Research of Niobium Cavity Coating with Nb_3Sn Film at IHEP

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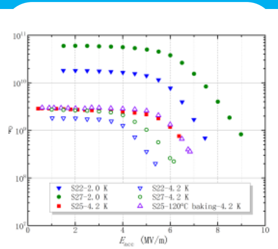
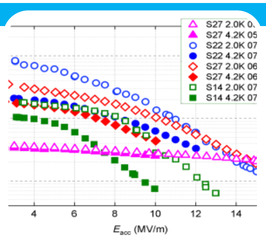
outline

- Introduction
- Setup and coating process
- Performance of Nb_3Sn grown on Nb cavities
- Characterizations of Nb_3Sn coated samples
- Summary and perspective

Introduction of our team





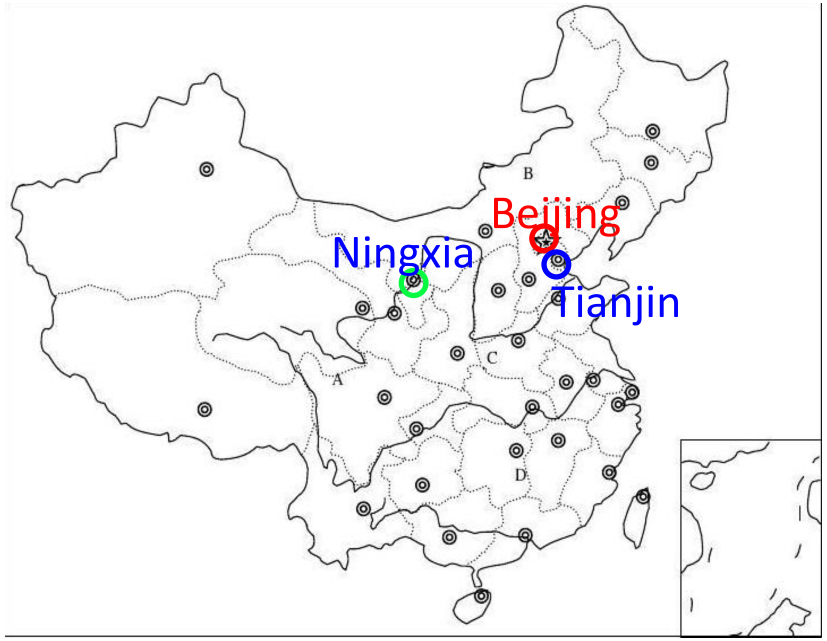
2020
- The furnace starts to operate normally.

2021
- 2.8E9 @ 4.0MV/m for 1.3 GHz 1-cell

2024
- 1.1E10 @ 10.5MV/m for 1.3 GHz 1-cell

Items	Person in charge + key participants
Leader	Weimin PAN + Peng SHA
Pre-process before Coating	Song JIN + Feisi HE
Anodization	Chao DONG + Guangwei WANG
Coating Process	Peng SHA + Baiqi LIU
Sample Characterization	Chao DONG + Jinxin YU
Vertical Test	Zhenghui MI + Lingxi YE
Conduction Cooling	Rui GE + Peng SHA

In addition to the personnel (11) mentioned above, there are more than 5 persons from companies (OTIC + HERT) involved.



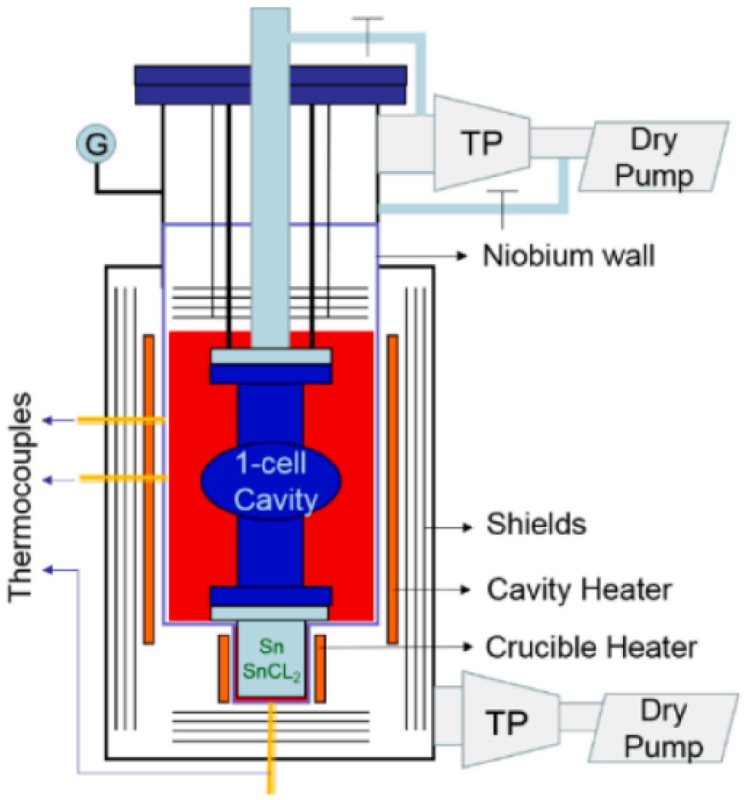
- Pre-process before coating

EP(Ningxia, ~1100km from Beijng)

BCP (Tianjin, ~150km from Beijng)

HPR + Assembly (Beijng PAPS)

The Coating Furnace for Nb_3Sn



A schematic of the furnace

Main Technical Specifications:

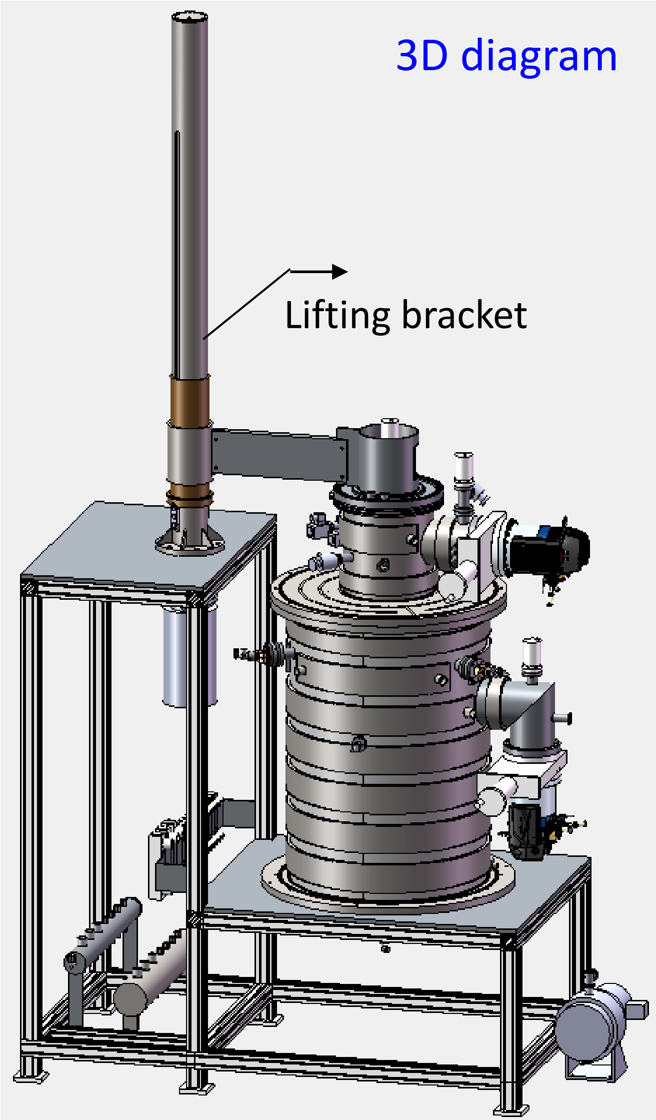
1. Type: vertical double vacuum furnace.

2.Effective Uniform Temperature Zone
The diameter is not less than 300mm, with a height of 500mm.

The inner wall material of the high-temperature zone furnace is niobium.

3. Vacuum System
Ultimate vacuum: ≤ 5.0×10^{-5} Pa (empty furnace, room temperature, fully degassed)
Working vacuum: ≤ 9.0×10^{-4} Pa (1300°C, empty furnace, fully degassed)
Vacuum pumps: oil-free molecular pump unit.

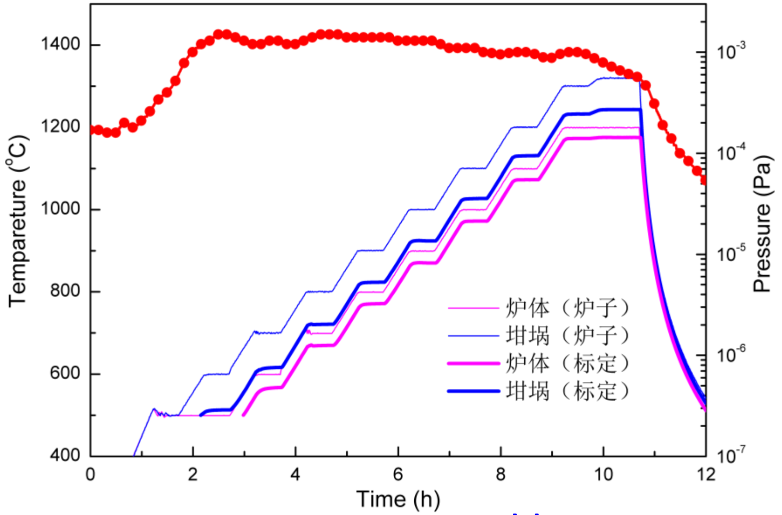
4. Temperature Control System
Maximum furnace temperature: 1200°C
Maximum crucible temperature: 1300°C
Temperature uniformity: Better than ±4°C



Cleanroom and Furnace Temperature Calibration

Cleanliness level:
Class 10,00

Time (min)	Furnace (℃)			Crucible(℃)		
	measured	calibrated	△T	measured	calibrated	△T
160	510	--	--	600	513.0	87
221	609	567.3	41.7	701	616.3	84.7
340	807	771.2	35.8	901	823.4	77.6
400	905	870.3	34.7	1001	924.1	76.9
462	1005	972.1	32.9	1100	1027.3	72.7
521	1104	1072.8	31.2	1201	1131.3	69.7
580	1202	1173.0	29	1300	1232.5	67.5
640	1203	1175.4	27.6	1320	1243.3	76.7



