THIN FILMS DEPOSITION OF Nb_3Sn ON COPPER

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OUTLINE

- Motivation
- Sample preparation & analysis
- Critical temperature
- Microstructural properties of the films
- Substrate choice
- Summary

MOTIVATION

Proposed solutions to achieve desired characteristics and operational cost reduction for SRF application using A15 materials:

- **Replace expensive Nb bulk cavities with coated copper ones**
  - ✓ **Copper cavities** offer high thermal conductivity at low temperature, which should greatly help to increase the stability of the cavity against breakdown.
  - ✓ **Nb coated copper cavities** are successfully **used in CERN** (LEP, LHC and HIE-ISOLDE machines).
- **Replace Nb thin films with superconductor with superior parameters (A15 intermetallic compound)**

WHY Nb_3Sn?

Advantages

- **High critical temperature**

T_c	Nb_3Sn ~ 18.3 K
	Nb ~ 9.3 K

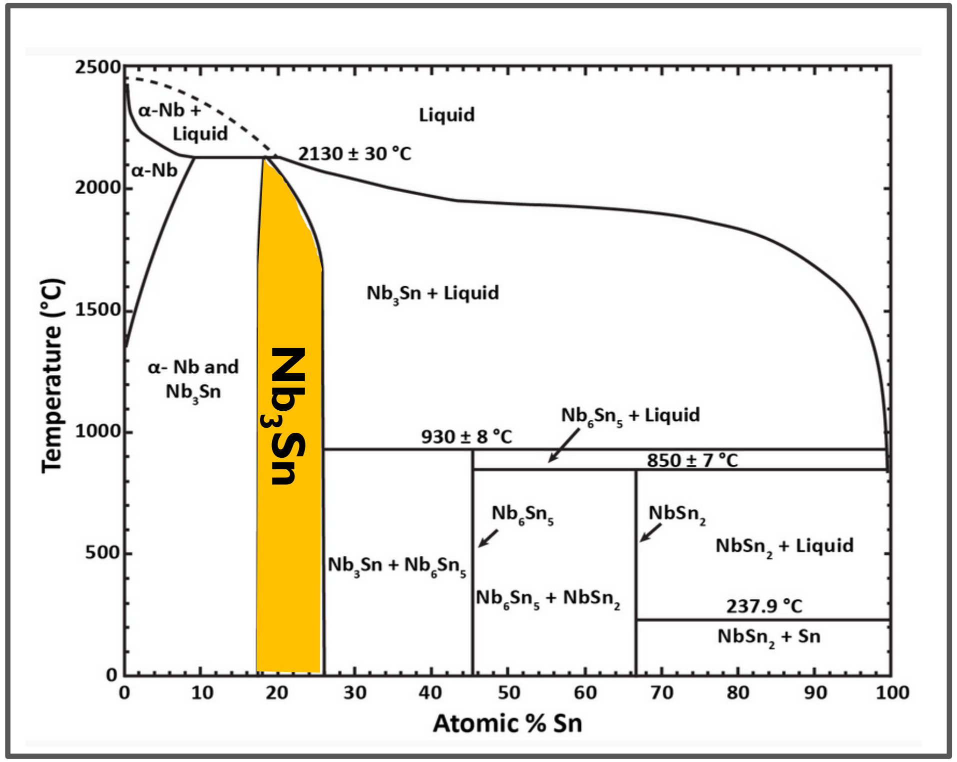
- **Low BCS resistance**

R_{BCS} @ 4.2K and 500MHz	Nb_3Sn ~ 0.4 nΩ
	Nb ~ 45 nΩ

Challenges

- Stoichiometry control (*Sn* At% 19-26 %)
- Requires high temperature treatment
- Limited range of annealing temperatures
- Substrate importance

WHY Nb_3Sn?



Binary phase diagram of the Nb-Sn system [1]

[1] J. Charlesworth, I. MacPhail, and P. Madsen, J. Mater. Sci. 5, 580 (1970).

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	Nb ~ 9.3 K

- **Low BCS resistance**

R_{BCS} @ 4.2K and 500MHz	Nb_3Sn ~ 0.4 nΩ
	Nb ~ 45 nΩ

Challenges

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- Limited range of annealing temperatures
- Substrate importance

SAMPLE PREPARATION BY MAGNETRON SPUTTERING

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Reacted **After** Coating

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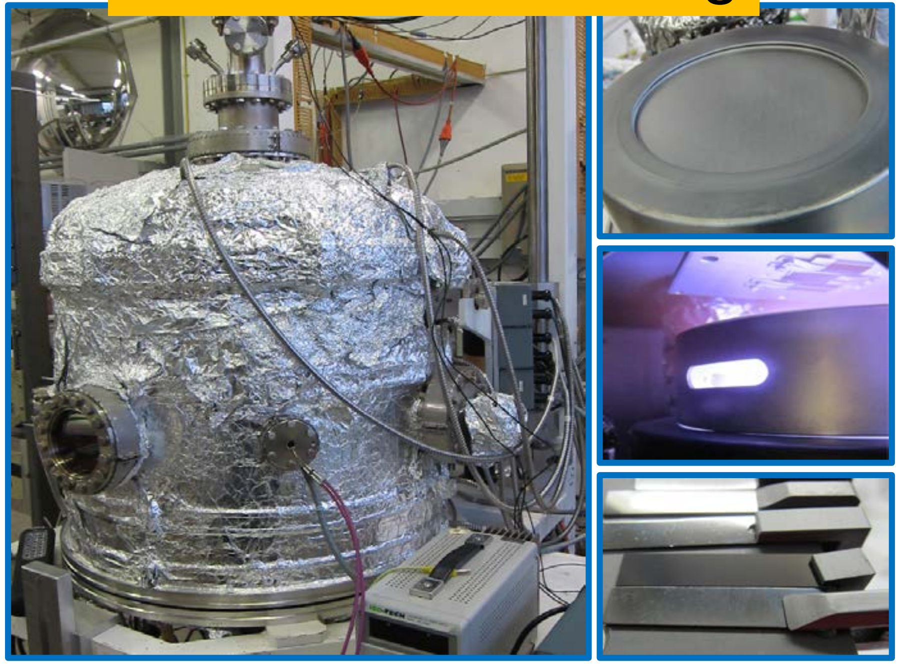

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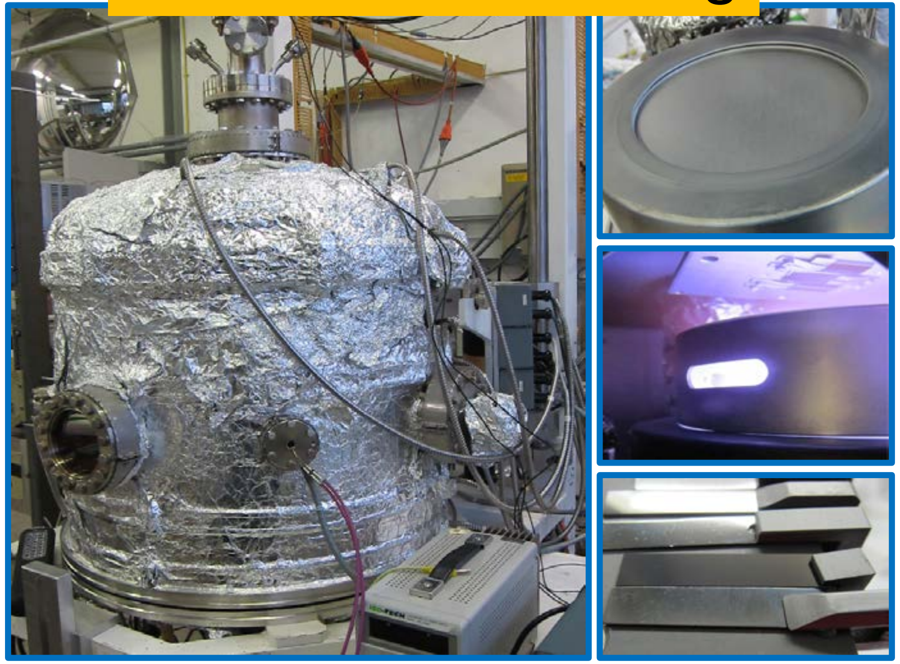

Compulsory Annealing

Annealing temperatures	**600 - 800°C**
Annealing time	24 h… 72 h

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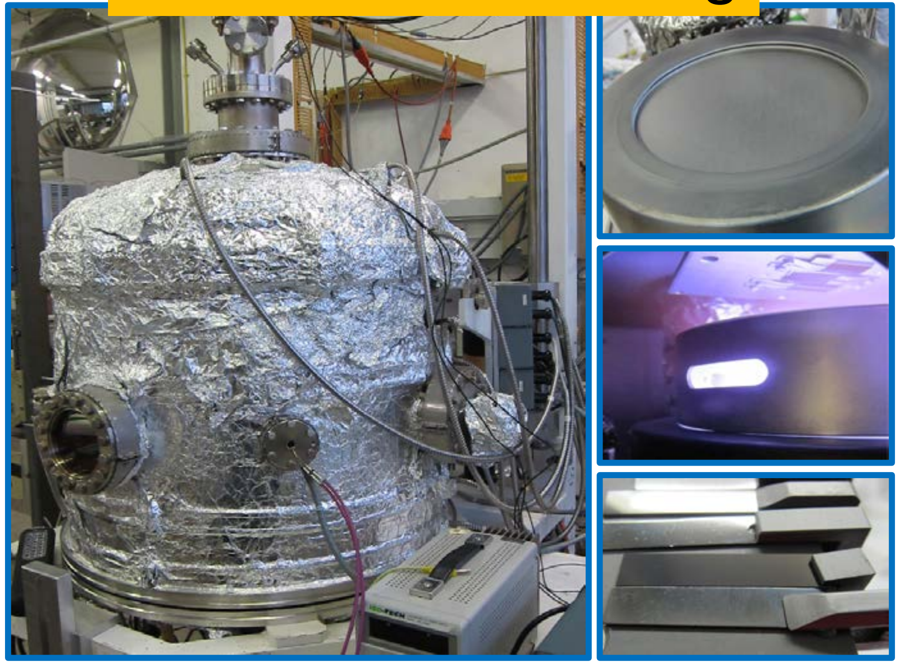

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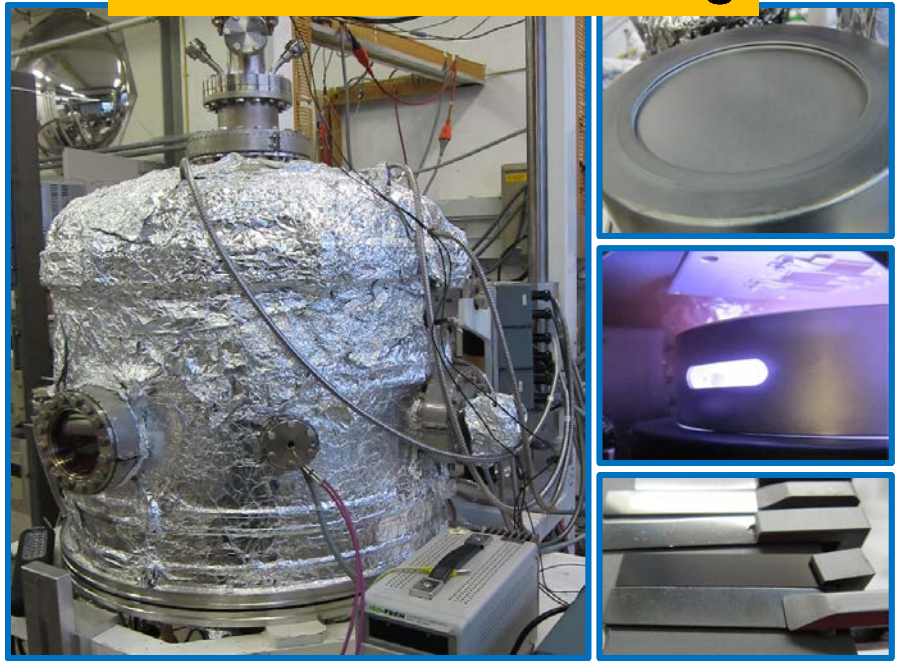

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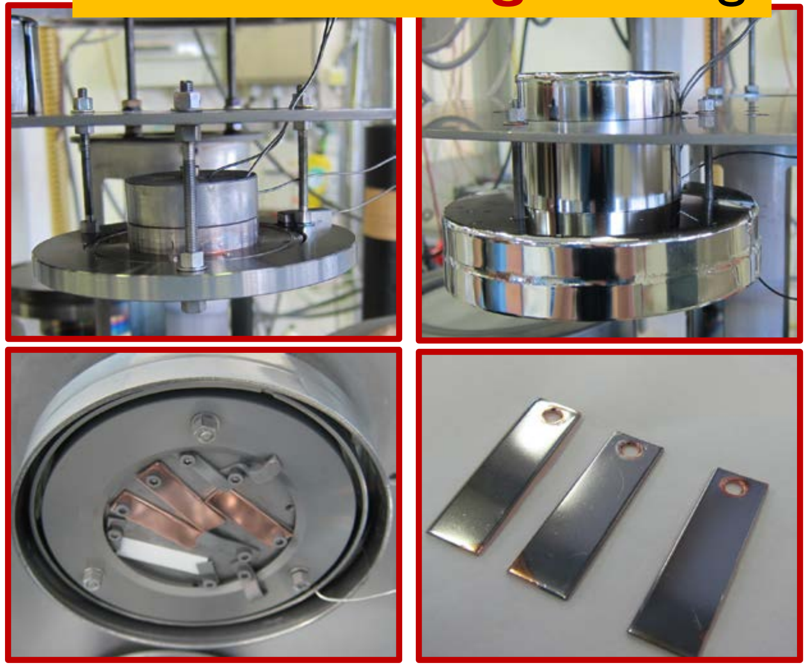

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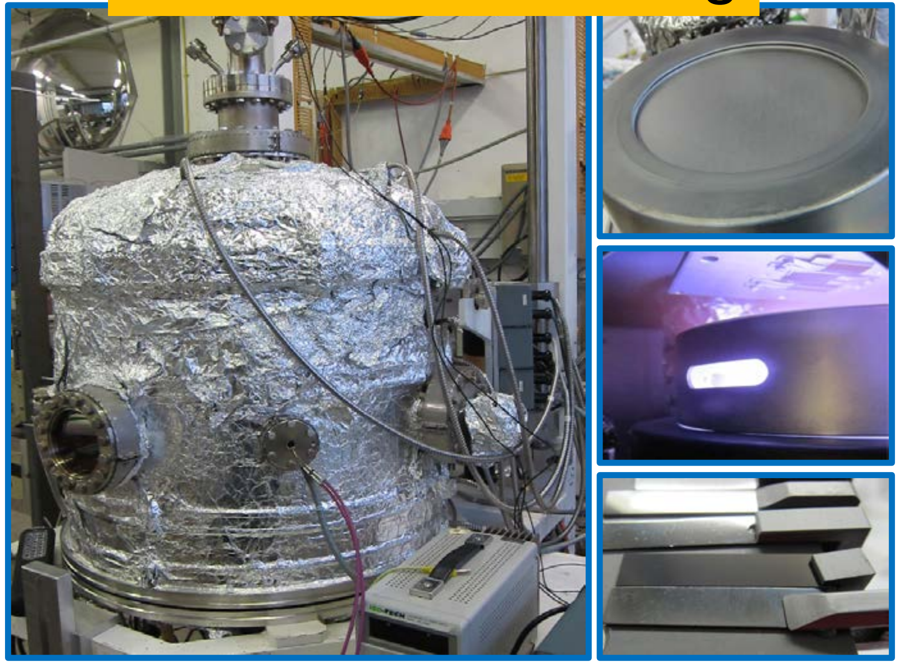

