Fermilab

Electrodeposition of Nb3Sn thin films

FERMILAB-SLIDES-19-0134-STUDENT

Electrodeposition of Nb_3Sn thin films

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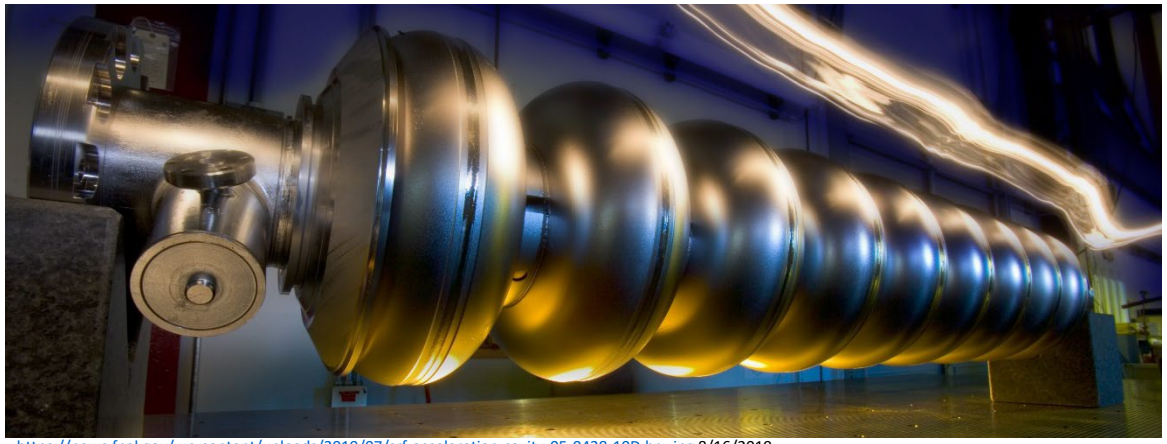
Supervisor: Emanuela Barzi

Summer School Project

24 September 2019

Aim of the project



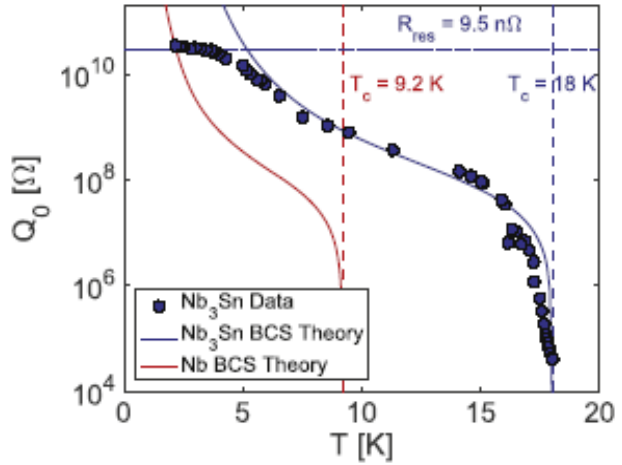
https://news.fnal.gov/wp-content/uploads/2019/07/srf-acceleration-cavity-05-0438-10D.hr_.jpg 8/16/2019

Nb SRF cavities are reaching their performance limit

Nb_3Sn is the best candidate to replace Nb as superconducting material for SRF cavities

Nb_3Sn :

- B_{C20} = 30 T
- Tc = 18 K



How to deposit Nb_3Sn?

Electrodeposition

Vapor diffusion

Sputtering techniques

Electrodeposition from aqueous electrolytes

STEP 1:
Electrodeposition

GOAL:
Deposition on Nb substrate of 3 precursor layers:

- **Cu seed layer**
- **Sn layer**
- **Cu barrier layer**

STEP 2:
Baking in inert atmosphere

GOAL:

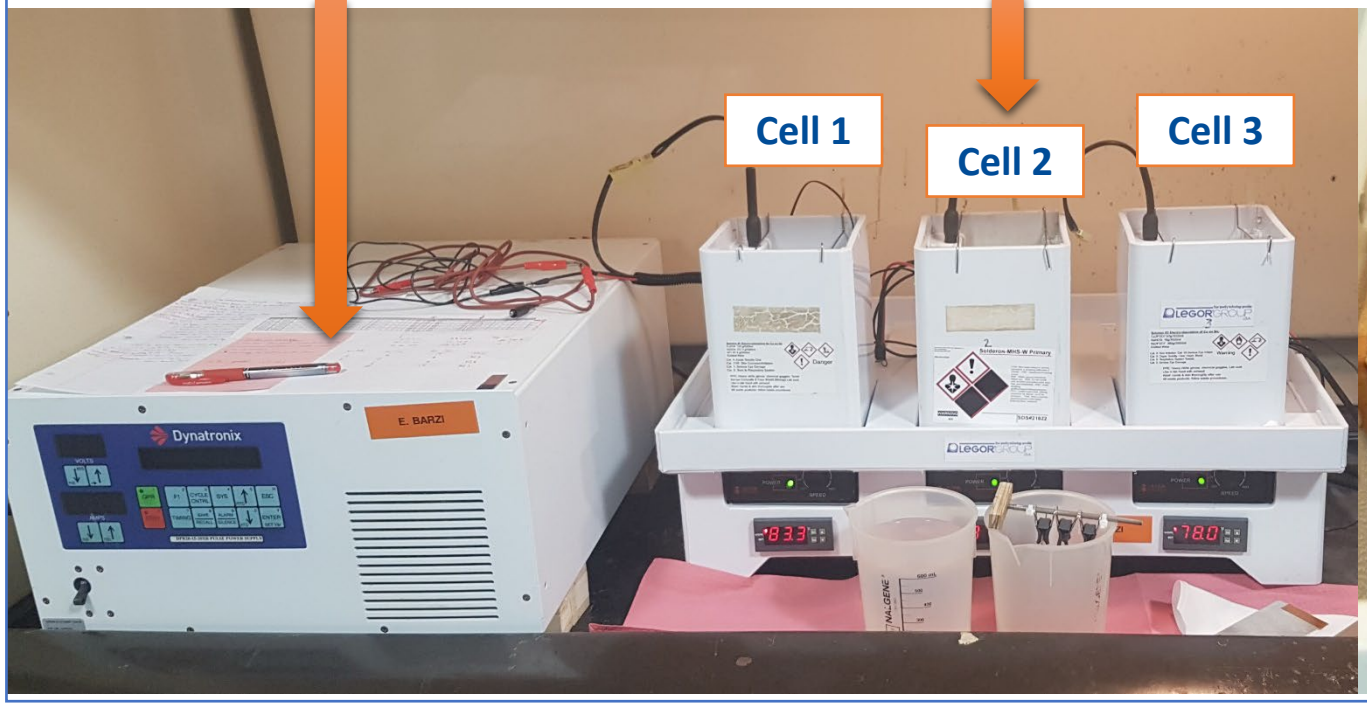
Formation of the superconductive phase Nb_3Sn during the thermal treatment

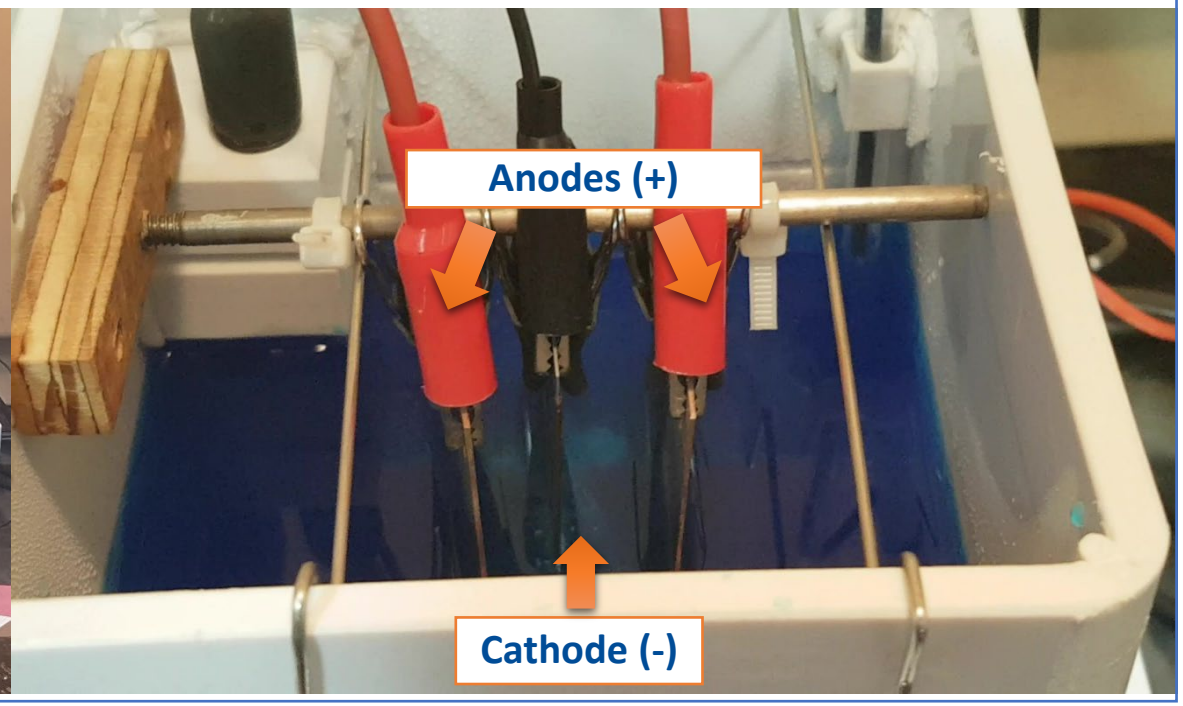
Experimental Setup: Electrochemical Cell

