Recent Progress of Nb_3Sn Thin Film SRF Cavity Coated by Vapor Diffusion Method at IMP

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Outlines

1 Background

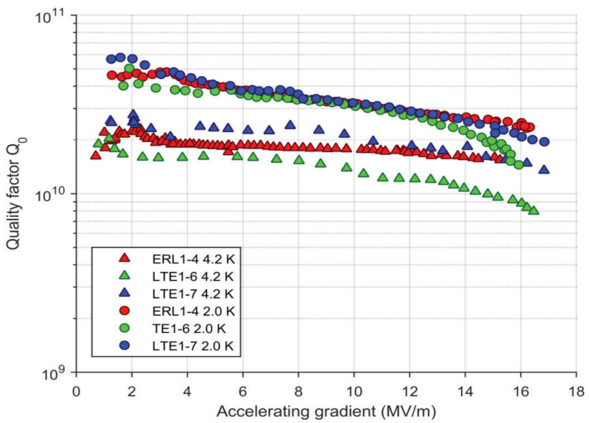
2 Coating process optimization

3 Conclusion

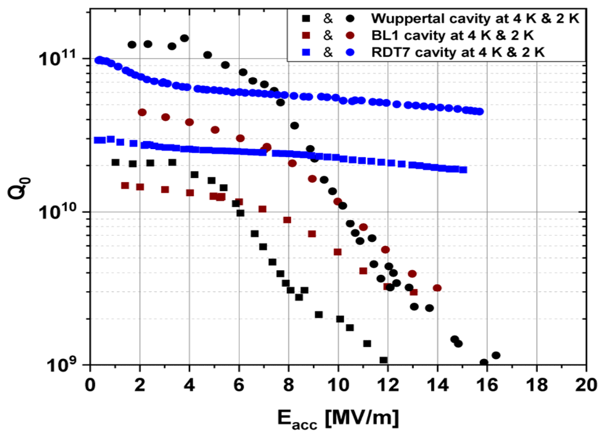
Background

- Amongst the research activities conducted since the 1970s, only **Cornell University, JLab, and FNAL** have managed to successfully coat high-performance Nb_3Sn SRF cavities by the vapor diffusion method

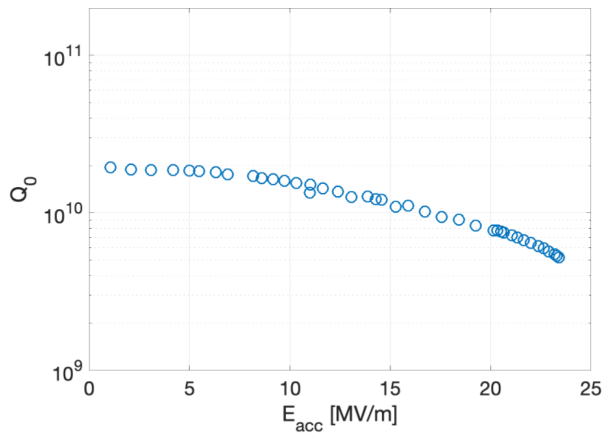
- **Further investigations and optimizing the coating process** of high-performance Nb_3Sn SRF cavities using the vapor diffusion method remains an essential research problem for further studies



