Construction of Nb_3Sn coating system and trial of sample coating at KEK

2020 Nov/11 Nb3Sn SRF'20

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Outline

- Motivation of Nb_3Sn R&D at KEK
- Design and simulation of the coating system
- Construction of the coating system
- Sample coating result at KEK
- Discussion (coating problems)
- Summary and outlook

Nb_3Sn R&D at KEK

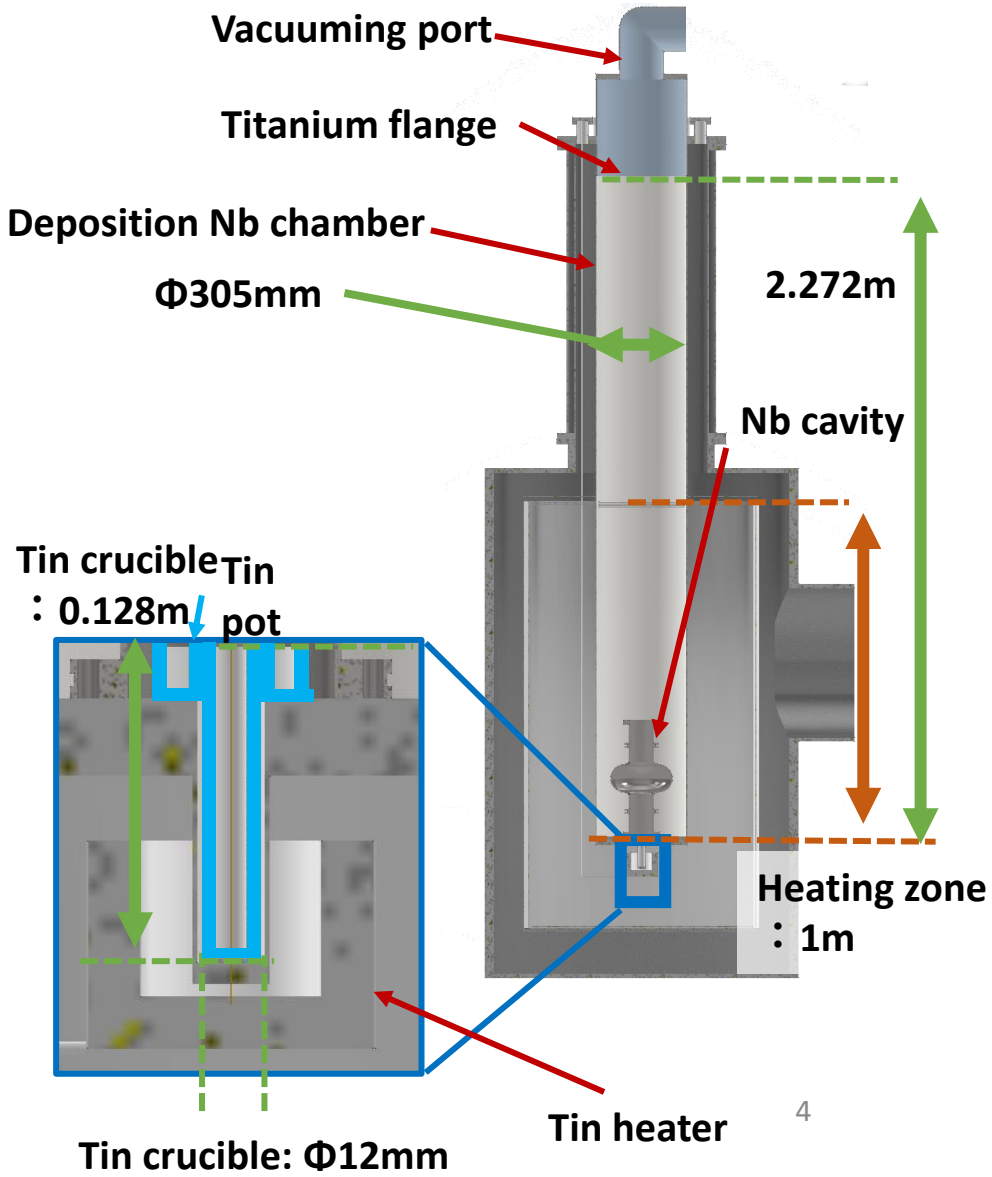
Nb_3Sn vapor diffusion R&D for High-Q has just started at KEK

- Motivation
  - Development of High-Q Nb_3Sn cavity
  - Compact SRF accelerator with cryocooler

- Vapor diffusion system (Furnace, Nb coating chamber, vacuum pump and etc.) was constructed.
- Coating tests were carried out.
- Characteristics of several coating samples were observed.
- Nb cavity for coating was prepared.

