Electrochemical and thermal synthesis of Nb_3Sn coatings on Nb substrates

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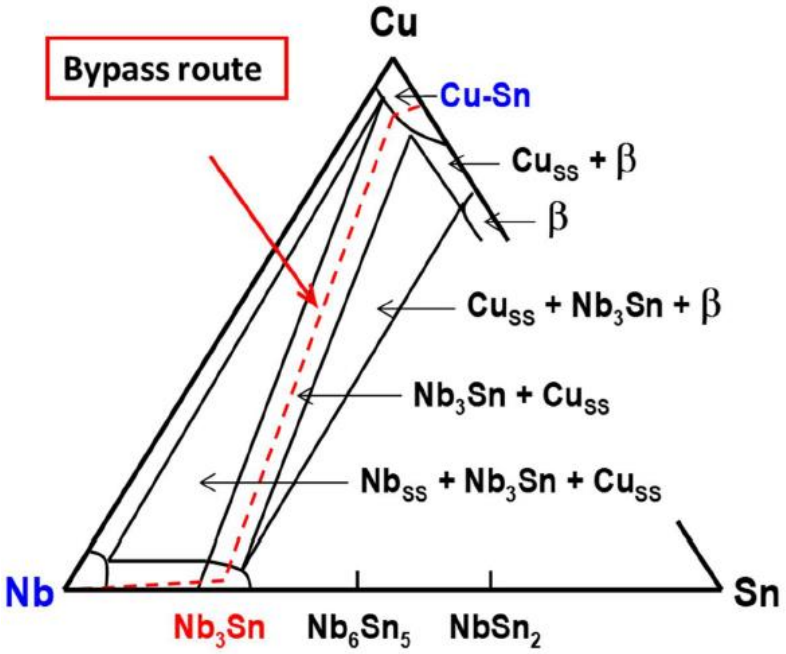
2021-03-16

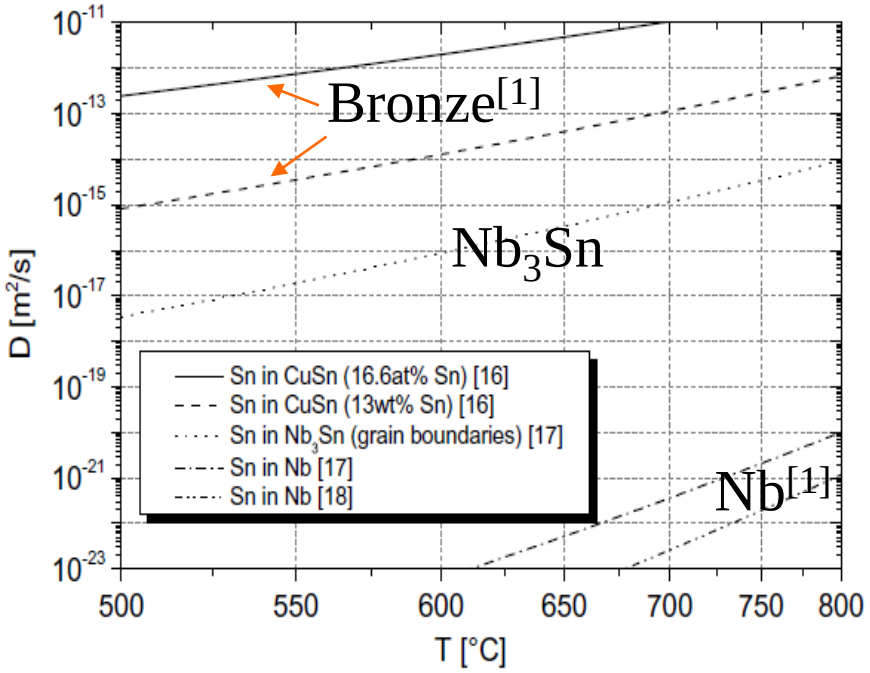
Outlines

1. Background
2. Nb_3Sn thin film activities
   - Route 1: Nb/Cu/Sn/Cu multilayer films
   - Route 2: Nb/Bronze layer films
   - Route 3: Cu/Nb/Bronze multilayer films
3. Conclusion

Background: Nb_3Sn bronze process

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



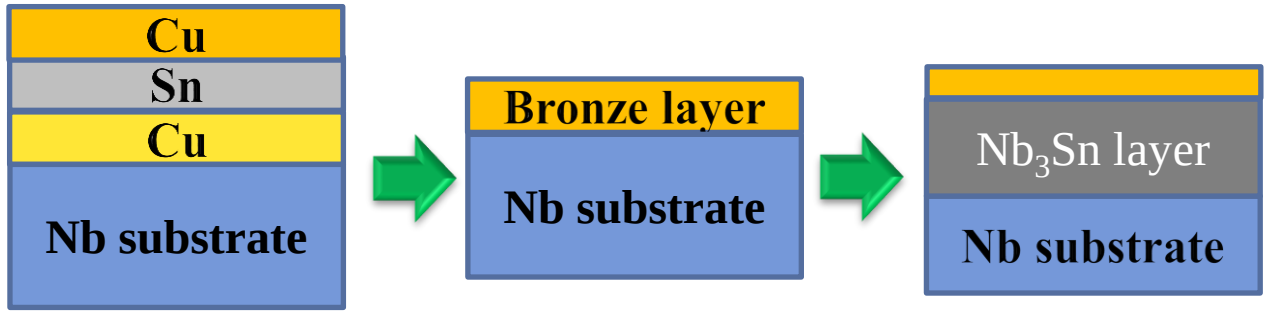


1. The copper can facilitate the interdiffusion between Nb and Sn by 7~10 magnitudes of orders.
2. The copper alloy will be excluded from the Nb_3Sn phase by itself.