Power Supply

Electrochemical Cells

3 Electrodes Geometry

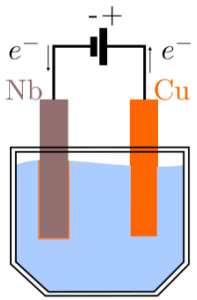




STEP 1

Deposition of Cu seed layer

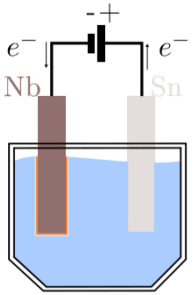
- HCl: $67.8ml$
- H_2SO_4: $217.4ml$
- $CuSO_4$: $120g$
- H_2O: up to 2L



STEP 2

Deposition of Sn layer

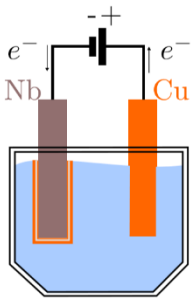
- Solderon Acid HC: $430ml$
- Solderon Tin HS-300: $333ml$
- Solderon MHS-W: $200ml$
- H_2O: up to 2L



STEP 3

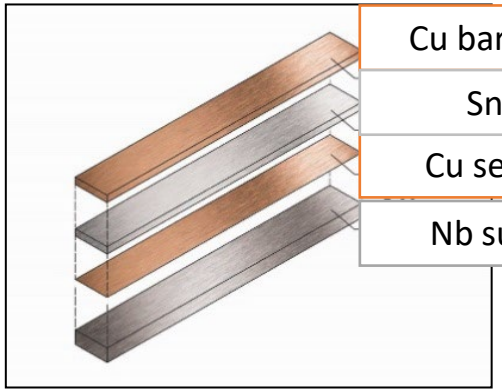
Deposition of Cu barrier layer

- $Cu_2P_2O_7$: $52g$
- $NaNO_3$: $10g$
- $Na_4P_2O_7$: $360g$
- H_2O: up to 2L



Experimental Setup: Baking

Final Structure

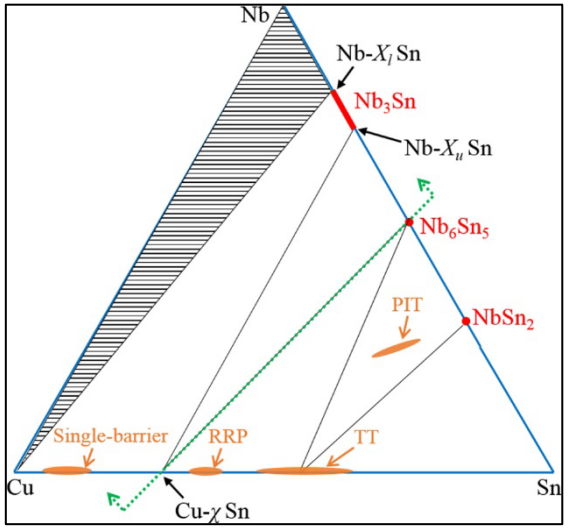


Cu barrier layer

Sn layer

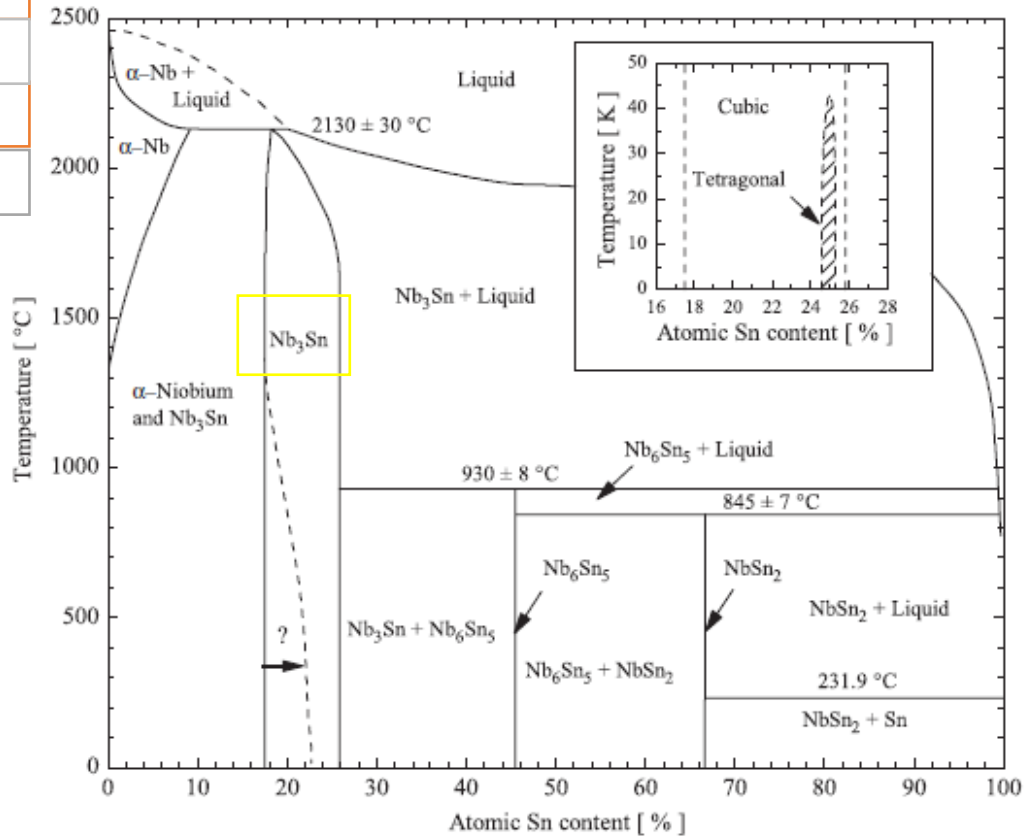
Cu seed layer

Nb substrate

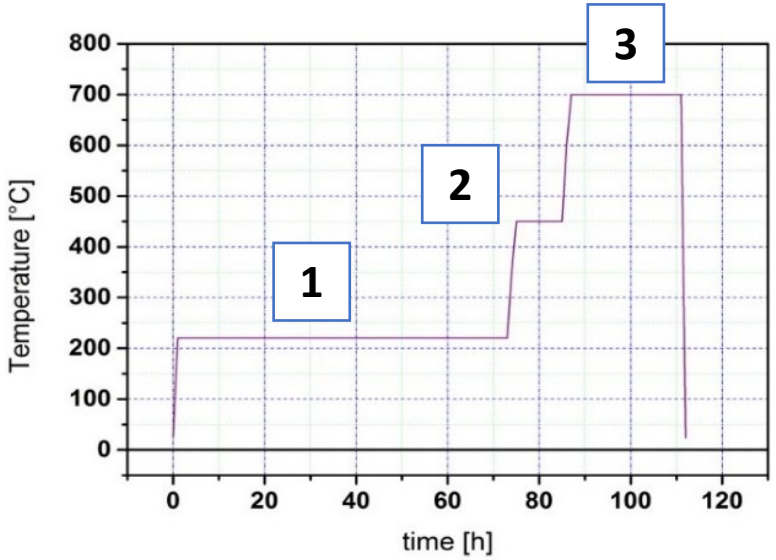


A review and prospects for Nb_3Sn superconductor development, X. Xu, Superconductor Science and Technology, 2017