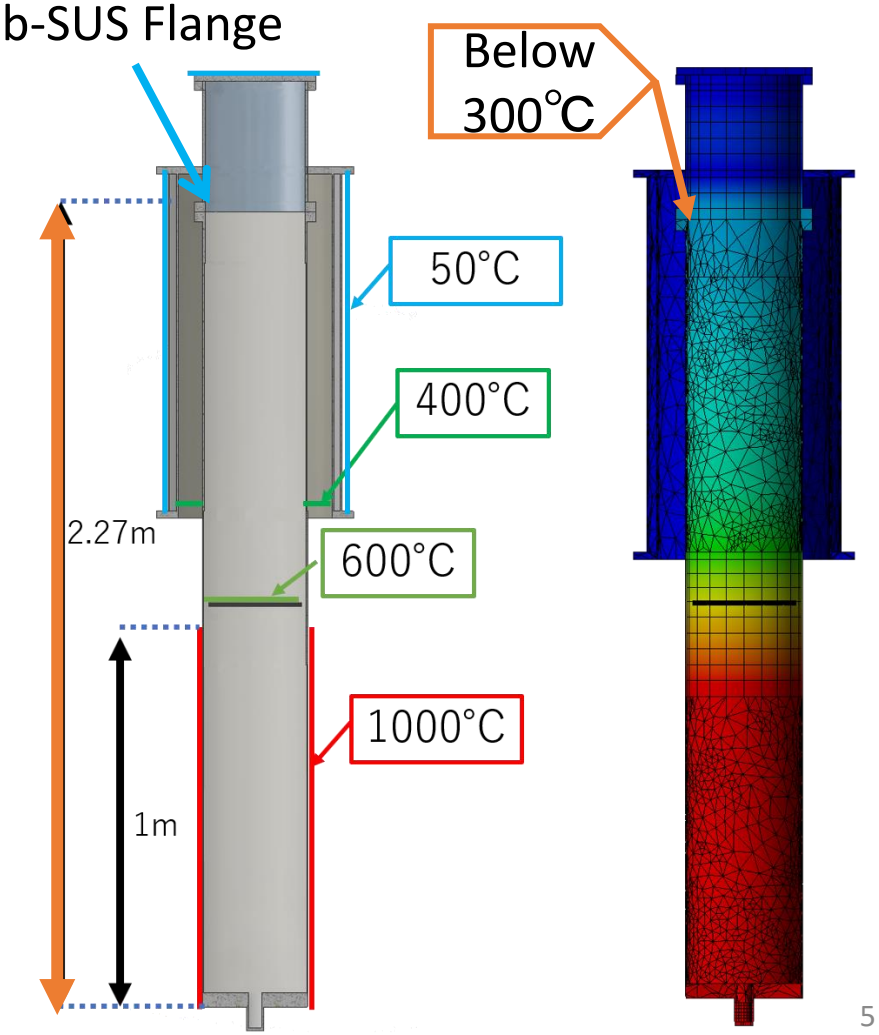
Design of KEK coating system

- Furnace temperature is 1100℃ and Tin heater temperature is above 1300℃ in coating process.
- Nb chamber vacuum and furnace vacuum are isolated.
  - Prevent contamination
- Nb chamber heatproof temperature is 1400℃.
  - Titanium flange was welded with TIG welding.
  - The chamber connects to SUS vacuum port.
    - Cu gasket is used for sealing.
    - Titanium flange temperature should be below 300℃
- Tin heater is made of Mo, and rated power is 1kW
  - Maximum temperature ＞ 1400℃
- Vacuuming system: **Cryo pump** +Dry pump
  - Vacuum pressure ＜ 1×10^{-5}Pa at 1100℃ (furnace)
- Furnace and Tin heater are independently controlled.



Thermal simulation of Nb chamber

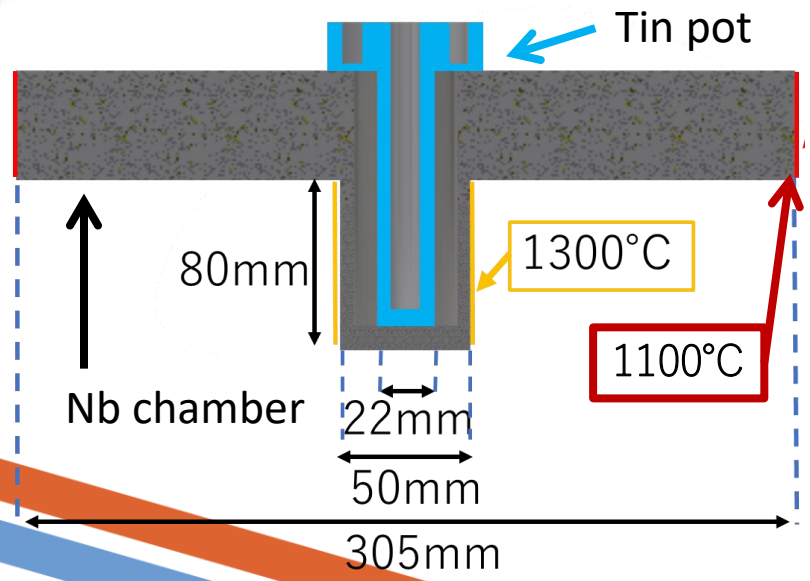
- The Nb chamber Flange temperature was simulated with ANSYS.
  - Flange between Nb-SUS : Operating temperature limit is below 300℃
  - Heating part：1000℃ or higher
  - Simplification model was used.
  - Heat transfer : Heat conduction and radiation
  - Confirmed the flange temperature was below 300℃.