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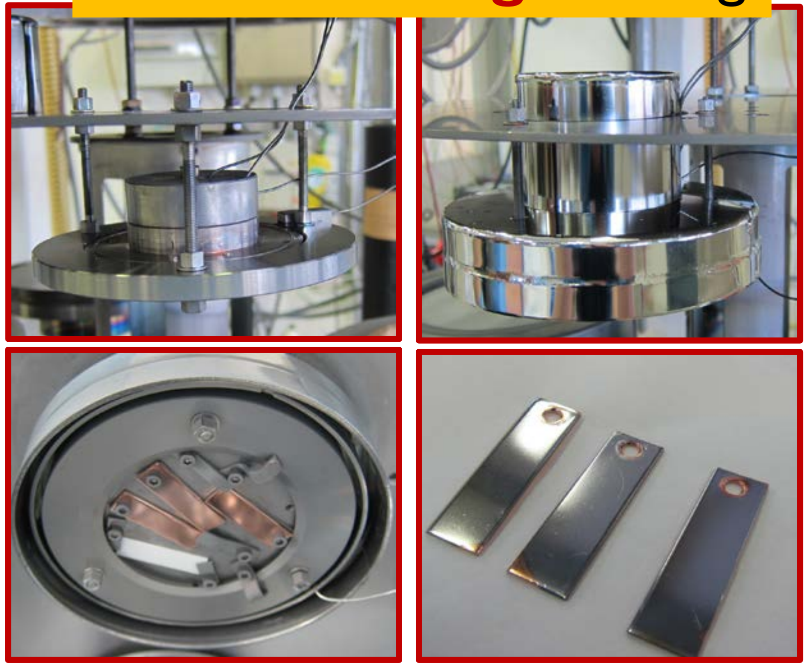

Alternative Annealing

Coating temperatures	600 - 735°C
Alternative Additional Annealing	24 h… 72 h

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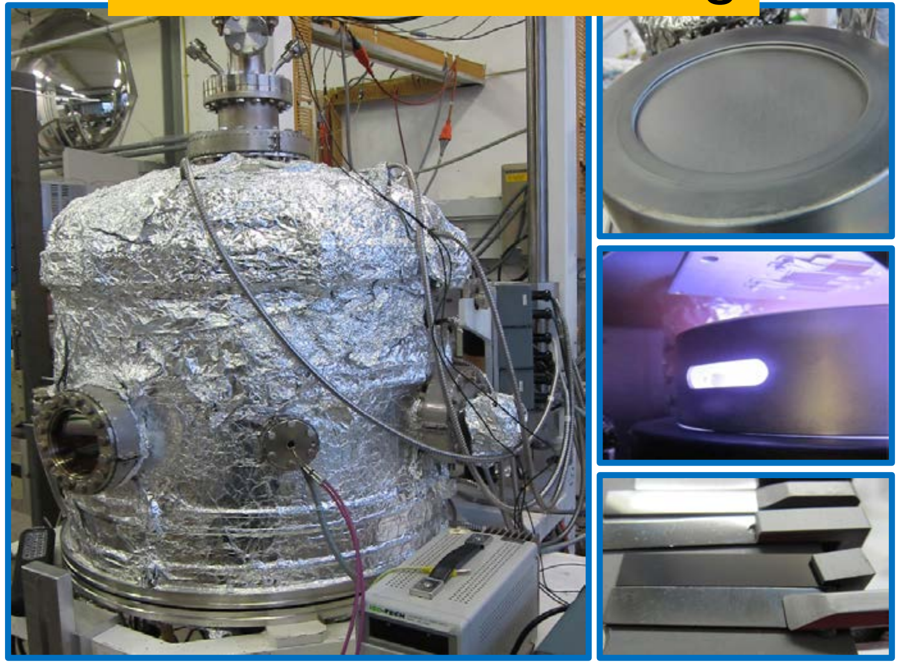

Coating parameters:

Coating gas: Ar or Kr

Coating pressures:
$7 \cdot 10^{-4}$ *mbar* … $5 \cdot 10^{-2}$ *mbar*

Composition:
Sn 20 At% to 27 At%,

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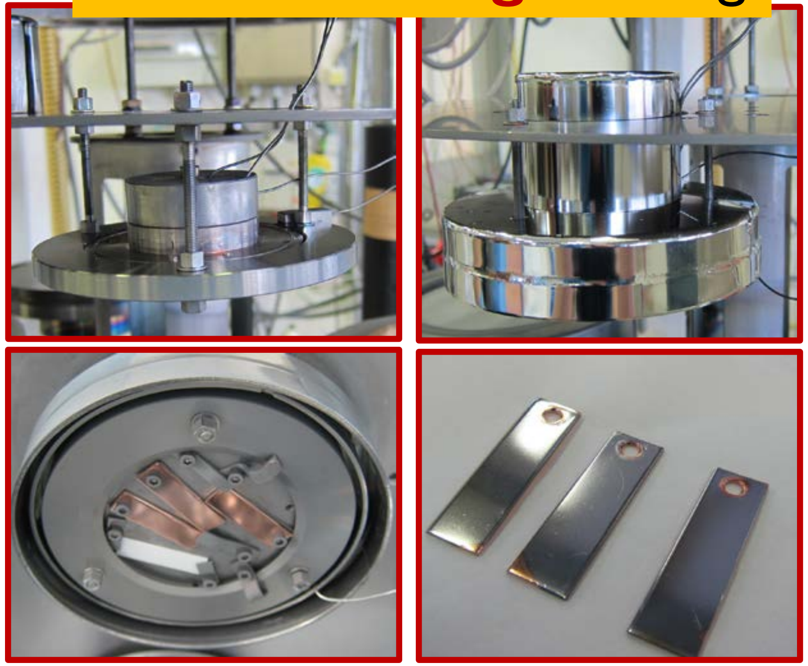

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FILMS REACTED **AFTER** COATING

A15 phase formation. XRD analysis & Morphology

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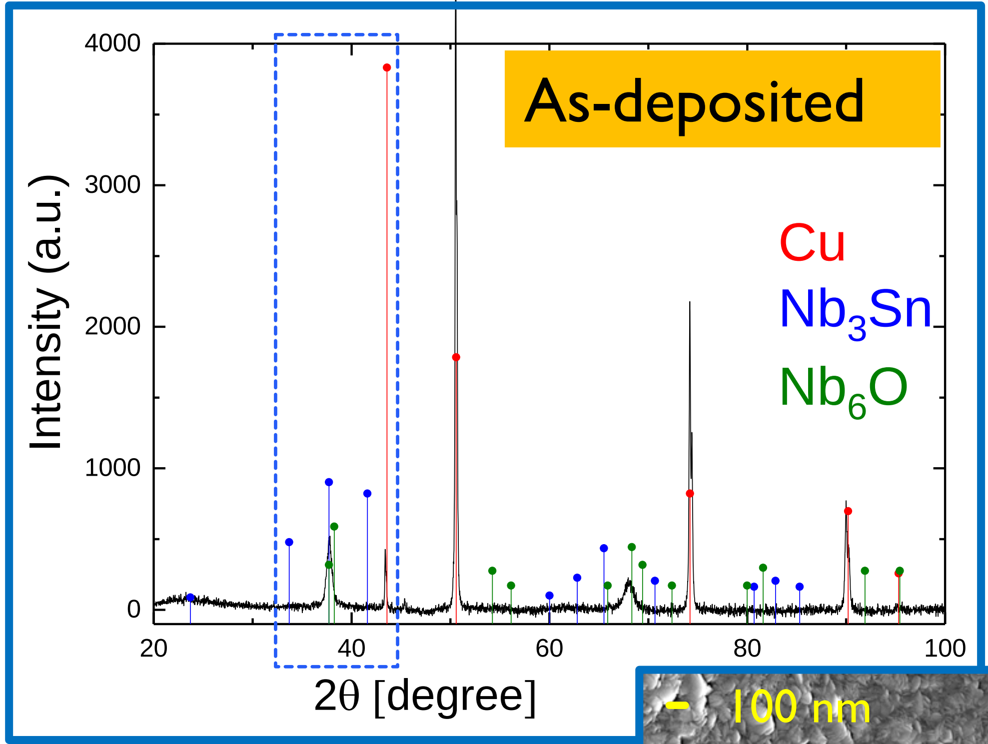

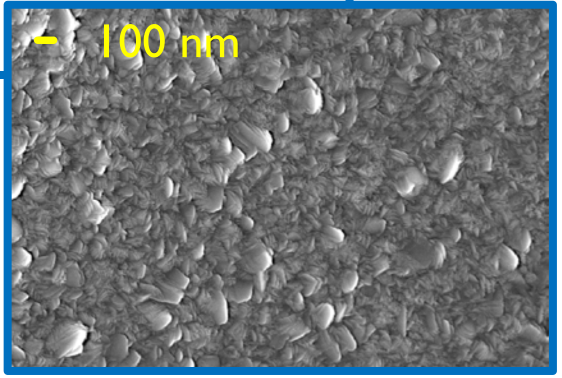

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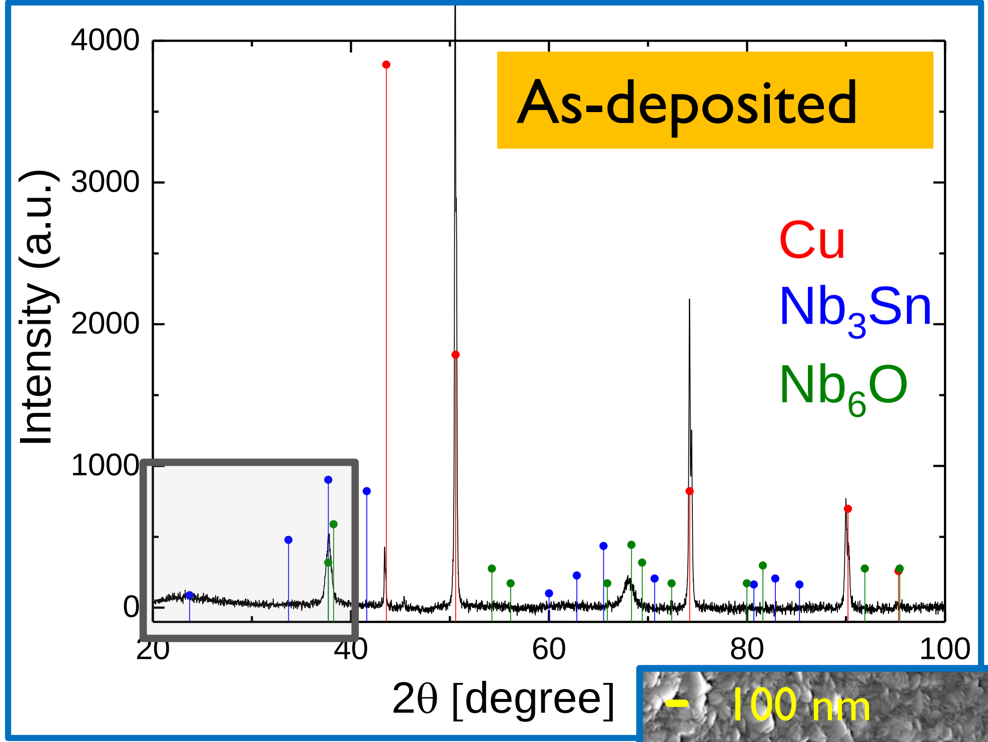

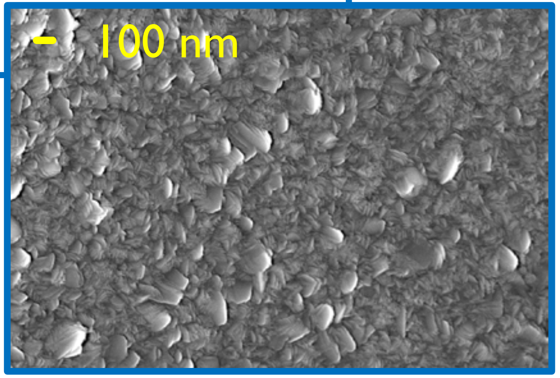

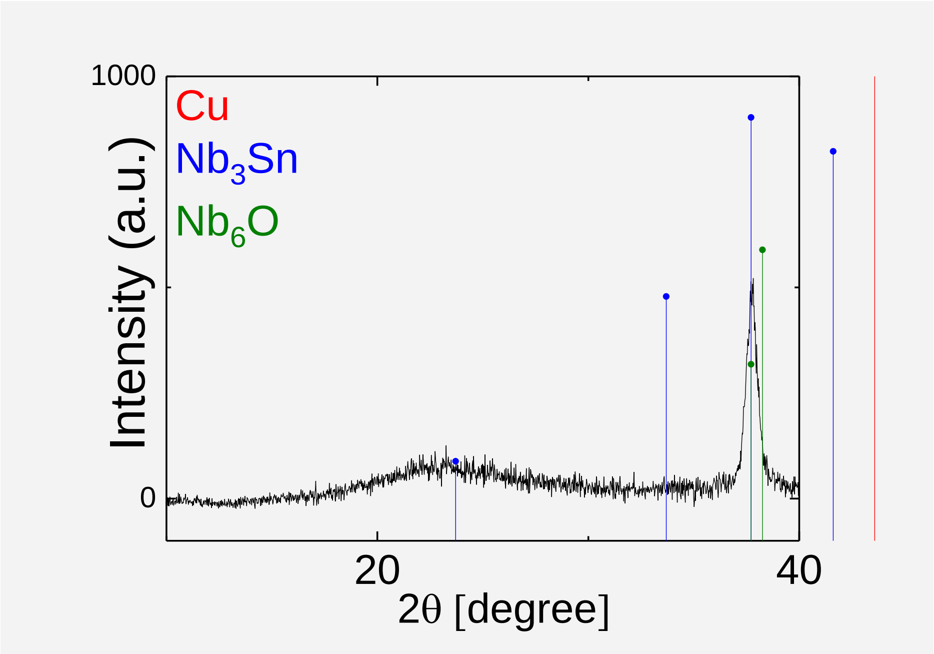

Amorphous / nanocrystalline phase

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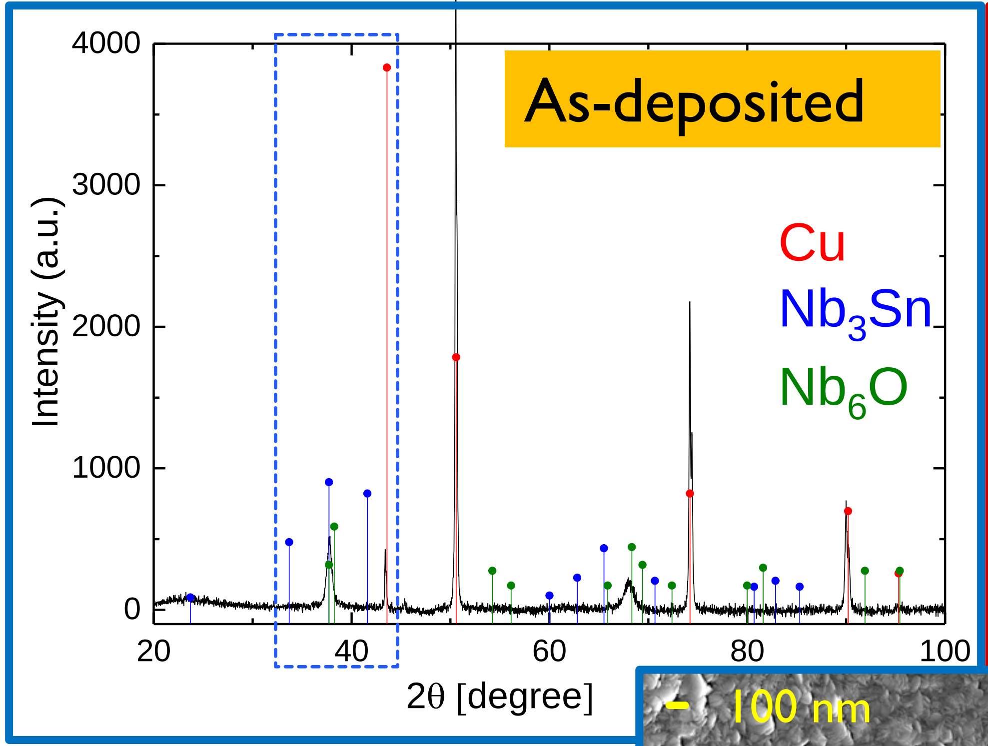