Nb-Sn Phase Diagram



Thermal Treatment



1] Relaxation of internal stresses

2] Melting of Sn layer and interdiffusion Cu-Nb

3]Formation of Nb_3Sn phase

Bronze

Nb_3Sn and Nb/Sn phases

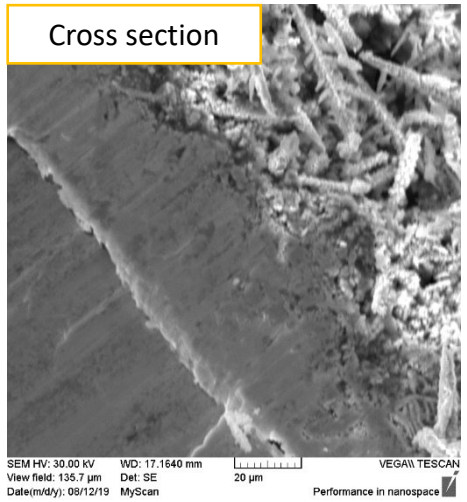
Nb substrate

Characterization and Superconductivity Test

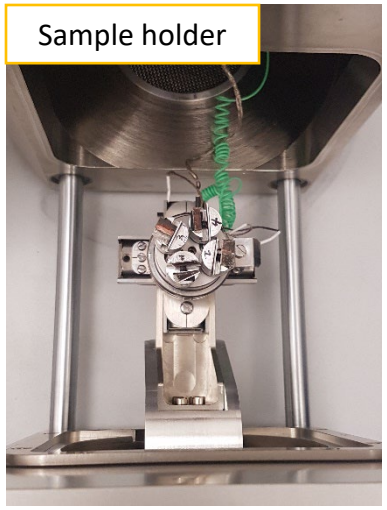
SEM & EDS

SEM apparatus

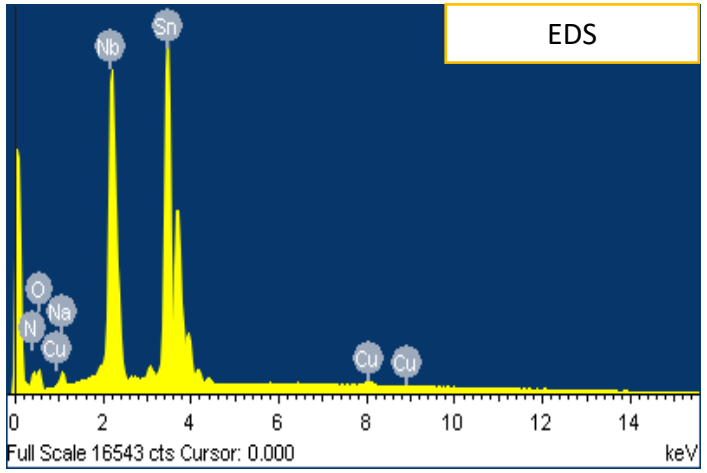
Cross section



Sample holder



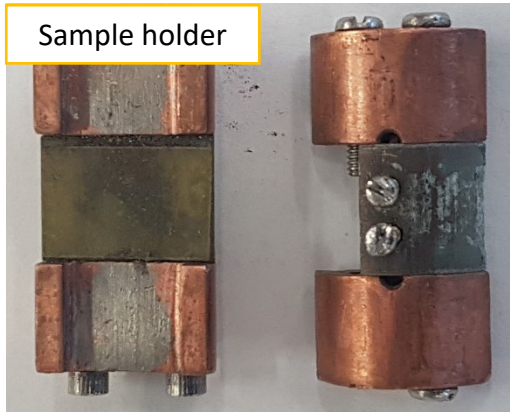
EDS



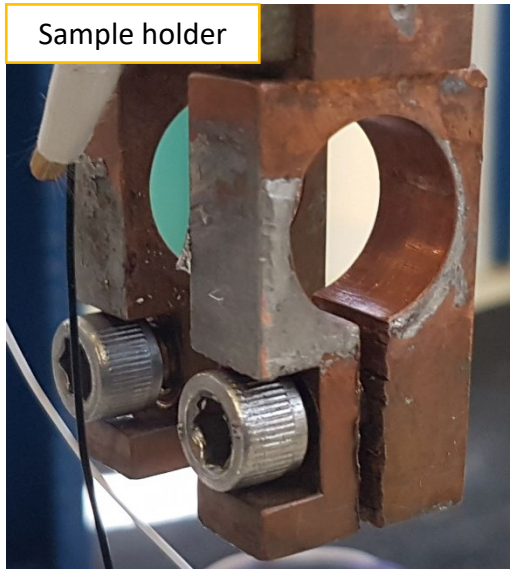
Superconductivity

Cryostat

Sample holder



Sample holder



Goals

1. Definition of the current densities range that allows for the deposition of a continuous and homogeneous Nb_3Sn superconducting thin-film
2. Improvement of the surface homogeneity (border effect)
3. Substrate roughness/ peeling off relationship
4. Testing the method feasibility on curved surfaces (Cylinders)
5. Deposition on Nb/Cu substrates

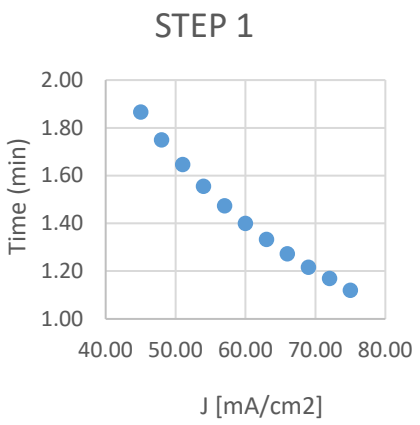
1: Definition of the current densities range

Starting from the electrochemical parameters of superconductive samples obtained last year, σ is kept constant, while J and t are varied across different samples

$$\sigma\ [C/cm^2] = J[A/cm^2] \cdot t[s]$$

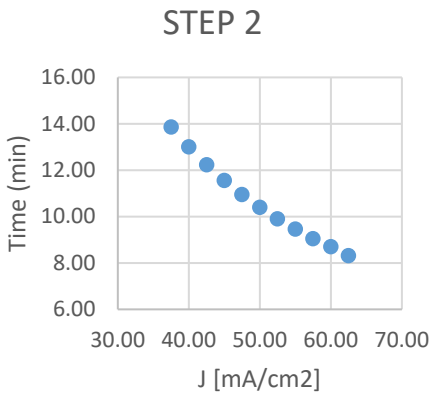
σ: *charge density*
J: *current density*

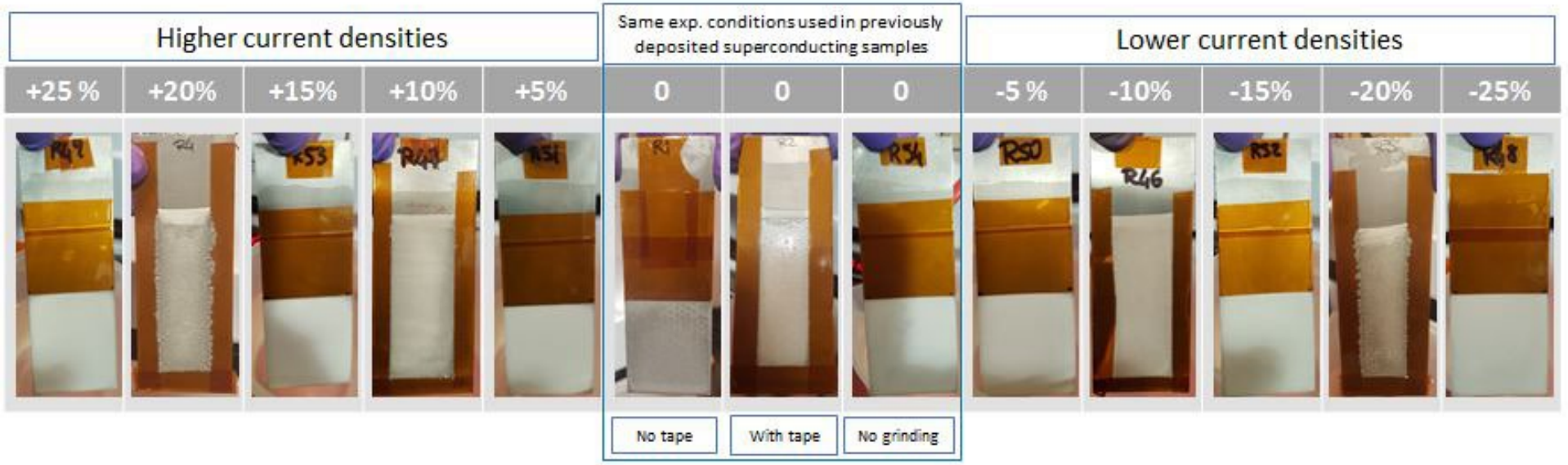
Deposition of Cu seed layer



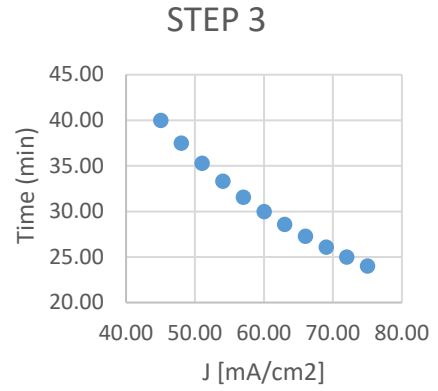
1: Definition of the current densities range

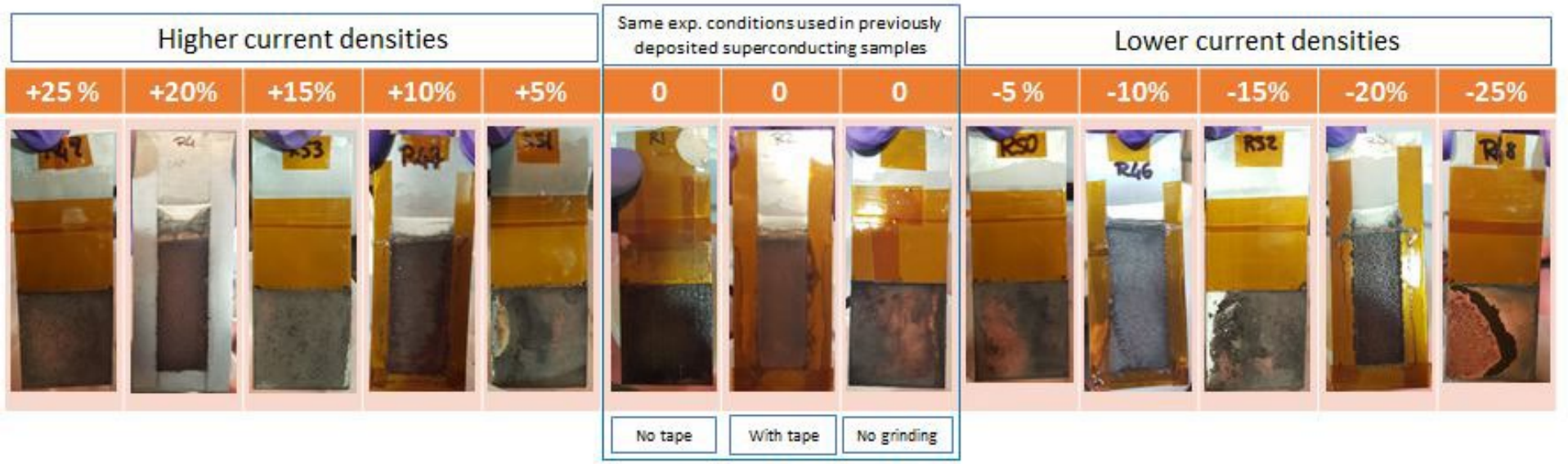
Deposition of Sn layer





Deposition of Cu barrier layer



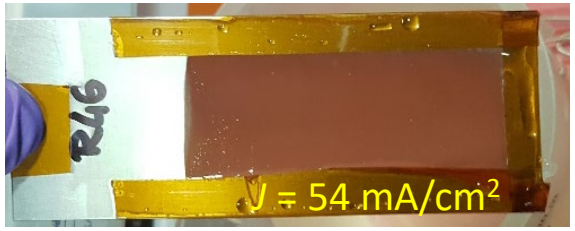


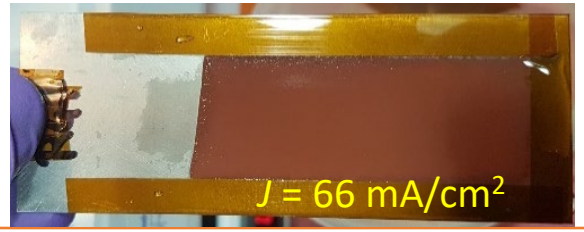
1: Definition of the current densities range

