Cu-based Nb_3Sn QPR sample preparation via bronze route

Ming Lu
Helmholtz-Zentrum Berlin (HZB)
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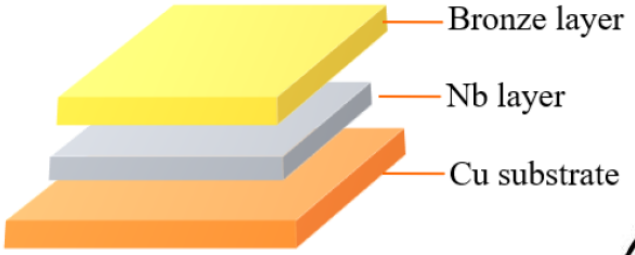
Outlines

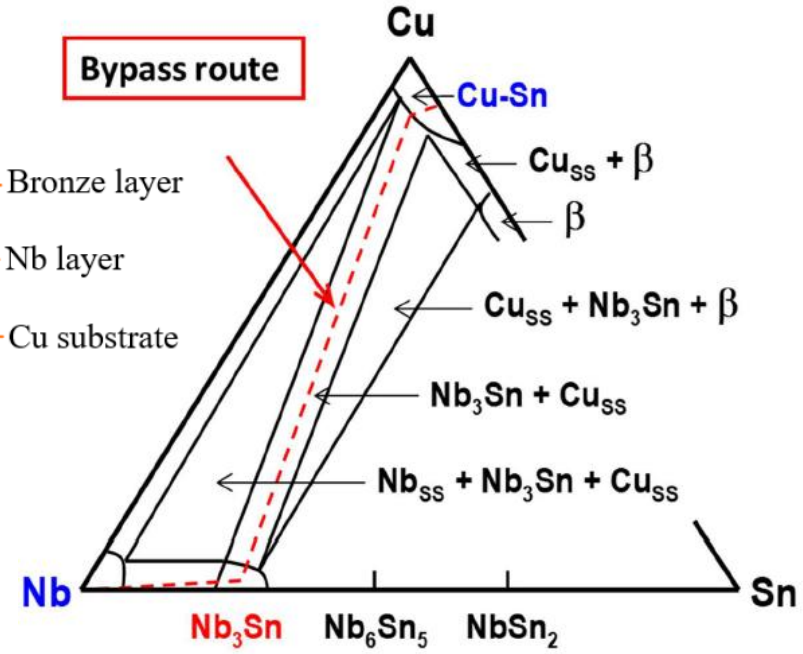
1. Background and research basis
2. Nb_3Sn sample preparation activities
    - 1: Nb_3Sn/Cu QPR sample
    - 2: Nb_3Sn/Nb QPR sample
    - 3: Optimization of bronze route Nb_3Sn
3. Next step work plan schedule

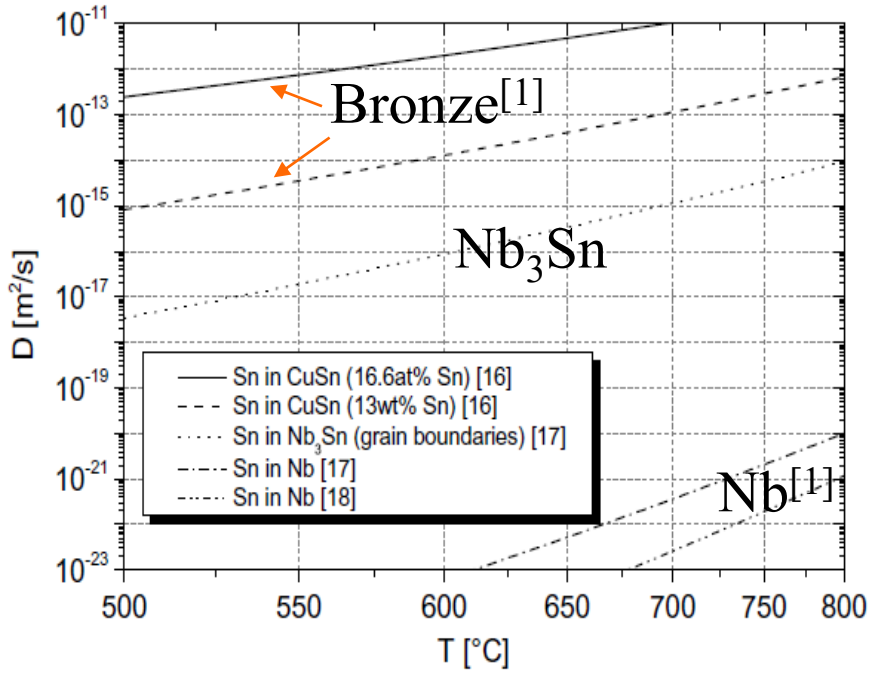
One achievable way : Nb_3Sn/Cu bronze route



Possible precursor structure for bronze route.

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



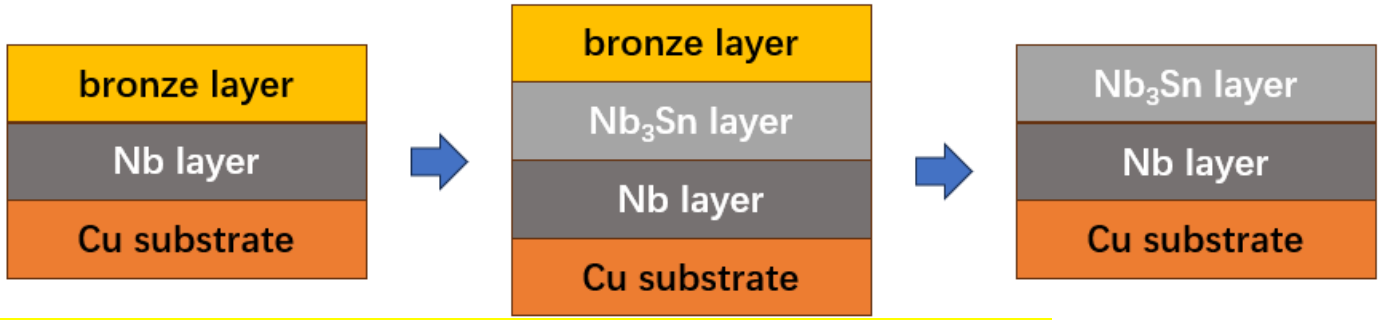


1. The copper can facilitate the interdiffusion between Nb and Sn by 7~10 magnitudes of orders.
2. The copper alloy will be excluded from the Nb_3Sn phase by itself.

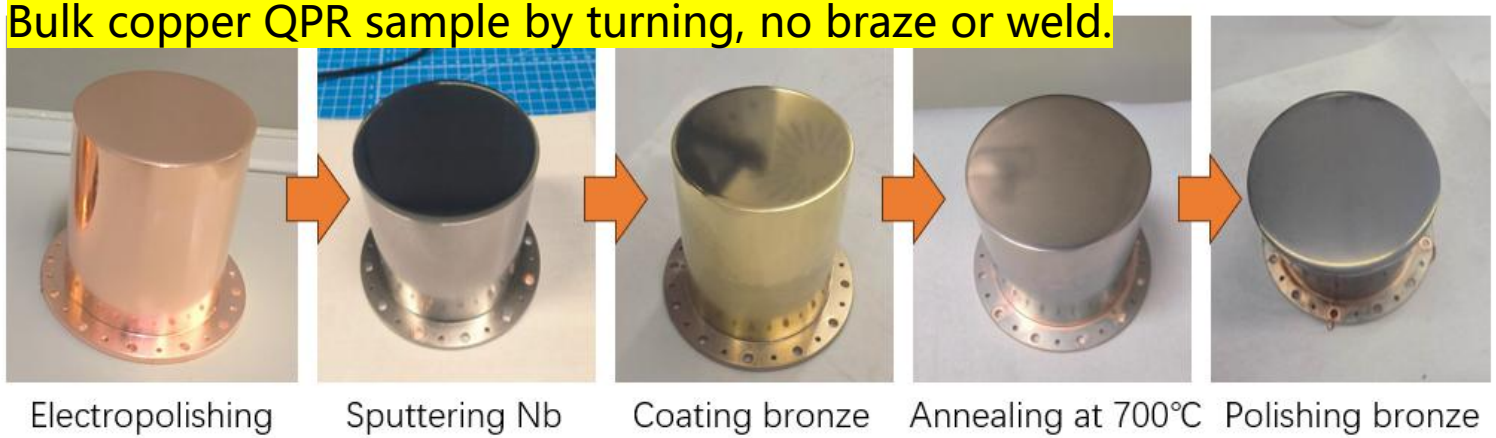
[1] H. Müller and T. Schneider, "Heat treatment of Nb3Sn conductors," Cryogenics, vol. 48, pp. 323-330, 2008/07/01/ 2008.
[2] L Mei, Z Du, C Guo, & C Li. (2009). Thermodynamic optimization of the cu-sn and cu-nb-sn systems. Journal of Alloys & Compounds, 477(1-2), 104-117.