Cornell results



JLab results



FNAL results

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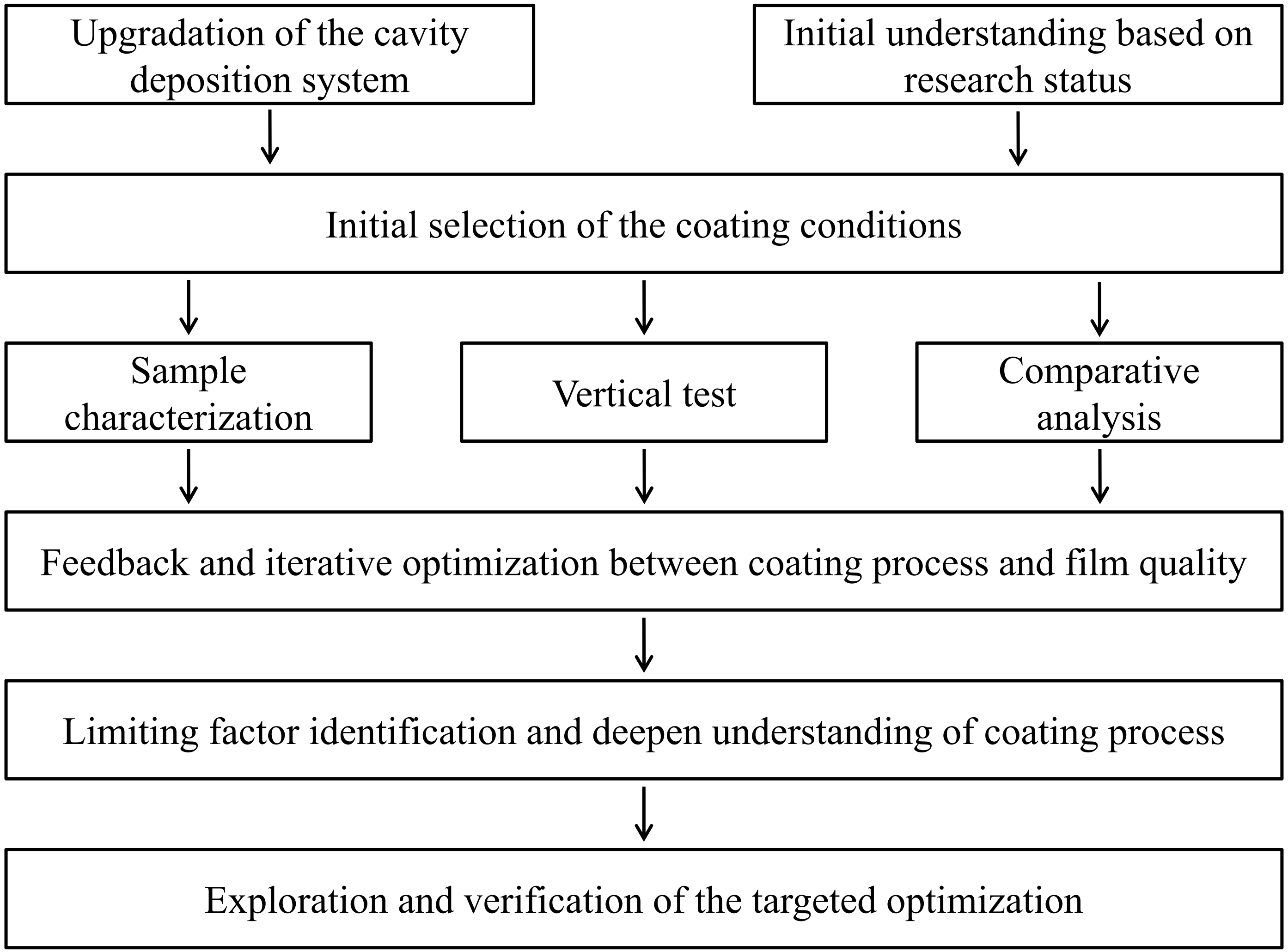
Research approach



Differences and optimizations of the subsequent 7 coating processes

Coating process	Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor			
			Sn power[1]	Valves[2]	Mo belt[3]	Ceramics[4]
1	1310	180	On	Closed	On	Without
2	1200	180	On	Closed	On	Without
3	1200	180	Off	Closed	On	Without
4	1200	180	On	Opened	On	Without
5	1200	180	On	Closed	Bottom On	Without
6	1200	180	On	Closed	On	With
7	1233	120	Off	Closed	Bottom On	With

[1]The condition of the Sn source power supply immediately after the film growth stage.

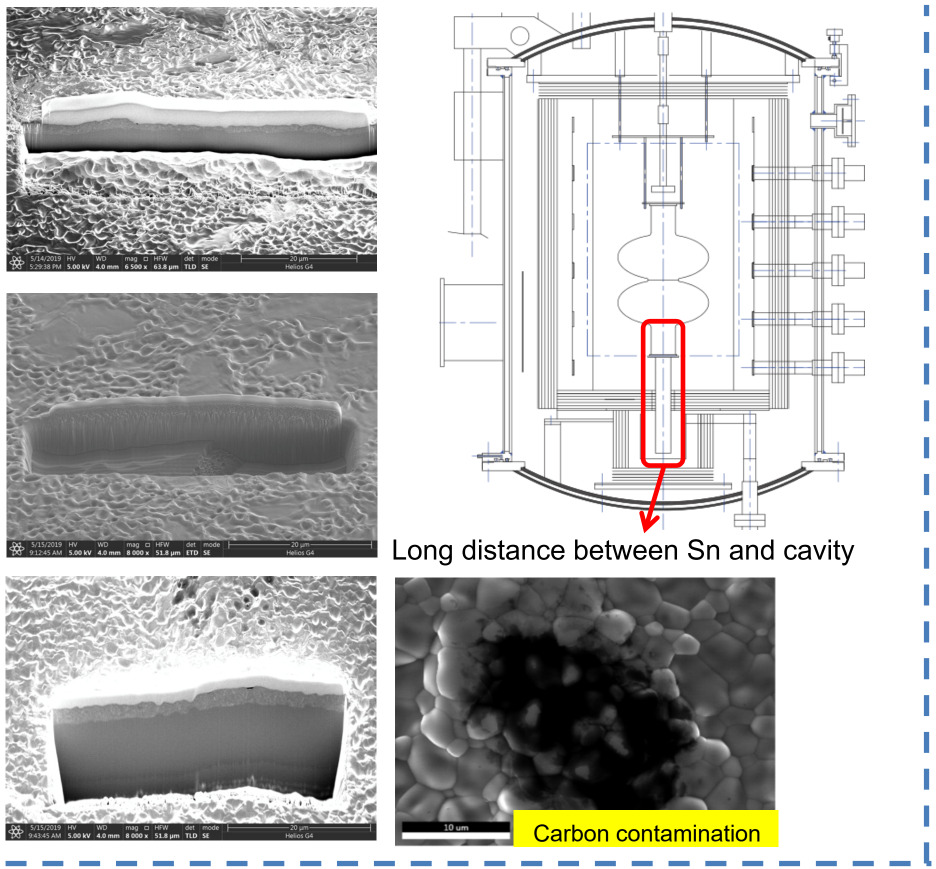
[2]The condition of the valves between the Nb coating chamber and the vacuum pump.

