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Task 9.3 @ INFN

iFAST 1st Annual meeting – CERN, May 2022

WP9 5th meeting

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Nb_3Sn on planar samples

4" planar magnetron

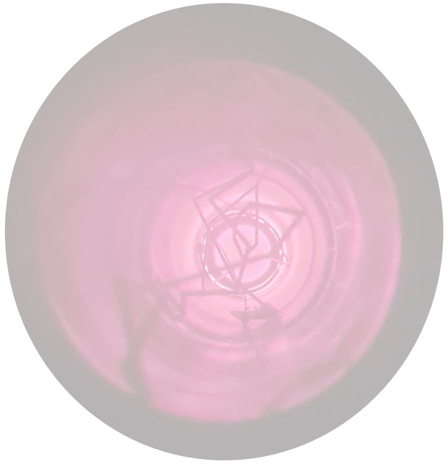
Nb_3Sn cylindrical target

Via liquid tin diffusion (dipping)

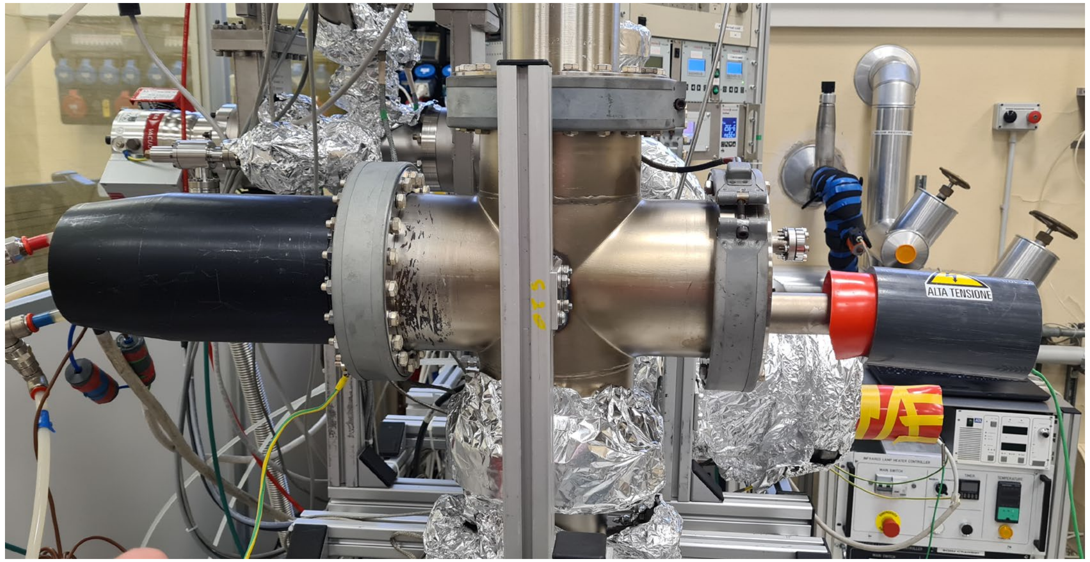
6 GHz cavities

RF characterization

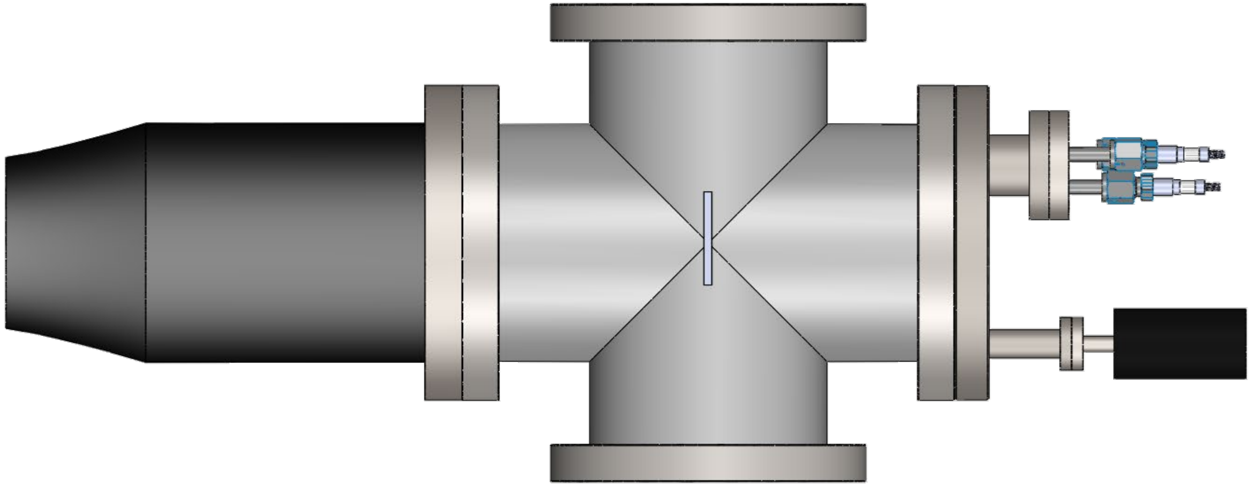
Nb$_3$Sn on planar samples	Nb$_3$Sn cylindrical target	6 GHz cavities
4" planar magnetron	Via liquid tin diffusion (dipping)	RF characterization

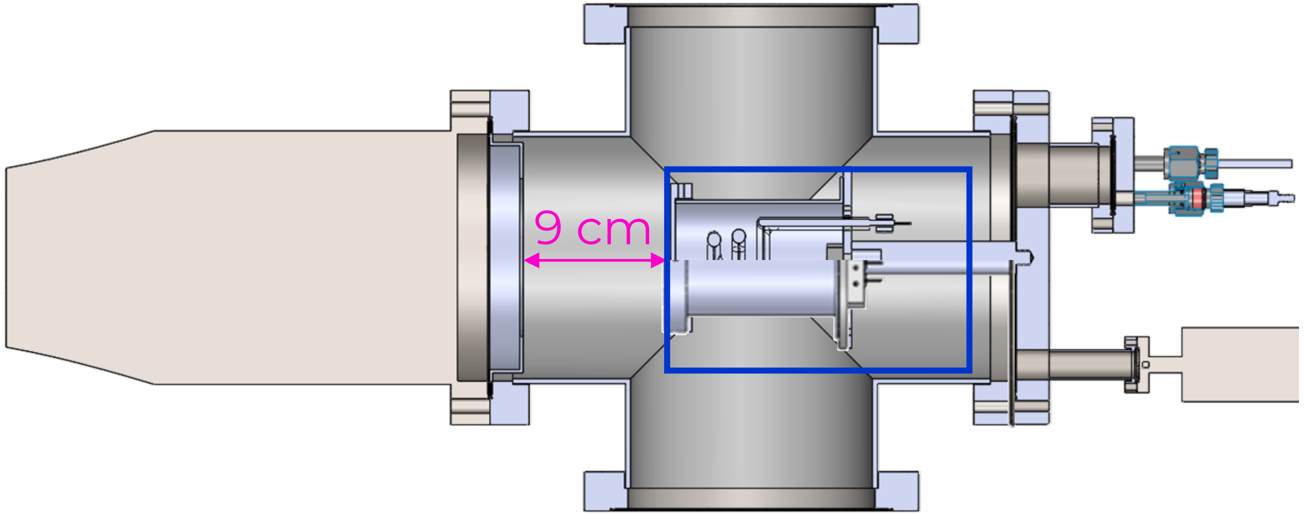


Nb_3Sn on planar samples by DCMS



LNL Nb_3Sn coating system

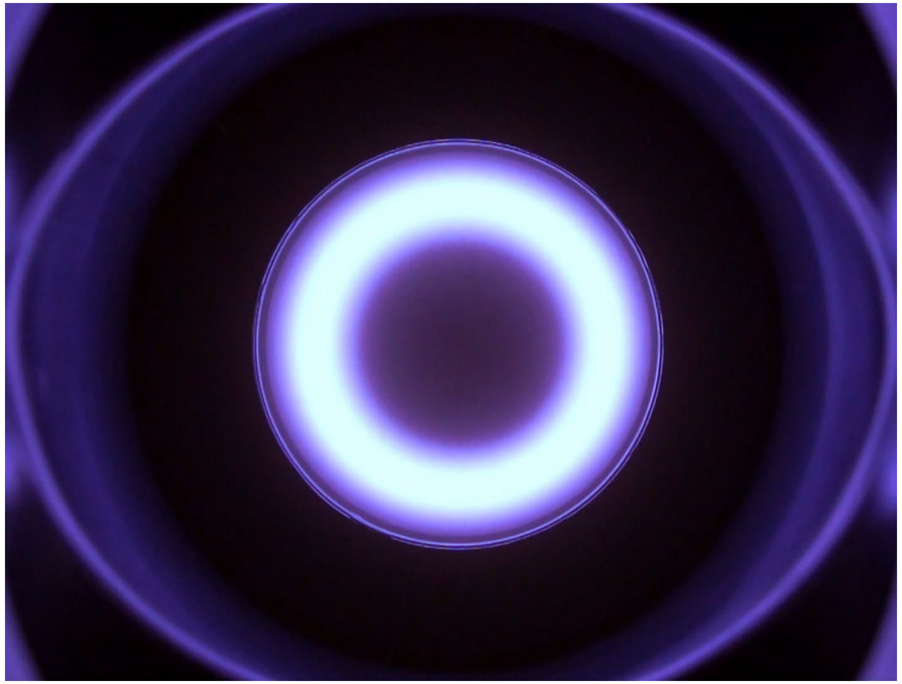




Nb_3Sn on planar samples by DCMS

Process optimization

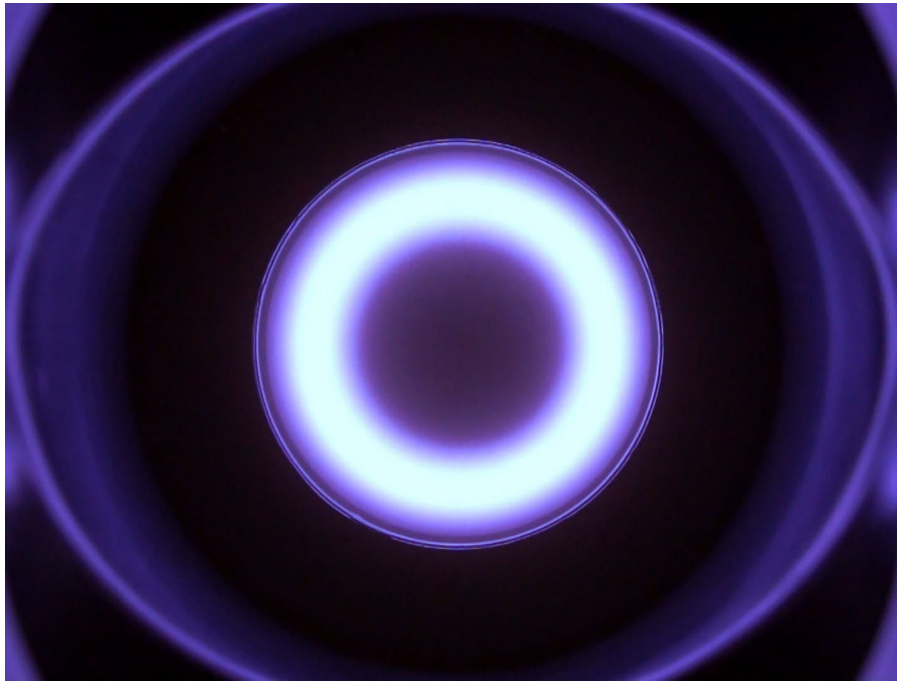
- Power
- Argon pressure
- Substrate temperature
- Substrate material
- UHV annealing



Nb_3Sn on planar samples by DCMS
Coating parameters

Process optimization

- Power — 0,25 A, 3mA/cm² — 550 V — 140 W, 1,7 W/cm²
- Argon pressure — $3x10^{-3}$ mbar
- Substrate temperature — 730°C
- Substrate material
- UHV annealing