Furnace temperature calibration curve

1. The actual furnace temperatures are approximately 30℃ lower than the measured value;

2. The actual crucible temperature is 65-80℃ lower than the measured value.

Coating Process

Degas: 150℃ 17h

↓

Nucleation temperature & time
Crucible/Furnace: 850℃/540℃ 5h

↓

Ramp-up: ~1h

↓

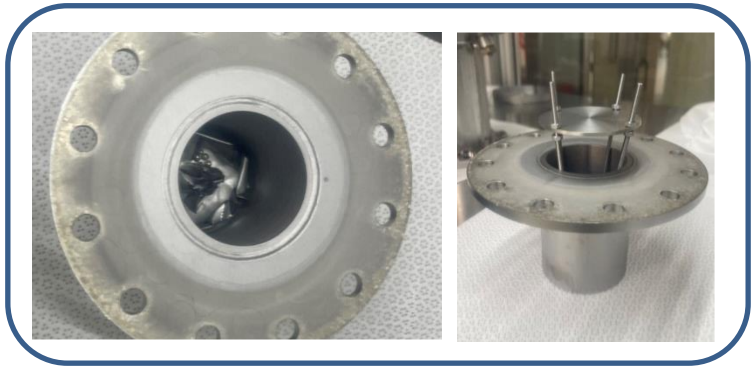
Coating temperature & time
Crucible/Furnace: 1290℃/1130℃ 1-3h

↓

Annealing temperature & time
Crucible/Furnace: None

↓

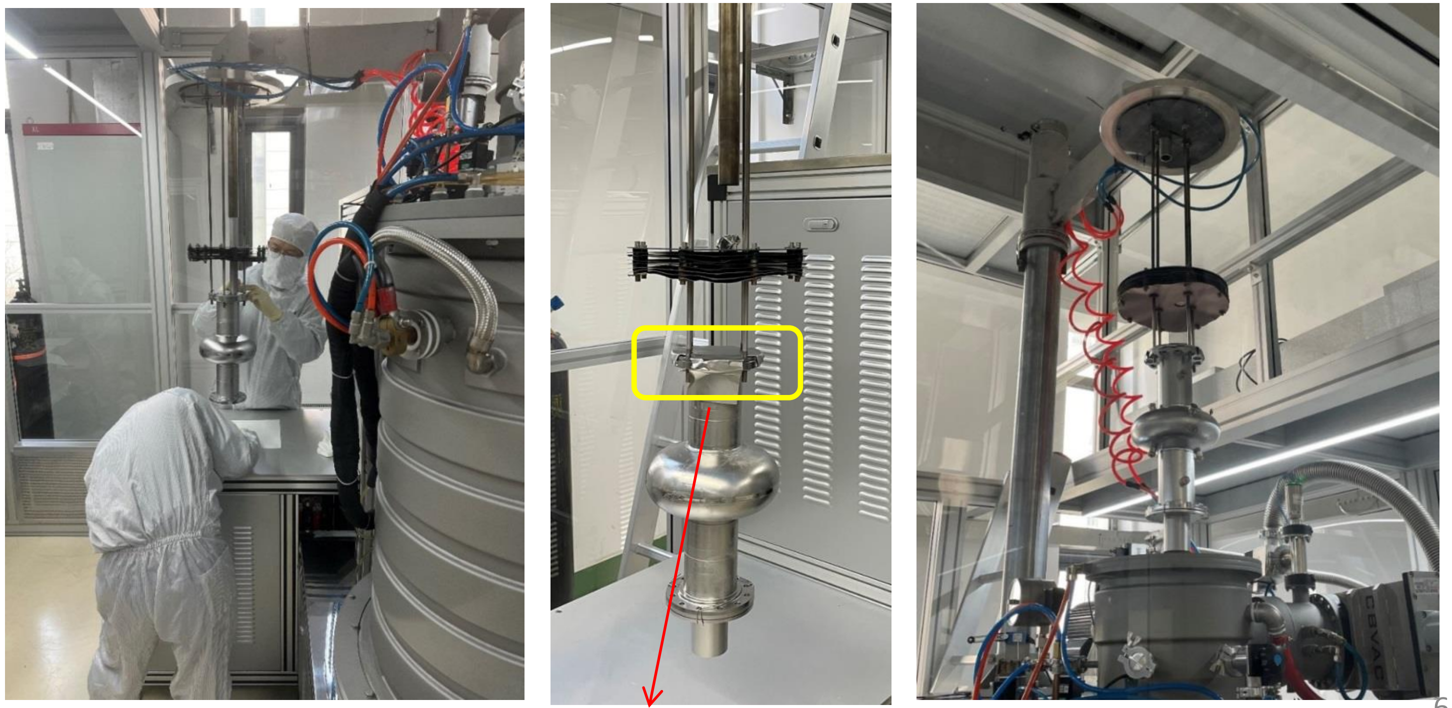
Ramp-down to 40°C: ~25h



A niobium crucible with a lid on top

The second tin source was suspended in the center of the cavity.

The small tray can also be used to place and suspend samples.



The top flange of the cavity was covered with niobium foil.

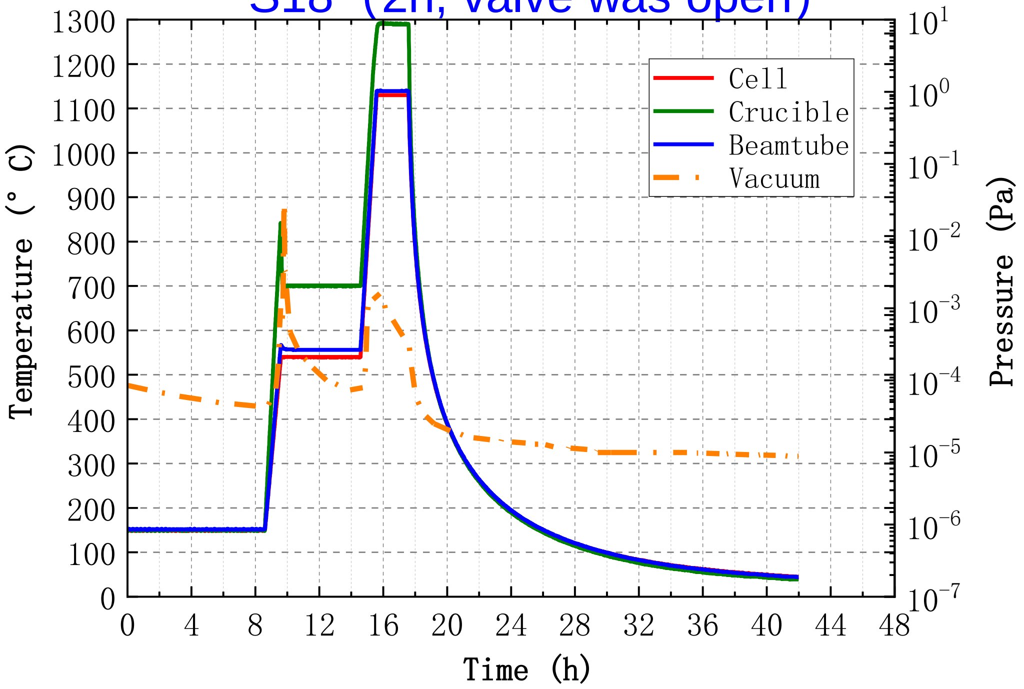
Coating Parameters of 1.3 GHz 1-cell Cavities

Coating Date+ Cavity No.	Degas Temperature & time	Nucleation Temperature& time Crucible/Furnace	Coating Temperature& time Crucible/Furnace	Annealing Temperature& time Crucible/Furnace	Sn (g)	SnCl2 (g)
20240520 + S27	150℃ 17h	Max：850/540℃ 5h	1290 / 1130 ℃ 1h	None	0.66 + 0.34	0.38
20240527 + S18	150℃ 17h	Max：850/540℃ 5h	1290 / 1130℃ 2h	None	0.64 + 0.42	0.4
20240603 + S7	150℃ 17h	Max：850/540℃ 5h	1290 / 1130℃ 3h	None	0.65 + 0.42	0.41
20240611 + S22	150℃ 17h	Max：850/540℃ 5h valve is closed	1290 / 1130℃ 1h valve is closed	None	0.67 + 0.43	0.4
20240620 + S27	150℃ 17h	Max：850/540℃ 5h valve is closed	1290 / 1130℃ 2h valve is closed	None	0.68 + 0.38	0.5
20240627 + S14	150℃ 17h	Max：850/540℃ 5h valve is closed	1290 / 1130℃ 3h valve is closed	None	0.66 + 0.34	0.49

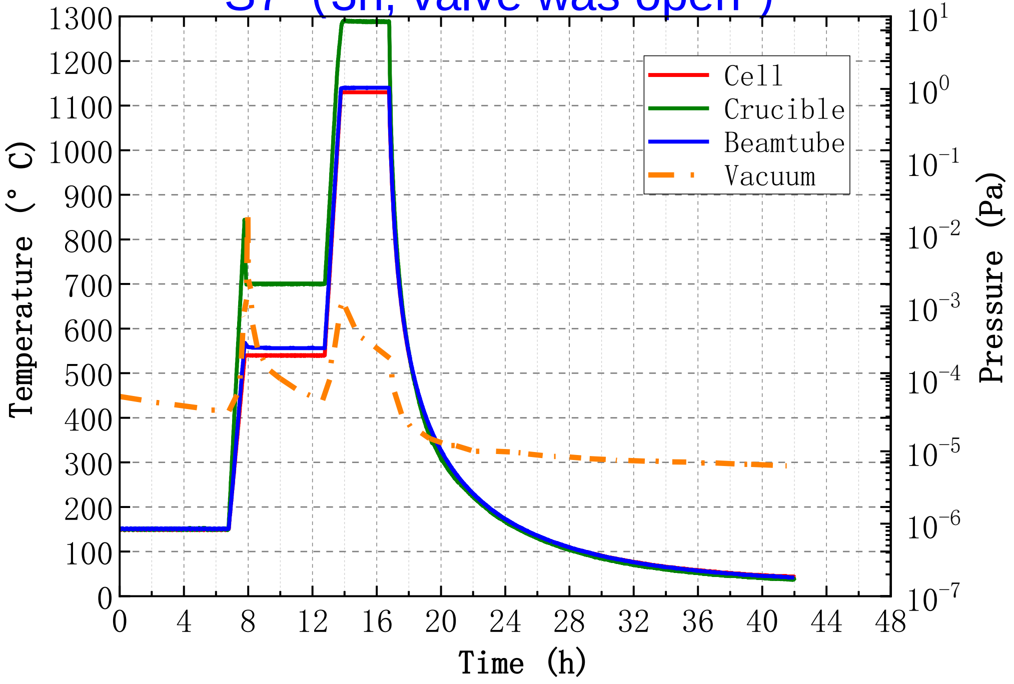
*The valve is open unless specifically stated otherwise.

