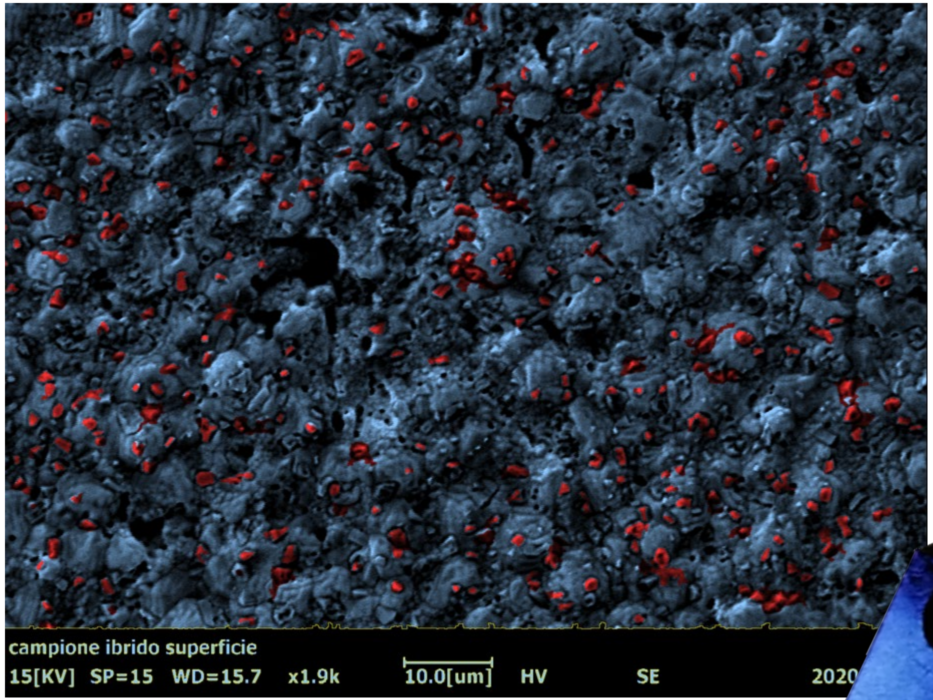


Nb substrate (BCP)

False color SEM

Nb_3Sn
Sn
Nb_6Sn_5

Sn 37%   Nb 63%
Sn 23%   Nb 77%

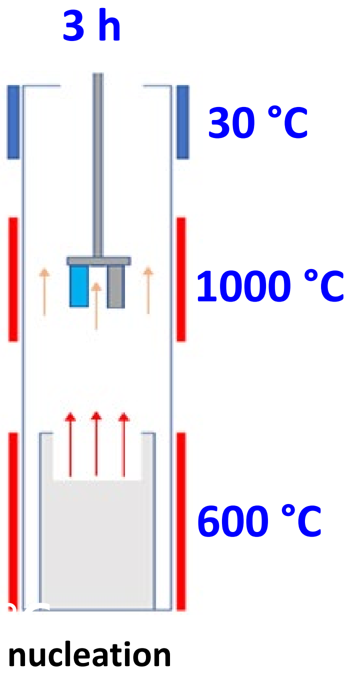


Nb_2O_5 substrate (Nb BCP anodized)

Nucleation is important!

(Already known in Vapor Diffusion)

New process

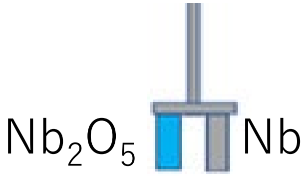


Analogous to Vapor Diffusion:

- Added an **initial Nucleation step** (without SnCl2) @ 600 °C for 3 h

NOTE:

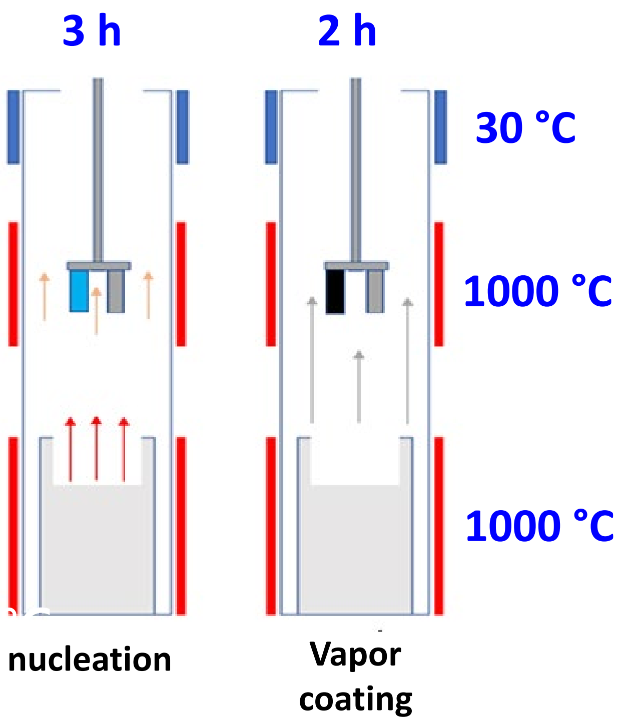
time process steps longer than vapor diffusion due to lower T

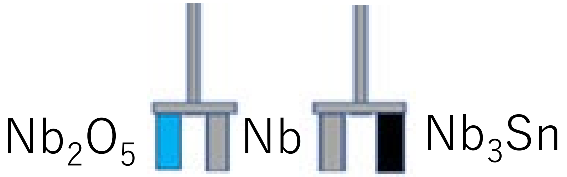


New process

Analogous to Vapor Diffusion:

- Added an **initial Nucleation step** (without SnCl2) @ 600 °C for 3 h
- Added a **Vapor coating step** @ 1000 °C for 2 h





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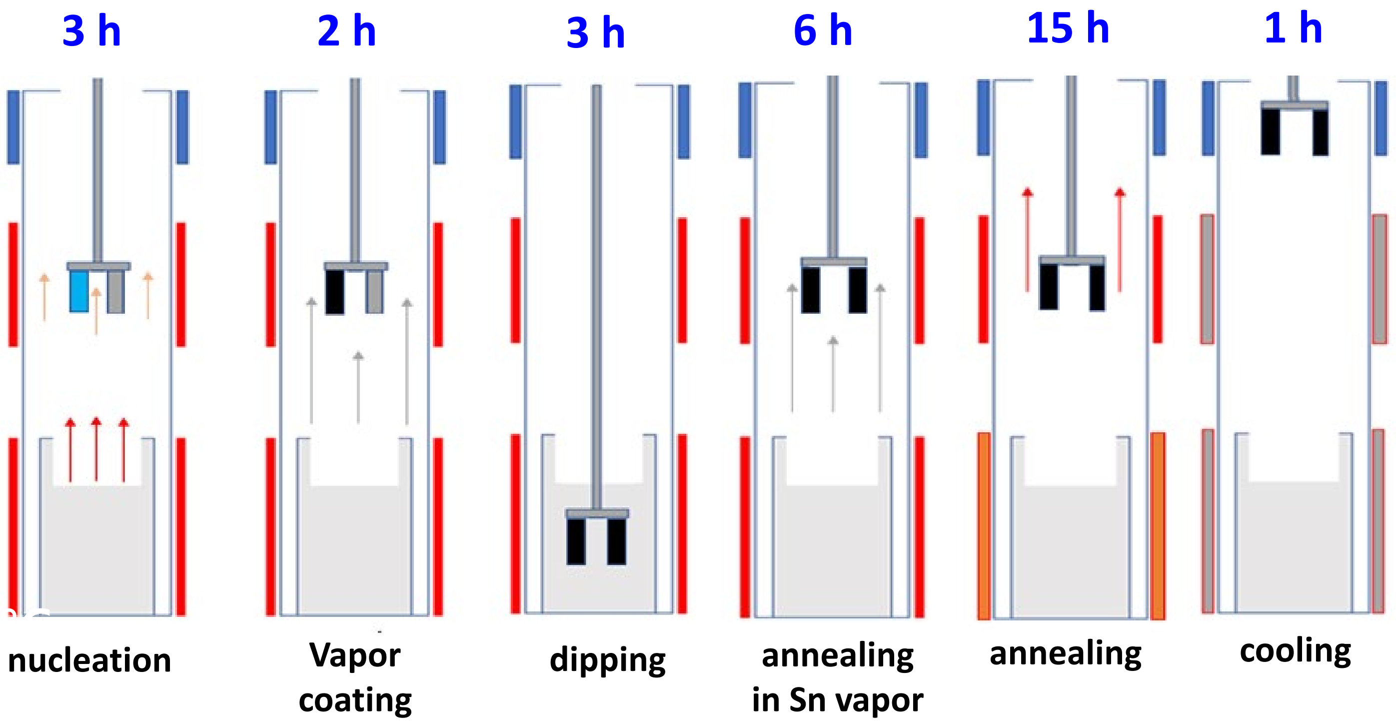
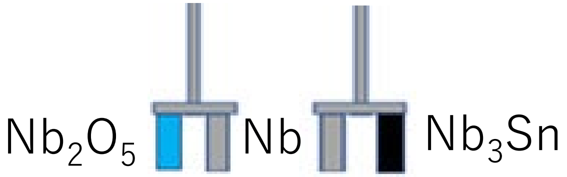