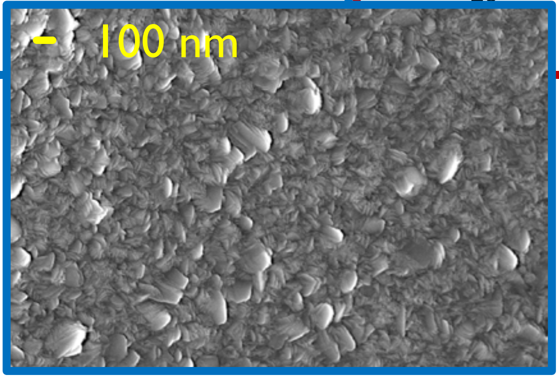

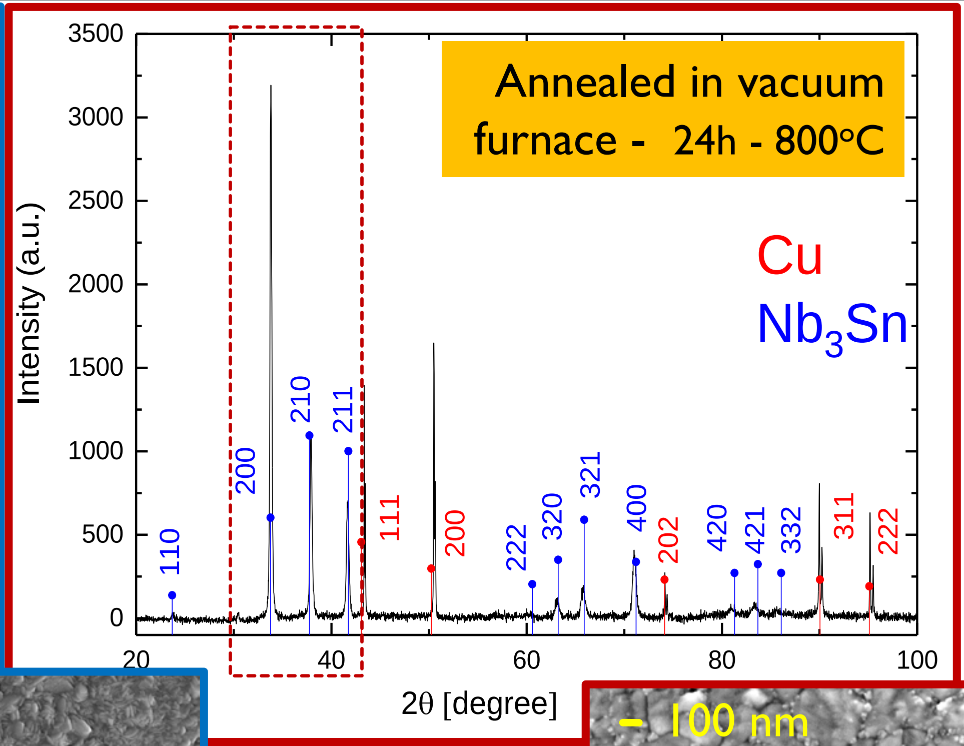

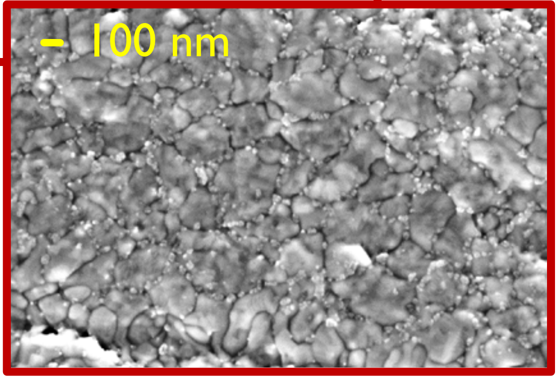

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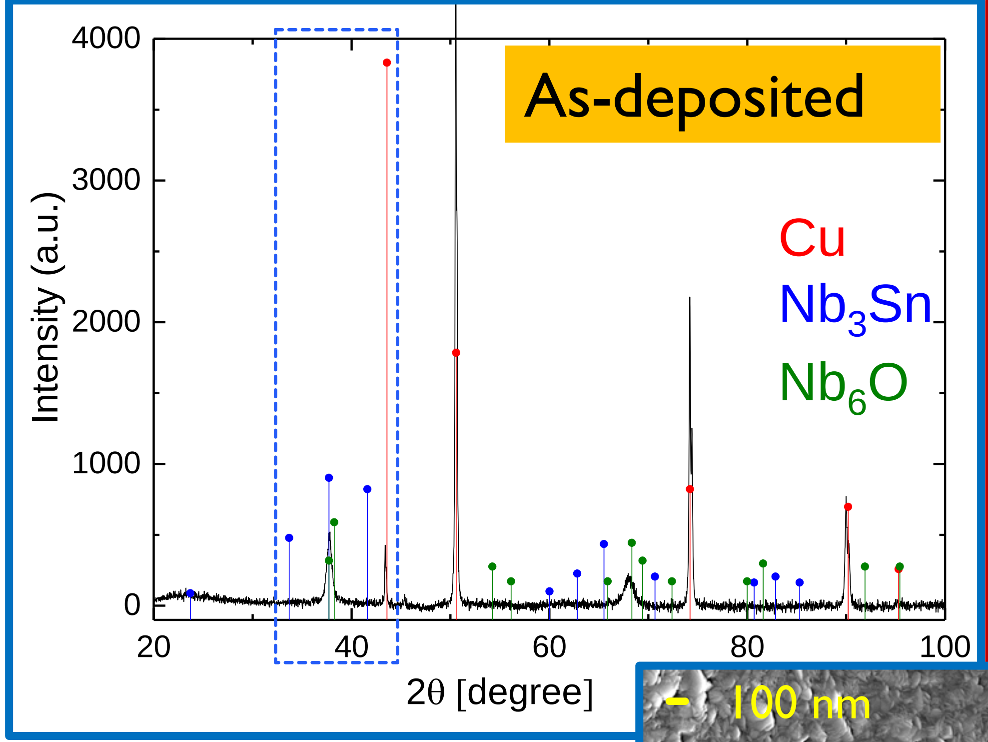
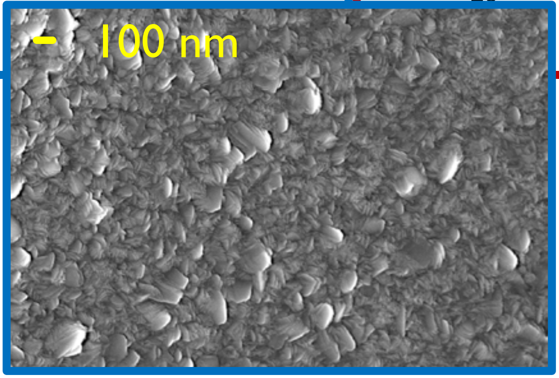
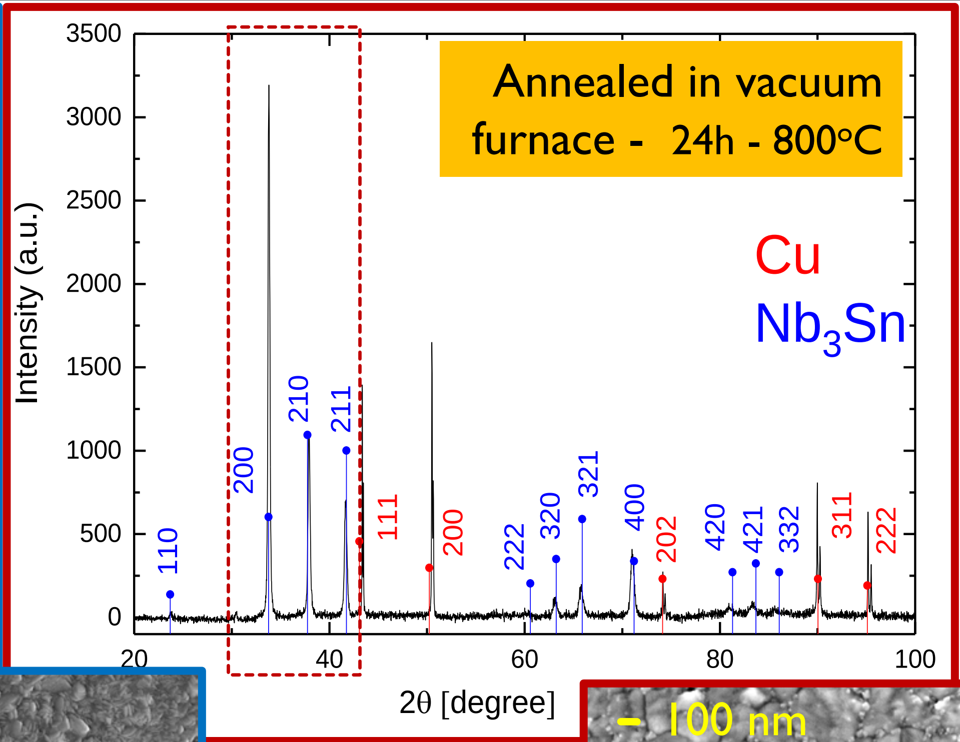
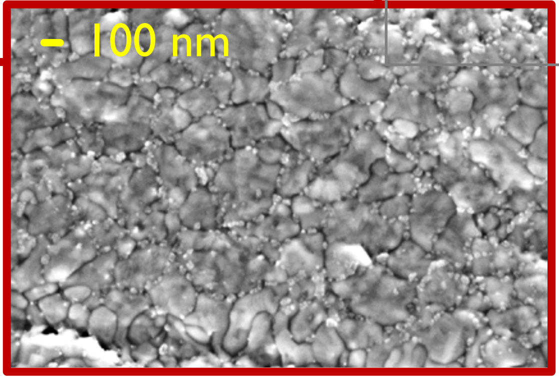






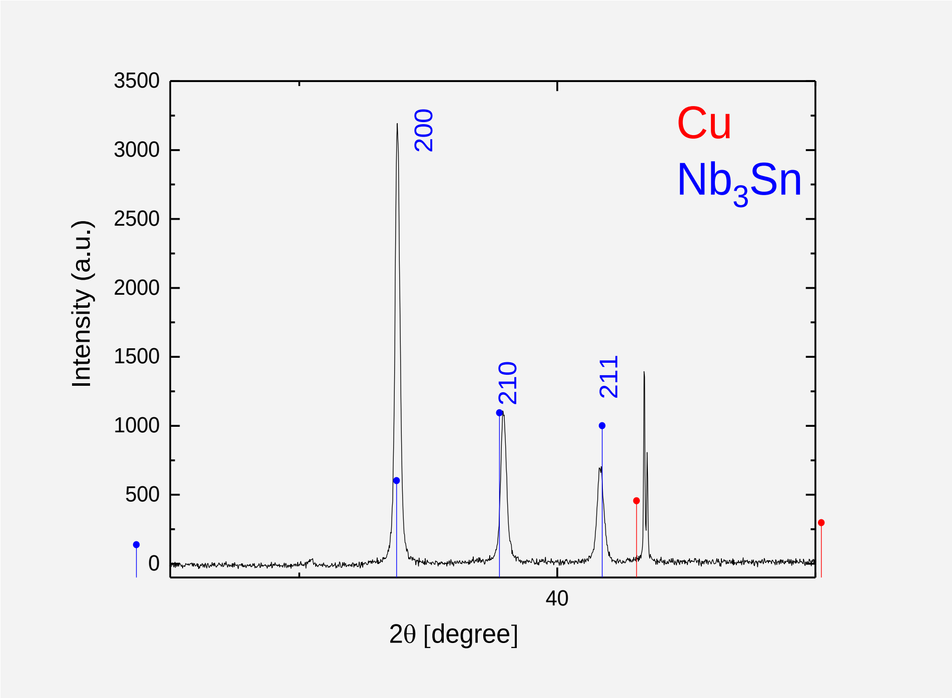


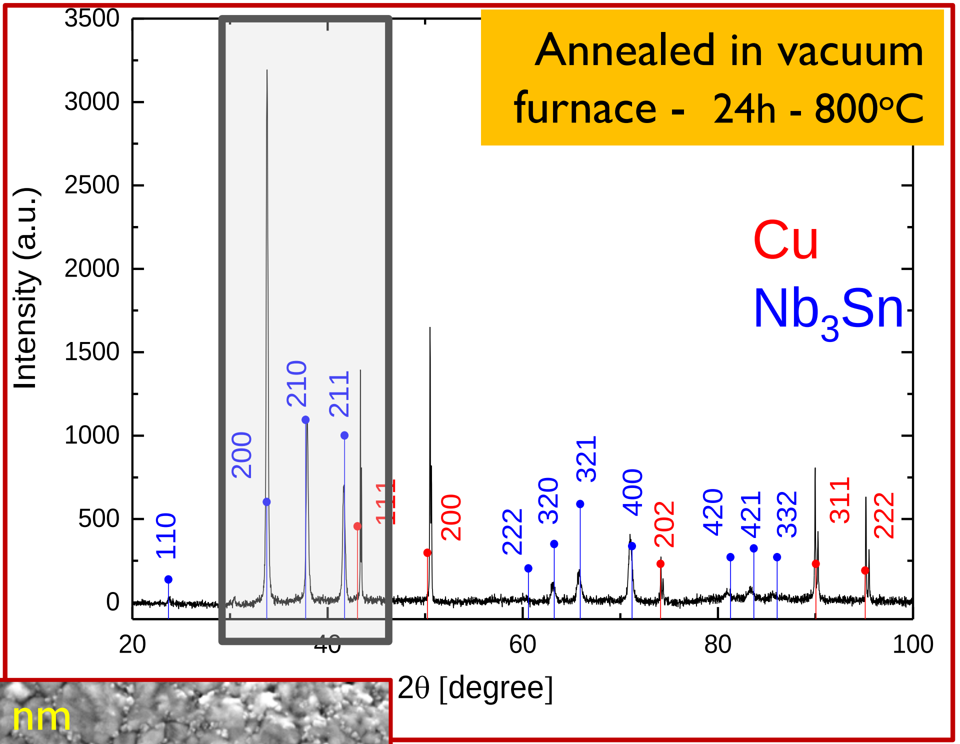
- **A15 phase confirmed** by the presence of Nb_3Sn characteristic peaks of (200), (210), (211) … (332)
- **Absence** of undesired phases ***$NbSn_2$, Nb_6Sn_5***

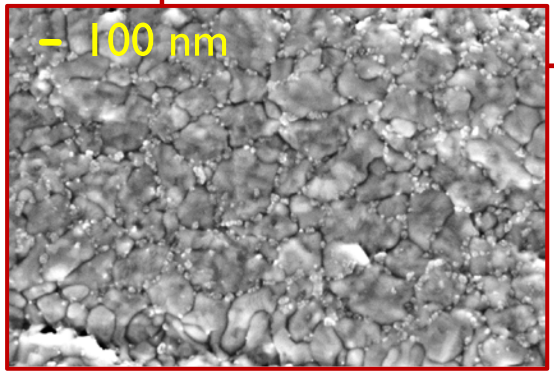
FILMS REACTED **AFTER** COATING

A15 phase formation. XRD analysis & Morphology

Texturation - Preferred orientation of (200)