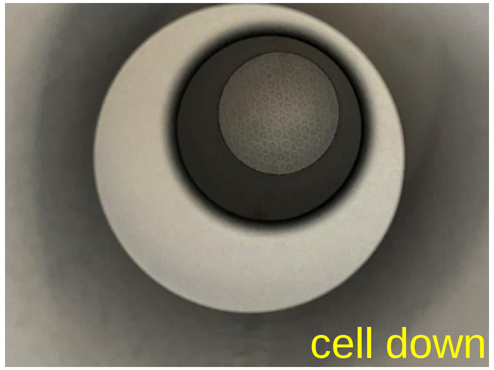
Typical Coating Curves

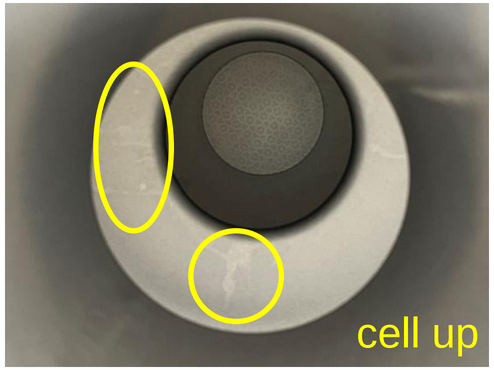
S18（2h, valve was open)

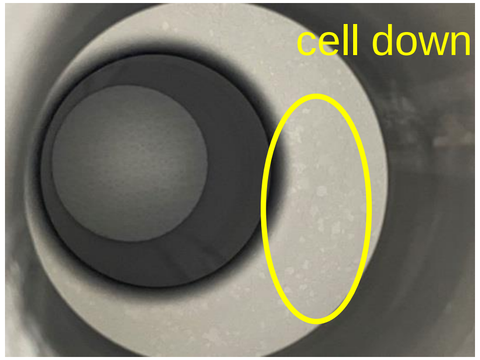


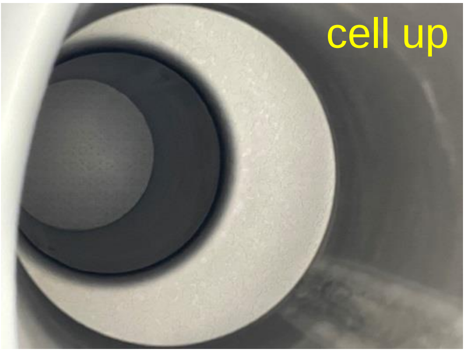
S7（3h, valve was open)