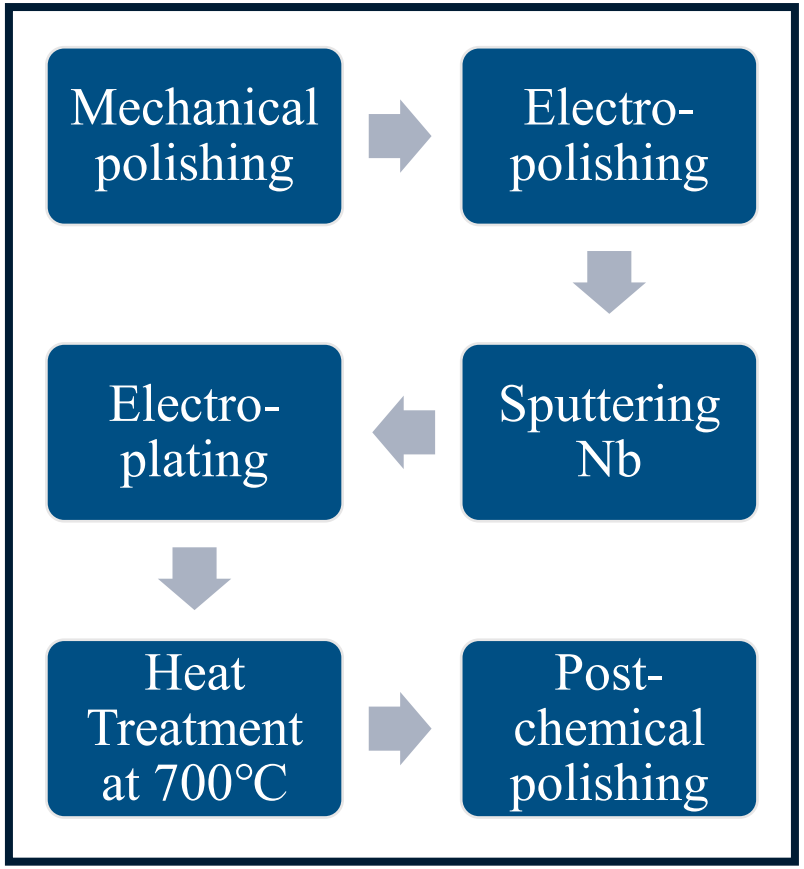
Nb_3Sn/Cu QPR sample prepare procedure

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



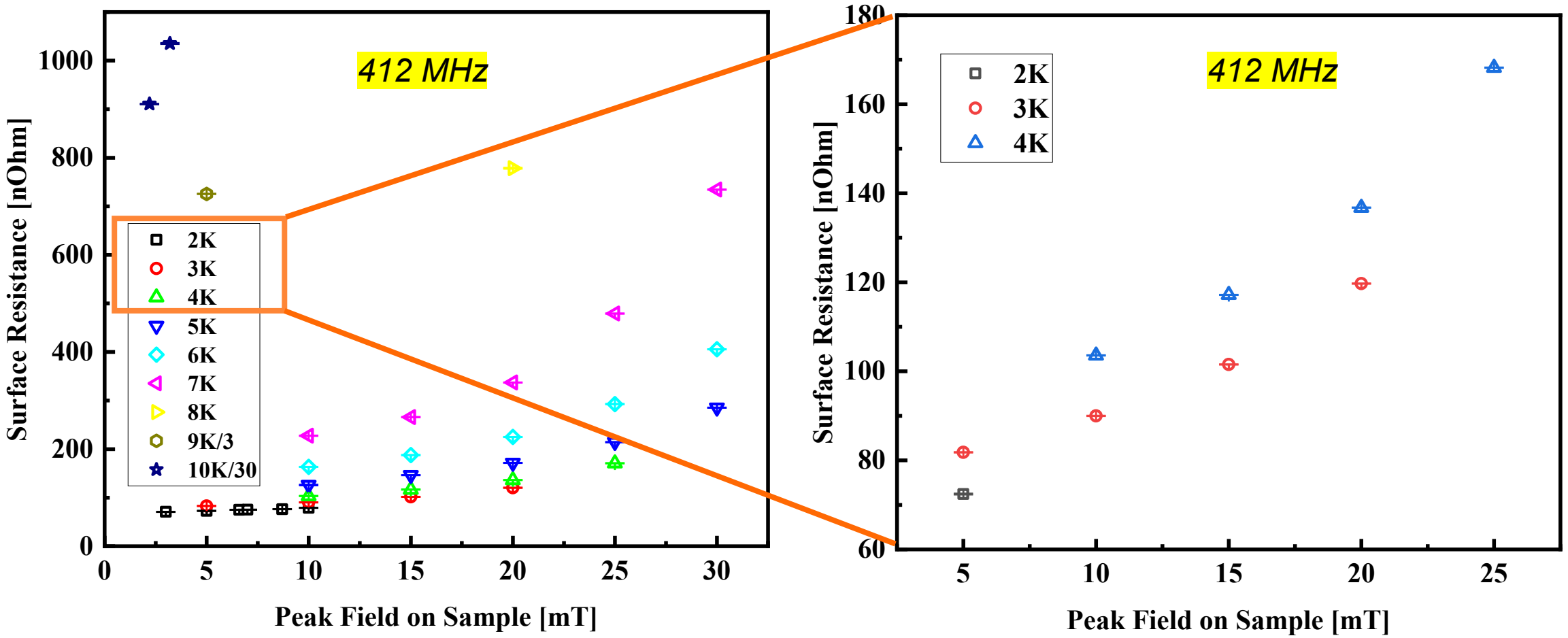
Bulk copper QPR sample by turning, no braze or weld.





- Several Cu-based Nb_3Sn QPR samples were successfully prepared.
- The first Nb_3Sn/Cu QPR sample RF properties have been tested at HZB.

