Cristian Pira
On behalf of INFN LNL SRF group

INFN LNL

iFAST

SAMARA
CSN5 INFN experiment

i.FAST has received funding from the European Union's Horizon 2020 programme under GA No 101004730.

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

TESLA TECHNOLOGY TTC COLLABORATION

Aomori
October 11-14, 2022

Outline

- **Project goal** and motivations
- Liquid Tin Diffusion **technique introduction**
- Target production **first results**
- First **sputtering test** with LTD target

Goal and motivations

Goal

Produce and test a Nb_3Sn on Cu 6 GHz elliptical cavity

Nb on Cu 6 GHz cavity

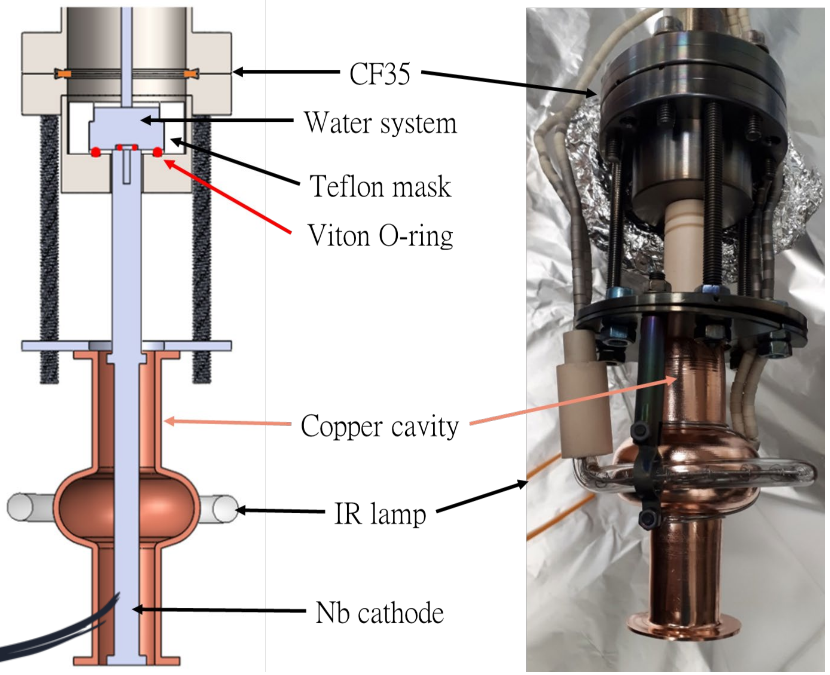
Motivations:

- **Cu** is a **cheaper** substrate
- Higher **thermal conductivity**
  (important also for applications beyond high energy physics with cryocooler cooling)
- **6 GHz** are a good test bench
  (handable, cheap, fast to test, test facility ready @LNL)

LNL Strategy

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

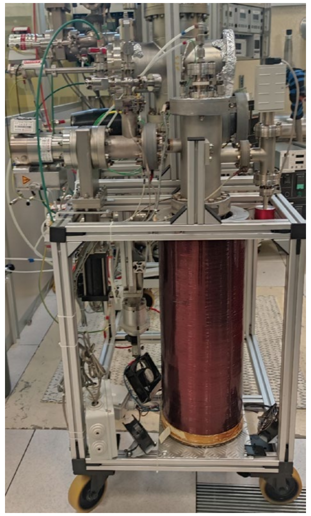
Nb on Cu 6 GHz cavity coating system



Replace Nb Cylindrical target with a Nb_3Sn one

Not so easy because
Nb_3Sn is extremely brittle!

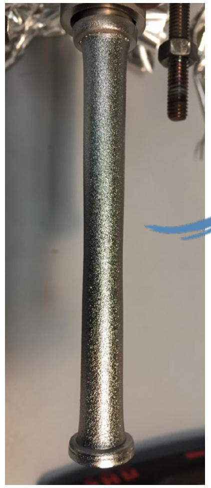
Sintered bulk target cannot be used



Nb_3Sn on Cu
LNL strategy for 6 GHz cavities

Nb target

Dipping
in Liquid Tin
+ Annealing



Nb_3Sn on Nb target

Magnetron
Sputtering

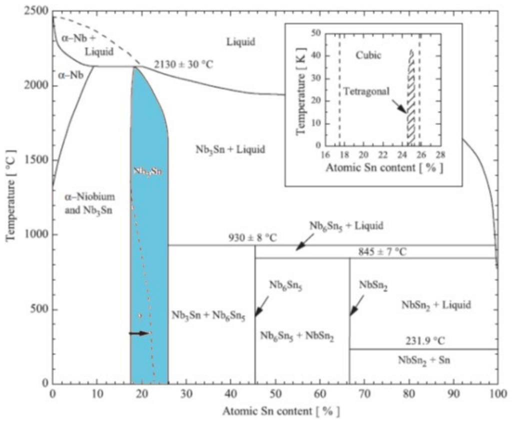
Liquid Tin Diffusion

in a couple of slides…

Vapor or Liquid Tin Diffusion

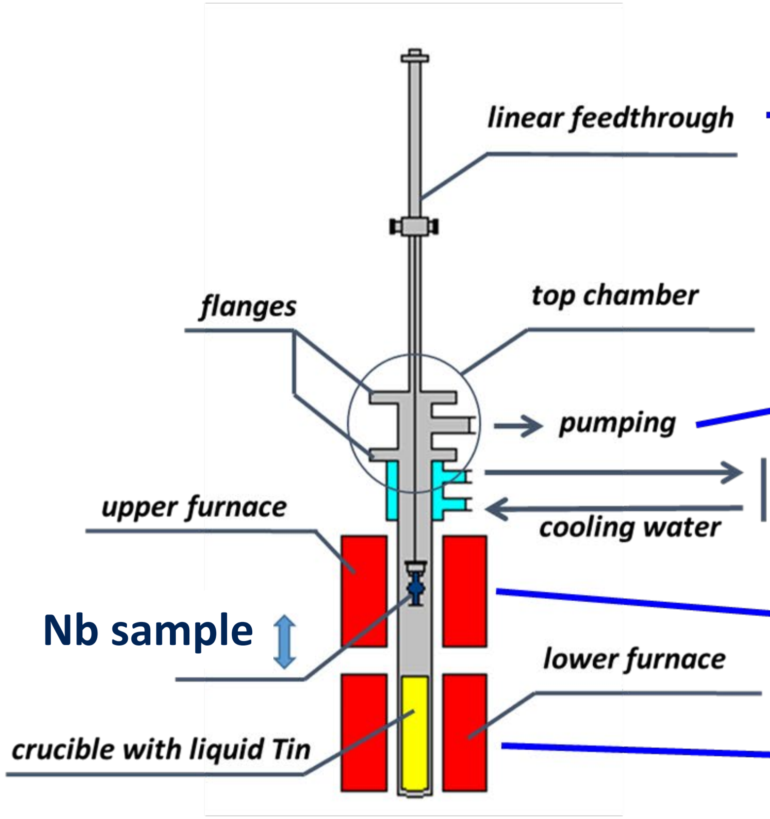
Tin Vapor Diffusion

Liquid Tin Diffusion
(Dipping)



- **Similar principle:** Sn diffuse onto Nb and react at High T
- Both suitable to growth **Nb_3Sn films on Nb substrates**
- Liquid Tin diffusion allows coating of **thick films**

Liquid Tin Diffusion Set Up @ INFN LNL



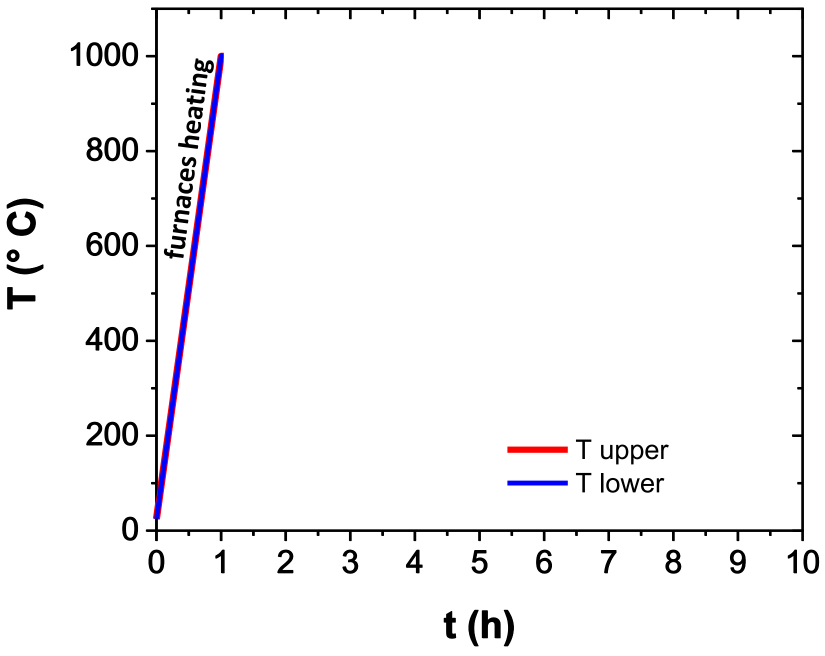
System **designed in 2005** for the coating of 6 GHz elliptical cavities

UHV Inconel chamber

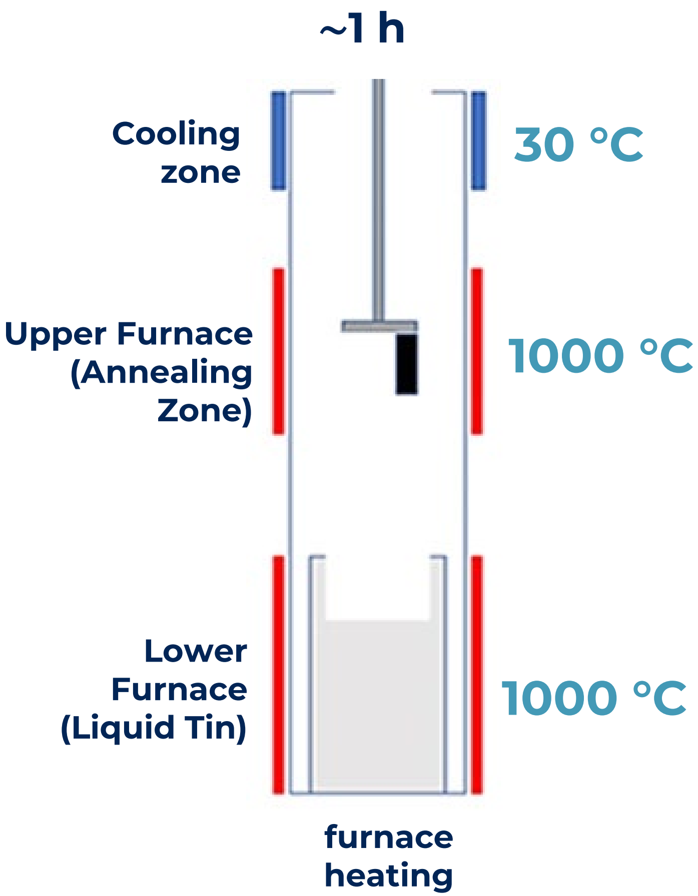
2 Separate **resistive furnaces**

Dipping process (LNL 2006)

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



Temperature furnaces profile

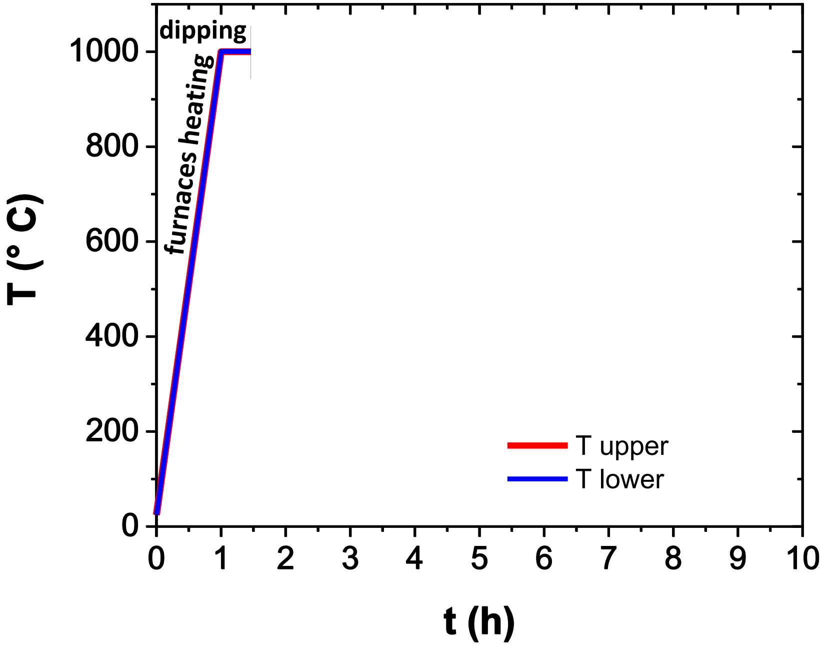


Sample temperature

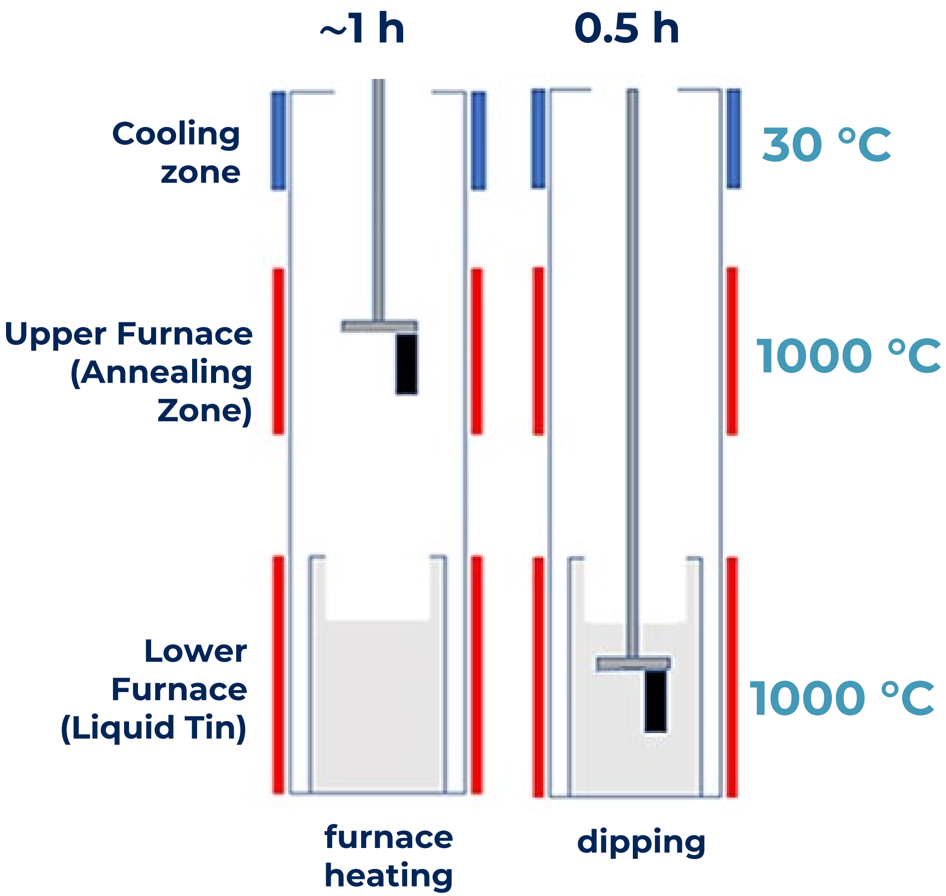
$\Delta T \sim$ - 100-150 °C

Dipping process (LNL 2006)