[3]The condition of the Mo heating belts surrounding the Nb coating chamber throughout the coating process.

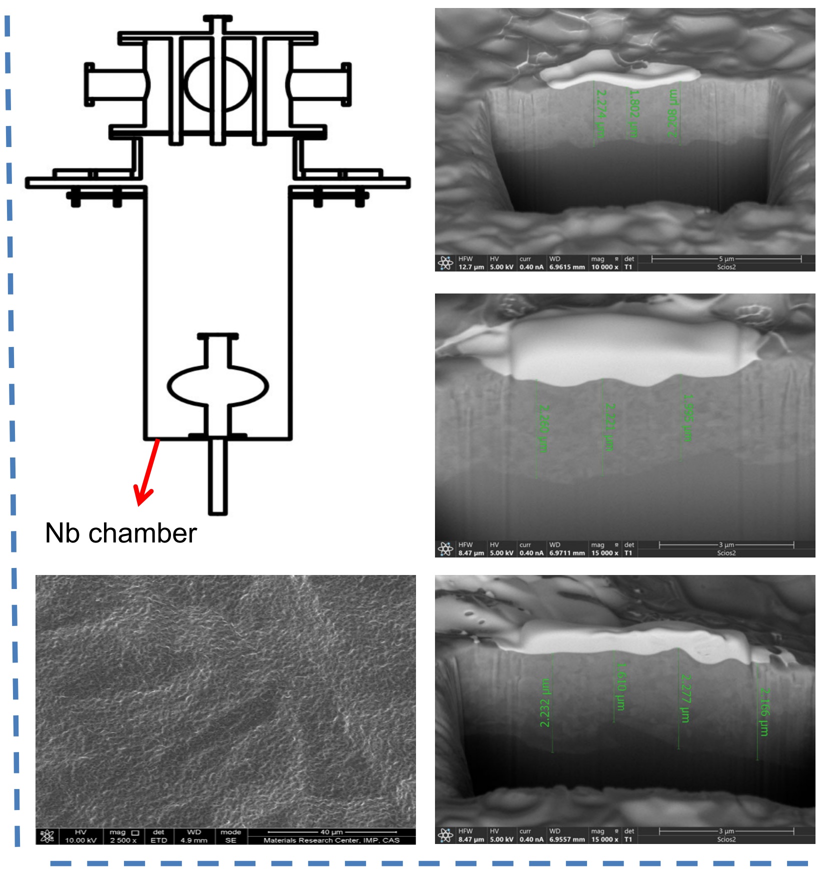
[4]Whether or not to hang high-purity absorbent ceramic bricks above the cavity.

The deposition system

Initial Design without Nb chamber



Upgrading of the deposition system

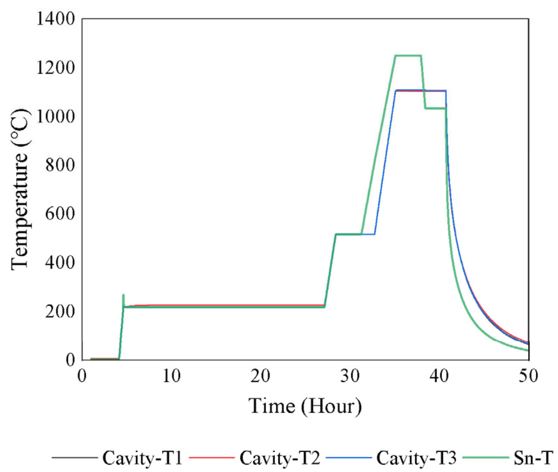


Initial design: C contamination from absence of Nb chamber, non-uniform growth of Nb_3Sn films from the distance of Sn and cavity

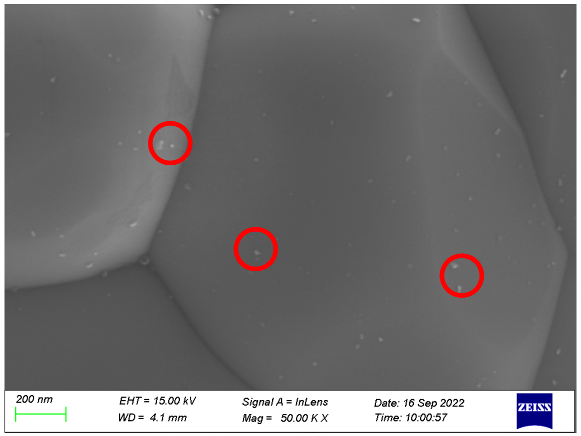
Upgradation: Nb chamber to prevent contamination, heat transfer along the chamber wall to reduce the distance of Sn and cavity

Coating process optimization

➢ *Coating Process (1)*



Temperature profiles of coating process (1)