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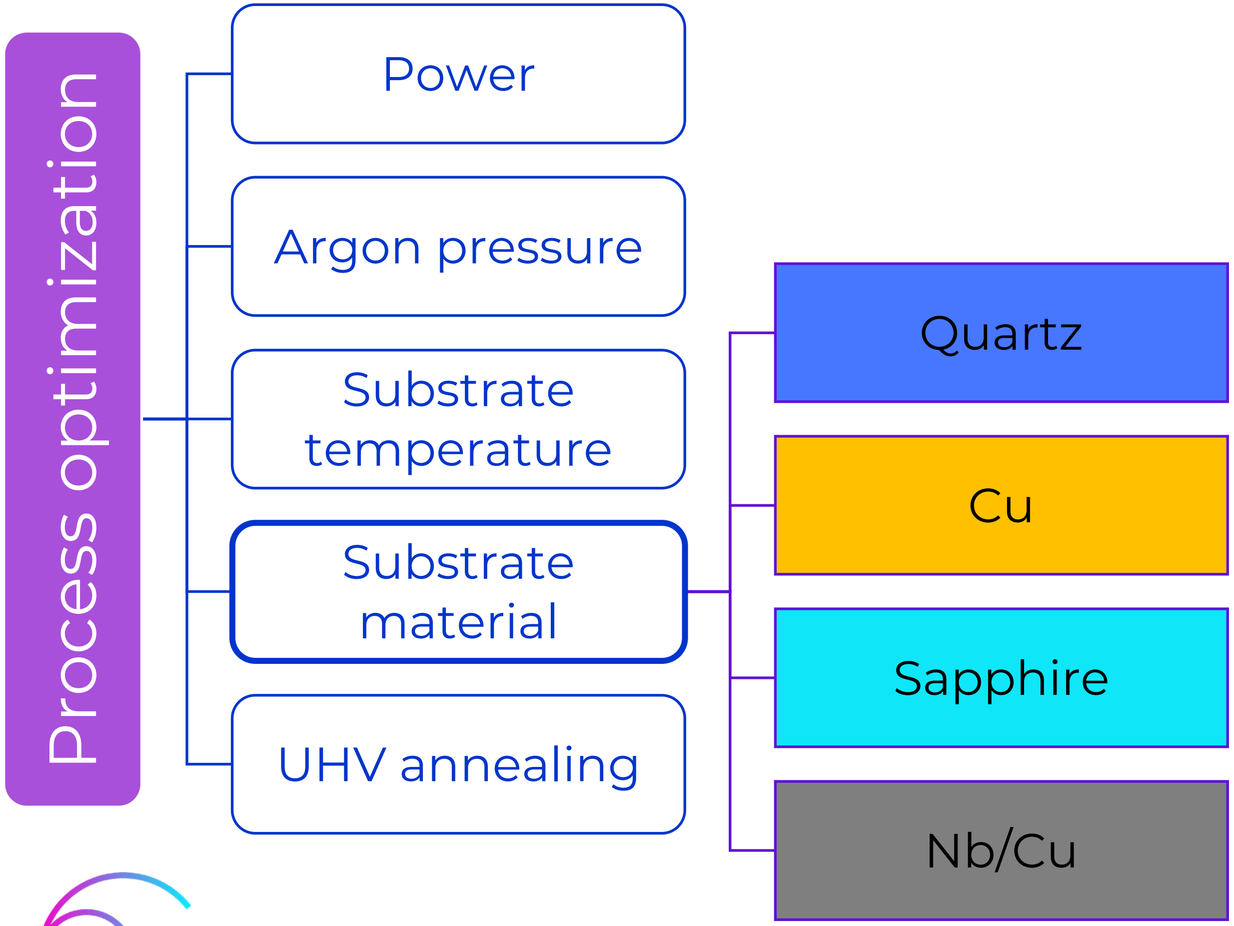
Nb_3Sn on planar samples by DCMS
Substrates

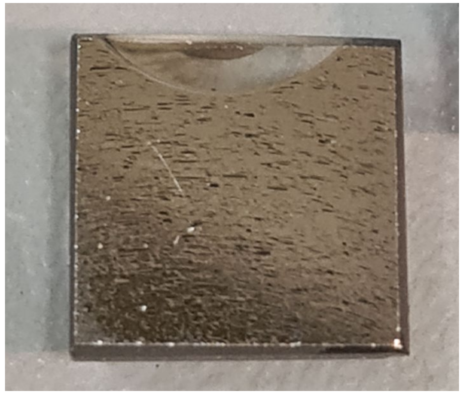


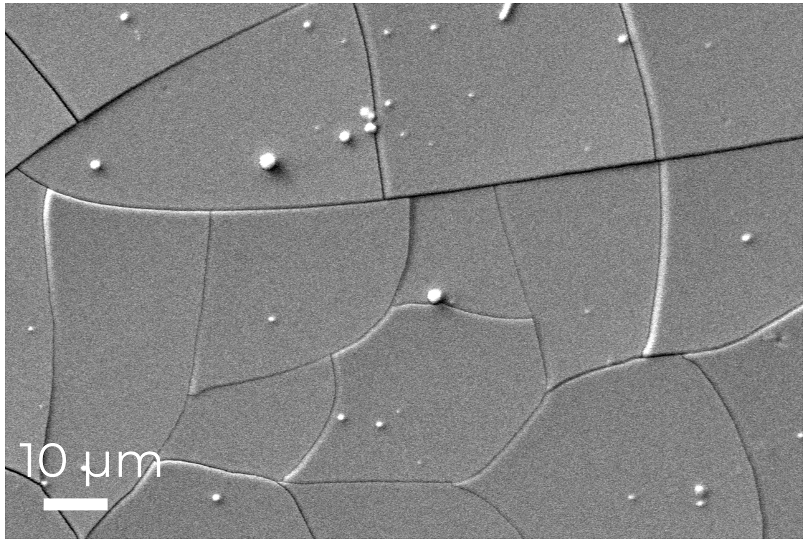
Nb_3Sn on planar samples by DCMS

Substrate effect on morphology

Process optimization

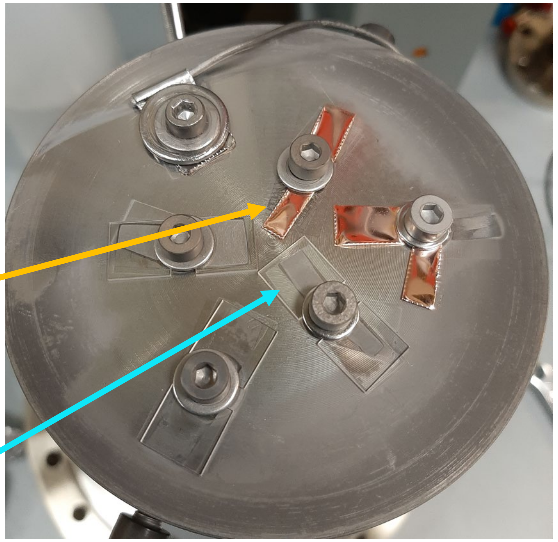
- Power
- Argon pressure
- Substrate temperature
- **Substrate material**
  - Quartz — Poor adhesion
    - α-quartz → β-quartz phase transition around **573 ℃**
  - Cu
  - Sapphire
  - Nb/Cu
- UHV annealing





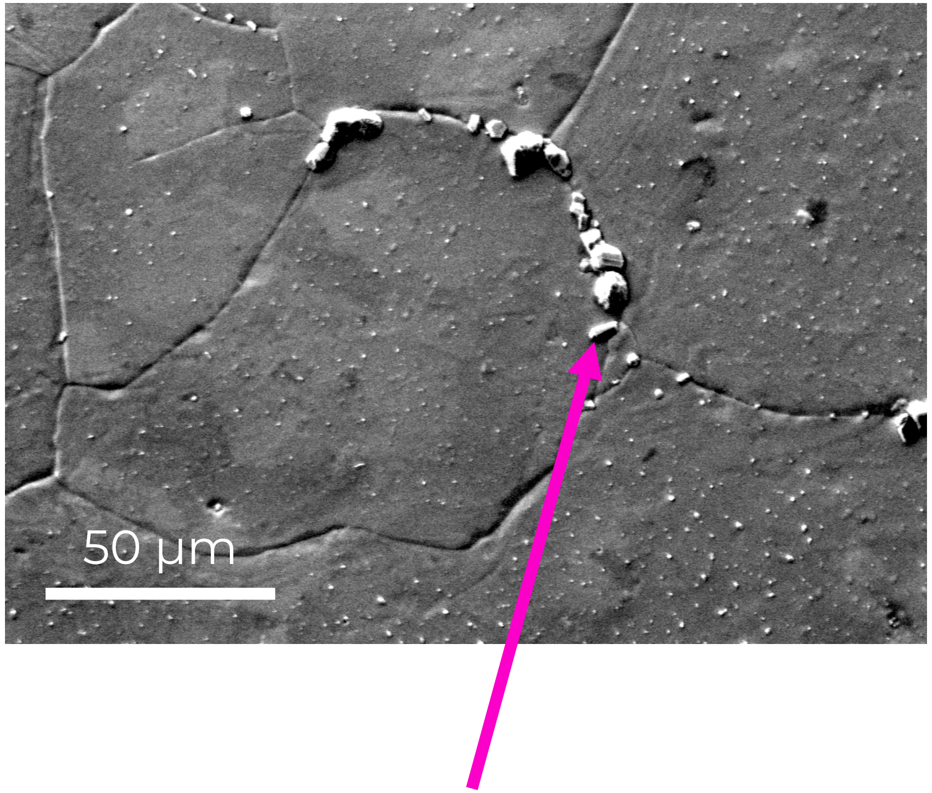
Nb_3Sn on planar samples by DCMS
Substrate effect on morphology

Process optimization
- Power
- Argon pressure
- Substrate temperature
- Substrate material
  - Cu
  - Sapphire
  - Nb/Cu
- UHV annealing



Nb_3Sn on planar samples by DCMS
Substrate effect on morphology

Process optimization
- Power
- Argon pressure
- Substrate temperature
- **Substrate material**
  - Cu
  - Sapphire
  - Nb/Cu
- UHV annealing