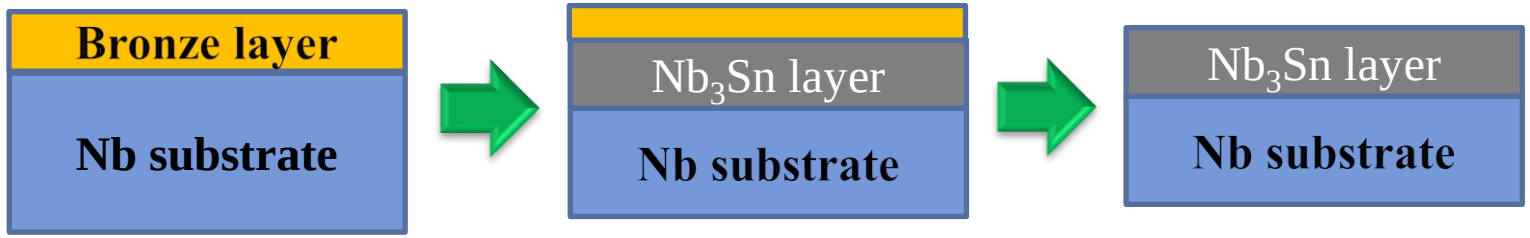
[1] H. Müller and T. Schneider, "Heat treatment of Nb3Sn conductors," Cryogenics, vol. 48, pp. 323-330, 2008/07/01/ 2008.
[2] L Mei, Z Du, C Guo, & C Li. (2009). Thermodynamic optimization of the cu-sn and cu-nb-sn systems. Journal of Alloys & Compounds, 477(1-2), 104-117.

Background: Nb_3Sn bronze process

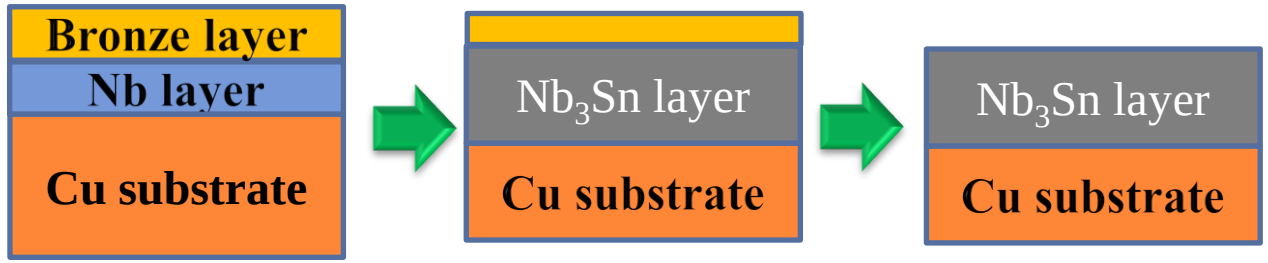
Route 1[1]:



Route 2[2]:



Route 3:



[1] E. Barzi, M. Bestetti, F. Reginato, D. Turrioni, S. Franz, Synthesis of superconducting Nb3Sn coatings on Nb substrates, Superconductor Science and Technology 29 (2016) 015009.
[2] M. Lu, F. Pan, H. Guo, S. Huang, Z. Yang, Q. Chu, F. Liu, T. Tan, Electrochemical and thermal synthesis of Nb3Sn coatings on Nb substrates, Materials Letters 292 (2021) 129557.

Pre-treatment procedures before electroplating

Roughness reduction

- Sand paper grinding and polishing

Degreasing

- 1%~2% micro-90®
- Ultrasonic cleaning
- 20 minutes

Removal of oxide (EP)

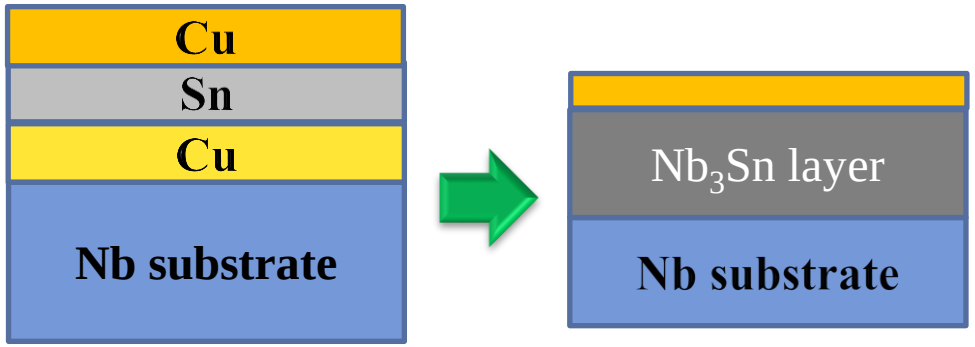
sulphuric acid : hydrofluoric acid about 9:1
10 minutes

