Cu-based Nb_3Sn QPR sample preparation via ETS bronze route

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Outlines

1. Nb_3Sn And QPR background
2. Overall ETS bronze route progress
3. Cu-based Nb_3Sn QPR sample preparation
4. Nb/Cu QPR sample baseline RF test
5. Future work and Summary

Background: Cu-based Nb_3Sn coating

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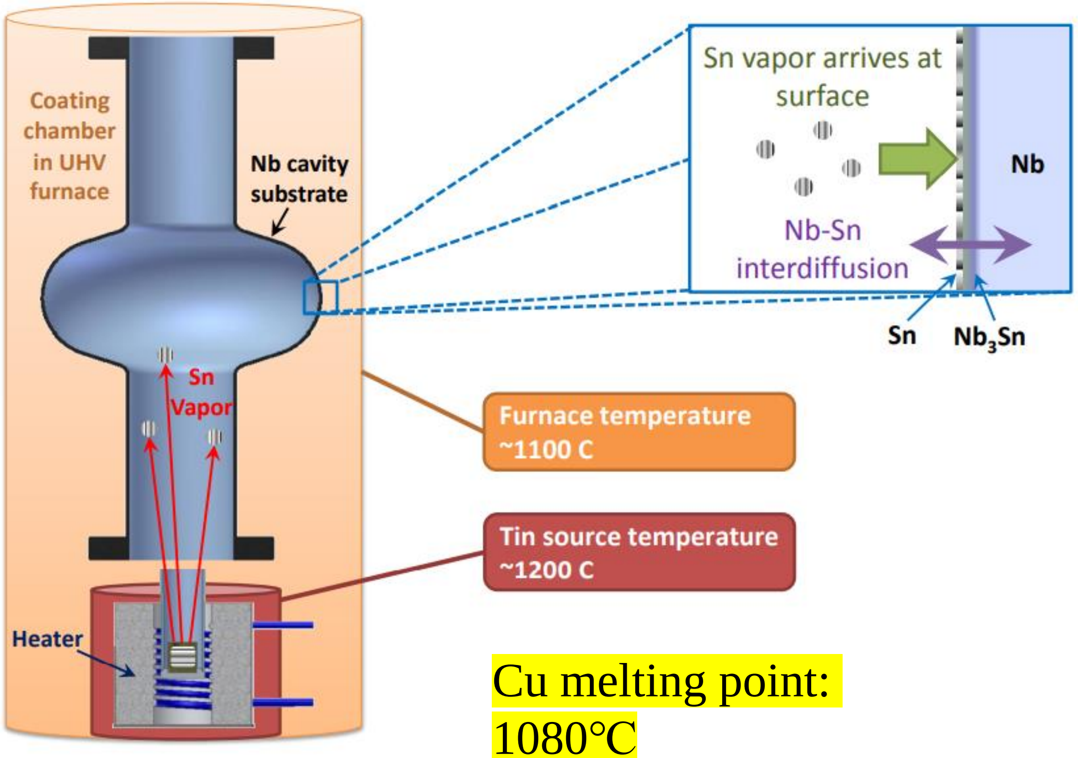
- Nb_3Sn thin film superconducting cavity is the key technology for next-generation accelerator, and its engineering application will lead to a technological revolution in the field of SRF.

Courtesy of G. Ciovati, JLab



Cu melting point: 1080°C

Diagram of the Tin Vapor Diffusion[1]

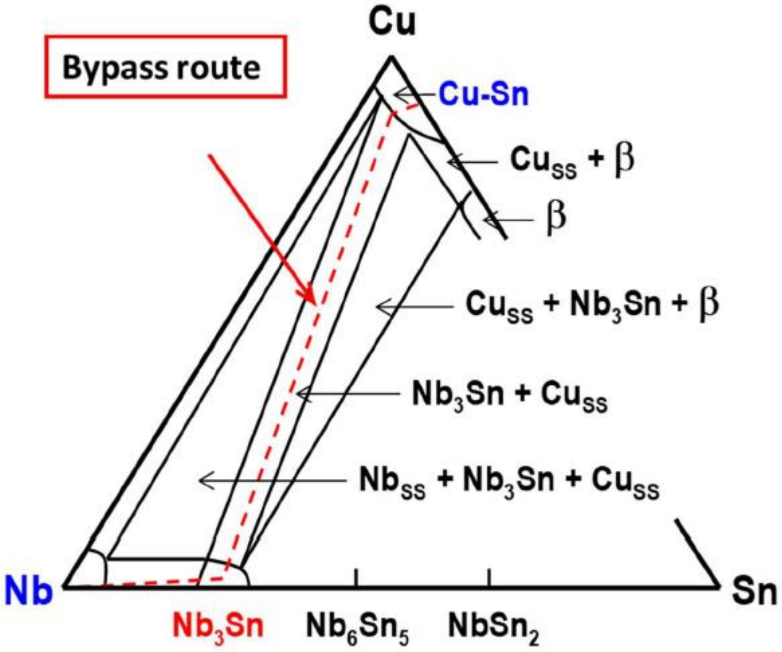
Nb_3Sn film on Cu cavity:

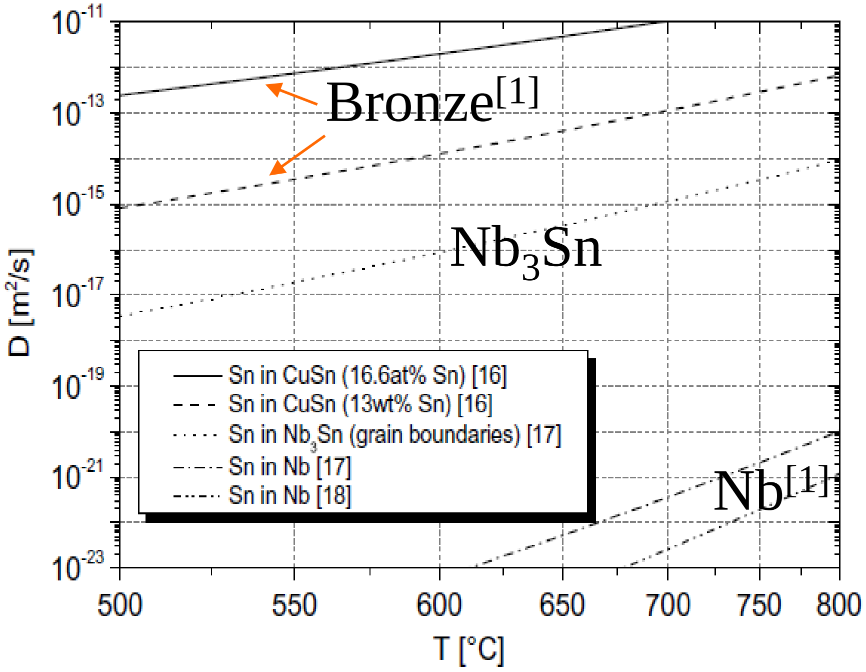
- Good thermal conductivity, better mechanical stability
- High performance@4.2 K, cooled by cryocooler

[1] Posen, Sam. "Understanding And Overcoming Limitation Mechanisms In Nb3Sn Superconducting Rf Cavities." (2015).

One achievable way : Nb_3Sn/Cu bronze route

Phase diagram of Nb-Cu-Sn ternary system [2] at 700°C





1. The copper can facilitate the interdiffusion between Nb and Sn by 7~10 magnitudes of orders.
2. The copper alloy will be excluded from the Nb_3Sn phase by itself.

[1] H. Müller and T. Schneider, "Heat treatment of Nb3Sn conductors," Cryogenics, vol. 48, pp. 323-330, 2008/07/01/ 2008.
[2] L Mei, Z Du, C Guo, & C Li. (2009). Thermodynamic optimization of the cu-sn and cu-nb-sn systems. Journal of Alloys & Compounds, 477(1-2), 104-117.

A powerful Tool: Quadrupole Resonator

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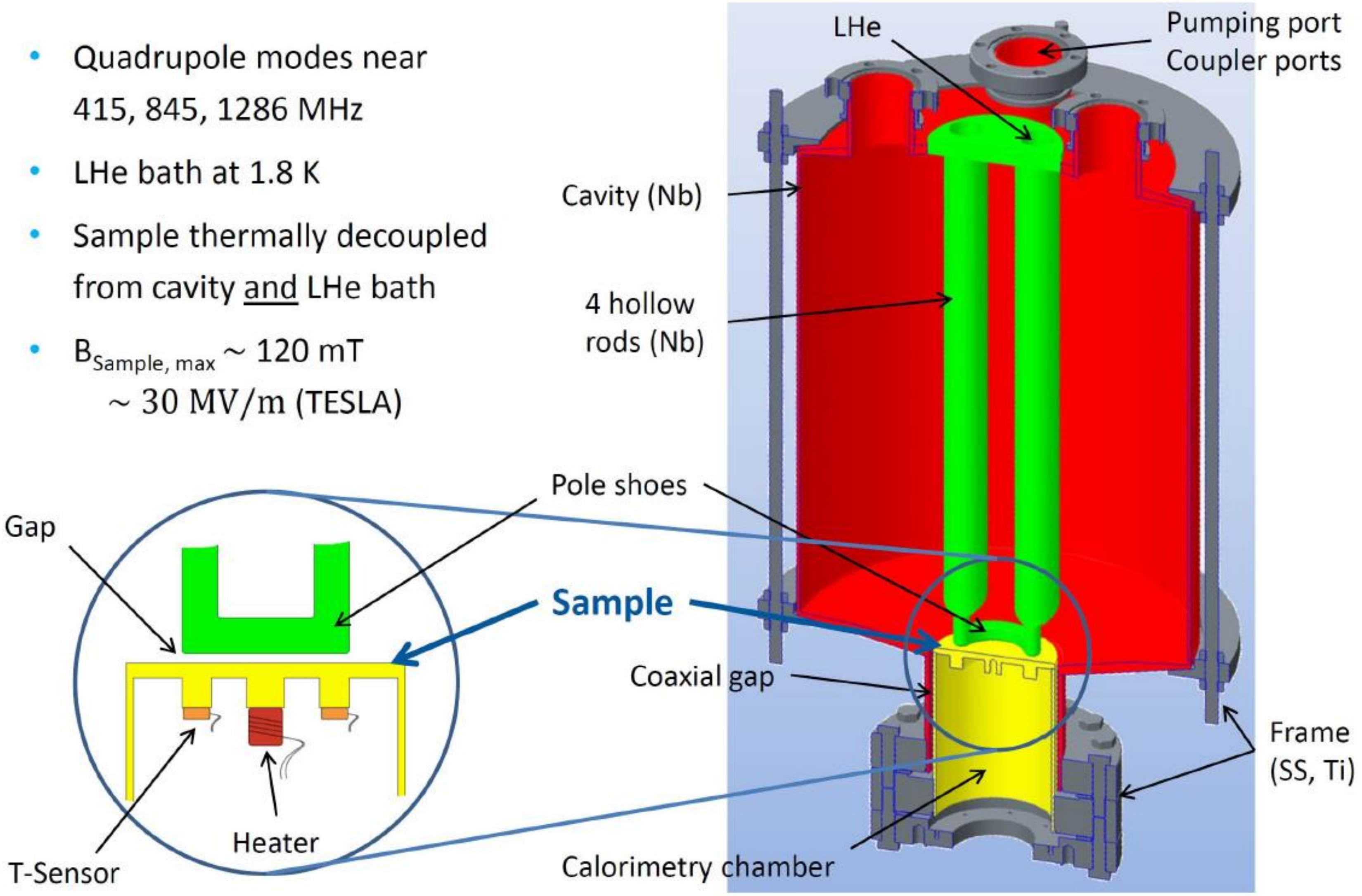
- Quadrupole modes near 415, 845, 1286 MHz
- LHe bath at 1.8 K
- Sample thermally decoupled from cavity and LHe bath
- $B_{Sample, max} \sim 120$ mT ~ 30 MV/m (TESLA)



Surface resistance

- $R_S(\omega, B_{RF}, T)$
- $R_{BCS} \leftrightarrow R_{res}$
- High resolution $R_S \approx 1\ n\Omega \leftrightarrow Q_0 > 10^{11}$
- Cooling conditions
- Trapped flux