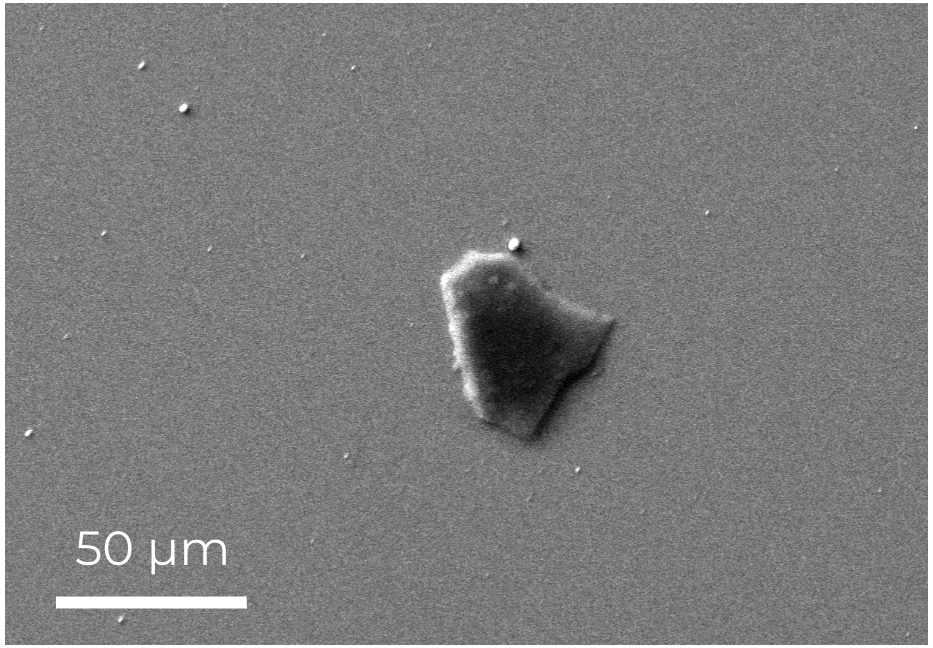


Cu inclusions in cracks

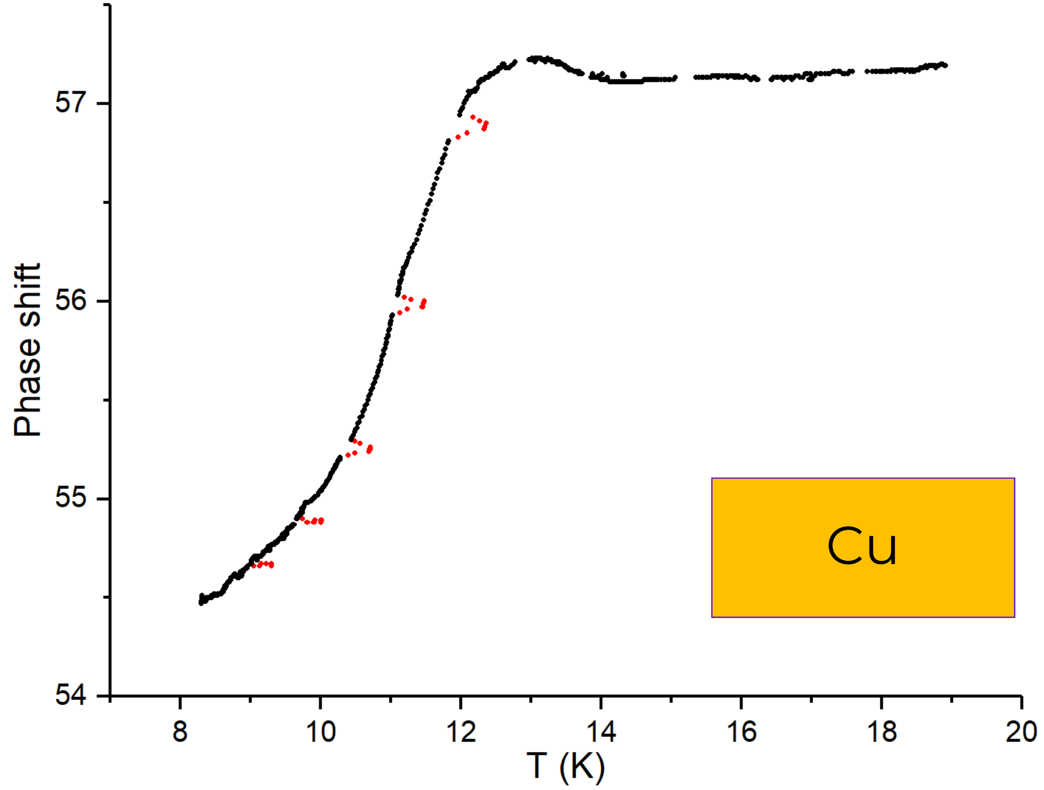
Nb_3Sn on planar samples by DCMS
Substrate effect on morphology

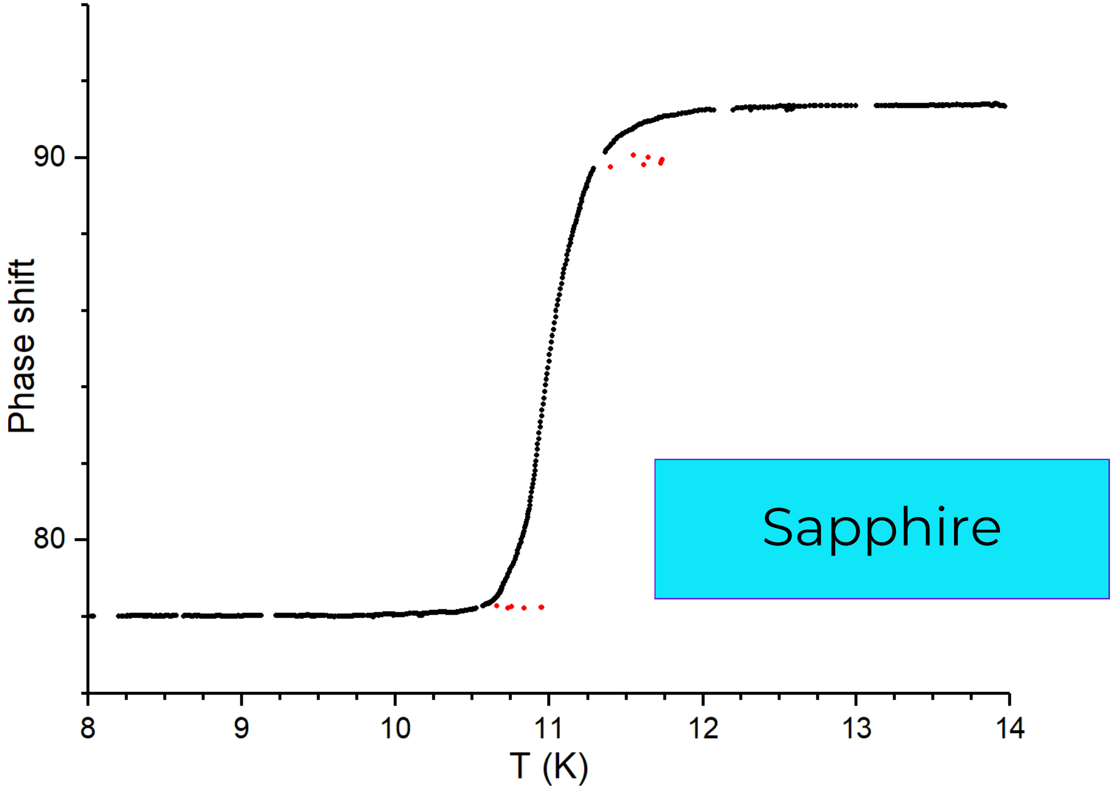
Process optimization

- Power
- Argon pressure
- Substrate temperature
- **Substrate material**
  - Cu
  - Sapphire
  - Nb/Cu
- UHV annealing



Nb_3Sn on planar samples by DCMS
Substrate effect on Tc





24 % Sn	at% by EDS (@20kV~1,2µm)	22 % Sn
~11 K	Tc (inductive)	11,±0,6 K

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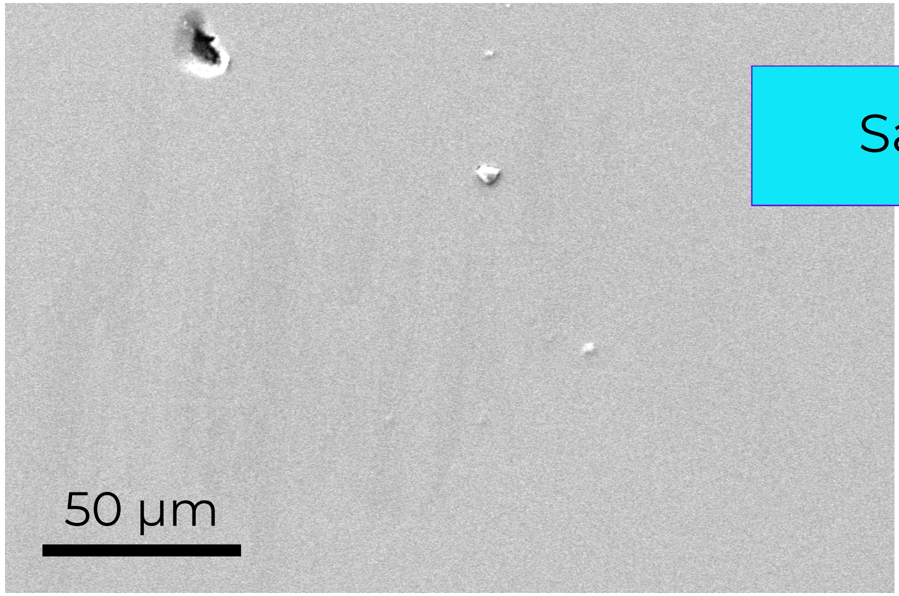
Nb_3Sn on planar samples by DCMS
Annealing effect

Process optimization

- Power
- Argon pressure
- Substrate temperature
- Substrate material
- UHV annealing
  - Temperature → 750°C (**higher point**: too high for copper cavity)
  - Duration → 24 h + slow cooling down



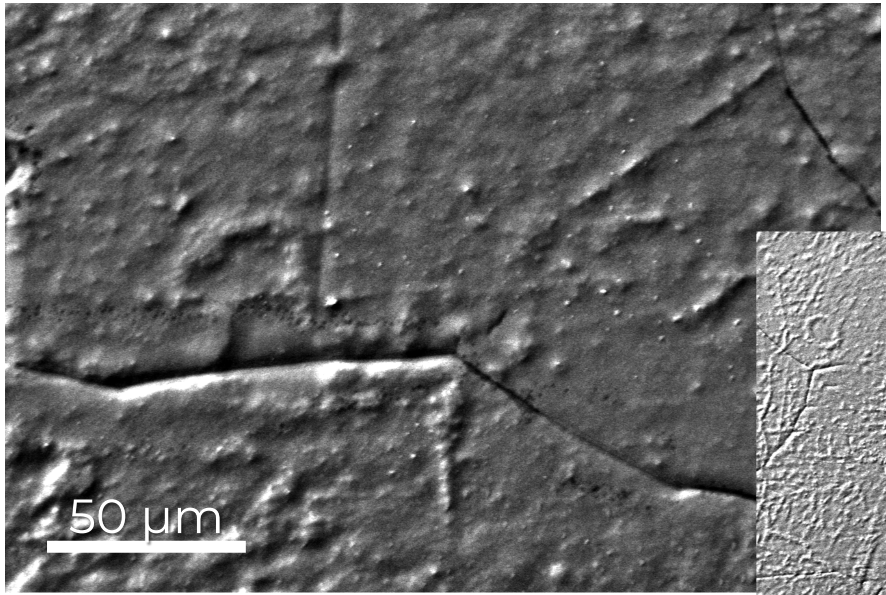
Sapphire

24 h annealing

Nb_3Sn on planar samples by DCMS
Annealing effect

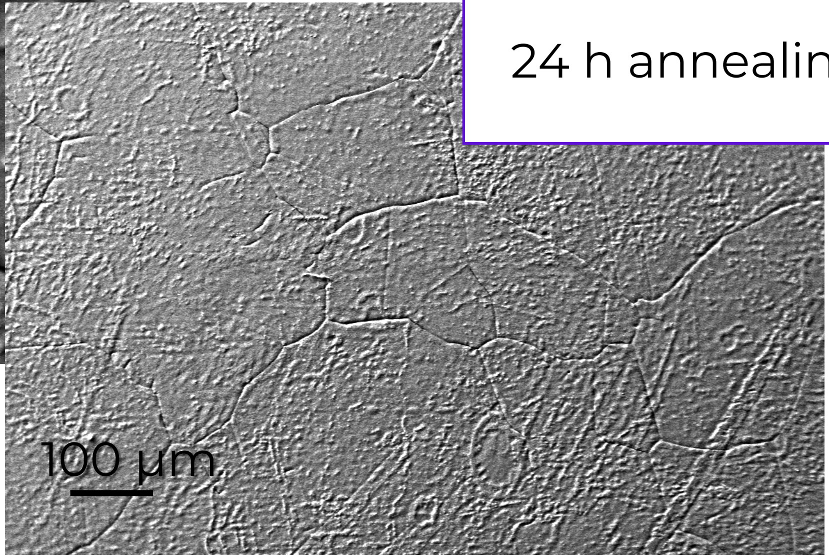
Process optimization

- Power
- Argon pressure
- Substrate temperature
- Substrate material
- UHV annealing
  - Temperature → 750°C
  - Duration → 24 h + slow cooling down