Nanometer-scale white spots on the surface

ICP-OES test results of the Sn content in the HNO_3 solution

Reagents	Sn (ppm)	Conditions
1#	0.26	Background
2#	0.47	Immersed with Nb_3Sn sample

XPS surface analysis results of the Nb_3Sn sample before and after immersion in HNO_3 solution

Samples	Nb Atomic %	Sn Atomic %	Sn/Nb
Original	45.22	54.78	1.21
HNO_3 rinse	71.03	28.97	0.41

Surface Sn content of samples measured by AES before and after immersion in HNO_3 solution

Samples	Nb Atomic %	Sn Atomic %	Sn/Nb
Original	54.39	45.61	0.84
HNO_3 rinse	67.90	32.10	0.47

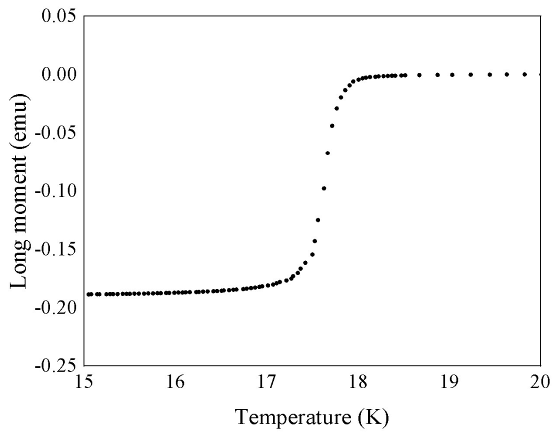
The nanometer-scale white spots are Sn droplets that are formed by condensation of the residual Sn vapor on the film surface during the natural cooling process

Coating conditions of process (1)

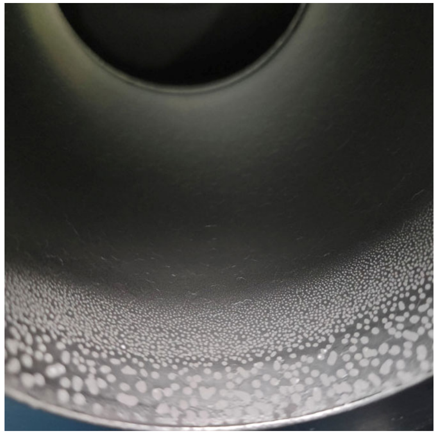
Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor			
		Sn power	**Valves**	**Mo belt**	**Ceramics**
1310	180	On	Closed	On	Without

Coating process optimization

➢ *Coating Process (1)*



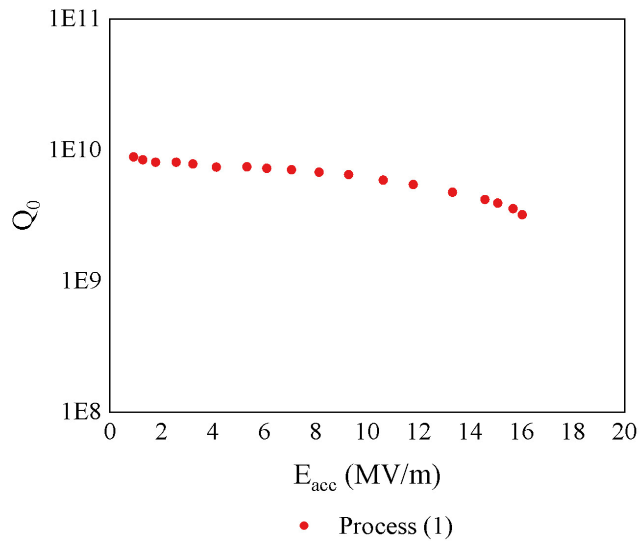
M-T measurements: T_c>18K



Visible millimeter-scale Sn spots near the bottom flange, where the temperature was about 10^0C lower than the rest of the cavity

EDS composition of the Nb_3Sn films by Coating Process (1)

Position	1	2	3
Sn content(at. %)	25.94	26.57	26.69
Average Sn content	26.40		



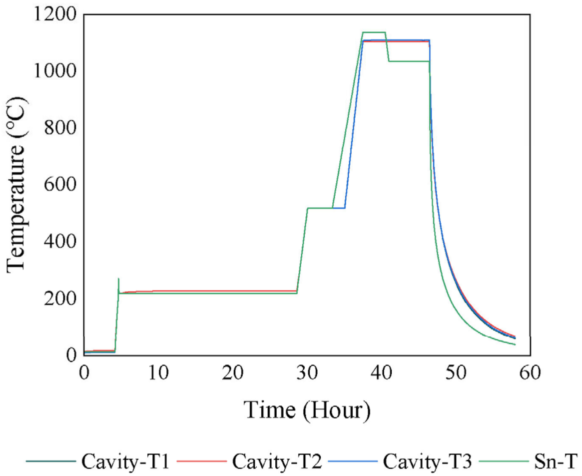
Cavity RF performance by Coating Process (1)

The Q_0 of this Nb_3Sn SRF cavity at low field reaches 8.8×10^9, degrading to 3.1×10^9 at the quench field of $E_{acc,max}$=16.11 MV/m.