Penetration depth

- Penetration depth $\lambda(T)$
- Critical temperature T_c

RF quench field

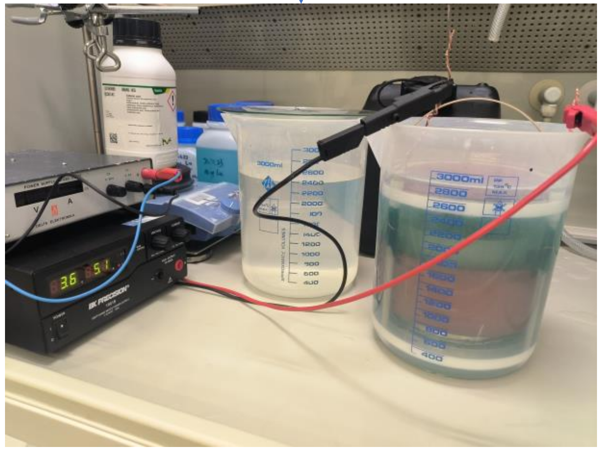
- $B_{vp,\,RF}(T, \omega)$
- T_c

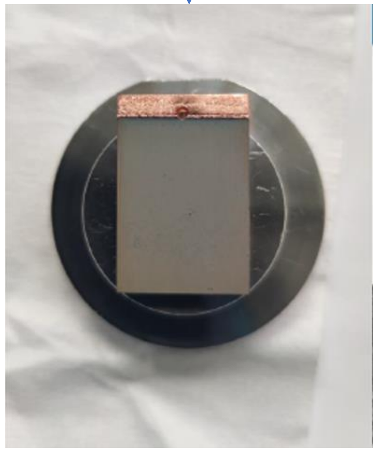
Oliver Kugeler

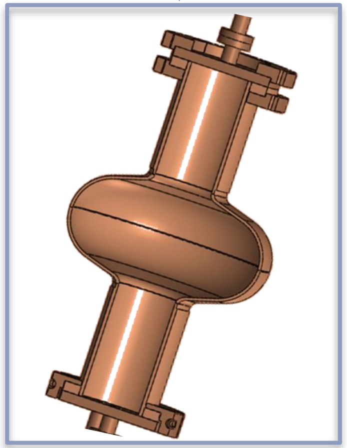
The bronze route work schedule at HZB

2023 | 2024 | 2025 | 2026

Study 1: Build an electrochemical platform √ → **Study 2:** Optimized small Nb_3Sn samples √ → **Study 3:** Optimized QPR Nb_3Sn samples √ → **Study 4:** QPR sample RF iterative optimization× → **Study 5:** Cavity RF iterative optimization×







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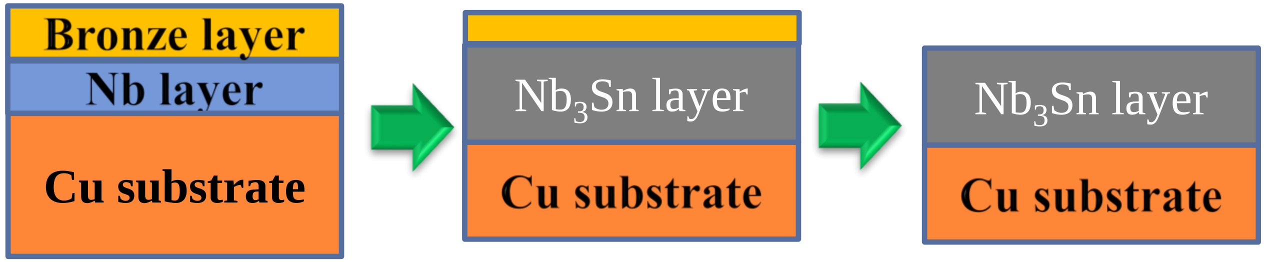
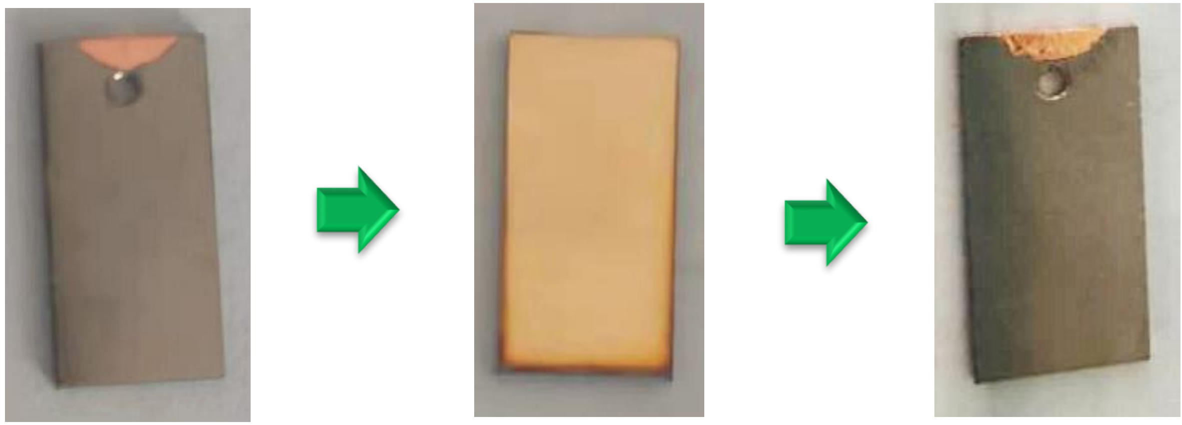
Previous work: Bronze/Nb/Cu flat samples

Nb_3Sn/Cu sample preparations: Electrochemical and Thermal Synthesis (ETS) bronze route

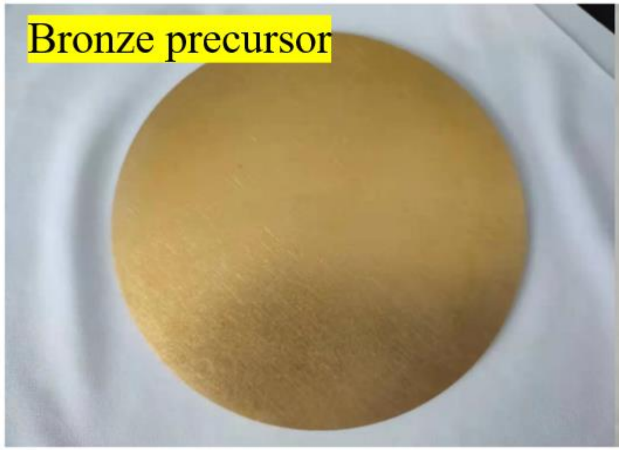
- ✓ low cost, simple operation.
- ✓ suitable for complex cavity types, mass production.

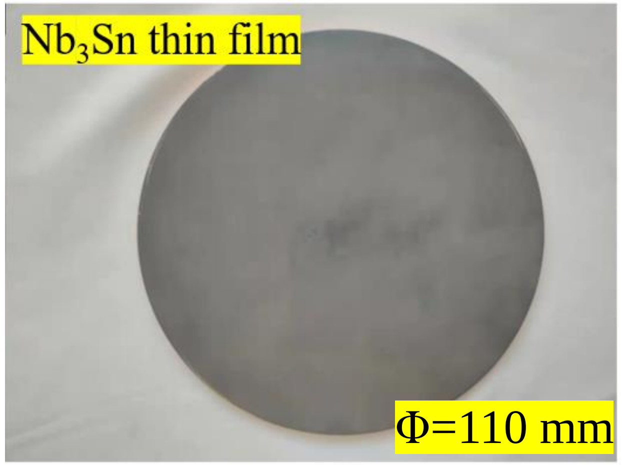


Nb layer: magnetron sputtering



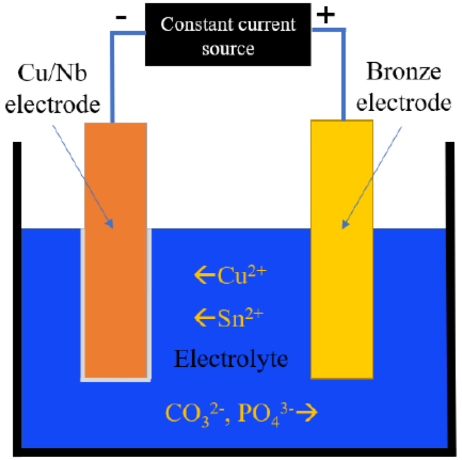
Nb/Cu | Bronze/Nb_3Sn/Cu | Nb_3Sn/Cu

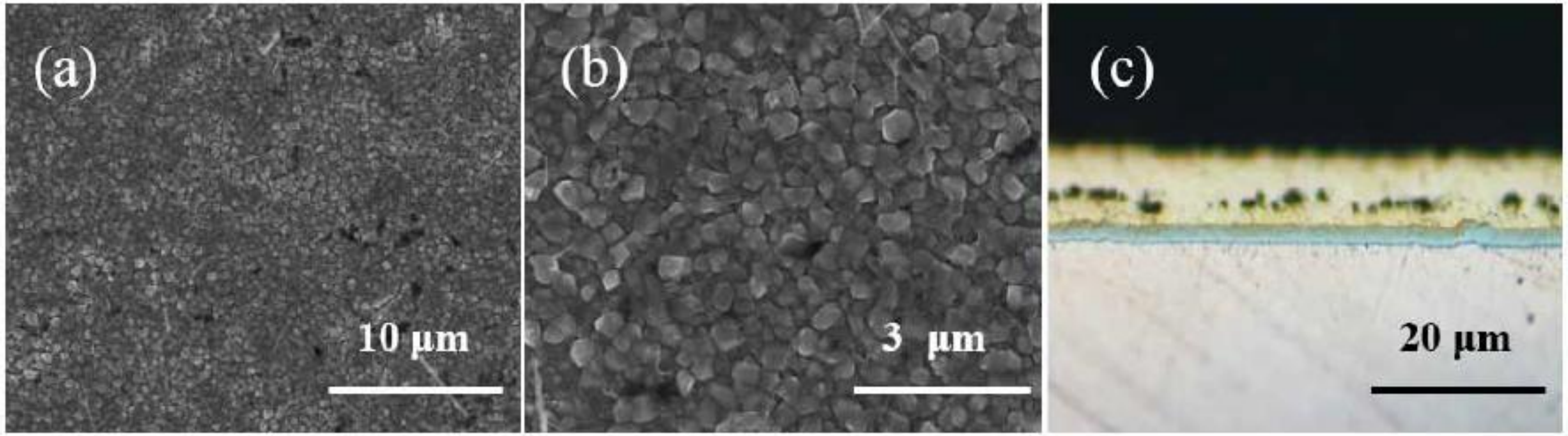


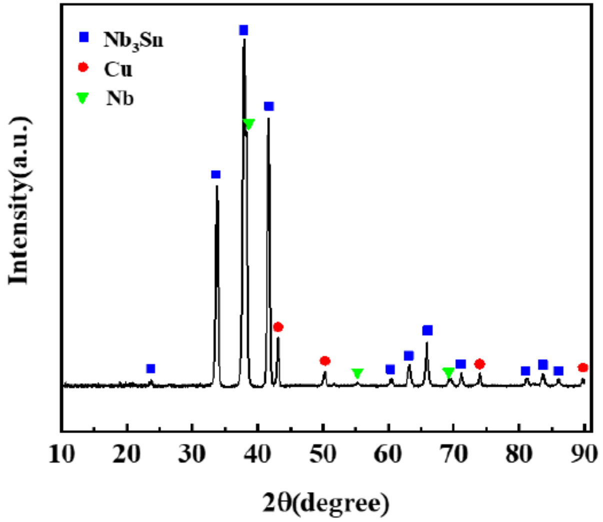


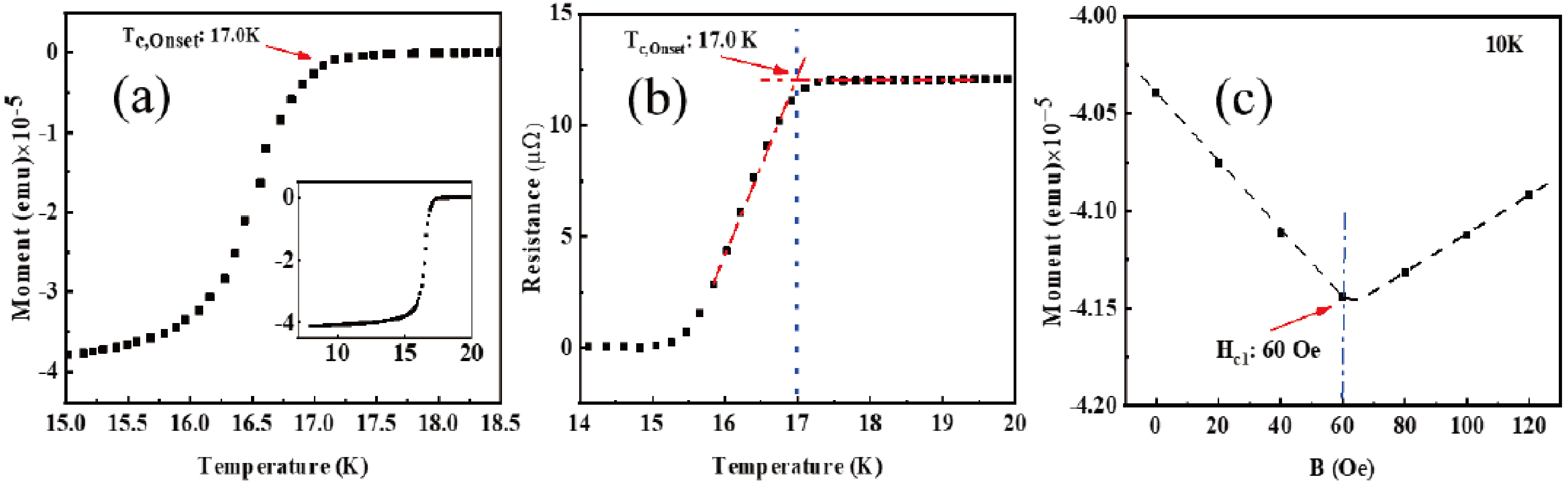
Small samples: Bronze/Nb/Cu multilayer films