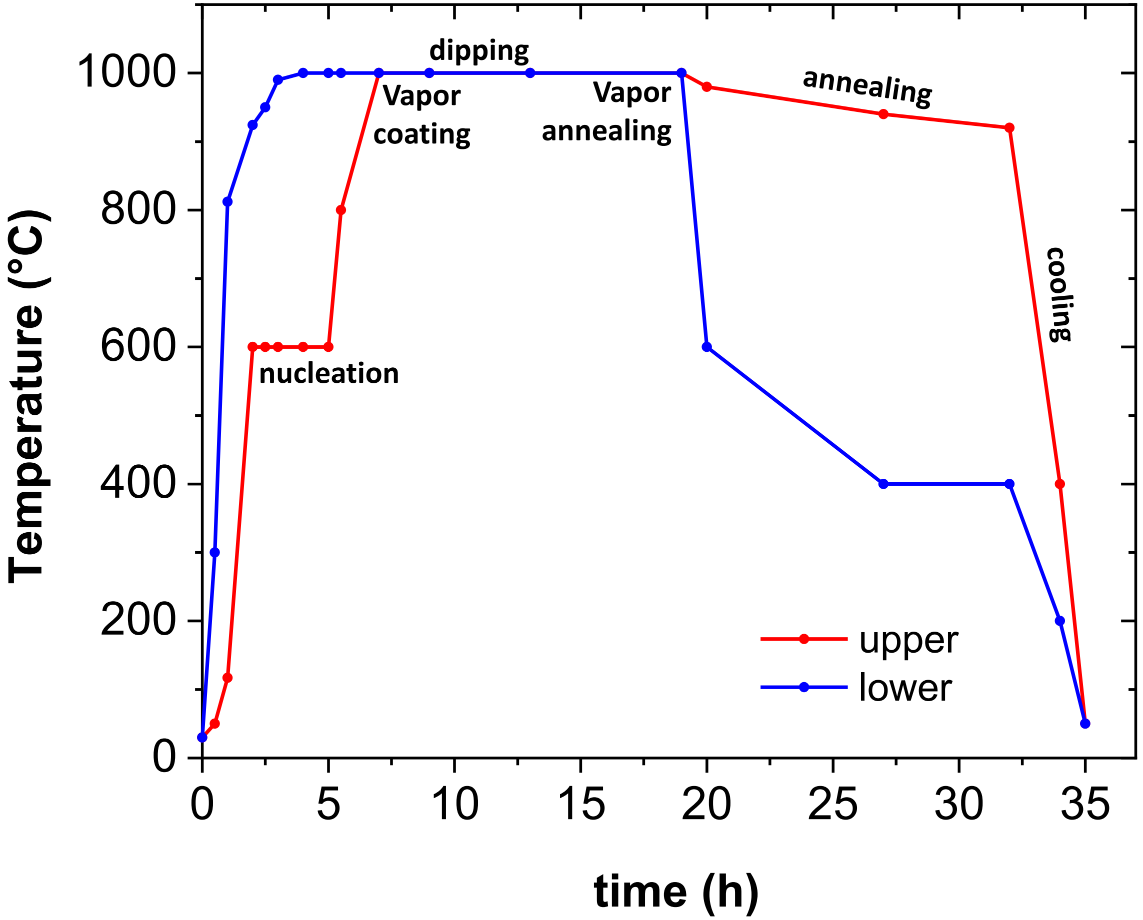
NOTE:

time process steps longer than vapor diffusion due to lower T

New process

Analogous to Vapor Diffusion:

- Added an **initial Nucleation step** (without SnCl2) @ 600 °C for 3 h
- Added a **Vapor coating step** @ 1000 °C for 2 h
- During the **Annealing step**
  Temperature decreased to ~ 920-940 °C



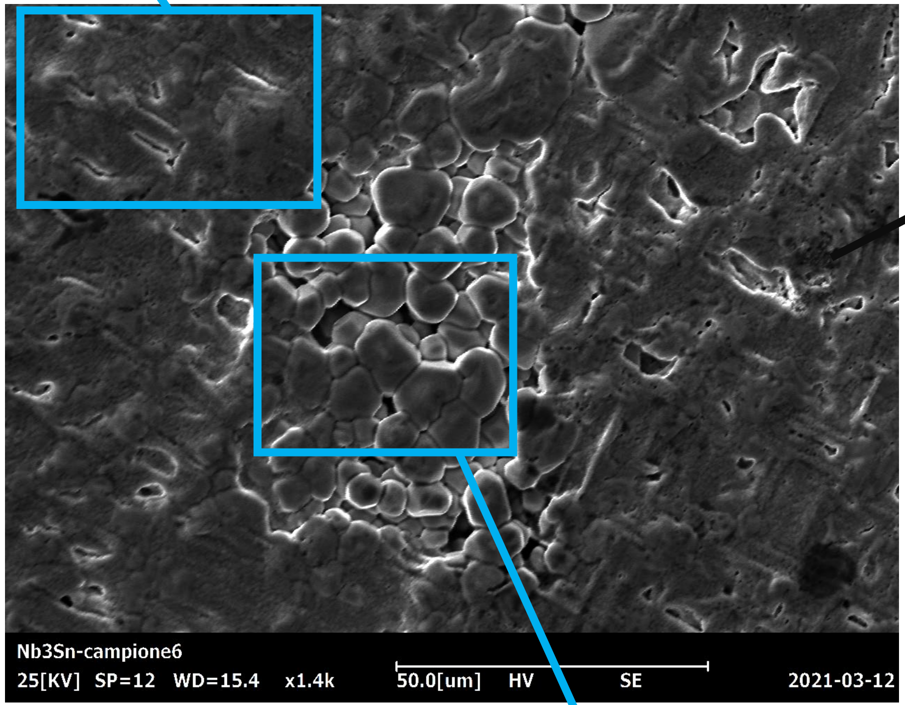
Temperature furnaces profile

NOTE:

time process steps longer than vapor diffusion due to lower T

Morphology (new process, 2021)

Grains + GB
Nb 70 %
Sn 30 %
Cr 0 %

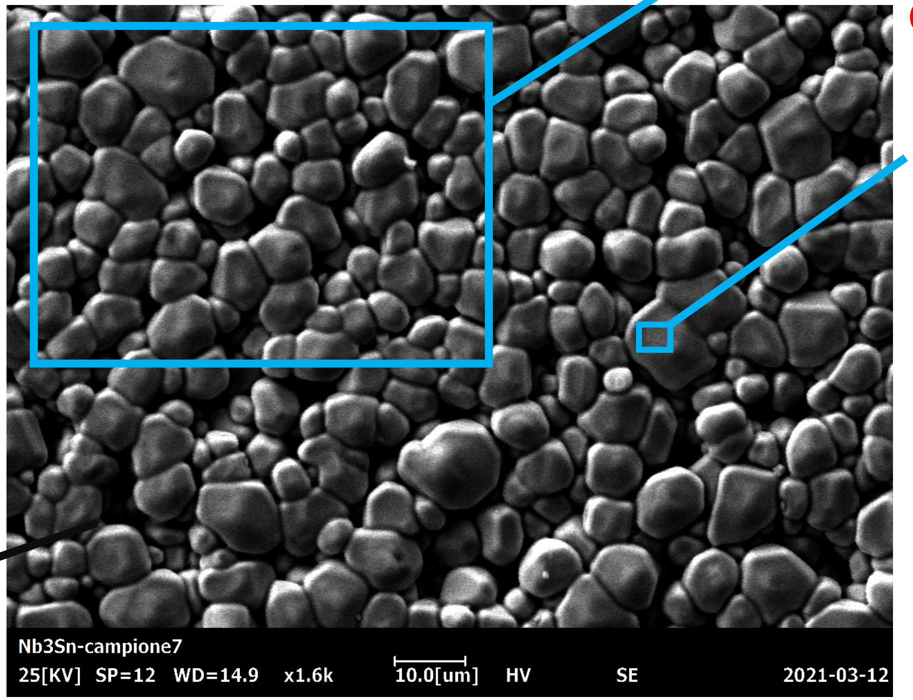


Nb substrate (BCP)