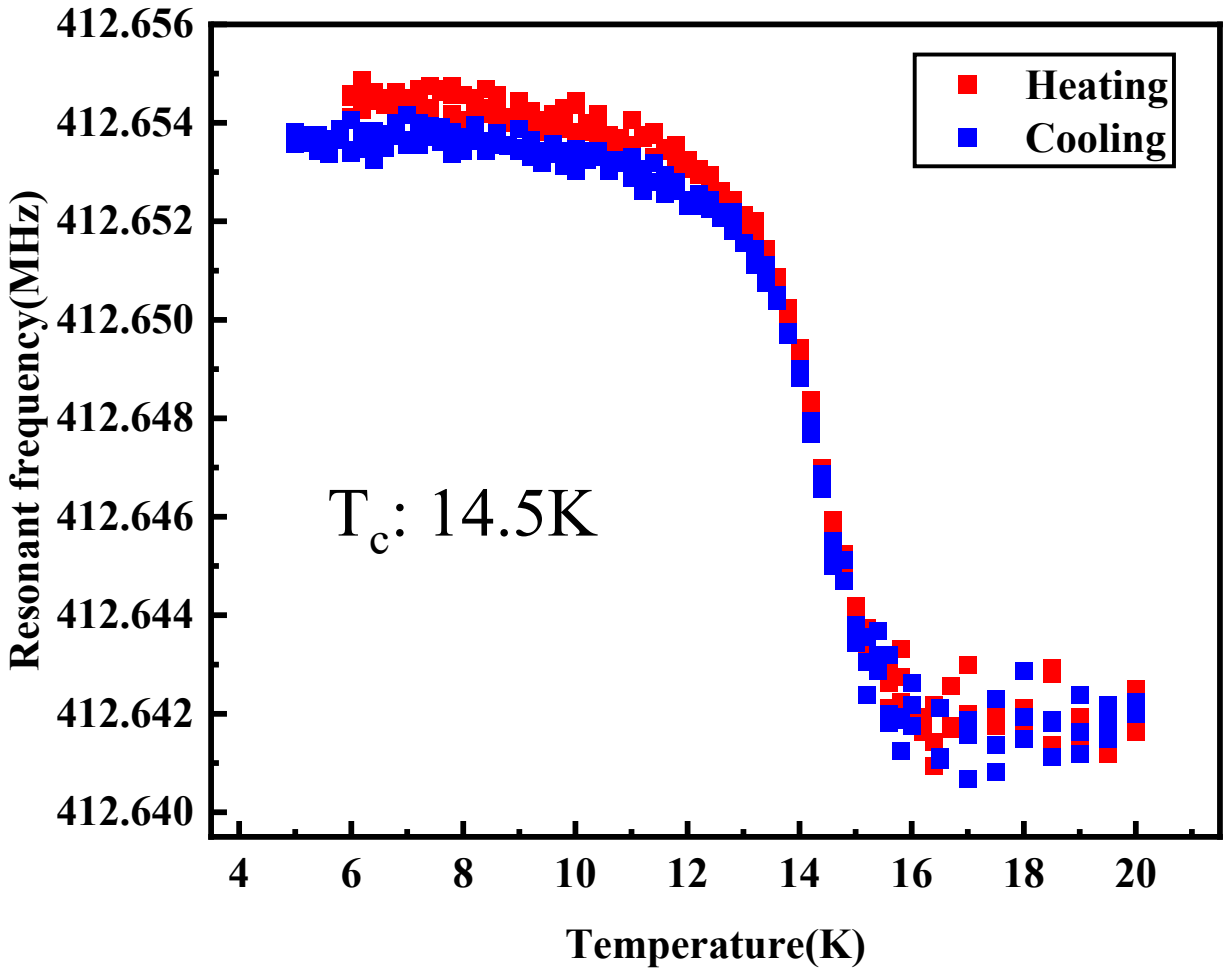
First Nb_3Sn/Cu QPR sample RF test

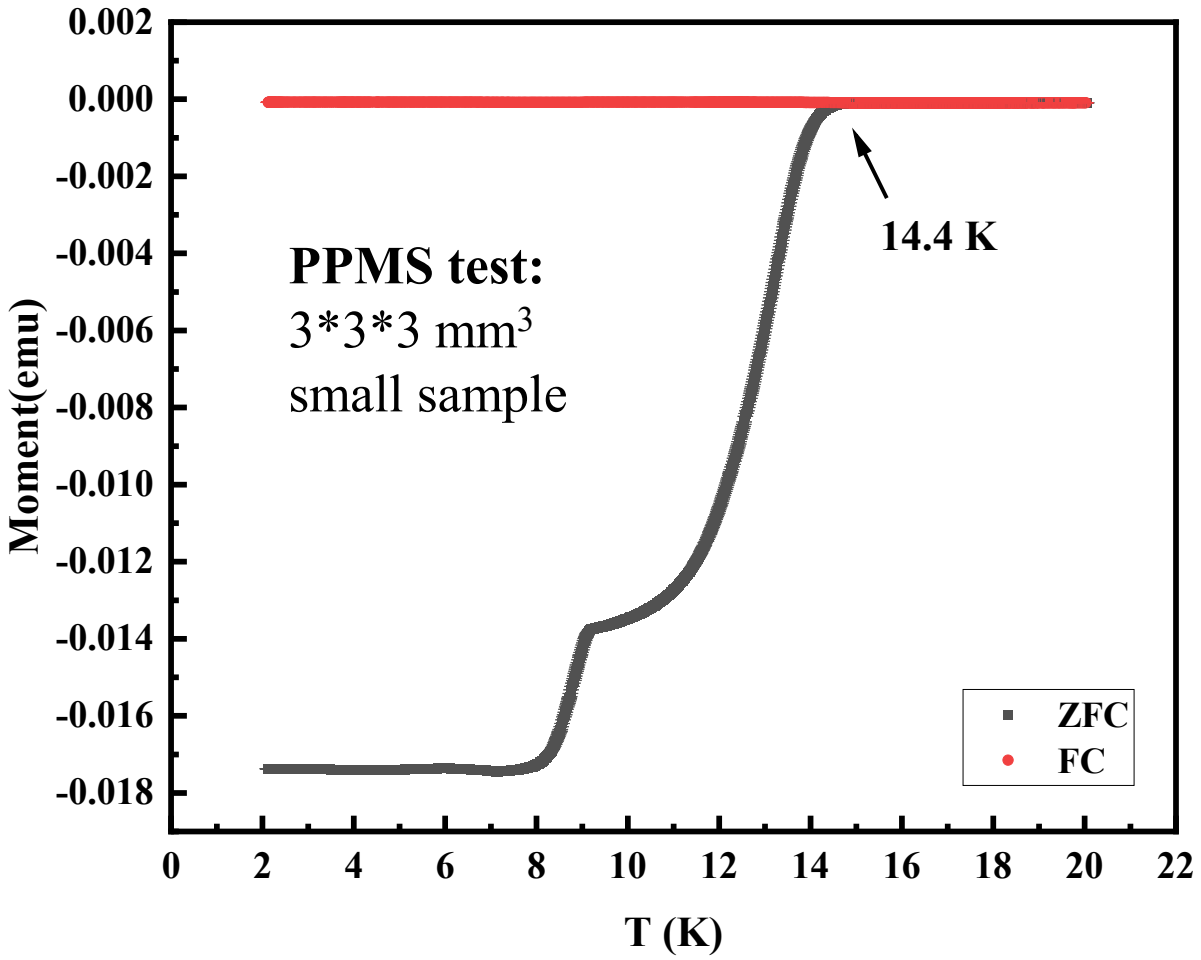


- *412 MHz:* R_s is measured to be about 104 nΩ at 4 K and 10 mT (main R_{res}) .
- *The Nb_3Sn/Cu coating quality is not very good:* Severe Q-slope and RF heat limit occur.

Sebastian Keckert

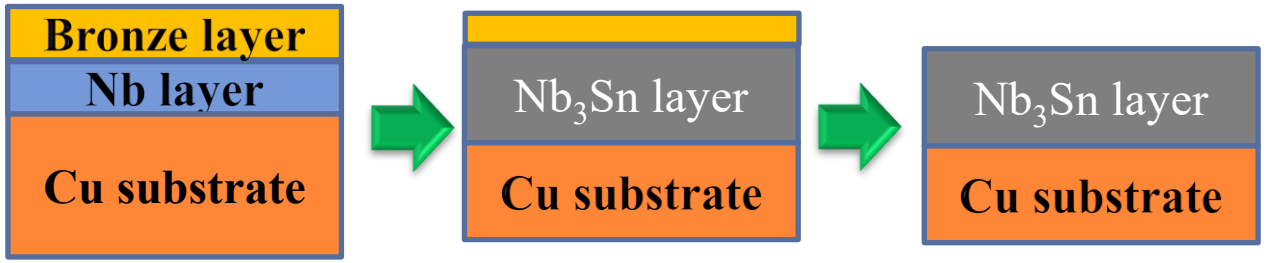
First Nb_3Sn/Cu QPR sample RF test





- The superconducting transition temperature of Nb_3Sn is low (VNA data), which is suspected to be caused by insufficient tin content in the Nb_3Sn sample. This has been verified in Nb_3Sn/Cu sample PPMS tests.

Current Nb_3Sn/Cu problems and solutions

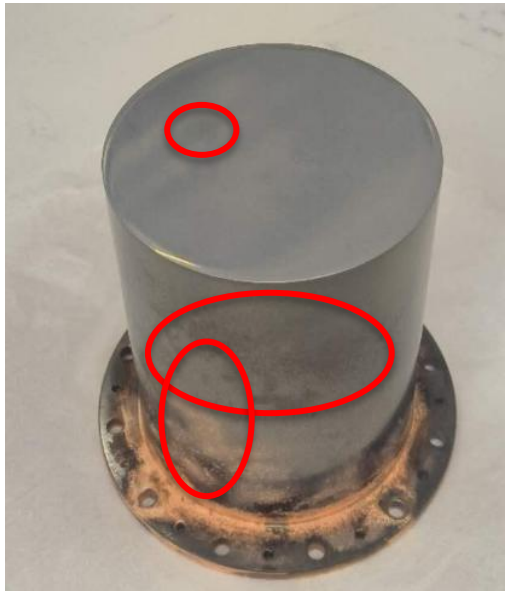


Nb_3Sn/Cu Problems:

① T_c: 14K, R_s: 104nΩ @4.0K,10mT

② Porous Nb_3Sn, Nb exposed on top, Cu exposed on side and bottom

③ Surface contamination, impurities



The first Nb_3Sn/Cu QPR sample

Several parts of challenges:

1. Nb layer: Porous Nb -> Porous Nb_3Sn

Solutions: **Hipims, Nb: T>600°C, Thickness~30μm, FE/laser treatment, Nb/Ta(Tantalum)/Cu layer**

2. Structure: Bottom of QPR sample expose Cu

Solutions: **Only coating bronze on top area**

3. Impurity: CuO_2,SnO_2,Nb_2O_5,C-H…

Solutions: **Ultrasonic cleaning, Aqua regia, HF activator**

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1. Nb/Cu QPR sample: Only coating top area

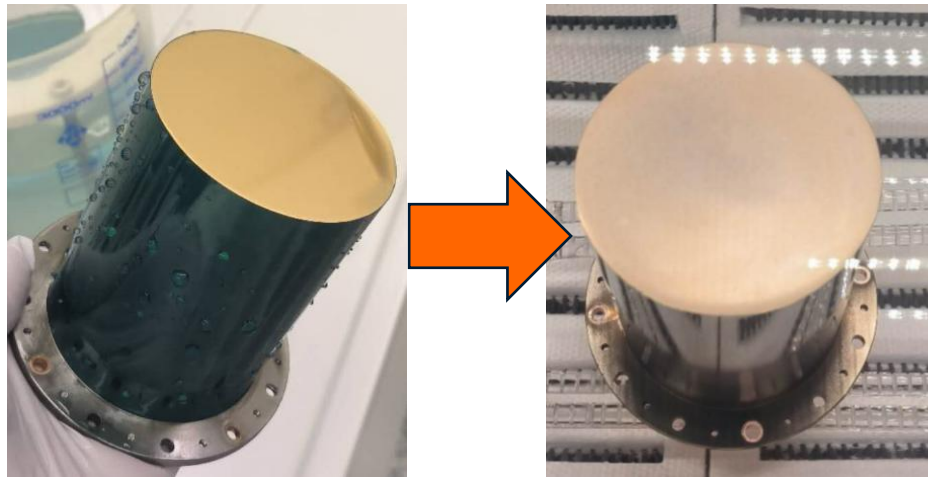
Optimized anode structure and key parameters:

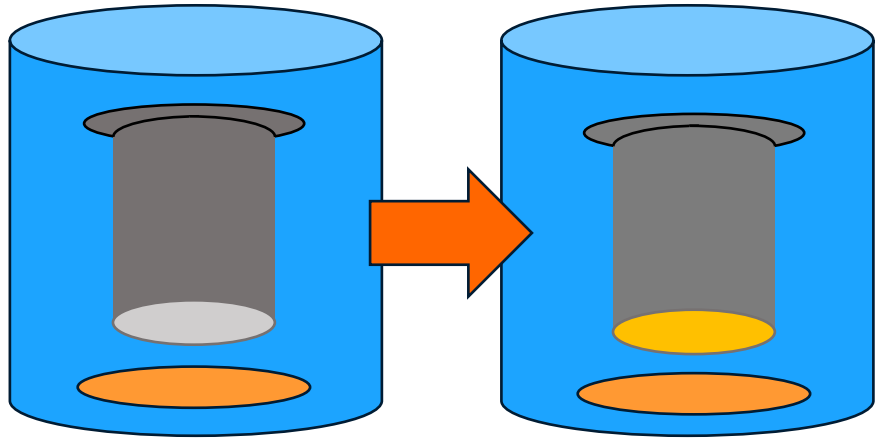
- **Anode structure:**
  1. Electroplating protective glue treatment, only the top area of the QPR sample is electroplated
  2. Only disk anode
- **COMSOL simulation:**