Nb_3Sn/Cu sample properties:



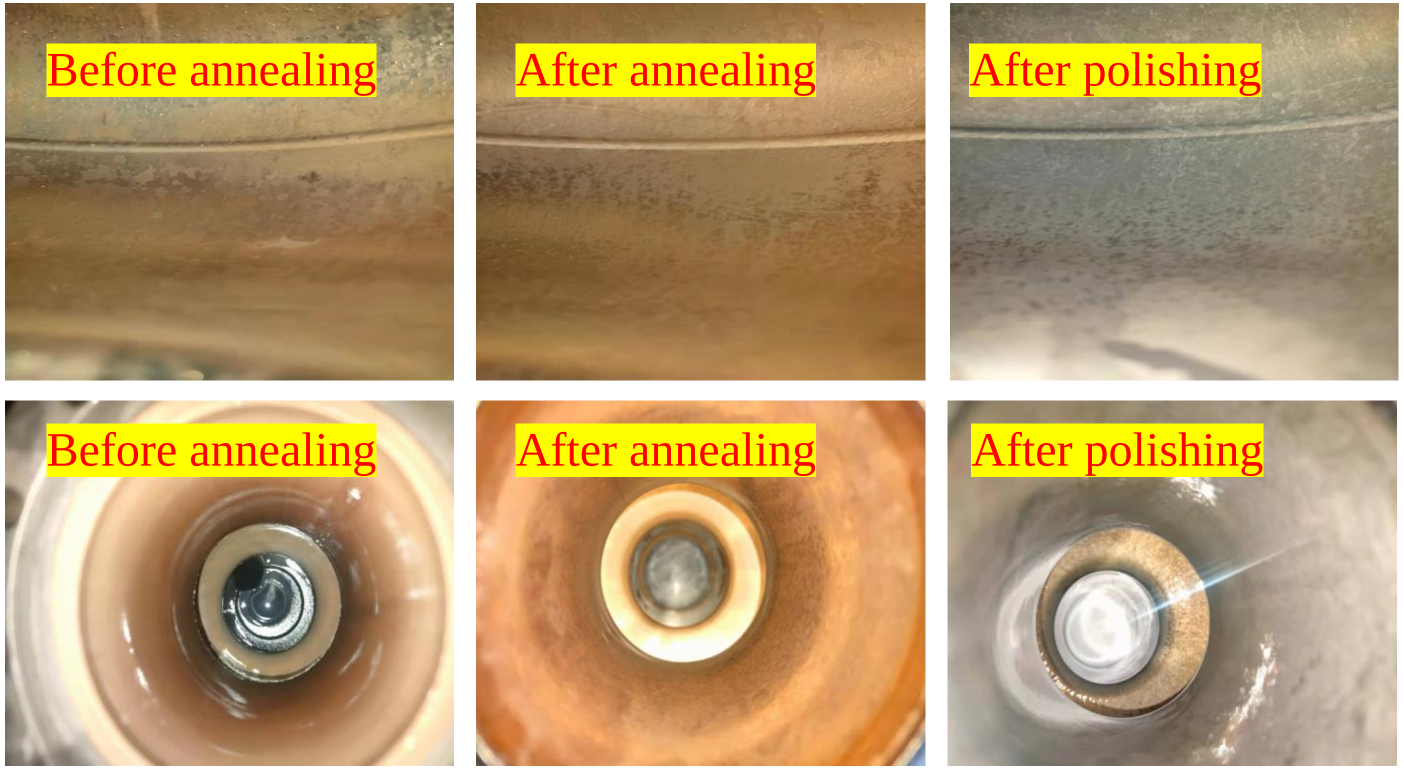


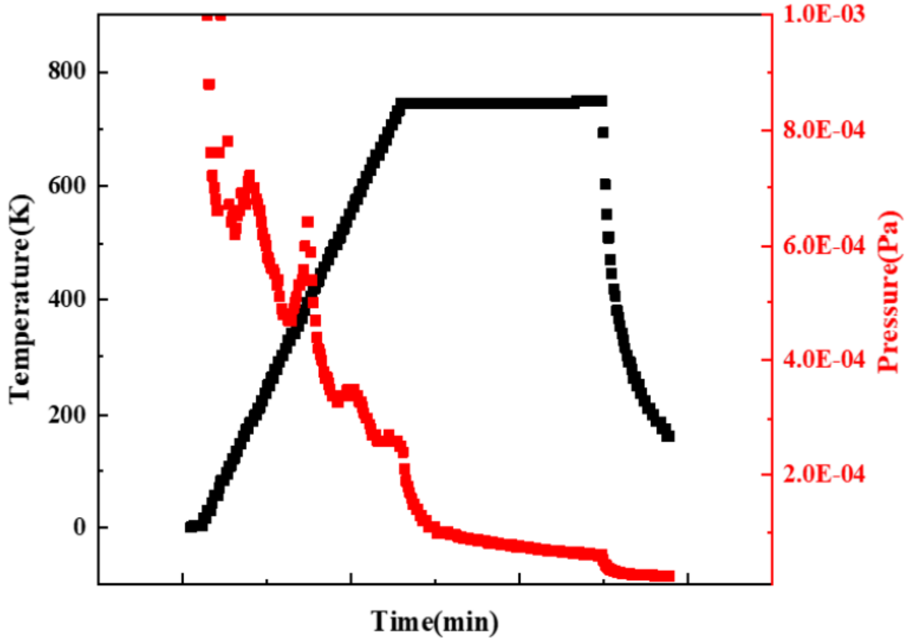




1.3 GHz Nb_3Sn/Nb cavity bronze route

➢ The surface morphology of the different stages of the coating is shown in the figure below:



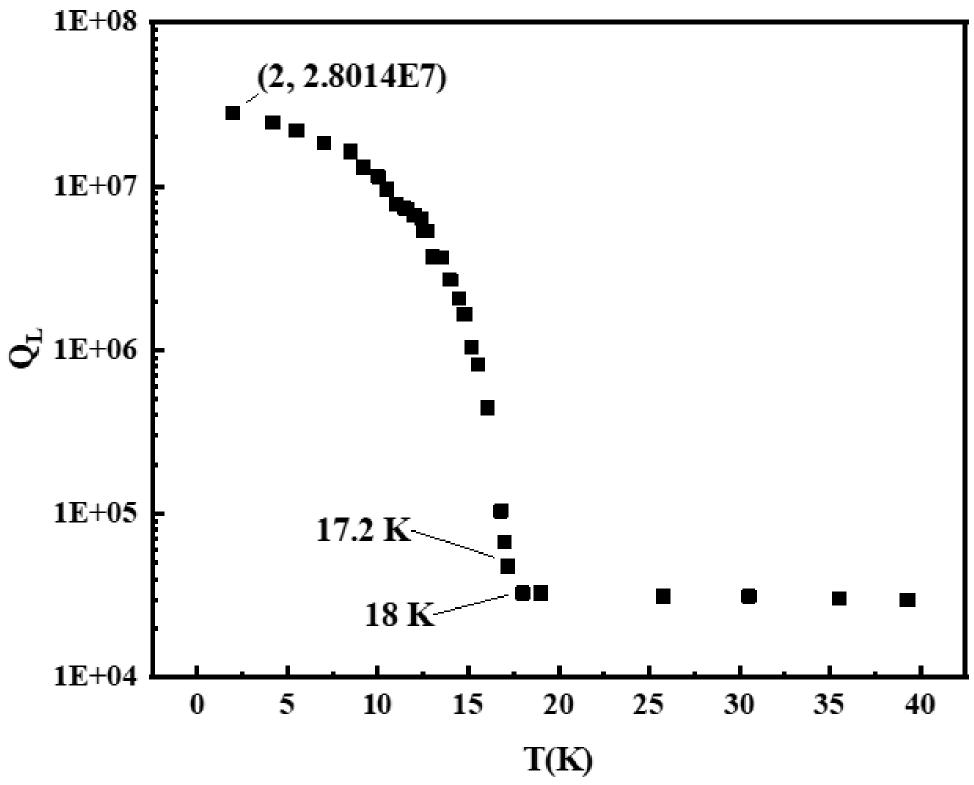


The relationship between annealing curve and air pressure with time.

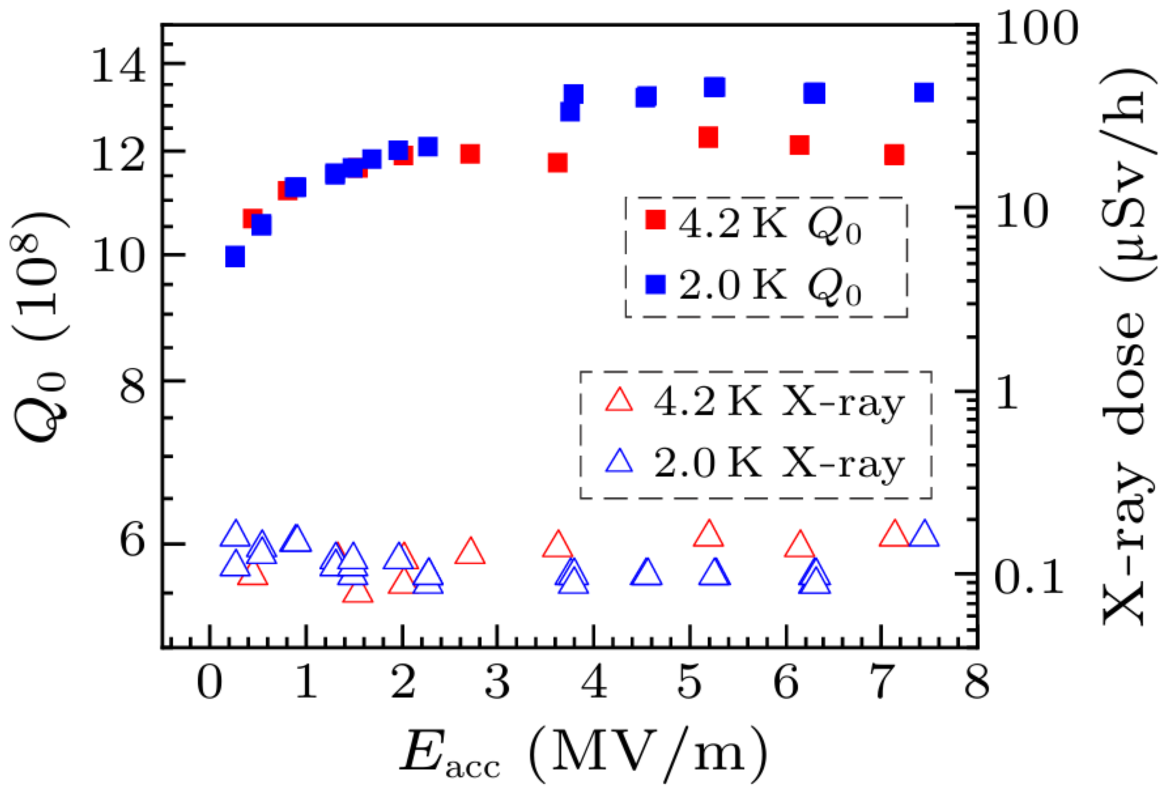
➢ Morphology analysis: The film is complete and the film-base bond is good, but there are differences in the optical area, and niobium oxide is suspected.

1.3 GHz Nb_3Sn/Nb cavity vertical test

Low temperature vertical test:



Q_L-T curve



Q_0-E_{acc} curve

- The Q_0 of the thin-film cavity at 4.2 K is about 1.2E+9, which is better than that of the bulk niobium cavity under the same conditions.
- Without Q-Slope and X-ray, it is inferred that the reason for limiting E_{acc} is thermal quench.