Grains + GB
Nb 50 %
Sn 29 %
Cr 20 %

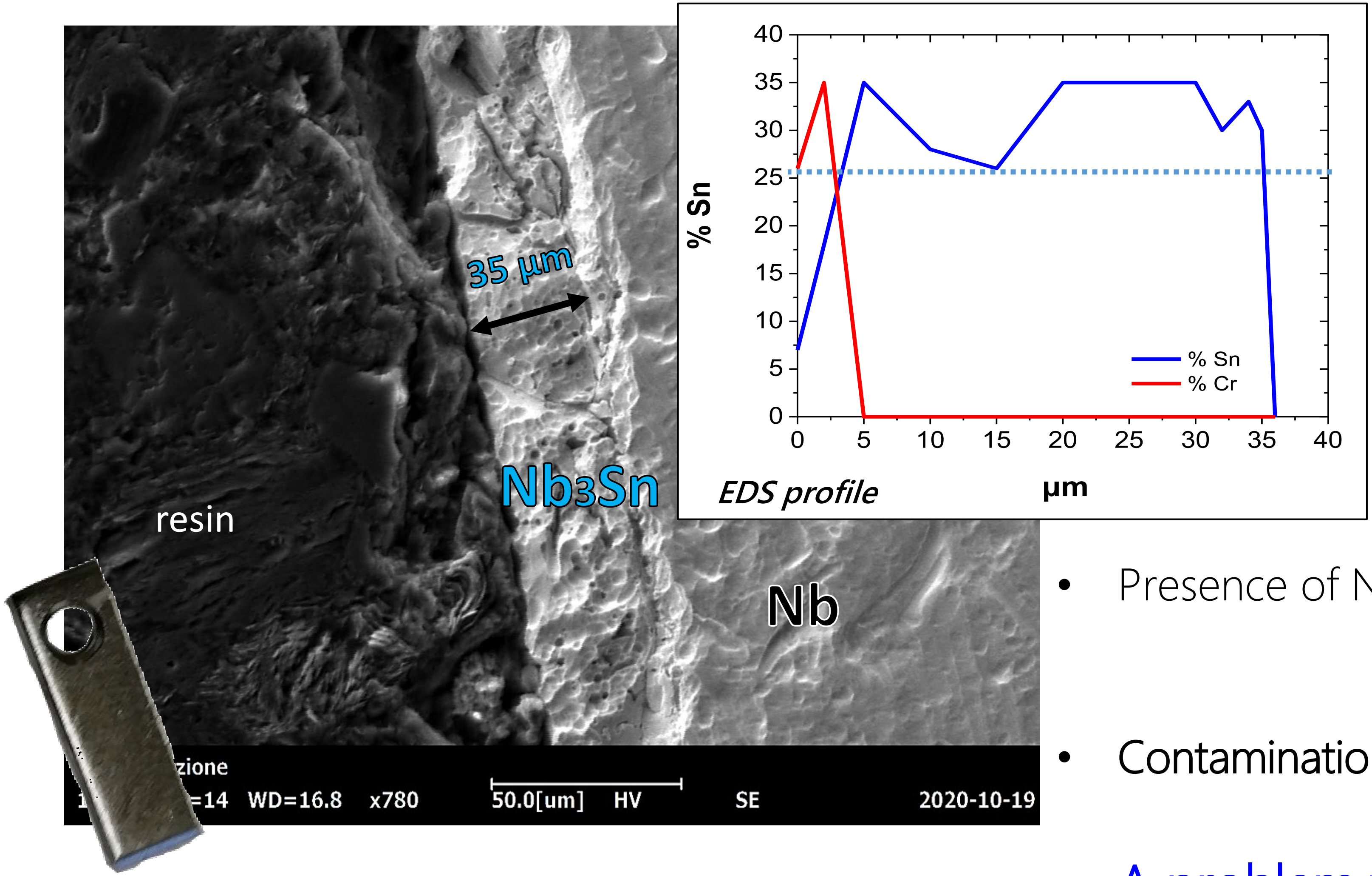
Grains + GB
Nb 58 %
Sn 30 %
Cr 10 %

Grain
Nb 74-76 %
Sn 24-26 %



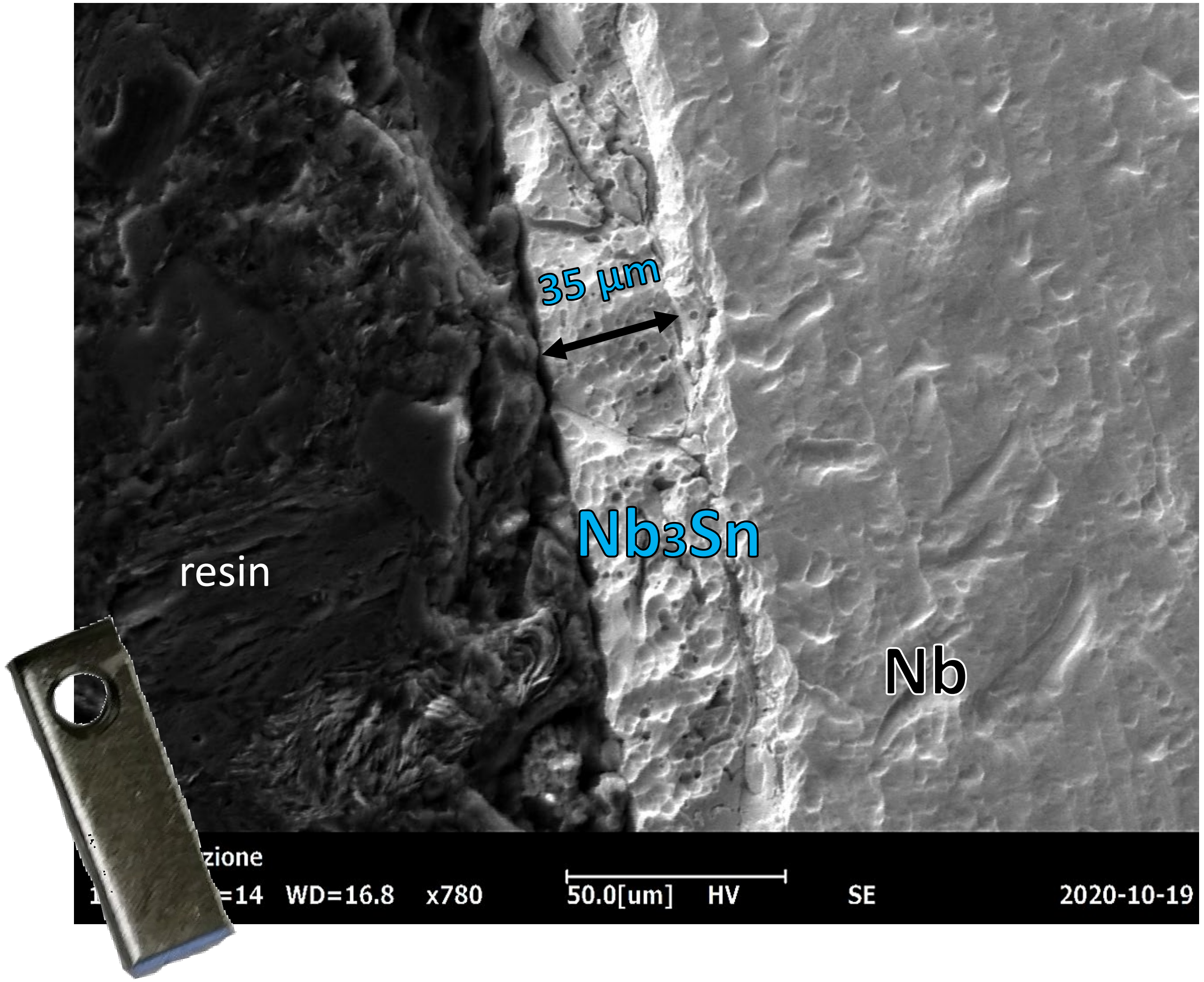
Nb_2O_5 substrate (Nb BCP anodized)

Chromium contamination



- Contamination is superficial
- No contamination in Sn crucible
- Presence of Ni and Fe as well ➡ Inconel Alloy of vacuum chamber
- Contamination present also in old samples
- A problem to face and solve

Dipping time - thickness relationship



7 hours of dipping → 35 µm

Dipping time - thickness relationship

We suppose that the **Nb_3Sn** **growing rate** by **dipping** is **related to the Sn diffusion in Nb**