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



Temperature furnaces profile



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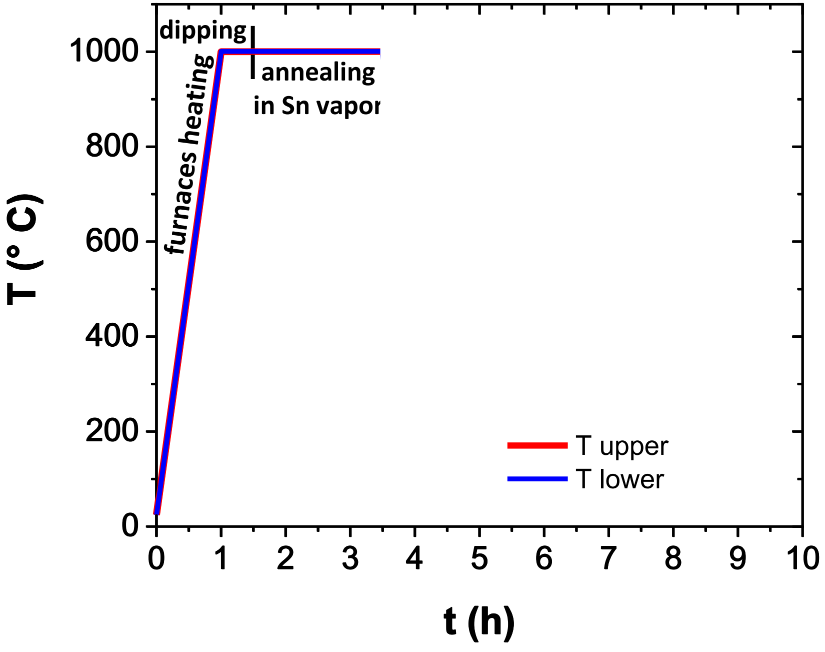
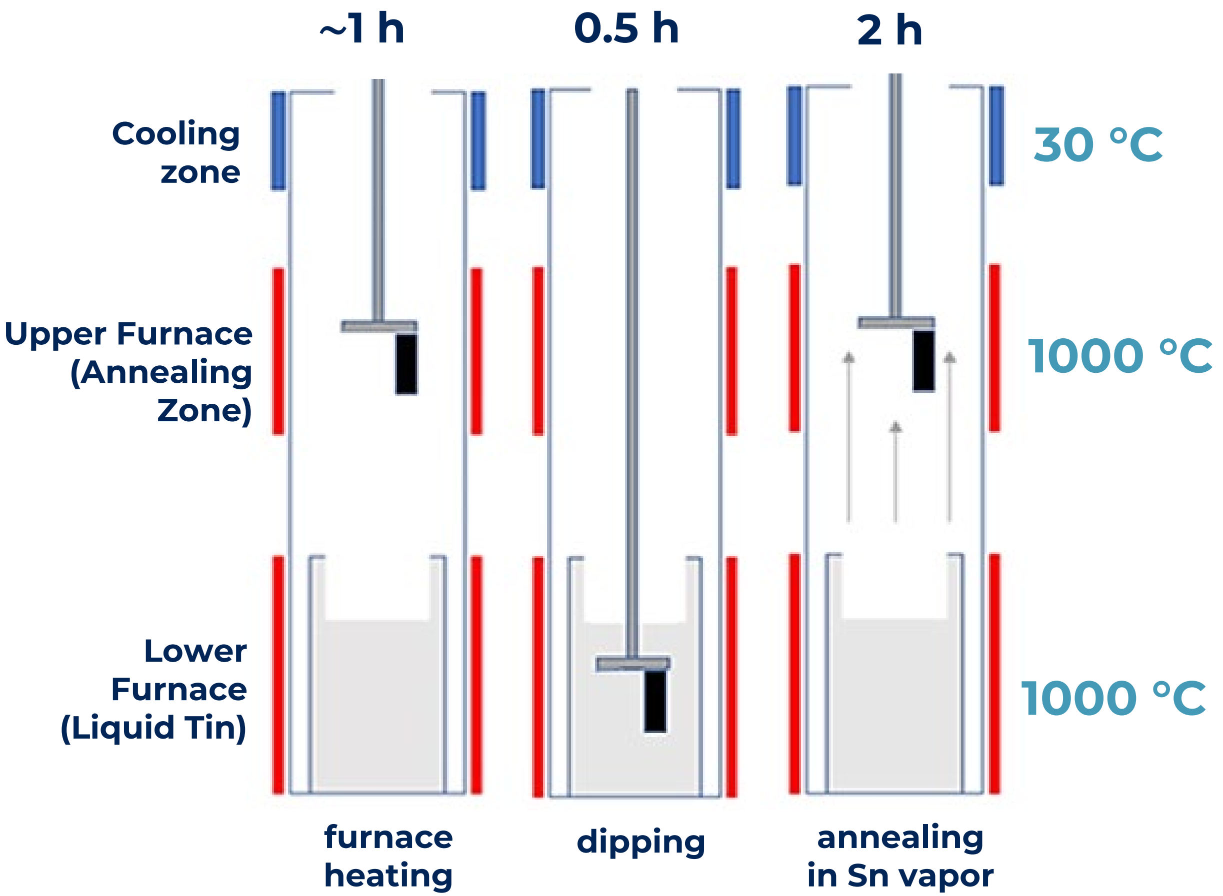


Temperature furnaces profile



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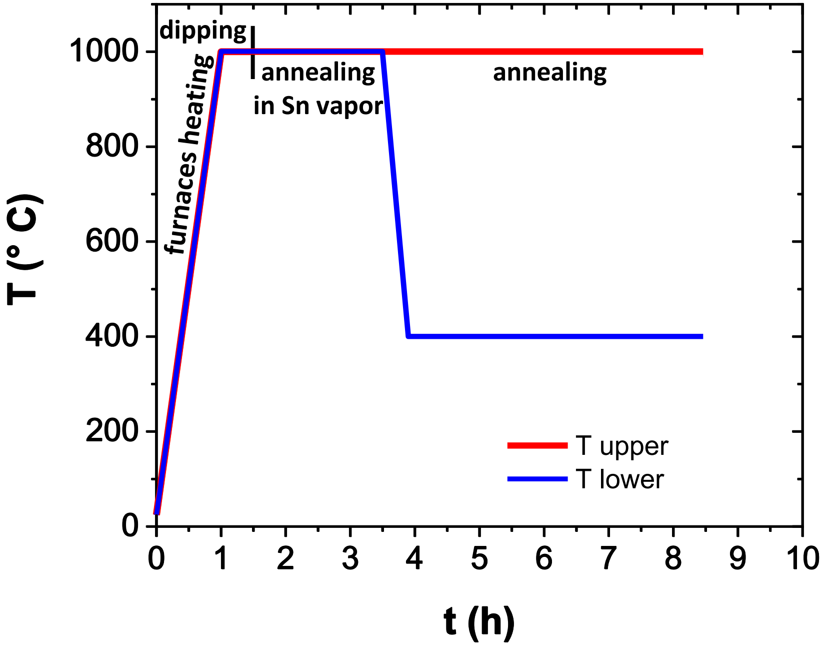
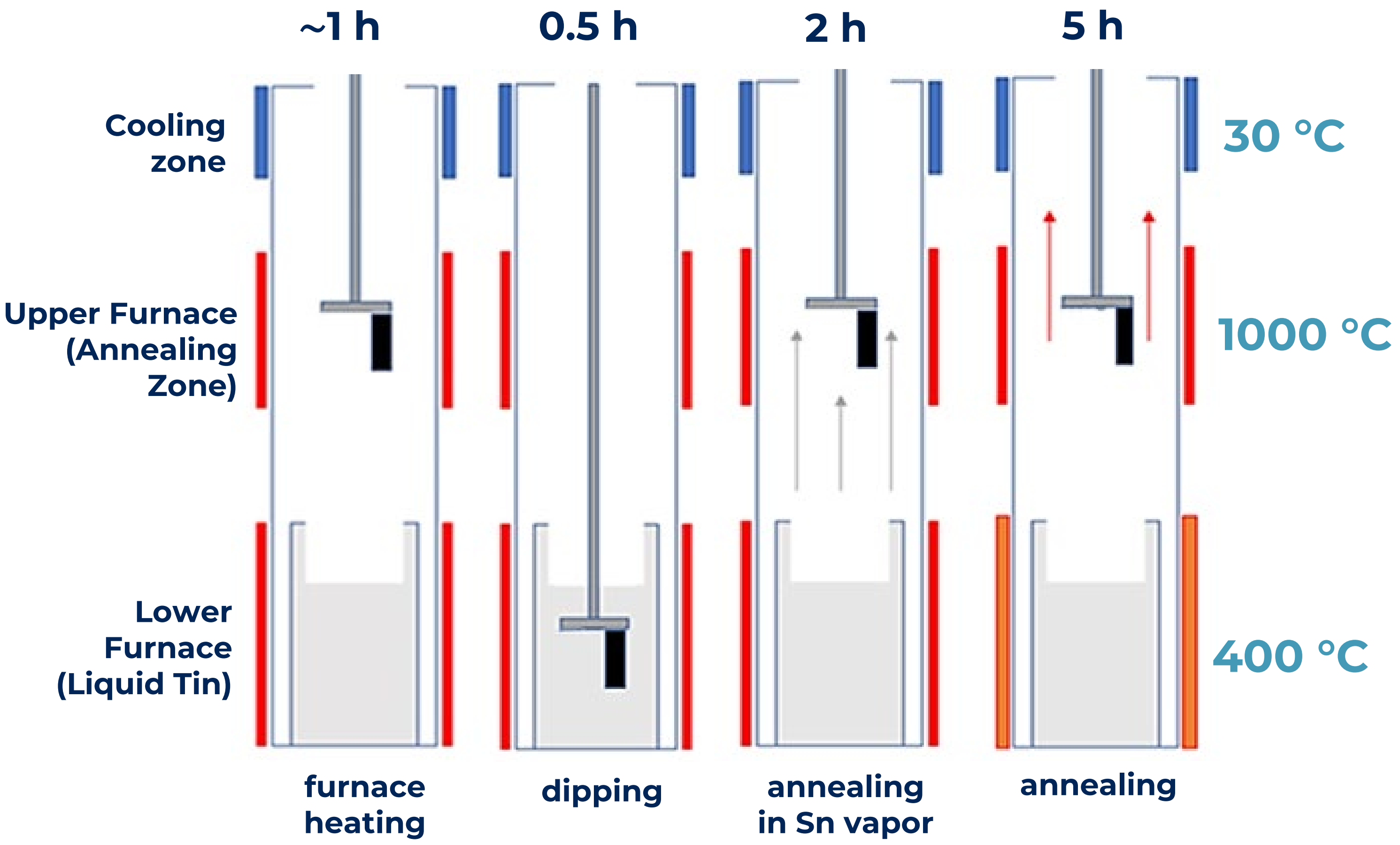


Temperature furnaces profile



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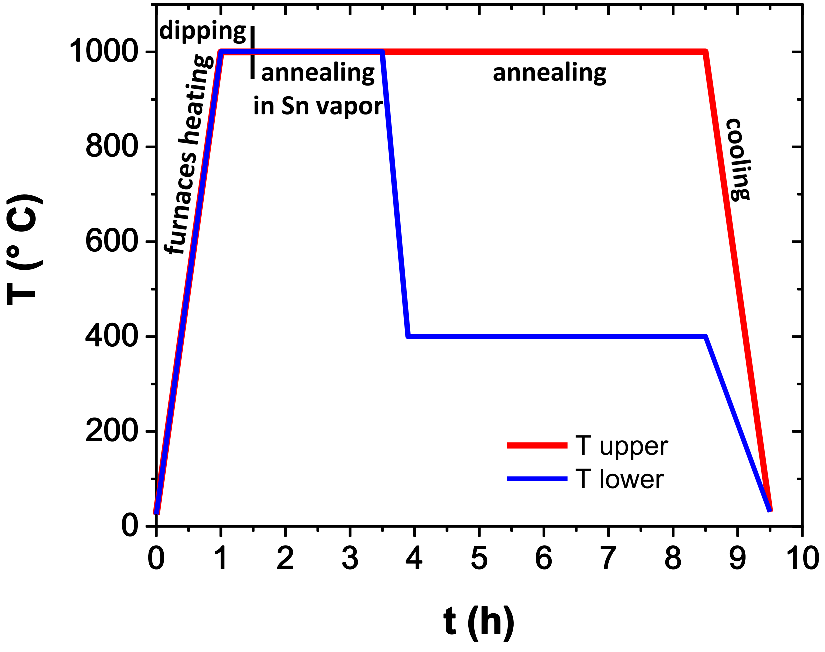
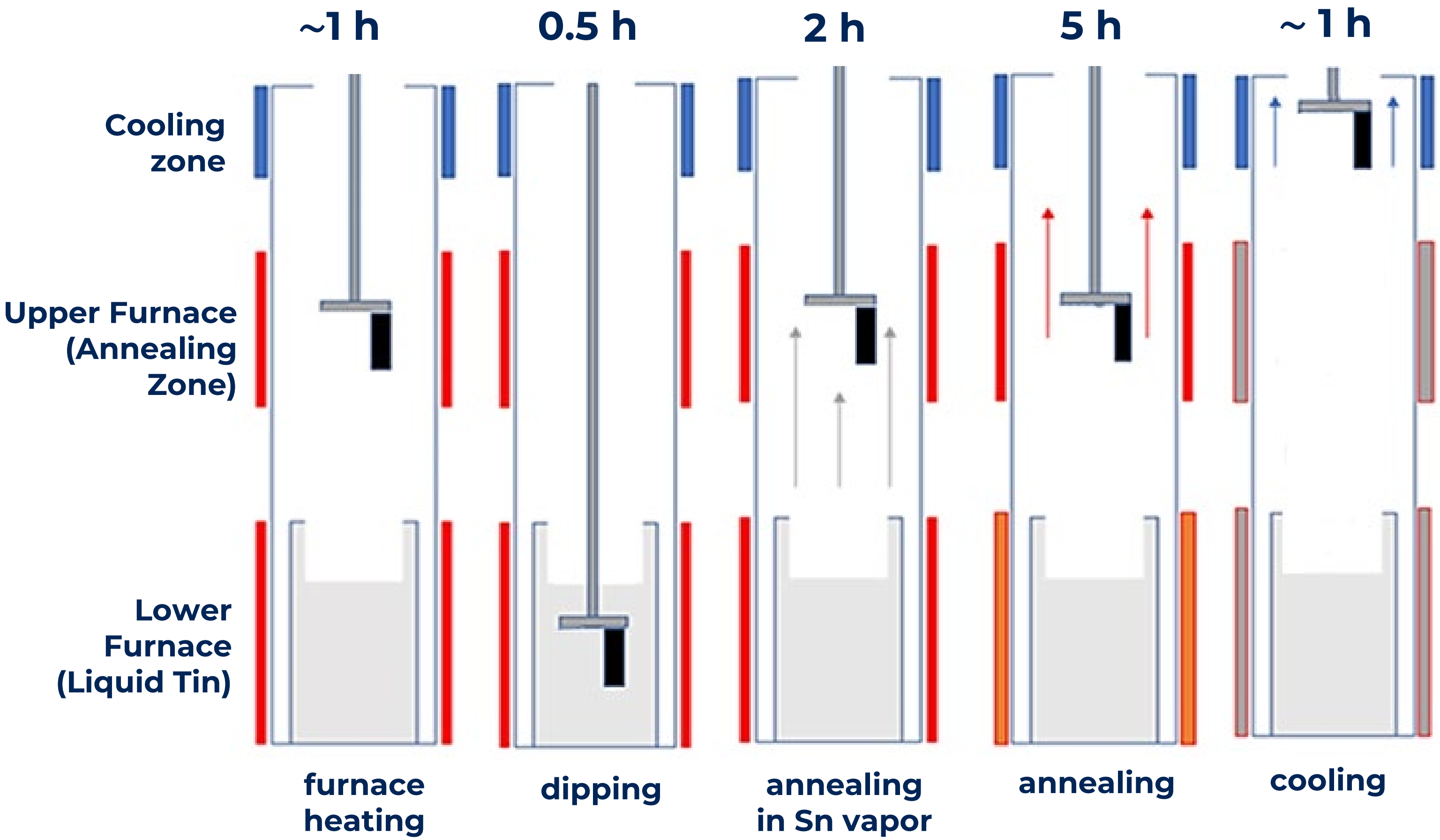
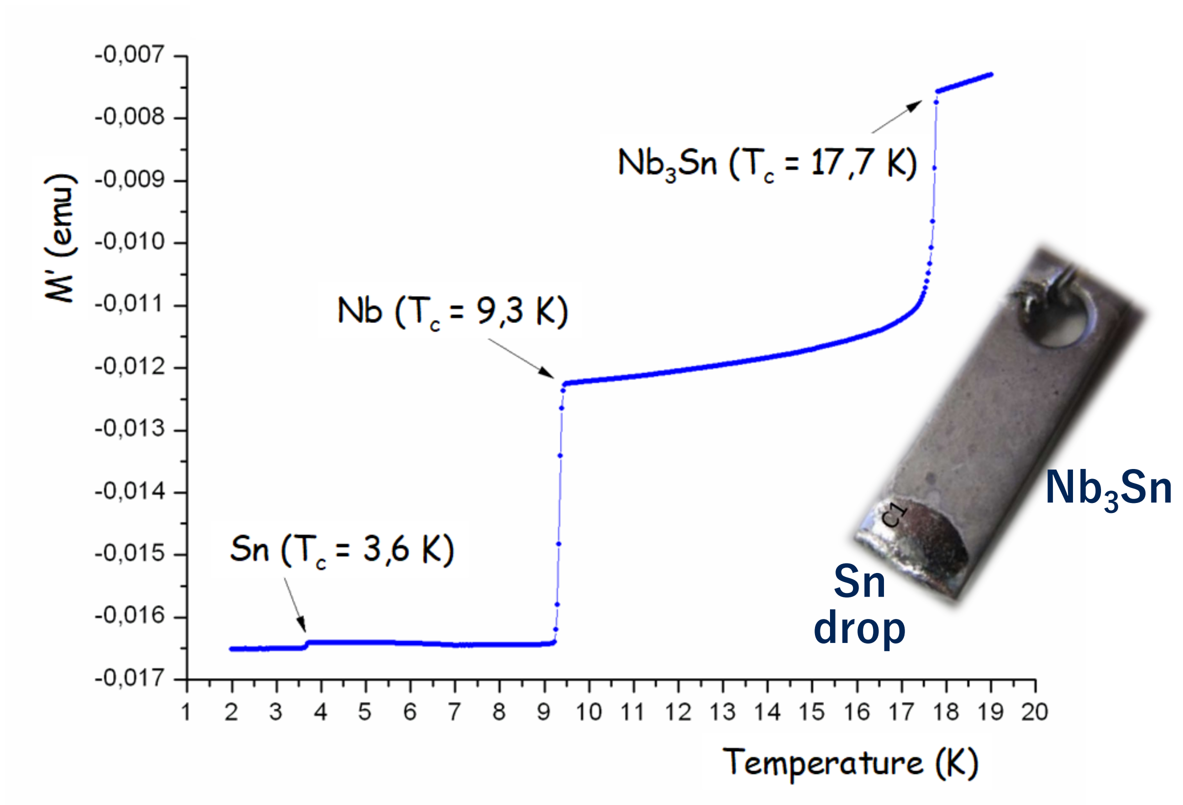