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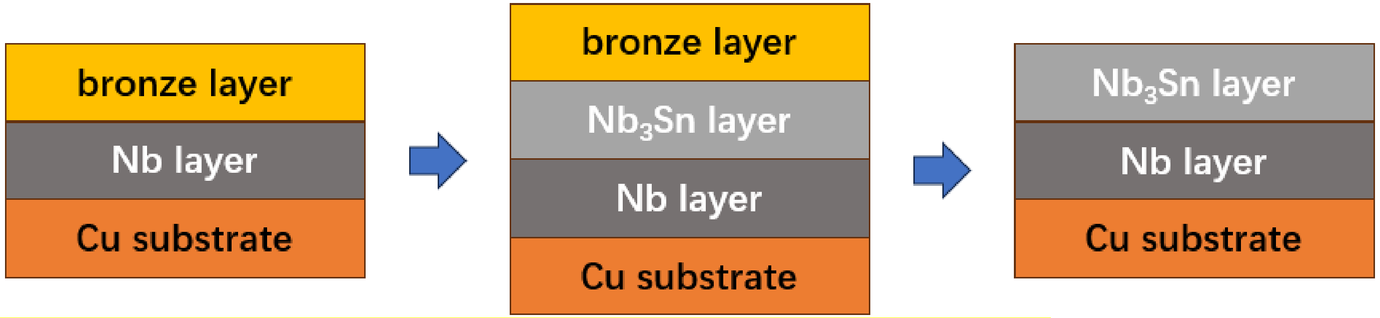
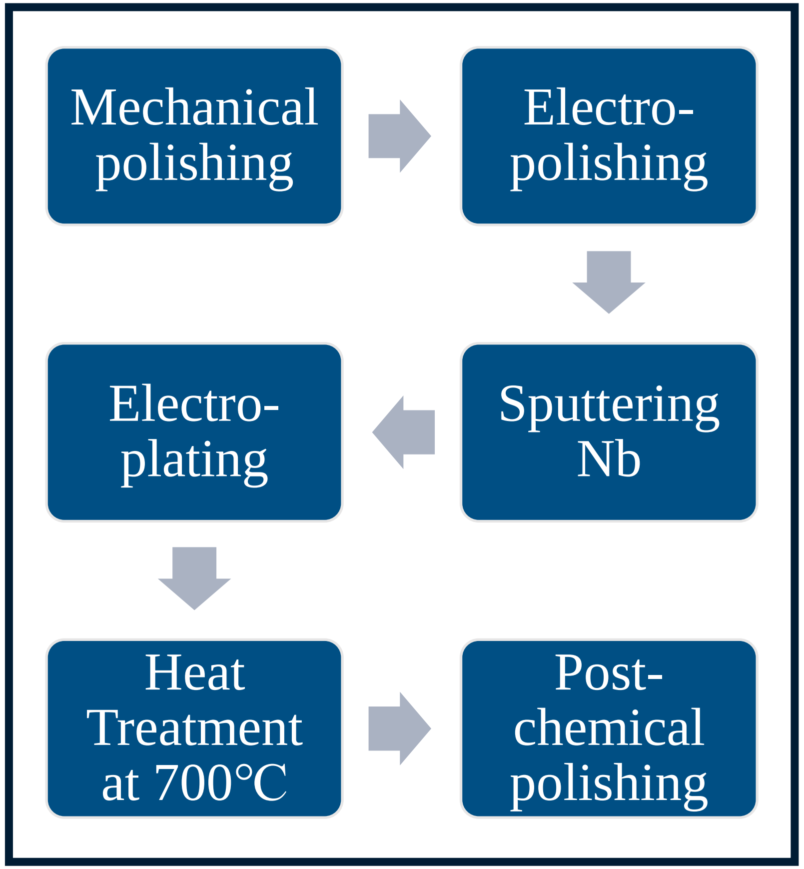
Nb_3Sn/Cu QPR sample prepare procedure

The Nb_3Sn/Cu QPR sample preparation process includes 6 steps:





Bulk copper QPR sample by turning, no braze or weld.

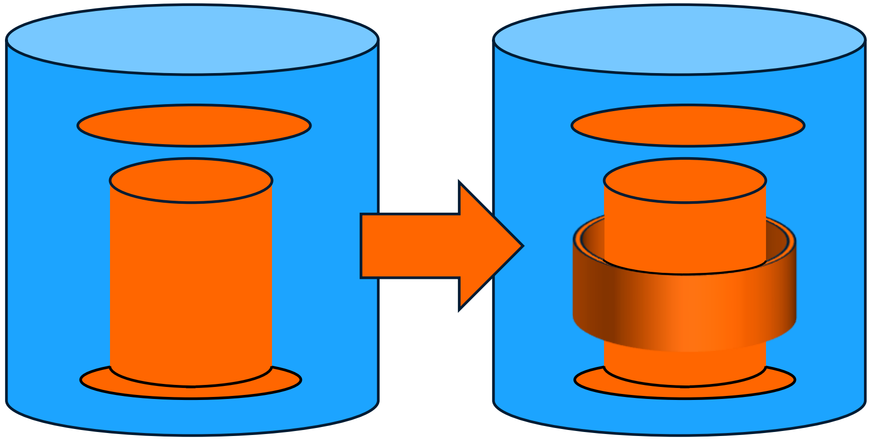
Electropolishing | Sputtering Nb | Coating bronze | Annealing at 700℃ | Polishing bronze

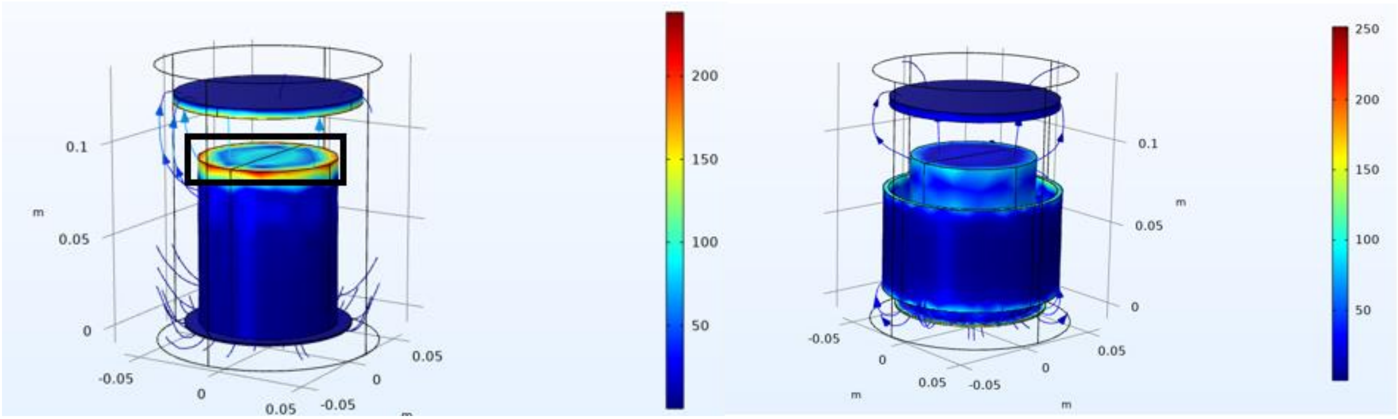
- A new Cu-based Nb_3Sn QPR sample was successfully prepared.
- The Nb_3Sn/Cu QPR sample RF properties will be tested at HZB soon.

Step 2: Cu QPR sample electropolishing process

Optimized cathode structure and key parameters:

- **New cathode structure:**
  1. Only disk cathode before
  2. Disk cathode and Round belt cathode
- **COMSOL simulation:**
  The difference in current density is reduced from 16 times to 2-3 times
- **Parameters:**
  1. CV mode: 2.1 V
  2. Electrode distance: 20 mm
  3. Temperature: 15 °C
  4. Stirring speed: 100 rpm
  5. Polishing time: 1 h (30 μm)





Surface Roughness test of EP QPR sample

Optimized electropolished Cu QPR sample:

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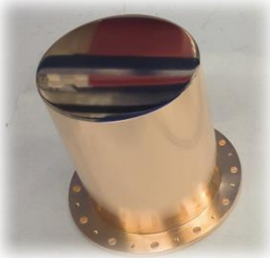
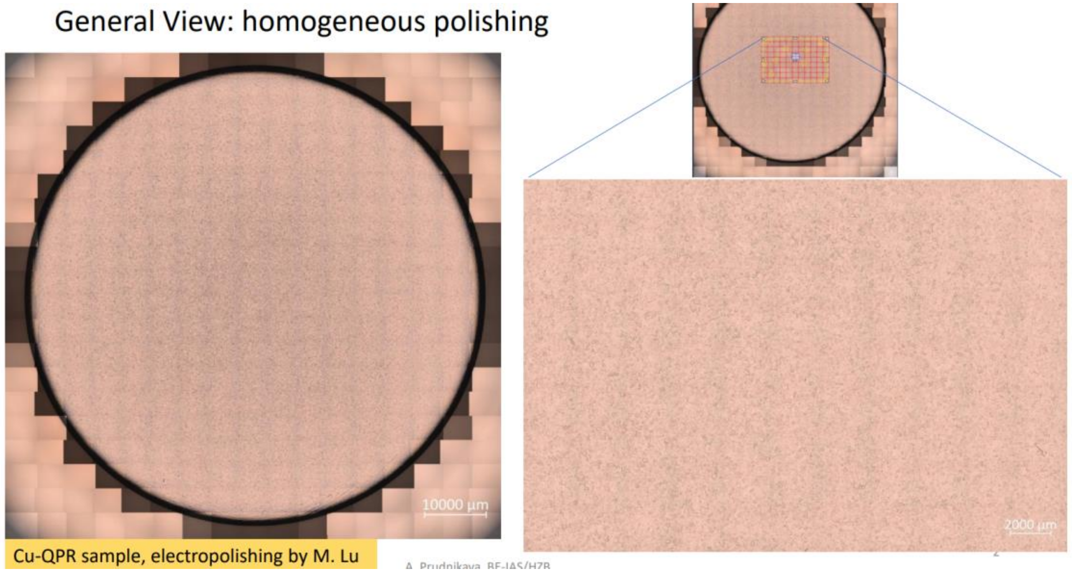
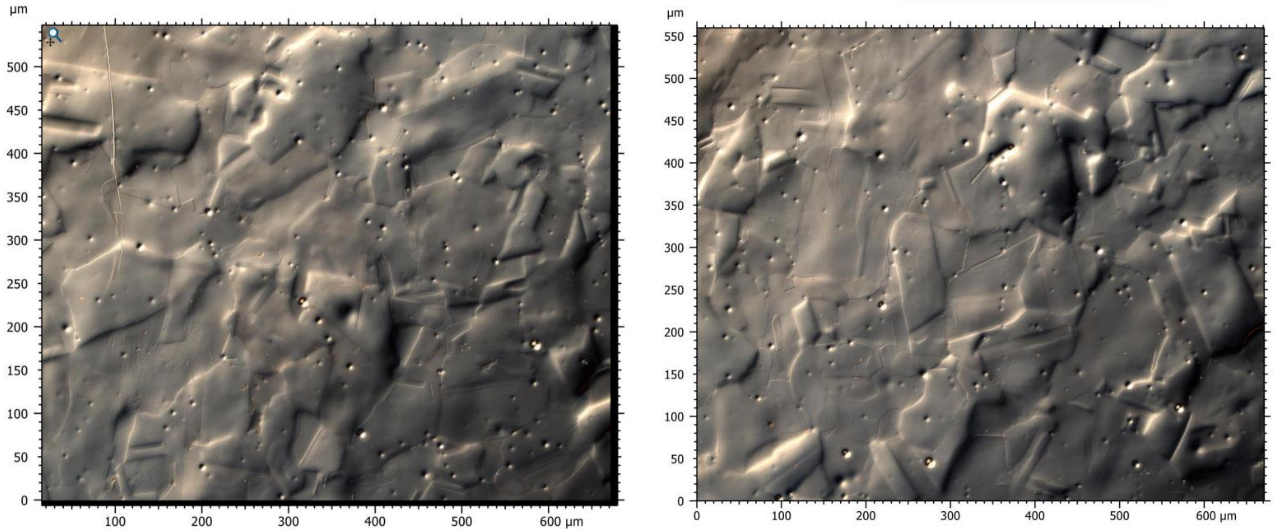




Optical (Polarized Light) Images



- ➢ Observing surface morphology using LSM and measuring surface roughness.
- ➢ There are small pitting (1µm) and corrosion on the surface, difficult to completely remove.