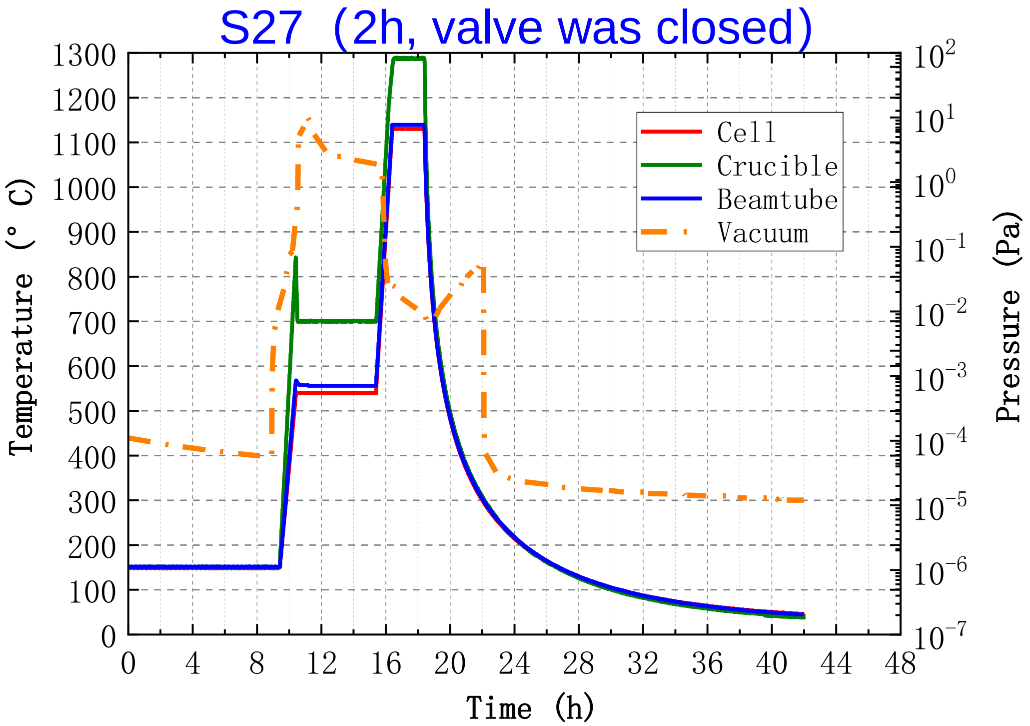


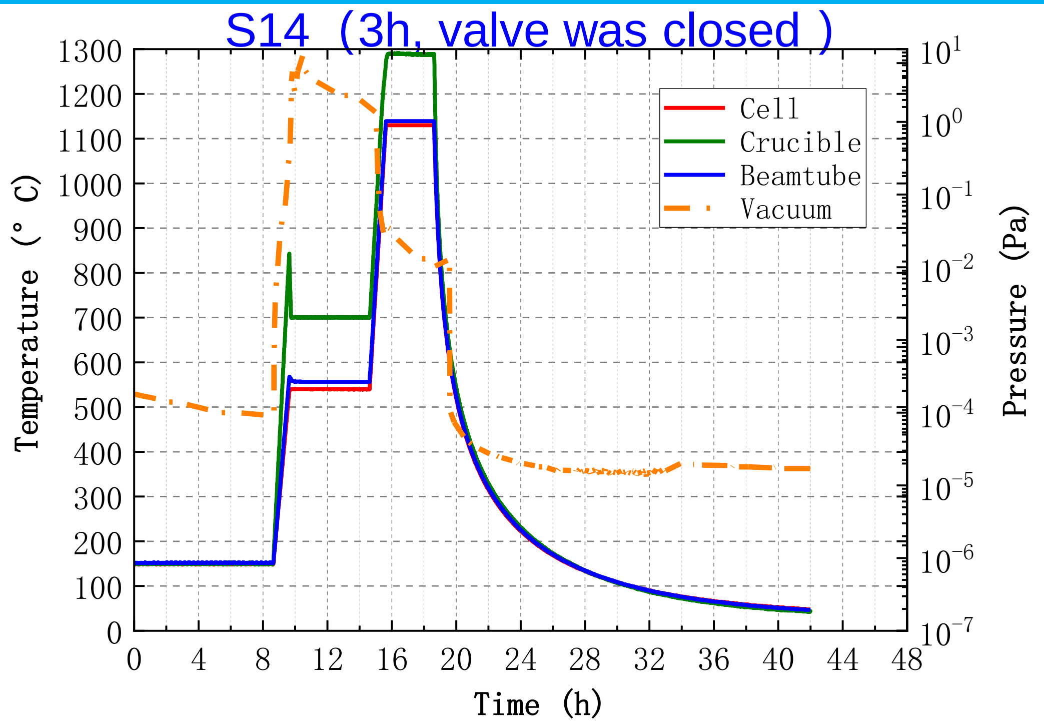


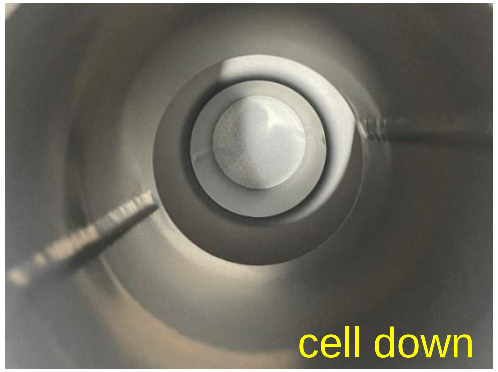


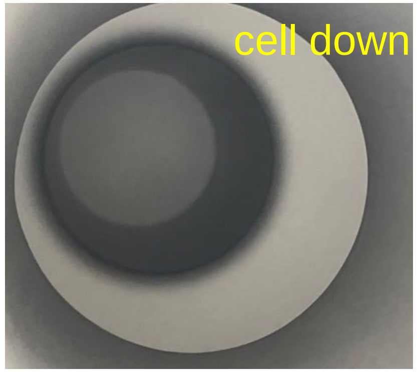
Large patchy regions

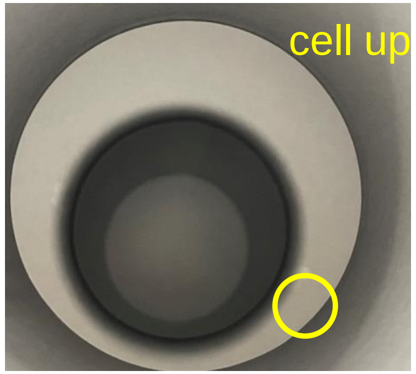
Typical Coating Curves





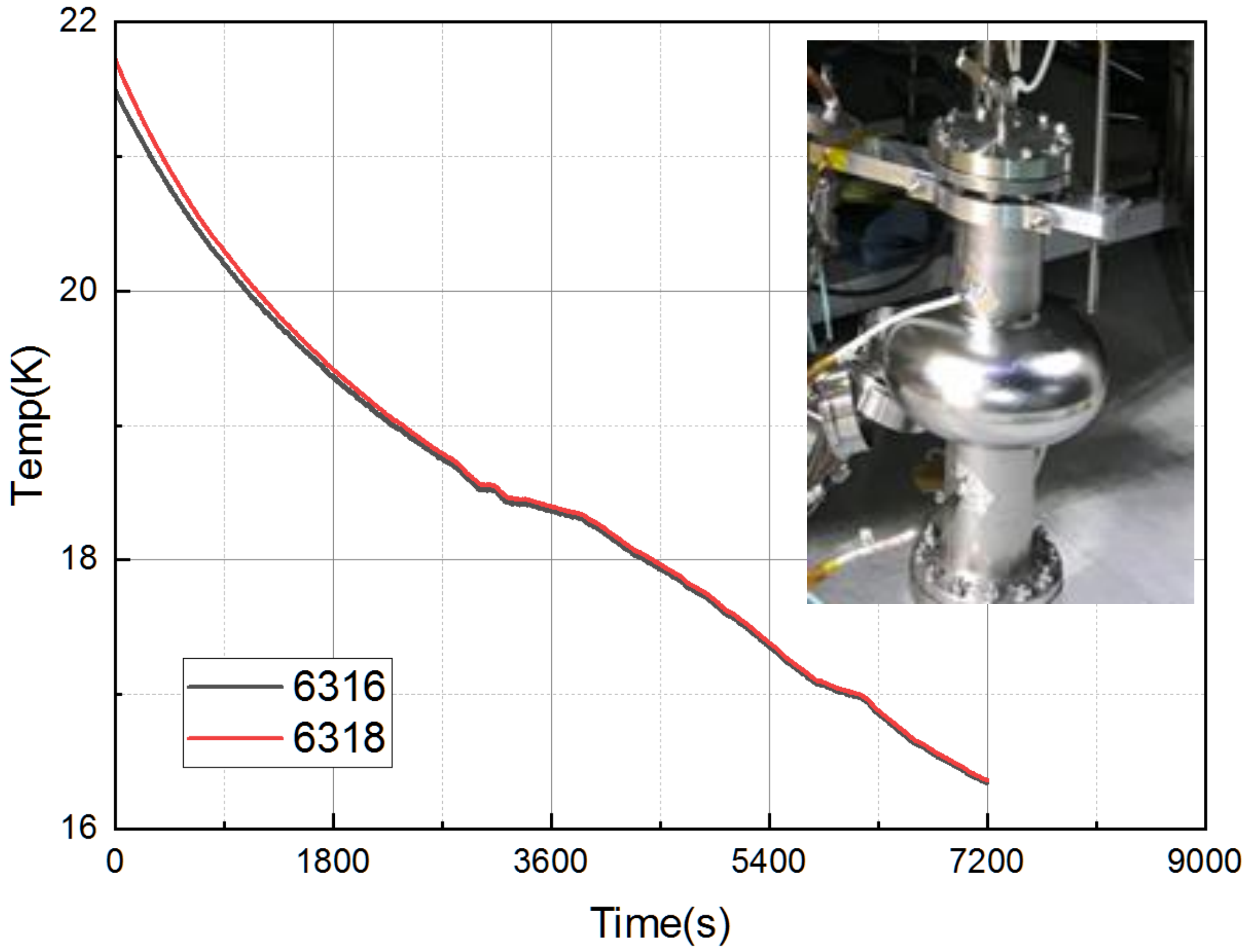






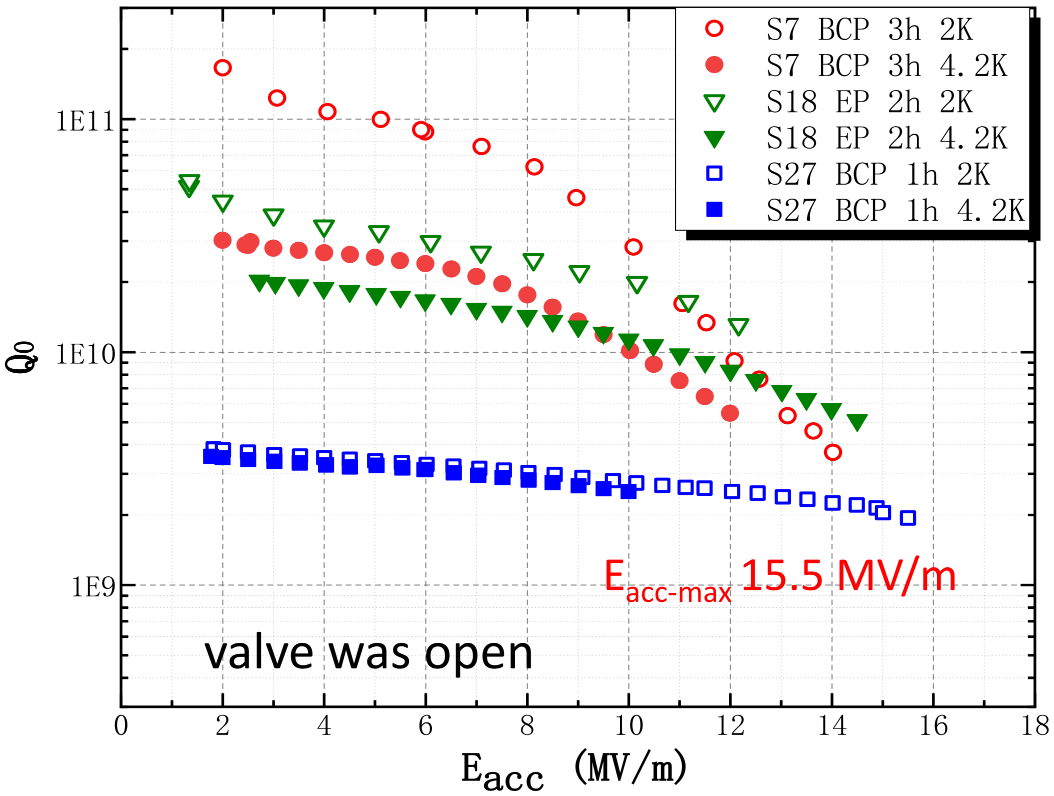
small patchy areas

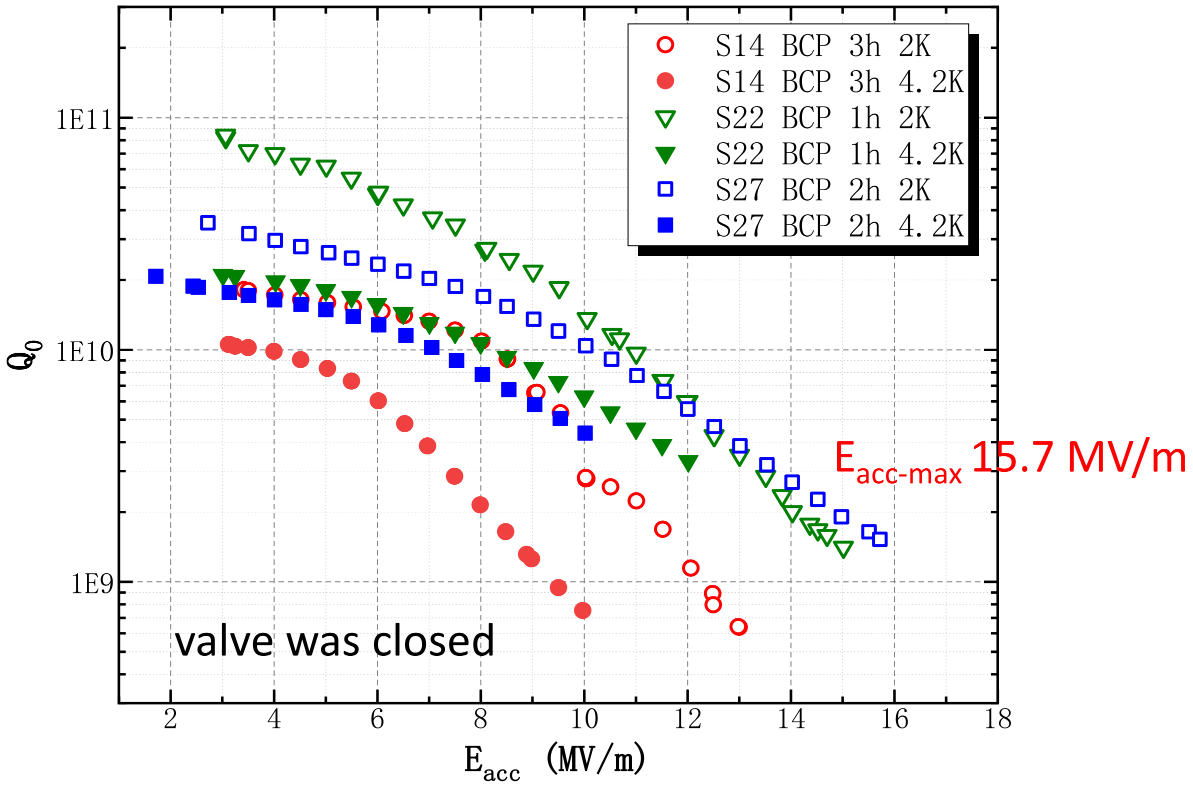
Slow Cooling



During the cooling process, the temperature difference between the upper and lower beamtubes (TI6318, TI6316) on the superconducting cavity is less than 0.02K.