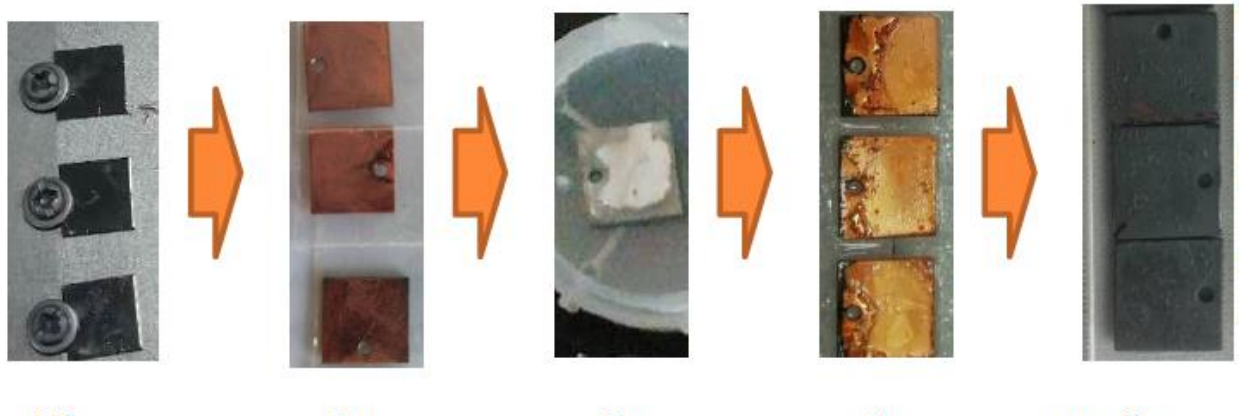
Surface activation

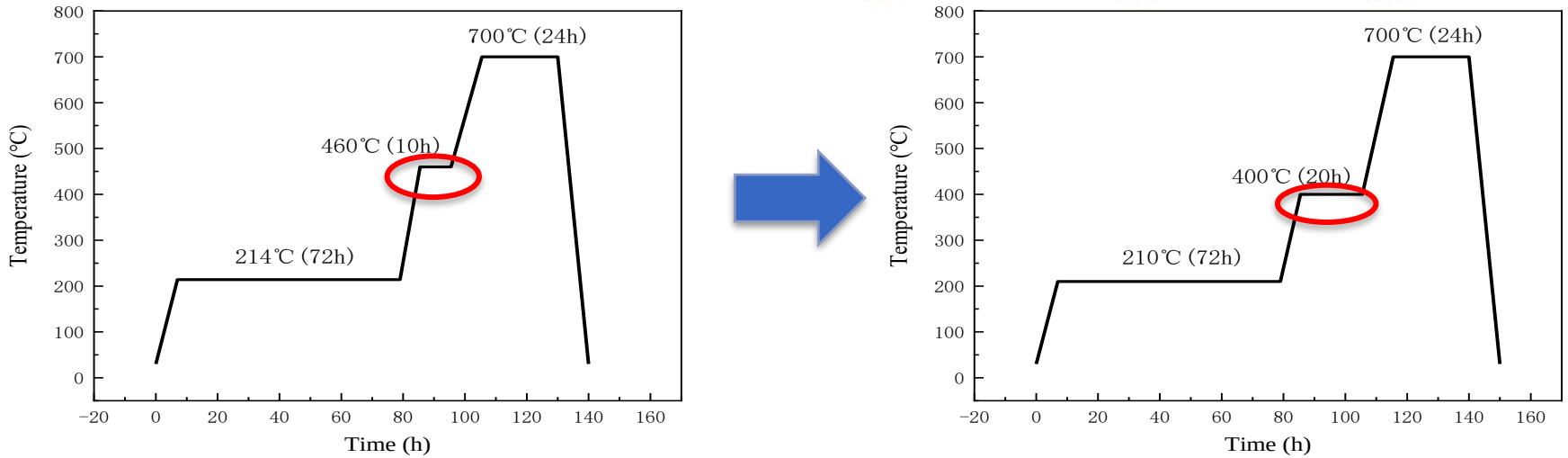
- 10% dilute sulfuric acid
- Wash with deionized water

Before EP After EP

Route 1: Nb/Cu/Sn/Cu multilayer films







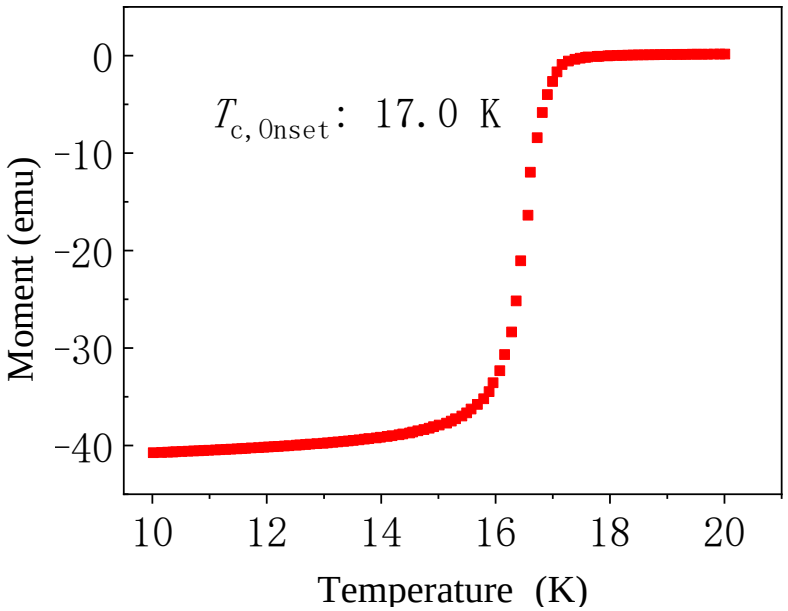
210℃(72h):Relieve the stress, give off water vapor and form copper-tin alloy

400℃(20h):Reduce the number of Kirkendall hole and increase grain size by extending medium treatment time

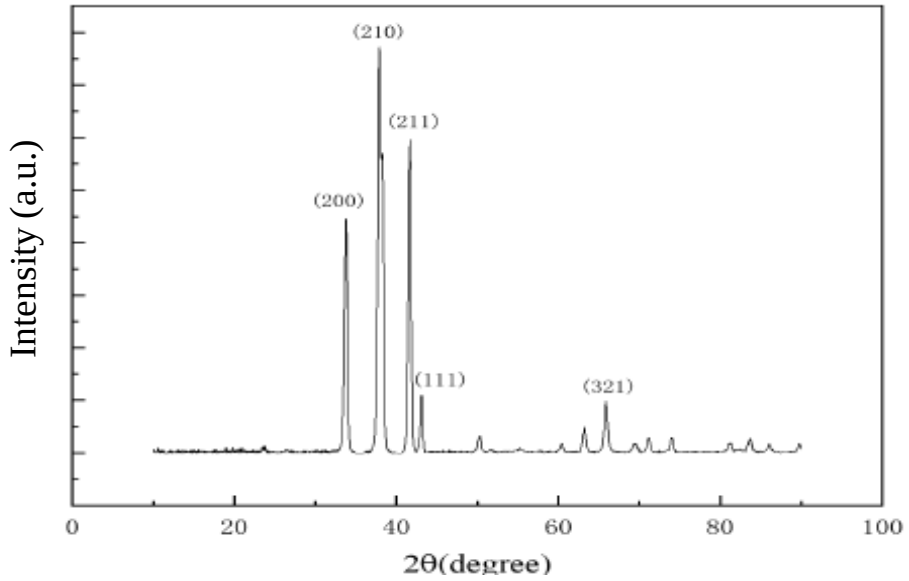
700℃(24h):Form the Nb_3Sn superconducting phase

Route 1: Nb/Cu/Sn/Cu multilayer films

Superconducting properties:



M-T measurements showed Tc 17.0K

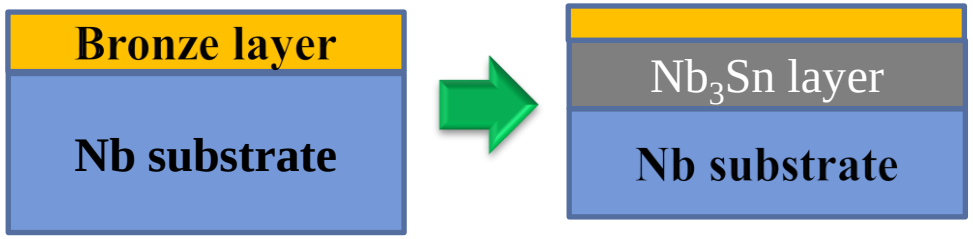


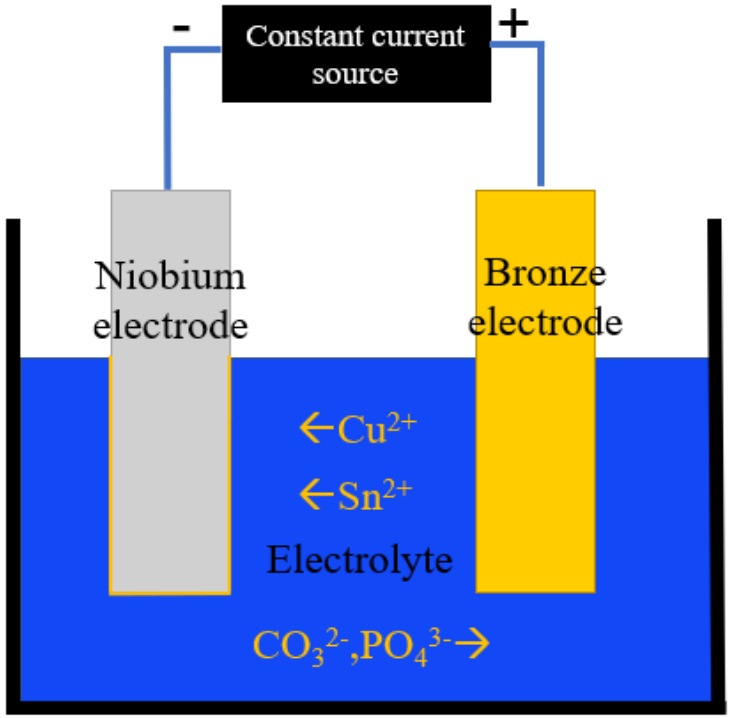
XRD patterns showed the formation of Nb_3Sn phase

Problems:

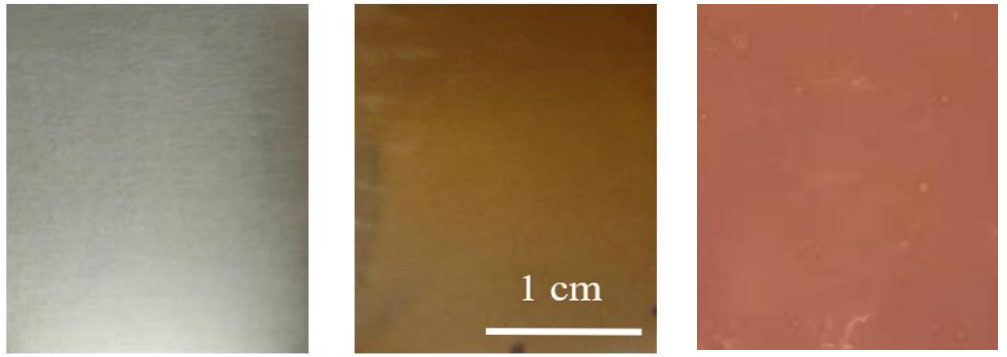
1. Films are easily broken, peel off or cracked.
2. Bubbles appear in the film after heat treatment.
3. There are copper, tin and other impurity phases in Nb_3Sn films.