Surface Roughness

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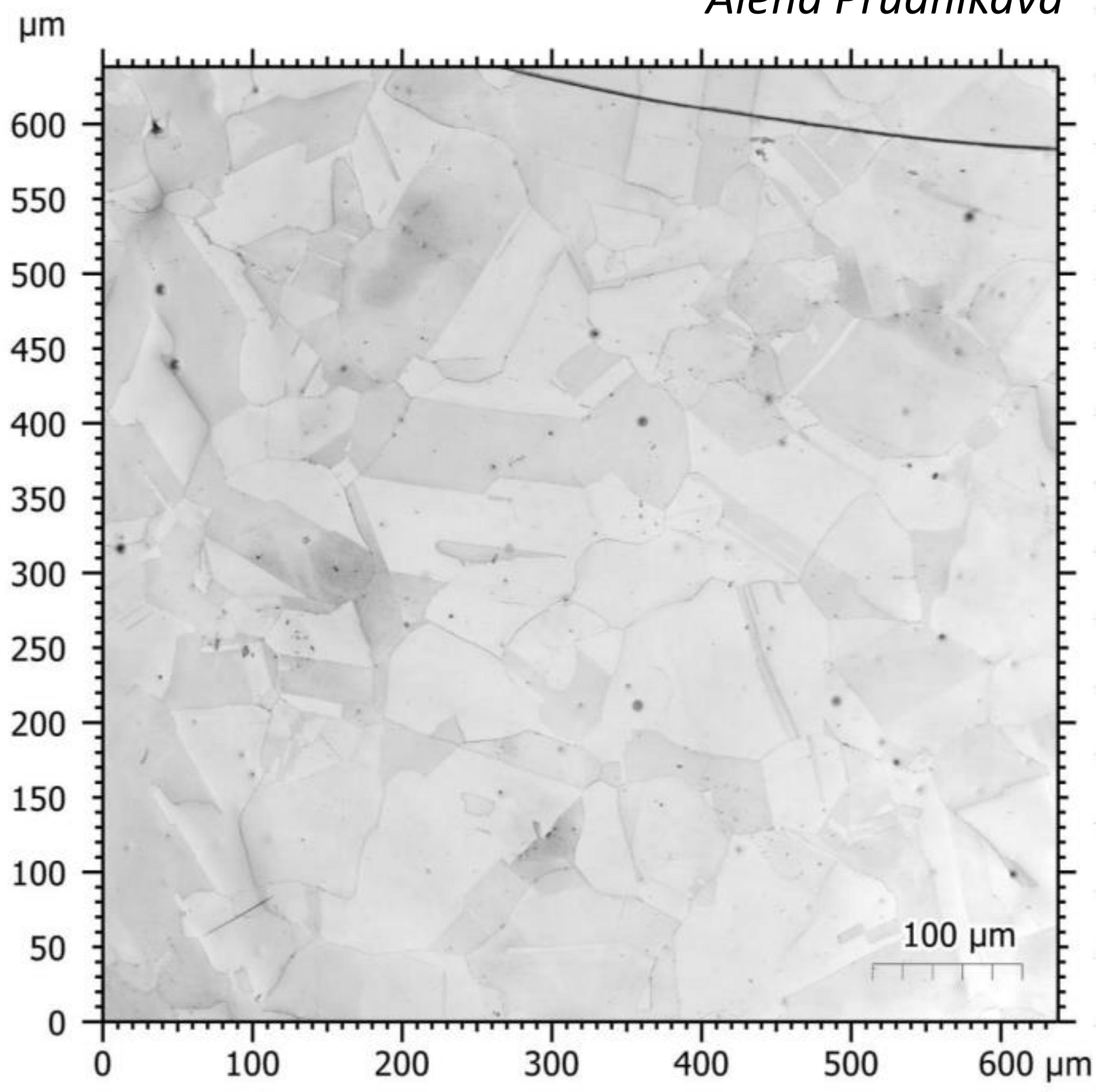
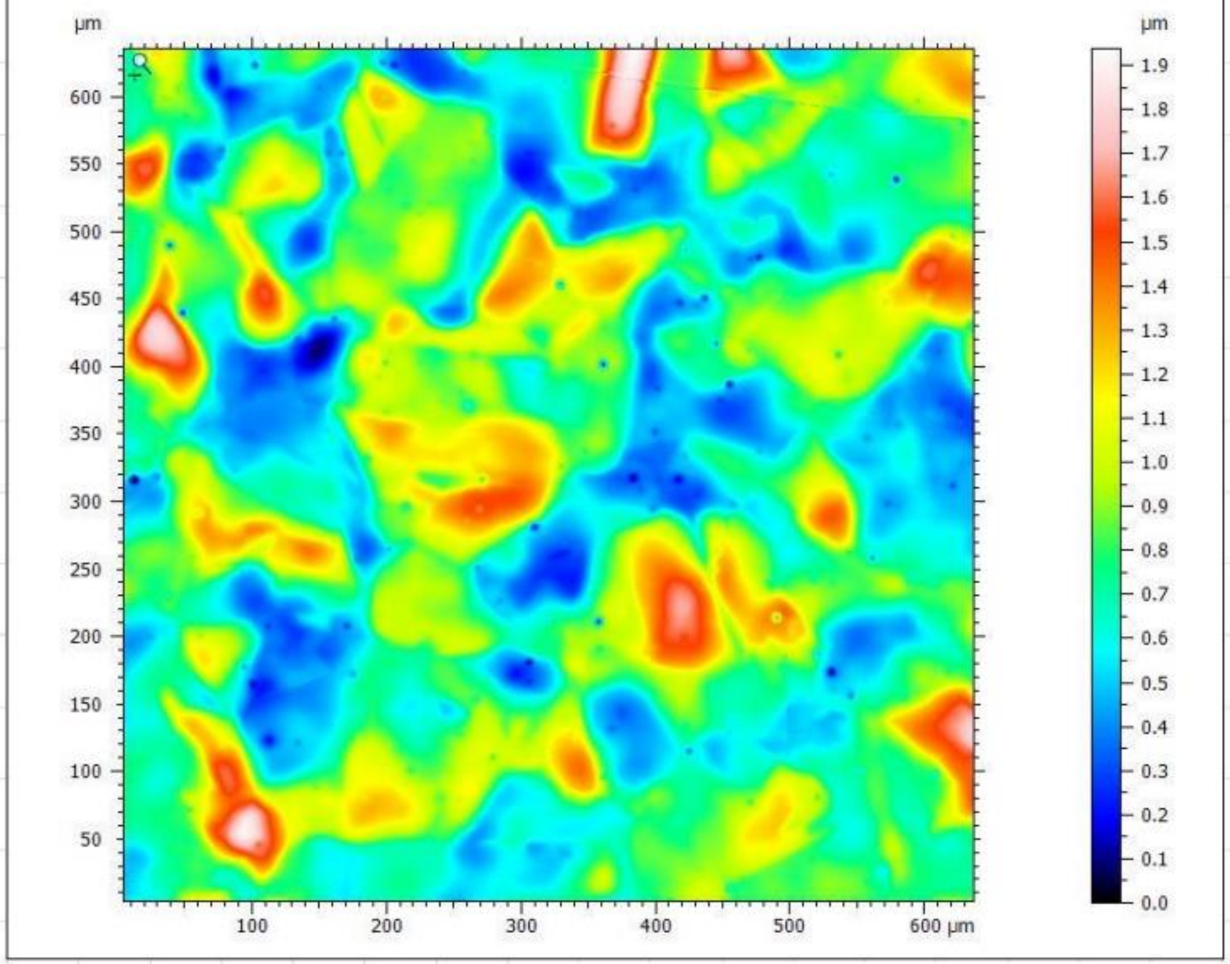
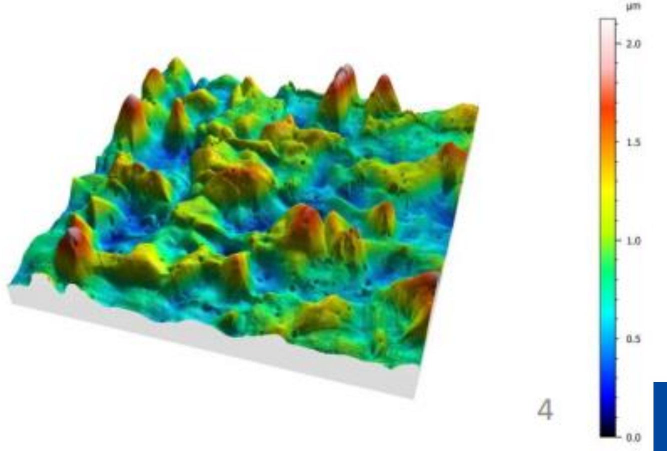


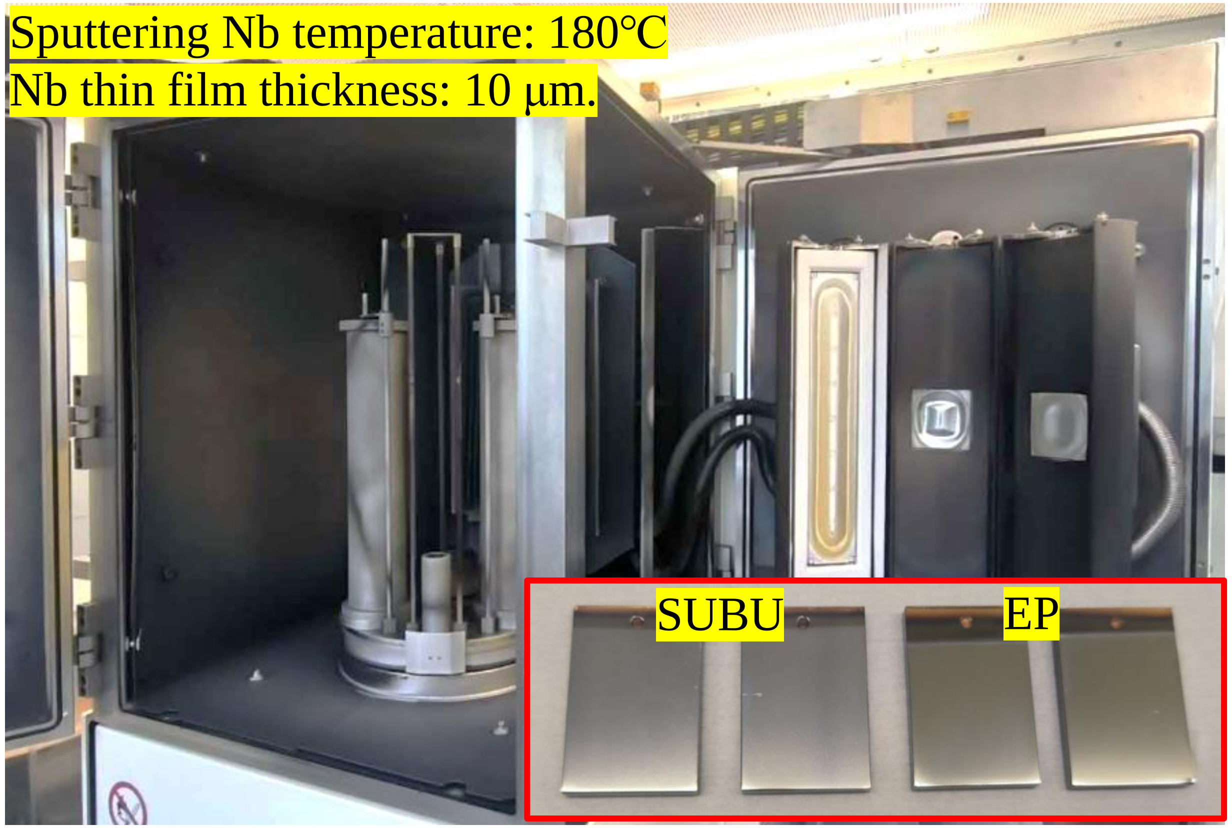
$S_Z = 1.94\ \mu m$

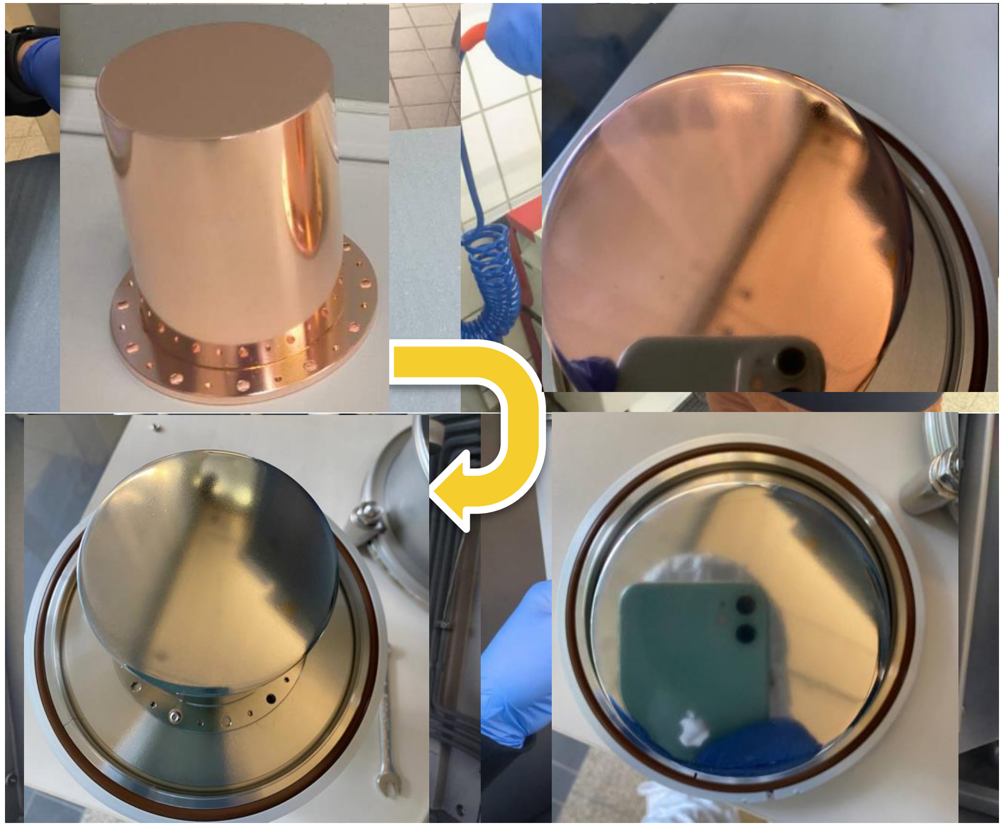


ISO 25178 - Primary surface		
F: [Workflow] Form removed (LS-poly 2)		
λs Filter: [Workflow] λs-filtered (λs 2.500 μm)		
Height parameters		
Sq	0.2881	μm
Ssk	0.6972	
Sku	3.504	
Sp	1.145	μm
Sv	0.7937	μm
Sz	1.938	μm
Sa	0.2259	μm



Step 3: Cu QPR sample sputtering Nb at Siegen





Dr. Aleksandr Zubtsovskii

- Cu QPR samples were sputtered with Nb thin films about 10 μm using HiPIMS at Siegen.
- The sputtering temperature is about 180°C, so annealing is required before bronze plating to eliminate thermal expansion mismatch problem.