- ☐ **The Sn source temperature of 1310^0C is rather excessive for our deposition system**

Coating process optimization

➢ *Coating Process (2)*



Temperature profiles of coating process (2)

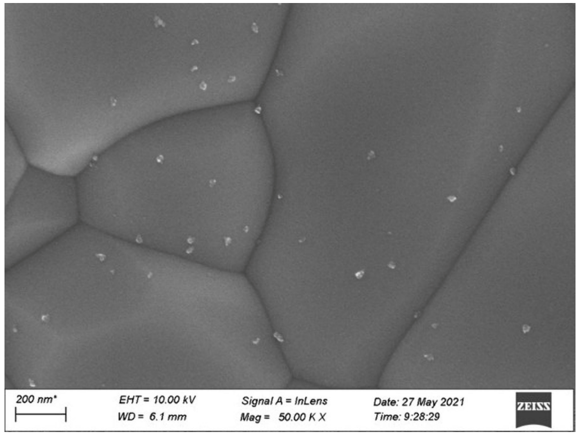
The Sn source temperature was lowered to 1200^{0}C to decrease the Sn vapor pressure and the Sn evaporation rate. This is to reduce or avoid the formation of Sn droplets and spots from the residual Sn vapor.

Coating conditions of process (2)

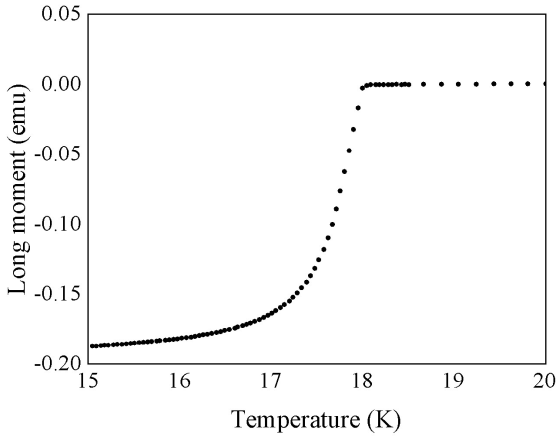
Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor			
		Sn power	Valves	Mo belt	Ceramics
1200	180	On	Closed	On	Without

Coating process optimization

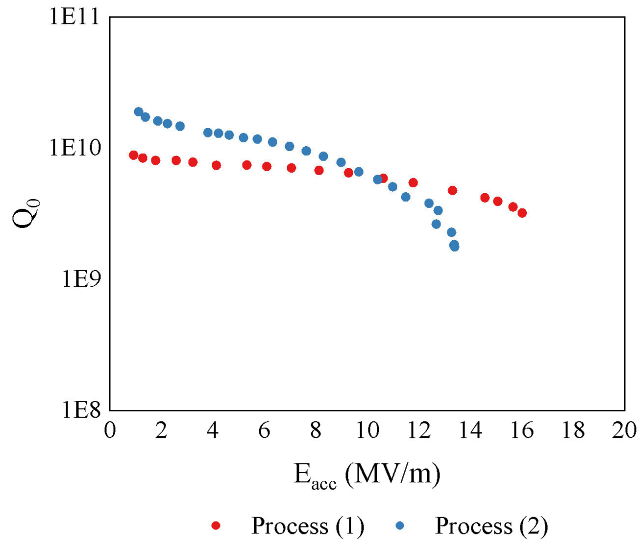
➢ *Coating Process (2)*



Nanometer-scale Sn droplets still exist on the surface of the Nb_3Sn film coated this time



M-T measurements: T_c>18K



Cavity RF performance by Coating Process (2)

EDS composition of the Nb_3Sn films by Coating Process (2)

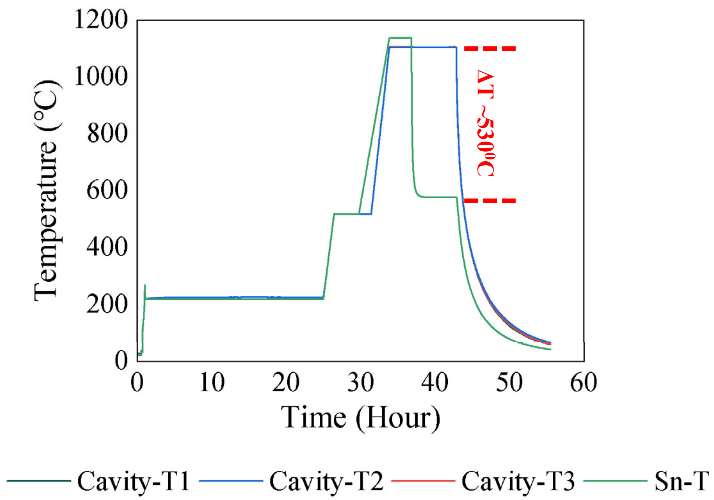
Position	1	2	3
Sn content(at. %)	26.46	26.32	26.31
Average Sn content	26.36		

The Q_0 of the Nb_3Sn SRF cavity by Process (2) reaches 1.9×10^{10} at 4.2 K in the low field region. The cavity quenches at $E_{acc,max}$=13.39 MV/m while the corresponding Q_0 drops to 2×10^9.