  The difference in current density: 1-2 times

  Current range: 3-4 mA/cm^2
- **Parameters:**

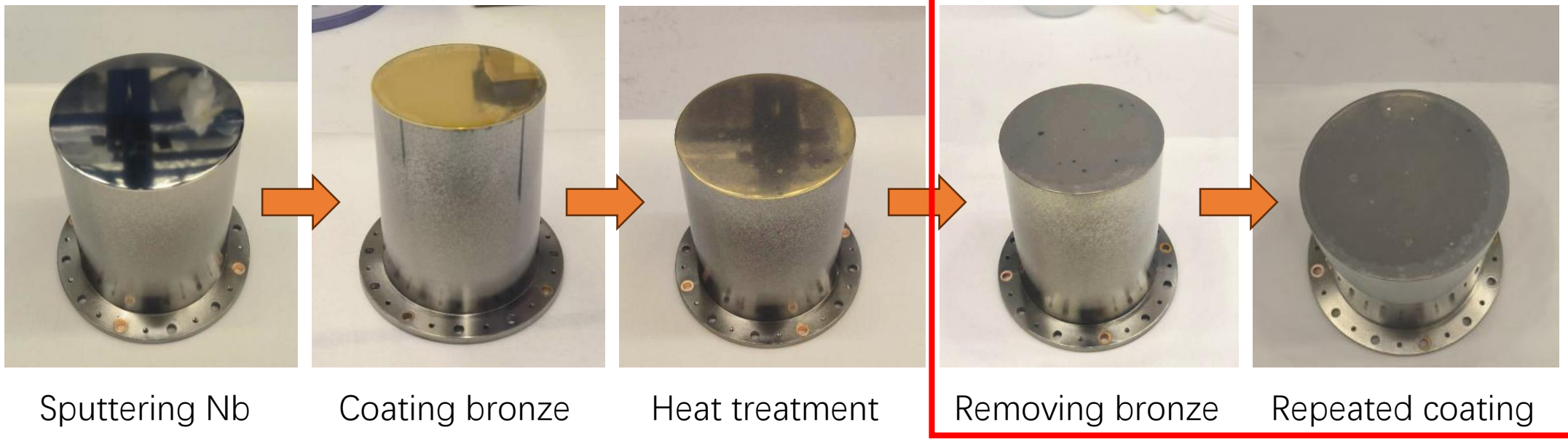
  1. CC mode: 0.1 A
  2. Electrode distance: 50 mm
  3. Temperature: 15 ℃
  4. Stirring speed: 100 rpm
  5. Electroplating time: 2 h (10 μm)





1. Nb/Cu QPR sample: Only coating top area

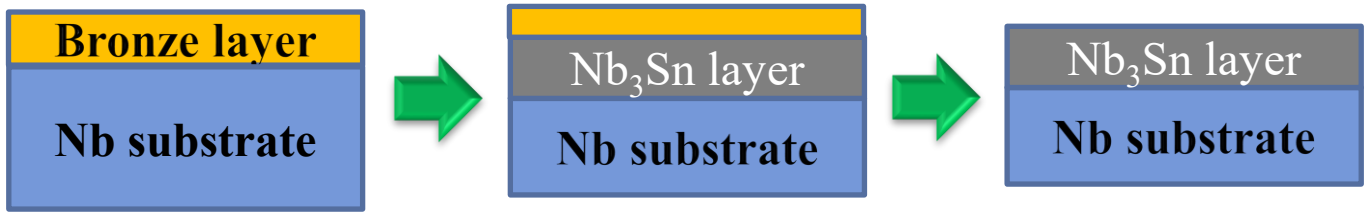
- ✓ To prevent copper exposure on the bottom, we only perform bronze coating on the top.
- ✓ This sample is testing for RF performance at HZB now (37 nΩ at 4 K and 10 mT).



- ➢ The QPR sample was bronze plated on the top and heat treated, but holes were found, which were caused by bubbles on the surface.
- ➢ We have conducted a repair experiment by slowly dipping the surface into the solution at the 30 angle to reduce surface bubbles.

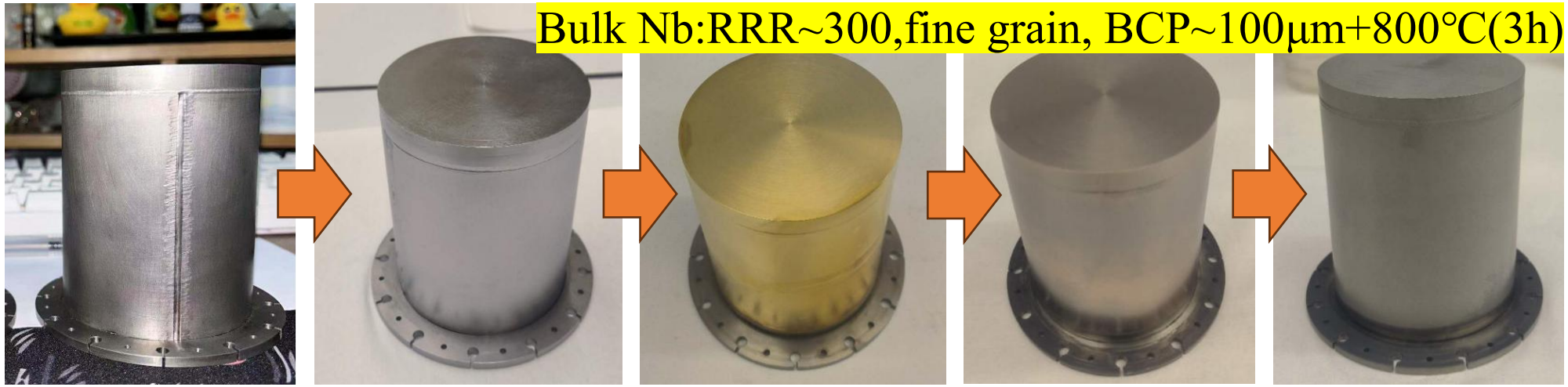
2. bulk Nb QPR sample preparation

- Bulk Nb QPR sample fabrication in China and baseline test at HZB.
- Nb_3Sn/Nb as an intermediate step to accelerate iterative optimization of bronze process.



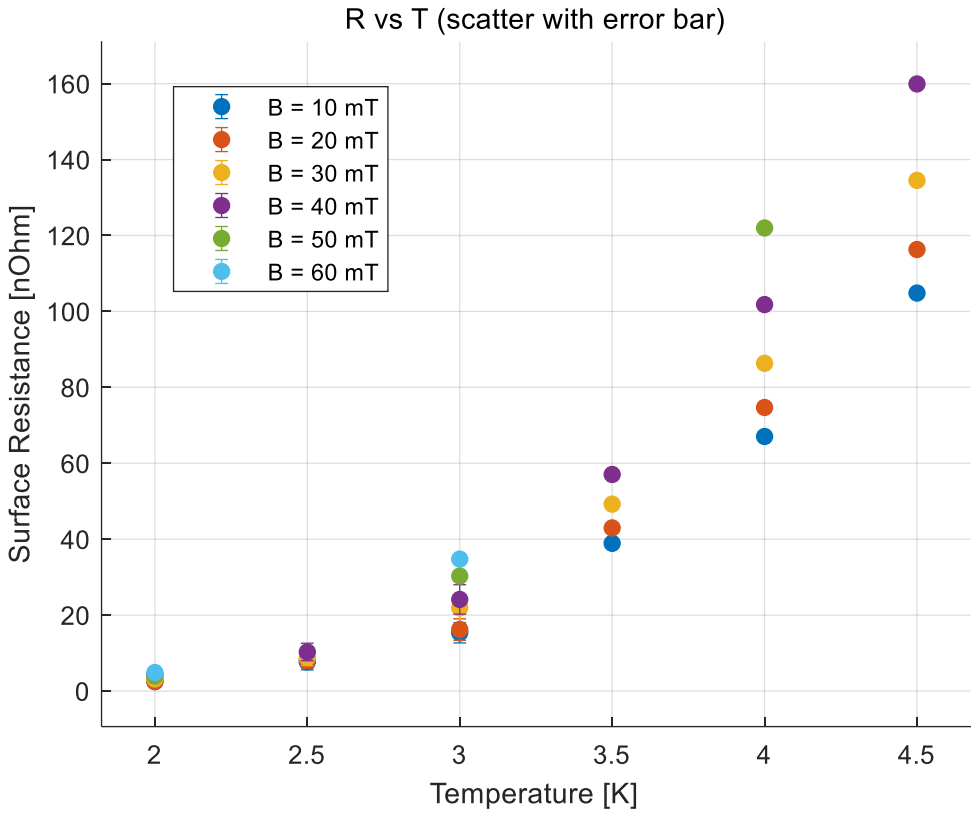
Bulk Nb sample, manufactured at Lanzhou, China
2025-01-14 labeled "F2"
2025-01-14 Weight measured 848.38 g +- 0.03 g
2025-01-14 avg distance flange to sample top 95.113 mm
2025-01-23 BCP 10μm Weight measured 845.55g+-0.05g
2025-02-07 BCP 70μm Weight measured 827.62g+-0.05g
2025-02-13 baking 800°C for 3h, heating rate 3°C /min
2025-02-17 BCP 24μm Weight measured 821.43g+-0.05g