If it is true, from the Fick's laws of diffusion we have that:

$$x^2 = kt$$

x: Nb_3Sn thickness

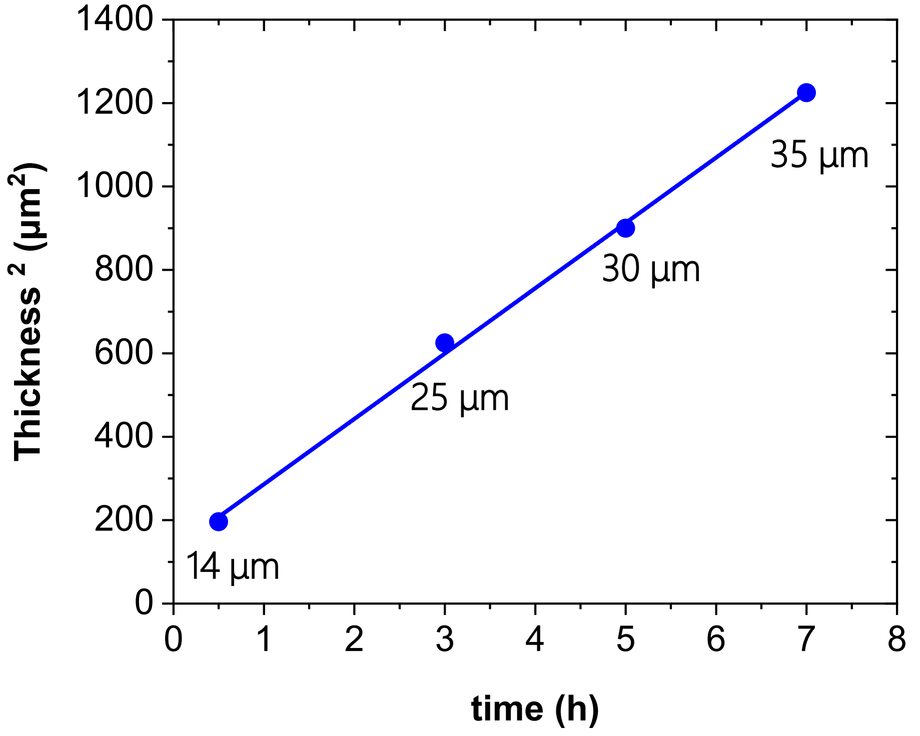
t: dipping time

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

Good agreement with the experimental data

100 µm → ~ 63 hours of dipping!

24 hours of dipping → ~ 62 µm



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

$$R^2 = 0.9983$$

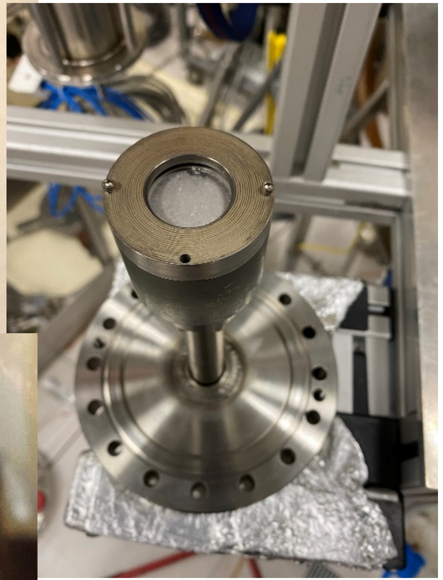
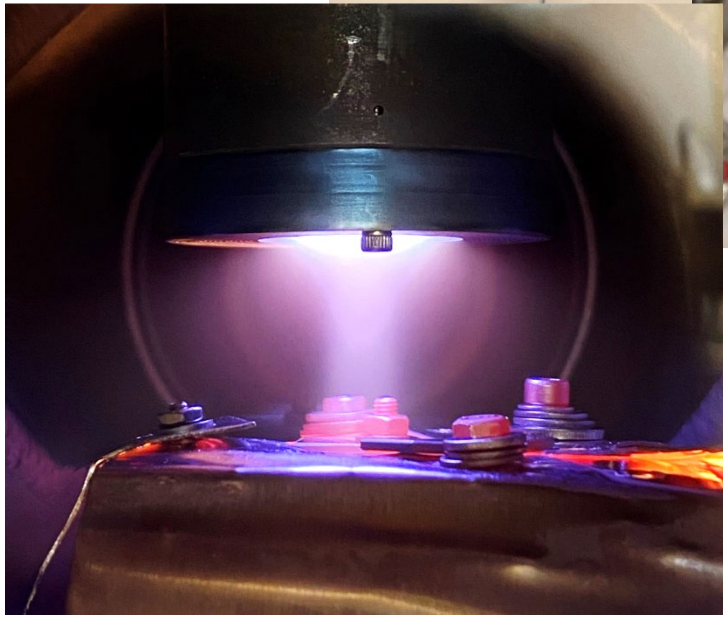
Proof of concept

- Preparation of a 1″ target (30 microns thick)

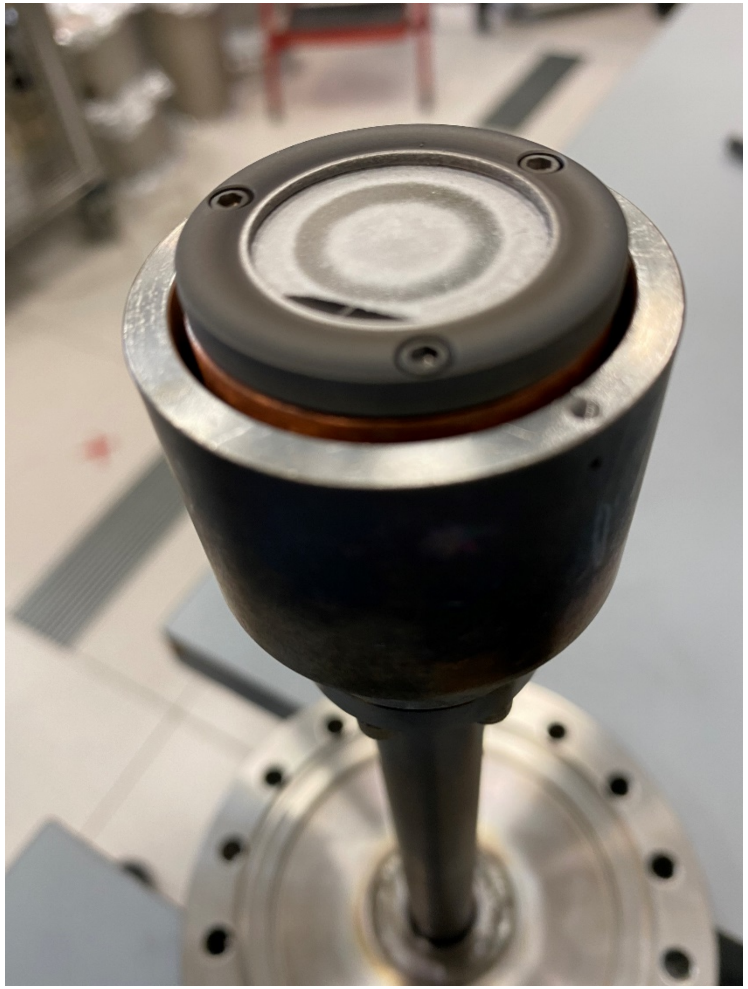
- Coating on quartz samples

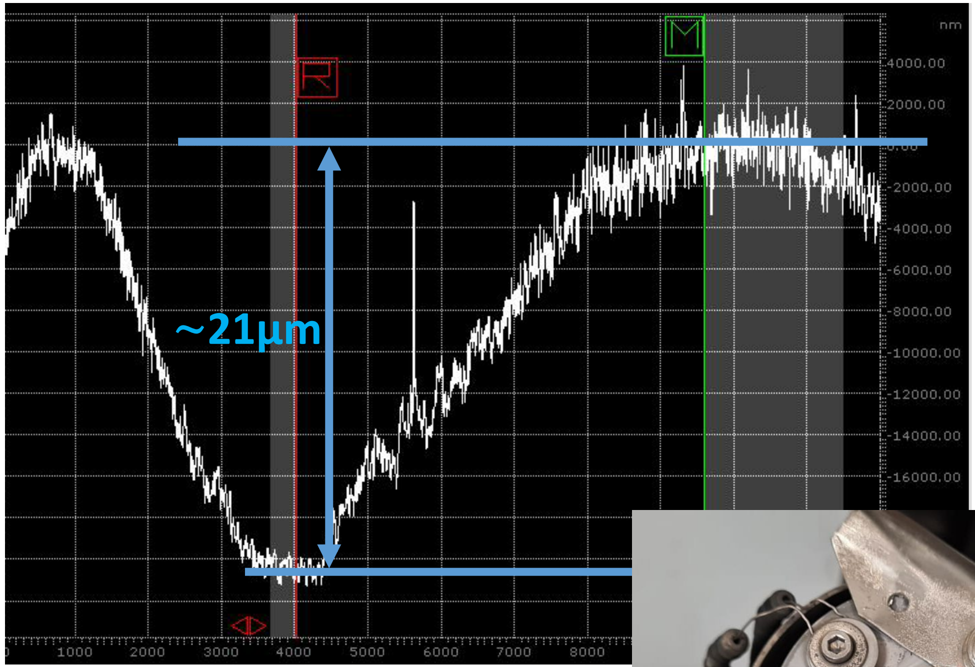
  $I = 0.1$ A (5 mA/cm^2)
  $t = 30$ min
  $T = 750$ °C
  $P_{base} = 2 \cdot 10^5$ mbar