- ☐ **Despite the reduced gradient, the nanometer-scale Sn droplets still indicates tin vapor is in excess?**

Coating process optimization

➢ *Coating Process (3)*



Temperature profiles of coating process (3): the temperature of the Sn source heater drops rapidly to about 570^{0}C after the film growth stage

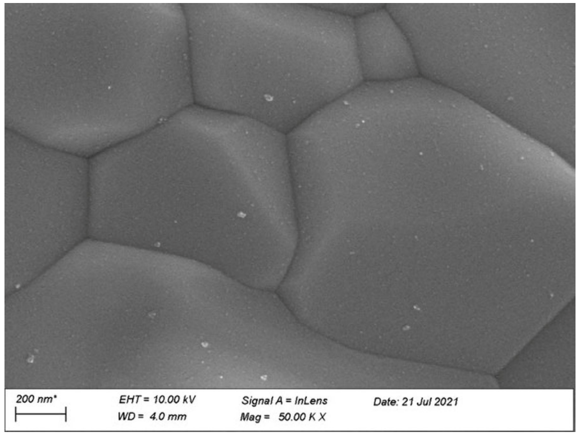
The conditions of the Coating Process (3) at the film growth stage were the same as those of the Coating Process (2). However, the Sn source of the Coating Process (3) was powered off immediately after the film growth stage to prevent additional Sn evaporation in the annealing stage. It is also expected that the lower temperature Sn source region adsorbs some Sn vapor during the annealing stage, hence reducing the Sn droplet density during the cooling stage.

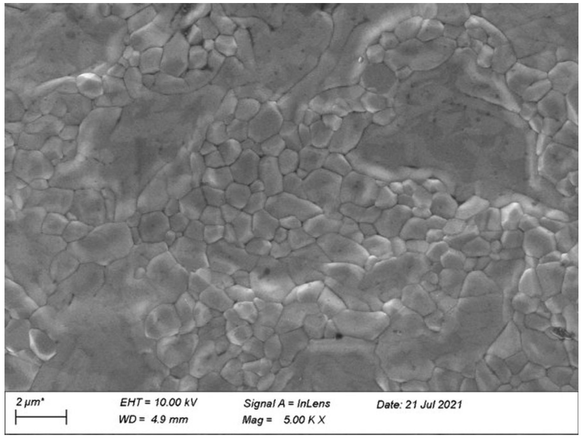
Coating conditions of process (3)

Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor			
		Sn power	Valves	Mo belt	Ceramics
1200	180	Off	Closed	On	Without

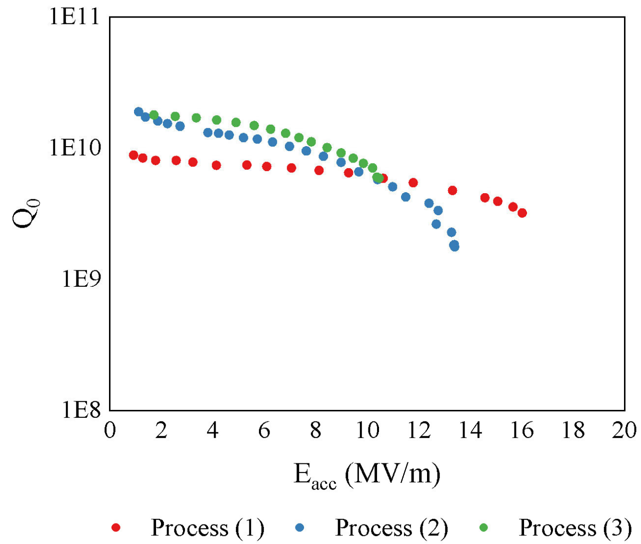
Coating process optimization

➢ *Coating Process (3)*





Nanometer-scale Sn droplets with **significantly reduced** density (left) and patchy areas (right) on the Nb3Sn film surface coated by the Coating Process (3). This confirms the **effectiveness** of the **adsorption** in reducing the formation of Sn droplets



Cavity RF performance by Coating Process (3)

EDS composition of the Nb_3Sn films by Coating Process (3)

Position	1	2	3
Sn content(at. %)	26.23	26.50	25.96
Average Sn content	26.23		

The Q_0 of the Nb_3Sn SRF cavity by Process (3) reaches 1.8×10^{10} at 4.2 K in the low field region. The cavity quenches at $E_{acc,max}$ = 10.46 MV/m because of the patchy areas found on the surface.

- ☐ Q-slope of process 3 is more gentle than process 2 at E_{acc} < ~5MV/m, but at E_{acc}>5MV/m Q-slope of process 2 becomes more precipitous and the Q_0 drops to the same level of process 3.
- ☐ Nanometer-scale Sn droplets mainly affect the RF performance in the low-field region. patchy areas have a more pronounced impact on the RF performance in the higher-field region, leading to a precipitous Q-slope and early quench.