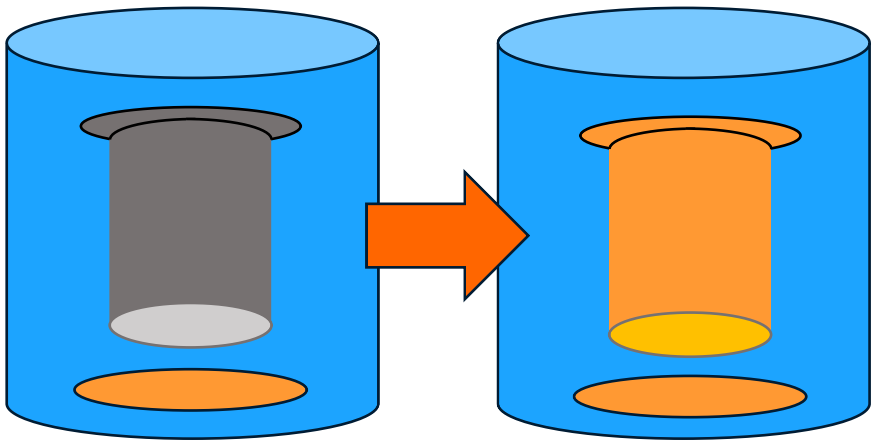
Step 4: Nb/Cu QPR sample electroplating process

Optimized anode structure and key parameters:

- **Anode structure:**

  1. QPR samples are placed upside down to reduce the deposition of impurities from the anode

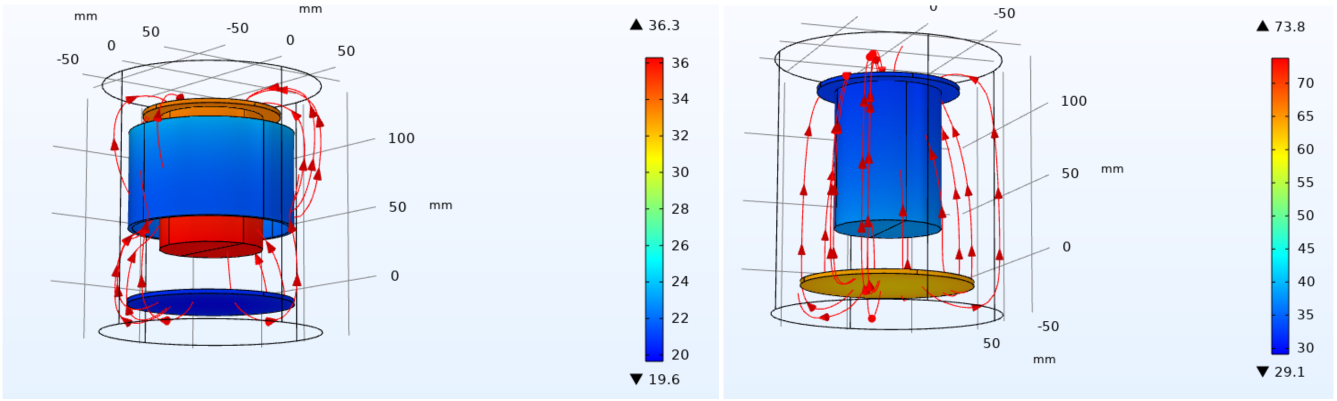
  2. Only disk anode



- **COMSOL simulation:**

  The difference in current density: 1-2 times

  Range: 3-4 mA/cm^2



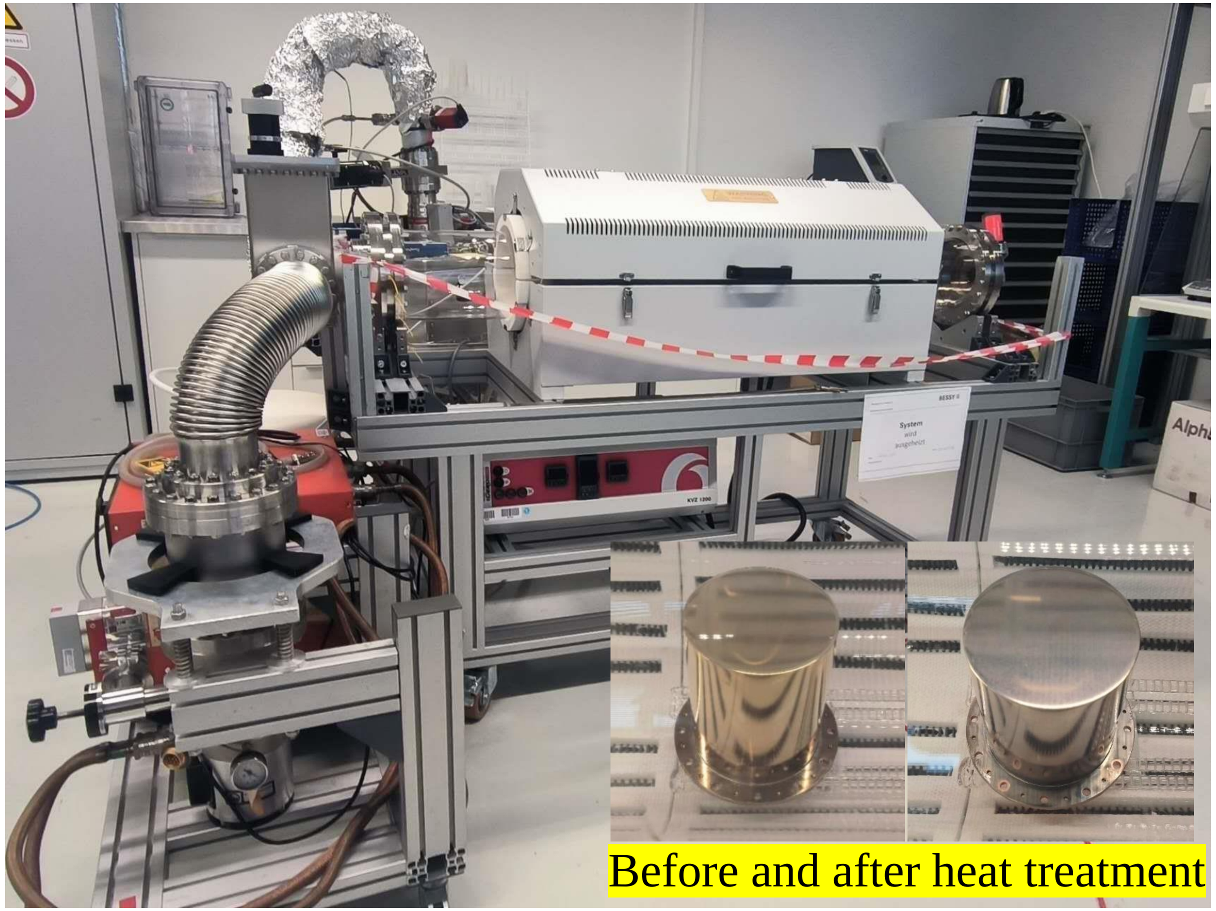
- **Parameters:**

  1. CC mode: 0.3 A
  2. Electrode distance: 50 mm
  3. Temperature: 15 ℃
  4. Stirring speed: 100 rpm
  5. Electroplating time: 2 h (10 μm)

Step 5: The QPR sample Heat Treatment at 700°C

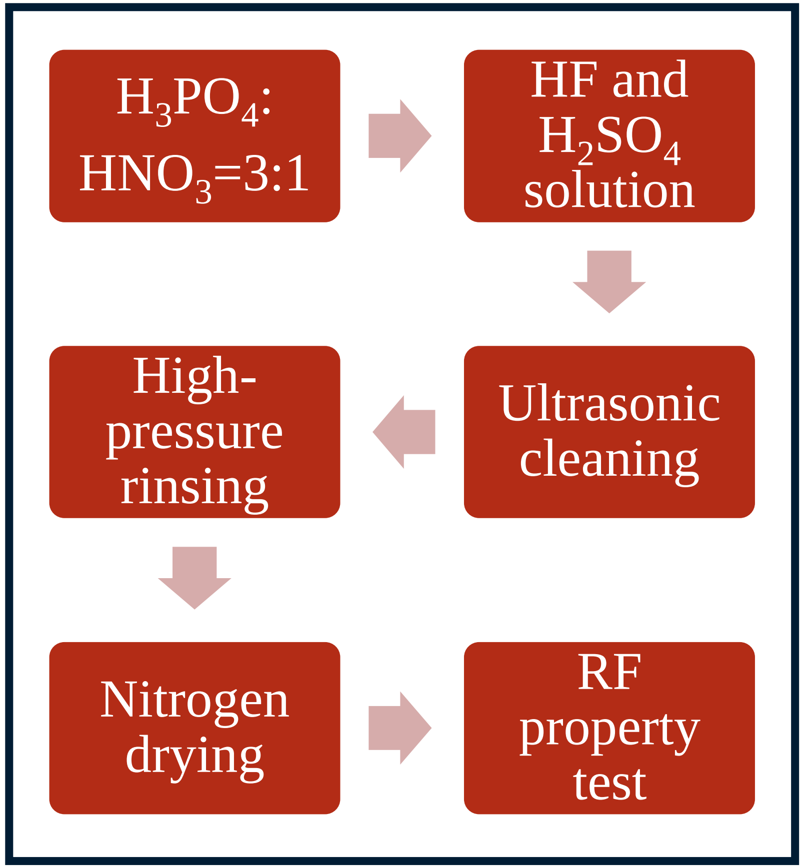
Optimized furnace structure and vacuum pressure:

- **Specific process:**
  1. Placed vertically in a vacuum tube annealing furnace.
  2. QPR samples are placed vertically with glass sample holders at the bottom.
- **Parameters:**
  1. Heat treatment curve: 600°C(30h)+700°C(30h).
  2. Heating and cooling rates:
     0.5°C/min and 9°C/min .
  3. Vacuum pressure:
     5E-9mbar (room temperature)
     6E-8mbar (at 700°C)



Before and after heat treatment

Step 6: The QPR sample polishing top impurity



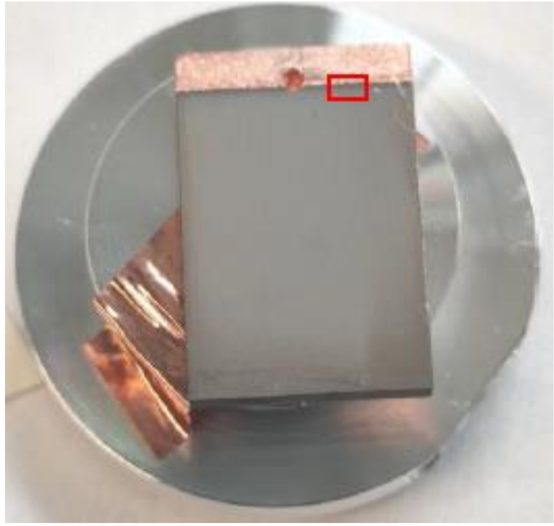
- The thickness of the Nb film at the bottom of the QPR sample is too thin, causing the Nb_3Sn film to fall off after polishing, exposing the Cu substrate.
- By removing impurities such as bronze, niobium oxide, and carbide on the surface, we obtained a clean Nb_3Sn/Cu QPR sample.