- Process stopped when V decreased

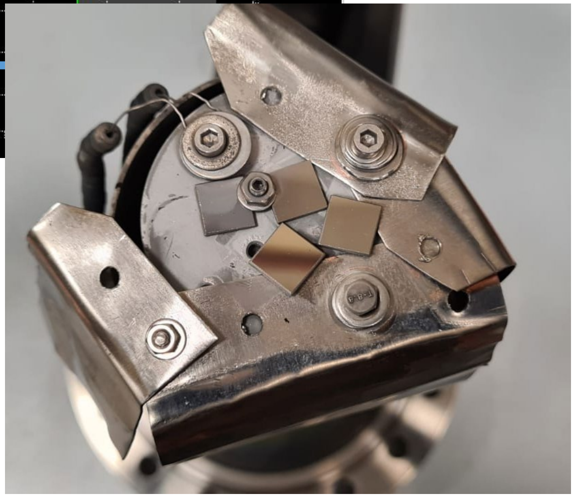




Results - Target erosion



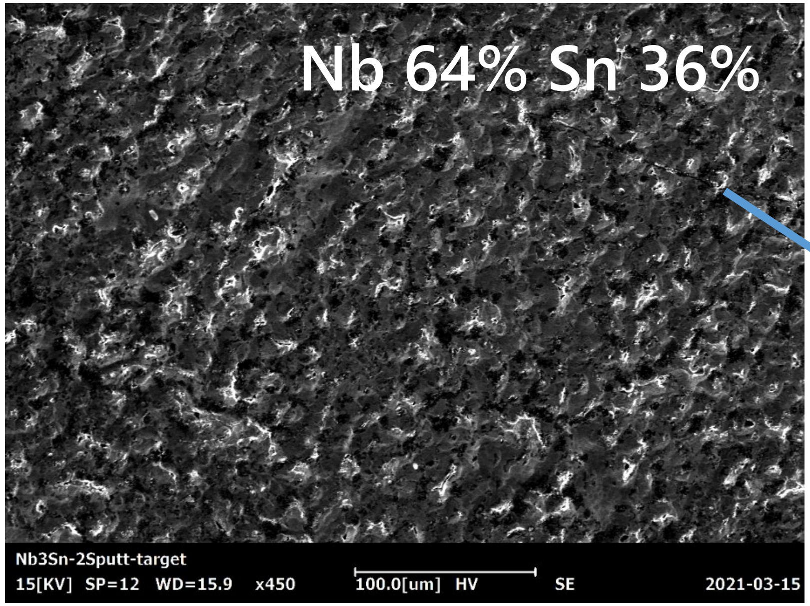


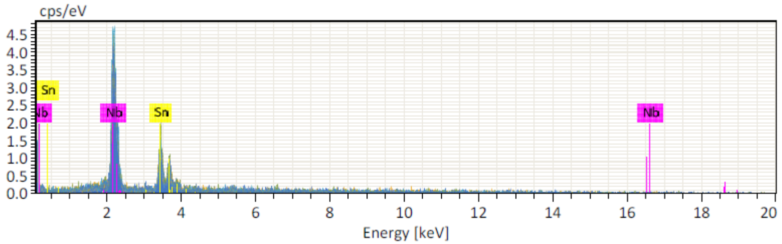
Target erosion profile by profilometer

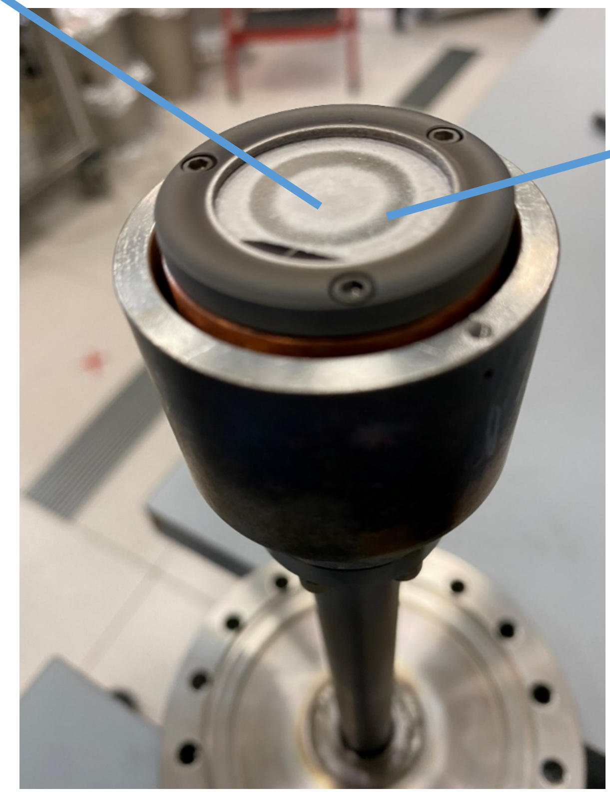


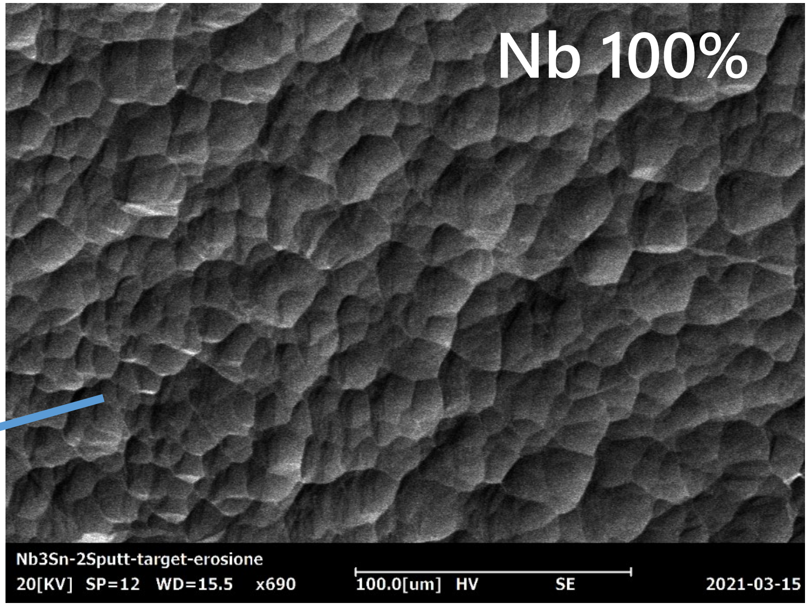
Coating thickness on samples: 1.2 µm

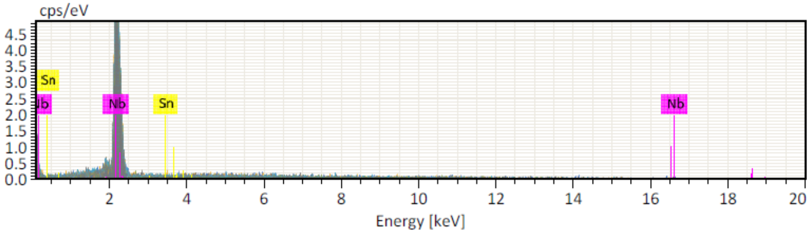
Results target



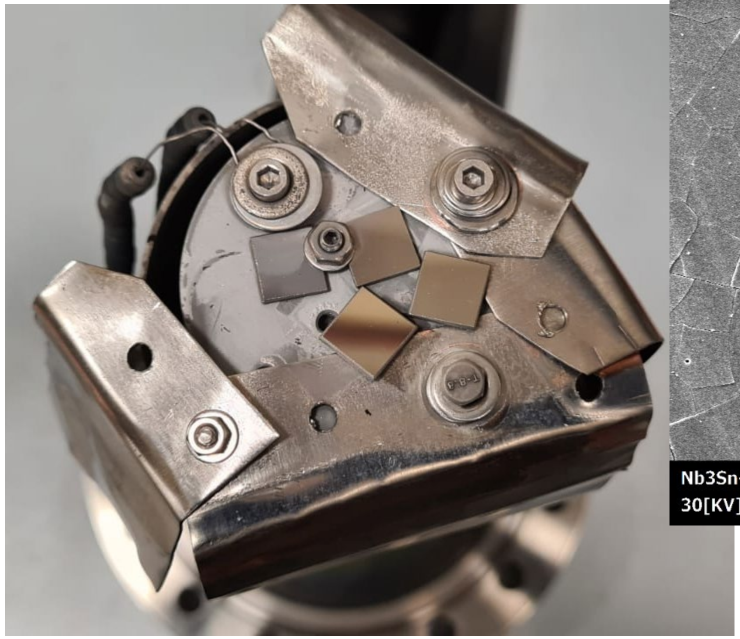


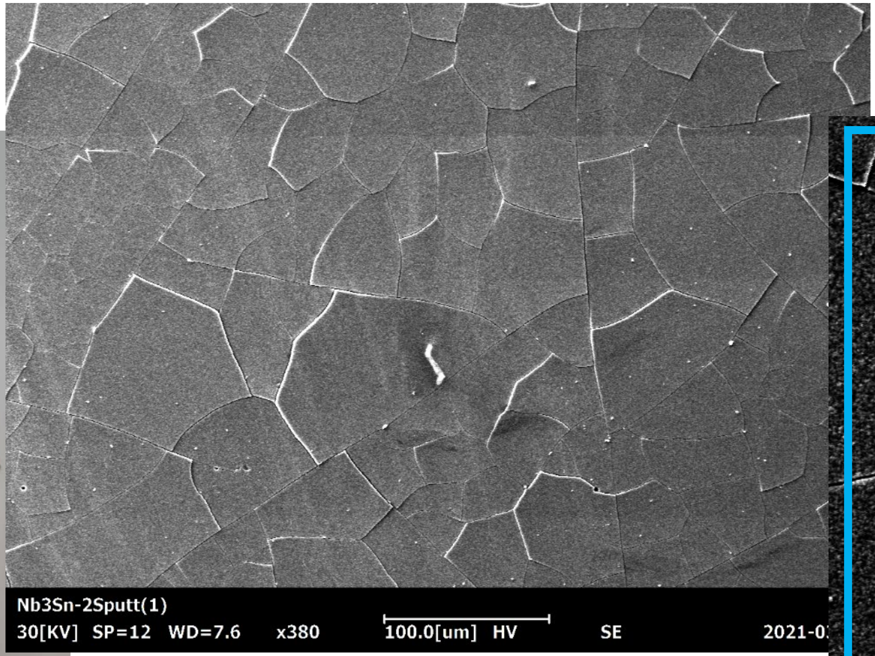






Sputtered samples





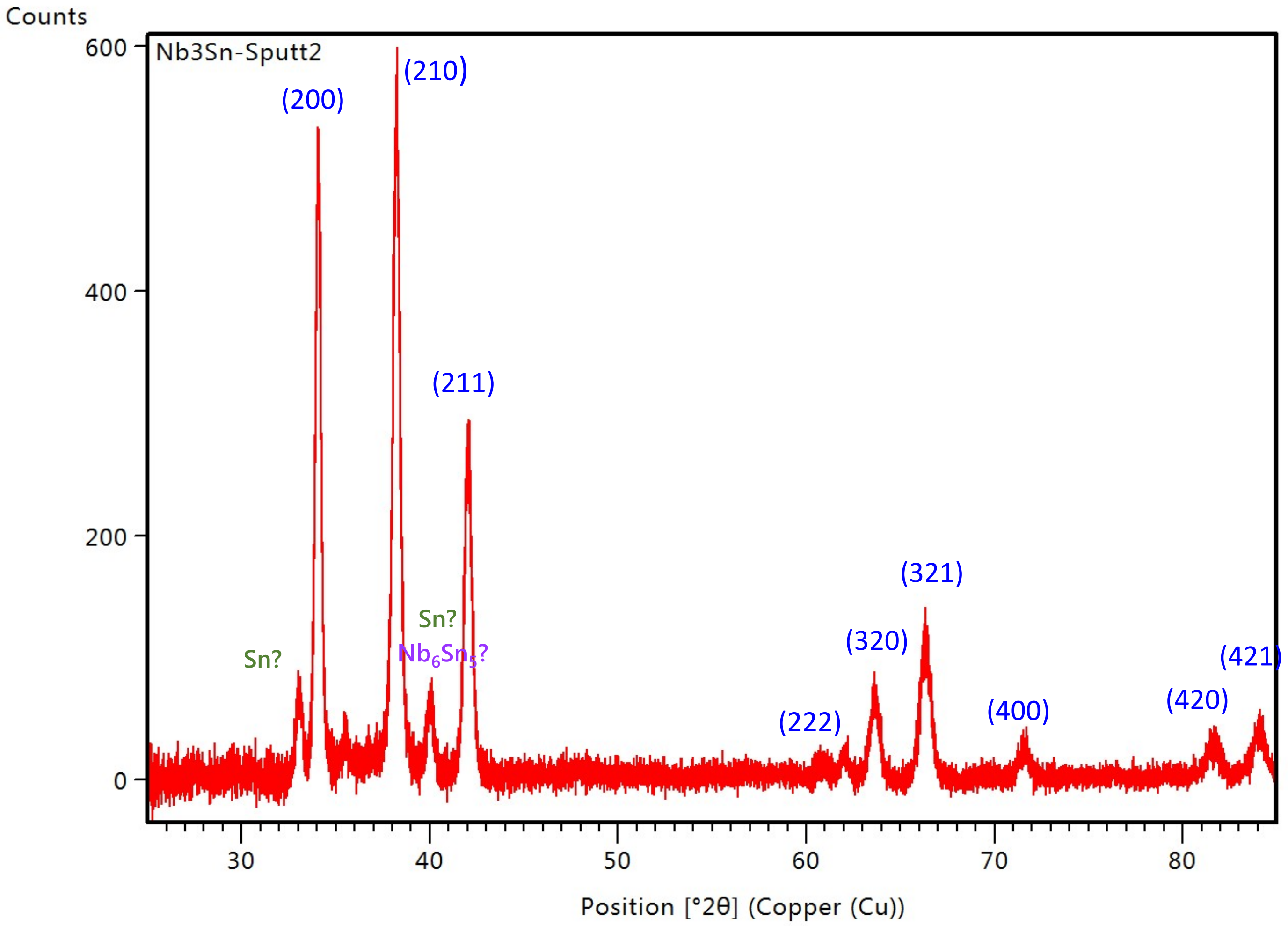
Nb 80% Sn 20%

Nb 74% Sn 26%