Temperature furnaces profile

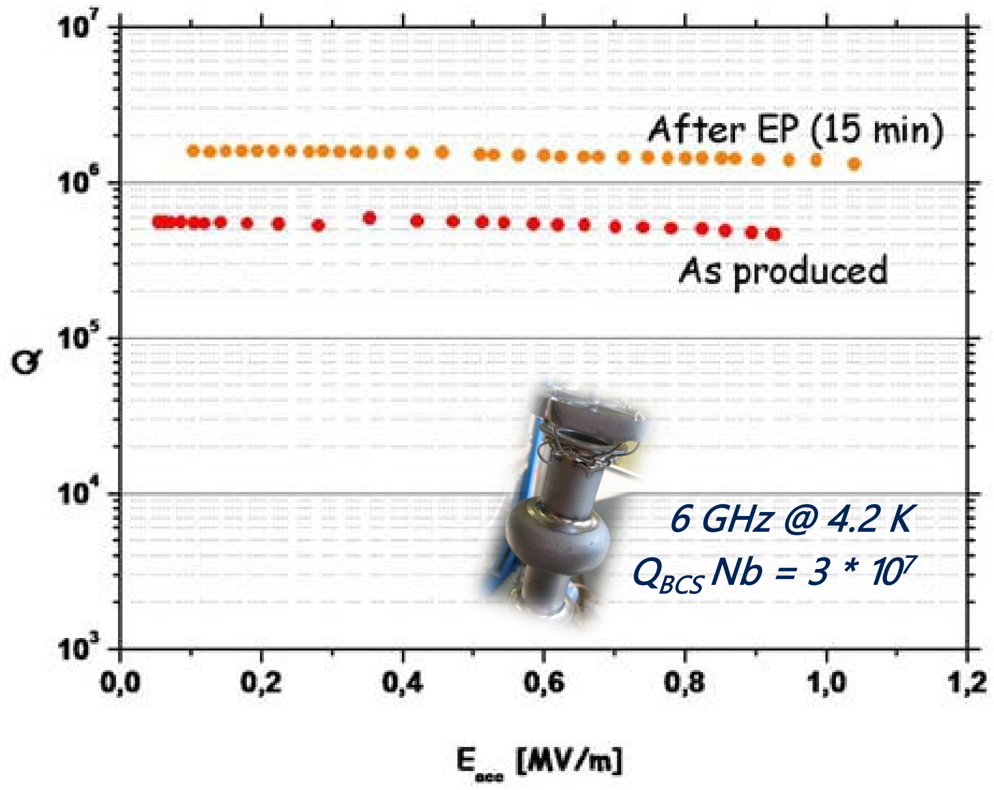


Results (LNL 2006)

S. M. Deambrosis et al., "A15 superconductors: An alternative to niobium for RF cavities," Physica C, 2006
S. M. Deambrosis, SRF 2009



Good performance on **planar samples**

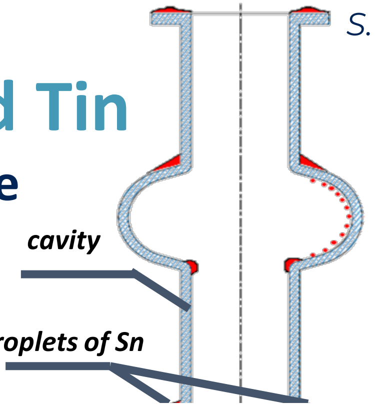


Poor performance on 6 GHz **cavity**

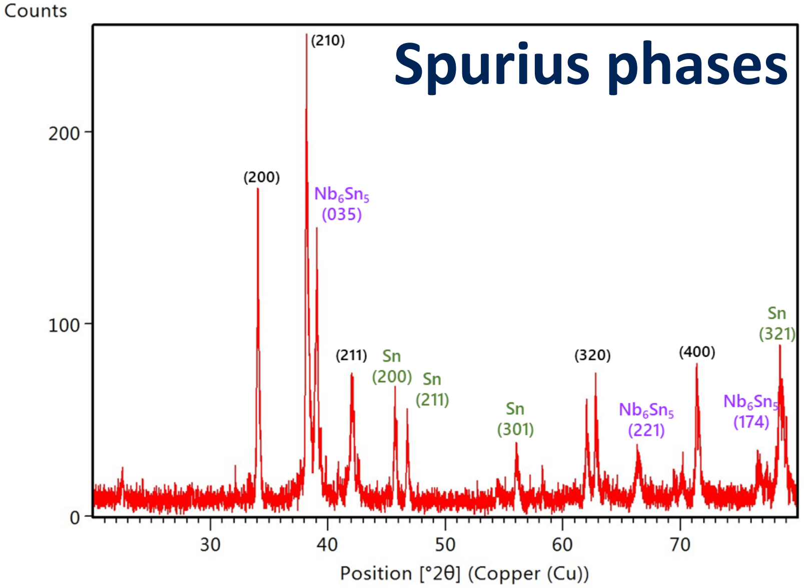
Results (LNL 2006)

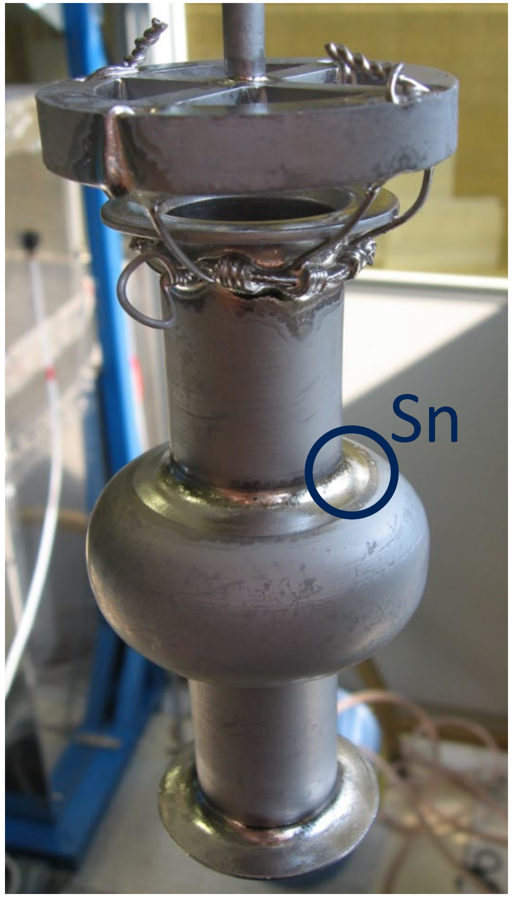
S. M. Deambrosis et al., "A15 superconductors: An alternative to niobium for RF cavities," Physica C, 2006
S. M. Deambrosis, SRF 2019

Drops of
unreacted Tin
on the surface

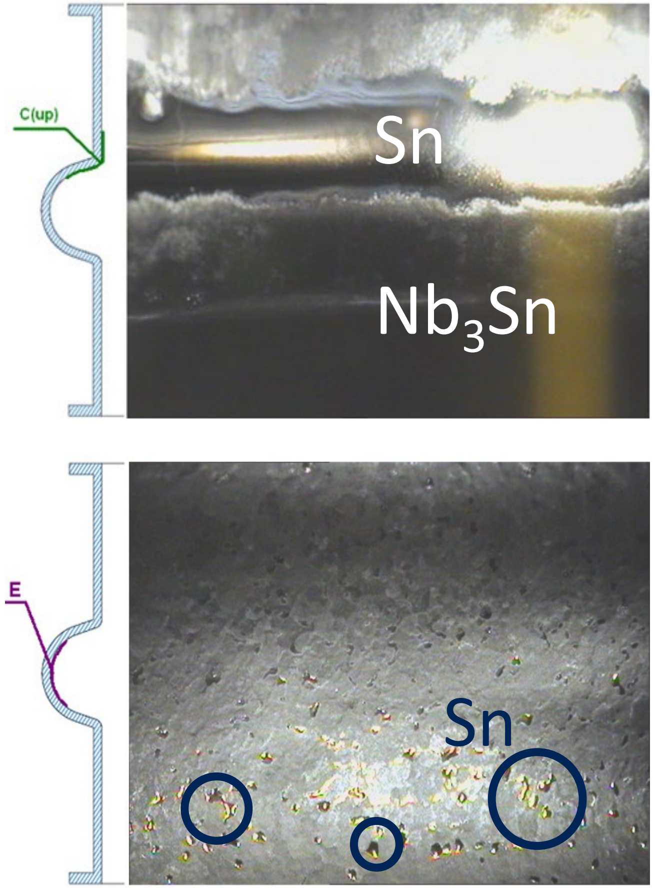


Spurius phases





External surface

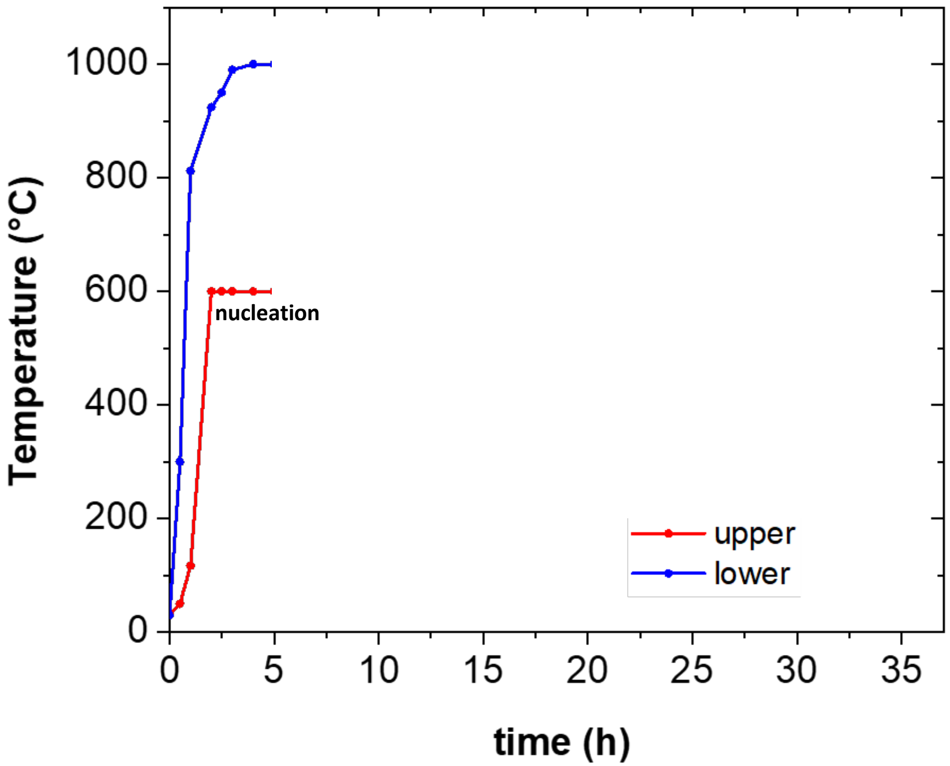


Internal surface

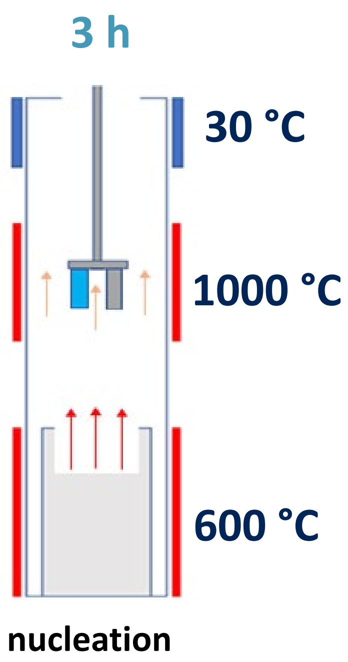
Dipping process optimization and Nb_3Sn target production