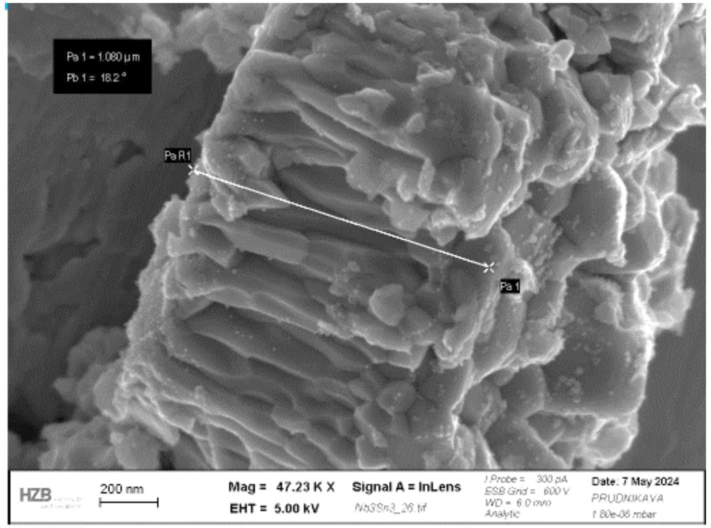
Small Nb_3Sn/Cu samples Heat Treatment at 700°C

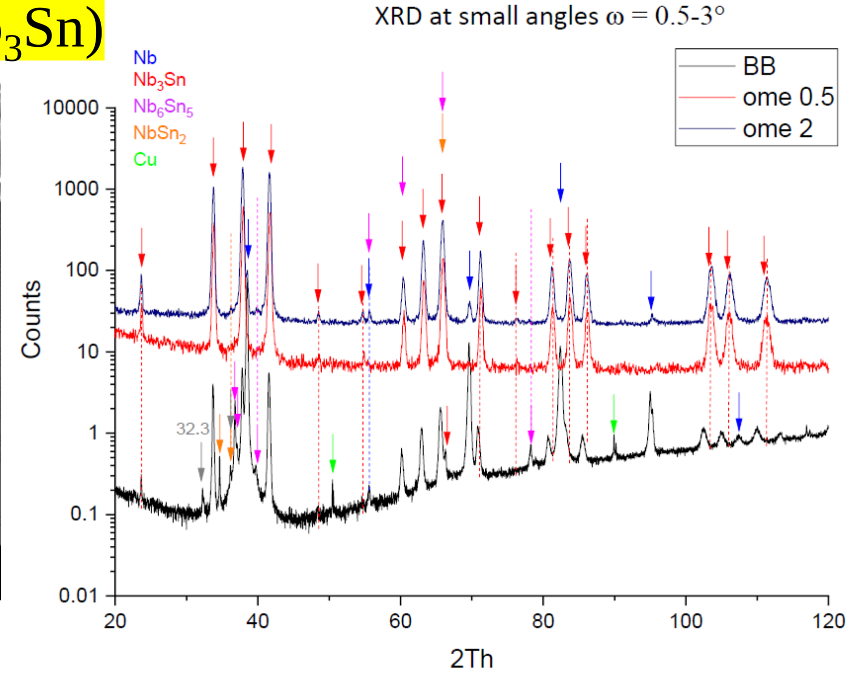
- I have prepared 6 small Nb_3Sn/Cu samples using the same process and characterized the Nb_3Sn coatings by LSM, SEM, EDX, XRD, PPMS, etc.

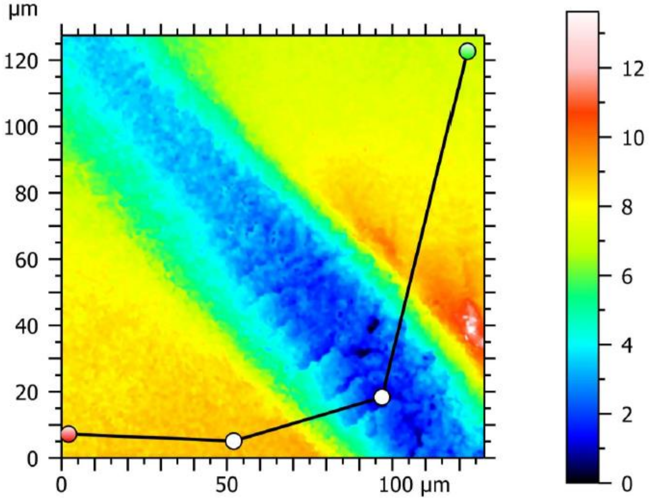
Small Nb_3Sn/Cu samples characterization

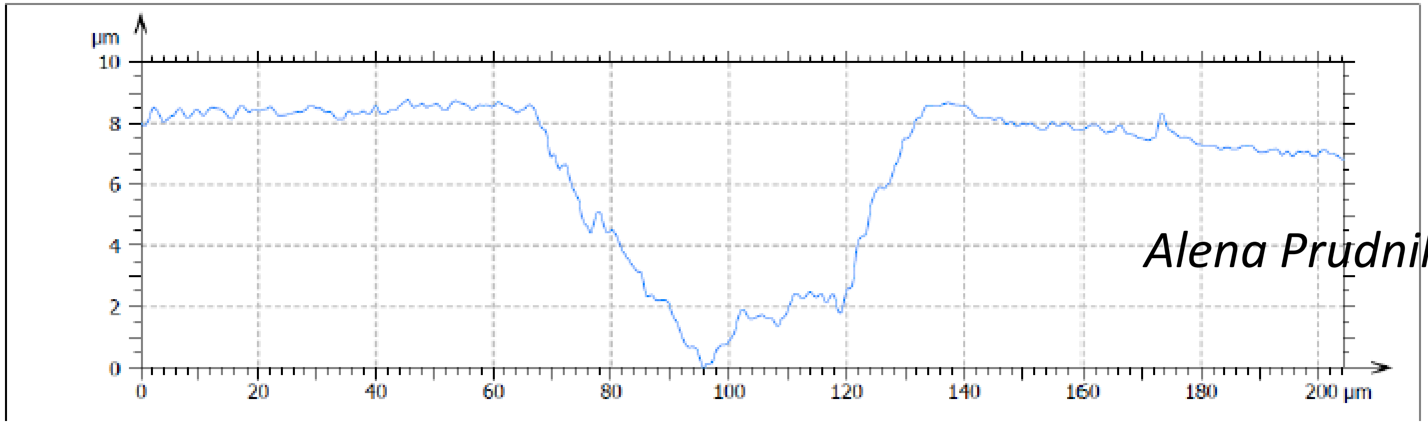
Zeiss LSM: total film thickness 9 µm (8 µm Nb+1 µm Nb_3Sn)











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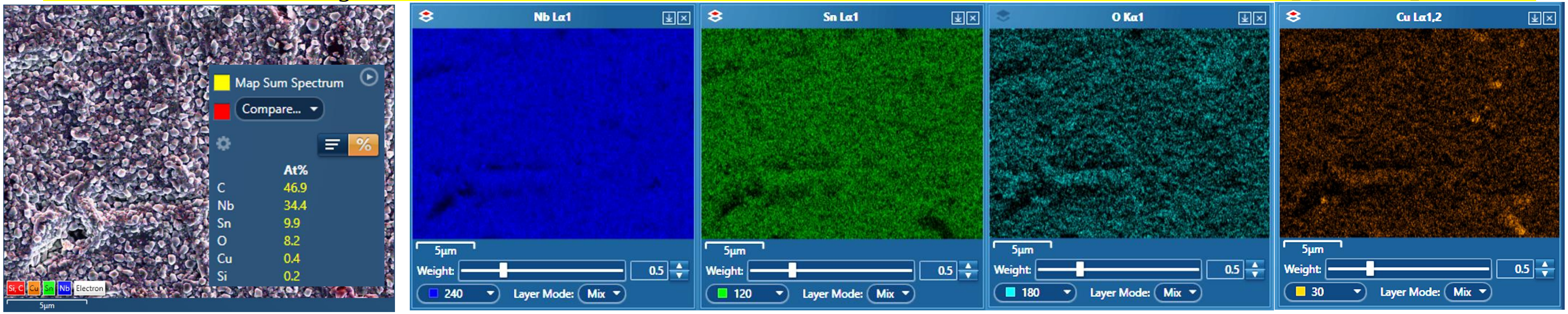
Small Nb_3Sn/Cu samples characterization

SEM and EDS: Nb_3Sn surface Sn content 22.35 at%, contains oxides and rare copper impurities.



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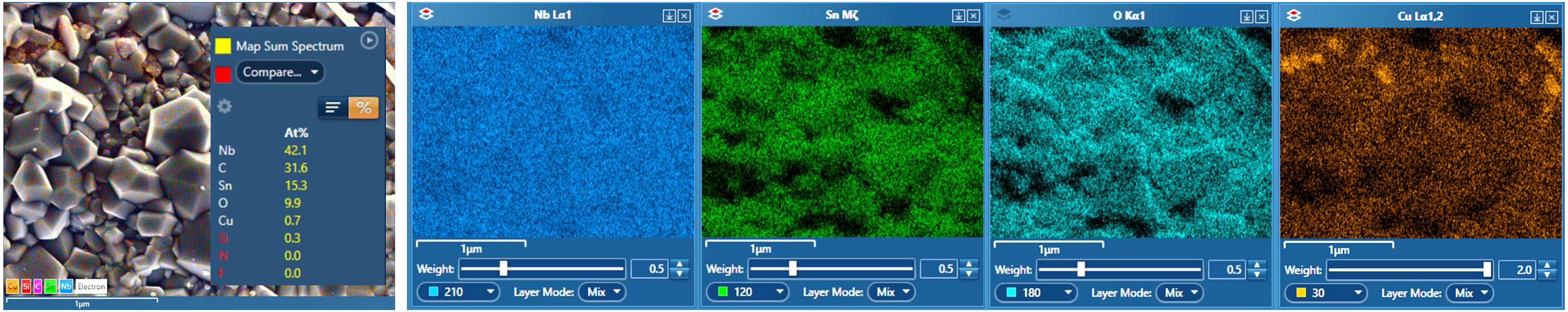

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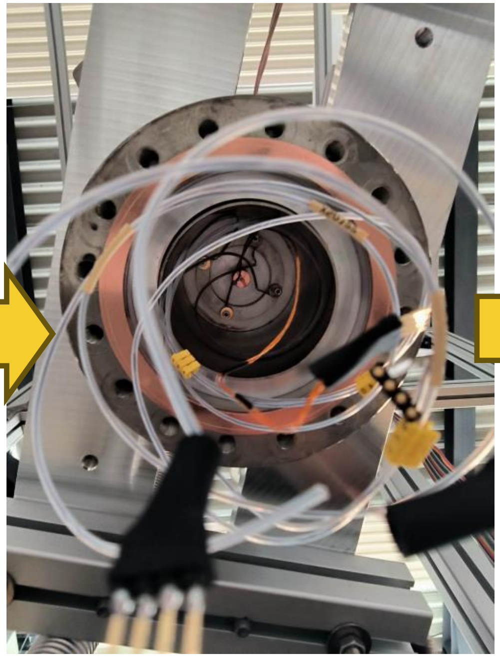
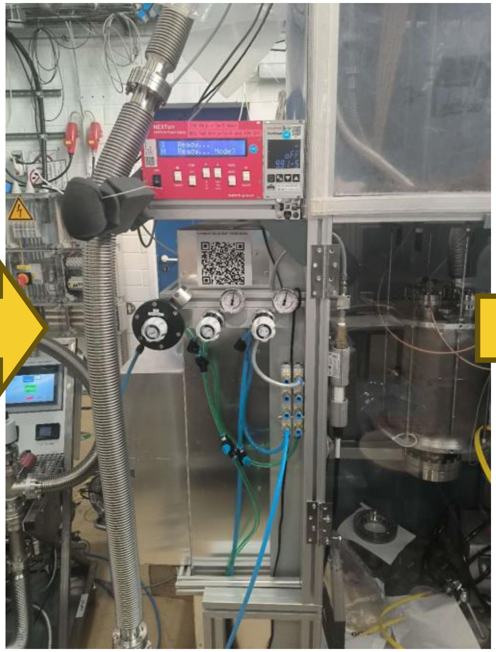
Nb/Cu QPR sample baseline test