Strange behaviour R vs T → not possible measure Tc

No optimized deposition conditions

Sputtered sample XRD



XRD shows Nb_3Sn phase

Presence of **unreacted Sn**

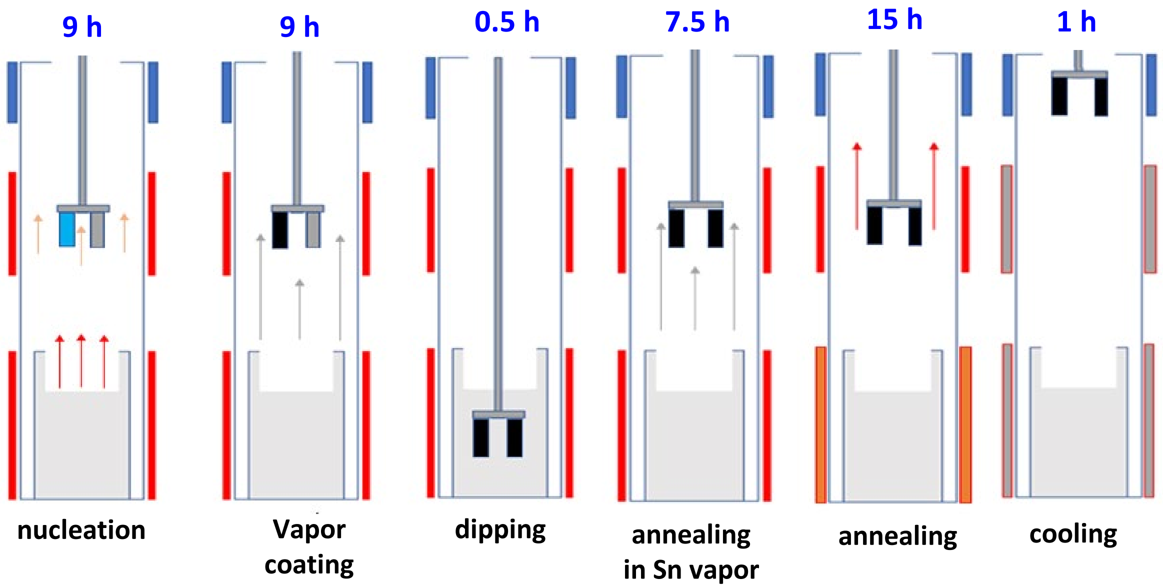
and/or **Nb_6Sn_5** spurious phase

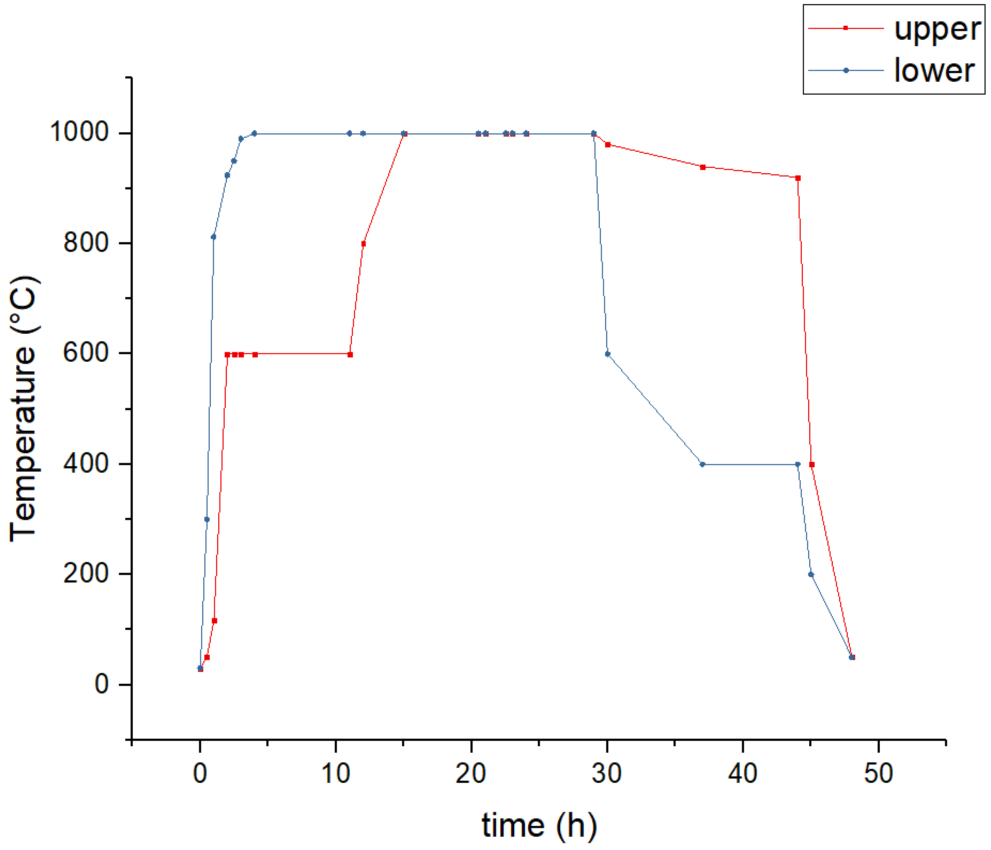
How does the new process work in RF?

How does the new process work in RF?

Process has been adapted to reduce Nb_3Sn thickness

(5-10 microns as the old cavities)





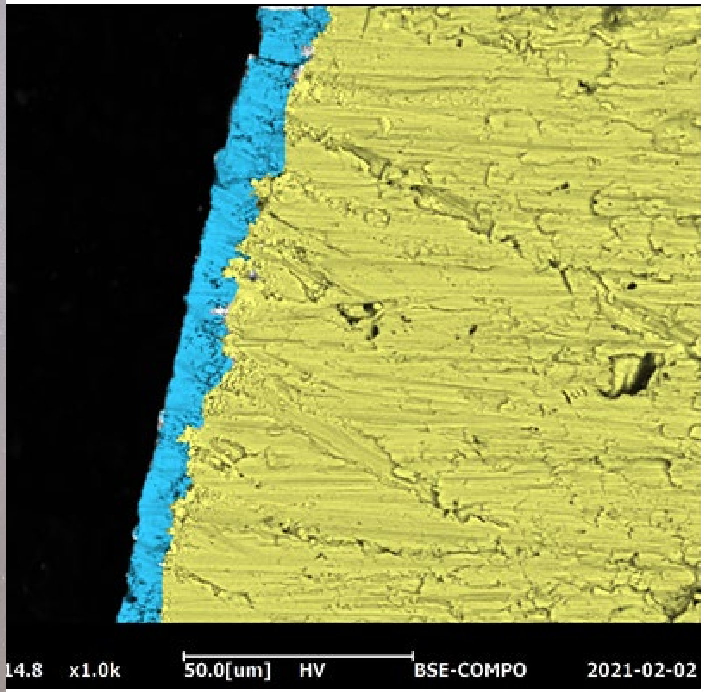
6 GHz cavity #1 (only geometry test)

First test in a broken cavity to test possible geometry effect

No tin drops!