Cu

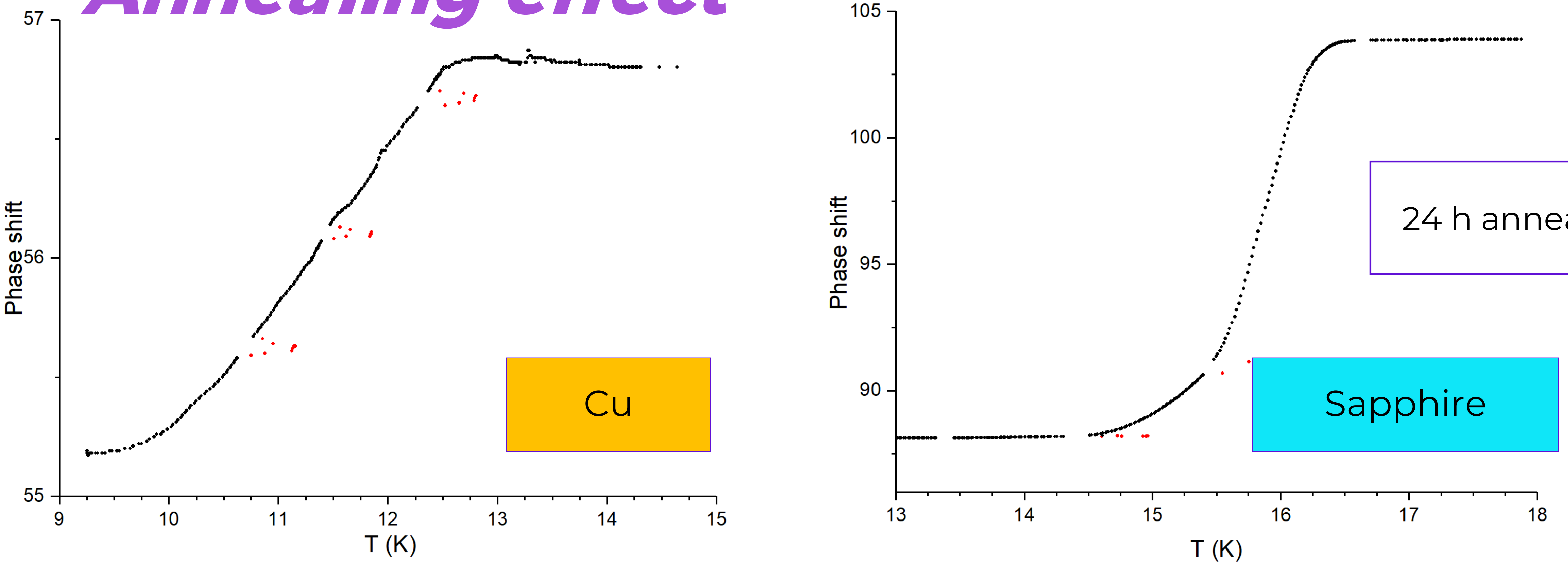
24 h annealing



No Cu inclusions

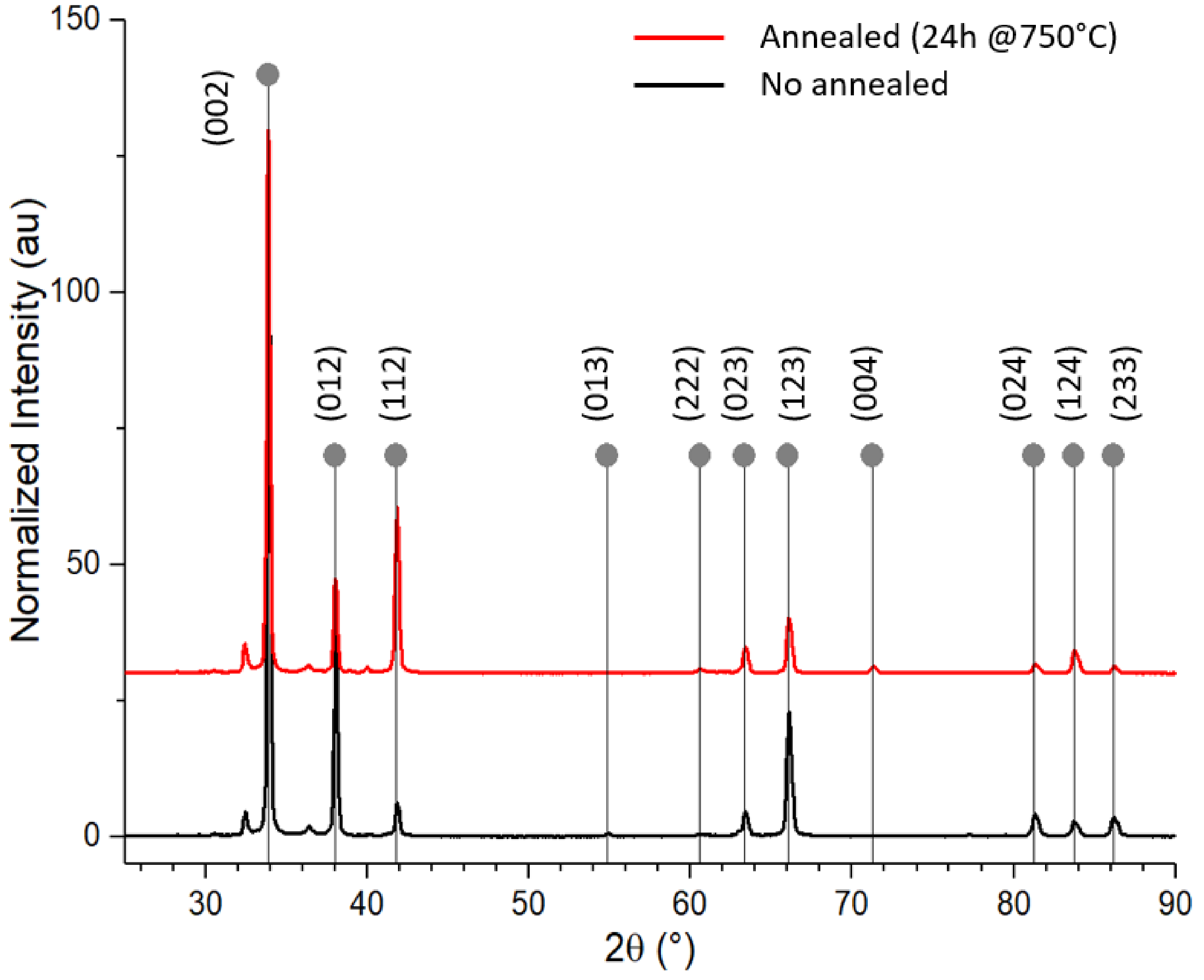
Nb_3Sn on planar samples by DCMS

Annealing effect



23 % Sn	at% by EDS (@20kV~1,2µm)	22 % Sn
~11,5 K	Tc (inductive)	15,8±0,8 K

Nb_3Sn on planar samples by DCMS
Annealing effect

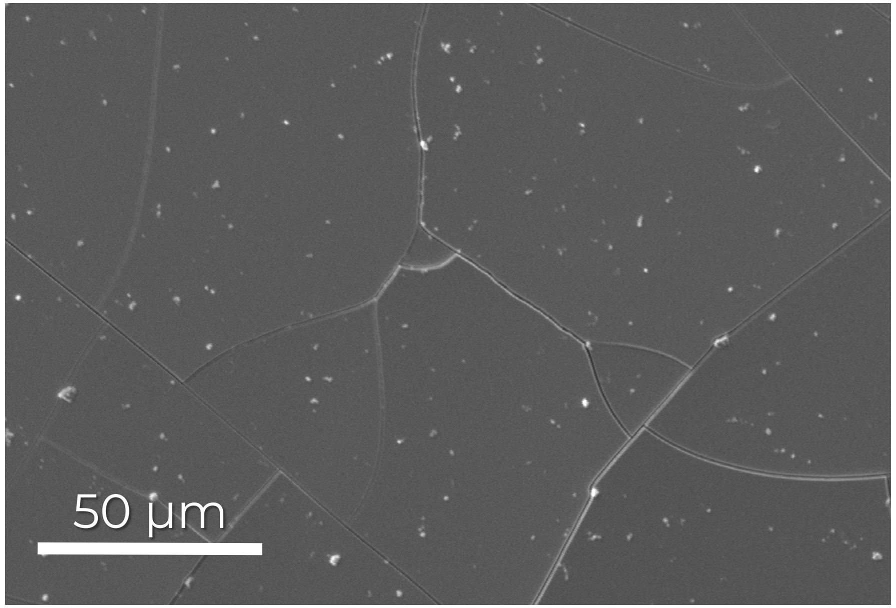


Sapphire

24 h annealing

Nb_3Sn on planar samples by DCMS
Annealing time effect: 24 → 48 h

Process optimization
- Power
- Argon pressure
- Substrate temperature
- Substrate material
- **UHV annealing**
  - Temperature → 750°C (**higher point**: too high for copper cavity)
  - Duration → 48 h



Sapphire

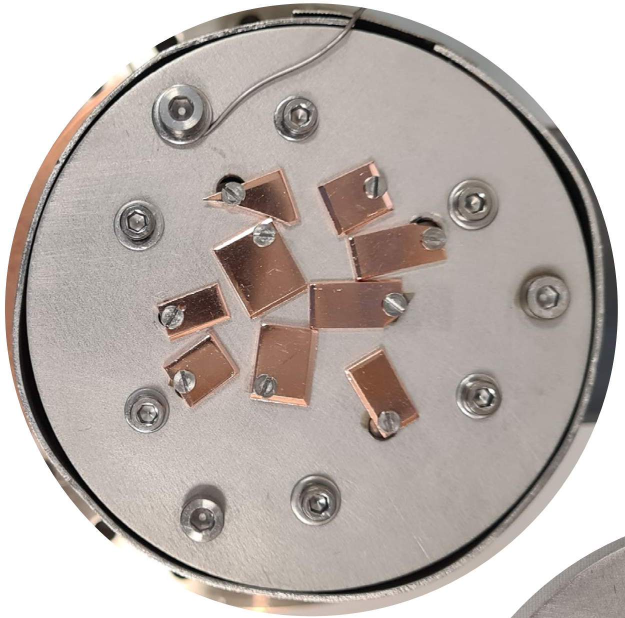
48 h annealing

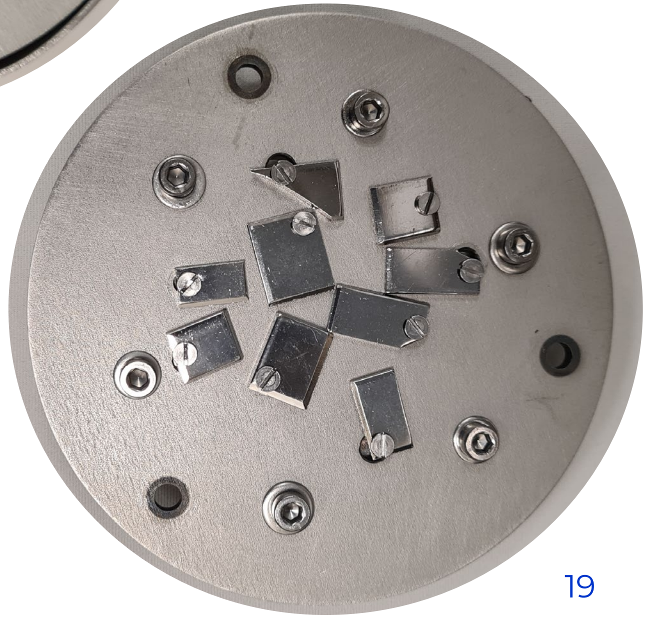
Film stressed and cracked

Nb_3Sn on planar samples by DCMS
Nb Buffer Layer

Process optimization

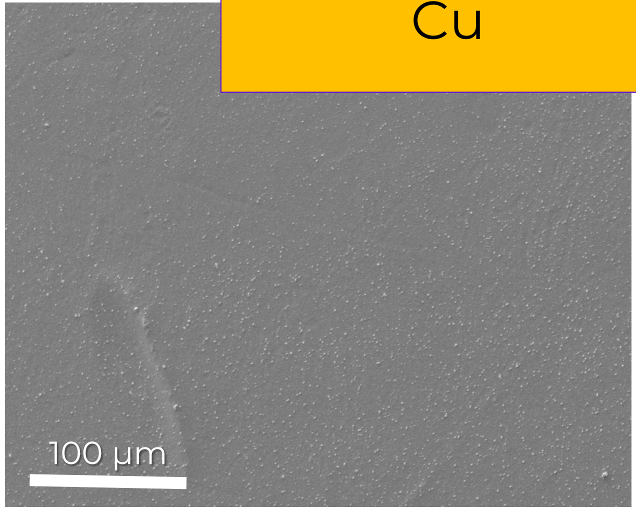
- Power
- Argon pressure
- Substrate temperature
- Substrate material
  - Cu
  - Sapphire
  - Nb/Cu
    - 500 nm
    - 1 µm
- UHV annealing