Dipping Optimized Process

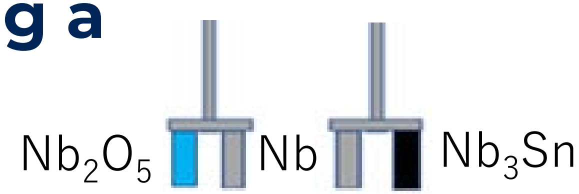
M. Zanierato, SRF 2021



Temperature furnaces profile

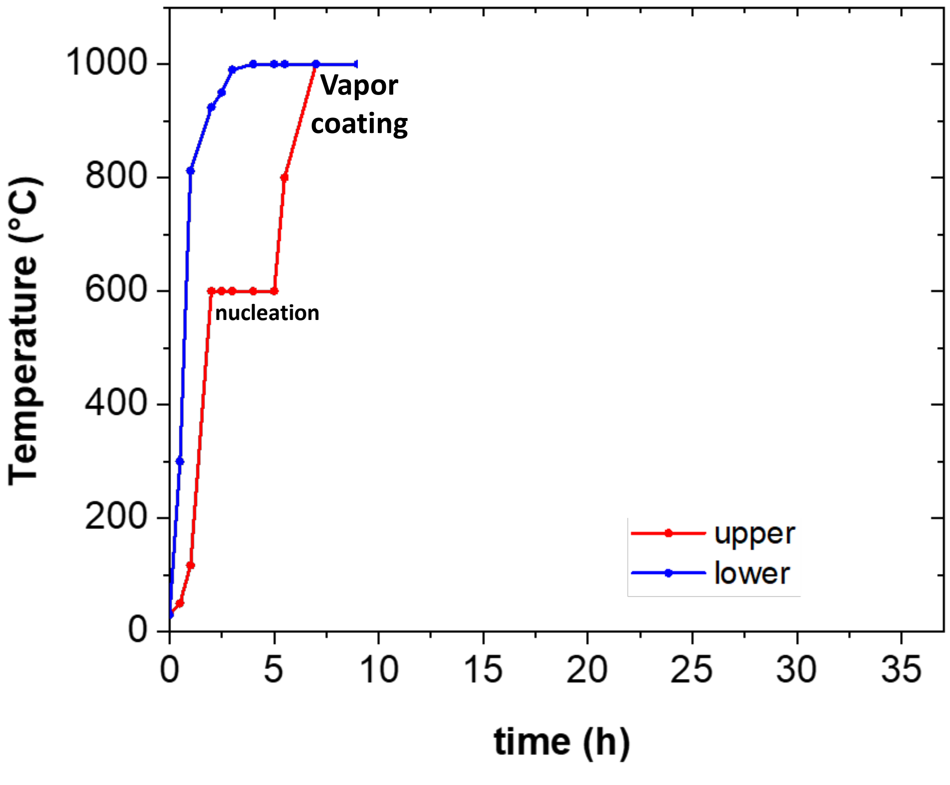


Nucleation improved adding a vapor coating step on anodized Nb samples

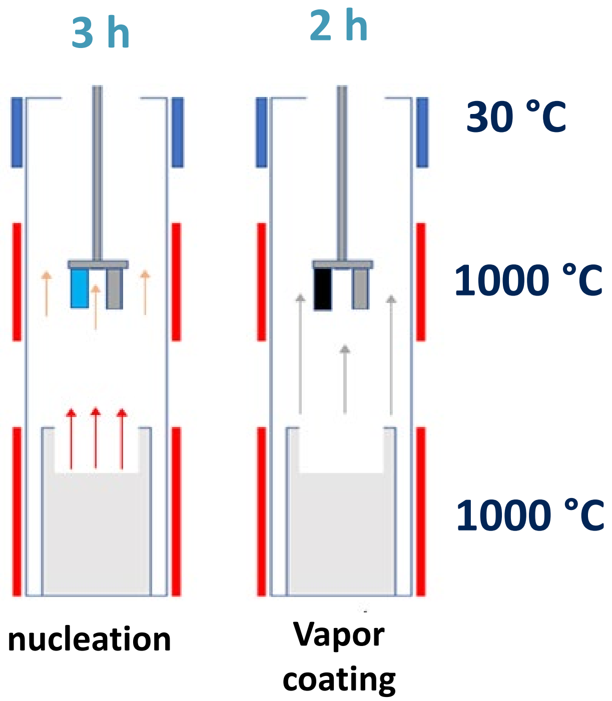


Dipping Optimized Process

M. Zanierato, SRF 2021



Temperature furnaces profile



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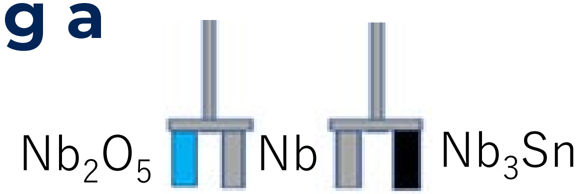
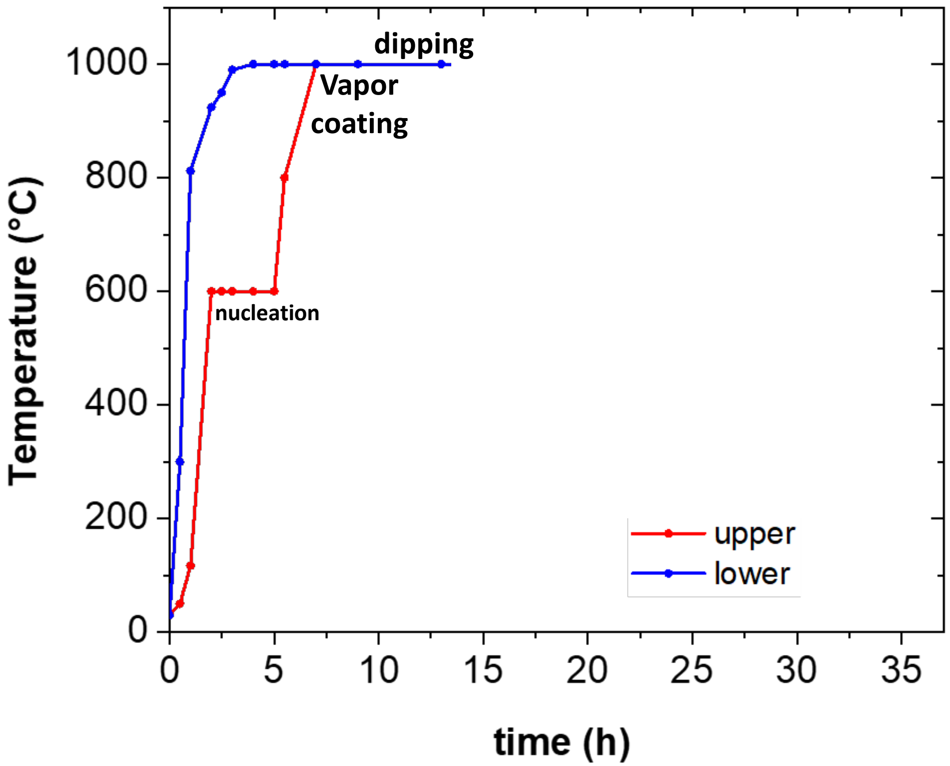
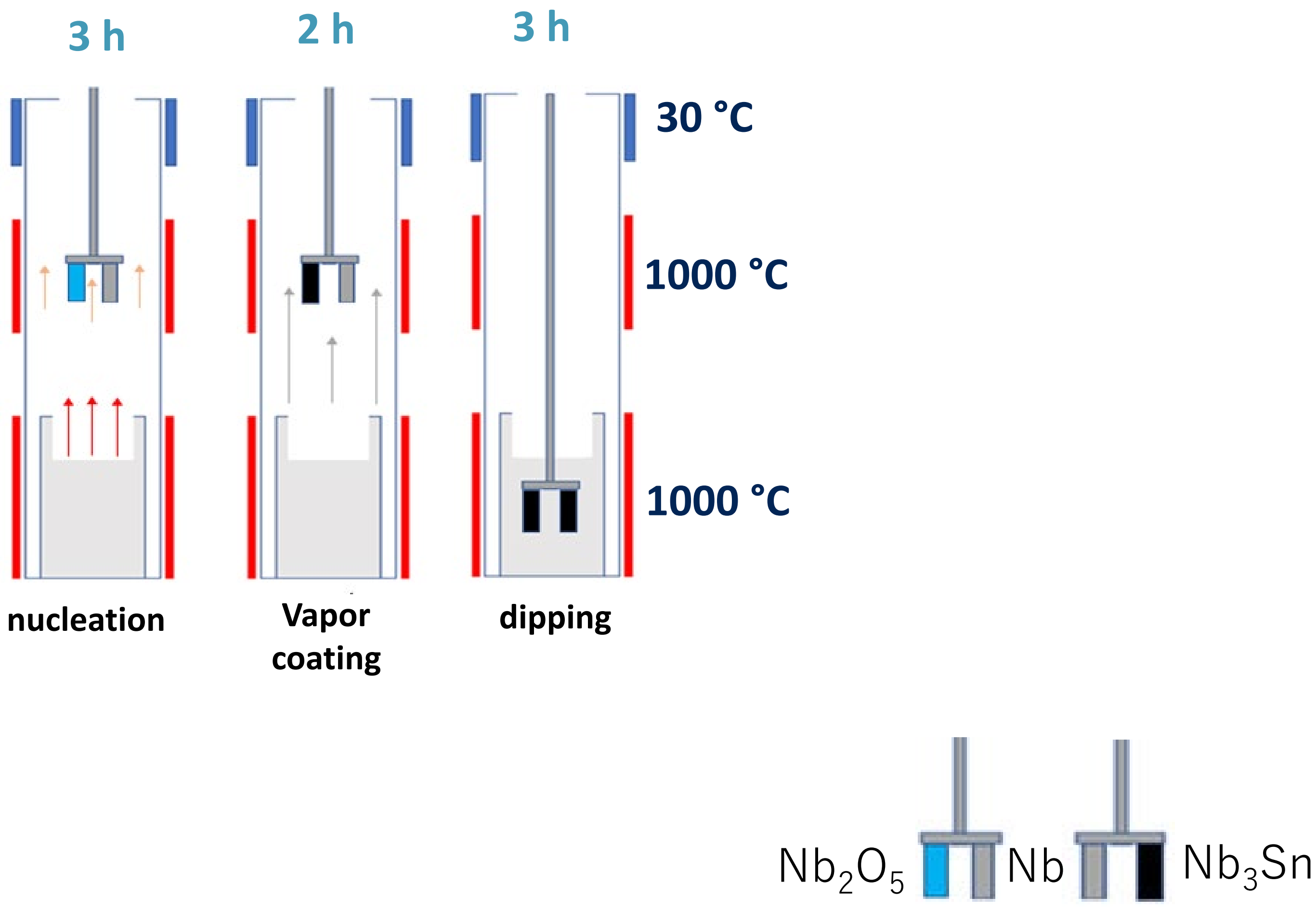