Route 2: Nb/Bronze layer films

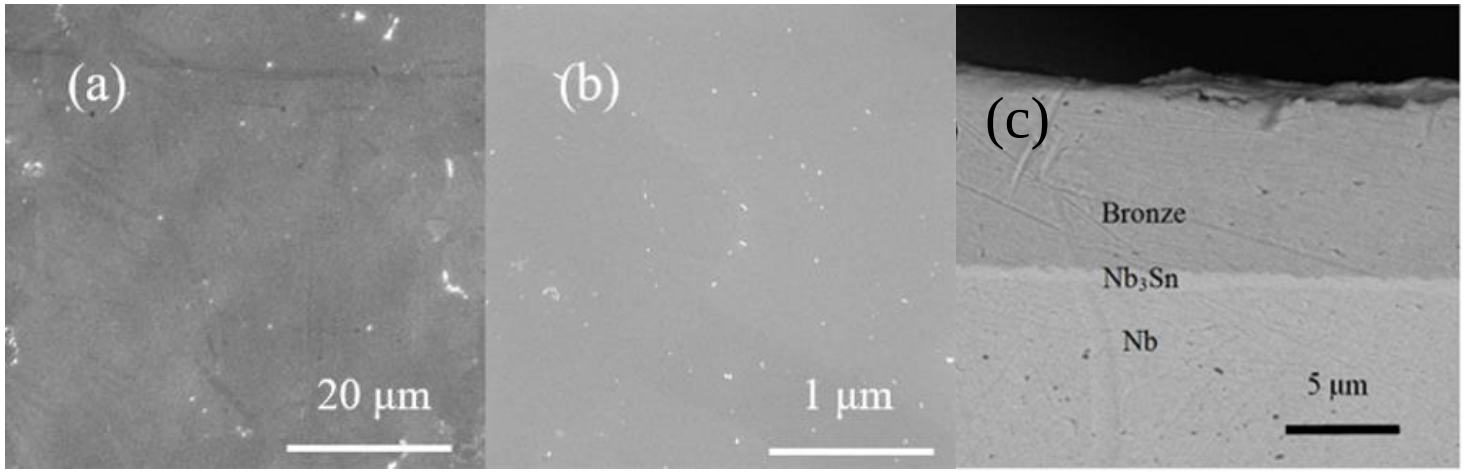




Schematic diagram of Cu-Sn co-deposition

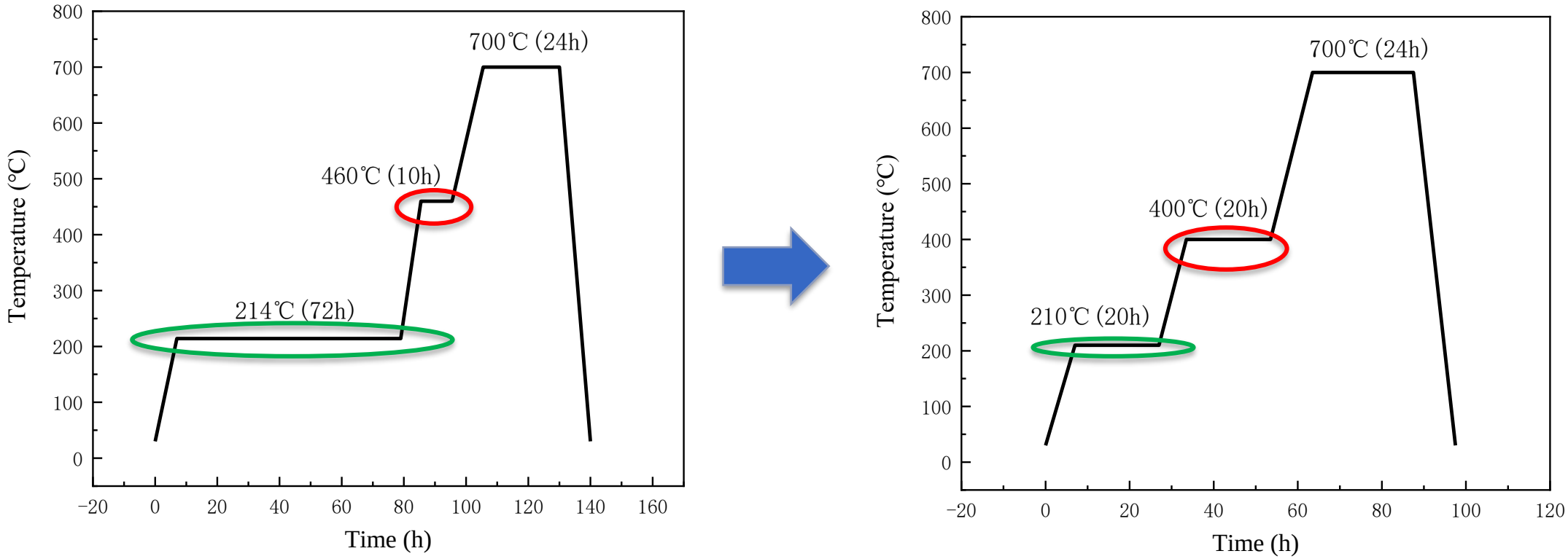


Nb Nb/Bronze Nb/Nb_3Sn/Cu



SEM picture of sample surface (a, b) before electropolishing under different magnification.

Route 2: Optimization of heat treatment curve

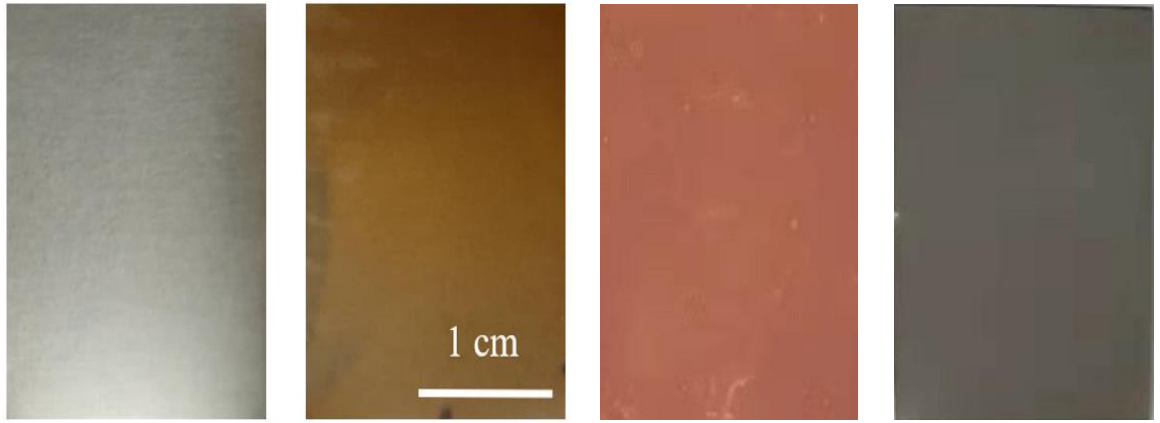


210℃(20h):The treatment time of low temperature bronzing is greatly reduced

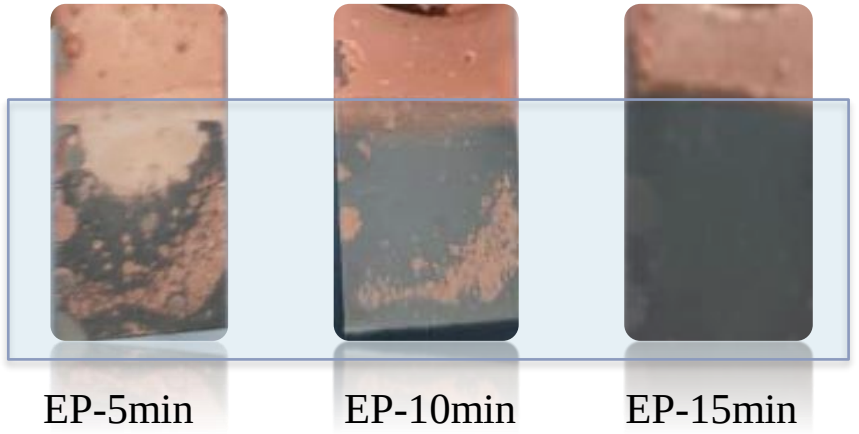
400℃(20h): Reducing the number of Kirkendall hole and increase grain size by extending medium treatment time