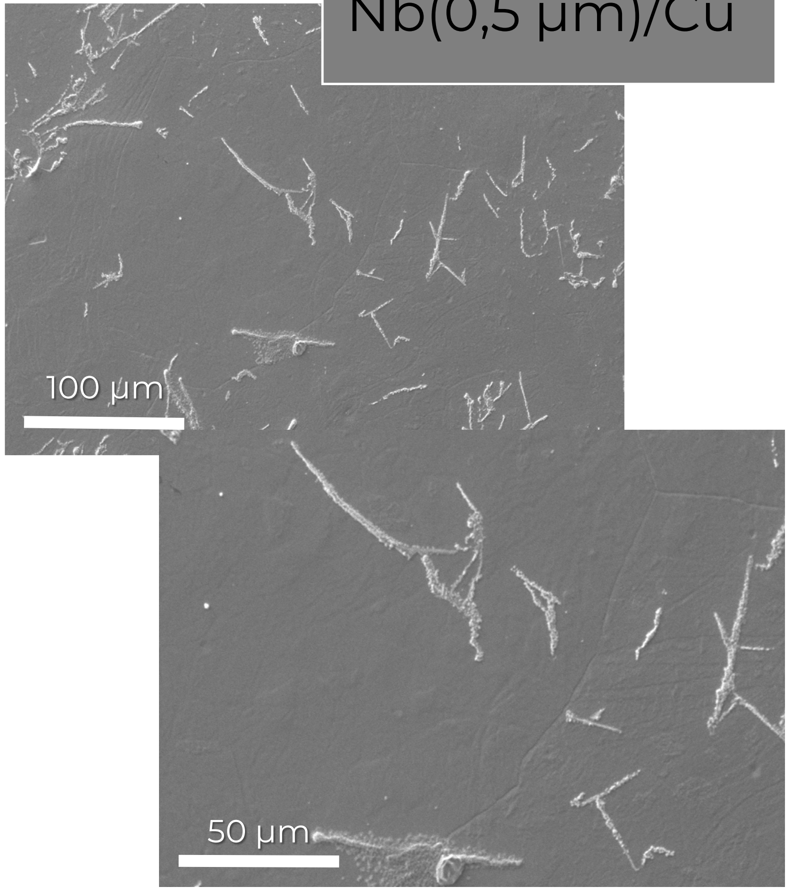


Nb_3Sn on planar samples by DCMS
Nb Buffer Layer

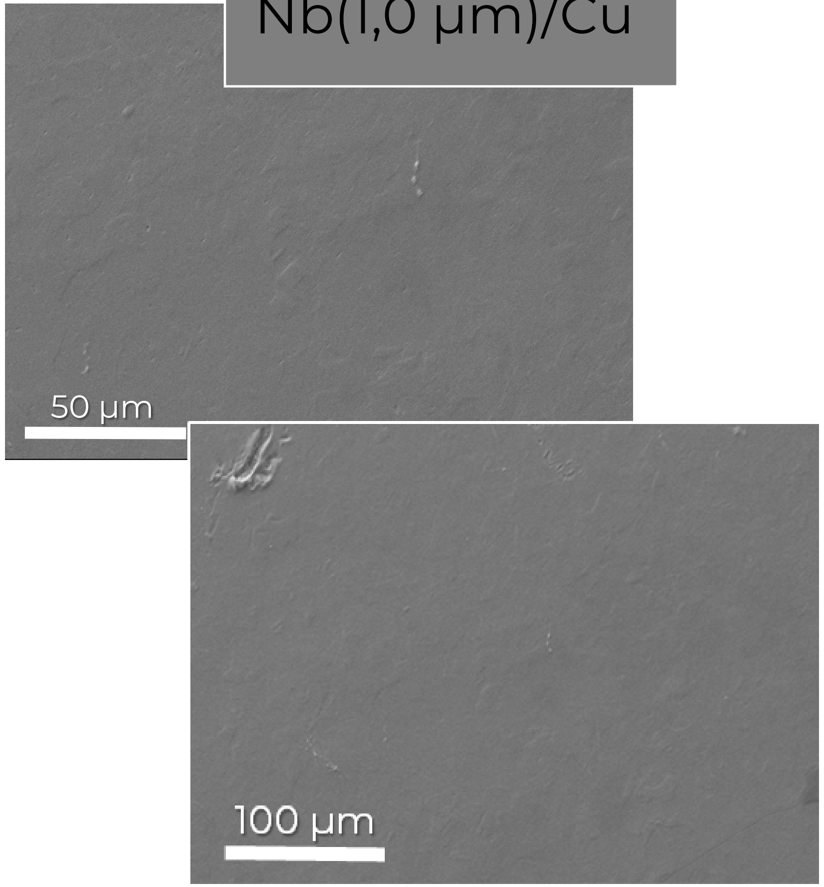
Cu



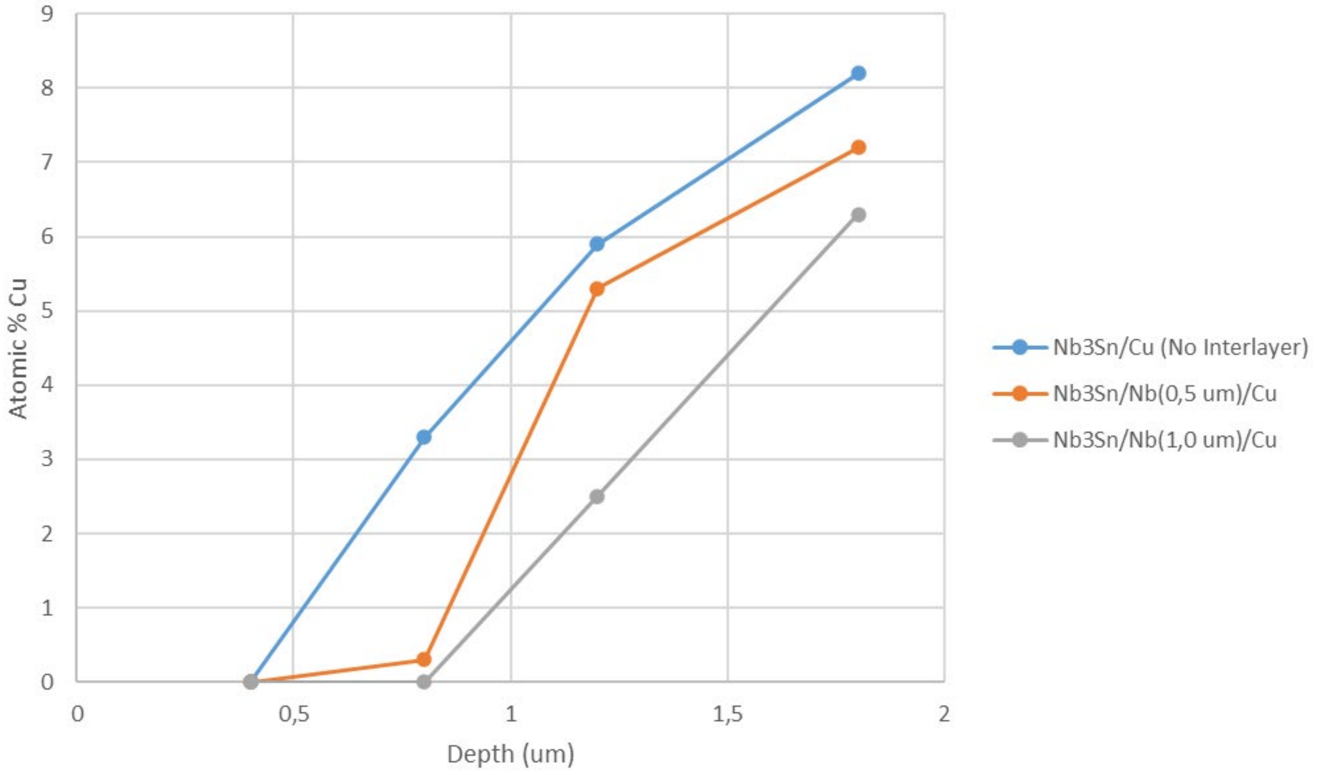
Nb(0,5 µm)/Cu



Nb(1,0 µm)/Cu



Nb_3Sn on planar samples by DCMS
Nb Buffer Layer



Copper interdiffusion

48 h annealing

Film thickness 1,5 μm

Atomic Sn % = ~22 %

Nb$_3$Sn on planar samples

4" planar magnetron

Nb$_3$Sn cylindrical target

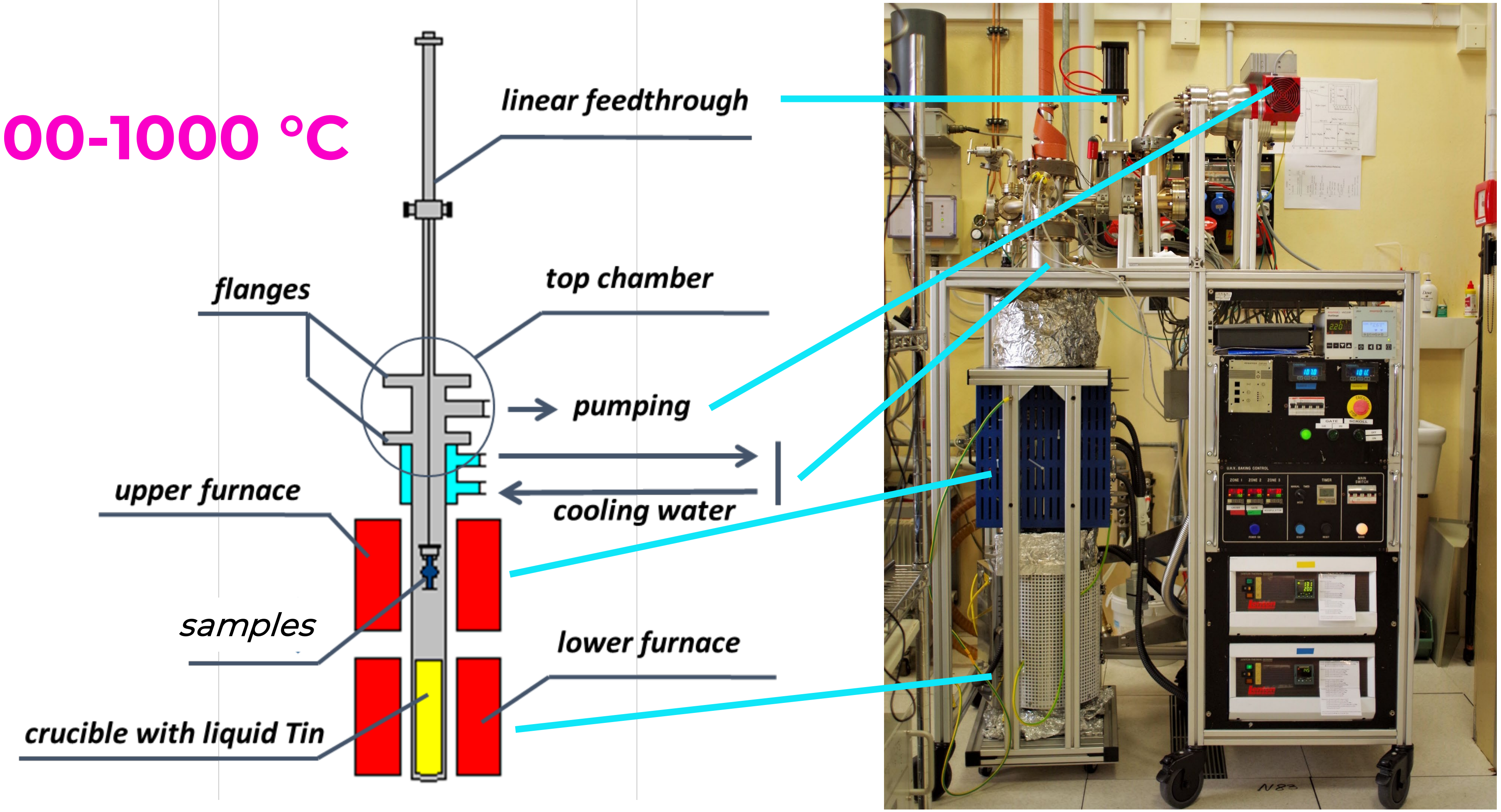
Via liquid tin diffusion (dipping)