Mount QPR sample

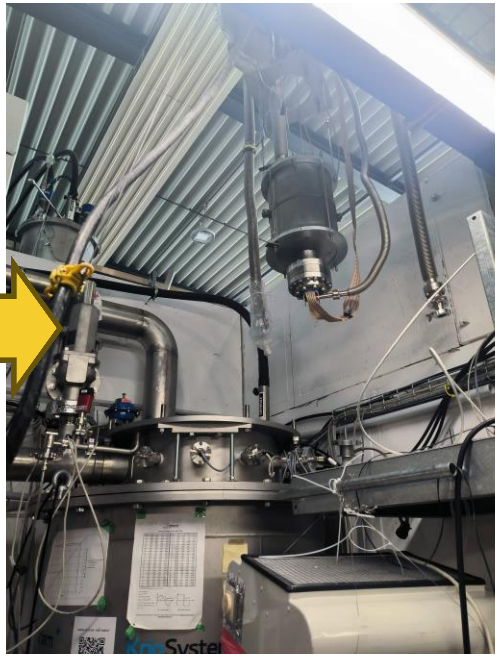
Mount sensor and heater



Check vacuum pressure



Move QPR to cryostat

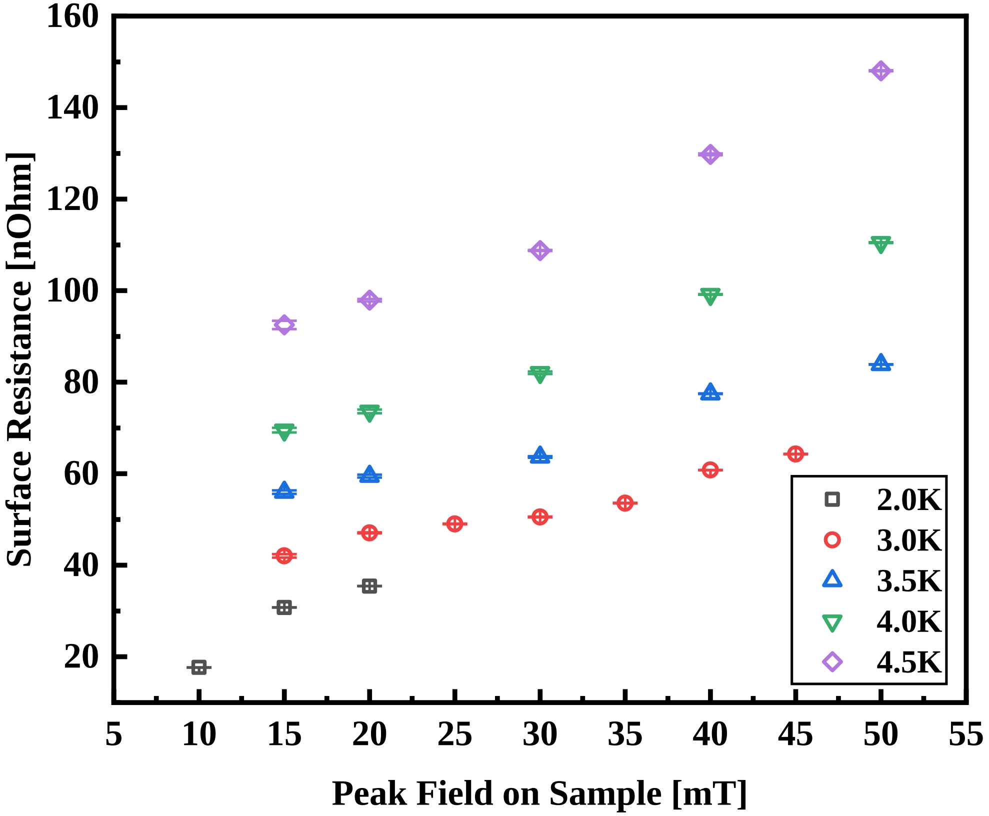


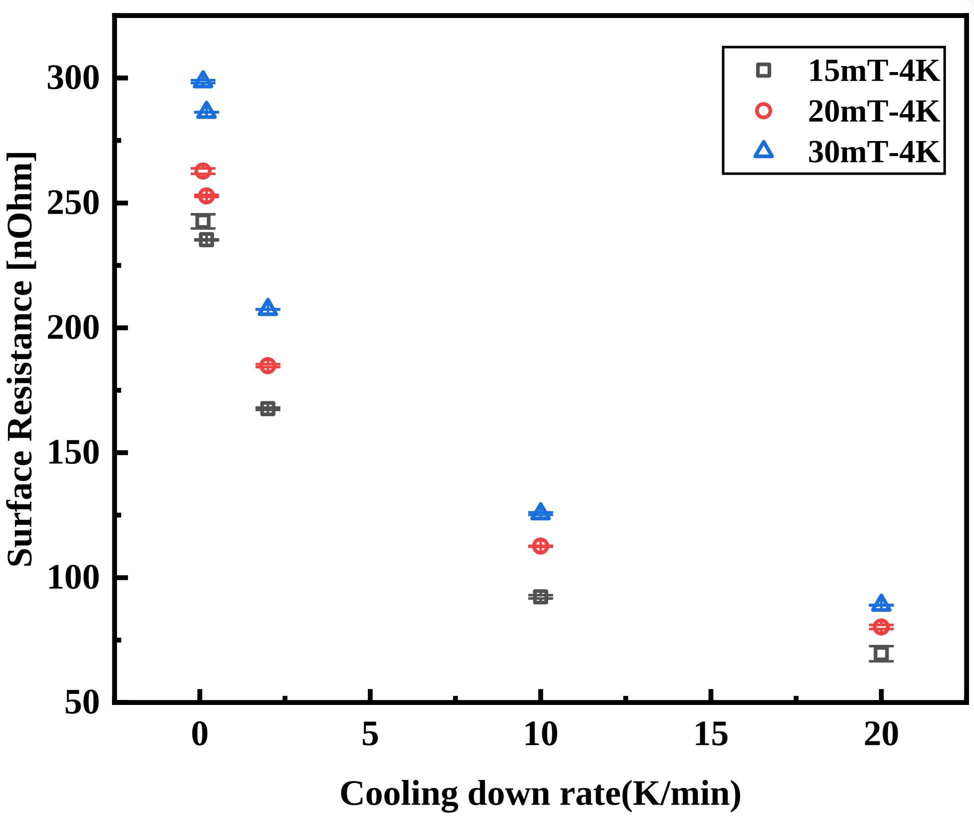
- The Nb/Cu QPR sample is not sealed with indium wire, but only connected with bolts, resulting in poor vacuum (resonator chamber: 7E-6 mbar, thermometry chamber: 2E-5 mbar).
- Control the gap distance by selecting the flange with the appropriate height and vector network analyzer test. (a total of 7 flanges with different heights)

Sebastian Keckert

Nb/Cu baseline QPR sample test

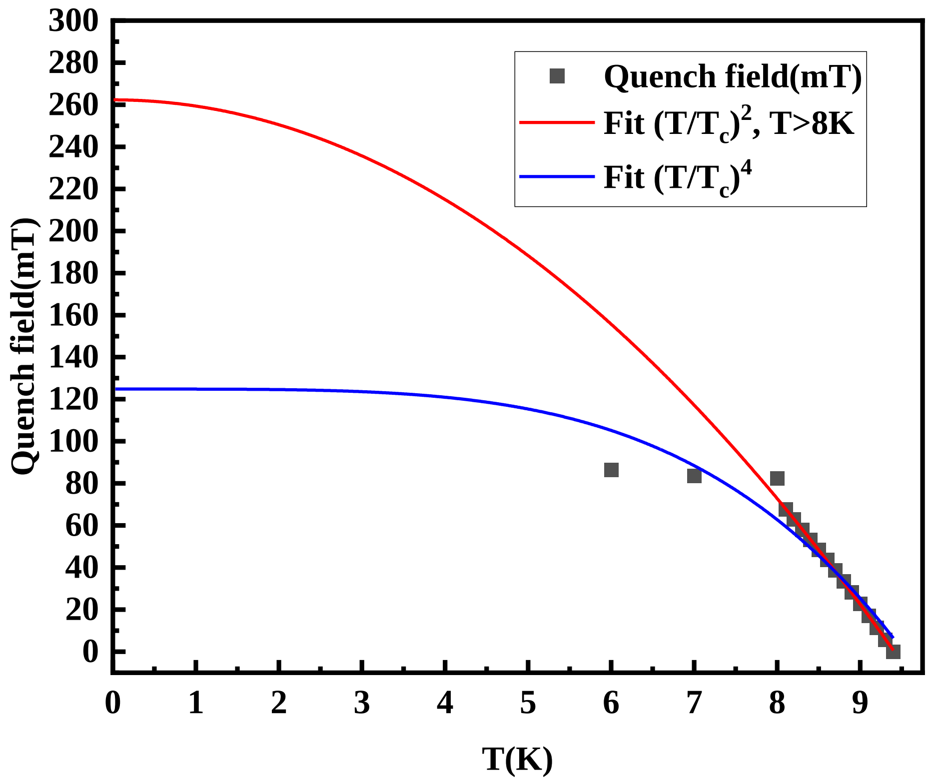
- *413 MHz: R_s* is measured to be about 33 nΩ at 2 K and 15 mT (R_{BCS} 1 nΩ +R_{res} 32 nΩ) .
- *The Nb coating quality is average:* Severe Q-slope and RF heat limit occur.

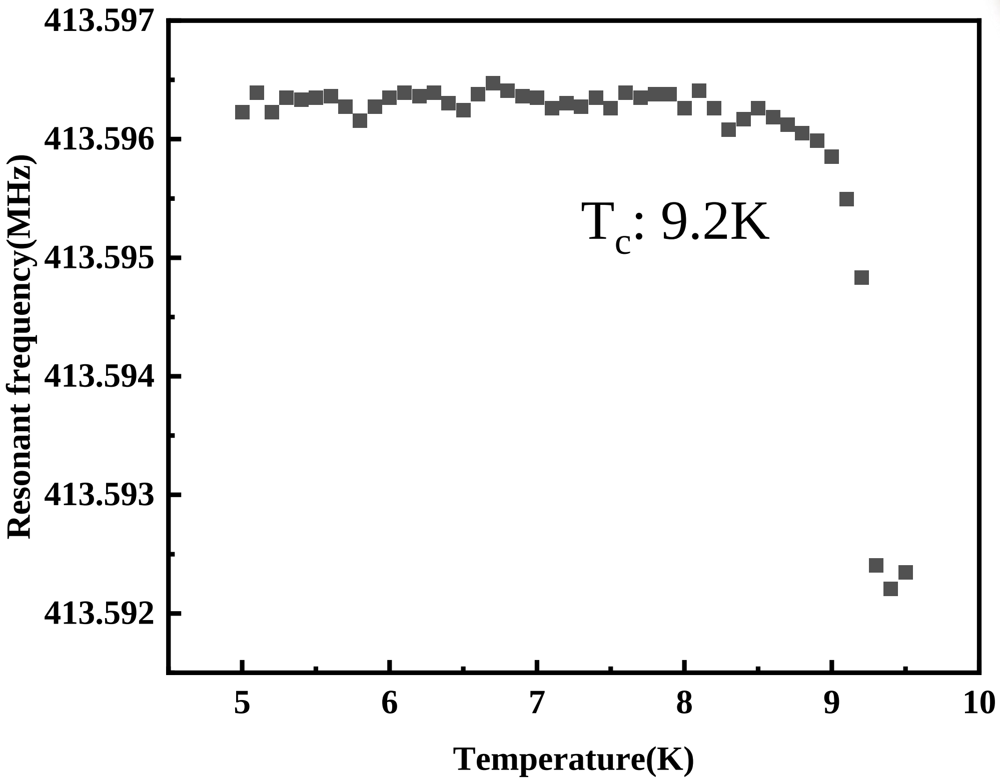




- *Performed multiple thermal cycles from 10-2K: 120-20* nΩ at 2K and 10mT, *240-60* nΩ at 4K and 15mT.
- Nb/Cu QPR sample: When cooling through the critical temperature, the larger the rate, the less thermal current and less magnetic flux is trapped.

Nb/Cu baseline QPR sample test

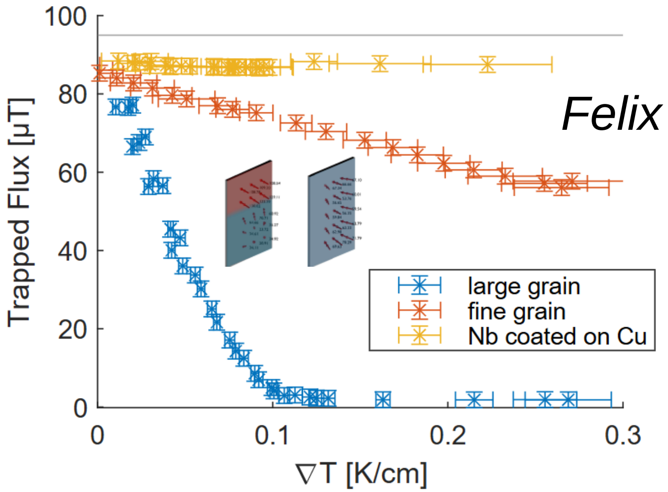




➢ From T-B_{quench} curve fitting $(T/T_c)^2$, T>8K, we got B_{quench}(0K): 260mT and T_c: 9.24K (VNA data)

Future work: From sample to Nb_3Sn/Cu cavity

- **Aim:**
  - Nb_3Sn/Cu surface impurities optimization. (Cu, Oxides, Carbides).
  - Nb_3Sn/Cu RF loss analysis and loss mechanism study.
  - Nb_3Sn coating growth mechanism study and grain control.
- **Next:**
  - Nb_3Sn/Cu QPR sample test scheduled in Oct 2024.
  - Trap flux study of Nb_3Sn coating on Cu substrate (TraMaFlu).
- **Future:**
  - Small samples -> larger samples -> QPR samples -> 1.3GHz cavities.
  - 1.3GHz copper cavity Nb sputtering + bronze route Nb_3Sn coating.



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Summary

- Cu-based Nb_3Sn combines the excellent thermal conductivity of copper and the superior superconducting properties of Nb_3Sn in SRF field.
- The ETS bronze route is one method to achieve Nb_3Sn coating on copper. Its advantages are low cost, simple operation, suitable for complex cavity types and mass production.
- We provide a complete set of QPR sample preparation processes from copper electropolishing, Nb sputtering, electrodeposition and heat treatment to synthesize Nb_3Sn.
- By optimizing the entire preparation process and key parameters, a new Cu-based Nb_3Sn QPR sample was successfully prepared, and its RF properties will be characterized by QPR testing system at HZB soon.

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