Performance of Nb_3Sn Grown on Nb Cavities

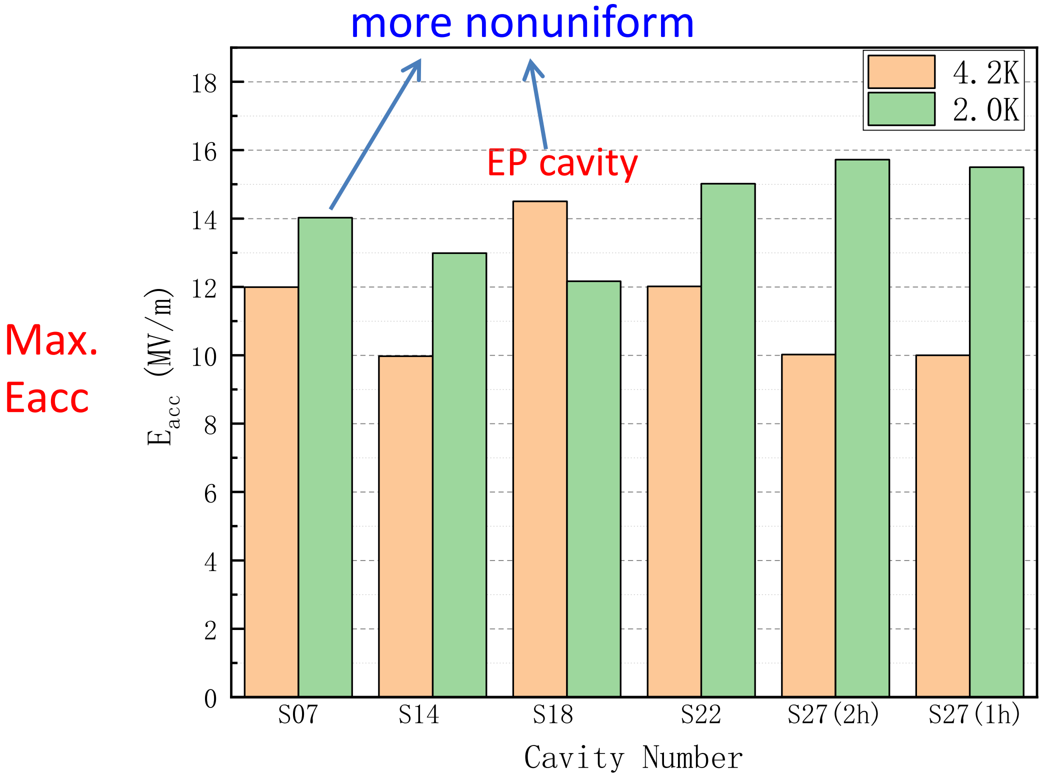


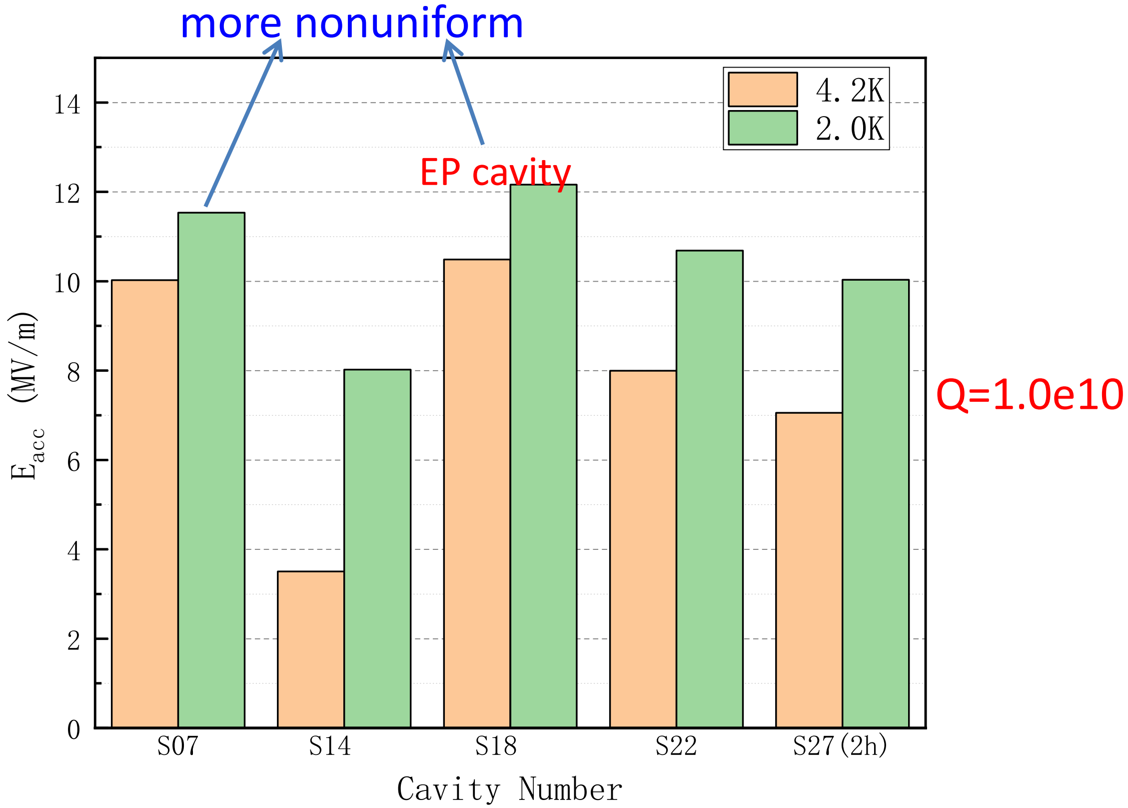


The gradient is almost the same whether the valve was opened or closed during nucleation and coating , but from the perspective of Q value, the coating effect is better when the valve was open.

The shorter the coating time with the valve closed, the better the vertical test results?

Performance of Nb_3Sn Grown on Nb Cavities





1. The only EP cavity S18 is the best-performing overall.
2. Based on the surface morphology, cavities with more patchy regions did not perform worse in the end.

Characterizations of Nb_3Sn Coated Samples

Characterizations of Nb_3Sn coated samples typically involve the following aspects :

(1) Structure characterization : Rigaku SmartLa X-ray Diffractometer (XRD)

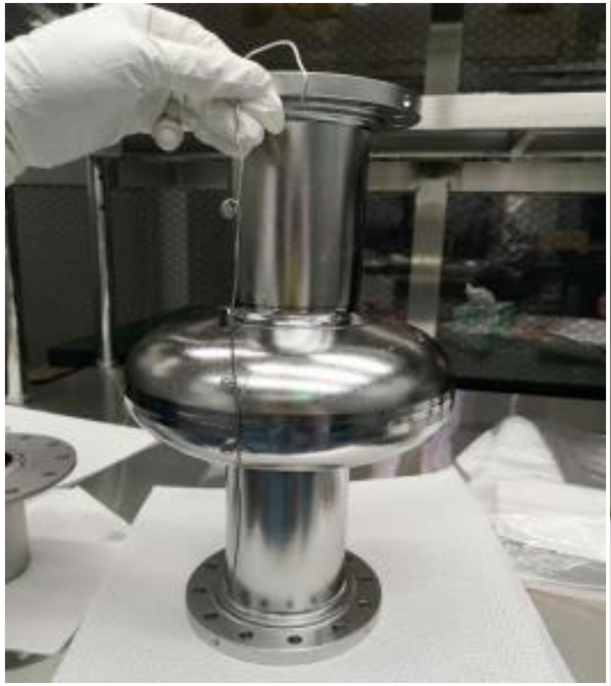
(2) Surface morphology: Hitachi SU5000/8100 Scanning Electron Microscope (SEM)

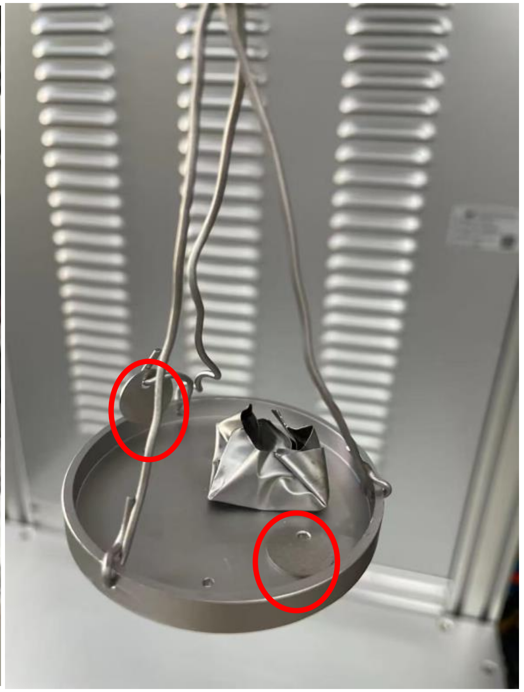
(3) Electromagnetic properties:
- R-T—Quantum Design Physical Property Measurement System (PPMS-9T)
- M-T/M-H—Quantum Design Magnetic Property Measurement System (MPMS-3)

(4) Composition characterization: Energy Dispersive X-ray Spectroscopy (EDS) and Wavelength Dispersive X-ray Spectroscopy (WDS).

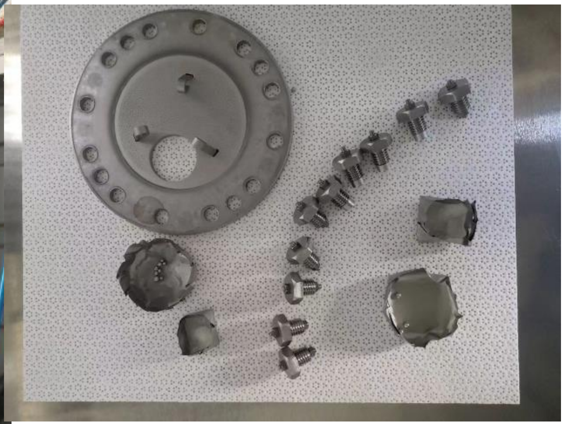
(5) Film thickness measurement: Focused Ion Beam (FIB) + SEM.

Nb_3Sn Coated Samples





sample cavity

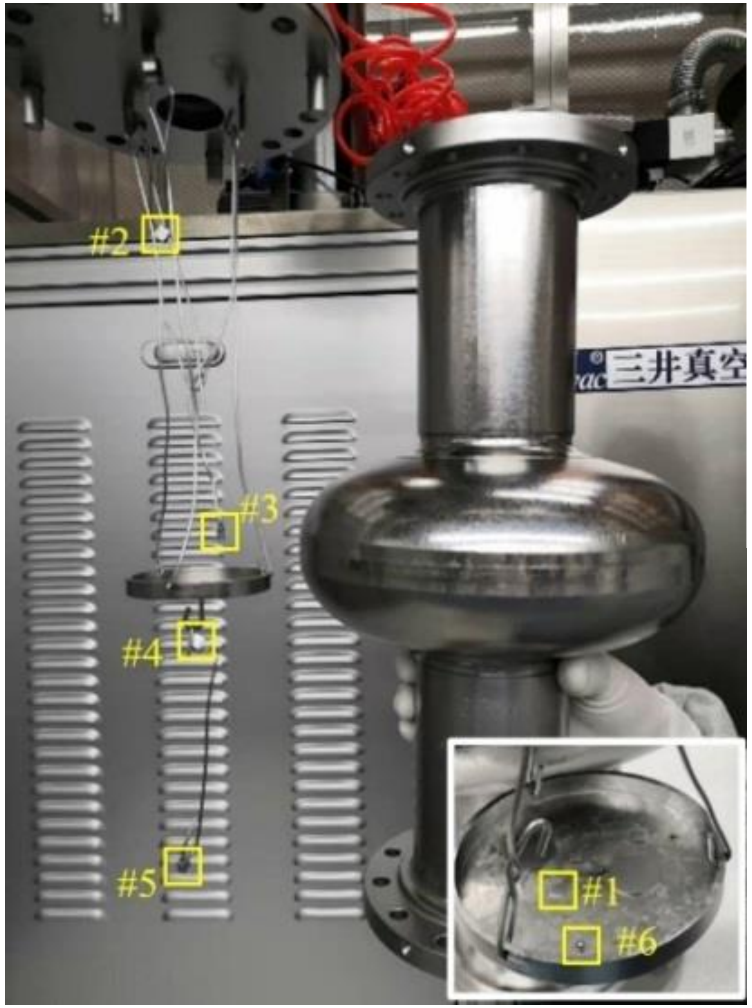


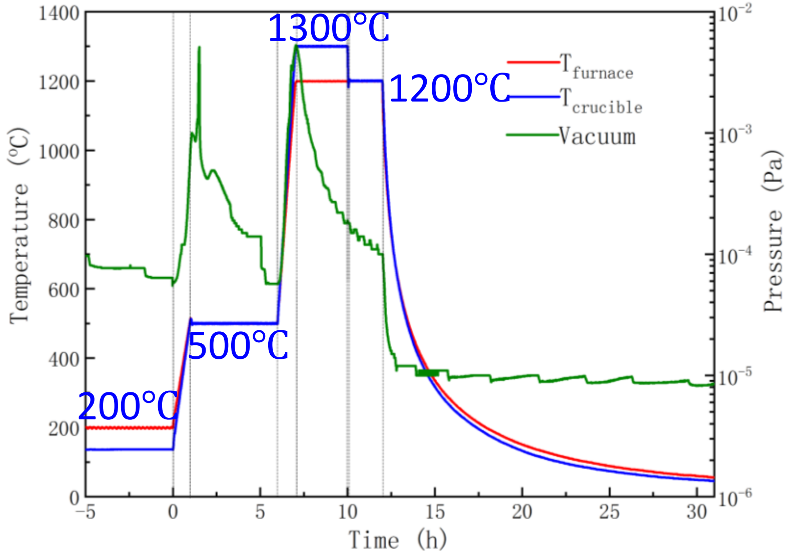
Ring-shaped sample

The first type: samples that are coated together with cavities, suspended in the middle of the cavity, or placed on the central tray.

The second type: a sample cavity capable of accommodating 10 samples (upper and lower beamtubes: 2, upper and lower cells: 8).