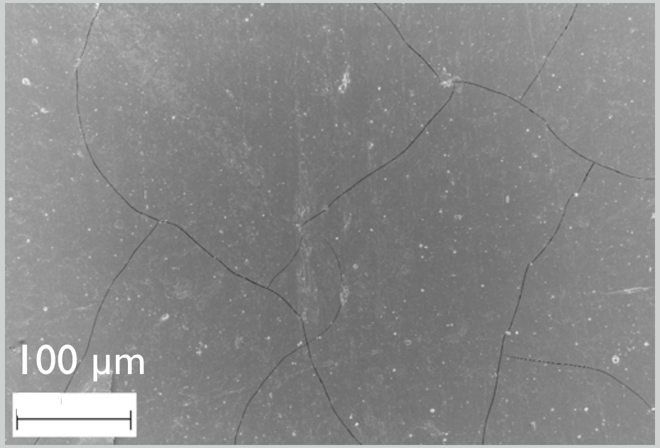




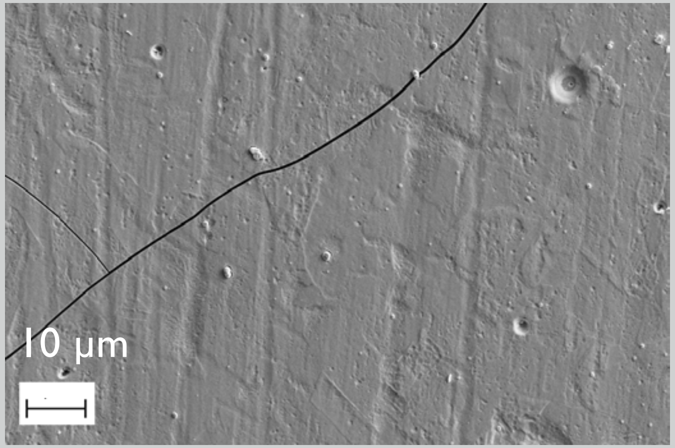


CRACKING OF THE FILMS **AFTER** ANNEALING

$T_{annealing}$ = 700°C



$T_{annealing}$ = 750°C



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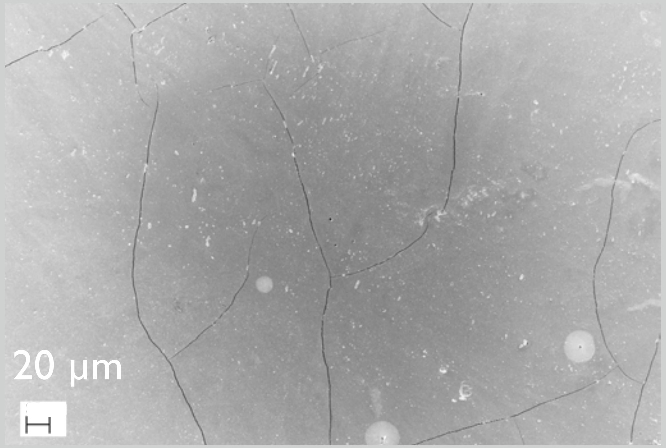
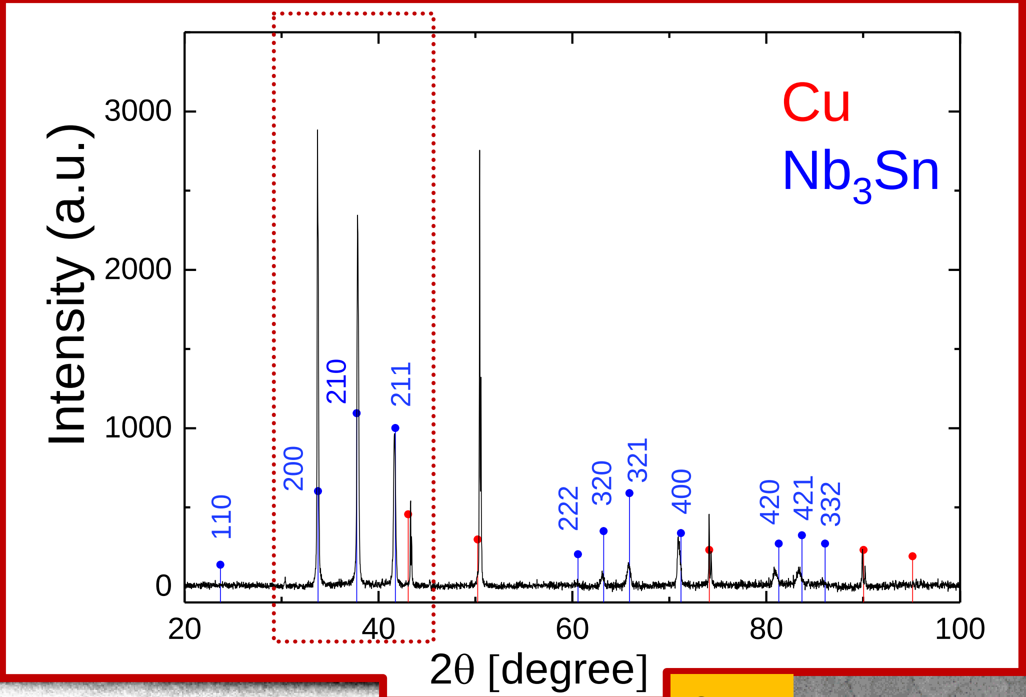
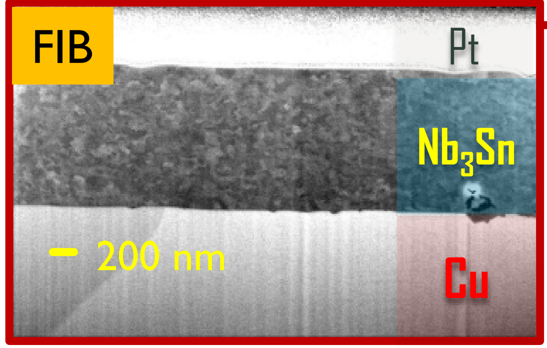
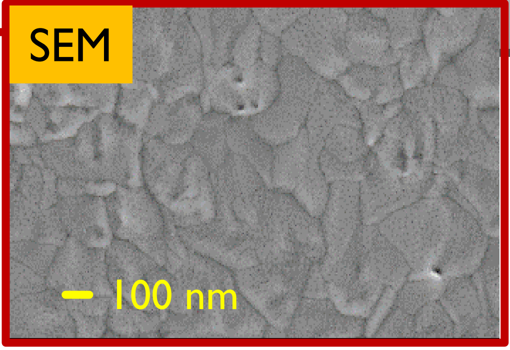


Without solving "cracking" problem not RF compatible

FILMS REACTED **DURING** COATINGS

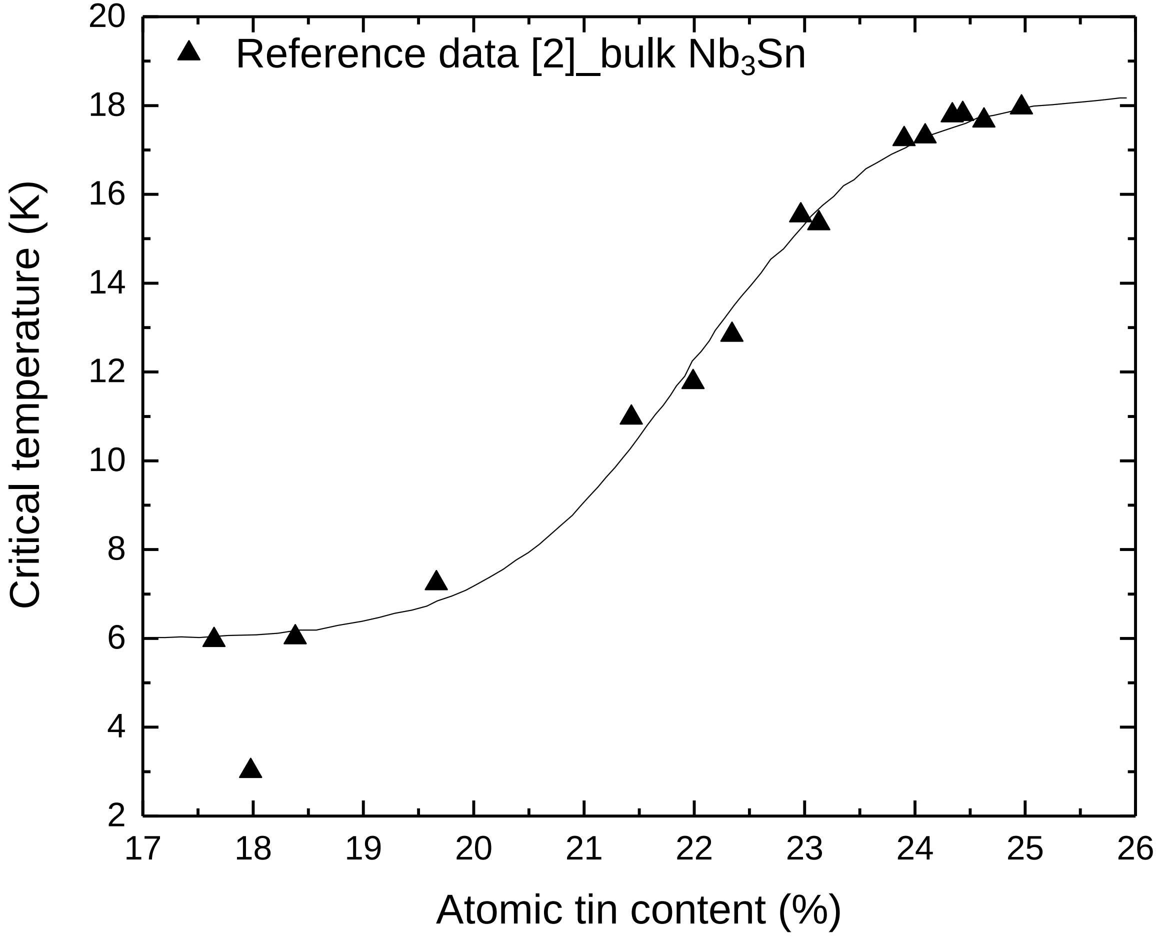






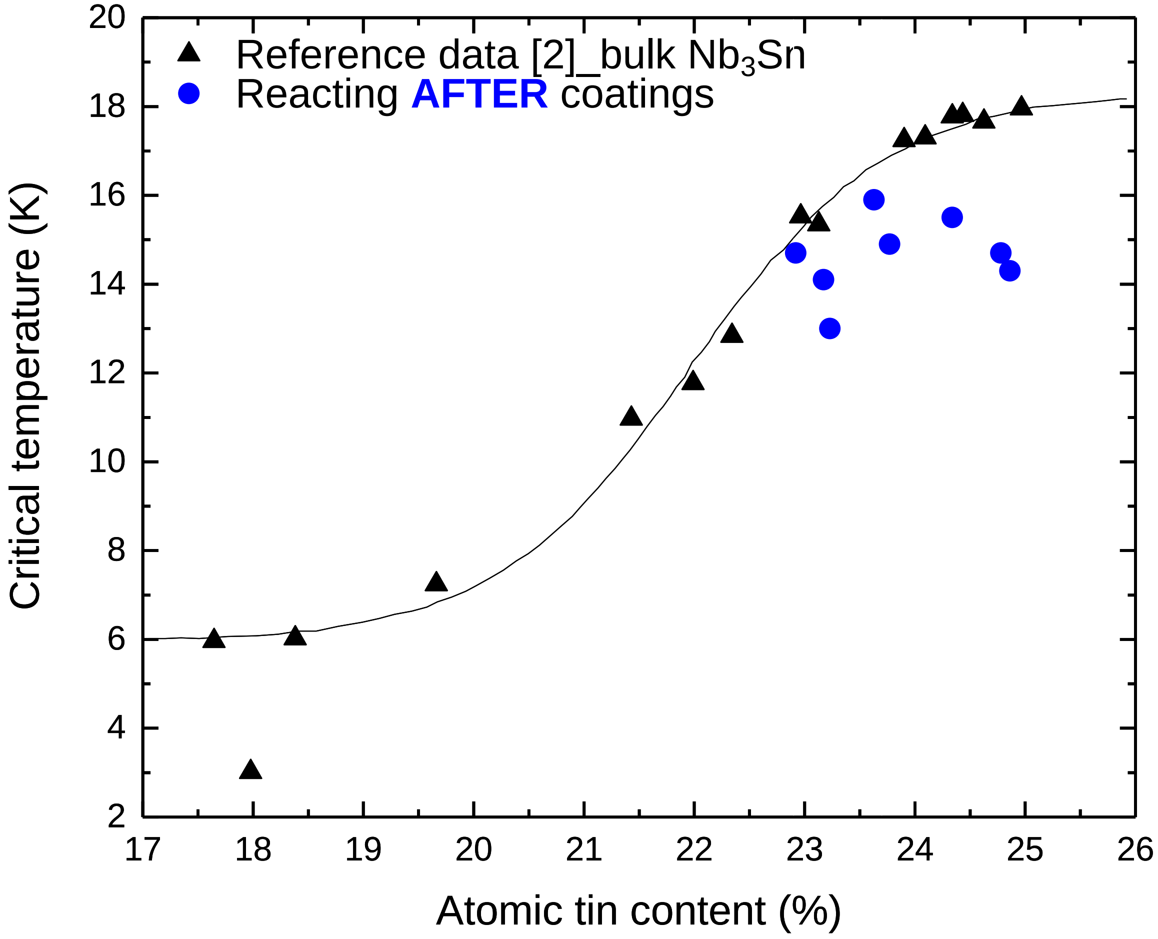
- Characteristics peaks after the coating of (200), (210), (211) … (332) – **Confirmation of the superconducting A15 phase**
- **Absence** of undesired phases ***$NbSn_2$, Nb_6Sn_5***
- **Dense, crack-free morphology**

CRITICAL TEMPERATURE



[2] *A. Godeke. Supercond. Sci. Technol., 19 (2006) R68-R80*

CRITICAL TEMPERATURE



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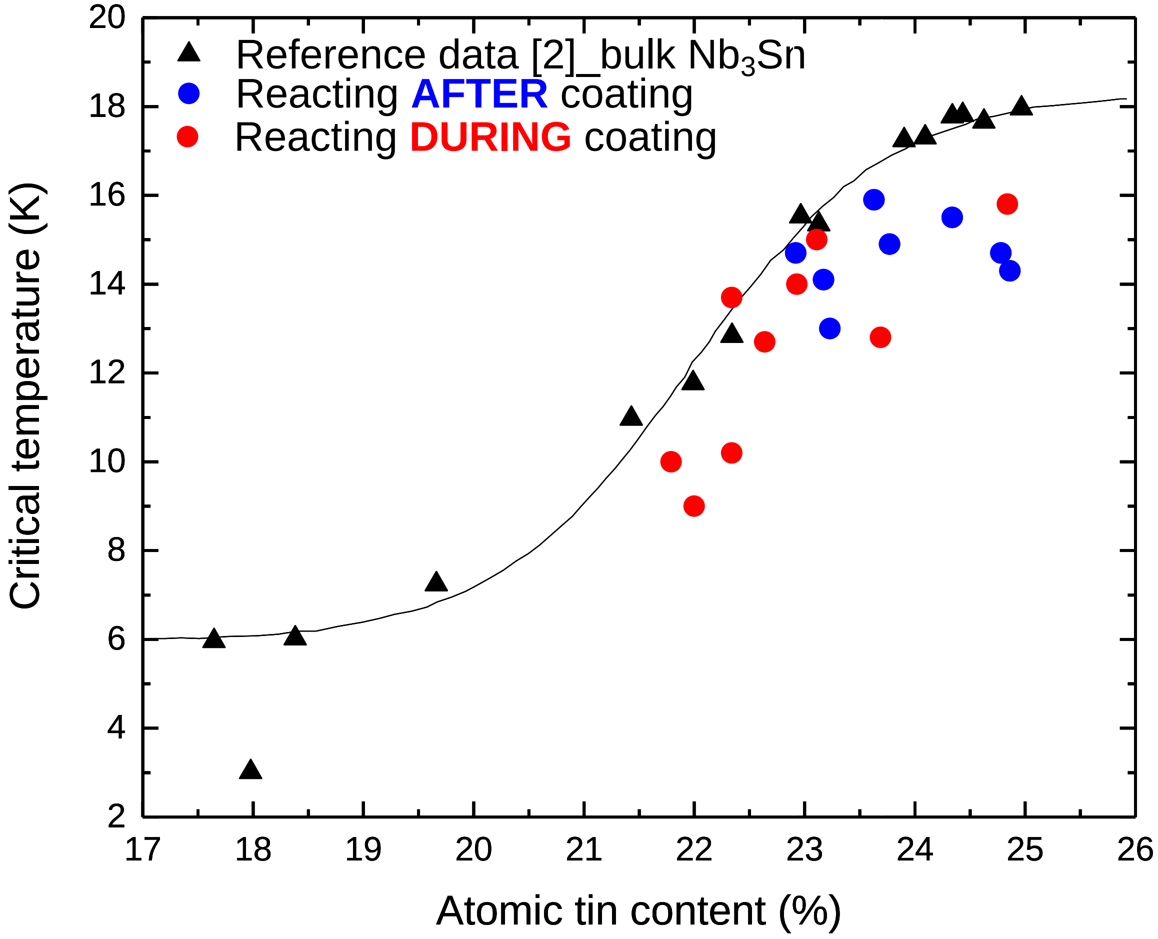