Dipping Optimized Process

M. Zanierato, SRF 2021

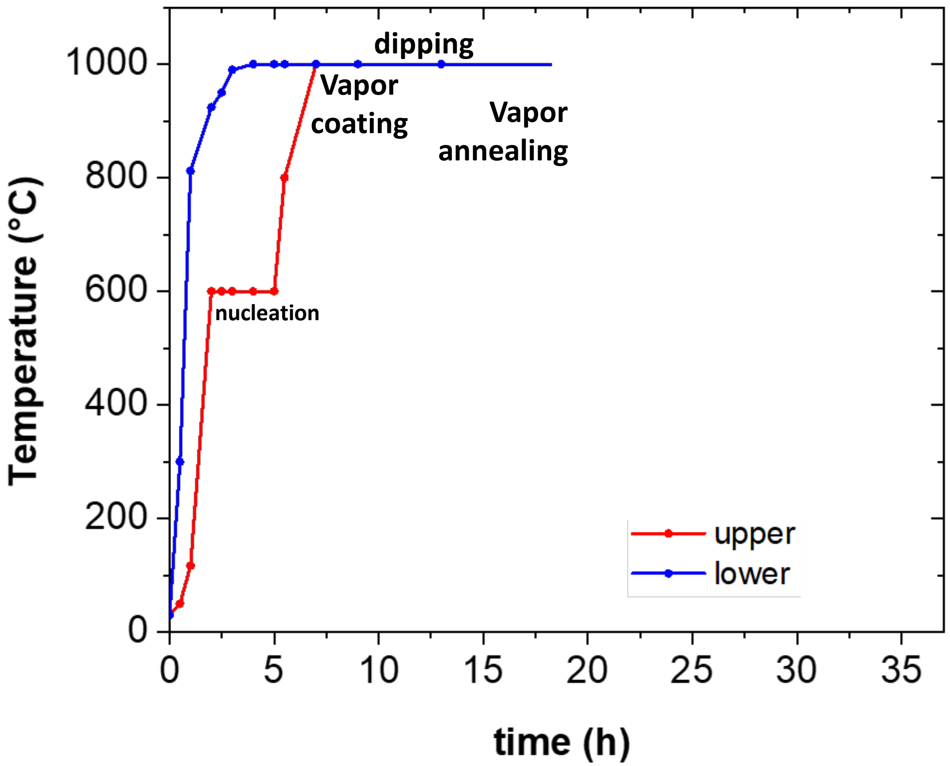


Temperature furnaces profile

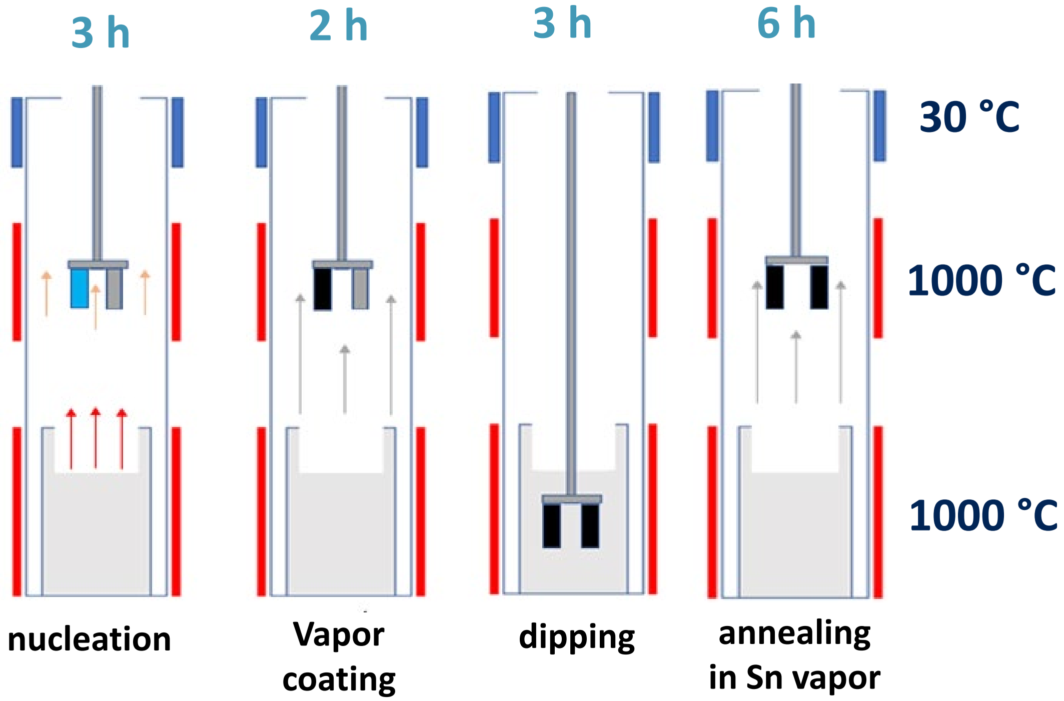


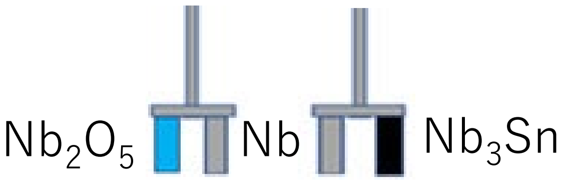
Dipping Optimized Process

M. Zanierato, SRF 2021



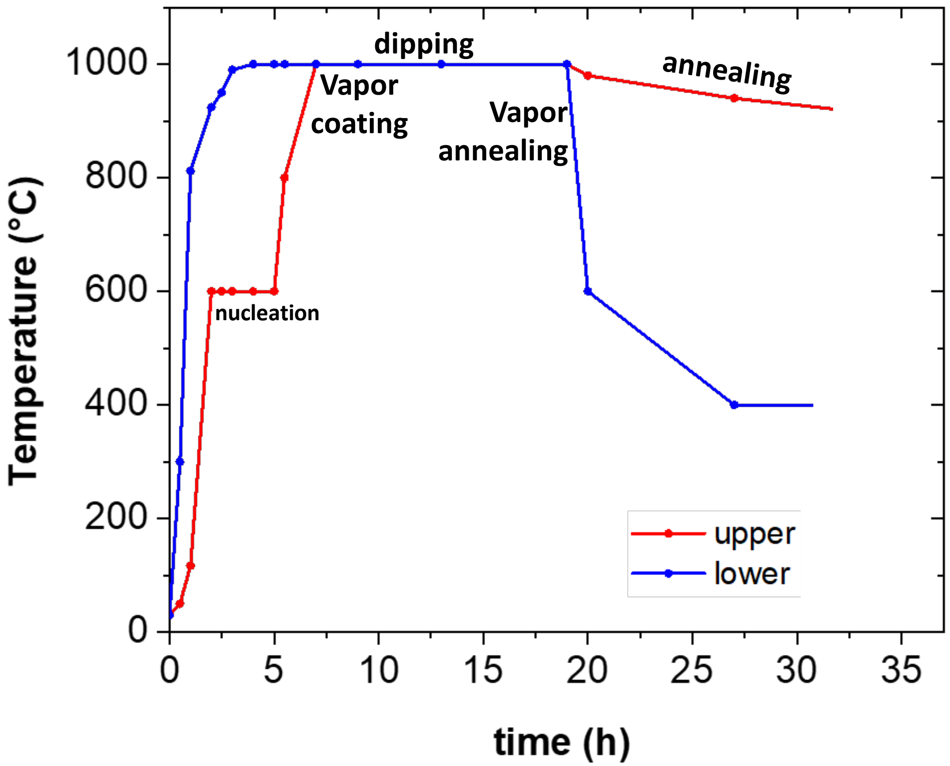
Temperature furnaces profile



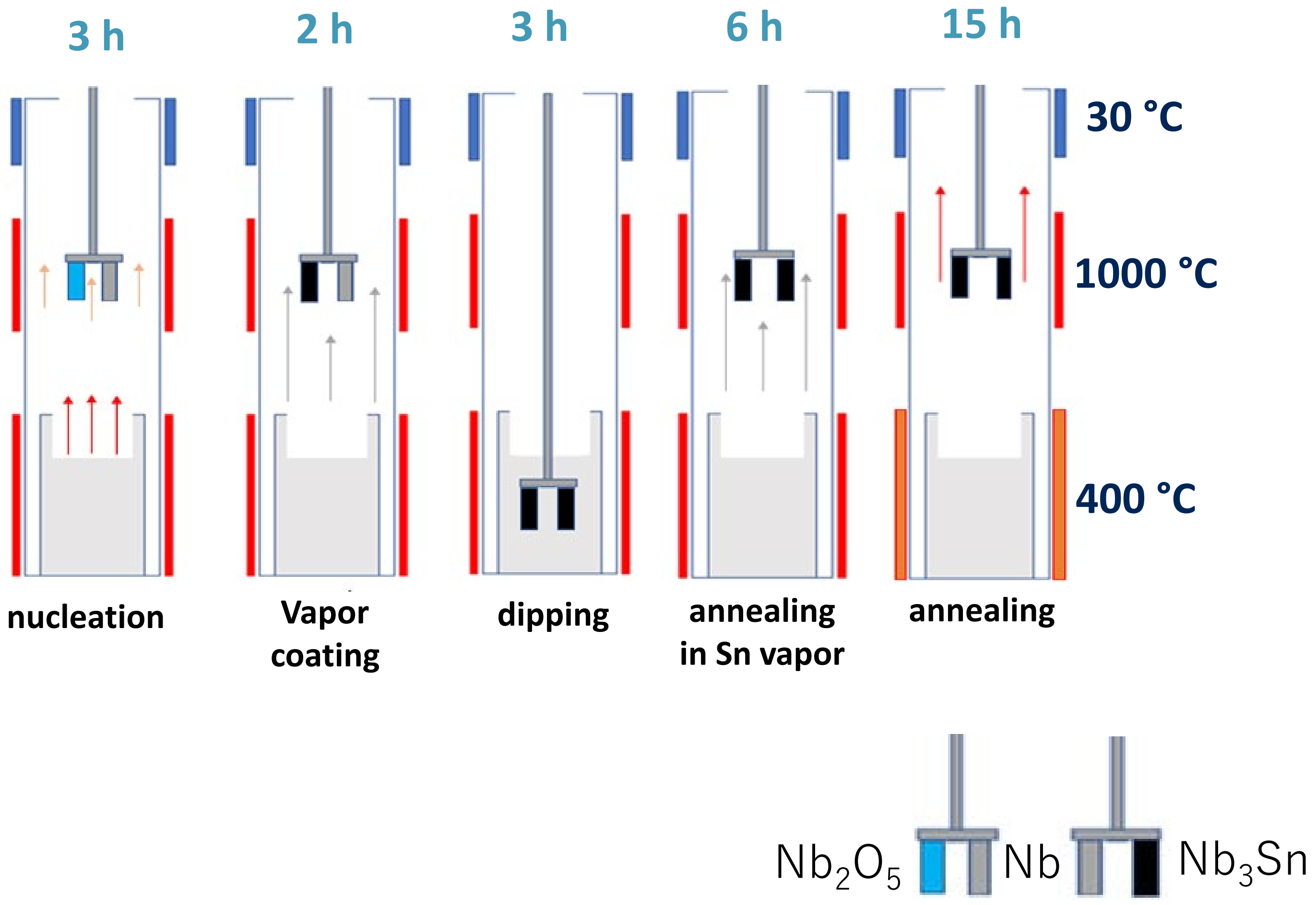


Dipping Optimized Process

M. Zanierato, SRF 2021

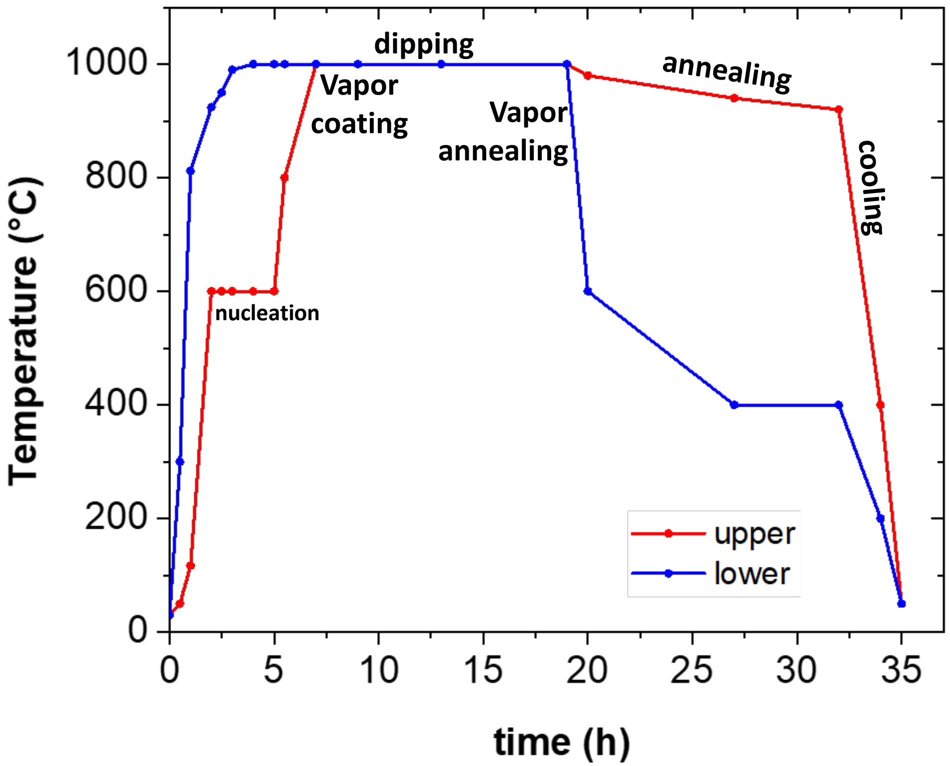


Temperature furnaces profile

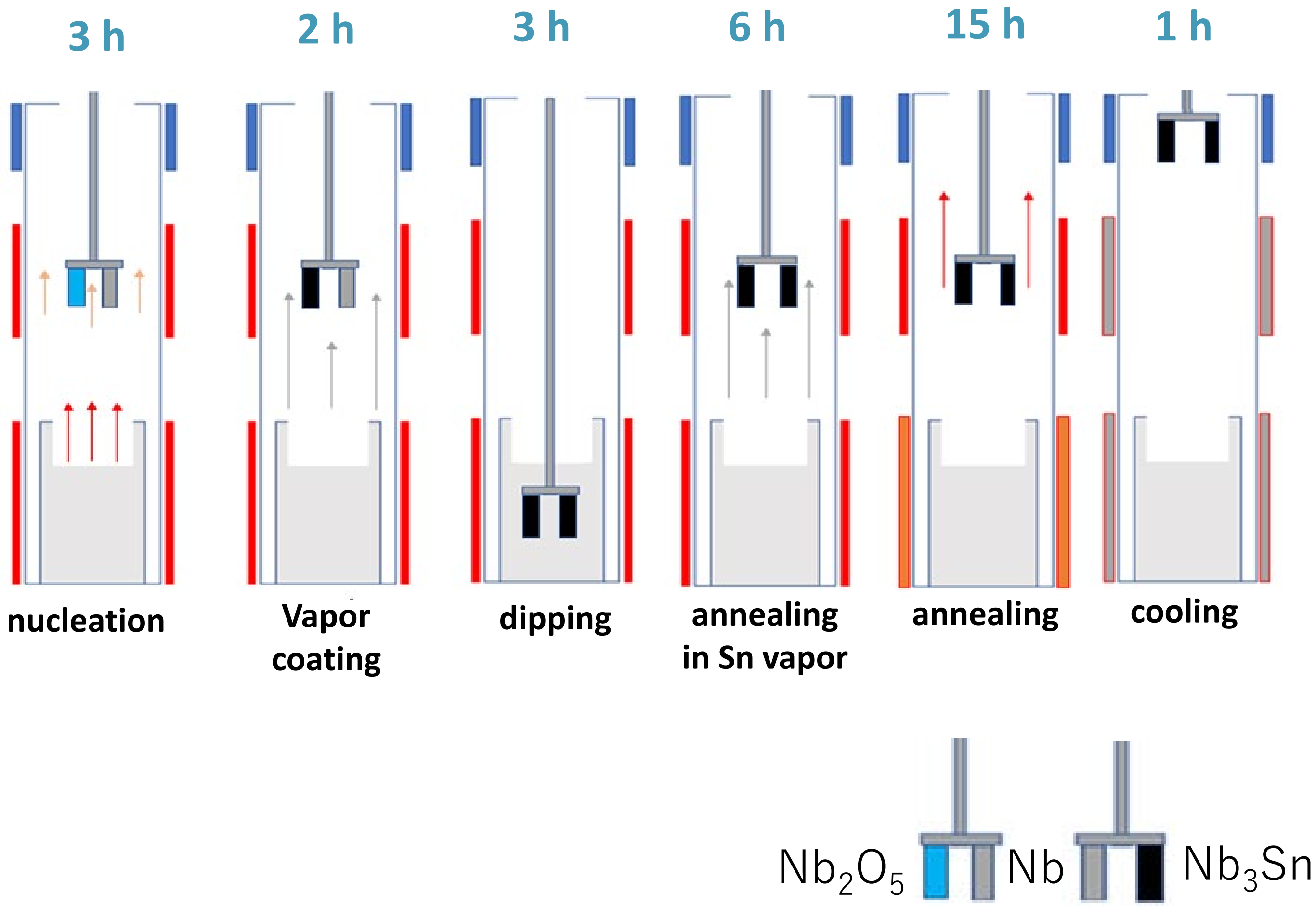


Dipping Optimized Process

M. Zanierato, SRF 2021



Temperature furnaces profile

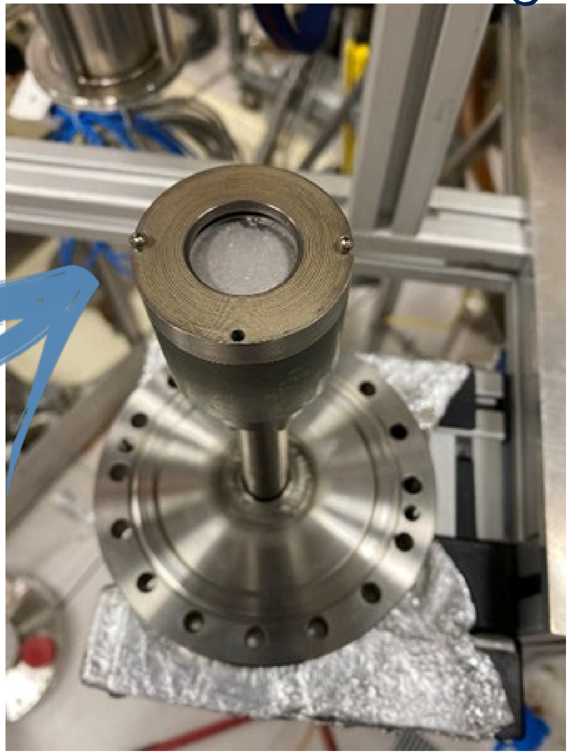


Test with 1" target

Anodized samples

Nb_3Sn coated samples

1" Magnetron ready



Shape optimized

to collect and remove Tin excess

Dipping time - thickness

C. Pira, Thin Films Workshop SRF 2021
M. Zanierato, SRF 2021

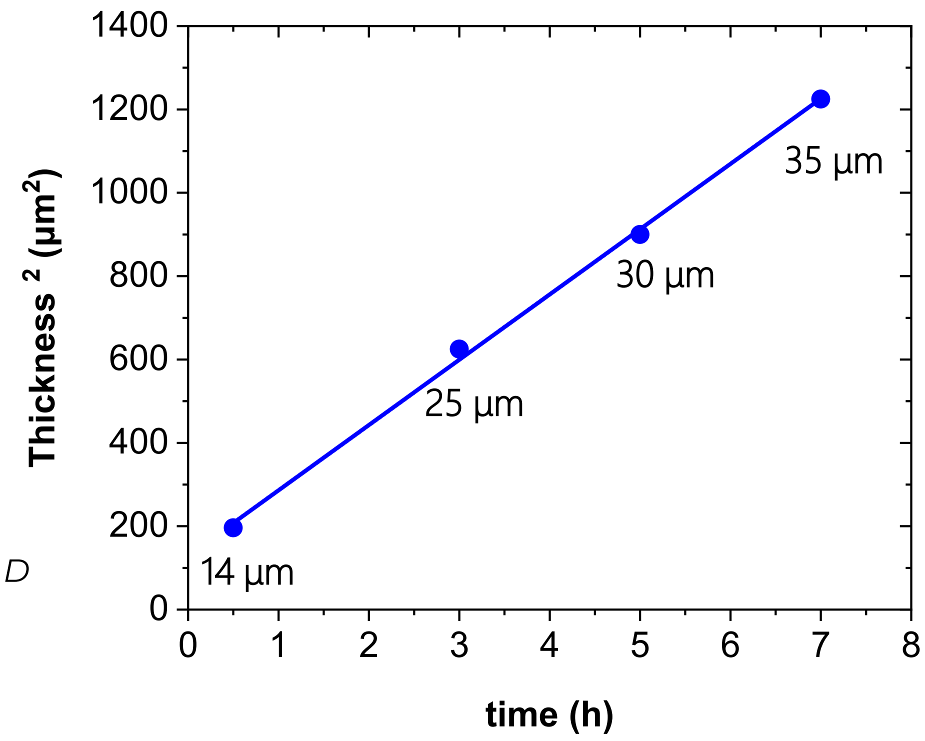
Nb_3Sn growing rate by **dipping** is **related to the Sn diffusion** in Nb and can be described by **Fick' laws of diffusion**

$$x^2 = kt$$

x: *Nb_3Sn thickness*

t: *dipping time*

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



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

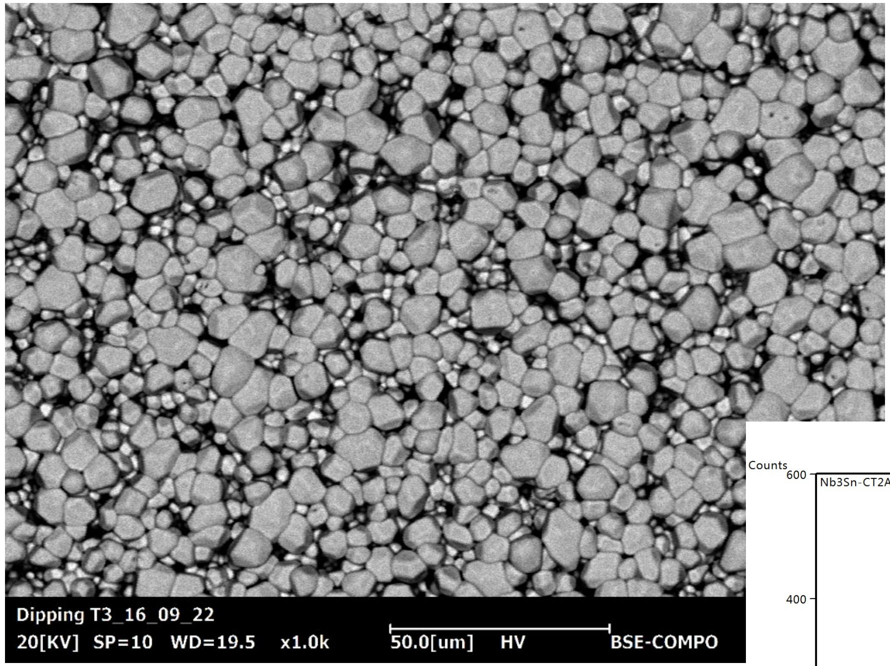
$$R^2 = 0.9983$$

100 µm → ~ 63 hours of dipping!