Cu seed layer

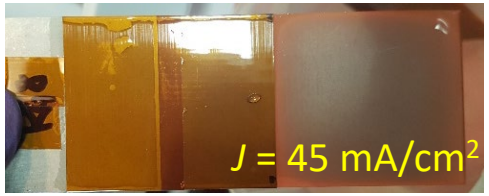
-10% ≤ *J* ≤ +10%

54 mA/cm² ≤ *J* ≤ 66 mA/cm²

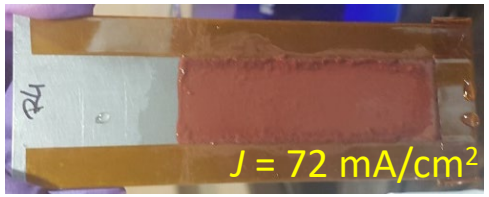




At **low *J***: non homogeneous deposition



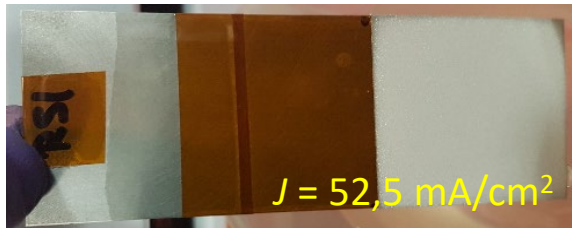
At **high *J***: formation of powder like deposits

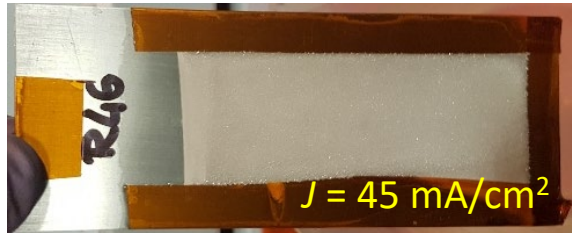


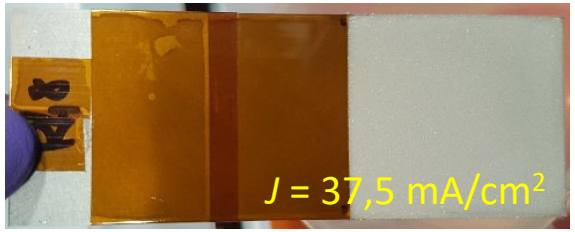
Sn layer

J ≤ +5%

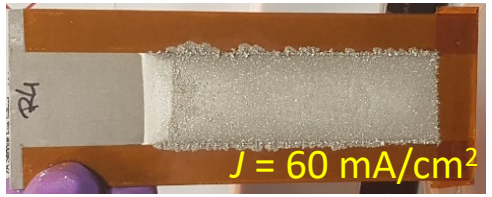
J ≤ 52,5 mA/cm²







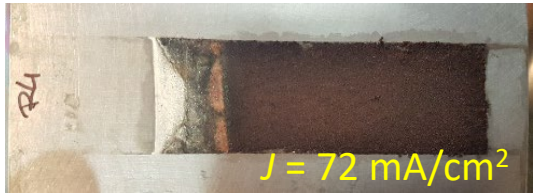
At **high *J***: formation of powder like deposits

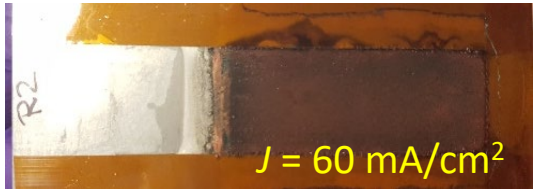


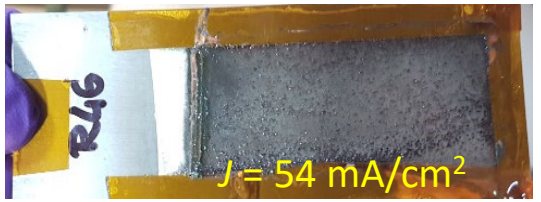
Cu barrier layer

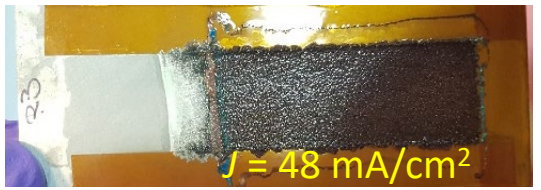
All samples are covered by dark powder like deposits = oxides