700℃(24h):The formation of Nb_3Sn superconducting phase

Route 2: Removing bronze layer



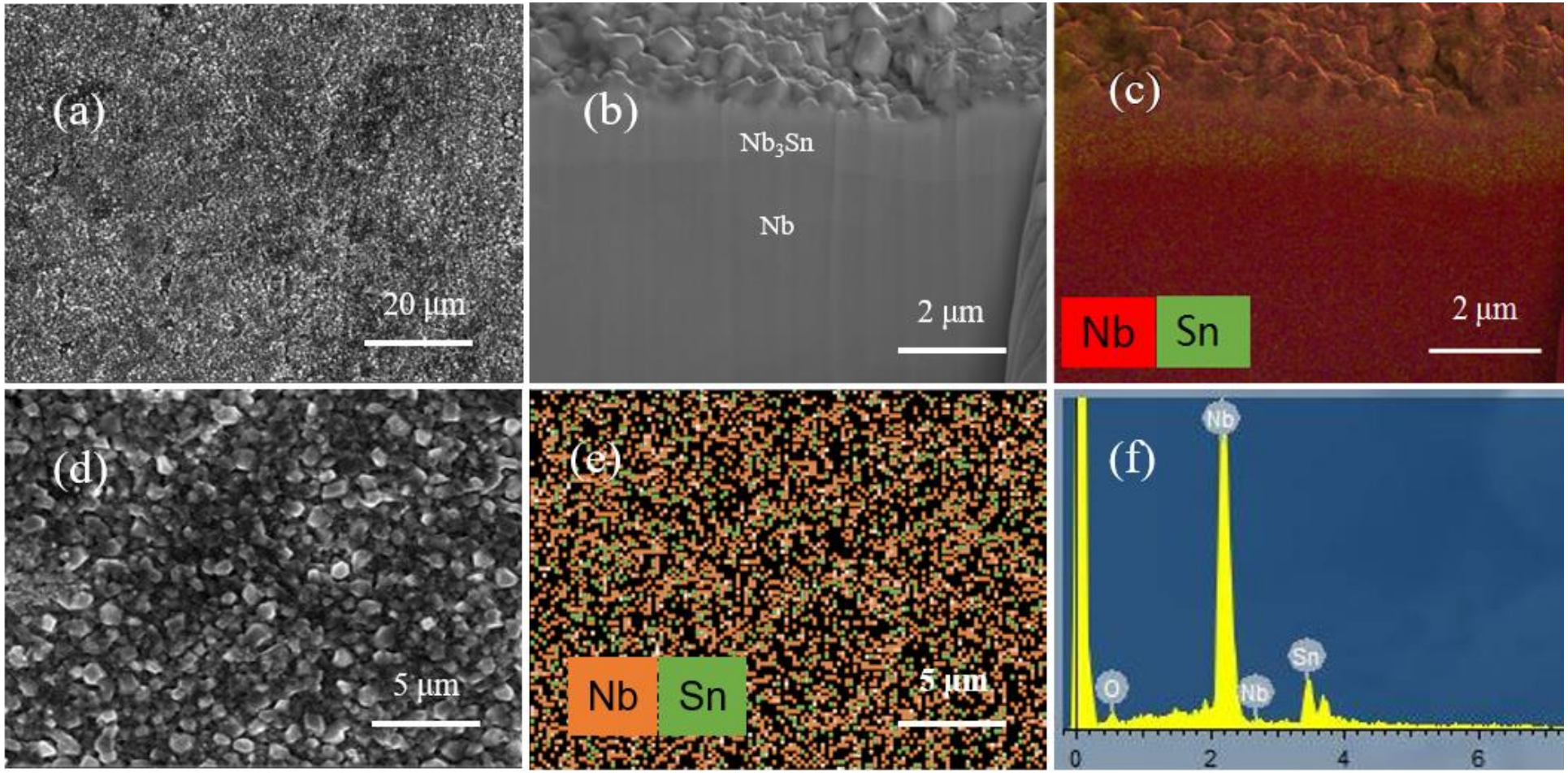
Nb Nb/Bronze $Nb/Nb_3Sn/Cu$ Nb/Nb_3Sn



Electropolishing experiment

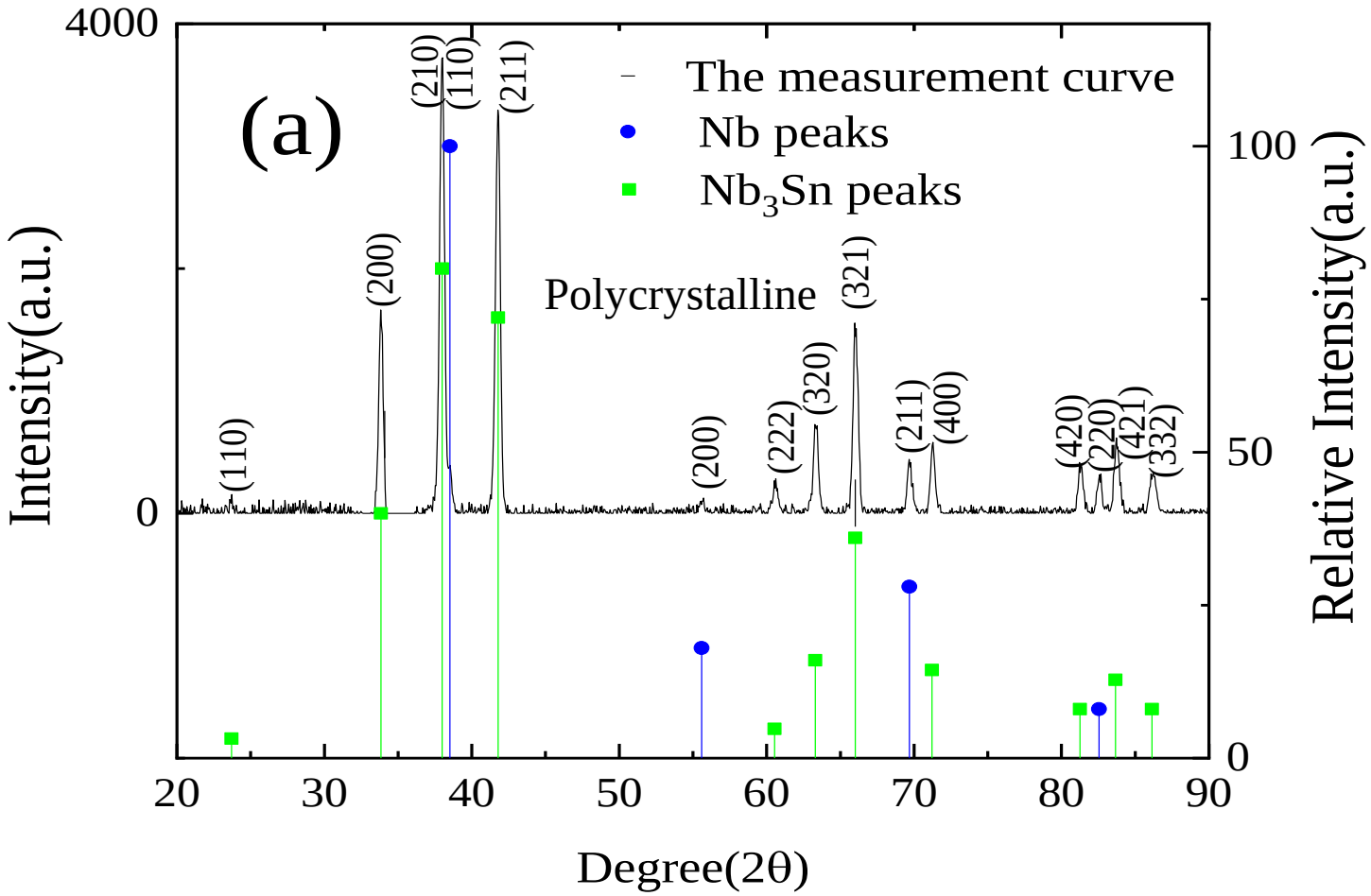
1. Phosphoric acid and ethylene glycol mixed in a volume ratio of 3:2
2. The electropolishing of surface bronze layer was performed at a current density of 50 mA/cm^2 and bath temperature of 25˚C. Polishing time is about 15 minutes