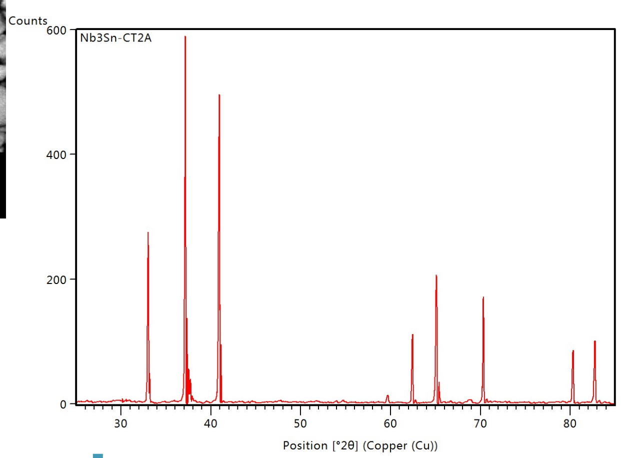
24 hours of dipping → ~ 62 µm

in progress

Target Morphology
(SEM + EDS characterization)



Stoichiometric Nb-Sn composition

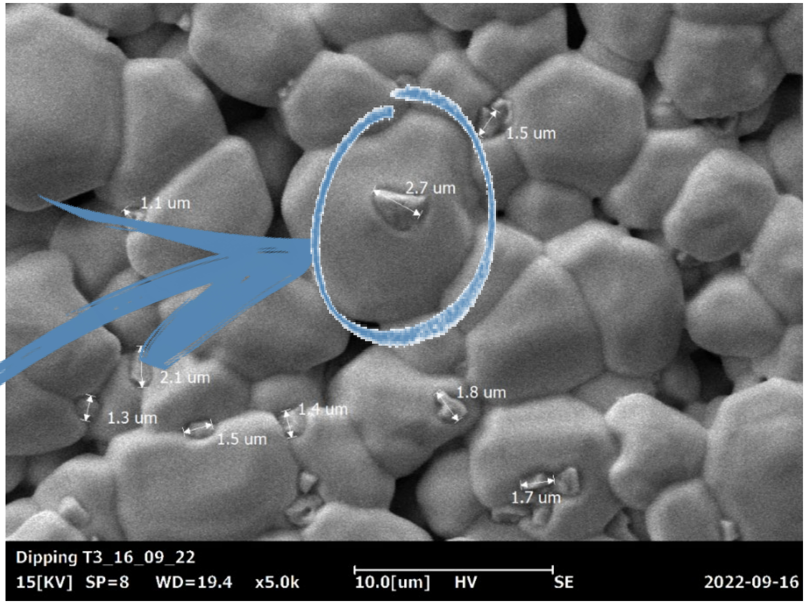


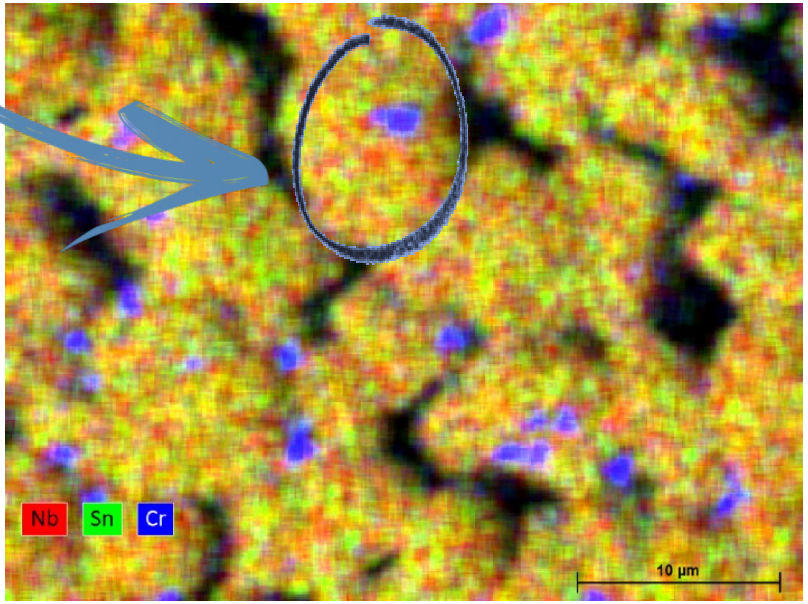
XRD shows only Nb_3Sn phase

Chromium contamination

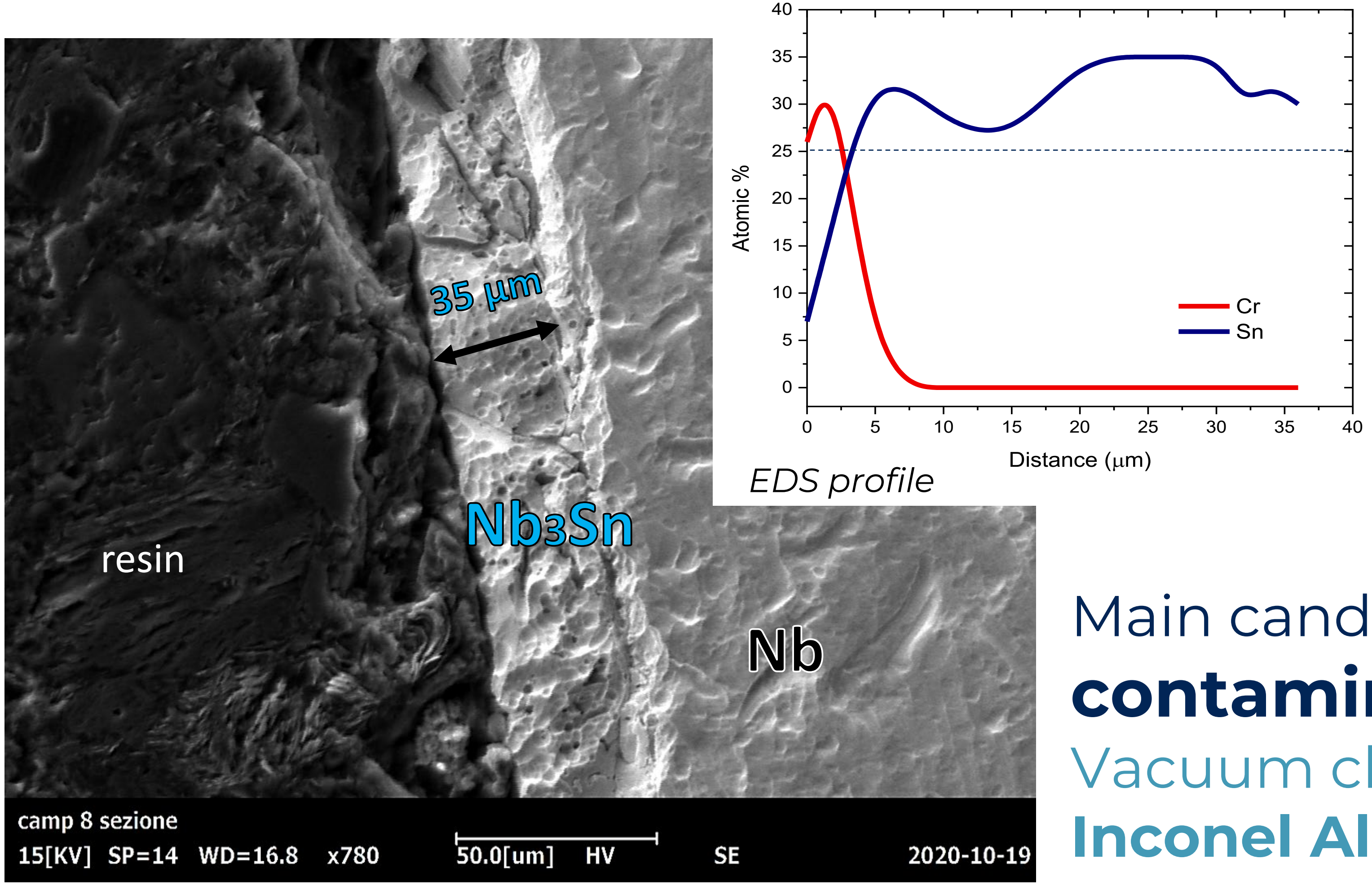
(10-20% on surface)

Ni and Fe are also present





Target cross section



Contamination is superficial

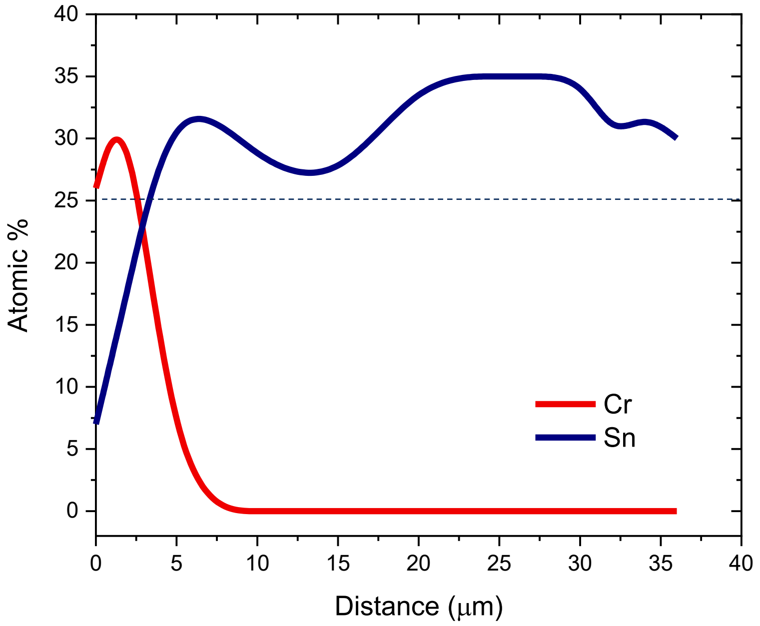
No contamination in Sn crucible

Main candidate for **Cr contamination:**

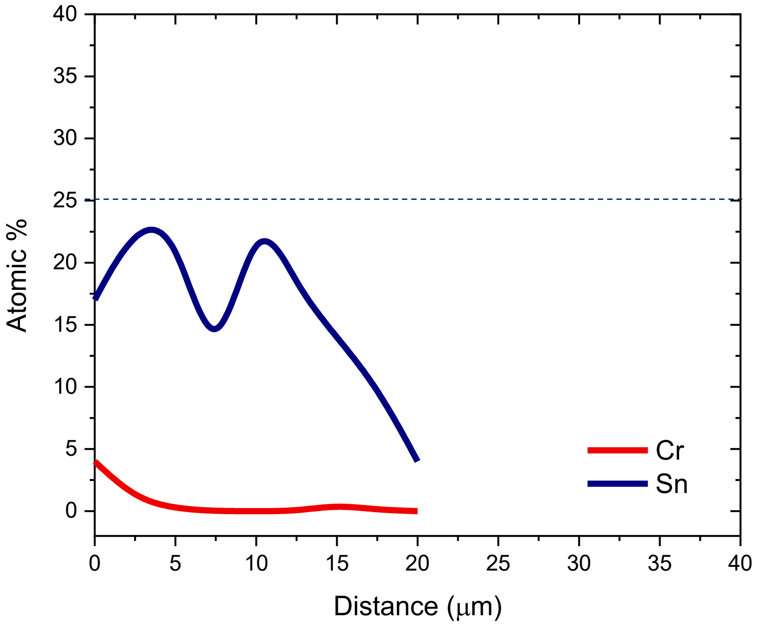
Vacuum chamber made by **Inconel Alloy**