6 GHz cavities

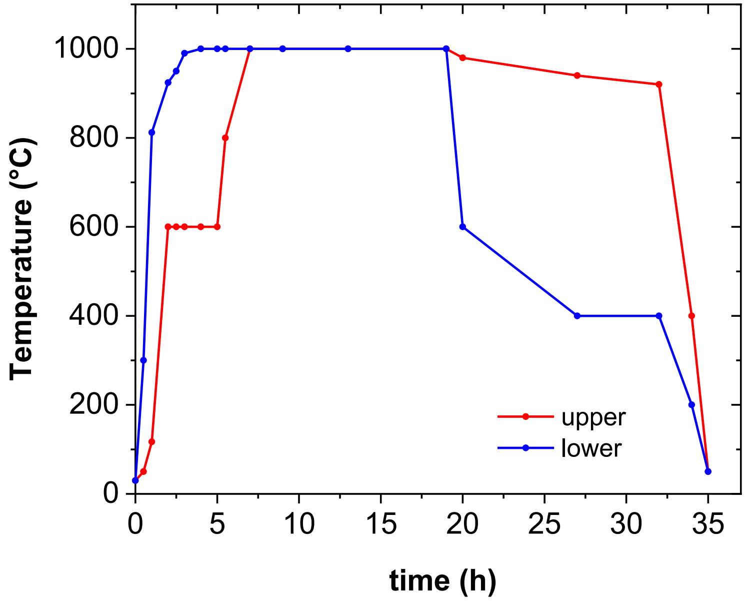
RF characterization

Nb_3Sn Cylindrical target production **by tin liquid diffusion (dipping)**

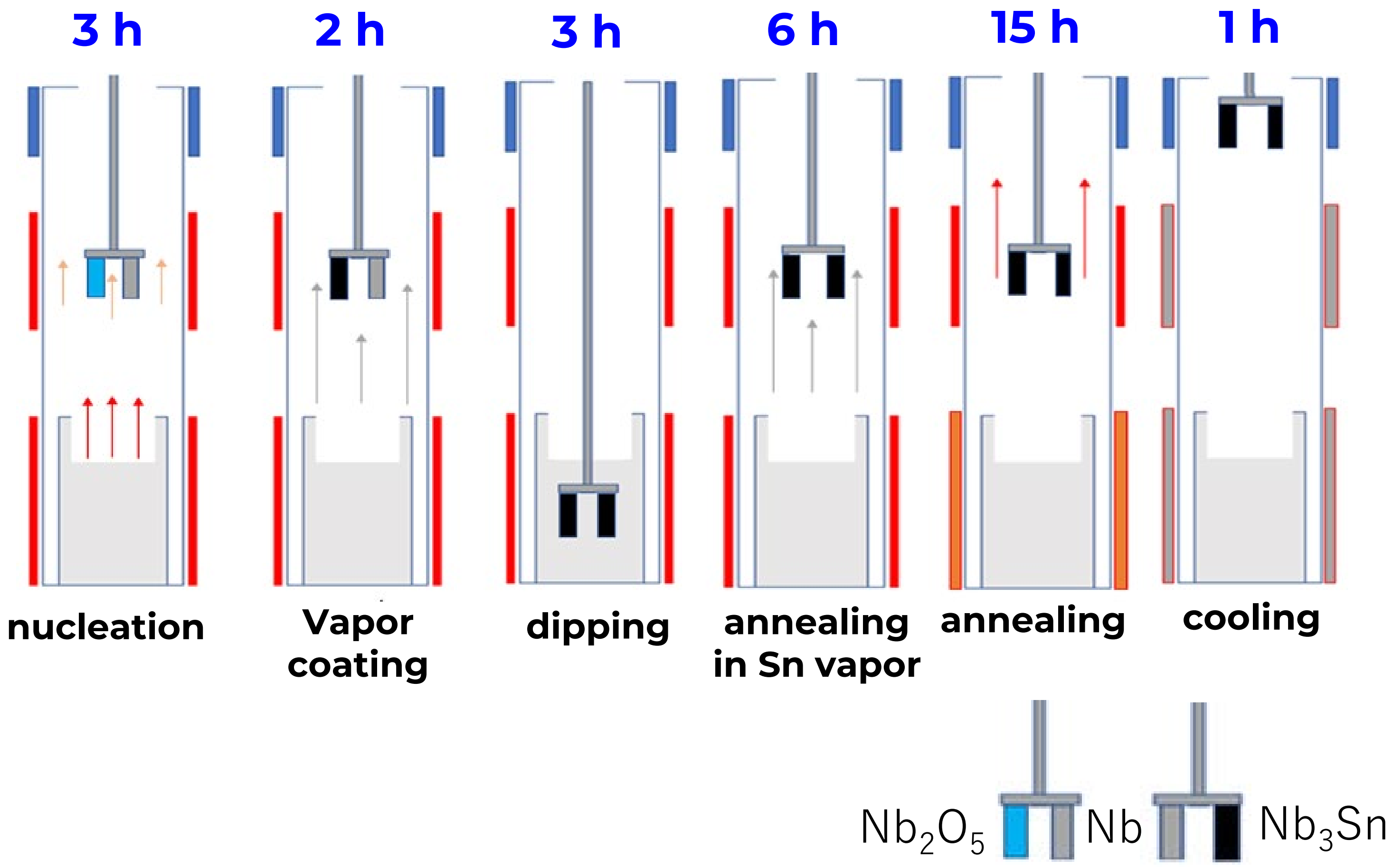
T ~ 900-1000 °C



Nb_3Sn Cylindrical target production **by tin liquid diffusion (dipping)**



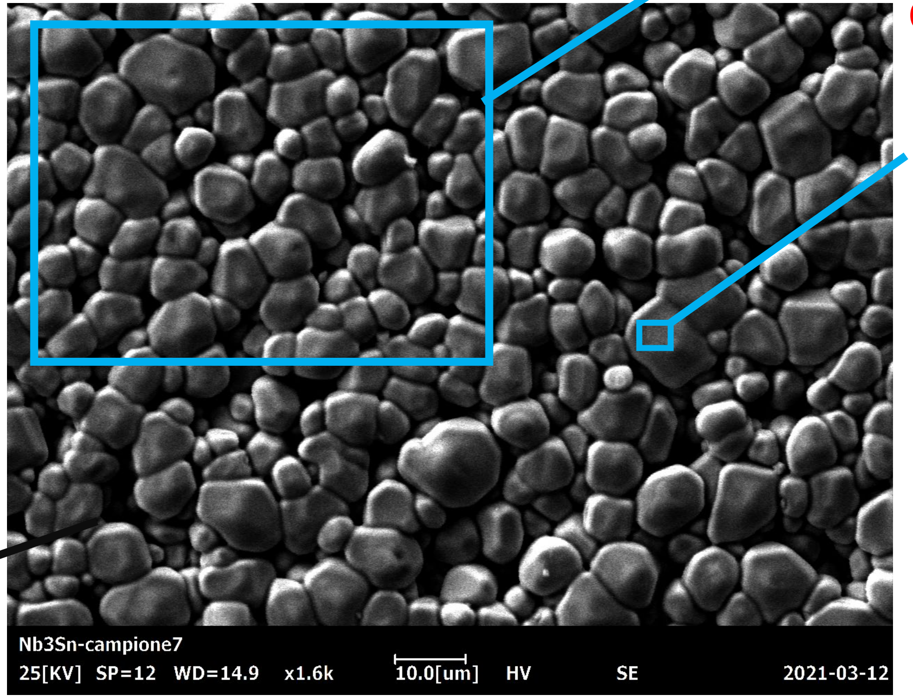
Temperature furnaces profile



Nb_3Sn Cylindrical target production **by tin liquid diffusion (dipping)**

Superficial Chromium contamination!

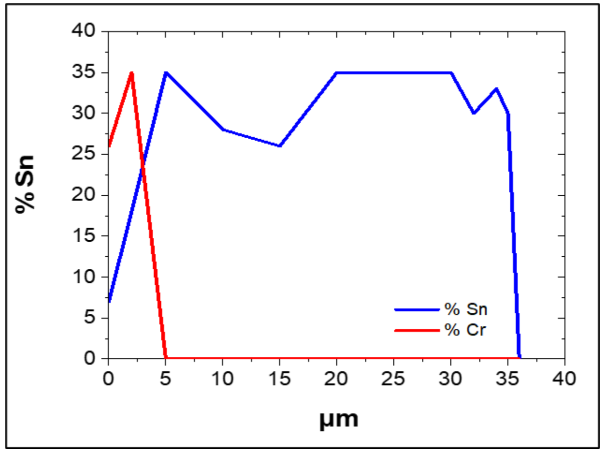
Main candidate:
Inconel Alloy
of vacuum chamber (presence of Ni and Fe as well)



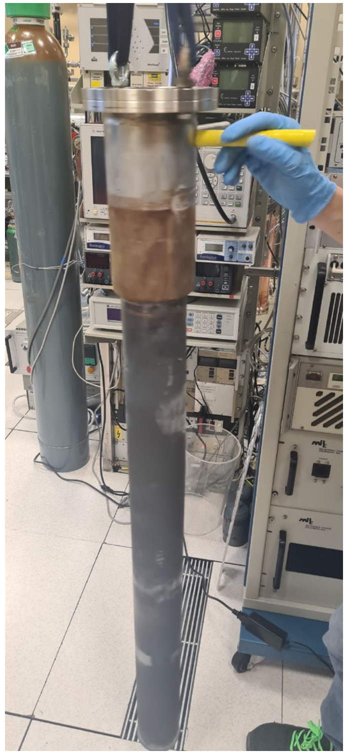
Nb_2O_5 substrate (Nb BCP anodized)

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

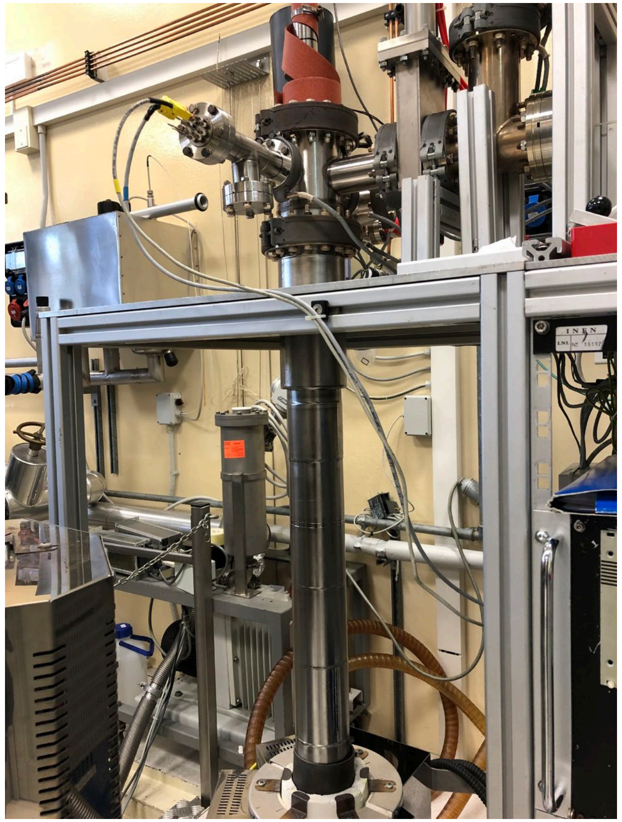
Grain
Nb 74-76 %
Sn 24-26 %



Nb_3Sn Cylindrical target production **by tin liquid diffusion (dipping)**



Replacement of old inconel chamber



With a new one in bulk Nb produced in Zanon

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Nb_3Sn Cylindrical target production **by tin liquid diffusion (dipping)**

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

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

Protective layer mandatory

Any advice?