Route 2: Nb/Nb_3Sn layer films



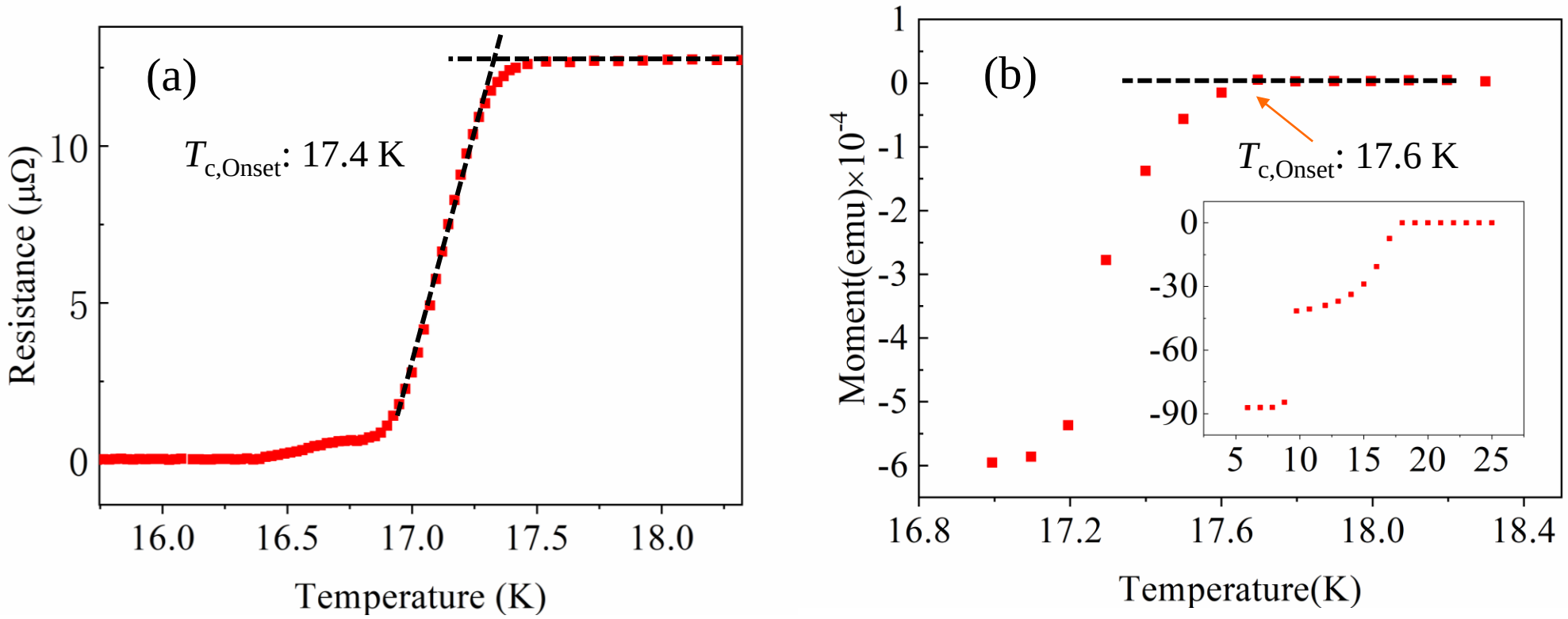
Morphology and Composition: Sample (a) surface and (b) cross-section SEM pictures after electropolishing, (c) the elemental mapping. The top-view SEM picture (d) morphology, (e) the elemental mapping, and (f) the EDX analysis of surface after electropolishing.

Route 2: Nb/Nb_3Sn layer films



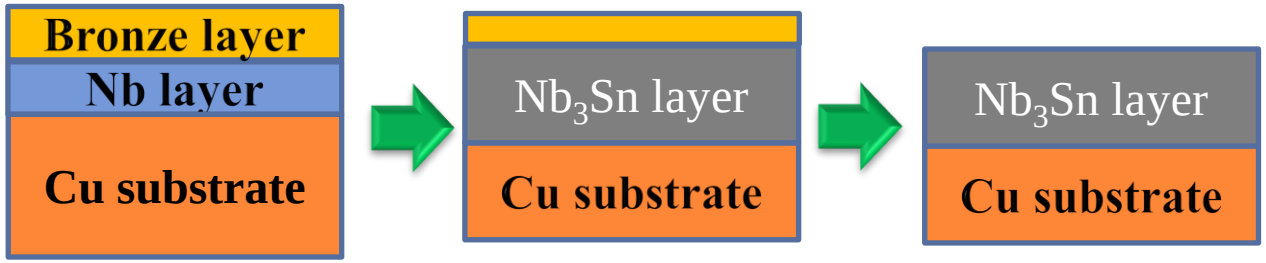
The XRD pattern of sample consisting of Nb/Nb_3Sn surface.