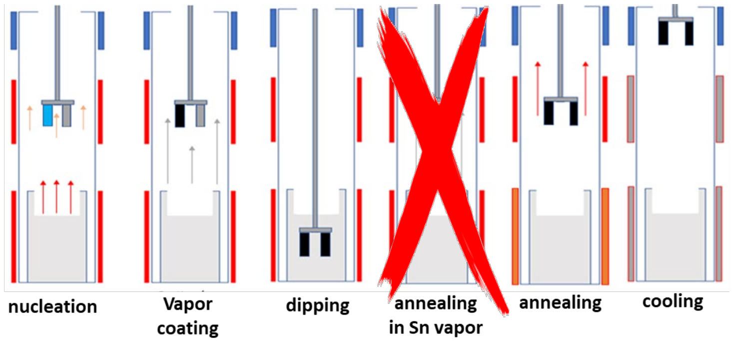
Tin content modulation

With Vapor Annealing Step



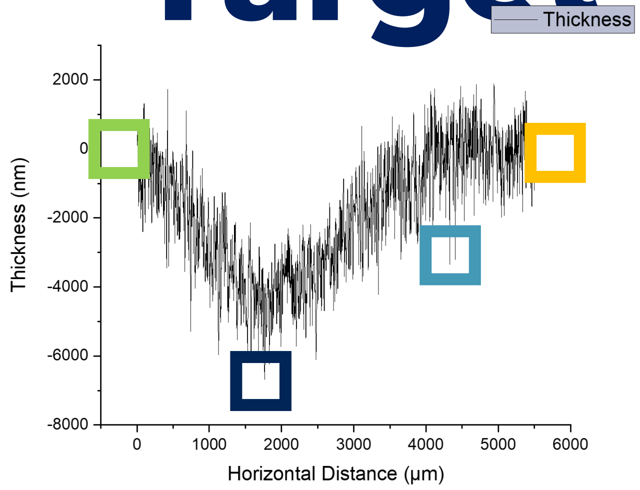
Without Vapor Annealing Step



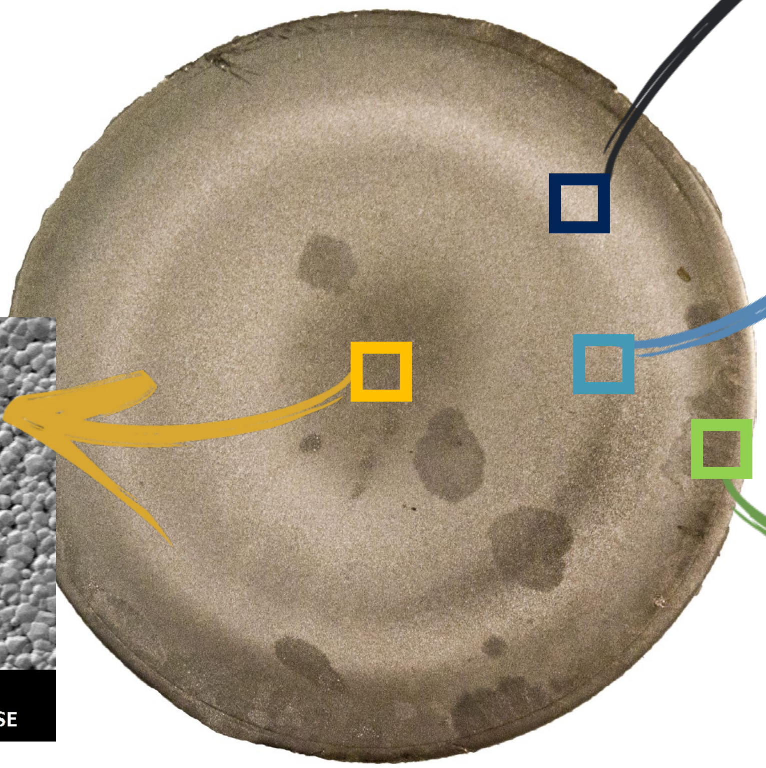


Vapor annealing step can be used to modulate Tin content

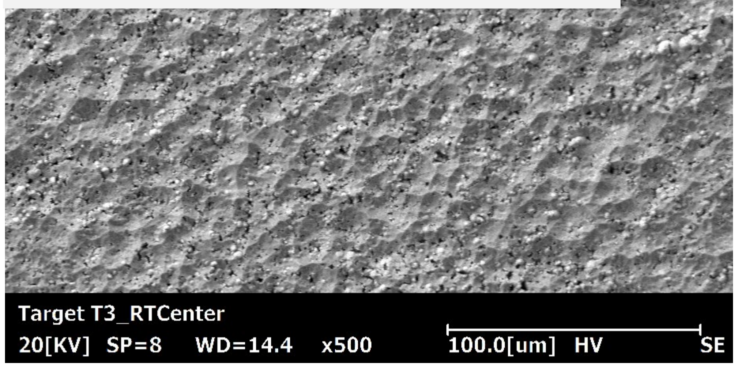
Target surface



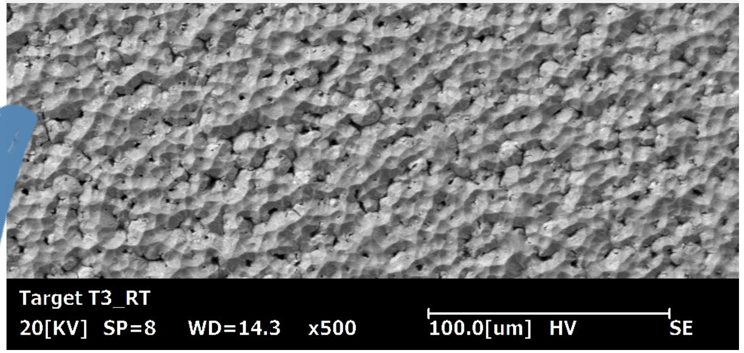
~6 microns eroded
700 nm coated



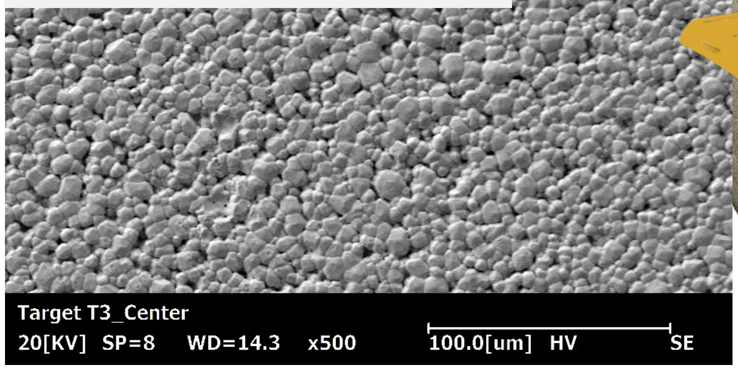
Race Track: **Sn 22.8%**



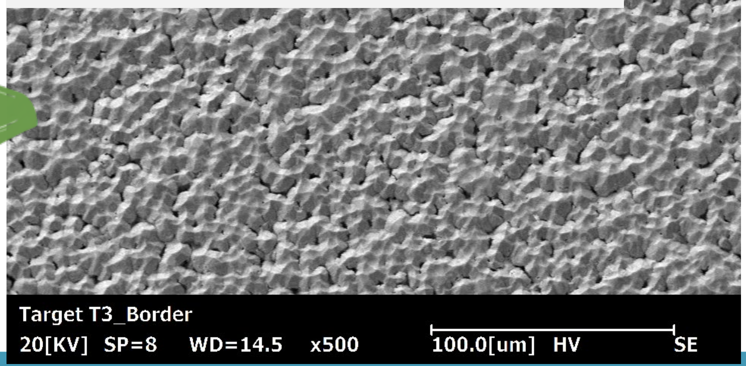
Race Track side: **Sn 23.2%**



Center: **Sn 23.5%**



Ext. border: **Sn 23.0%**

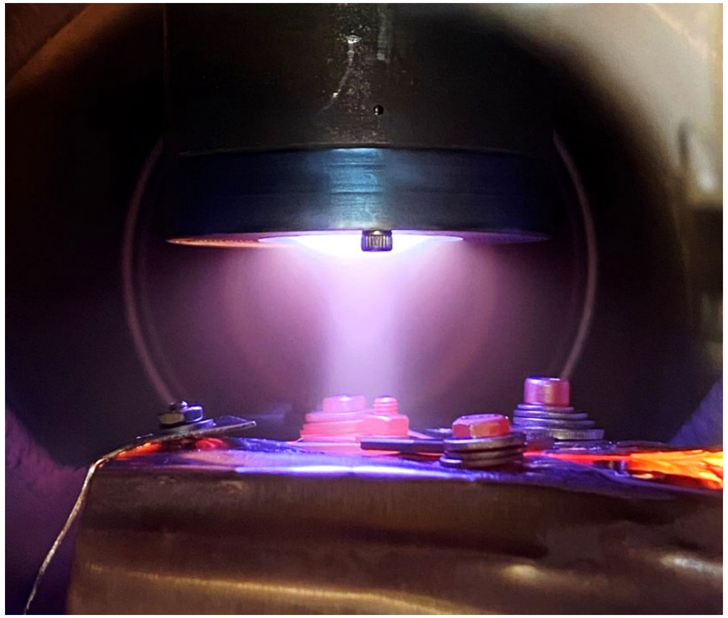


First sputtering test on Quartz

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

- Coating on **quartz** samples by MS

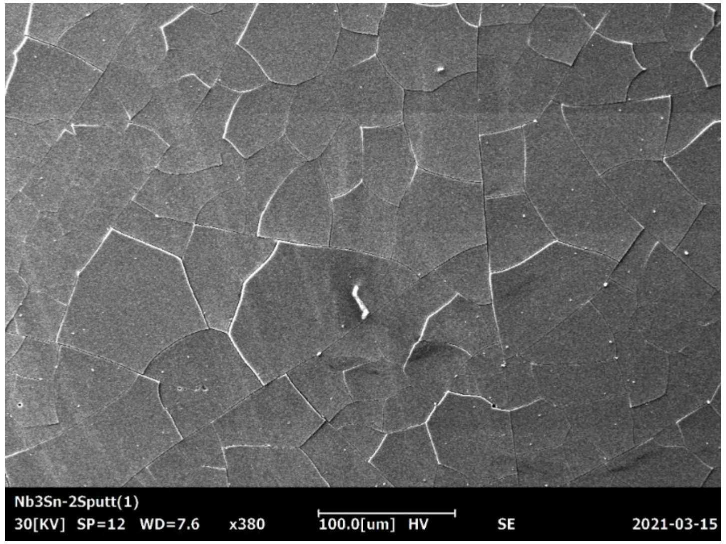
  $I = 0.1$ A (5 mA/cm^2)
  $t = 30$ min
  $T = 750$ °C



Zanierato et al,
SRF Proceedings 2021

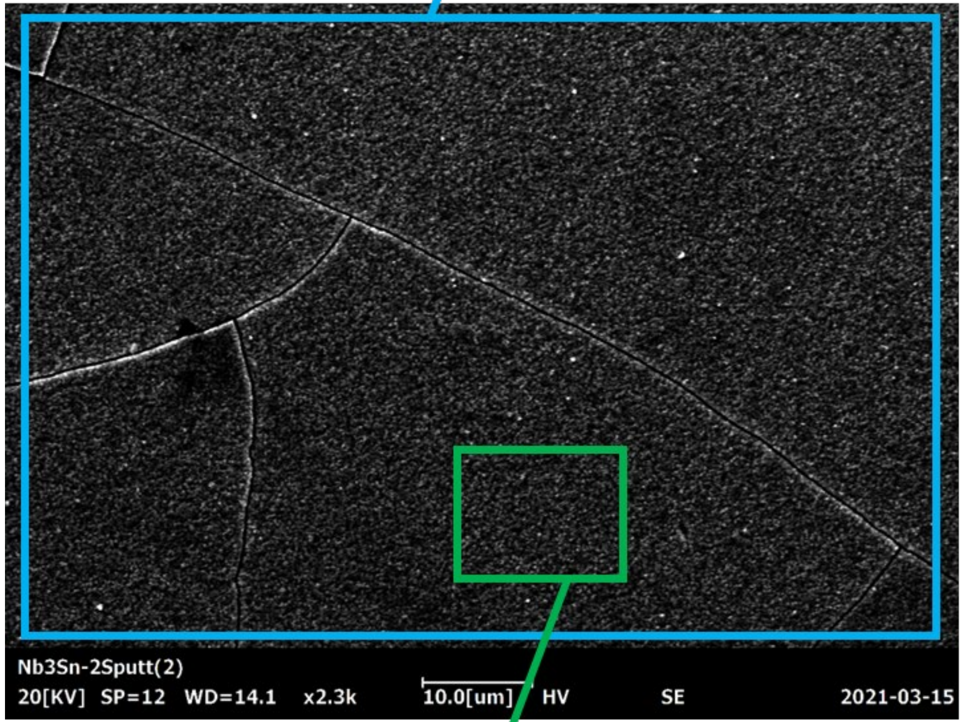
- Process stopped when V started to decrease

First sputtering test on Quartz



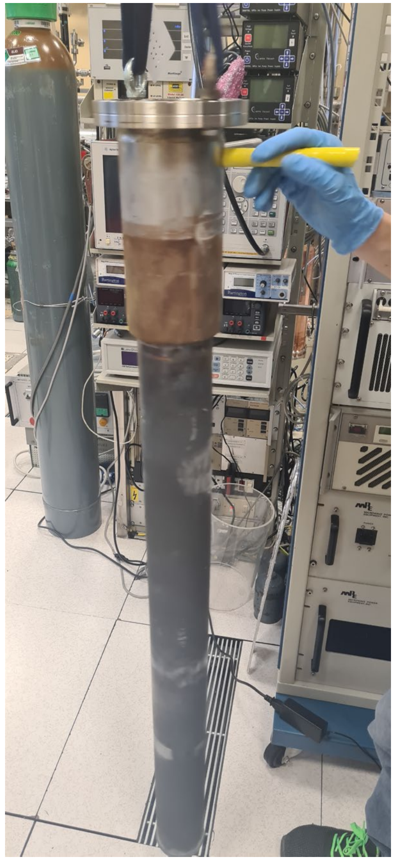
- **Good composition**
- **No Cr** visible by EDS
- **Cracks due to Quartz substrate**
- **No Tc measured**
- **2 New targets ready for new tests**

Nb 80% Sn 20%



Nb 74% Sn 26%

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

Conclusions

- Proved the possibility of realizing targets via LTD
- Easy modulation of target thickness and Tin content

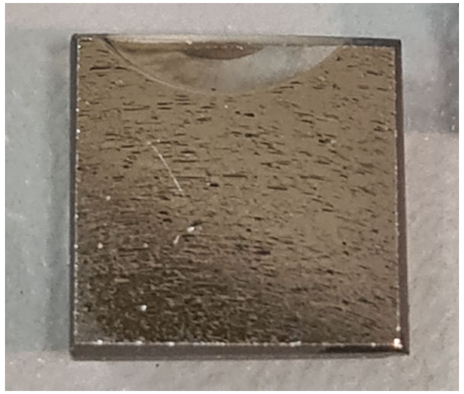
Two main aspect to optimize:

Chromium poisoning

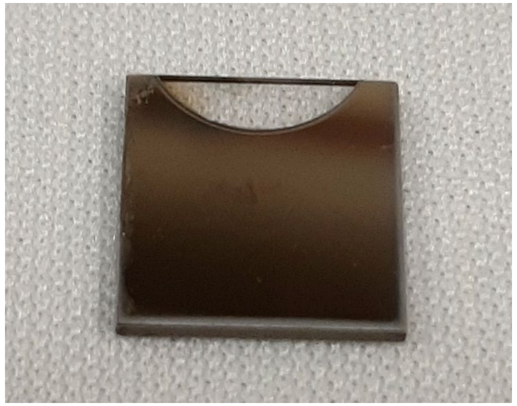
Study the magnetron sputtering deposition parameters

in progress with commercial sintered Nb_3Sn targets

Take home message

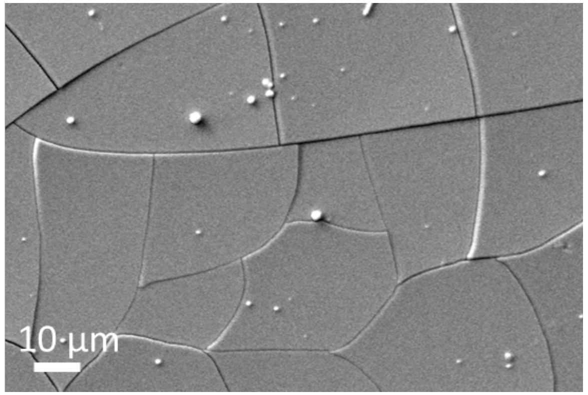


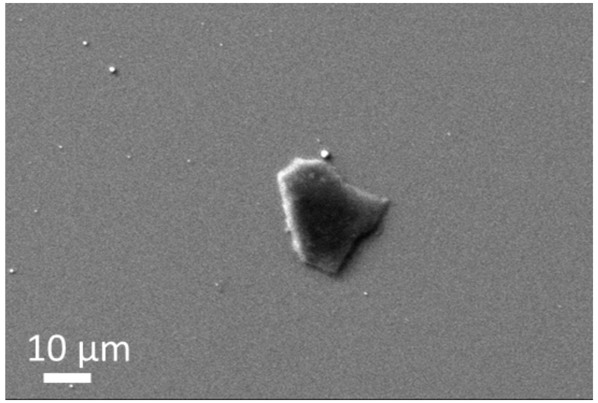
Quartz

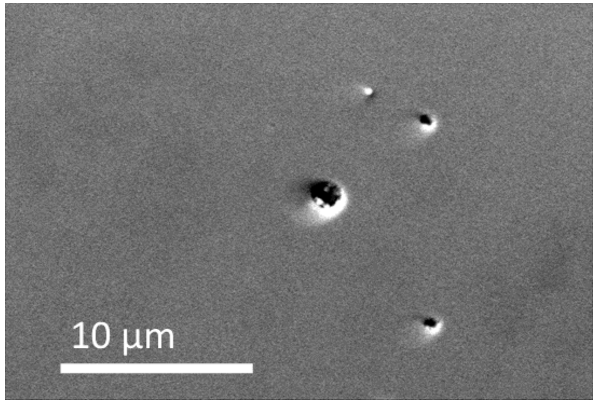


Sapphire

Copper







Do not use quartz for temperatures above 550 C!

Thanks to the LNL INFN team:

Alessandro Salmaso, Davide Ford, Luca Torrassa, Eduard Chyhyrynets, Fabrizio Stivanello, Giorgio Keppel, Oscar Azzolini

Special thanks to **Vanessa Garcia Diaz**

Open postdoc position @LNL:
(deadline October 30th)

https://l.infn.it/job

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INFN

Thanks for your attention

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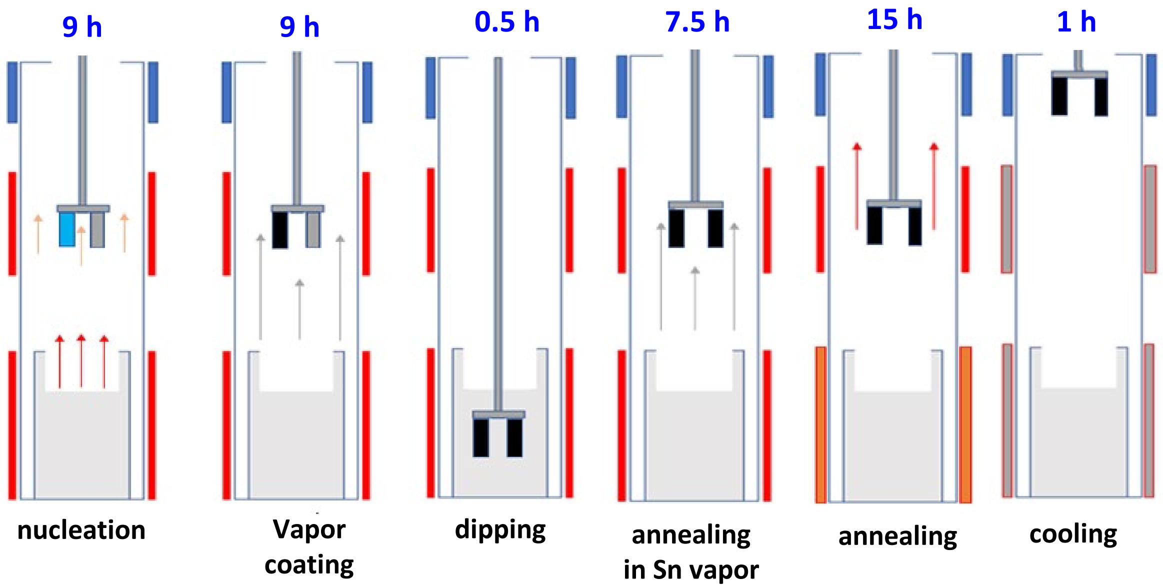
Backup slides

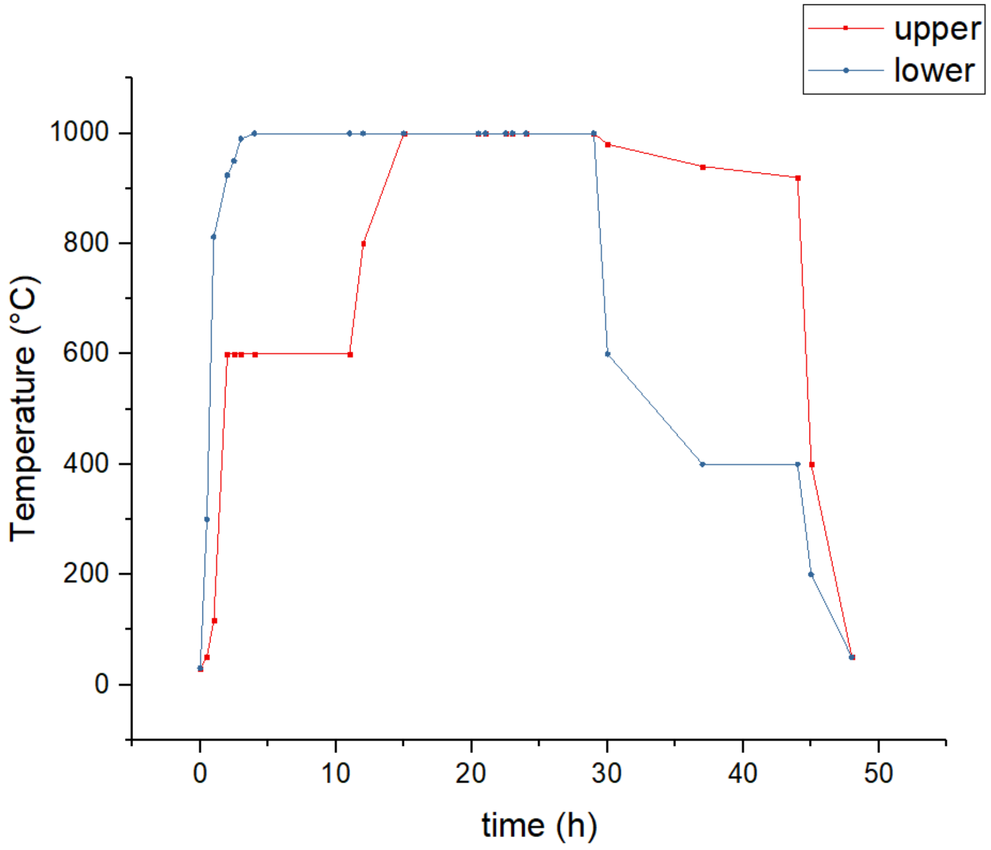
How does the new process work in RF?