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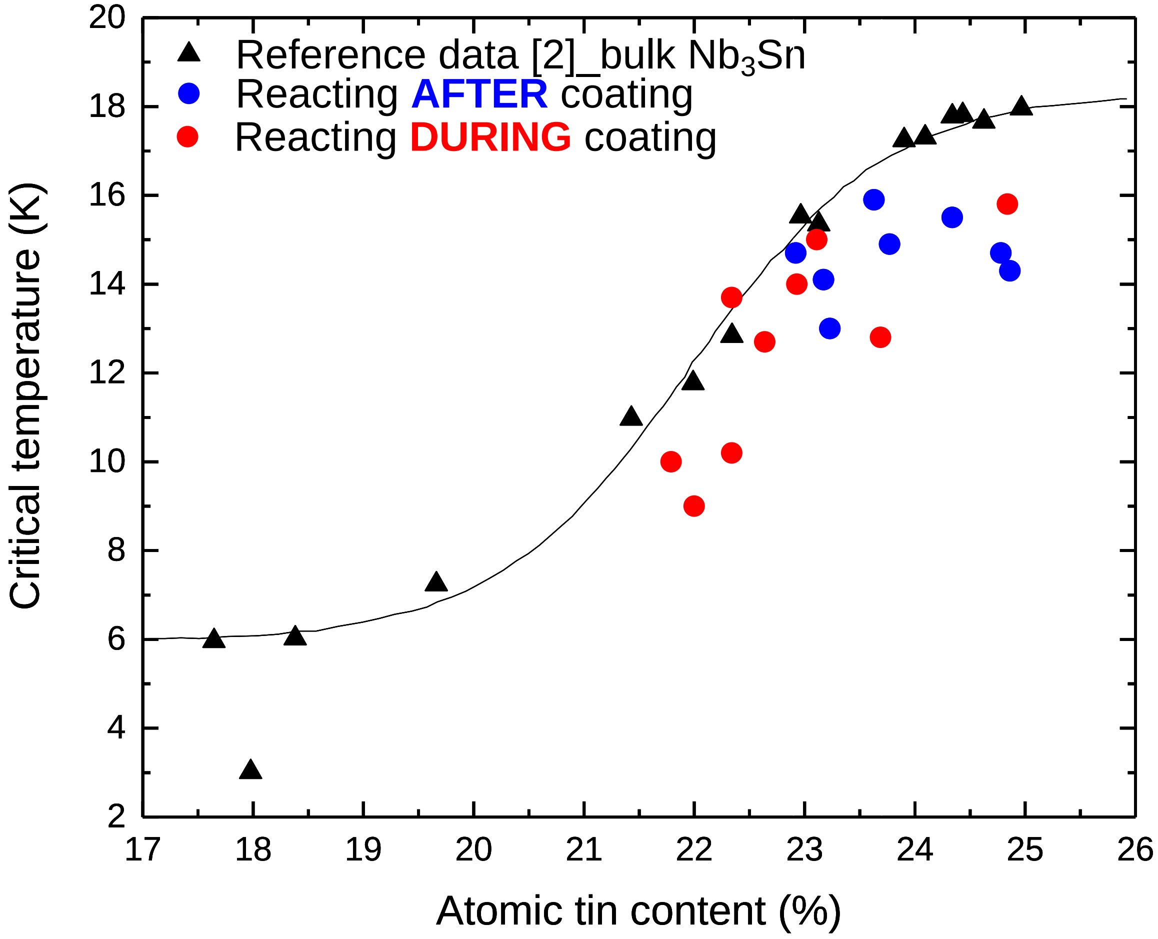

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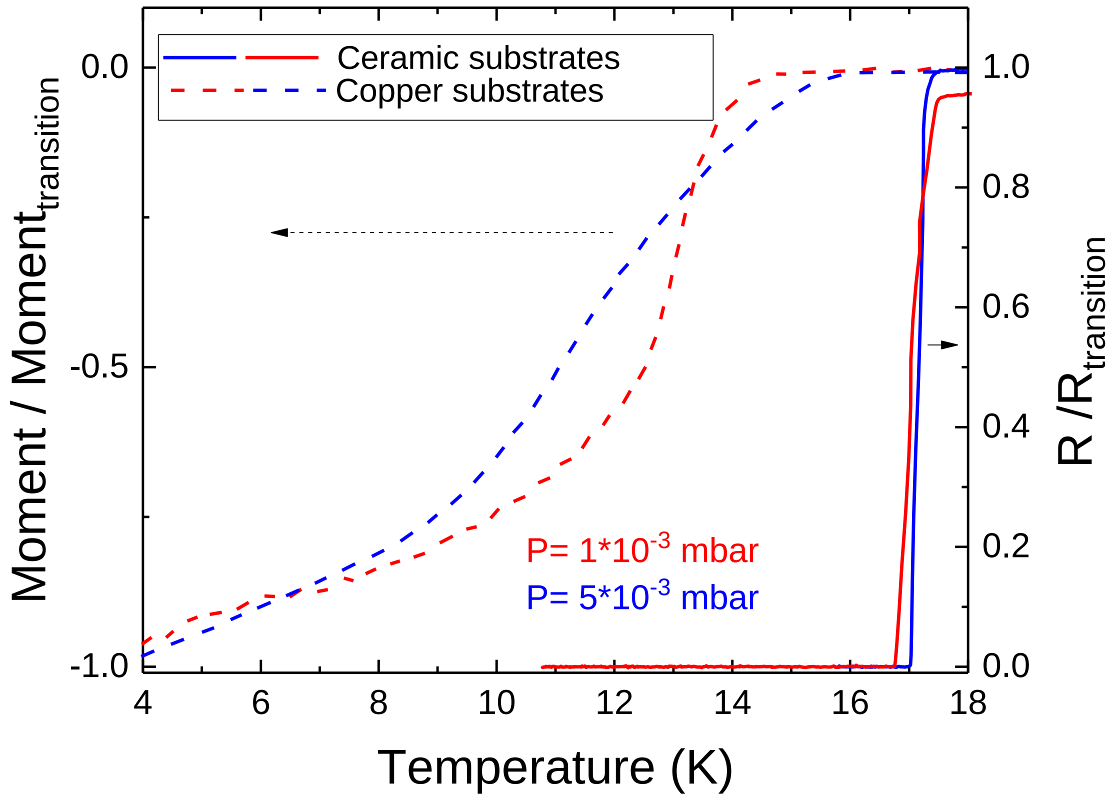
How to increase T_c?

- Composition
- Films of Nb_3Sn on copper substrate
- Reacting AFTER /DURING coating
- High temperature treatment duration
- Additional Annealing

IMPACT OF THE SUBSTRATE CHOICE ON T_C TRANSITION

- Increase of the *Tc* on 2-4K for the films grown on ceramic substrates (Al_2O_3)
- Sharper transition for Nb_3Sn synthesised on ceramics



Best *Tc*

on copper ~ 16 K

on ceramic (Al_2O_3) ~ 17 K

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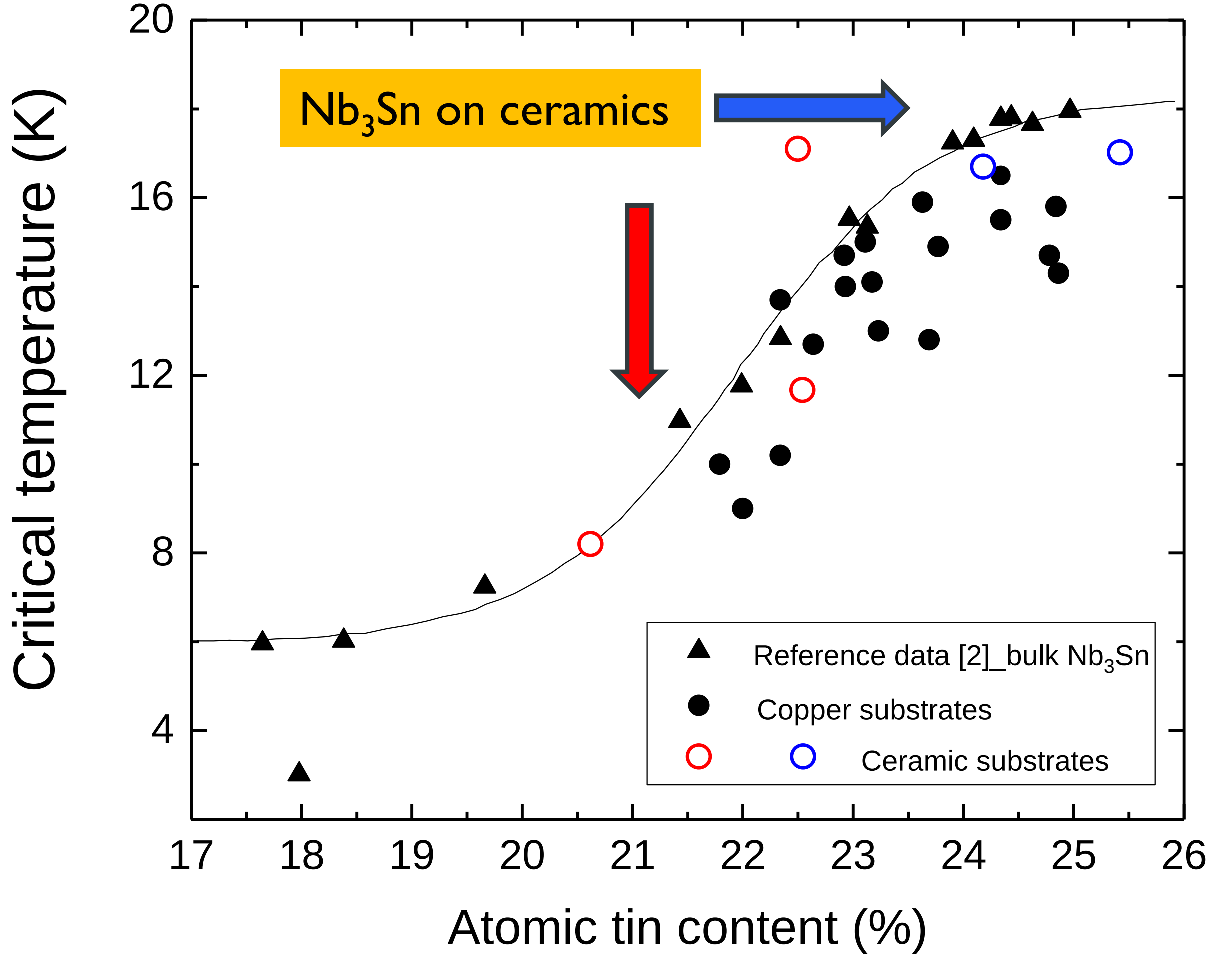

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COPPER SUBSTRATE "CHALLENGE"

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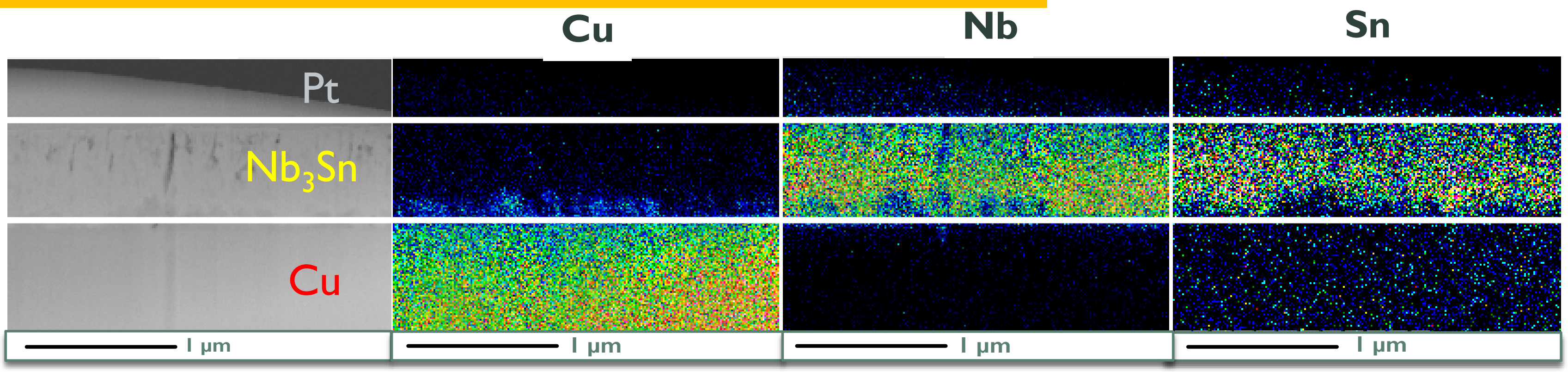
Reacted **After** Coating (24h High Temperature Treatment)



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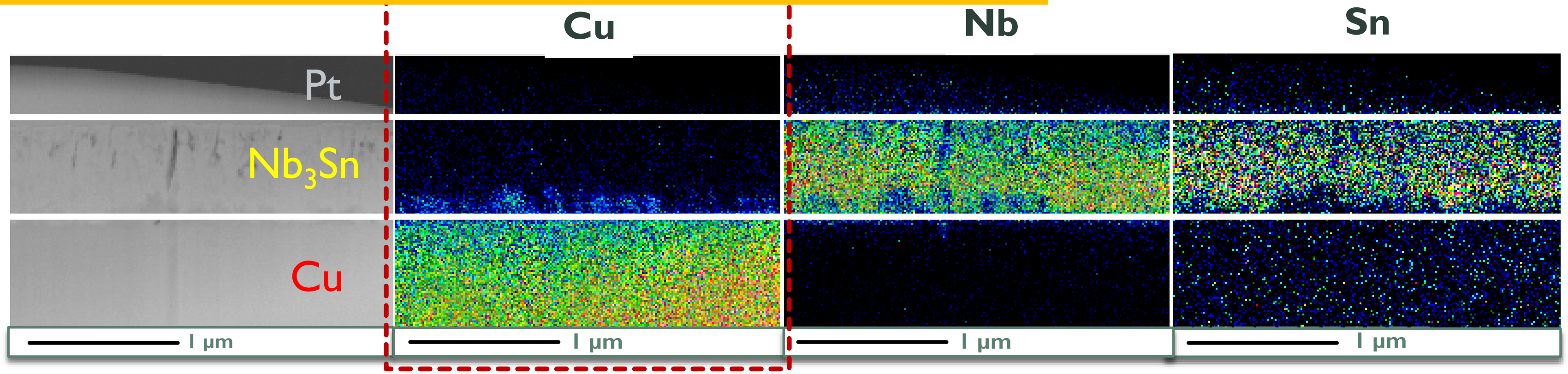

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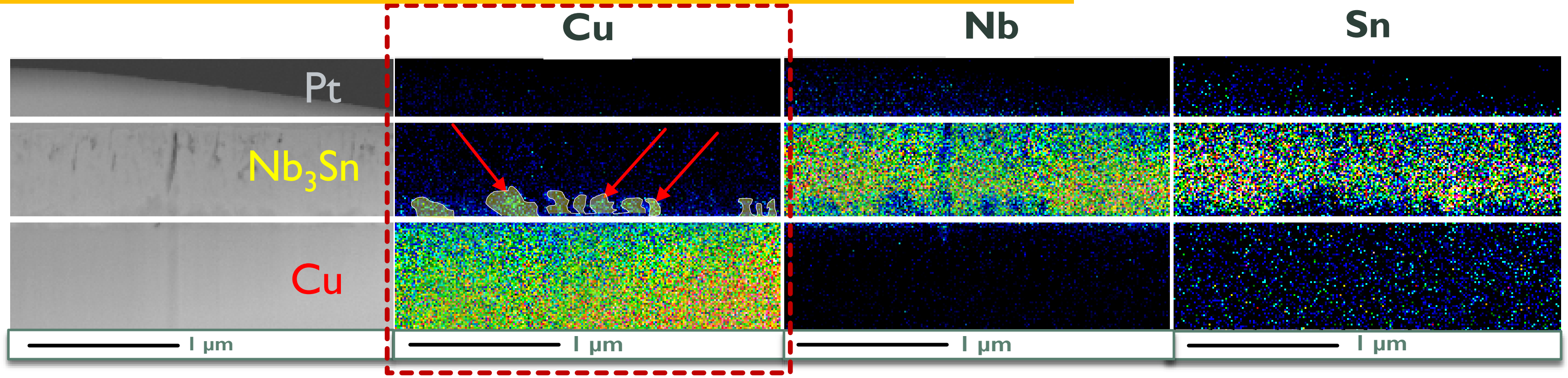