Structural Characterization

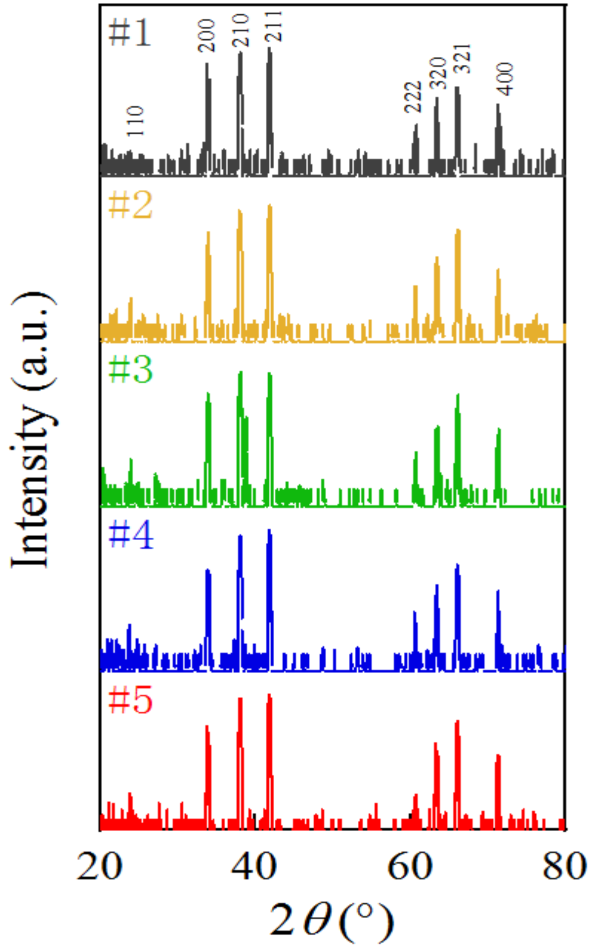




#1: $3 \times 3 \times 0.5\ mm^3$

#2 – #5: $5 \times 5 \times 2.8\ mm^3$

#6: $\emptyset 1\ mm^3$



θ-2θ scans of samples #1-#5

The crystallinity was good and there were no impurity phases.

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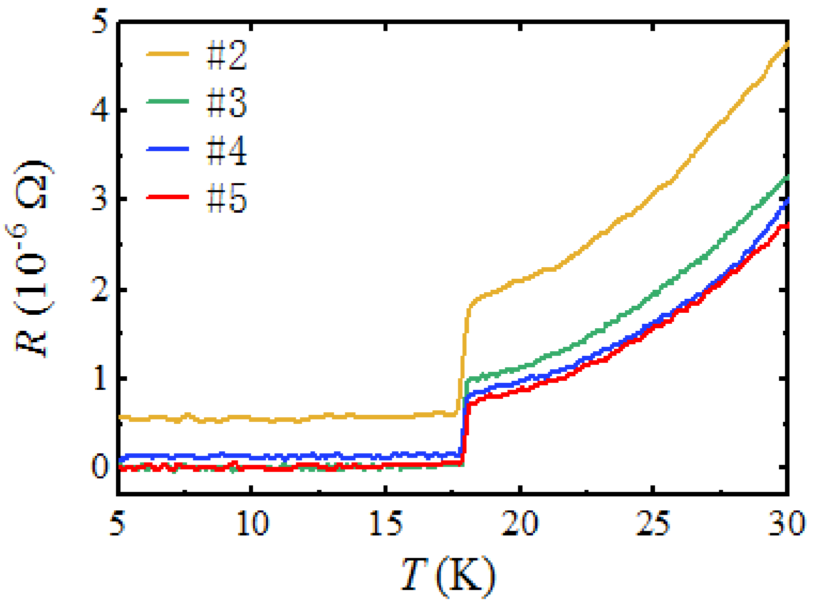
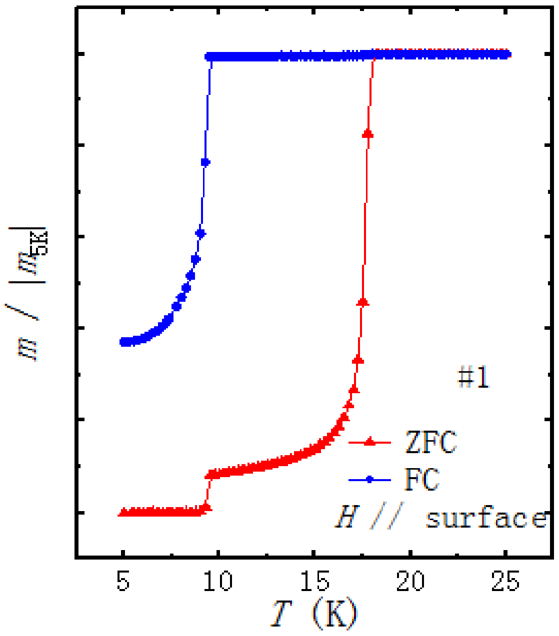
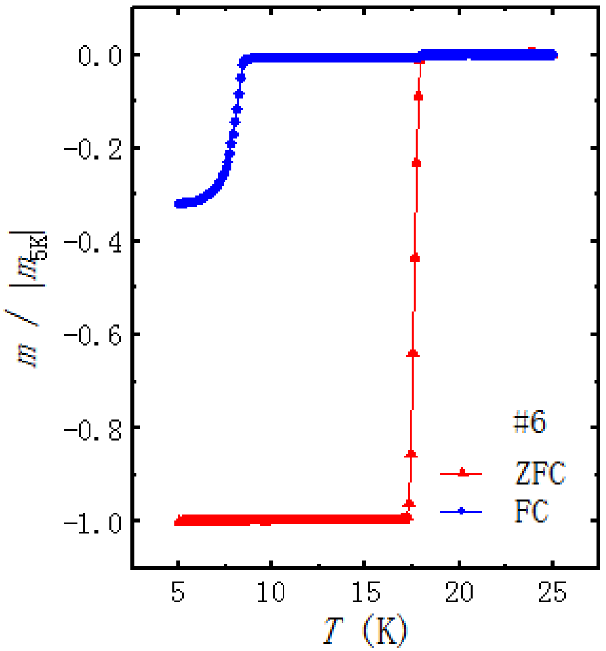
R-T/ m-T　Measurements







- Samples #2-#5 had a consistent T_c value of about 18 K.
- Both XRD and R-T data confirmed the almost identical coating quality at different locations of the cavity.
- Two transitions occurred at 9.4 K and 18.0 K in ZFC for sample #1, confirming the superconductivity of both Nb and Nb_3Sn.
- Only one transition occurred in ZFC for sample #6, which suggested the Nb sphere was well enclosed by the Nb_3Sn layer, and the magnetic field was entirely excluded outside the Nb_3Sn layer.

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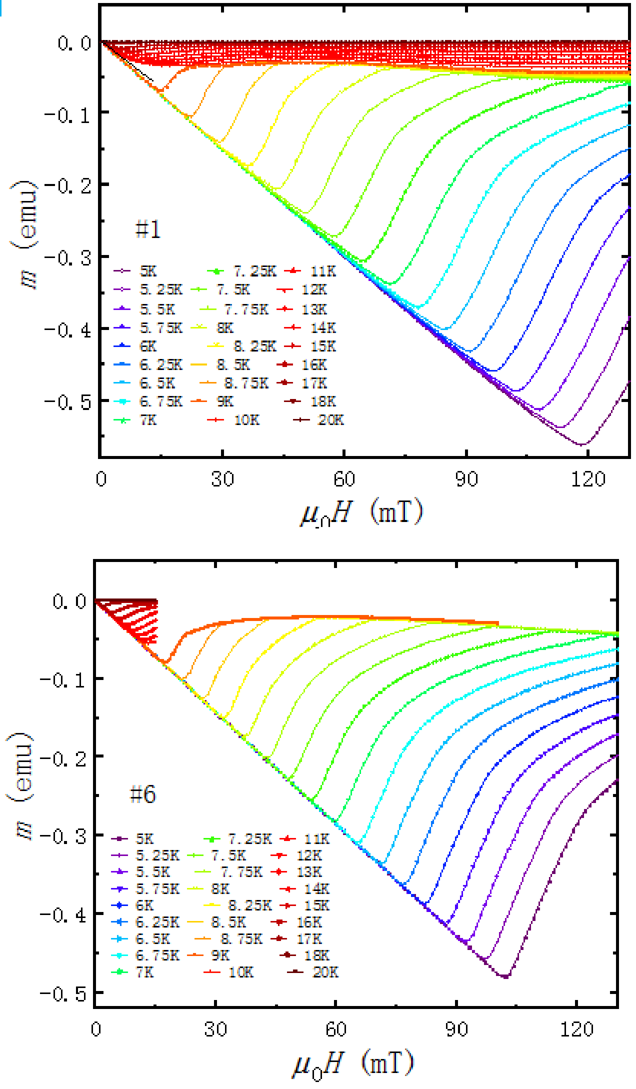
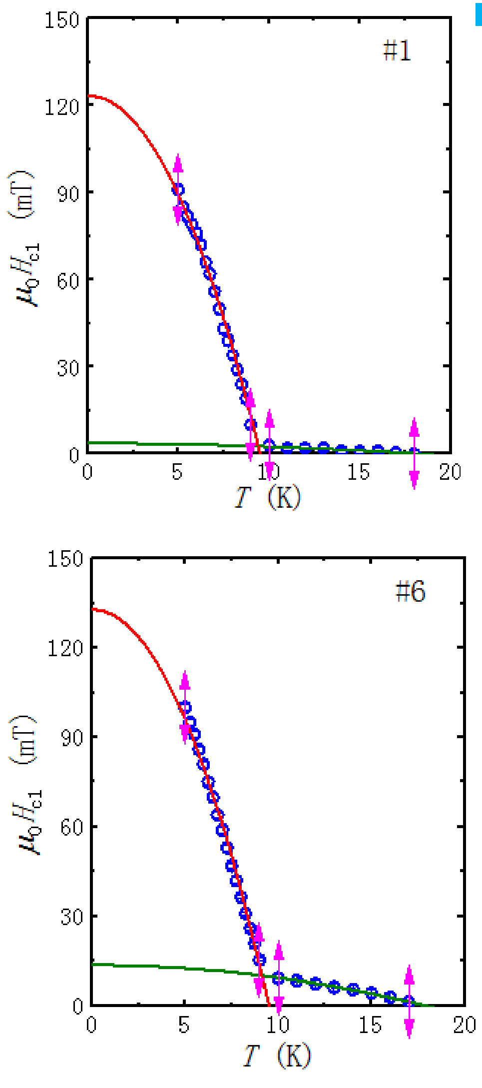
B_{c1} in Nb_3Sn/Nb



Magnetization curves at various temperatures for #1 and #6 .



Temperature dependences of B_{c1} for #1 and #6.

- Fitting formula：

  $\mu_0 H_{c1}(T) = \mu_0 H_{c1}(0)[1-(T/T_c)^2]$;

- The $B_{c1}(0)$ of #6 becomes 130 mT when only the data lower than 10 K is fitted. This is consistent with bulk Nb.
- The $B_{c1}(0)$ of #6 is about 14 mT when only the data higher than 10 K is fitted, less than that of Nb_3Sn.