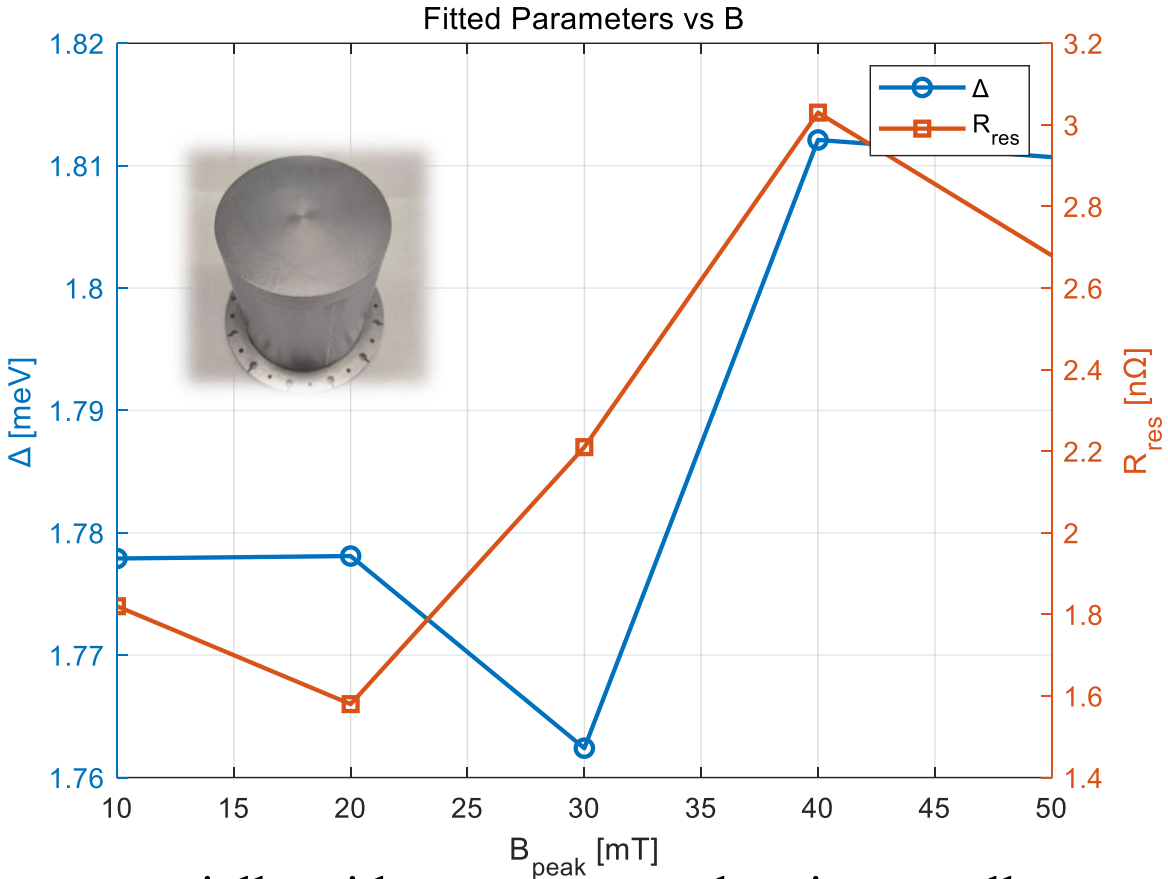
Bulk Nb:RRR~300,fine grain, BCP~100μm+800°C(3h)



Initial Nb | BCP 120μm | Coating bronze | Heat treatment | Remove top

2. bulk Nb QPR sample baseline test

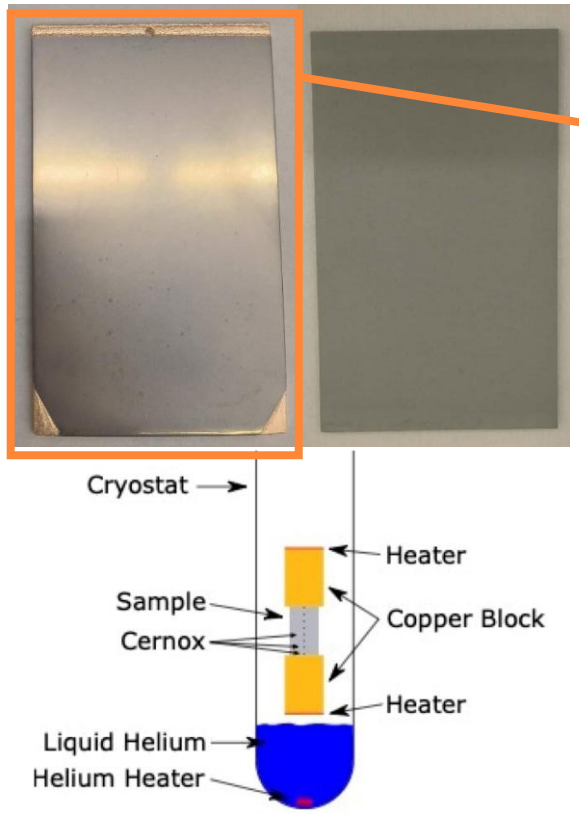


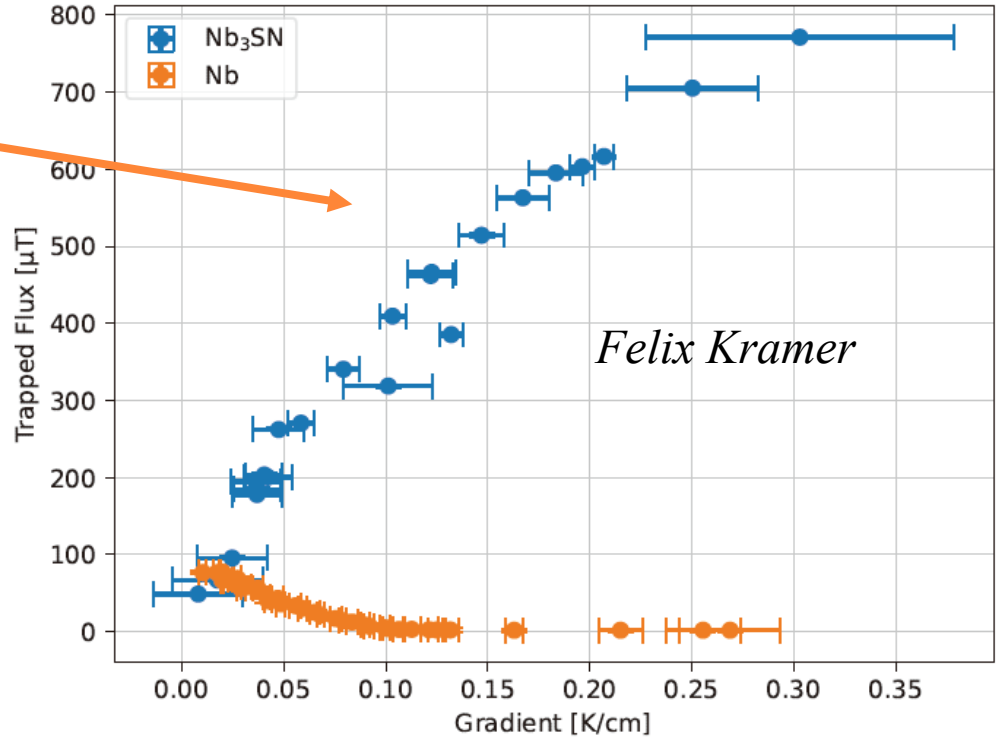


- The measured R_s (2–5 nΩ at 2K) decreases exponentially with temperature, showing excellent agreement with BCS theory.
- The extracted energy gap Δ remains in the range of 1.76–1.81 meV, indicating high-purity niobium with low residual resistance (1.6–3 nΩ), reflecting good surface quality.

2. large Nb_3Sn/Cu sample preparation

Trap flux test of bronze route Nb_3Sn samples using TraMaFlu devices





- We have prepared large-sized Nb_3Sn samples of 100*60*3mm and will conduct flux trap tests.
- The results of trapped flux of Nb_3Sn/Nb/Cu are different from those of bulk niobium, which is very valuable for the study of multilayer films.

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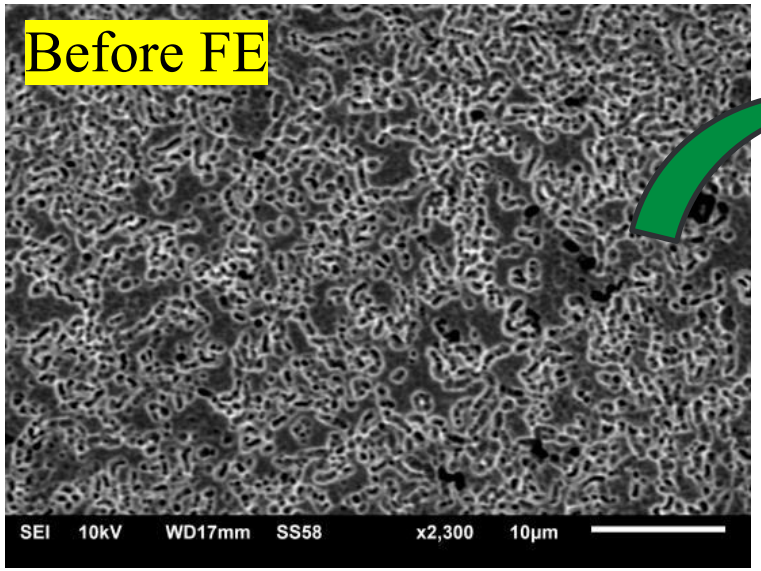
3. **Next step work plan schedule**