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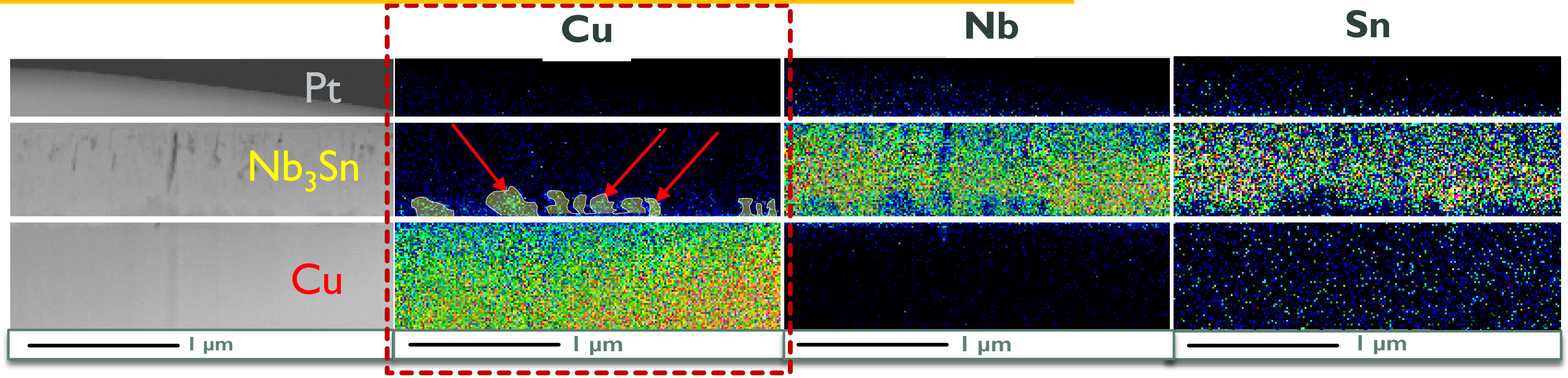

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- High temperature treatment causes copper interdiffusion

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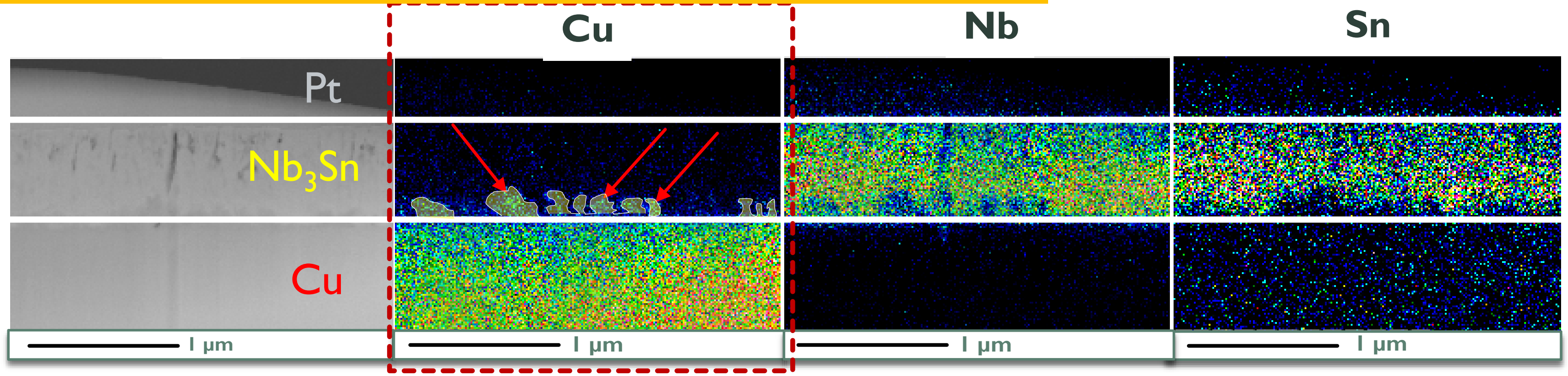
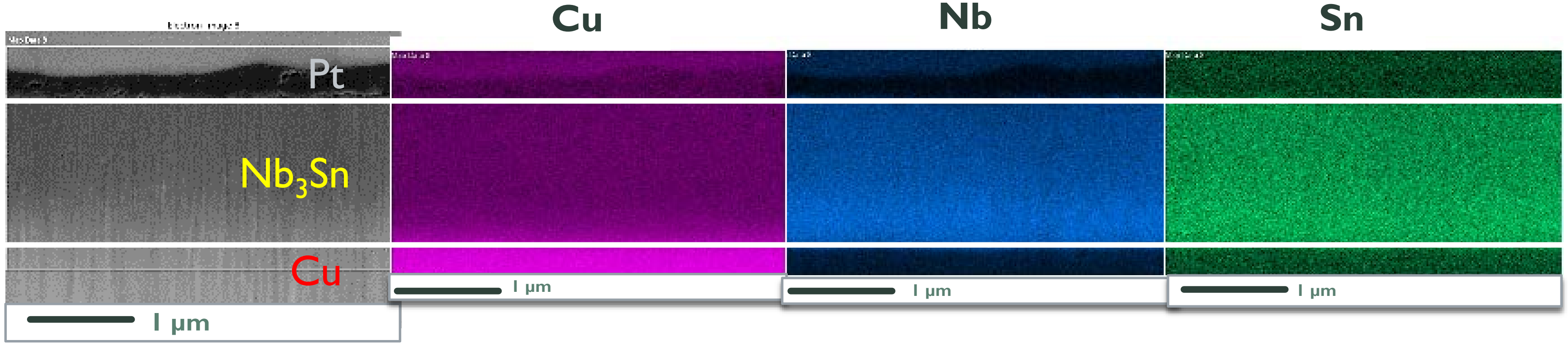


Reacted **During** Coating (1h High Temperature treatment)



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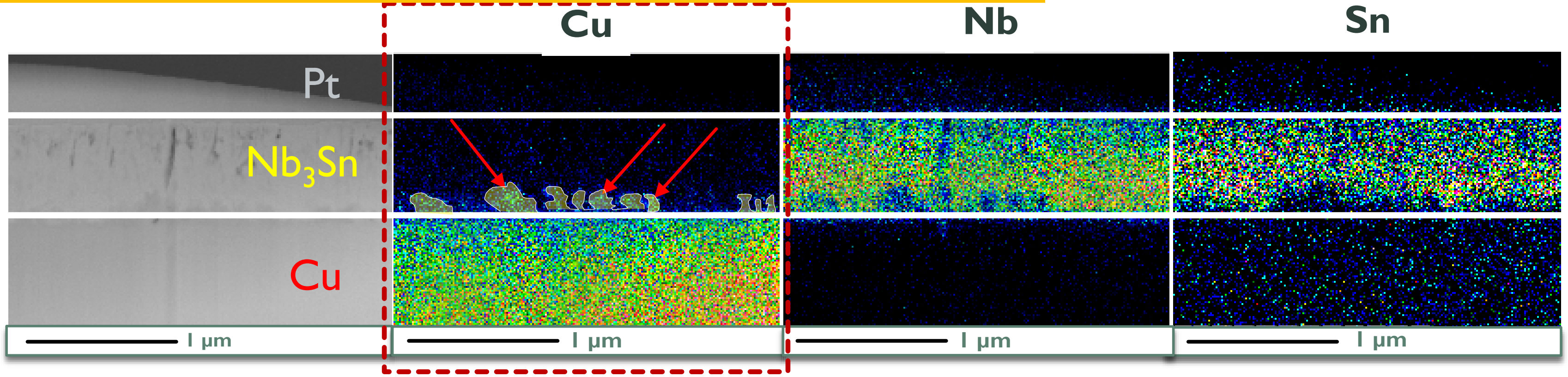

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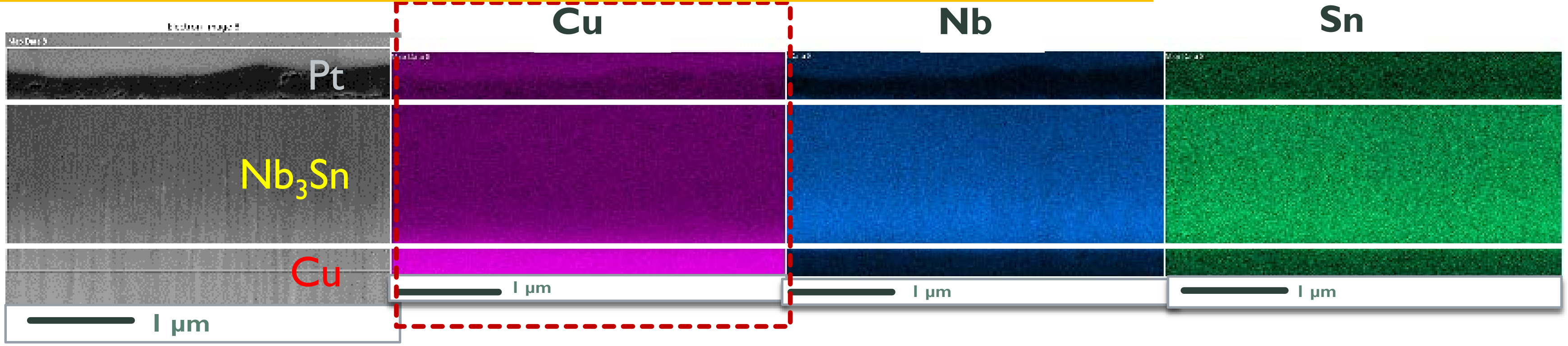

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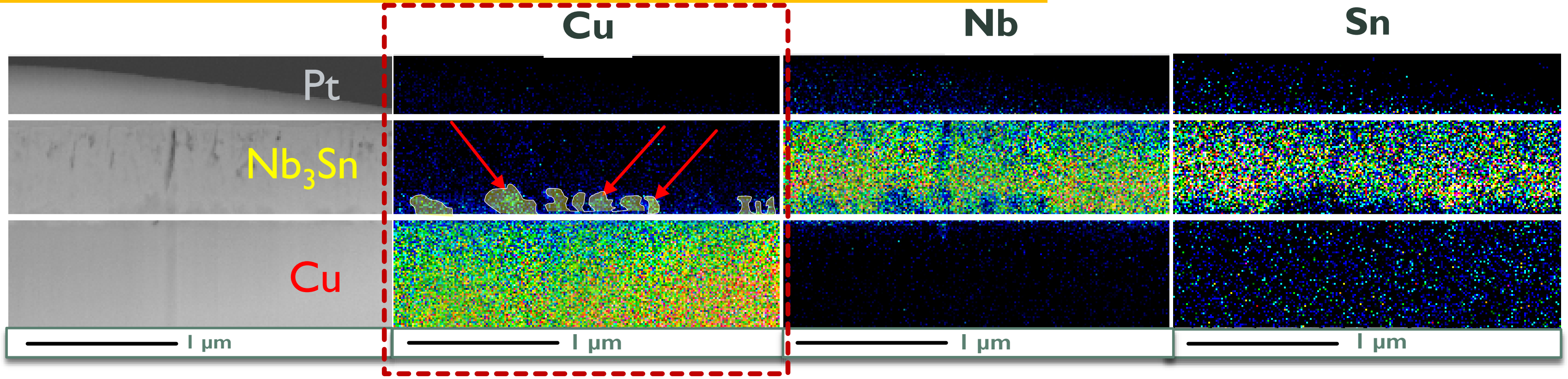

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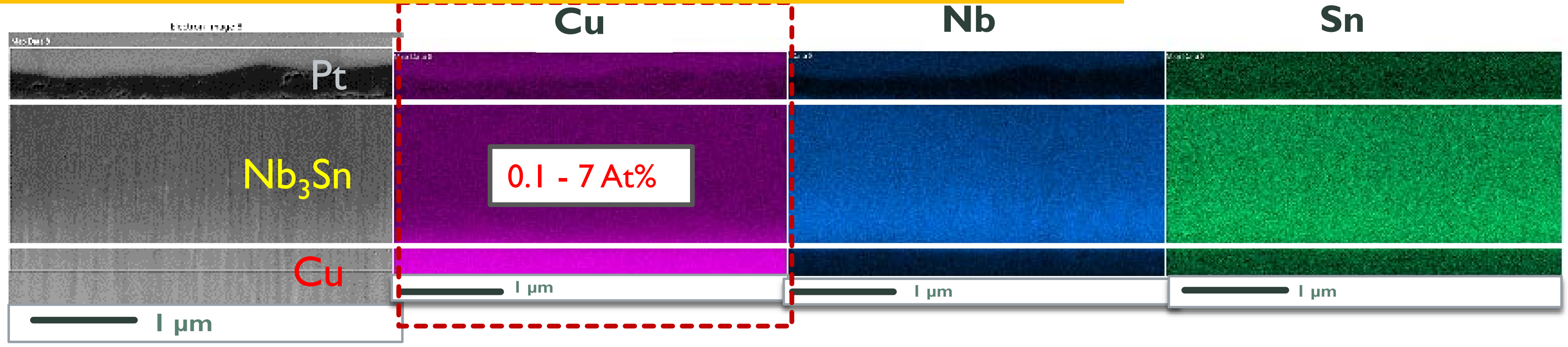


- *Nb* and *Sn* distribution are **homogenous**
- High temperature treatment causes copper interdiffusion

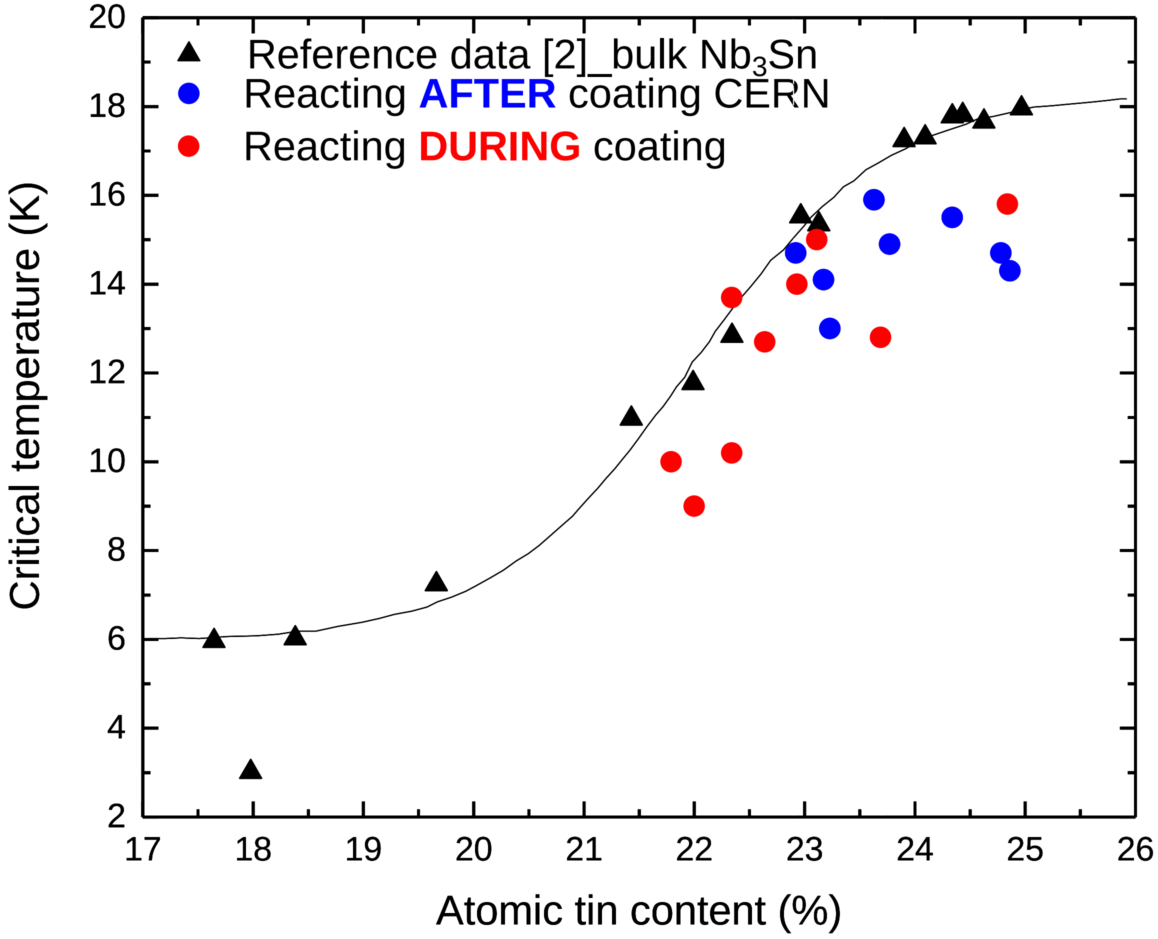
SOLUTION: INTERMEDIATE LAYER

- Works as "buffer" layer to reduce residual stresses in the films (to solve cracking problem for the films reacted **AFTER** the coating)
- Decrease lattice mismatch, i.e. improving crystalline lattice order (to avoid T_c depression) [3]
- Diffusion barrier layer (to prevent copper interdiffusion into Nb_3Sn layer)