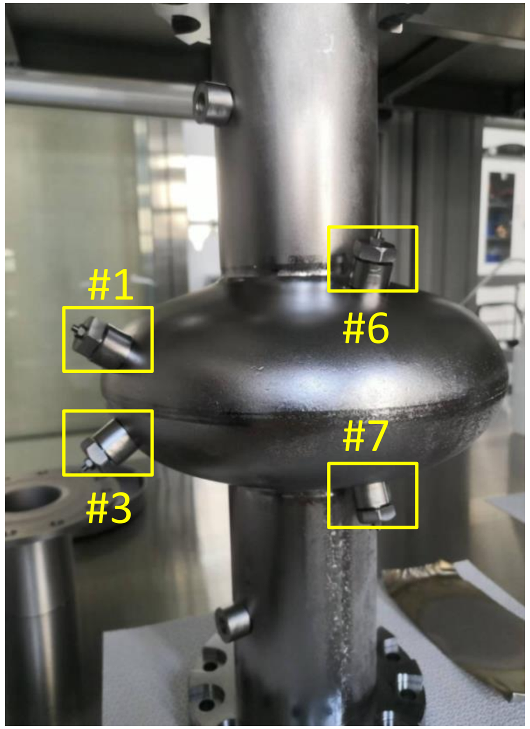
Therefore, there is still much room for the improvement of the critical parameters of Nb_3Sn coatings.

Coating Parameters of the Sample Cavity

Coating Date	Degas Temperature & time	Nucleation Temperature& time Crucible/Furnace	Coating Temperature& time Crucible/Furnace	Annealing Temperature& time Crucible/Furnace	Sn (g)	SnCl2 (g)
20240506	150℃ 17h	Max：850/540℃ 5h	1290C / 1130 3h	1170C / 1130 2h	0.6+0.33	0.32+0.2
20240516	150℃ 17h	Max：850/540℃ 5h	1290C / 1130 1h	None	0.6+0.36	0.32
20240523	150℃ 17h	Max：850/540℃ 5h	1290C / 1130 2h	None	0.61+0.4	0.4
20240530	150℃ 17h	Max：850/540℃ 5h	1290C / 1130 3h	None	0.65+0.42	0.41
20240606	150℃ 17h	Max：850/540℃ 5h valve closed	1290C / 1130 1h valve closed	None	0.65+0.42	0.41
20240617	150℃ 17h	Max：850/540℃ 5h valve closed	1290C / 1130 2h valve closed	None	0.65+0.44	0.4
20240624	150℃ 17h	Max：850/540℃ 5h valve closed	1290C / 1130 3h valve closed	None	0.65+0.41	0.53

*The valve is open unless specifically stated otherwise.

Sn content % by WDS



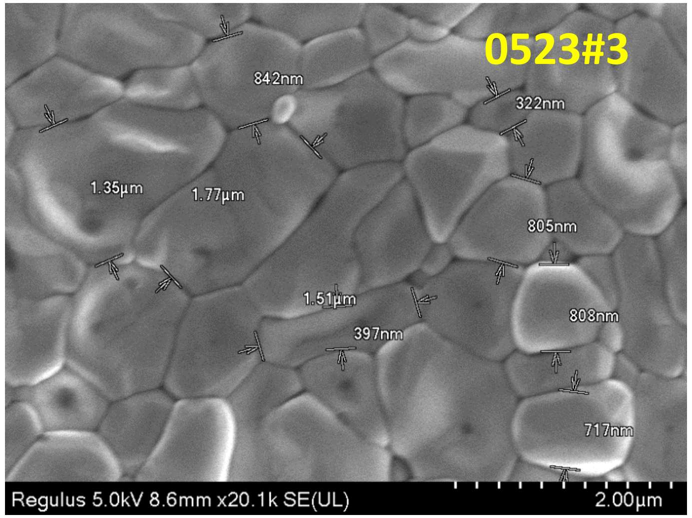
Date	#1	#3	#6	#7	avg.	Sn rich /poor	#P	#D
0506	25.6	25.3	25.5	24.6	25.3	Rich	--	--
0516	20.7	21.0	21.4	20.0	20.7	Severely poor	3.3	12.7
0523	22.6	22.5	21.7	22.5	22.3	Severely poor	24.6	24.9
0530	24.1	24.2	24.0	23.9	24.1	Poor	--	25.8
0606	23.9	23.8	23.8	24.0	23.9	Poor	--	24.5
0617	25.7	25.4	25.1	26.0	25.6	Rich	--	25.5
0624	25.8	26.0	--	25.7	25.8	Rich	--	26.3

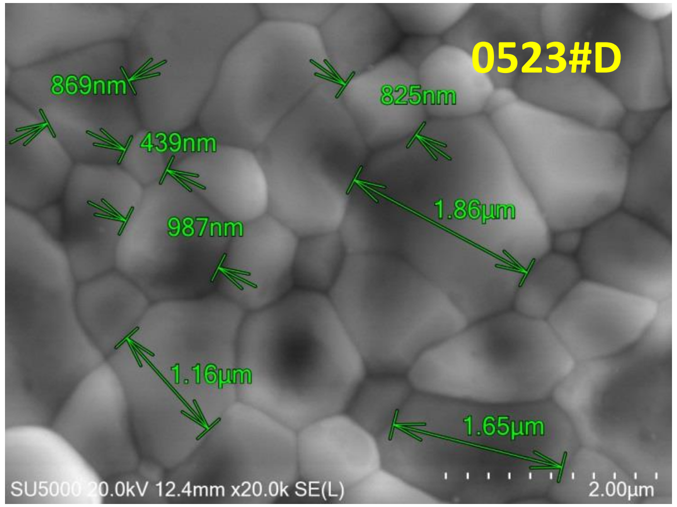
1.Samples at different locations on the inner surface of the cavity: #1 #3 #6 #7;
2. The sample suspended in the center of the cavity: #D;
3. The sample placed on the tray: #P.

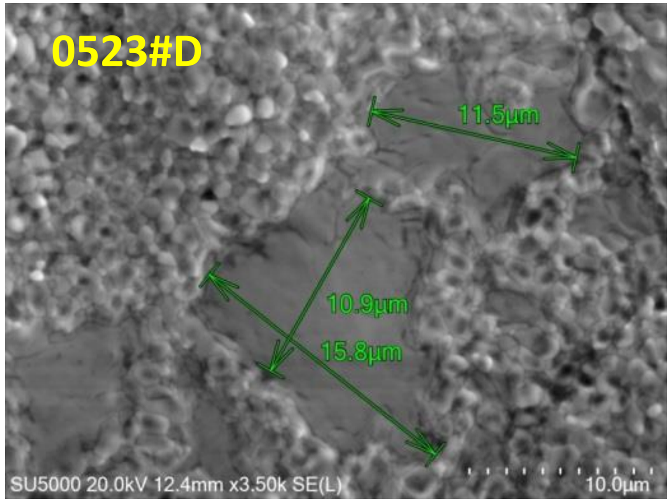
The differences in Sn content between different positions on the inner surface are not significant. **Nonuniformity: main form Sn poor regions.**

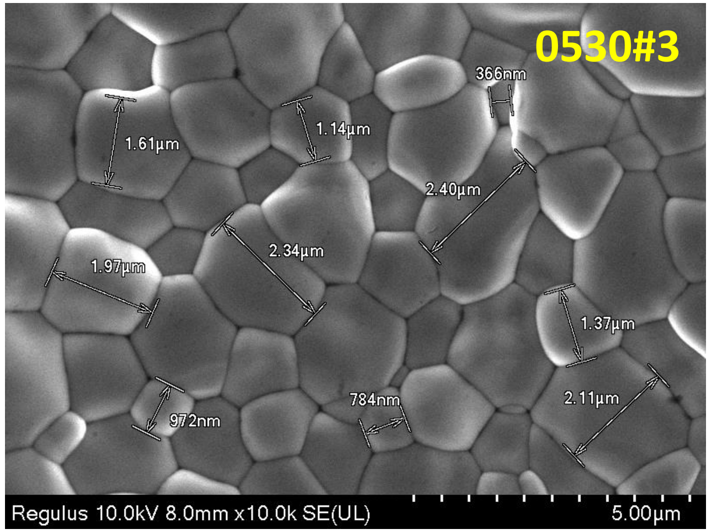
The Sn content results of the sample suspended in the center of the cavity cannot represent the actual values well.

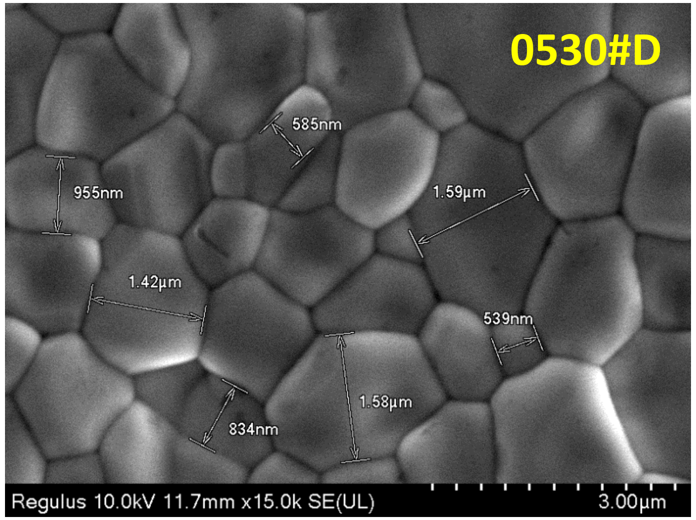
Surface Morphology (SEM)