Dipping furnace oxidation
possible solutions

Paint option

HIGH TEMPERATURE REFRACTORY COATINGS

Technical Bulletin A5-S5

Aremco's refractory paint systems offer the ultimate protection of high temperature structures used in the processing of metals, glasses and plastics.

PRODUCT HIGHLIGHTS

Graphi-Coat™ 623
This coating system, originally patented by Aremco, is a two-part, silica-bonded, titanium diboride filled, oxidation resistant coating for protecting graphite from oxidation to 2000 °F (1093 °C). Used on graphite crucibles, heat-treating fixtures, and electrodes in the metallurgical industry.

Pyro-Paint™ 634-AS and 634-AS1
These alumina-silica based advanced coatings are rated for continuous service temperatures up to 2300 °F. Provides excellent adhesion to ceramic fiber blankets, modules and boards and resists wetting by nonferrous molten metals, increasing the durability and erosion resistance of the underlying material. High dry film thicknesses from 10-50 mils can be achieved with this coating. Select 634-AS for thin coating applications and 634-AS1 for high build applications.

Pyro-Paint™ 634-AL
This pure, alumina based compound creates a hard, high temperature resistant coating for refractory fiber boards and shapes, providing exceptional resistance to molten metals and open flames to 3200 °F (1760 °C). Increases heat reflectivity in furnaces to improve efficiency and ramp up temperatures more rapidly.

Pyro-Paint™ 634-ALP
This phosphate-bonded, alumina coating system bonds exceptionally well to dense refractory ceramics, providing high abrasion and corrosion resistance for operating temperatures to 3200 °F (1760 °C).

Pyro-Paint™ 634-BN and 634-BN(SC)
These highly-filled boron nitride solutions are extremely lubricious and inert. They are non-wetted by molten salts, glasses, plastics, and most metals including aluminum and magnesium. Select 634-BN for hard coat; 634-BN(SC) for softer, lubricious coat.

Pyro-Paint™ 634-CA and 634-GR
These carbon and graphite based coatings are formulated for parting of aluminum permanent molds, non-sticking in glass forming applications, and lubrication and stop-off in metalworking and wire drawing. Provides superior release, surface finish and mold protection.

Pyro-Paint™ 634-SIC
This advanced silicon carbide, water-based coating reduces significantly the oxidation of graphite and carbon components and structures at temperatures to 2550 °F (1400 °C). Provides a hard surface and withstands thermal cycling.

Pyro-Paint™ 634-YO
This ultra high temperature yttrium oxide coating provides exceptional protection of graphite, ceramic and metal components exposed to reactive molten metals such as titanium, uranium and their alloys. Usable in vacuum and inert atmospheres to 2732 °F (1500 °C).

Pyro-Paint™ 634-ZO
This highly-filled zirconium oxide-based coating produces a hard, chemically-resistant protective layer which is stable with aluminum, molybdenum, platinum, rhodium, and titanium. It is ideal for sealing porous ceramics and protecting other ceramic, graphite and metal structures up to 3270 °F. Exceptional for coating resistance wire heating elements in furnaces, protecting them from residue buildup which causes arcing and reduced element life.

Features

- *Ultra Hi-Temp Resistance*
- *Non-Wetted by Molten Metals, Salts, Glass & Plastics*
- *High Lubricity for Easy Part Release*
- *Minimizes Cast Surface Defects*
- *Increases Mold & Die Life*
- *For Use in Oxidizing, Reducing & Vacuum Atmospheres*

Applications

- *Composite Forming*
- *Glass Forming*
- *Metal Casting*
- *Injection Molding*
- *Ceramic Hot-Pressing*
- *Metal Powder Sintering*
- *Welding*
- *Brazing*

HIGH TEMPERATURE REFRACTORY COATINGS PROPERTIES

Part Number	623	634-AL	634-ALP	634-AS	634-AS-1	634-BN	634-BNSC	634-CA	634-GR	634-SIC	634-YO	634-ZO
Tradename	Graphi-Coat™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™	Pyro-Paint™
Major Constituent	Titanium DiBoride	Aluminum Oxide		Alumina-Silica		Boron Nitride		Carbon		Silicon Carbide	Yttrium Oxide	Zirconium Oxide
Temp. Limit, °F (°C)	2000 (1093)	3200 (1760)	3200 (1760)	2300 (1260)	2300 (1260)	1560 (850)①	1560 (850)①	2200 (1200)	2200 (1200)	2550 (1400)	2732 (1500)	3270 (1800)
% Solids by Weight	78.0	76.8	75.8	61.2	77.0	38.6	28.5	25.4	45.5	74.5	42.7	65.0
% Solids by Volume	57.7	66.0	73.5	63.3	70.0	21.6	27.5	18.4	29.7	53.0	23.0	35.5
Theoretical Coverage ft²/gal @ 1 Mil DFT	890	1060	1180	1015	1120	345	440	295	475	850	370	570
Application Temp., °F	50-90	50-90	50-90	50-90	50-90	50-90	50-90	50-90	50-90	50-90	50-90	50-90
Recommend Curing Min Air Set, Hours	1	2	1	2	2	2	2	2	2	1	0.5	2
Cure, °F/Hours ②	1400/0.25	200/2	200/2, 800/1	200/2	200/2	200/2	200/2	200/2	200/2	200/2, 800/1	200/1	200/2
Color	Gray	White	White	Off-White	White	White	White	Black	Black	Gray	Off-White	Tan
No. Components	2	2	1	1	1	1	1	1	1	1	1	1
Mix Ratio ③	60 : 40	75 : 25	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Viscosity, cP	1,000 - 2,500	7,000 - 8,000	5,000 - 7,000	600 - 800	10,000-15,000	1,500-2,500	< 100	700 - 1,000	750 - 1,250	1,000-2,000	200-400	1,000 - 1,400
Thinner ④	623-T	634-AL-T	634-ALP-T	634-AS-T	634-AS-T	634-BN-T	634-BNSC-T	634-CA-T	634-GR-T	634-SIC-T	634-YO-T	634-ZO-T
Specific Gravity, g/cc	2.00	2.27	2.38	1.59	1.70	1.34	1.20	1.15	1.32	2.30	1.31	2.02
Coating pH	7-8	4-5	2-3	7-8	7-8	11-12	4-5	8-9	8-9	2-3	7-8	11-12
Flash Point, °F	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	75	N/A
Shelf Life, months	6	6	6	6	6	6	6	6	6	6	6	6
Storage, °F	40-90	40-90	40-90	40-90	40-90	40-90	40-90	40-90	40-90	40-90	40-90	40-90
Weight/Gallon, lbs ⑤	12.5	12.0	16.5	12.0	12.5	9.5	9.5	8.5	9.5	16.5	12.0	23.5

Reference Notes
① Temperature limit applies to oxidizing atmospheres only. Can be used in vacuum/inert atmospheres to 2000 °C.
② A short cure is recommended, however, most of these products can be air set them ramped up to operating temperature immediately.
③ Mix ratio is Powder : Liquid. Ratios may be altered as required to adjust viscosity.
④ Distilled water may also be used to thin all products. Use 1-2% distilled water by weight.
⑤ For two-part systems, this only refers to the weight per gallon for the powder portion of the mixture.

Dipping furnace oxidation
possible solutions

- **Paint option**
- **Cold spray coating (Tungsten?)**
- **Chromium protective layer (can diffuse…)**
- **Double wall chamber (filled with Ar)**

Other suggestions?

Nb_3Sn on planar samples

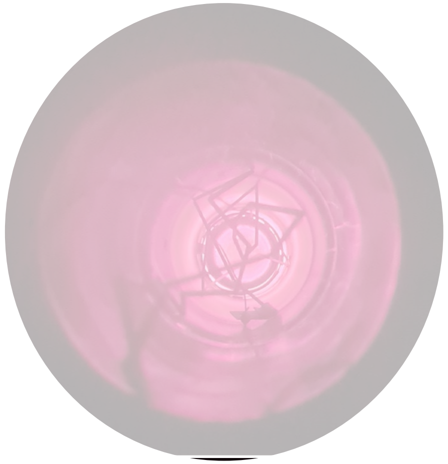
4" planar magnetron

Nb_3Sn cylindrical target

Via liquid tin diffusion (dipping)