3. Optimization of Nb_3Sn/Cu surface morphology

Porous surface for all areas

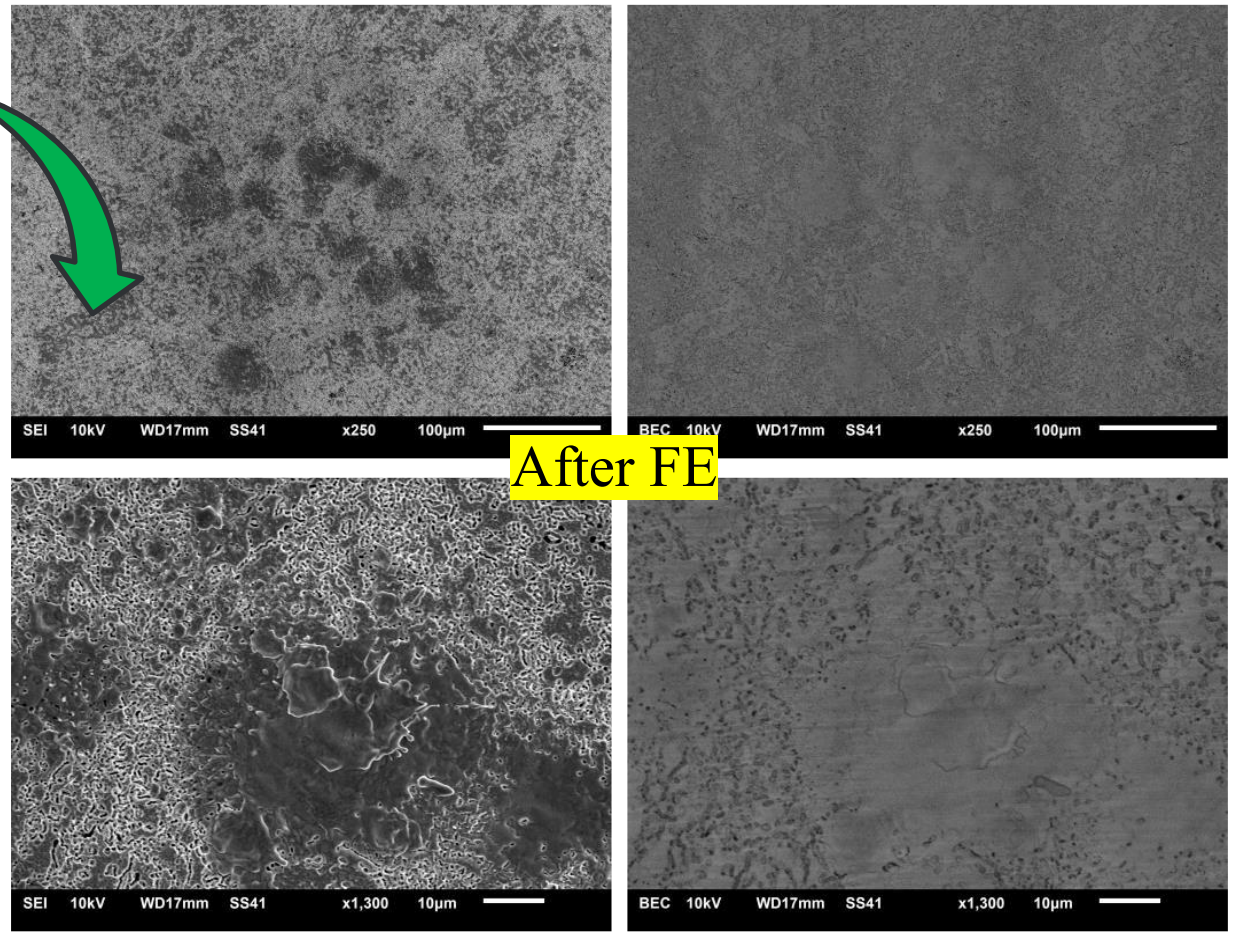


Frederic Braun

Nb_3Sn/Cu FE treatment at Wuppertal:

- ✓ FE strong enough to melt surface
- ✓ BSE Image shows homogenous surface after melting
  - ✓ Becomes Sn rich
  - ✓ Not porous anymore

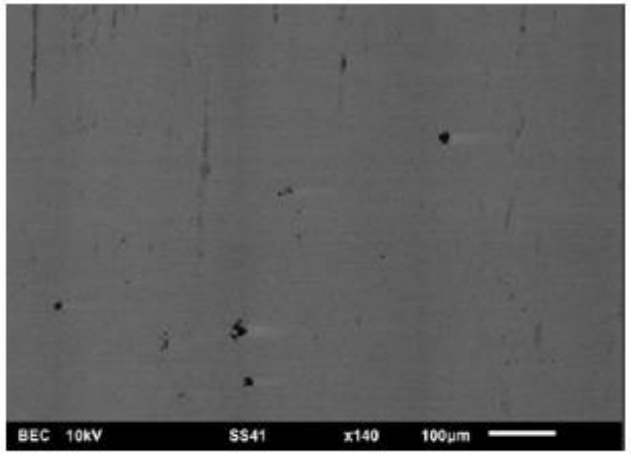
SEM Images after FESM measurements –Nb_3Sn/Cu

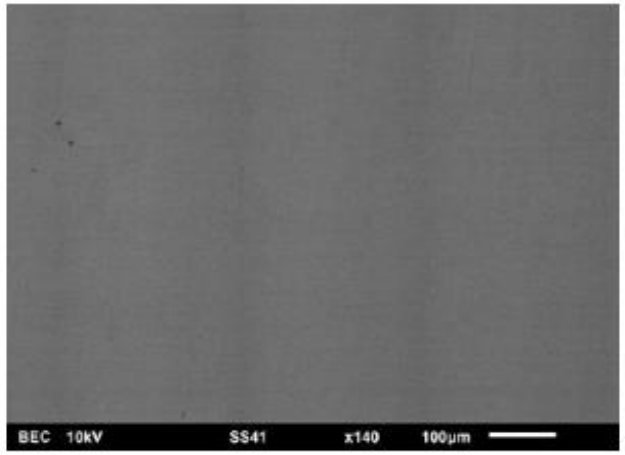


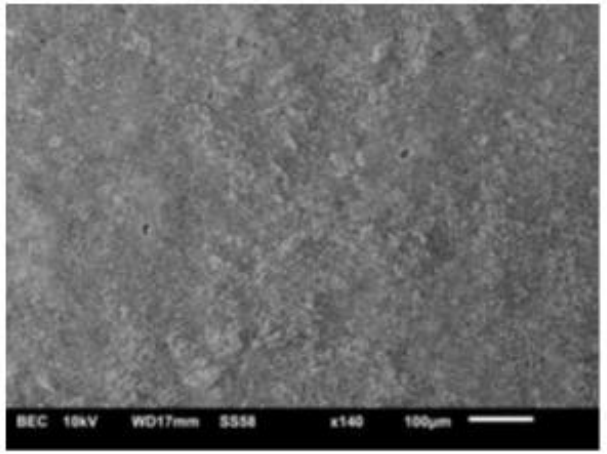
3. Optimization of Nb_3Sn surface impurity recipe