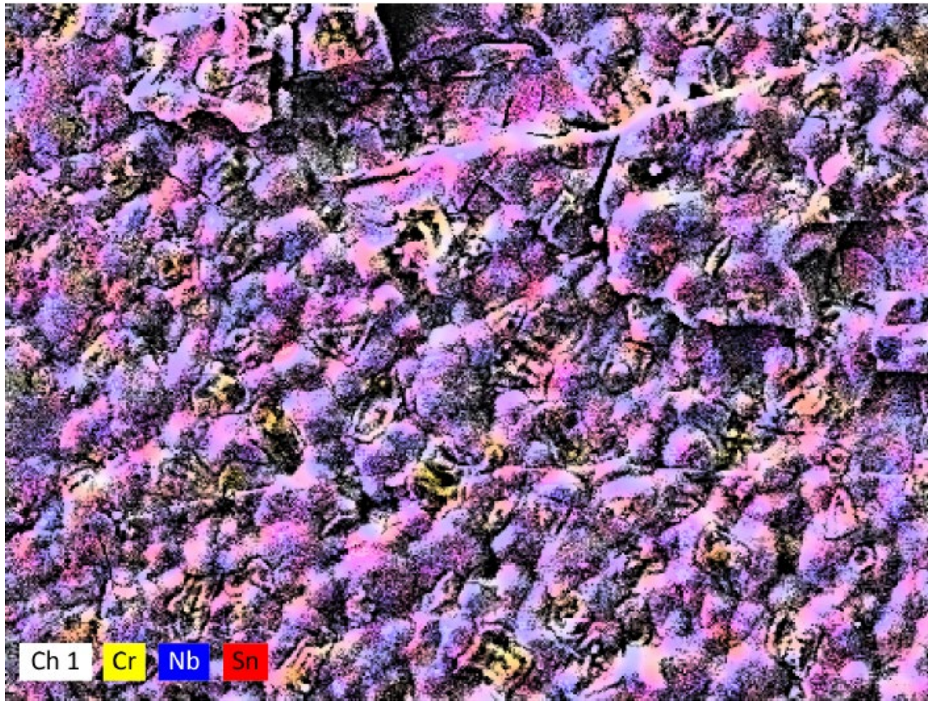
Very good surface

5-10 micron Nb_3Sn thickness



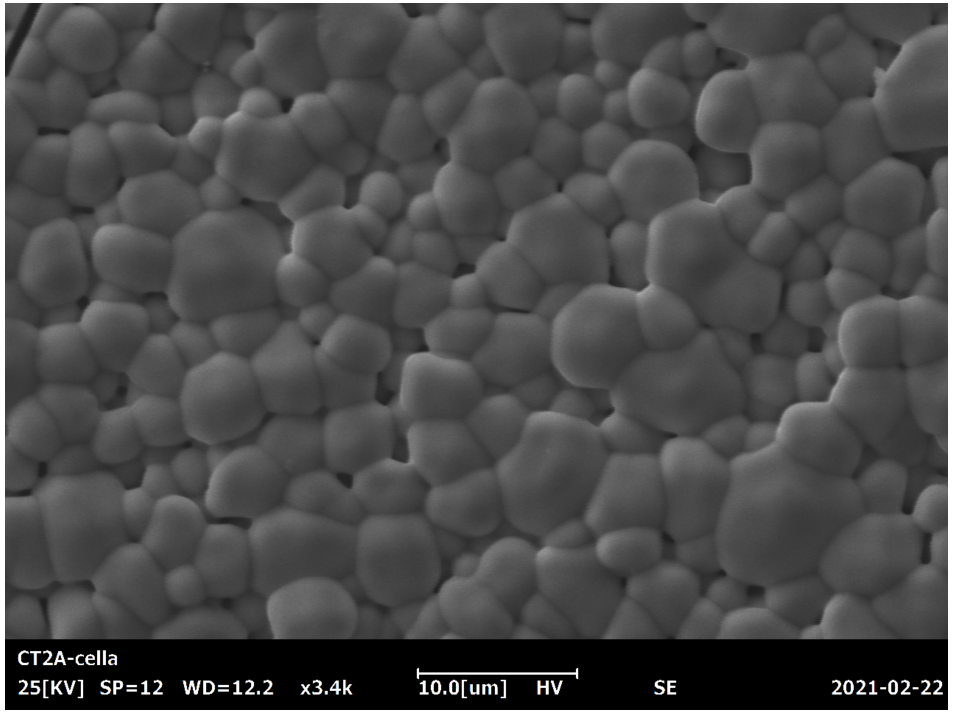
Cr-Ni contamination in 6 GHz cavity

External Surface of a 6 GHz elliptical cavity



Nb 42% Sn 13% Cr 38% Ni 7.2%

Inner Surface of a 6 GHz elliptical cavity



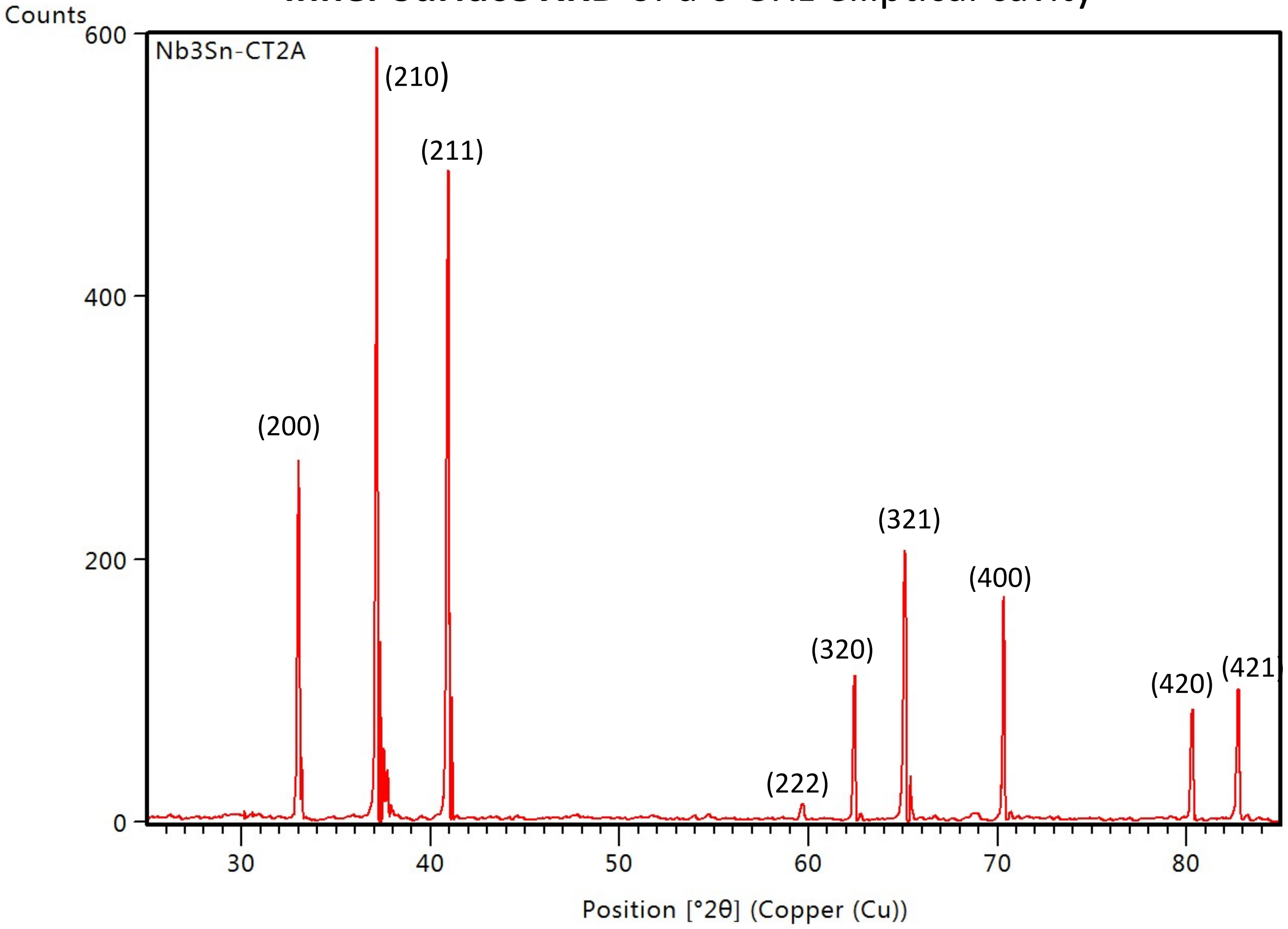
Nb 65% Sn 35% Cr 0%

The cavity **geometry preserves** the **inner surface** from **contamination**

6 GHz cavity #1 (only geometry test)

XRD shows only the **Nb_3Sn phase**

Inner Surface XRD of a 6 GHz elliptical cavity



6 GHz cavity #2

Same process of cavity #1

Problem with upper furnace: **annealing at 920 °C instead of 940 °C**

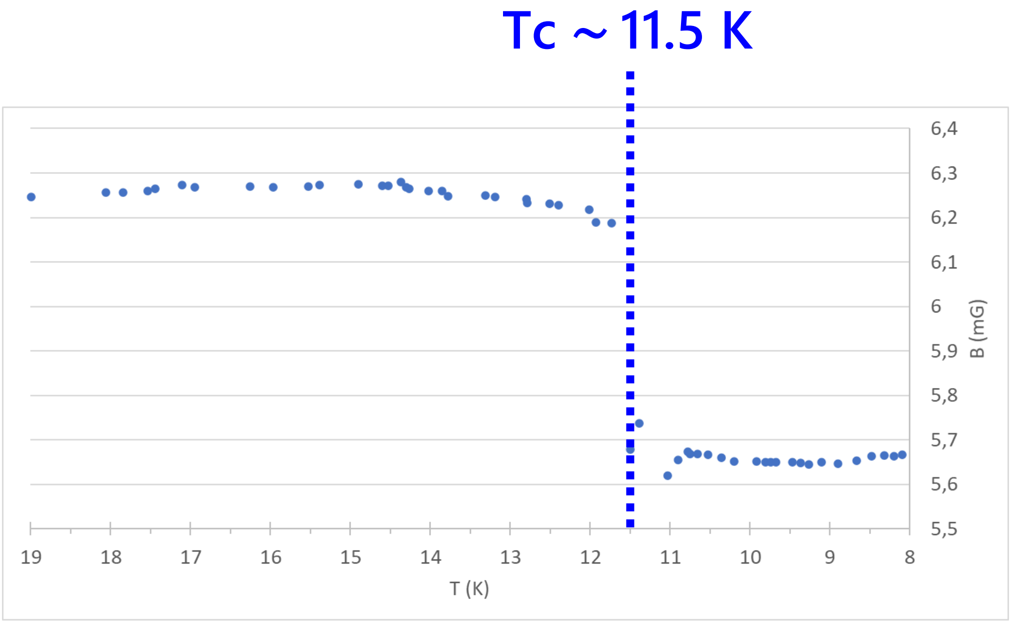
45 h of annealing instead of 15 h