Route 2: Nb/Nb_3Sn layer films



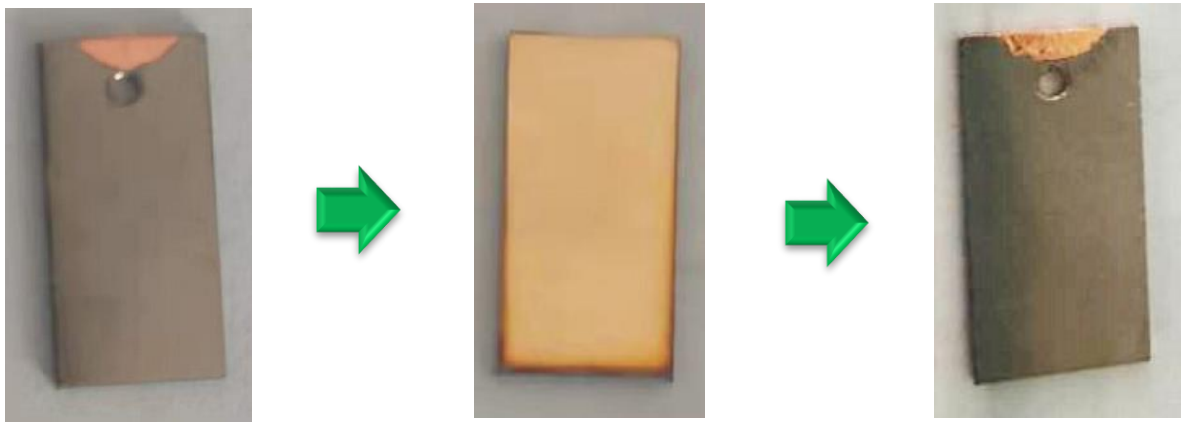
DC electrical measurement (a) of critical temperature T_c, and DC magnetic measurement (b) of critical temperature T_c

Route 3: Cu/Nb/Bronze multilayer films



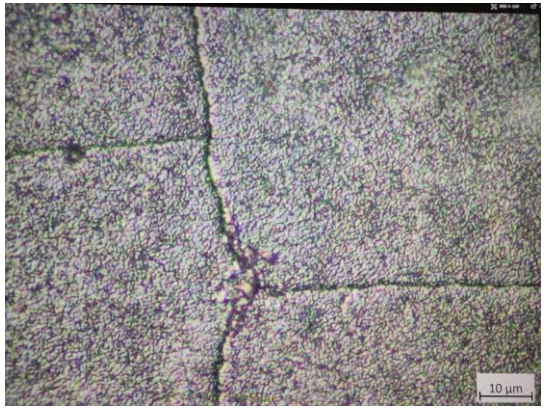
Removal of bronze layer by electropolishing after annealing

Nb: magnetron sputtering



Cu/Nb Cu/Nb_3Sn/Bronze Cu/Nb_3Sn

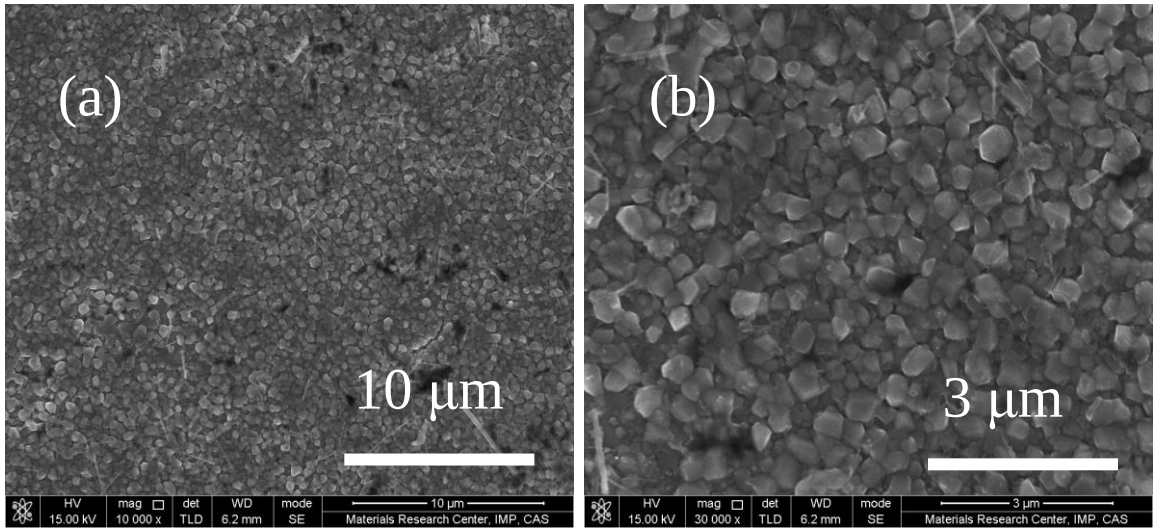
Early attempts: cracks



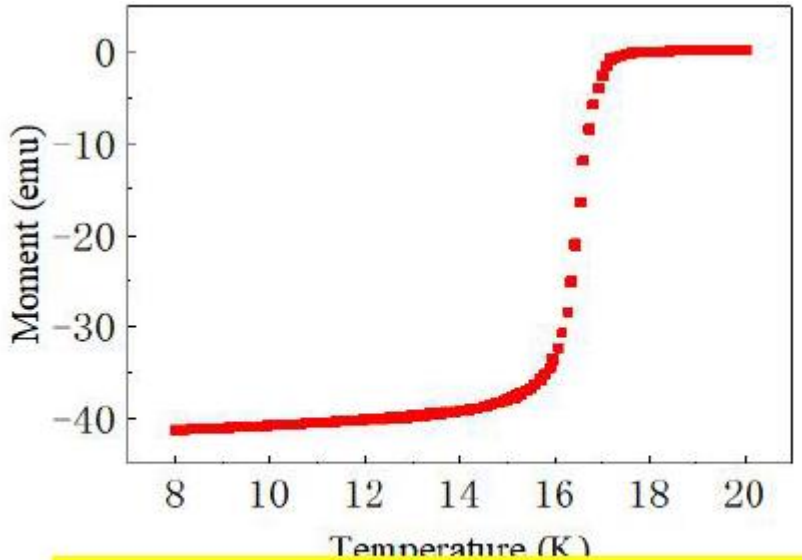
Early attempts: cracks

Route 3: Cu/Nb/Bronze multilayer films

Sample properties:



SEM picture of sample surface (a, b) before electropolishing under different magnification.



M-T measurement show $T_{c,\ Onset} \sim 17K$

Future plan

Conclusion

A combined electrochemical-thermal synthesis (ETS) technique of Nb_3Sn film on Nb substrates was presented.

- The medium temperature treatment technology has been improved.
- Direct bronze alloy precursor can avoid a long process of copper and tin alloying, requiring only a short period of low temperature pre-treatment to remove moisture, residual gas and stress. High quality Nb_3Sn film on Nb substrate can be obtained. The highest Tc observed on these samples was 17.6 K.
- After heat treatment, the residual copper and bronze layer on top of the sample could be completely removed by electropolishing.
- Its low temperature annealing allows the fabrication of Nb_3Sn thin film on copper cavity.

Thanks for your attention.