- Dark Red deposits = Cu_2O
- Black deposits = CuO
- Green areas = bronze patina

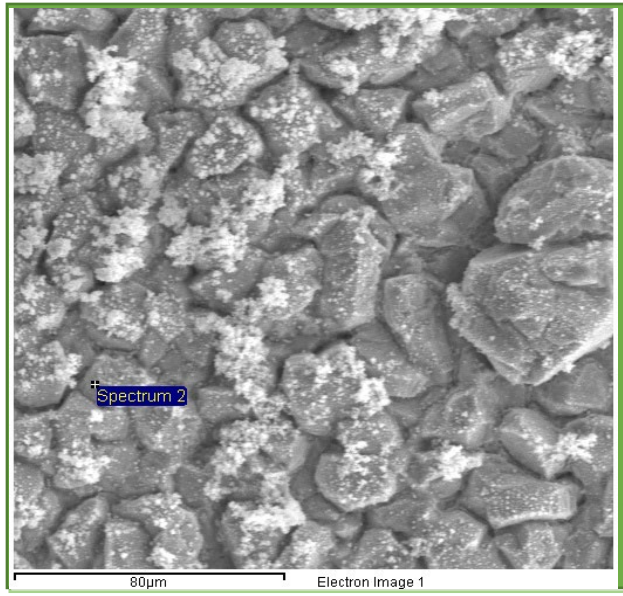


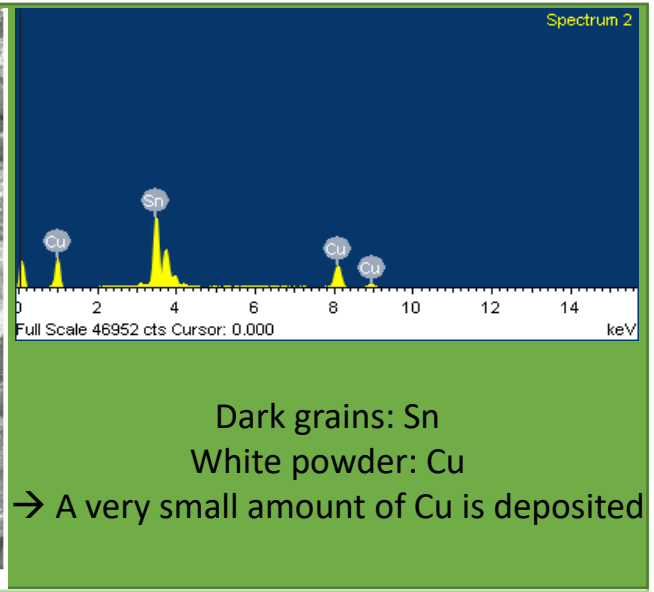




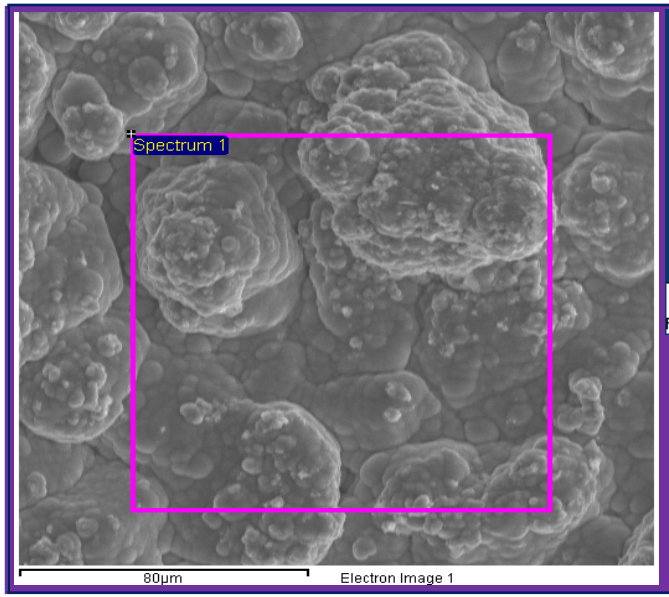


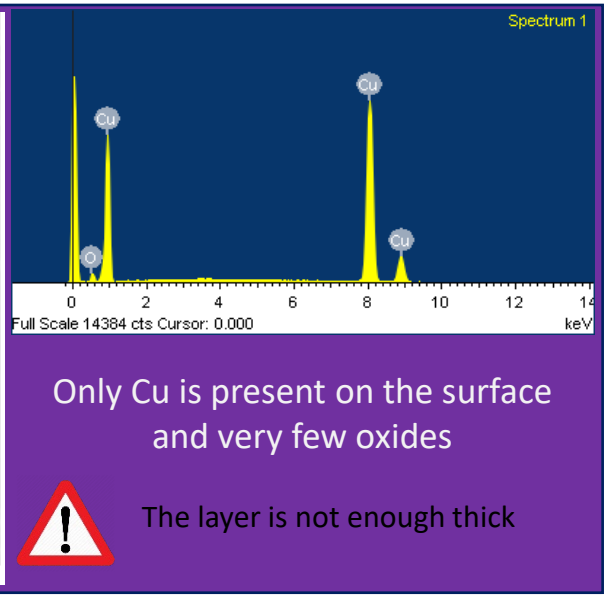
Deposition of the Cu barrier layer





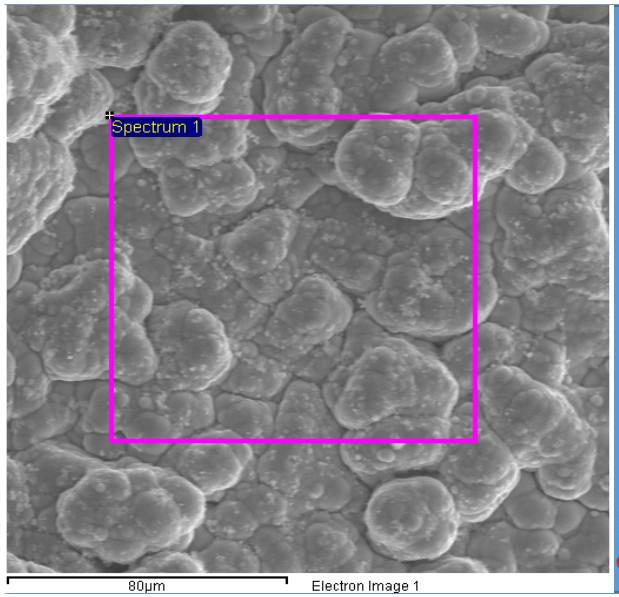
Dark grains: Sn
White powder: Cu
→ A very small amount of Cu is deposited

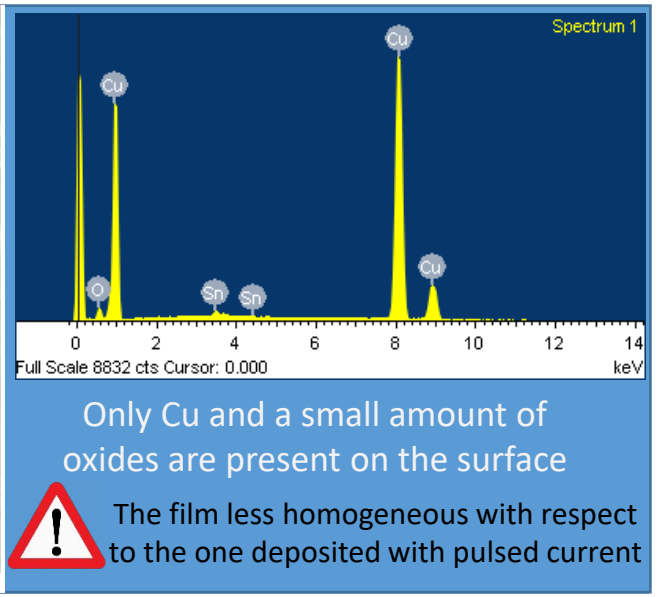




Only Cu is present on the surface and very few oxides

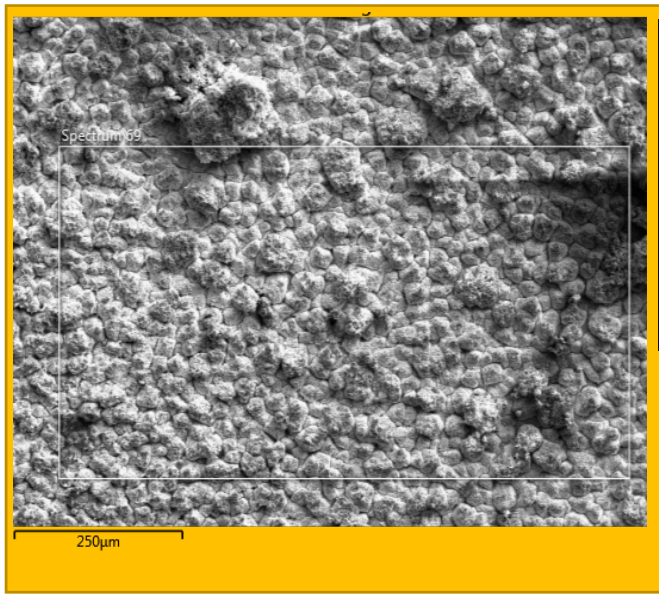
The layer is not enough thick

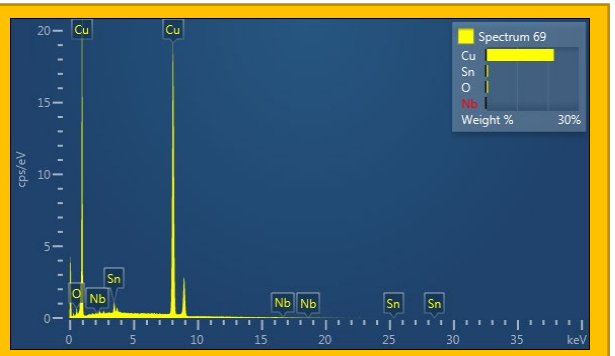




Only Cu and a small amount of oxides are present on the surface

The film less homogeneous with respect to the one deposited with pulsed current





Only Cu is present on the surface

Larger grains with respect to samples deposited in basic bath

2: Improvement of the surface homogeneity

Border effect: on the edges of the samples the electric field is higher than in the center → the current density on the edges is higher than in the center

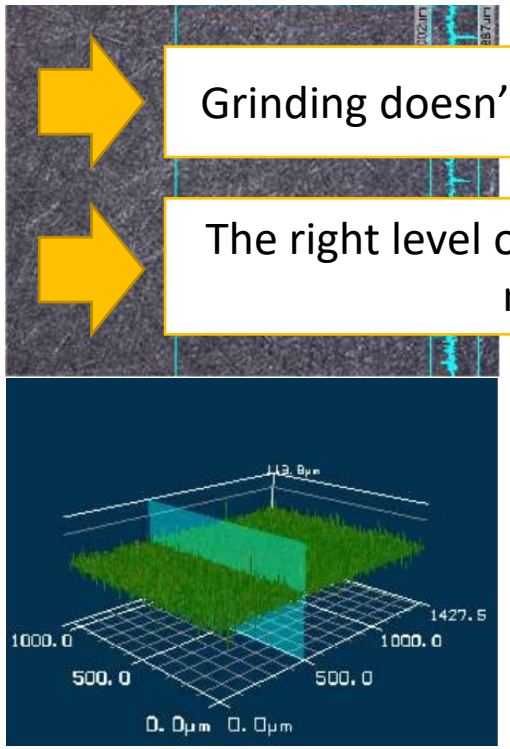
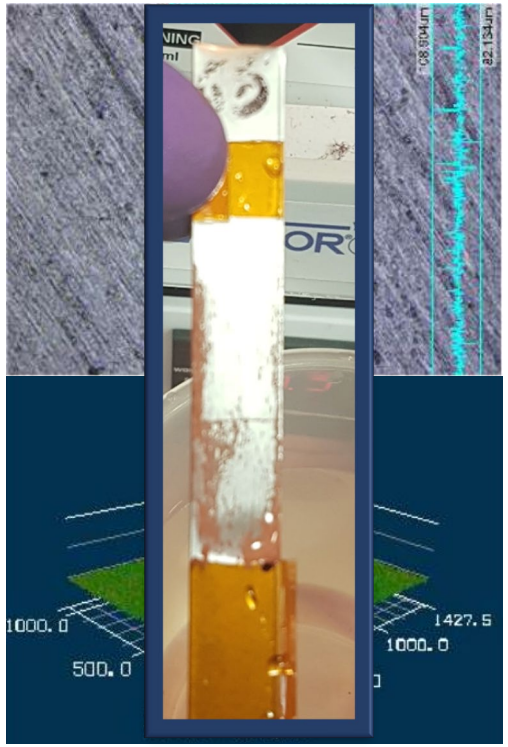
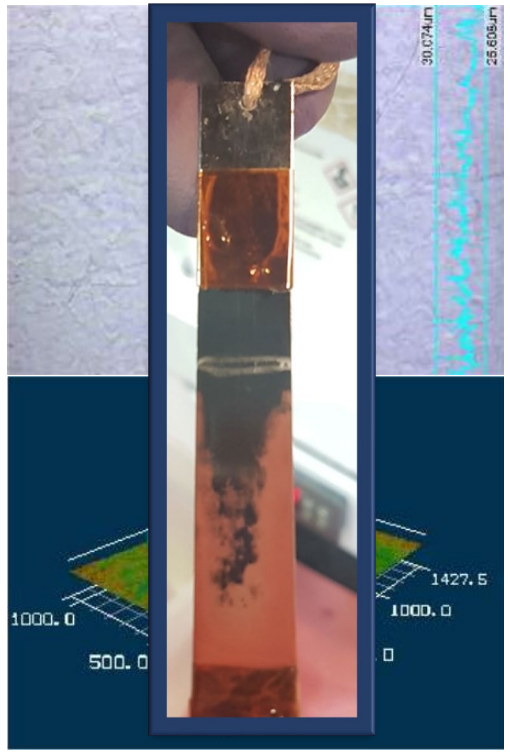
No tape

Tape

3: Substrate roughness/ peeling off relation

How roughness affects the adhesion of the deposited film?