How does the new process work in RF?

Process has been adapted to reduce Nb_3Sn thickness

(5-10 microns as the old cavities)





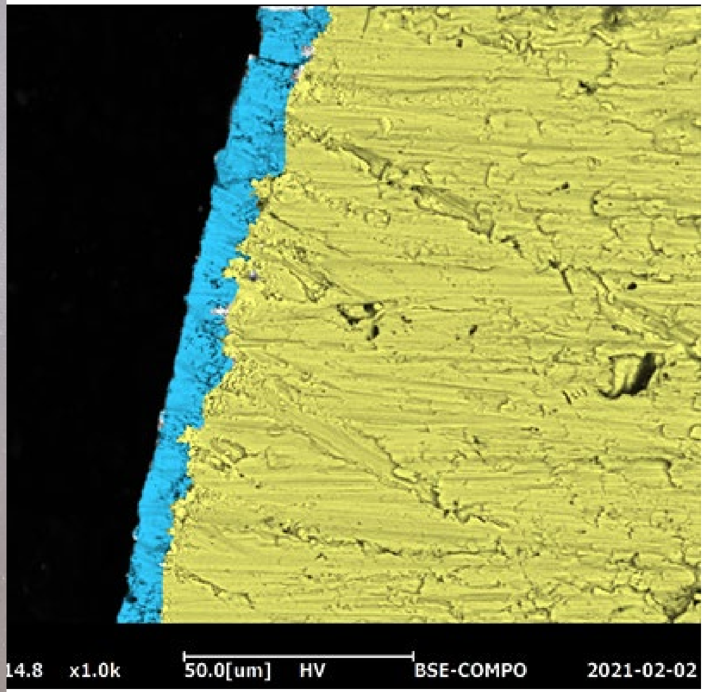
6 GHz cavity #1 (only geometry test)

First test in a broken cavity to test possible geometry effect

No tin drops!

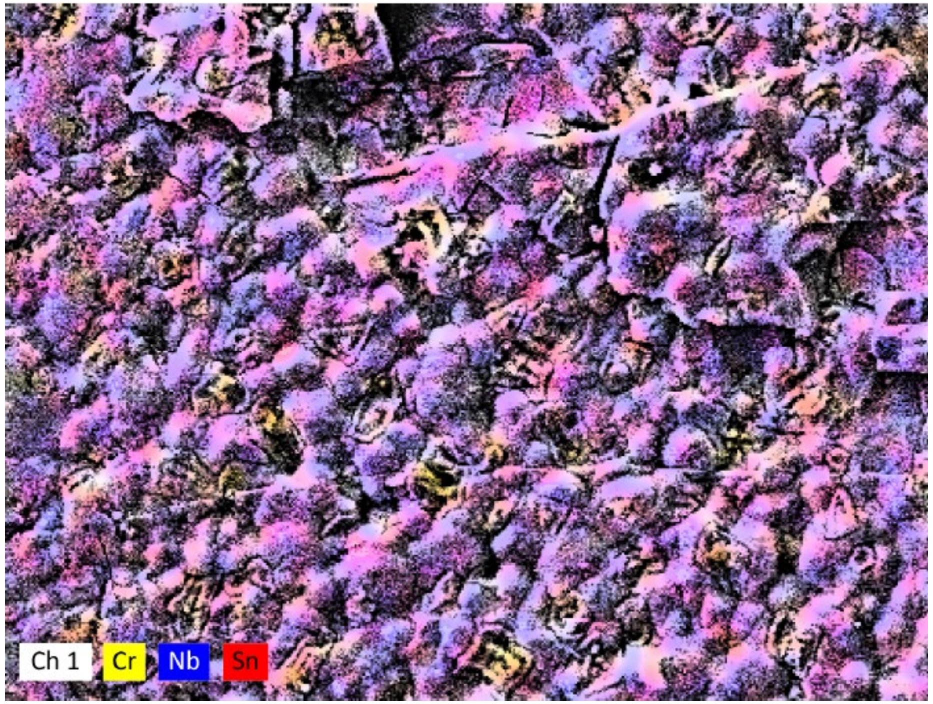
Very good surface

5-10 micron Nb_3Sn thickness



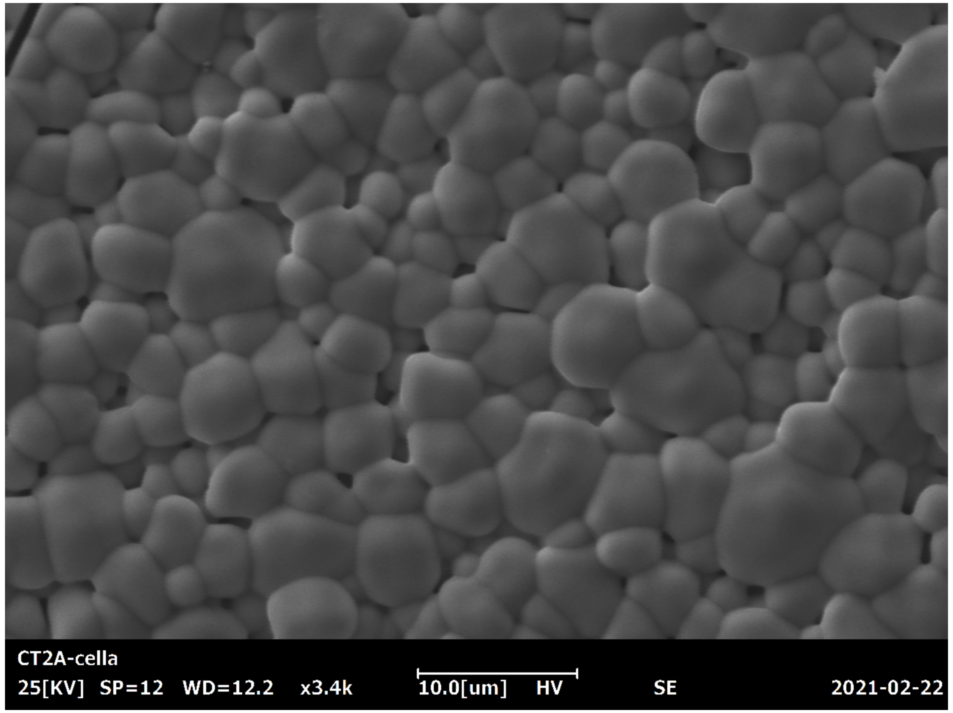
Cr-Ni contamination in 6 GHz cavity

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



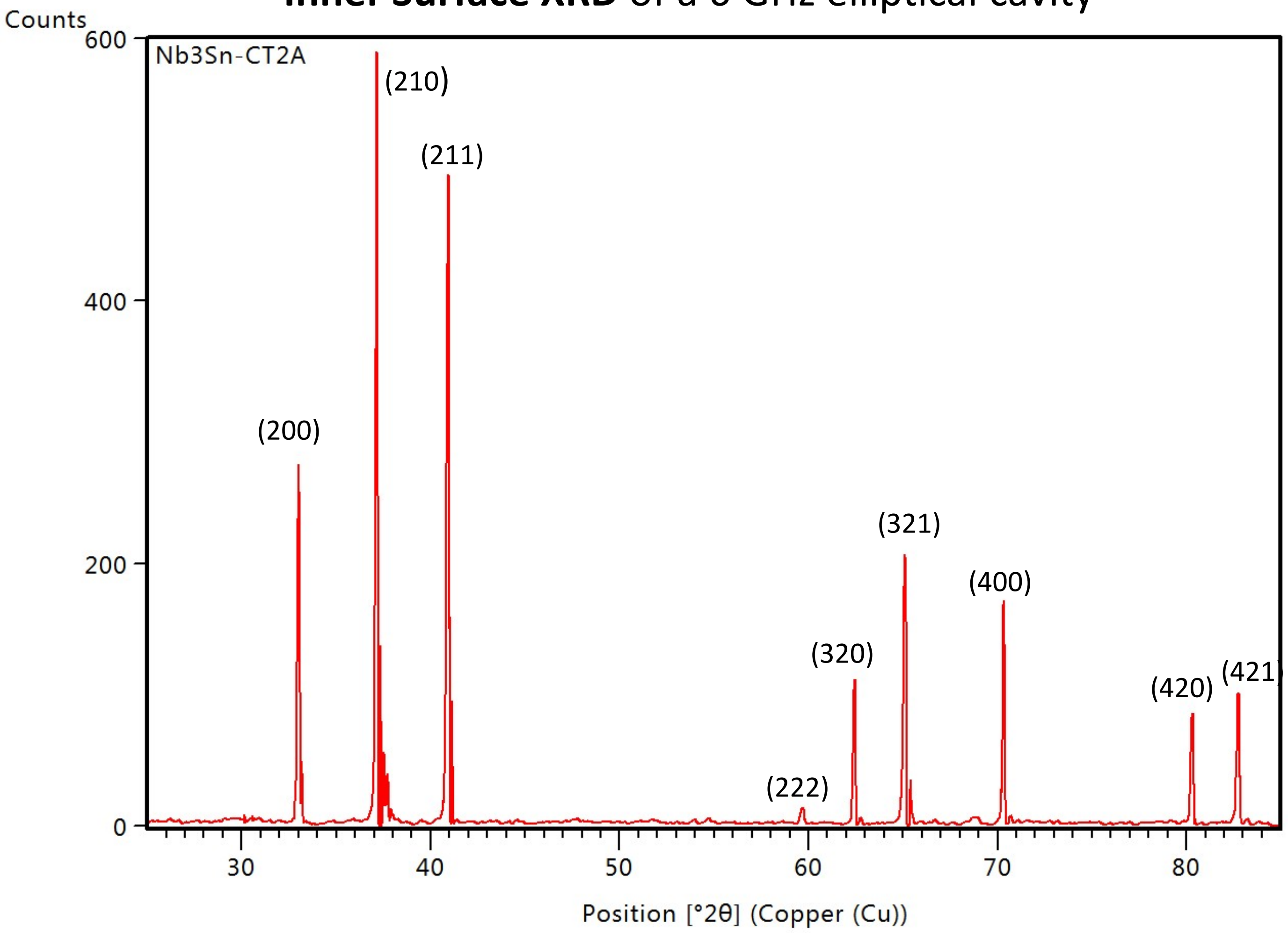
Nb 65% **Sn 35%** **Cr 0%**

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**

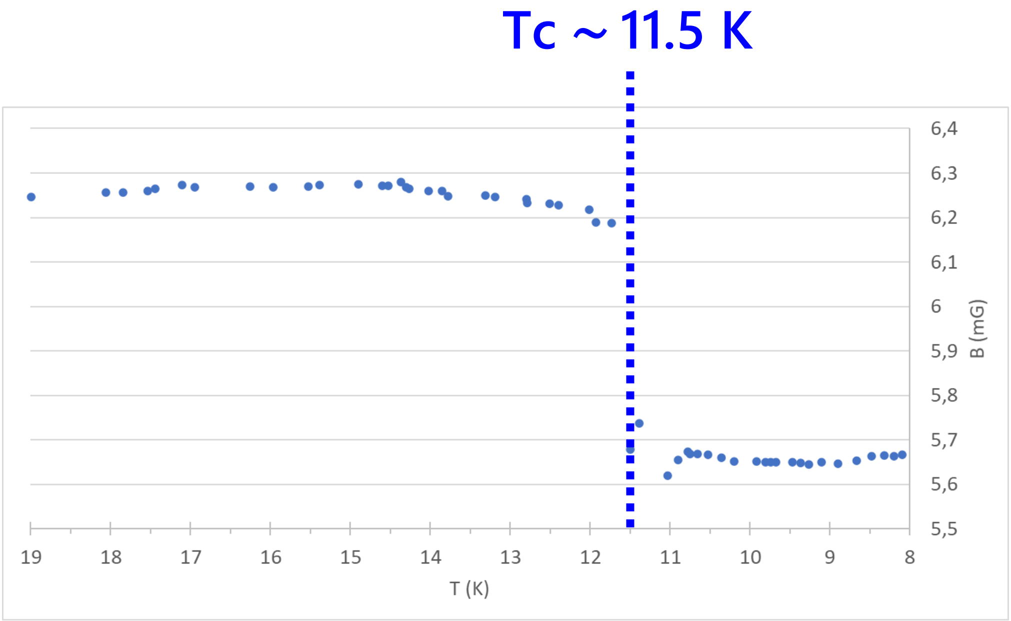
45 h of annealing instead of 15 h

Metallic tin outside

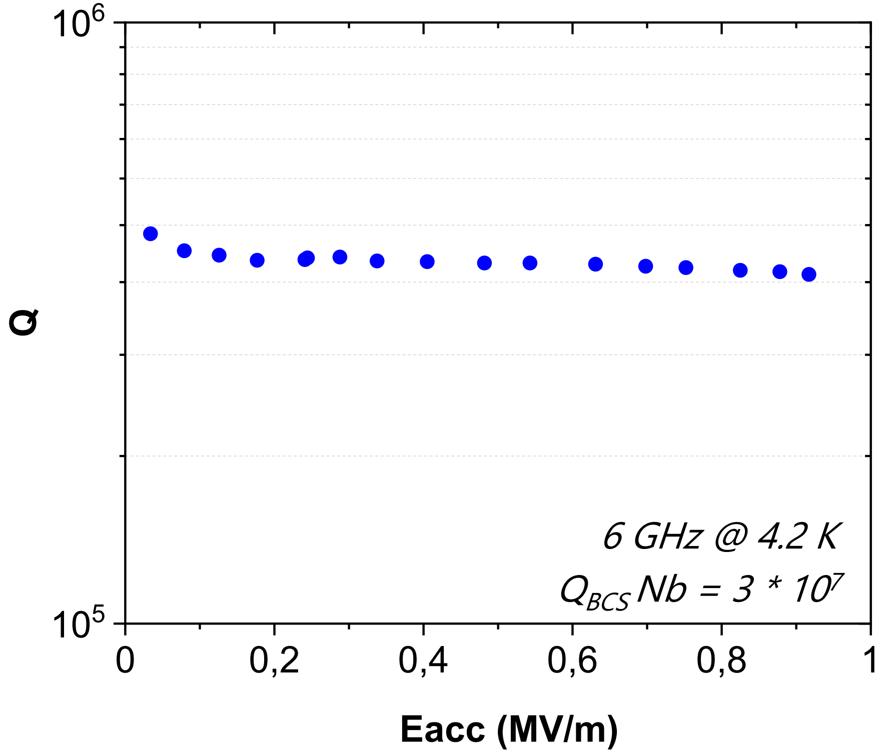
Dark spots in the inner surface

(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? Thick film?)