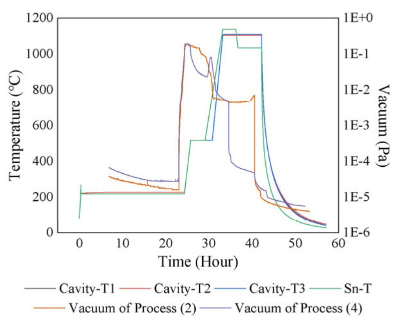
Coating process optimization

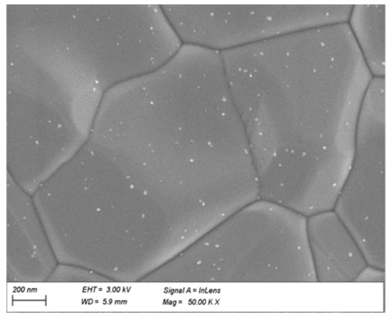
➢ Adsorption effect confirmation : *Coating Process (4),(5) and (6)*

Coating conditions of process (4), (5) and (6)

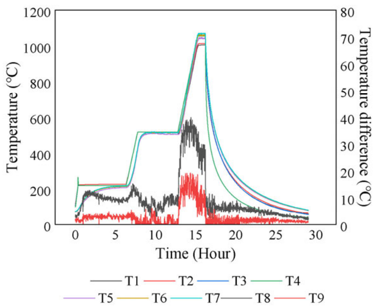
Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor*			
		Sn power	Valves	Mo belt	Ceramics
1200	180	On	Open	On	Without
		On	Closed	Bottom On	Without
		On	Closed	On	With

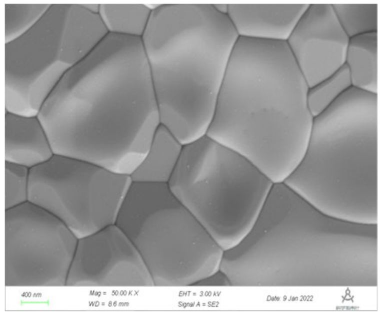
*The Sn source temperature and growth stage time of coating process (4), (5), (6) are the same as that of process (2)



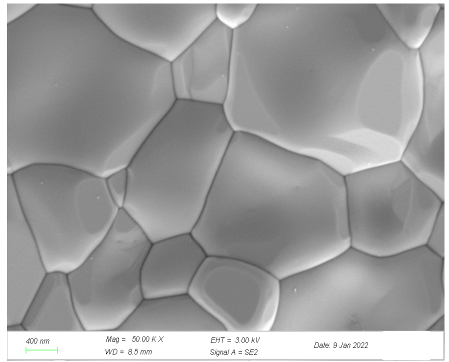


Process (4): Attempt at vacuum pumping by opening the valves between the coating chamber and the vacuum pump immediately after the film growth stage to pump away the residual Sn vapor(×)





Process (5): Manufacture of cold trap of ~27^{0}C by keeping the middle and upper Mo heating belts powered off all the process (√)

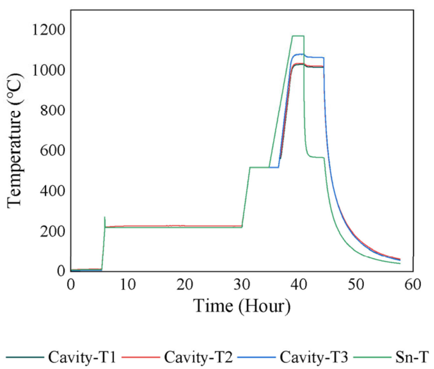


Process (6): High-purity ceramic bricks are suspended above the cavity to increase the adsorption of residual Sn vapor (√)

- ◻ By creating lower-temperature cold traps and suspending high-purity ceramic bricks, the adsorption of Sn vapor can be **effectively** enhanced to **prevent** the condensation of Sn droplets on the film surface.

Coating process optimization

➢ *Coating Process (7)*



Recipe of the Coating Process (7) with the Nb cavity at 1100^{0}C and the Sn source at 1233^{0}C

Optimization idea: under the premise of effectively inhibiting the condensation of Sn droplets, the Sn source temperature can be safely increased.

Targeted modifications to the Coating conditions:

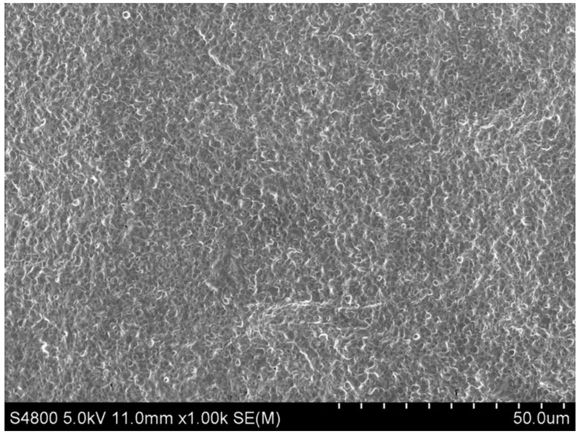
1. During the film growth stage, the temperature of the Sn source was kept at 1233^{0}C, but the duration was shortened to 2 hours.
2. Keeping the bottom Mo heating belts powered on while turning off the middle and upper Mo heating belts during the whole coating process.
3. The Sn source heater was turned off immediately after the film growth stage.
4. Two pieces of high-purity ceramic bricks were suspended above the cavity to further increase the adsorption of residual Sn vapor.