Nb substrate | Nb substrate | Cu substrate

BSE

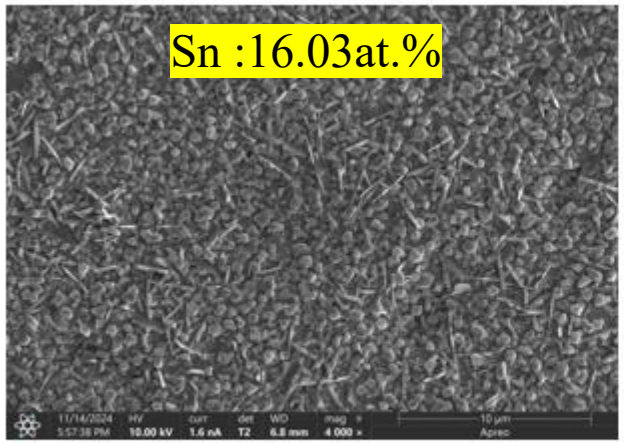


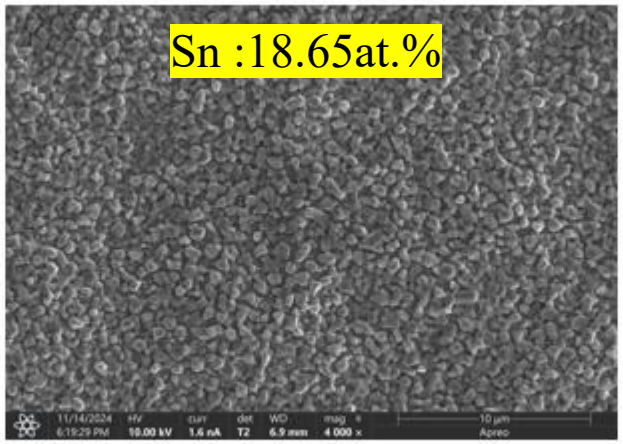


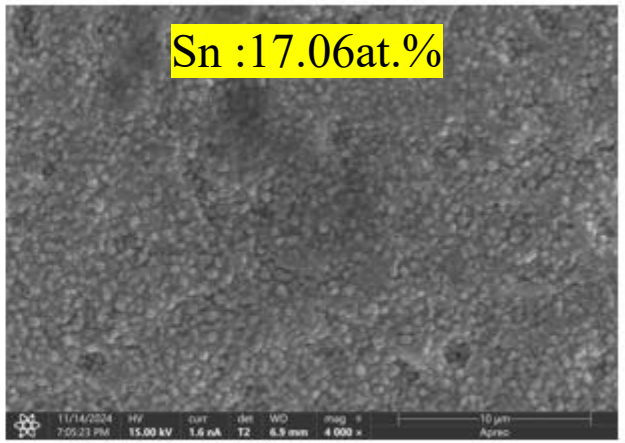


Aqua Regia | Aqua Regia + HF activator | H_3PO_4+HNO_3

SE



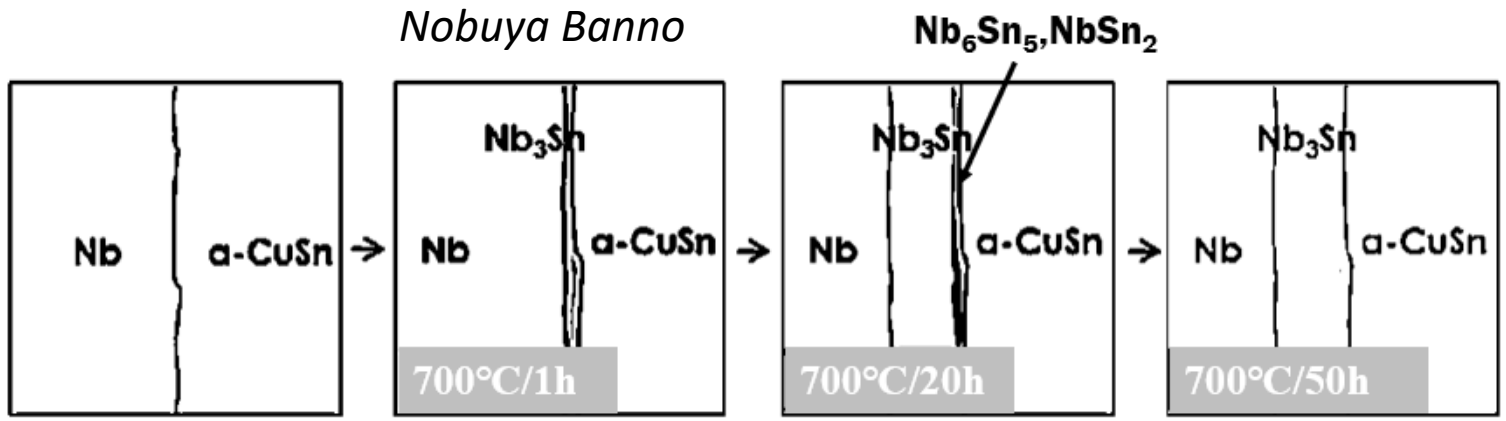


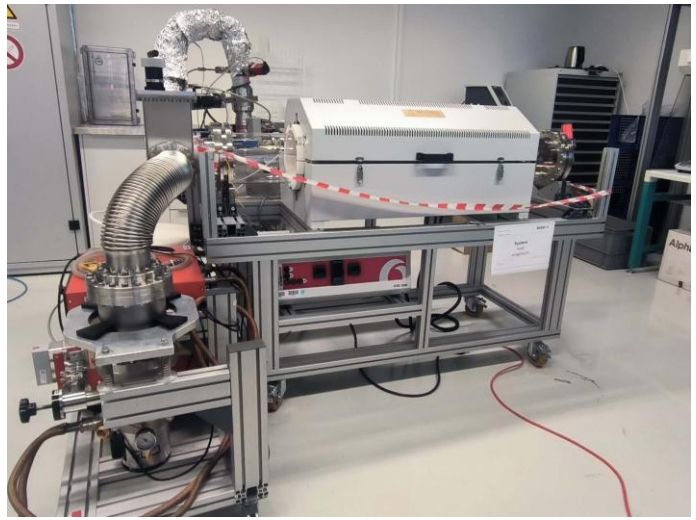


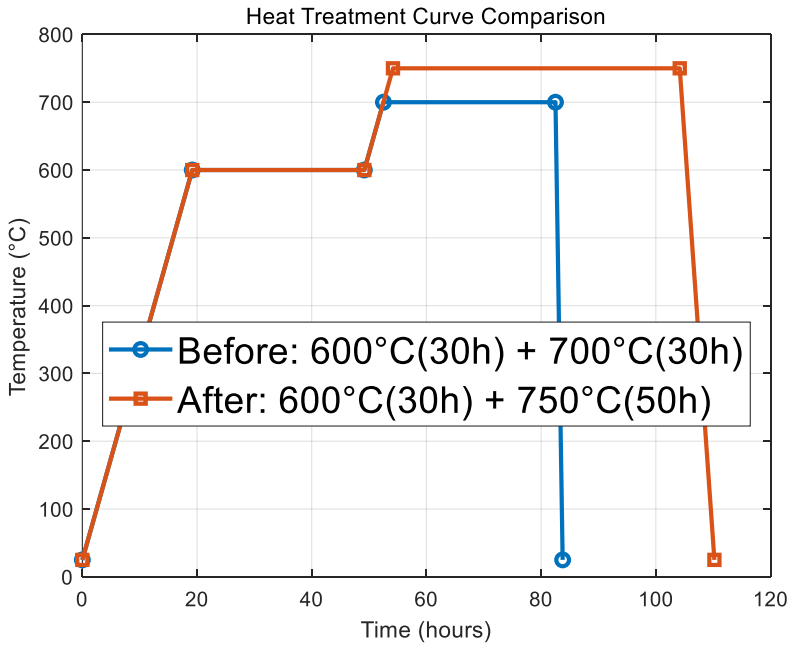
Nb substrate + BCP 10µm | Nb substrate + BCP 120µm | Cu substrate + Nb 10µm

- We studied the effects of different chemical polishing on the surface morphology, tin content and impurity content of Nb_3Sn.
- The results showed that the intermediate treatment process and polishing recipe worked best, and further characterization is ongoing.

3. Optimization of Nb_3Sn heat treatment curve ☺





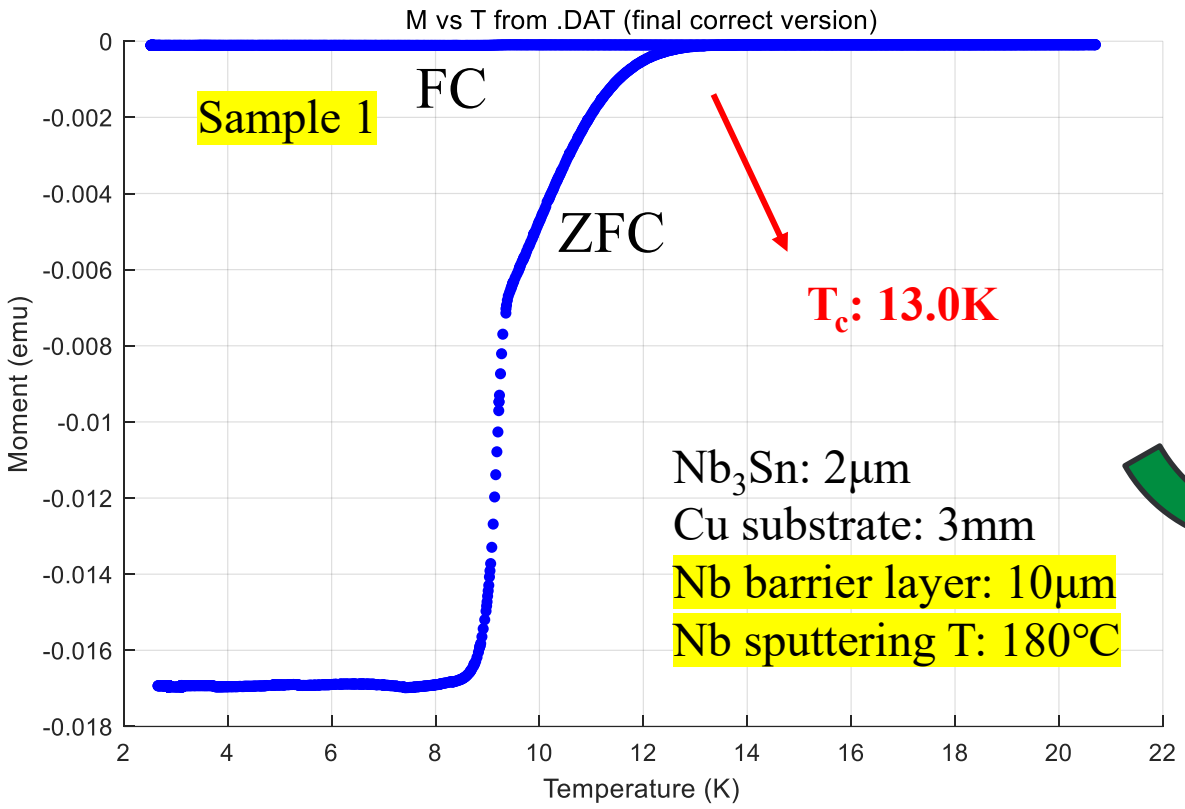


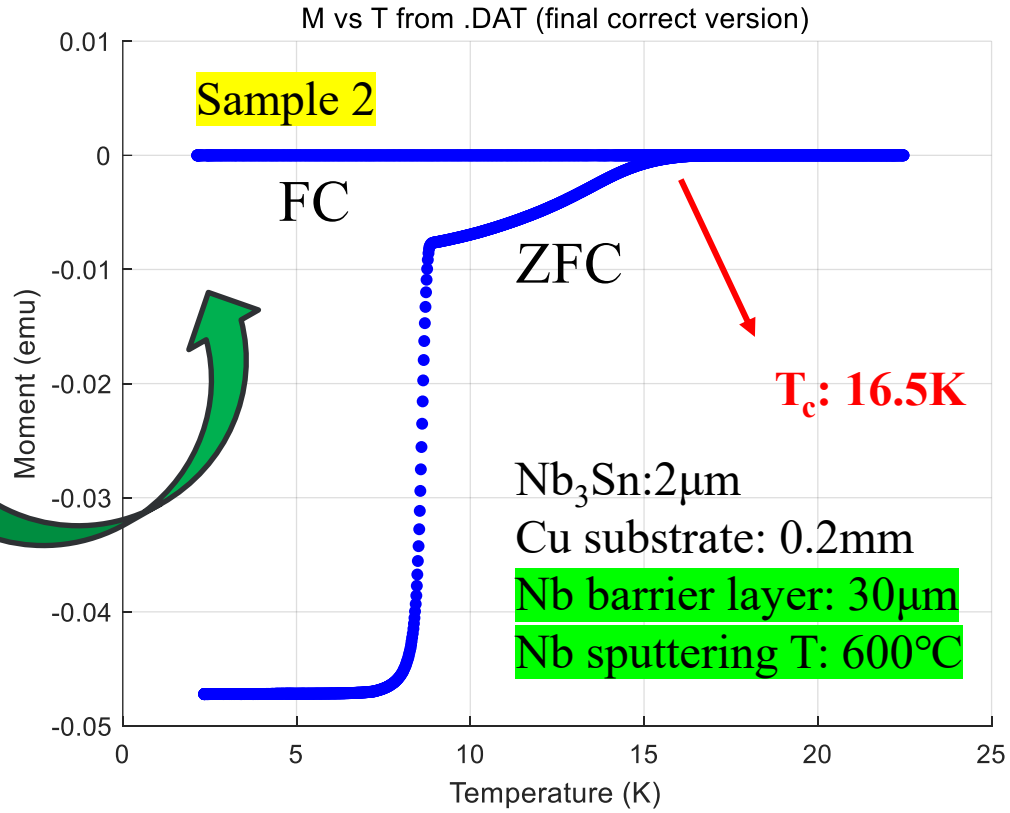
We optimized the heat treatment curve of the bronze route:

Increase Nb_3Sn growth temperature 700->750°C → Increase the grain size of Nb_3Sn, reduce pinning centers

Extend the 750°C high temperature heat treatment time to 50h → Make sure Nb_6Sn_5 and $NbSn_2$ impurity phases are transformed into Nb_3Sn

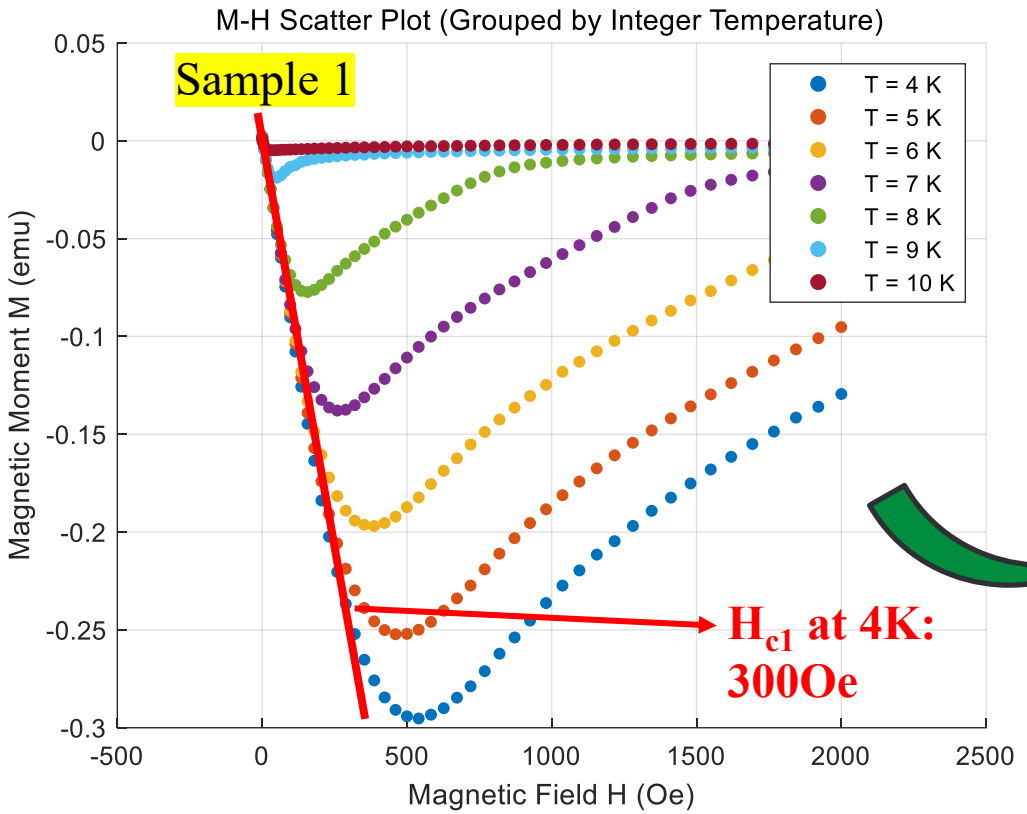
3. Nb_3Sn/Cu small sample T_c optimization☺



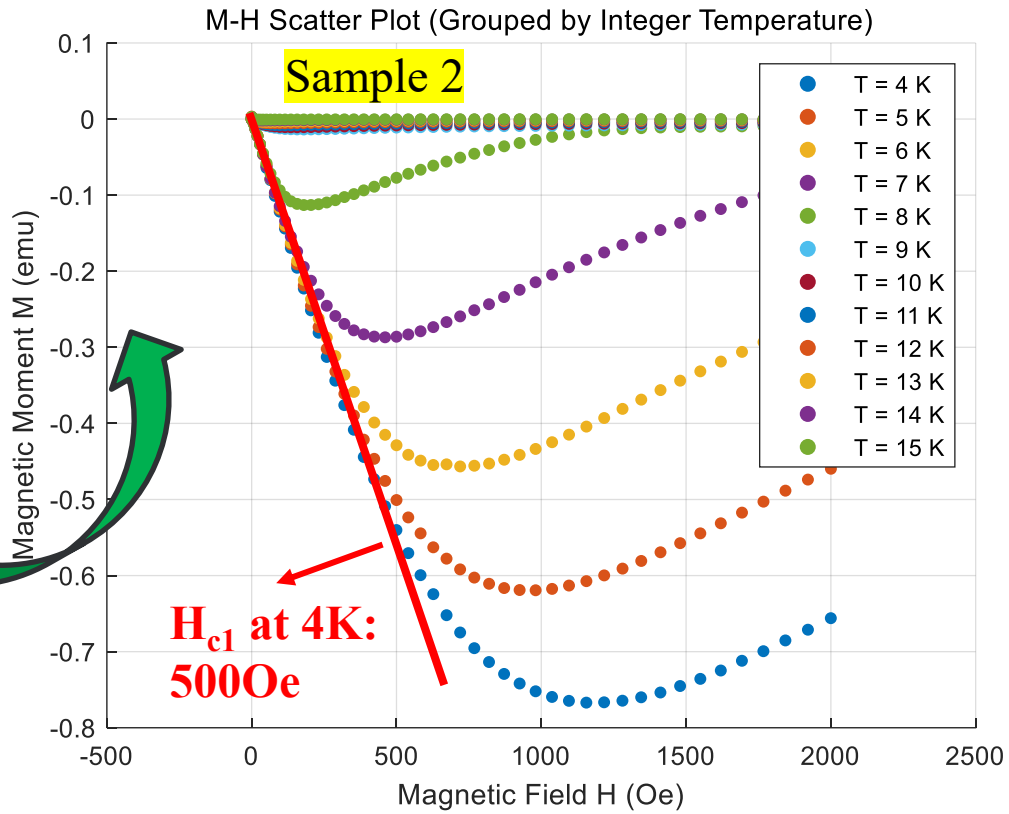


- Through PPMS testing, it was found that increasing the Nb sputtering temperature and increasing the thickness of the Nb barrier layer can effectively improve the quality of Nb_3Sn coating, and T_c was increased from 13.0K to 16.5K.

3. Nb_3Sn/Cu small sample H_{c1} optimization☺



Nb barrier layer: 10μm
Nb sputtering T: 180°C

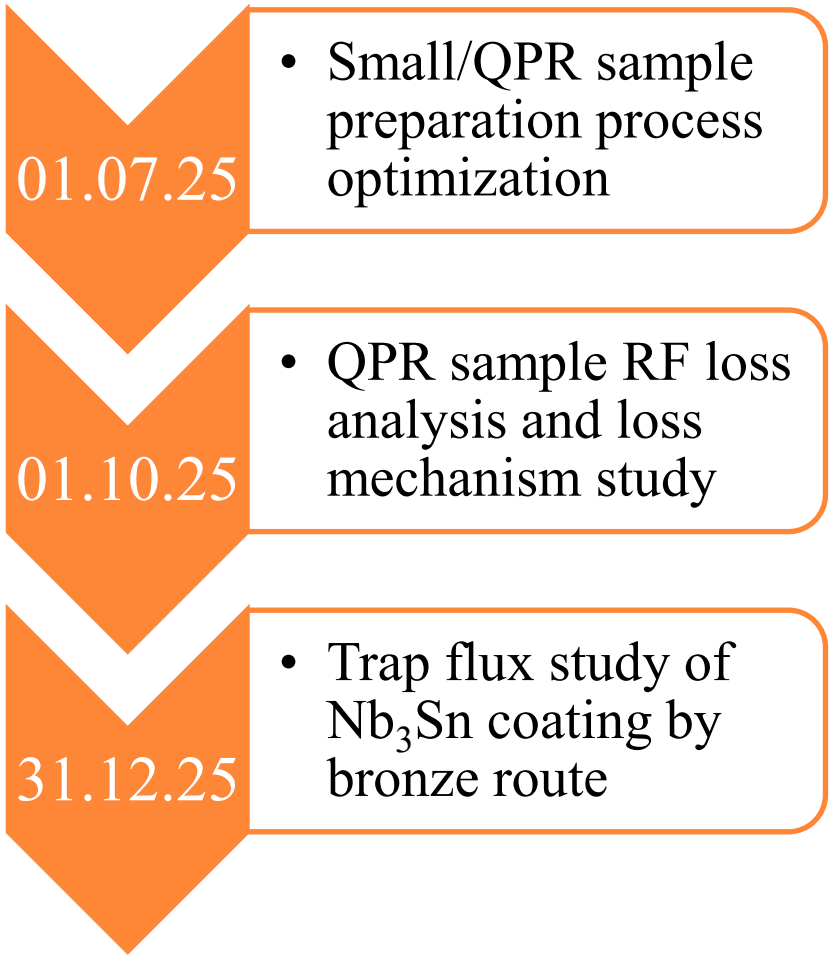


Nb barrier layer: 30μm
Nb sputtering T: 600°C

- In addition, M-H tests were conducted and it was found that the lower critical magnetic field H_{c1} also increased from 300Oe to 500Oe.

Next step work plan schedule

- **Aim: high quality Nb_3Sn by bronze route**
  - Nb_3Sn/Cu surface impurities optimization. (Cu, Oxides, Carbides).
  - Nb_3Sn coating growth mechanism study and grain control.
  - Nb_3Sn/Cu RF loss analysis and loss mechanism study.
- **Next work: next six months**
  - Nb_3Sn/Cu QPR sample will be optimized to increase T_c.
  - Trap flux study of Nb_3Sn coating on Nb substrate (TraMaFlu).
- **Future work: next two years**
  - Small samples -> larger samples -> QPR samples -> 1.3GHz cavities.
  - 1.3GHz copper cavity Nb sputtering + bronze route Nb_3Sn coating.



Thanks for your attention.