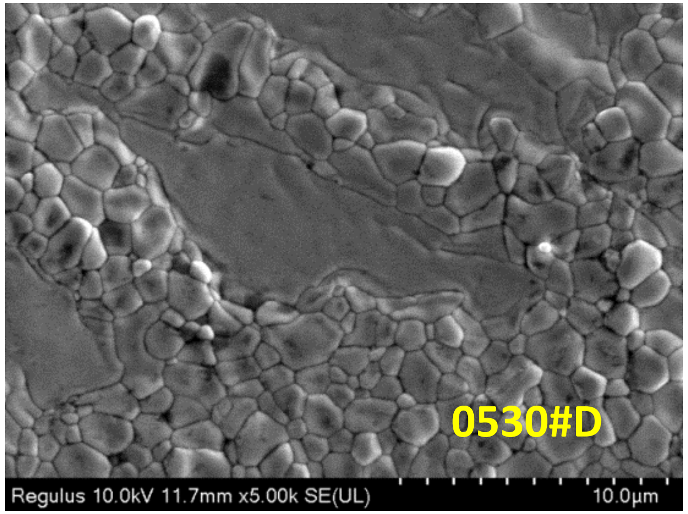








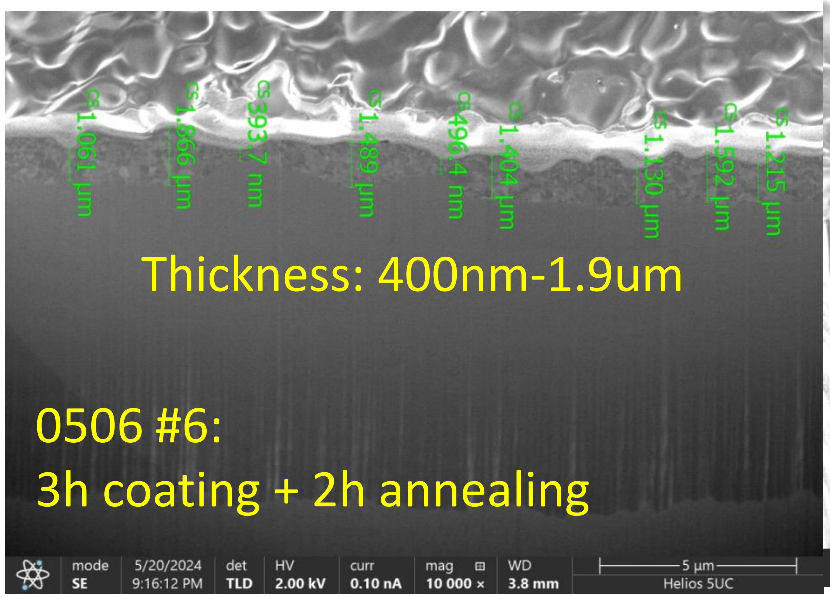


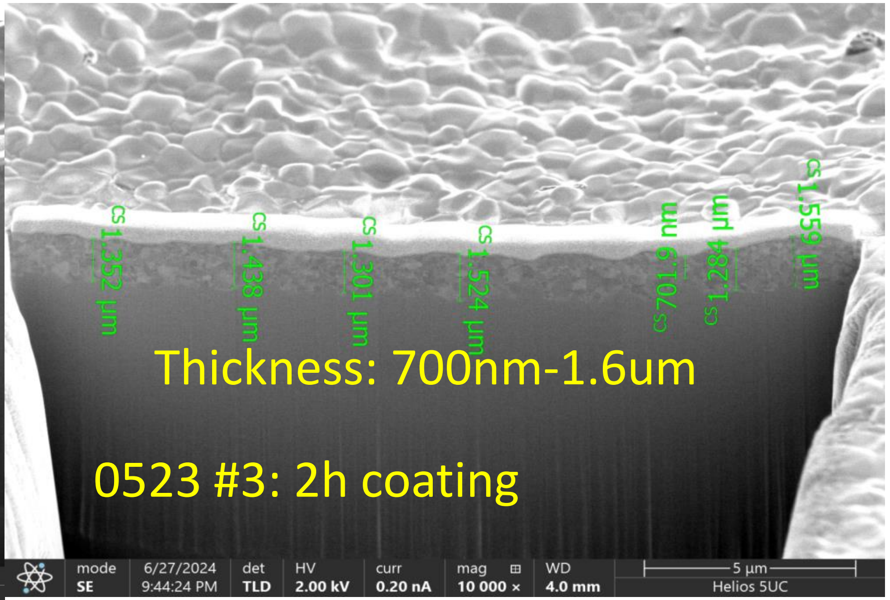


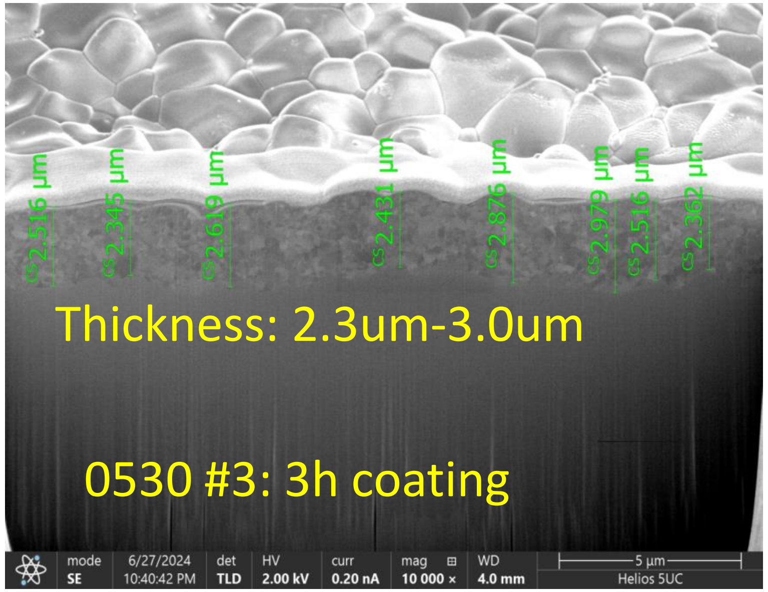
Max grain size: 3um

Grain sizes large than 10um only occurred on the samples suspended in the center of the cavity.

Film Thickness Measurement







The uniformity of film thickness is strongly correlated with the flatness of the substrate.

Film thickness is directly proportional to the growth time of the thin film.

Next step: investigate the variations in film thickness at different positions.

Technical Challenges

Nonuniformity

(1) Variation in film thickness

↓

Smoother substrate

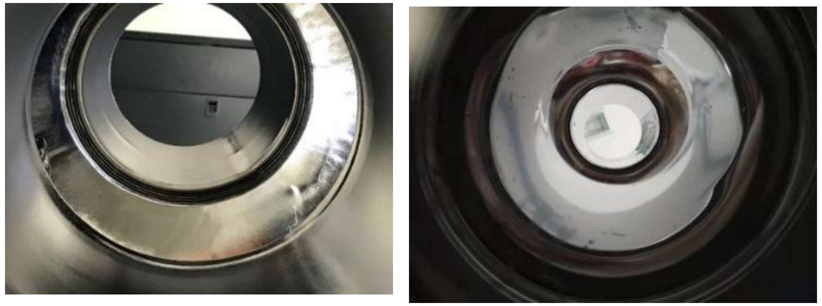
↓

Mechanical polishing + cold EP

↓

Thinner Nb_3Sn coating film

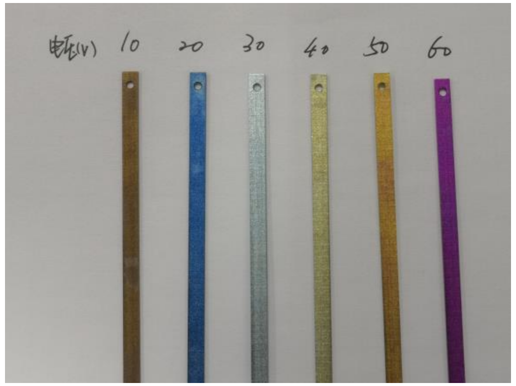
Mechanical polishing equipment



The inner surface of a large grain cavity before and after mechanical polishing.

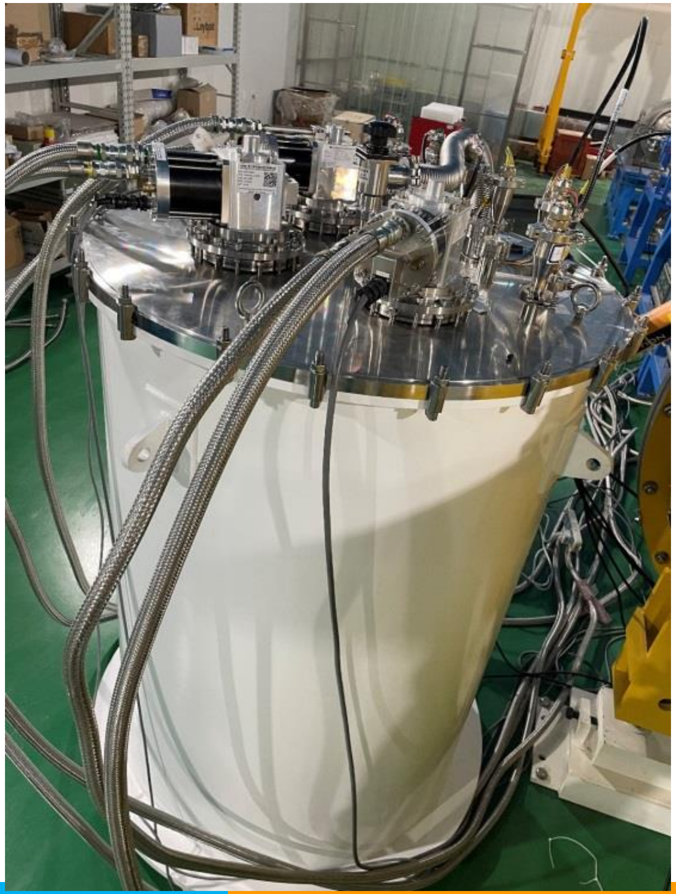
(2) Anodize the niobium cavity:

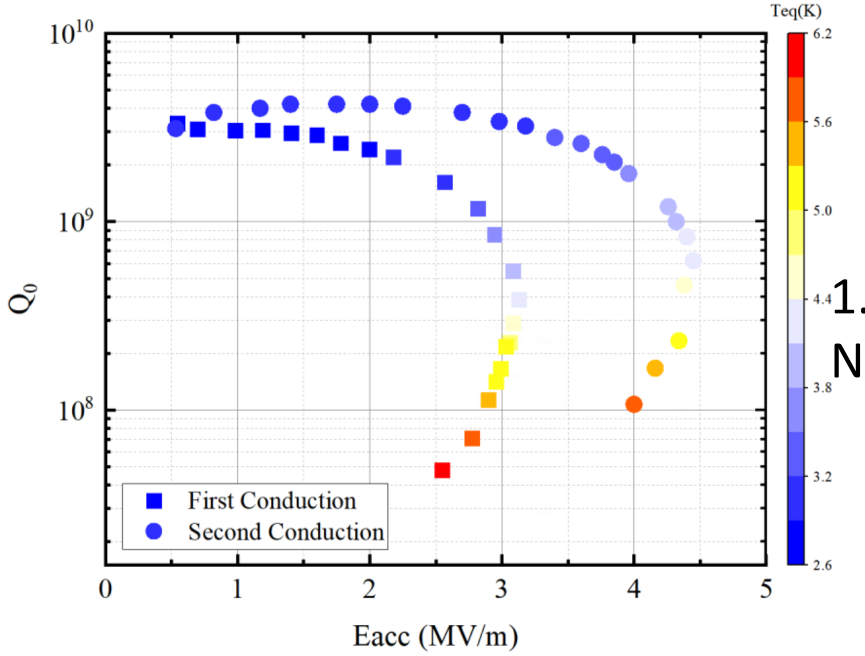
to improve the uniformity of tin nucleation.



Summary and Perspective

- In future, the coating process will be further optimized, in order to enhance Q_0 and E_{acc} of Nb_3Sn SRF cavities.
- Besides, the conduction cooling of Nb_3Sn SRF cavities will be studied, too.





1.3GHz 1-cell Nb cavity

Currently, we have successfully tested a niobium cavity using conduction cooling **with commercial cryocoolers.**

Thank you for your attention!