How to properly treat Nb before the deposition?

No grinding, only HF	Grinding + HF	BCP + HF	Electro polishing + HF
			
R_a ≈1,8 µm	R_a ≈1,8 µm	R_a ≈ 1,5 µm	R_a ≈ 0,3 µm

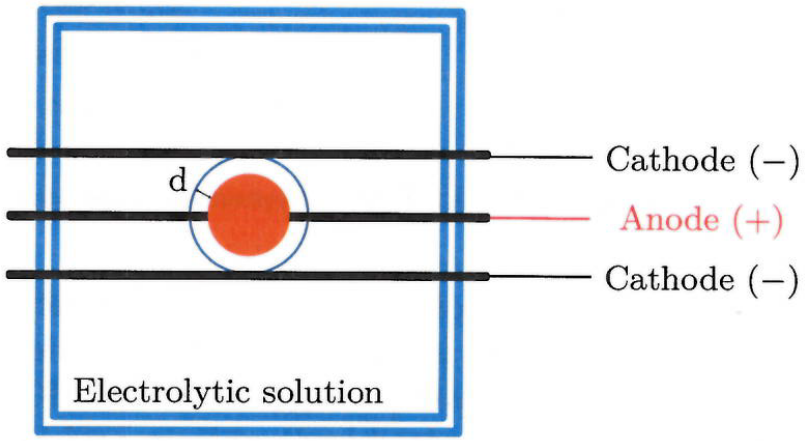
Grinding doesn't affect the roughness of the sample

The right level of roughness is needed to have good mechanical adhesion

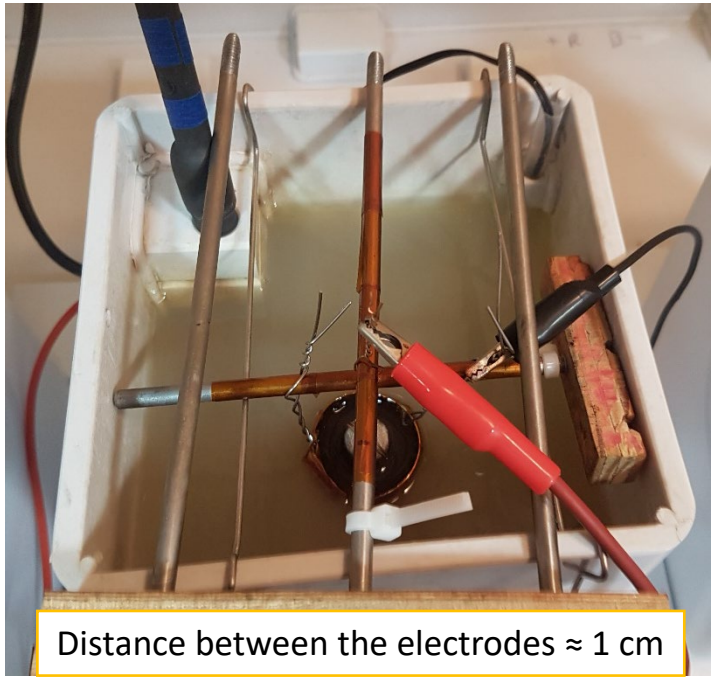
4: Testing the method feasibility on curved surfaces

Depositions on cylinders have been carried out once understood which are the best electrochemical conditions for plates