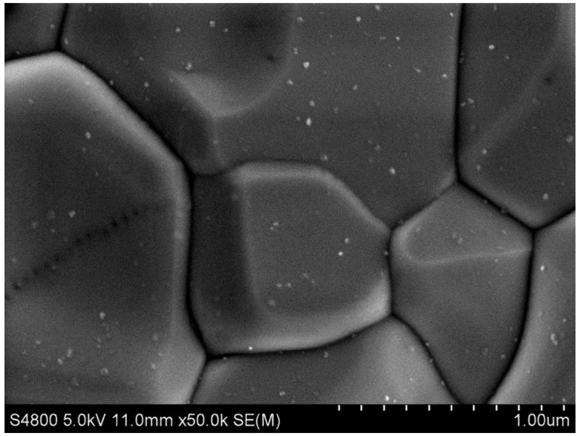
Coating conditions of process (7)

Sn source Temperature/℃	Growth stage time / Minute	Exploration of methods for adsorbing residual Sn vapor			
		Sn power	Valves	Mo belt	Ceramics
1233	120	Off	Closed	Bottom On	With

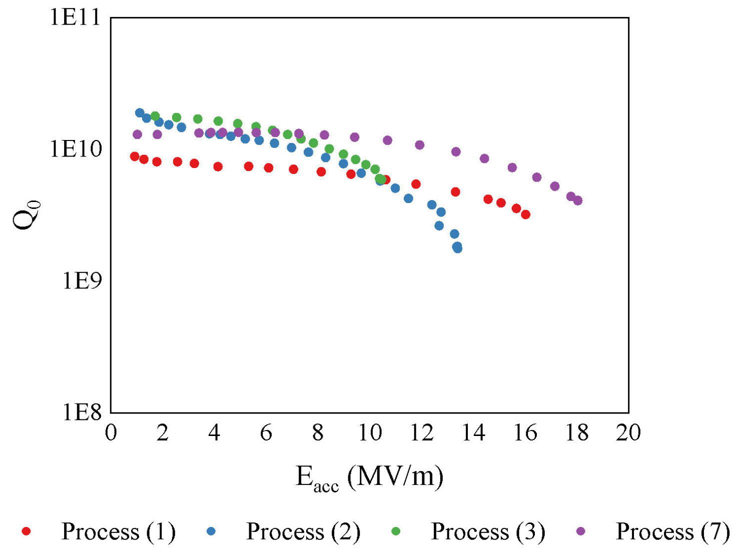
Coating process optimization

➢ *Coating Process (7)*





SEM images of the Nb_3Sn film coated by the Coating Process (7): no patchy areas are found in the 125 μm by 125 μm area (left) and nanometer-scale Sn droplets on the surface of the Nb_3Sn film (right)



EDS composition of the Nb_3Sn films by Coating Process (7)

Position	1	2	3
Sn content(at. %)	25.60	25.86	25.75
Average Sn content		25.74	

The Q_0 of the Nb_3Sn SRF cavity by Process (7) at 4.2 K in the low field region is about 1.4×10^{10} and the $E_{acc,max}$ reaches 18.03 MV/m. Moreover, despite the condensed Sn droplets, the Q-slope is relieved with the Q_0 larger than 1×10^{10} at E_{acc} = 12 MV/m and 4.2 K.

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Film characterization and RF performance comparison

Coating process	Sn droplets	Sn spots	Patchy aera	Q_0 at low field	$E_{acc,max}$	Onset of precipitous Q-slope
1	Yes	Yes	No	8.8E9	16.11MV/m	~12MV/m
2	Yes	No	Doubtful	1.9E10	13.39MV/m	~7MV/m
3	Yes	No	Yes	1.8E10	10.46MV/m	~5MV/m
4	Yes	No	Doubtful	No test	No test	No test
5	No	No	Doubtful	No test	No test	No test
6	No	No	Doubtful	No test	No test	No test
7	Yes	No	No	1.4E10	18.03MV/m	~12MV/m

- **The composition and causes of nanometer-scale Sn droplets, millimeter-scale Sn spots, and locally extremely thin patchy areas on the surface of Nb_3Sn films were confirmed.**
- **The impact of nanometer-scale Sn droplets, millimeter-scale Sn spots, and patchy areas on the RF performance of Nb_3Sn SRF cavities, such as Q_0, $E_{acc,max}$, and Q-slope, were also distinguished and clarified.**
- **The method of achieving high-quality Nb_3Sn films by increasing the adsorption of residual Sn vapor to suppress the generation of nanometer-scale Sn droplets was explored under the premise of ensuring uniform film growth by increasing the Sn vapor flux.**

Details in *Applied Surface Science 643 (2024) 158708 : https://doi.org/10.1016/j.apsusc.2023.158708*

Thanks for your attention.
Welcome worldwide cooperation!