Possible candidates – *Nb*, *Ta*, Al_2O_3

[3] Hein. The A15 story. The Science and Technology of Superconductivity pp 333-372

CRITICAL TEMPERATURE



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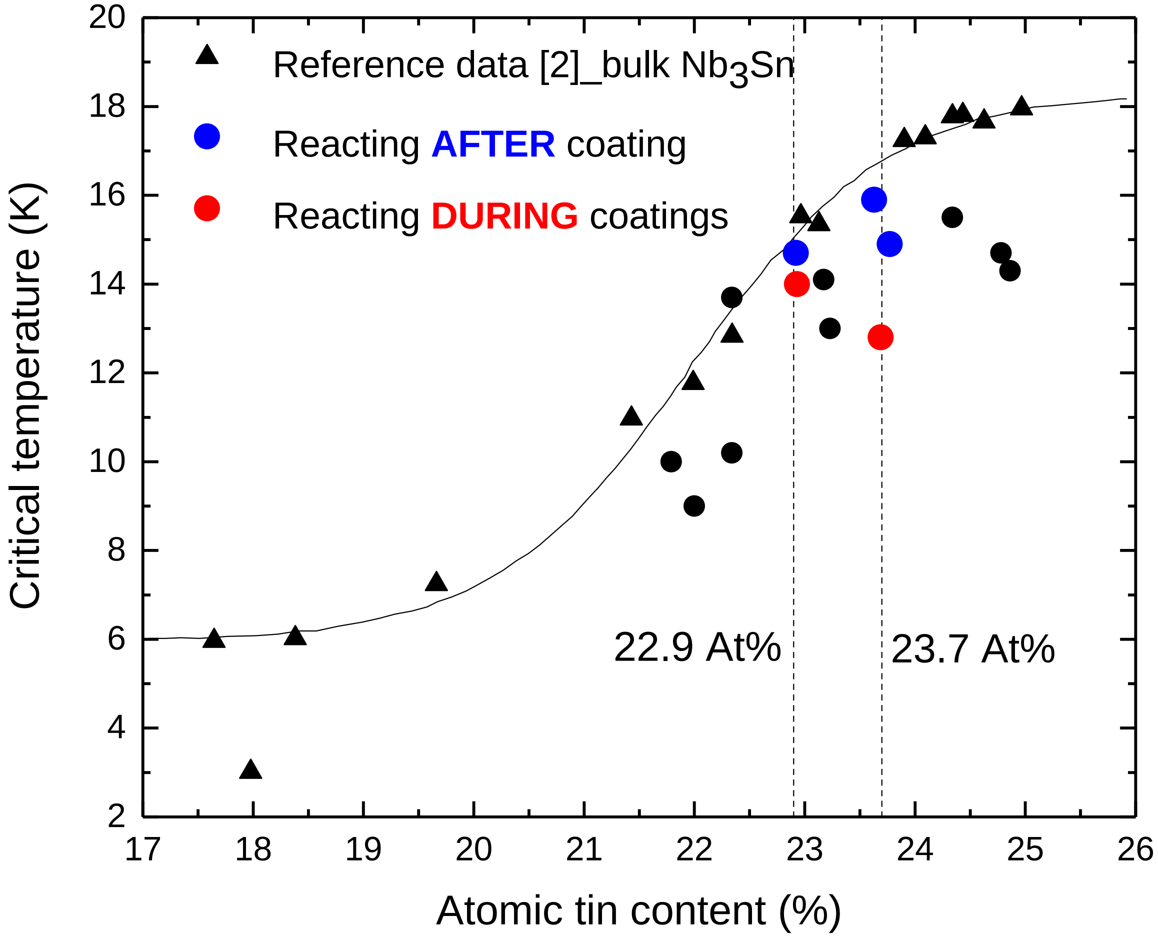

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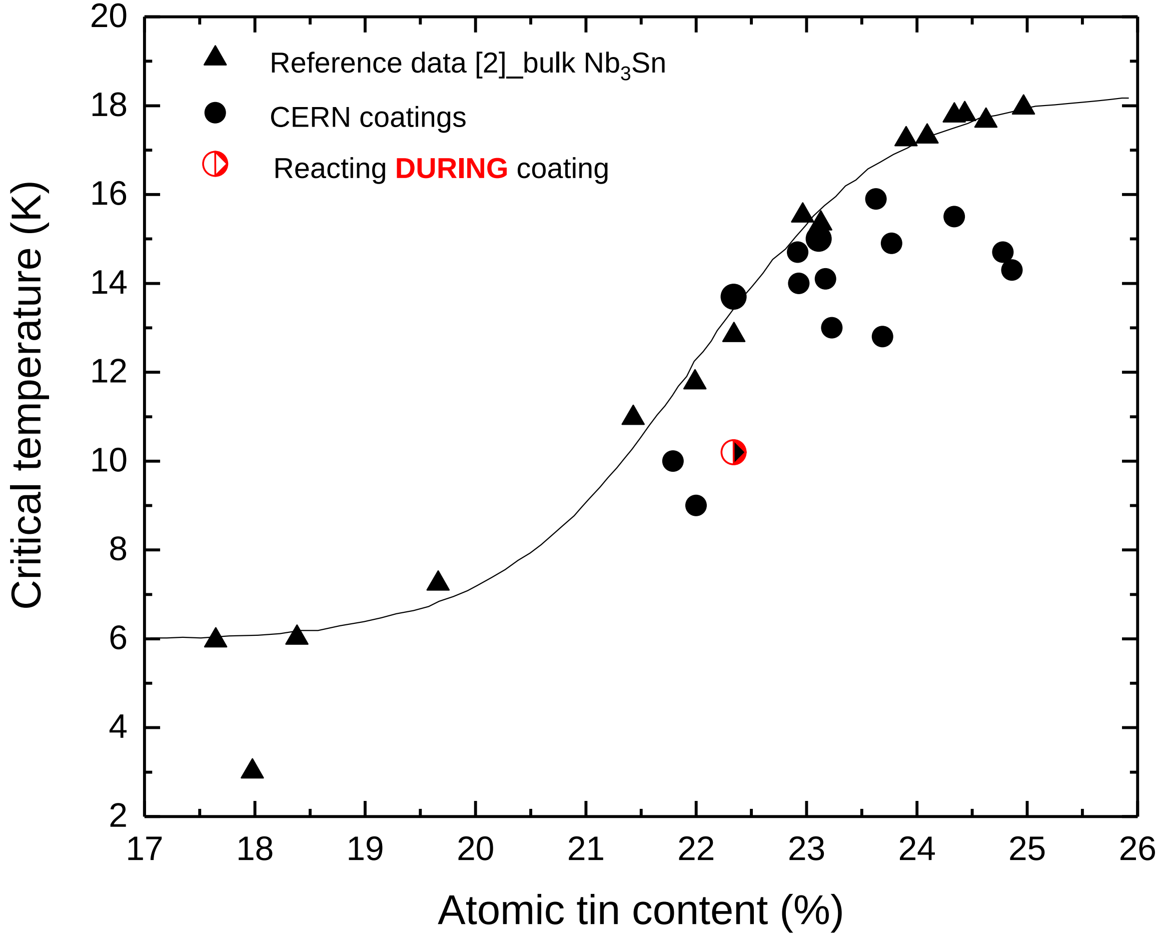

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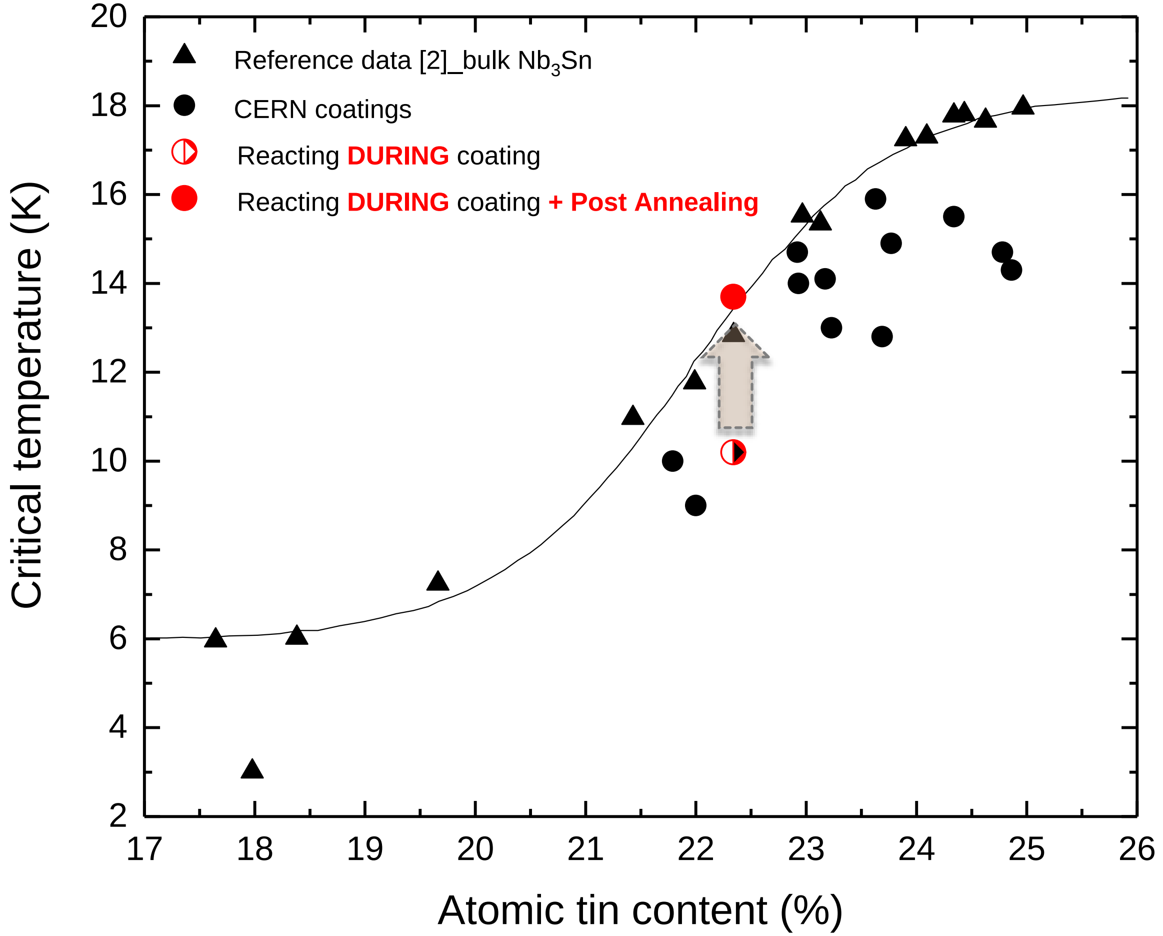
CRITICAL TEMPERATURE

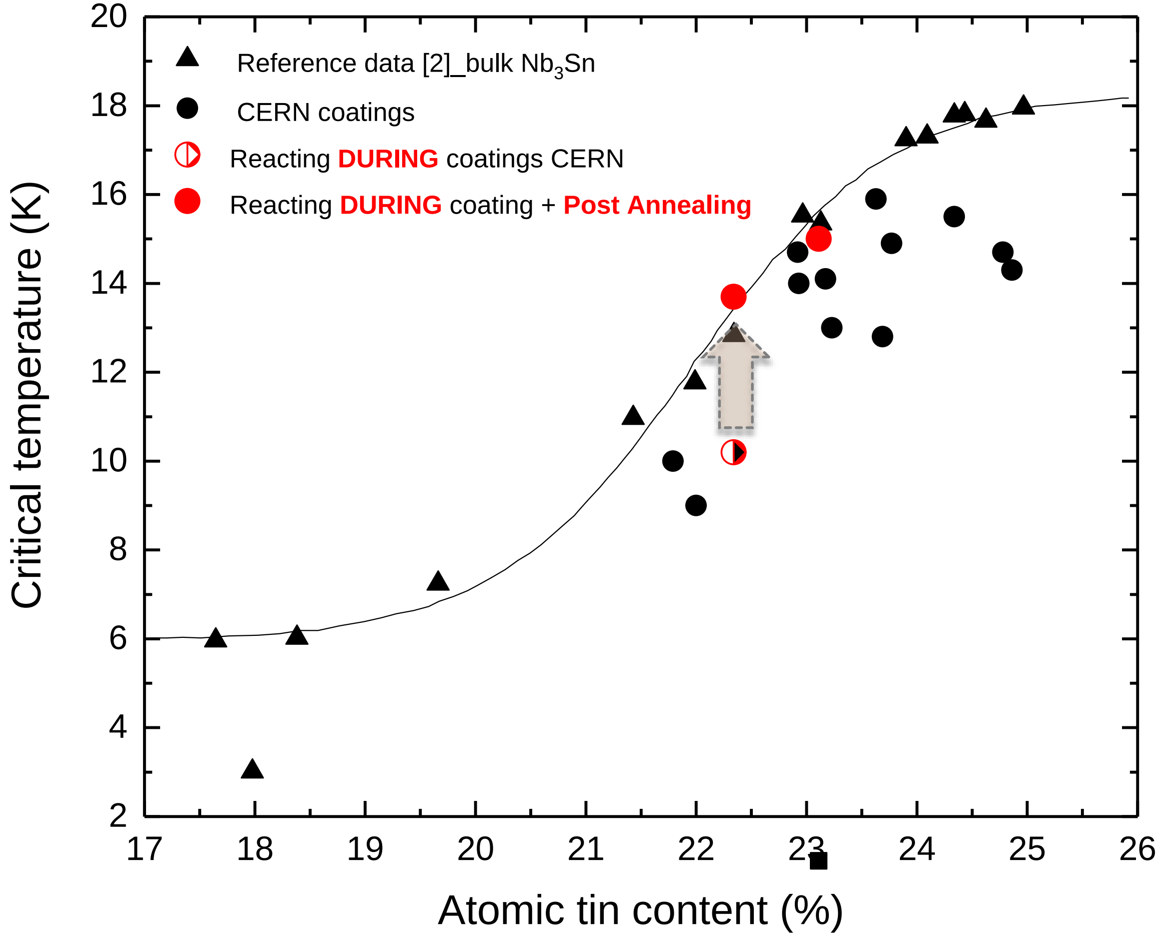


[2] *A. Godeke. Supercond. Sci. Technol., 19 (2006) R68-R80*

How to increase T_c?

- Composition
- Substrate choice
- High temperature treatment duration
- Additional Annealing

CRITICAL TEMPERATURE



[2] *A. Godeke. Supercond. Sci.Technol., 19 (2006) R68-R80*

How to increase T_c?