2 electrode geometry

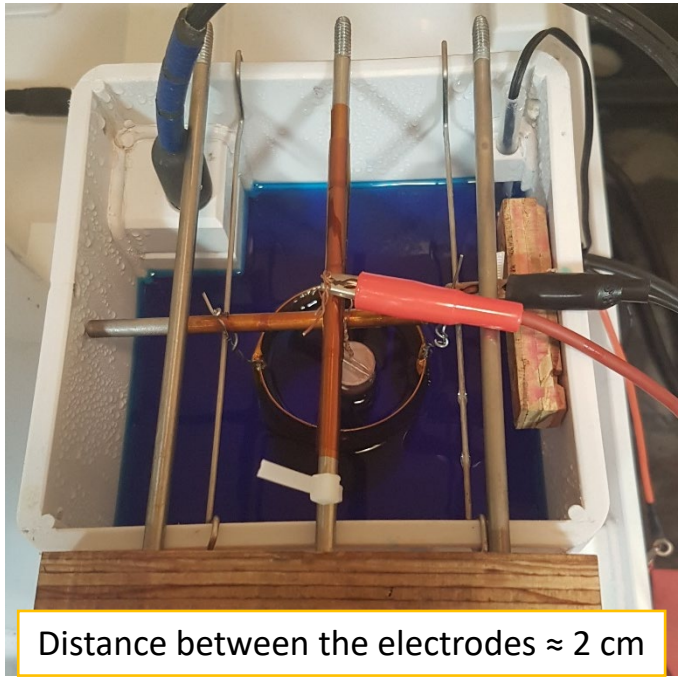


Small cylinders



Distance between the electrodes ≈ 1 cm

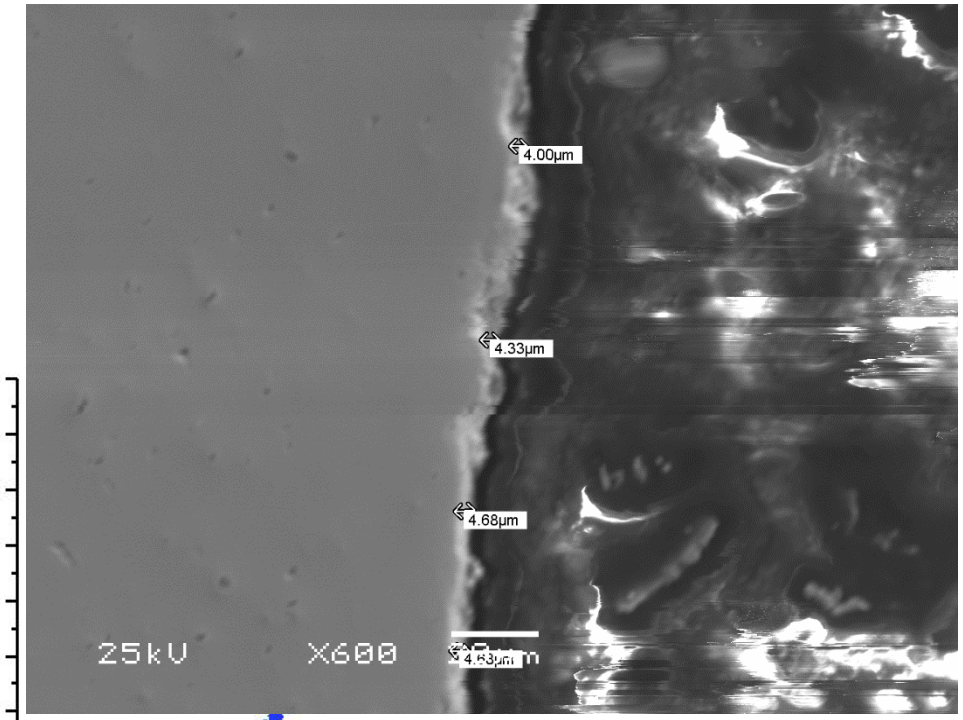
Large cylinders

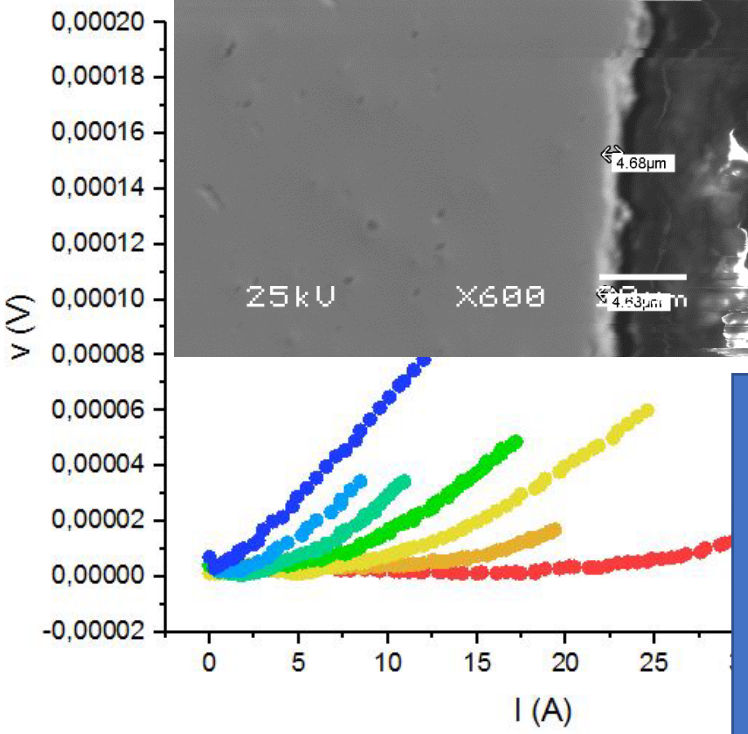


Distance between the electrodes ≈ 2 cm

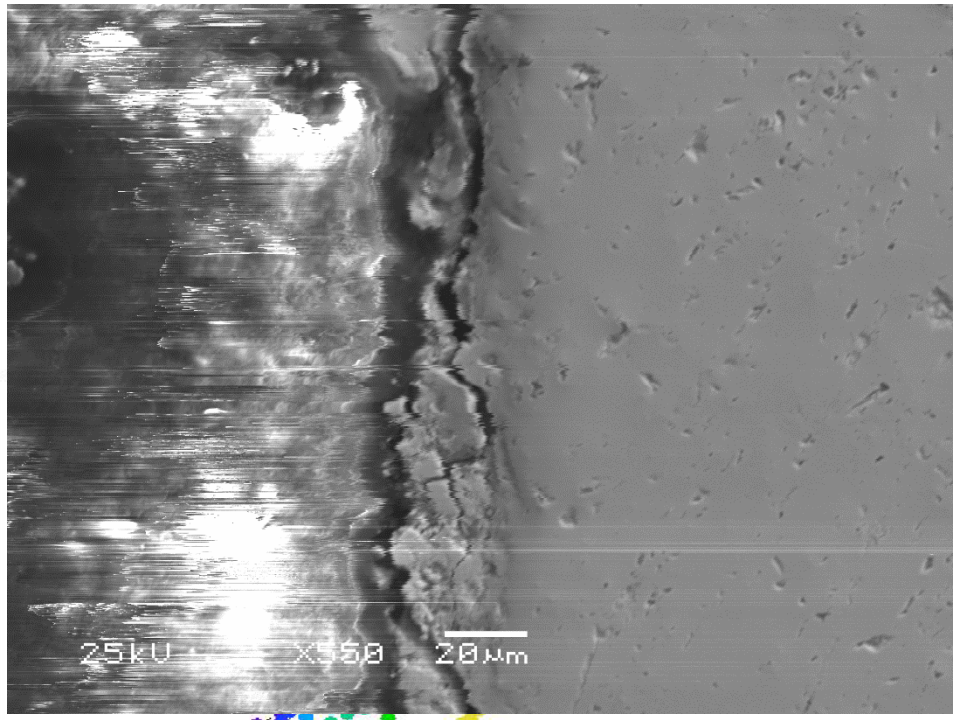
Superconductivity tests

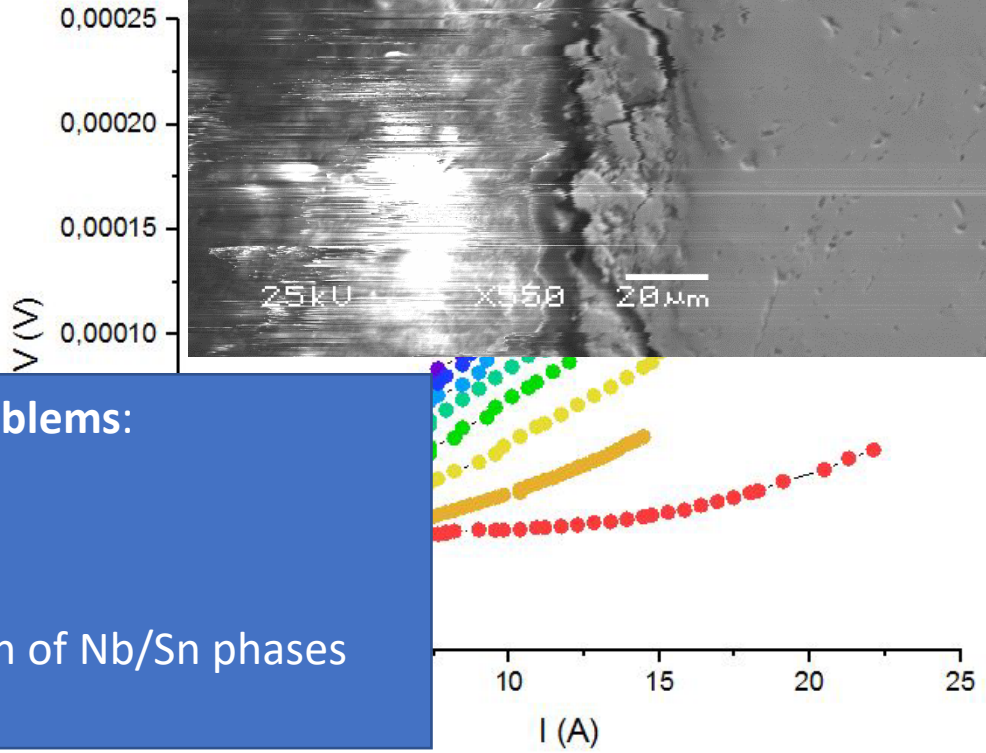
R 46





R 47





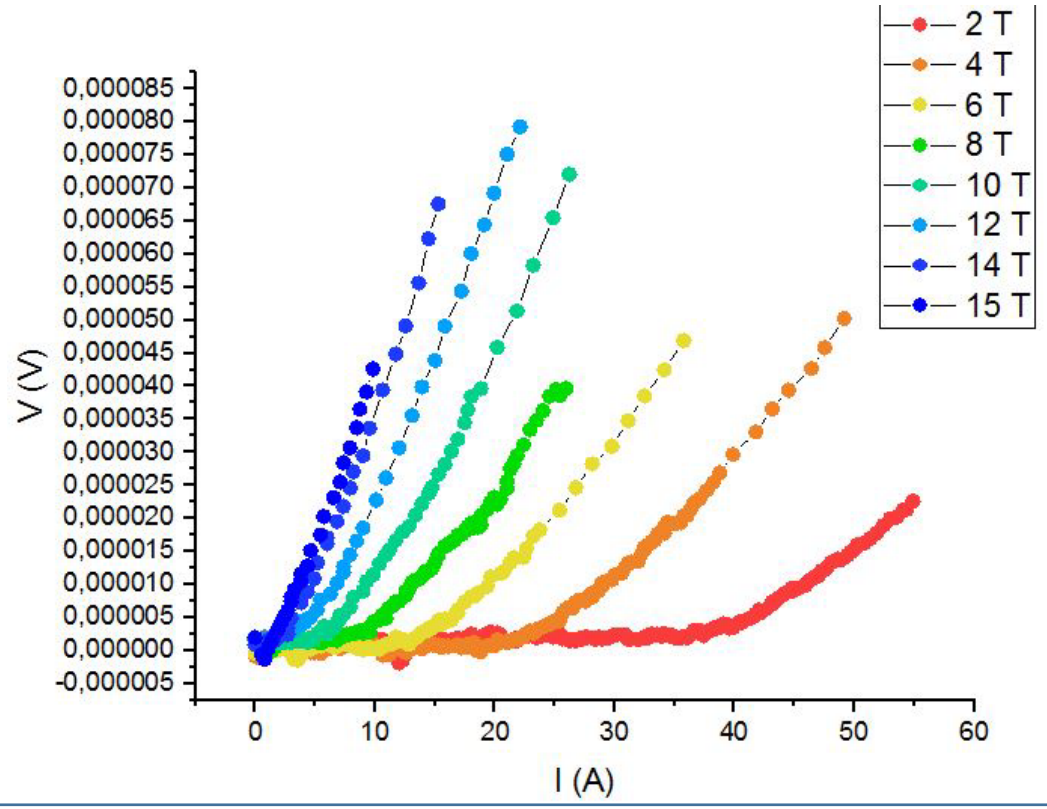
Main problems:

- Small thickness
- Inhomogeneity
- Maybe the formation of Nb/Sn phases other than Nb_3Sn