Metallic tin outside

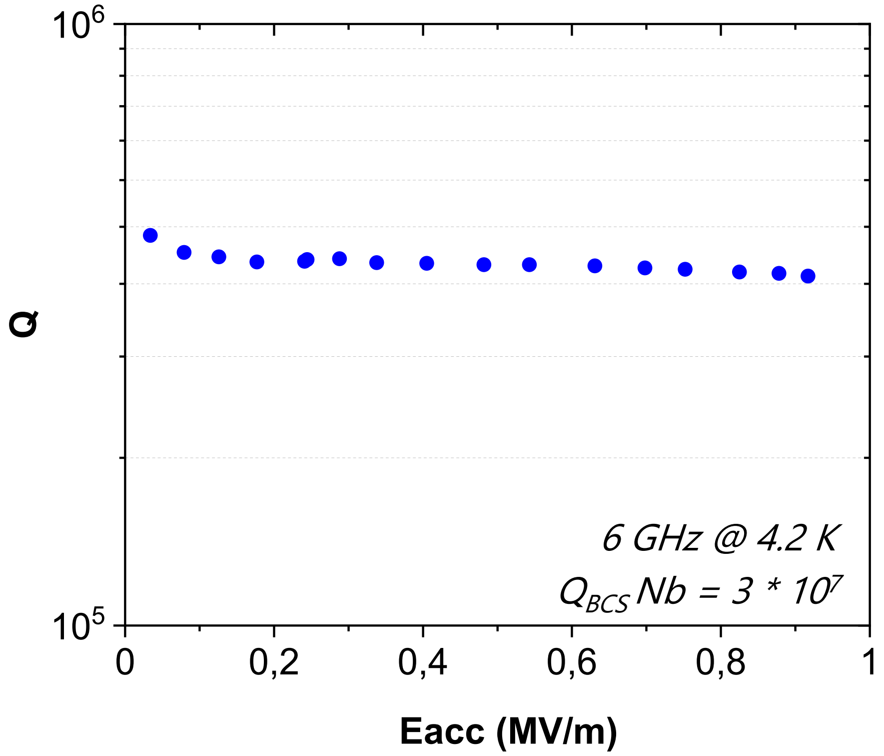
Dark spots in the inner surface

(signs of tin excess)

Tc and RF Test



Flux expulsion measured by a Flux Gate placed in the equator



Poor RF performances comparable to old process

(Sn excess? Cr-Ni contamination? Low T? Thick film?)

Conclusions

- Possibility to grow thick film → target production
- Good Stechiometry
- Proof of concept passed *(sputtering process must be optimized)*
- Cr-Ni contamination to solve *(Nb screen or New Nb chamber)*
- Poor SRF performance
  (Sn excess? Cr-Ni contamination? Low T? Thick film?)

Thank you for the attention!

Material Science and Technology Service for Nuclear Physics crew

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