6 GHz cavities

RF characterization

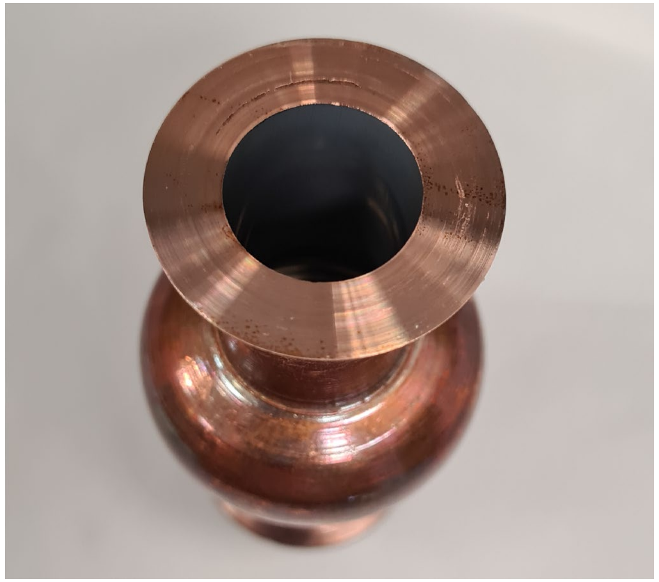


6 GHz cavities RF characterization

STFC first Nb on Cu 6 GHz cavity

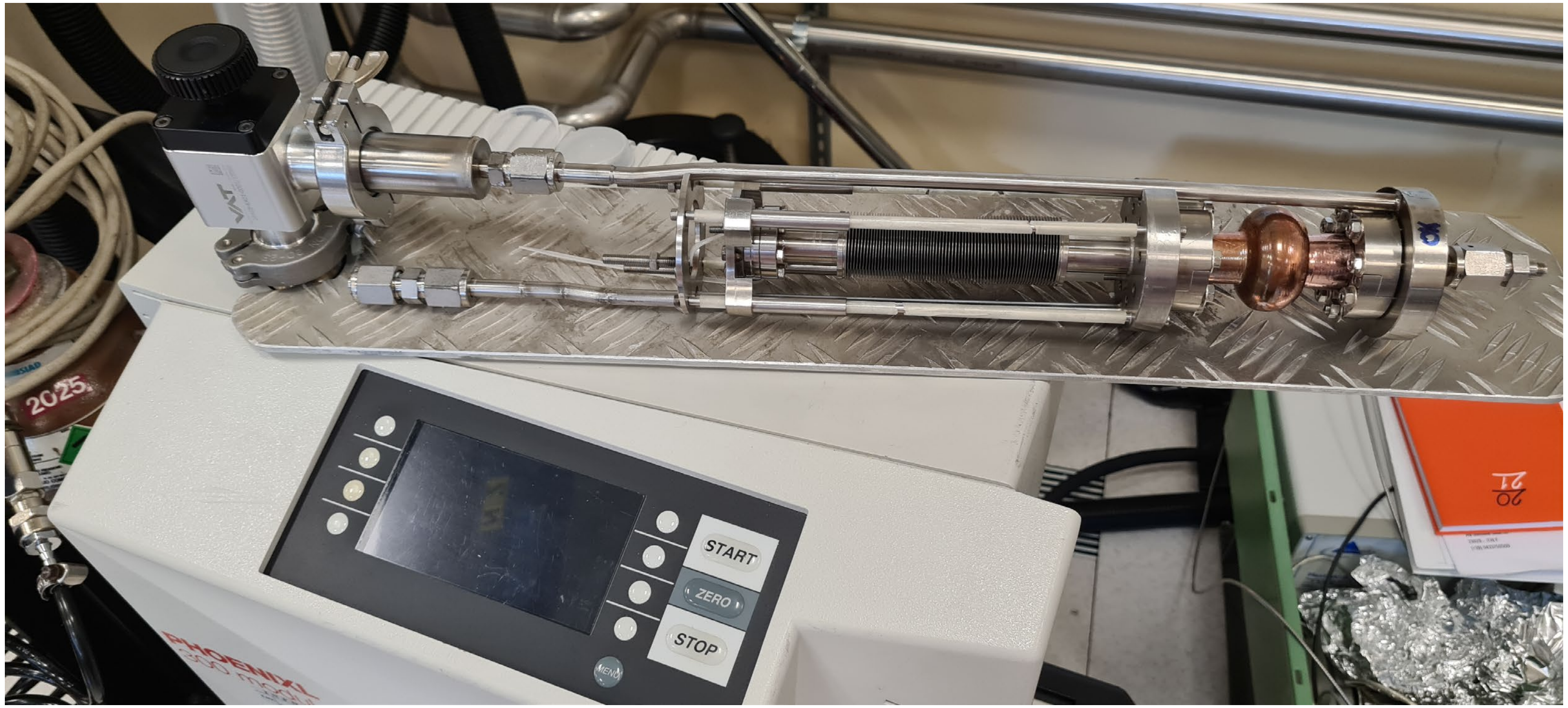
Leak test not passed because **flange was deformed**

6 GHz cavities RF characterization



Mechanical machining of the flange preserving the coating from poisoning

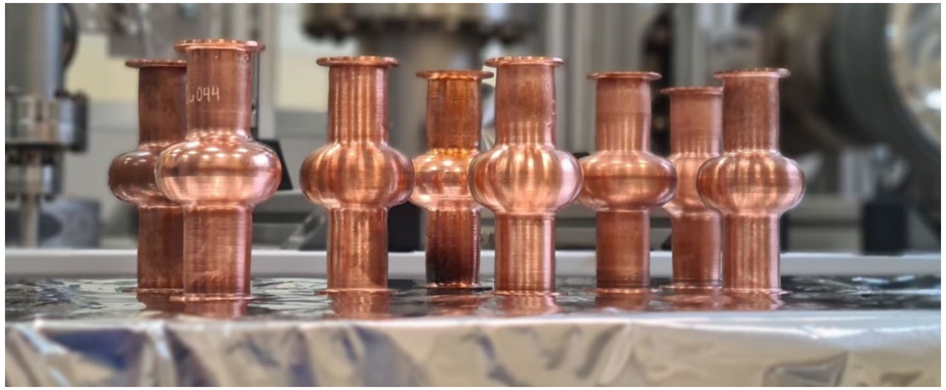
6 GHz cavities RF characterization



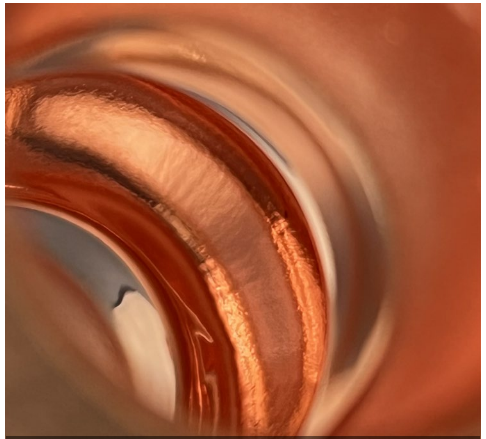
Leak test passed!

RF test scheduled on Thursday

6 GHz cavities production



Issue: one side – Mate surface



EP

cavity n° 53

№	Process	Data	Time	Removed PC	Delta mass	Removed Formula
1	EP	21/01/2022	71 min	54,8 um	2,21	52,5 um
2	EP	25/01/2022	70 min	55	2,22	52,8 um
3	SUBU	25/01/2022	4 min			
4	EP	27/01/2022	167 min	177 um	8.25	196 um
5	EP	30/01/2022	110 min	80,0 um	3.52	83.7 um
6	SUBU	30/01/2022	3 min			
7	HPR	30/01/2022				
Total removed				373,8 um		392 um

2 cavities almost ready

Science and Technology Facilities Council

Going forward to ***increase the 6 GHz production*** *in the future 3 months with a Stage Student –* ***Federico Valerio***

iFAST

Collaboration study on PEP - MP

Metallographic polishing path with final chemistry polishing
courtesy of Oleksandr Hryhorenko (IJClab/CNRS)

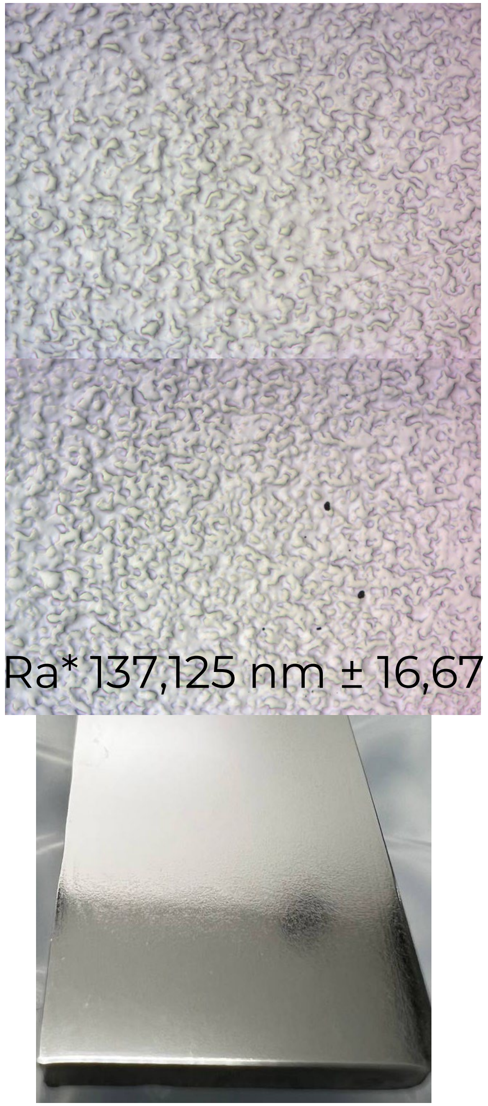
Plasma Electrolytic Polishing

1. Optical miscroscopy
2. Laser confocal miscroscopy
3. SIMS
4. Linear profilometry
5. …

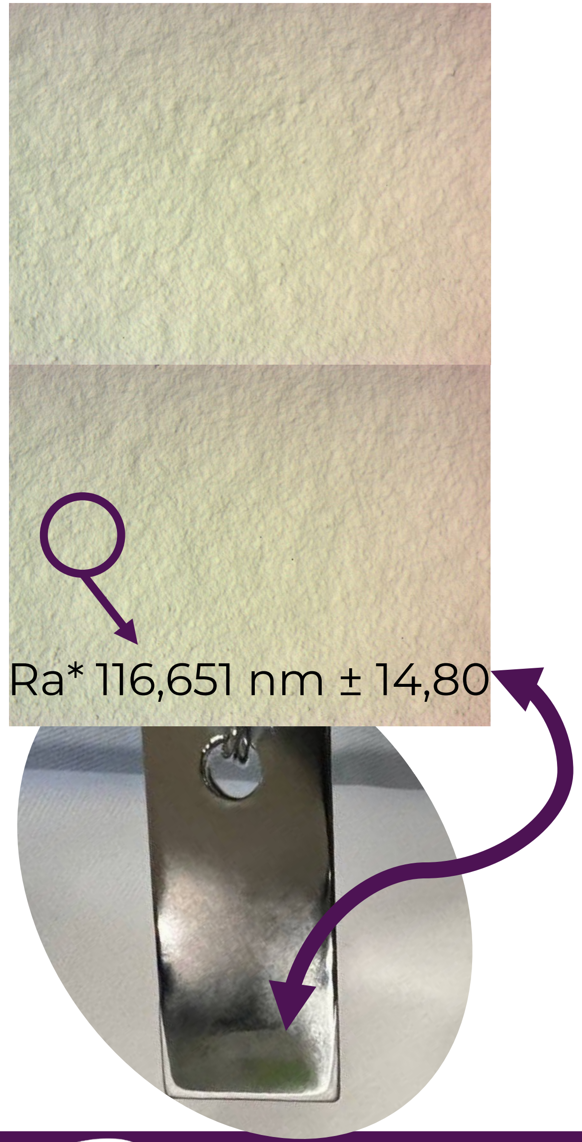
Studied samples

- Nb MP + multi-step PEP *11.2021*
- Nb MP + single-step PEP *04.2022*
- Cu MP + multi-step PEP *11.2021*
- Cu MP + single-step PEP *04.2022*
- More samples to study / check

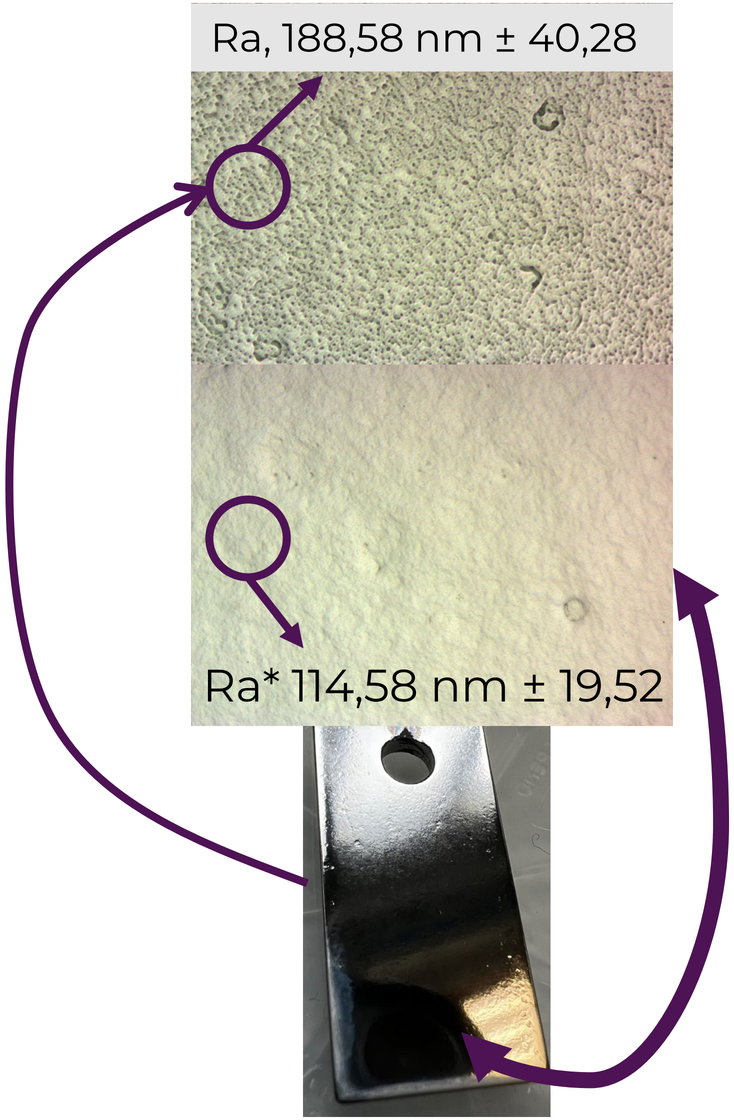
Initial
after MP



MP + Multi step PEP
6 step x 1-2min = 23 um



MP + Single step PEP
1 step x 6min = 23 um



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Thank you for your attention

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