Superconductivity tests

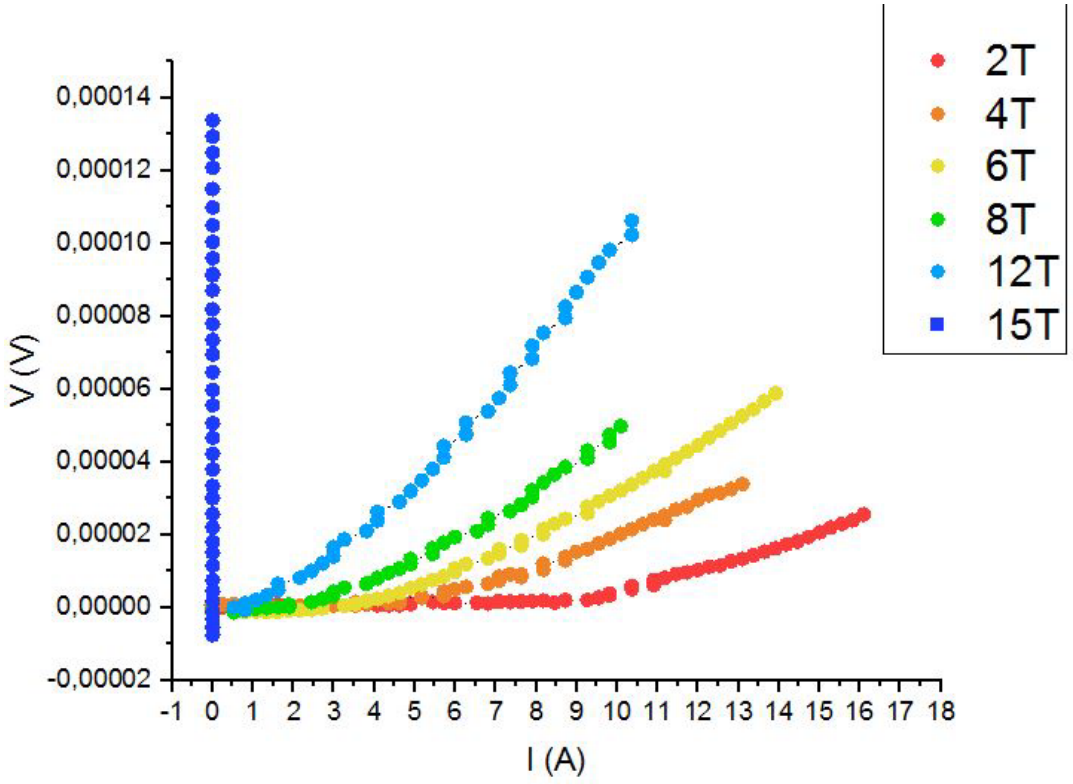
R 57

	1 step	2 step	3 step
J (mA/cm²)	54	45	16
t (s)	1.56	11.56	30.00



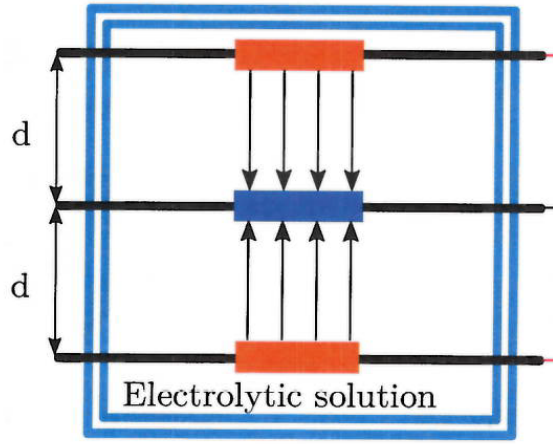
R 63

	1 step	2 step	3 step
J (mA/cm²)	66	40	12
t (s)	2	13.5	15.00



What's next?

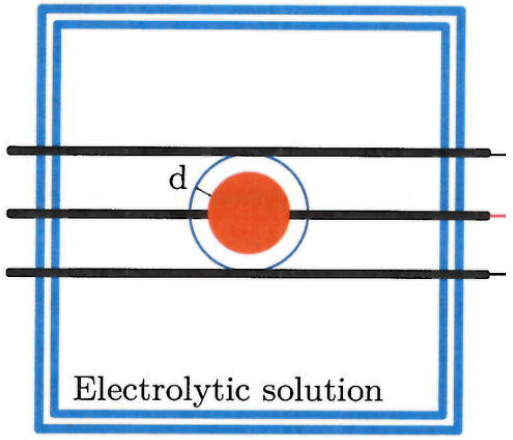
Deposition of new flat samples



Is this procedure highly reproducible?

Can geometry related problems be avoided?

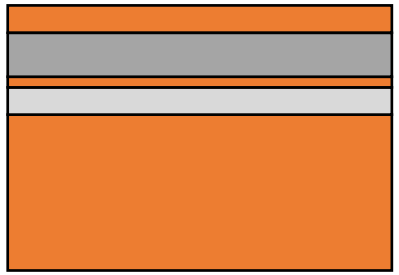
Deposition on new cylinders



Can curved and flat shapes be plated with the same exp. conditions?

Are the results in terms of morphology and superconductivity the same?

Deposition on Nb/Cu disks



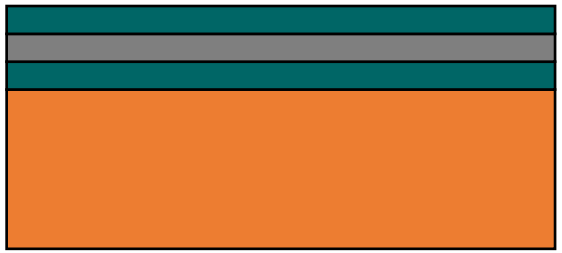
Cu barrier layer
Sn layer
Cu seed layer
Sputtered Nb layer
Cu substrate

Thermal treatment

If Sn < stoichiometric

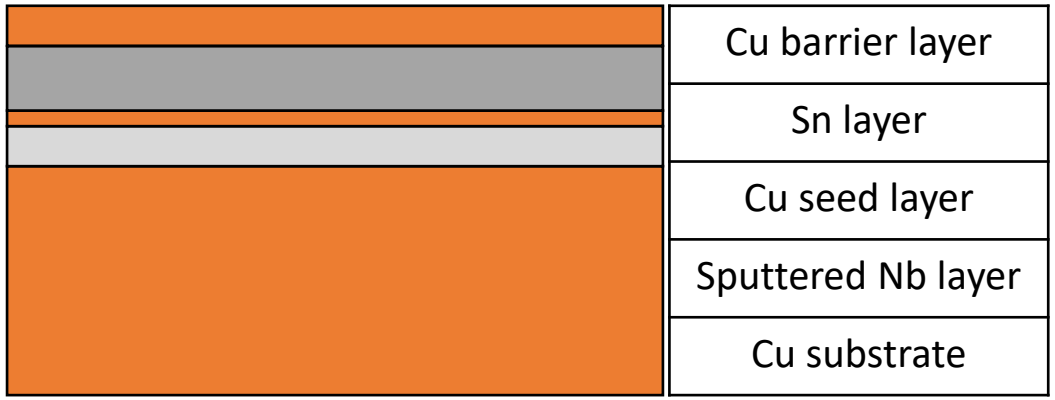
Bronze
Nb_3Sn
Nb
Cu

If Sn > stoichiometric

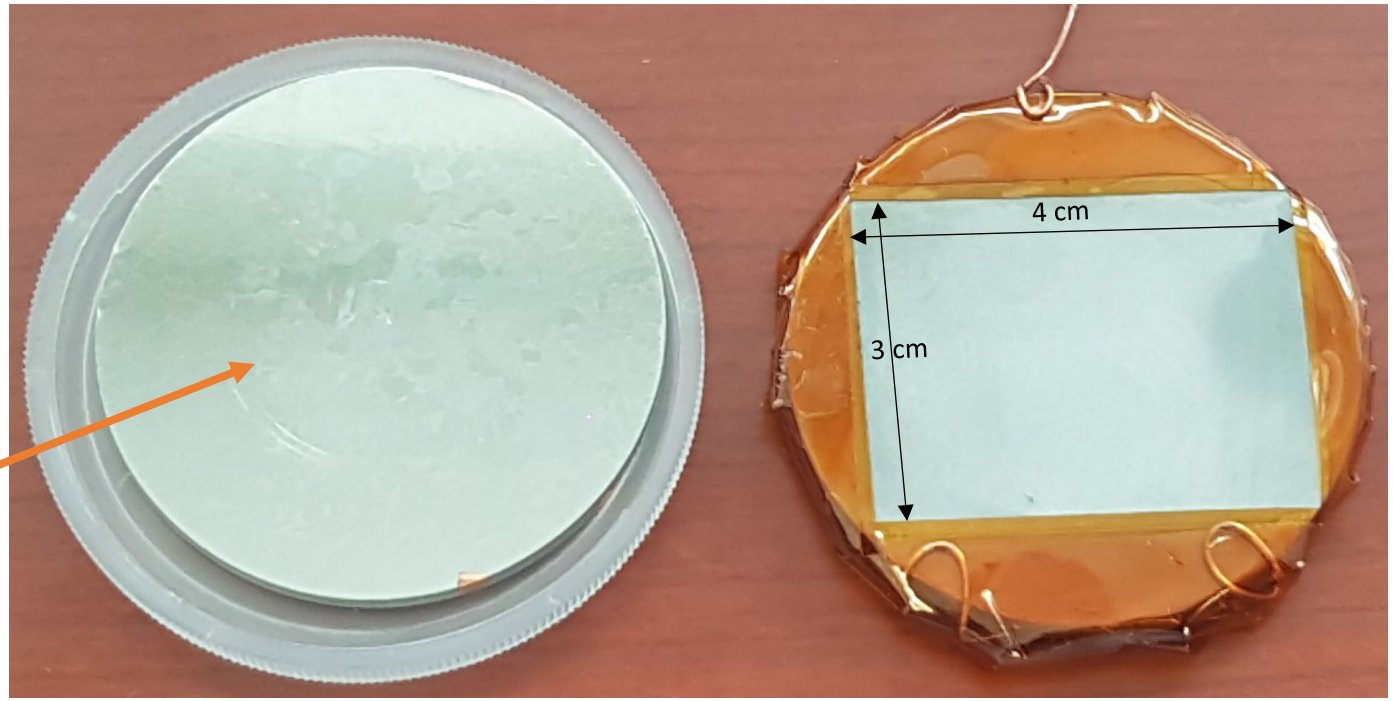


Bronze
Nb_3Sn
Bronze
Cu

5:Deposition on Nb/Cu substrates



Sputtered Nb on Cu



Extremely flat surface:
$R_a \approx 0{,}5\ \mu m$

Grinding procedure is needed