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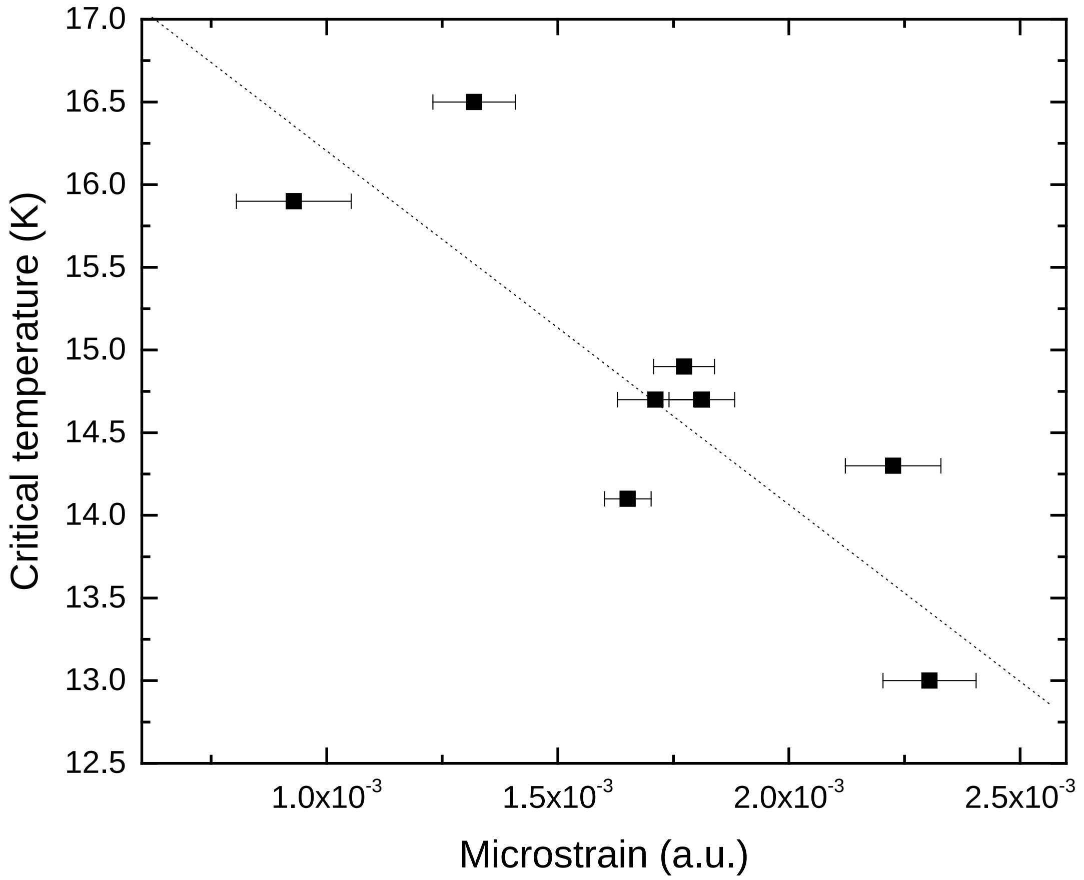
MICROSTRUCTURAL PROPERTIES



- *Calculated values from broadening of the diffraction lines using Rietveld analysis [4].*
- *Only samples reacted **AFTER** the coating were taken into consideration.*
- *Uniform residual stress is released but impact of microstrain remains important.*
- *Dependence of critical temperature on microstrain in the films is established.*

[4] L. Lutterotti, S. Matthies and H. -R. Wenk, *MAUD (Material Analysis Using Diffraction): a user friendly Java program for Rietveld Texture Analysis and more*, Proceeding of the Twelfth International Conference on Textures of Materials (ICOTOM-12), Vol. 1, 1599, 1999.

SUMMARY

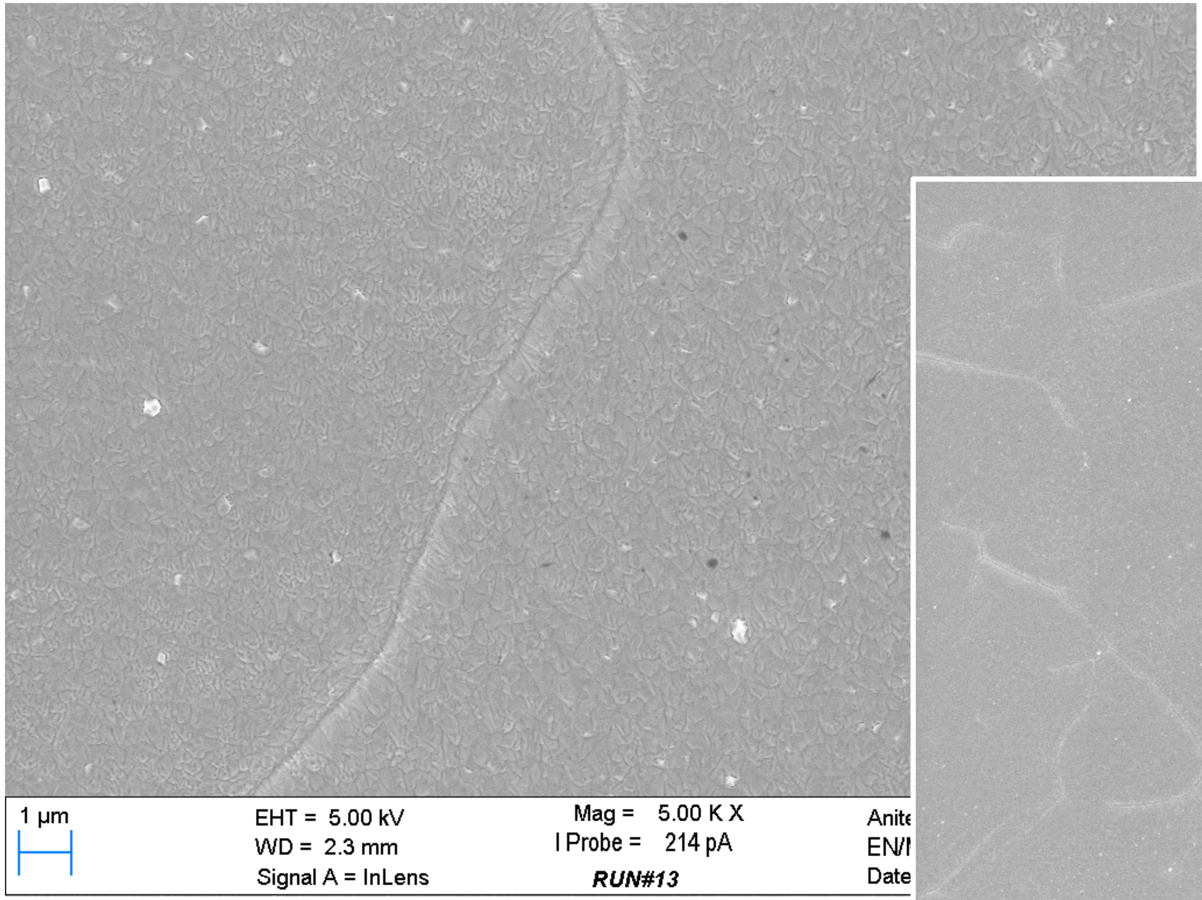
- We are able to produce high quality films (good composition, crack-free surface, *Tc* on ceramic ~ 17.2 K and on copper ~ 16 K)

- Impact of the copper substrate, i.e. increasing in disorder degree and copper interdiffusion after annealing, can be minimised by using intermediate layer.

- The recipes for the synthesis applicable for RF applications using both producing routes (reacting AFTER and DURING coating) will be produced.

- Coating of the QPR sample in order to test RF properties of the film is planned for the beginning of the next year.

Thank you for your attention!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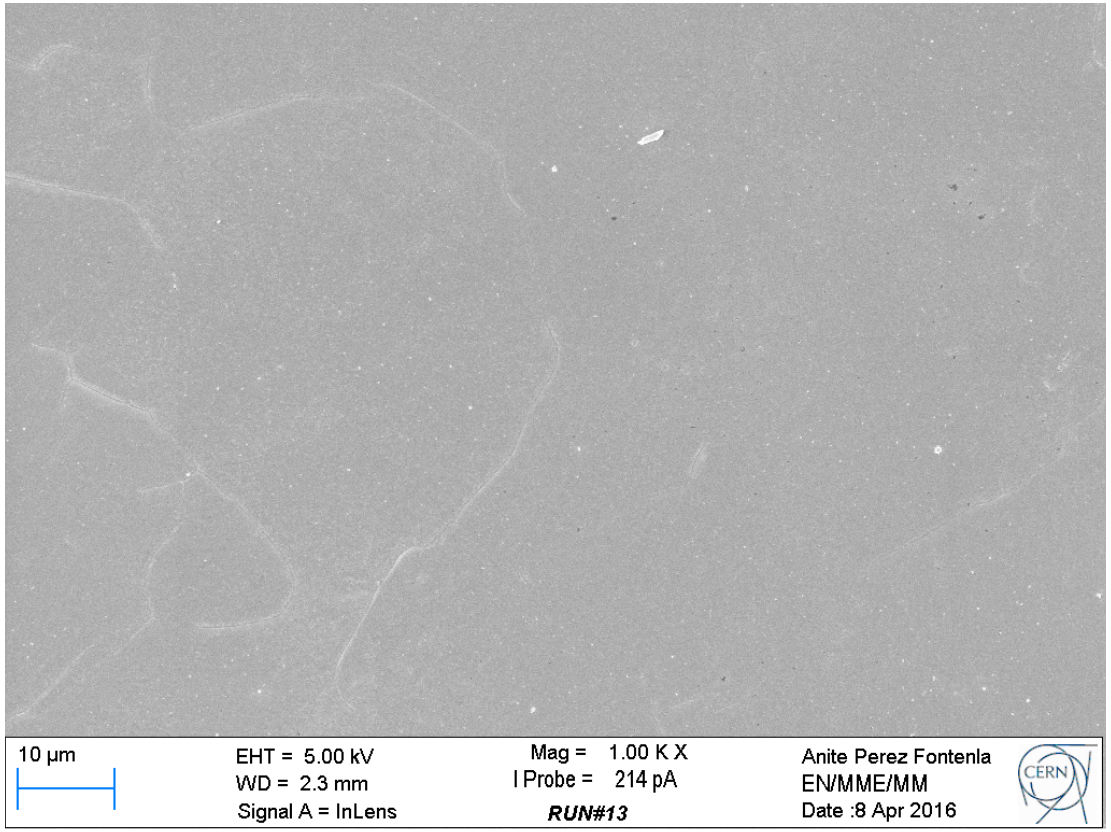
EHT = 5.00 kV
WD = 2.3 mm
Signal A = InLens
Mag = 5.00 K X
I Probe = 214 pA
RUN#13


10 µm
EHT = 5.00 kV
WD = 2.3 mm
Signal A = InLens
Mag = 1.00 K X
I Probe = 214 pA
RUN#13
Anite Perez Fontenla
EN/MME/MM
Date :8 Apr 2016

Difference in thermal expansion coefficient between substrate and Nb_3Sn contribute to disordering effect.

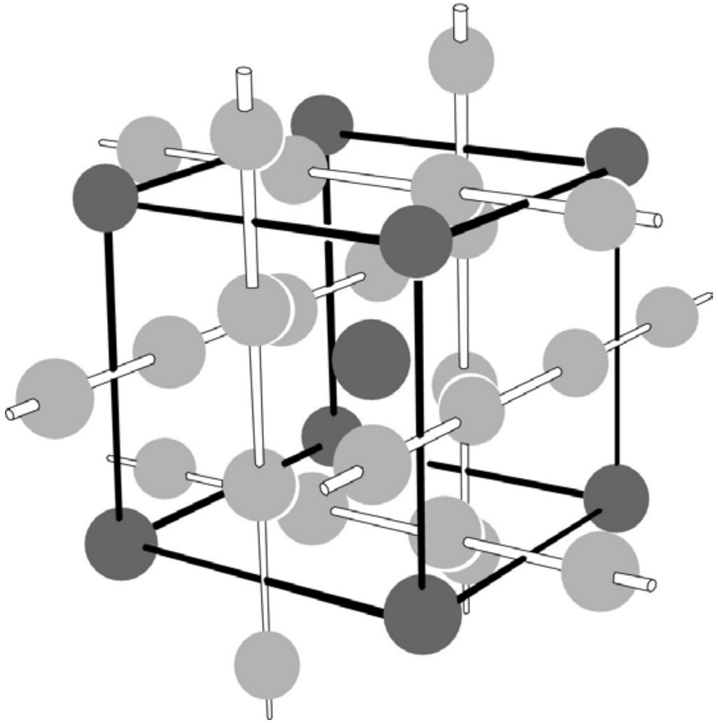
Element	α (x 10^{-6}) K^{-1}
Cu	16.8
Ta	7.64
Nb	7.02
Nb_3Sn	6.3

In BCS theory:

$$T_c \simeq \theta_D e^{-(1/N(0)\cdot V^*)}$$

θ_D – Debye temperature
$N(0)$ – density of state at the Fermi surface
V^* - interaction between electron and lattice vibration

From this expression one sees that a large N(0), implies a large T_c.
The BCS interaction parameter, V^*, is approximately a constant. [5]



"*A15 phases.*" *McGraw-Hill Concise Encyclopedia of Physics. 2002. The McGraw-Hill Companies, Inc. 3 Nov. 2017*
https://encyclopedia2.thefreedictionary.com/A15+phases

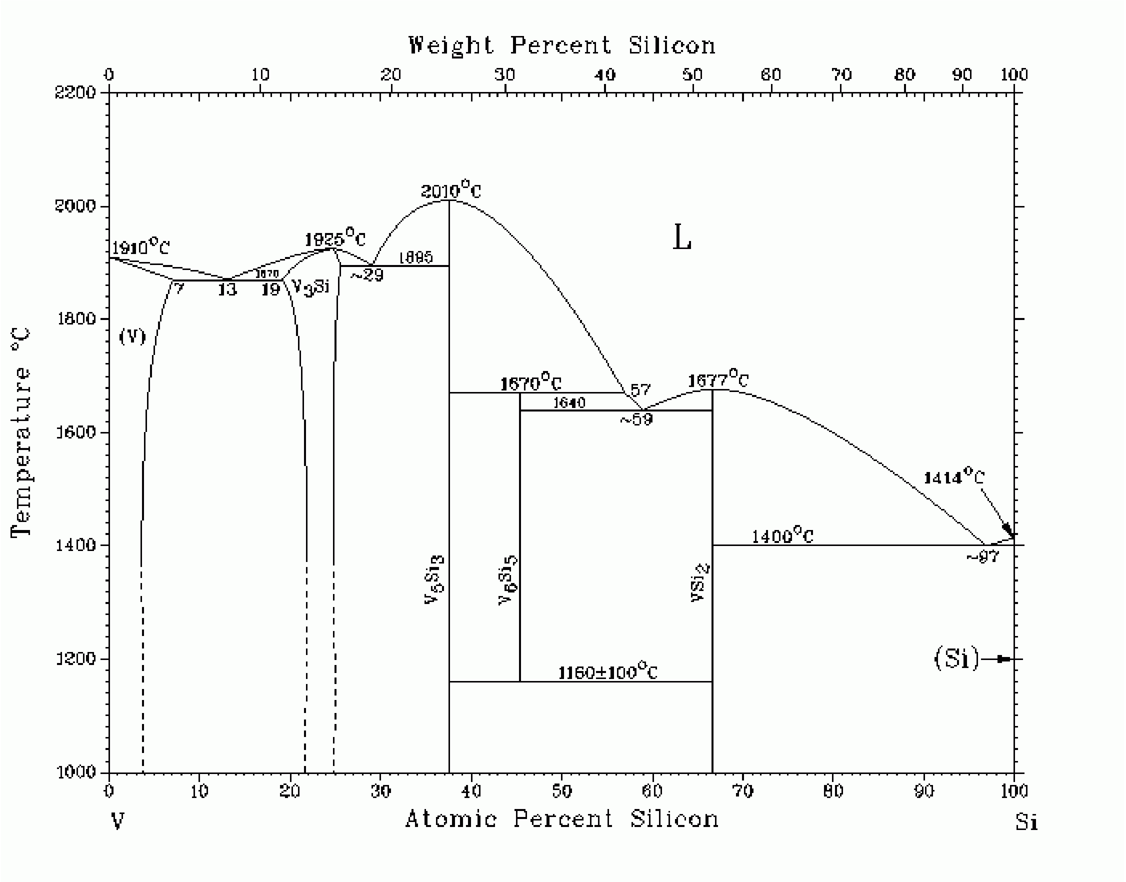
$$\downarrow d \Rightarrow \uparrow N(0)$$

d – interatomic distance between *Nb* atoms

$$\uparrow N(0) \Rightarrow \uparrow T_c$$

[5] SUPERCONDUCTING INTERMETALLIC COMPOUNDS - THE AIS STORY. Robert A. Hein. U. S. Naval Research Laboratory Washington, D. C. 20390

W. D. Gregory et al. (eds.), The Science and Technology of Superconductivity © Plenum Press, New York 1973


Weight Percent Silicon
Temperature °C
Atomic Percent Silicon
V
Si
L
(V)
(Si)
2010°C
1925°C
1910°C
1895
~29
7
13
19
V_3Si
V_5Si_3
V_6Si_5
VSi_2
1670°C
1640
57
~59
1677°C
1414°C
1400°C
~97
1160±100°C