Thermal simulation of Tin pot

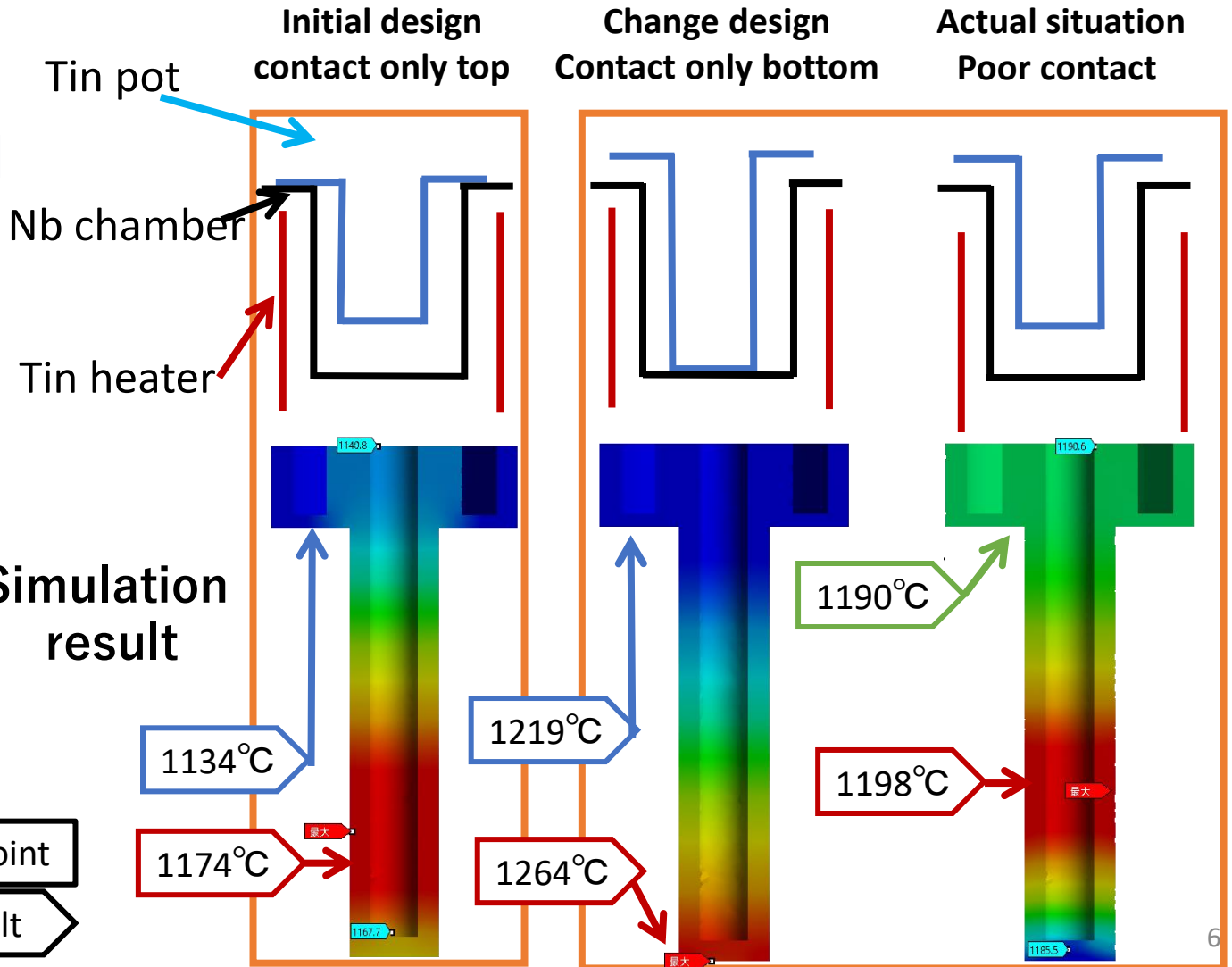
- Temperature of Tin pot is important for uniform coating
  - Dependent on contact condition at the bottom of Tin pot
  - The pot shape was slightly modified : a little longer

Simulation model

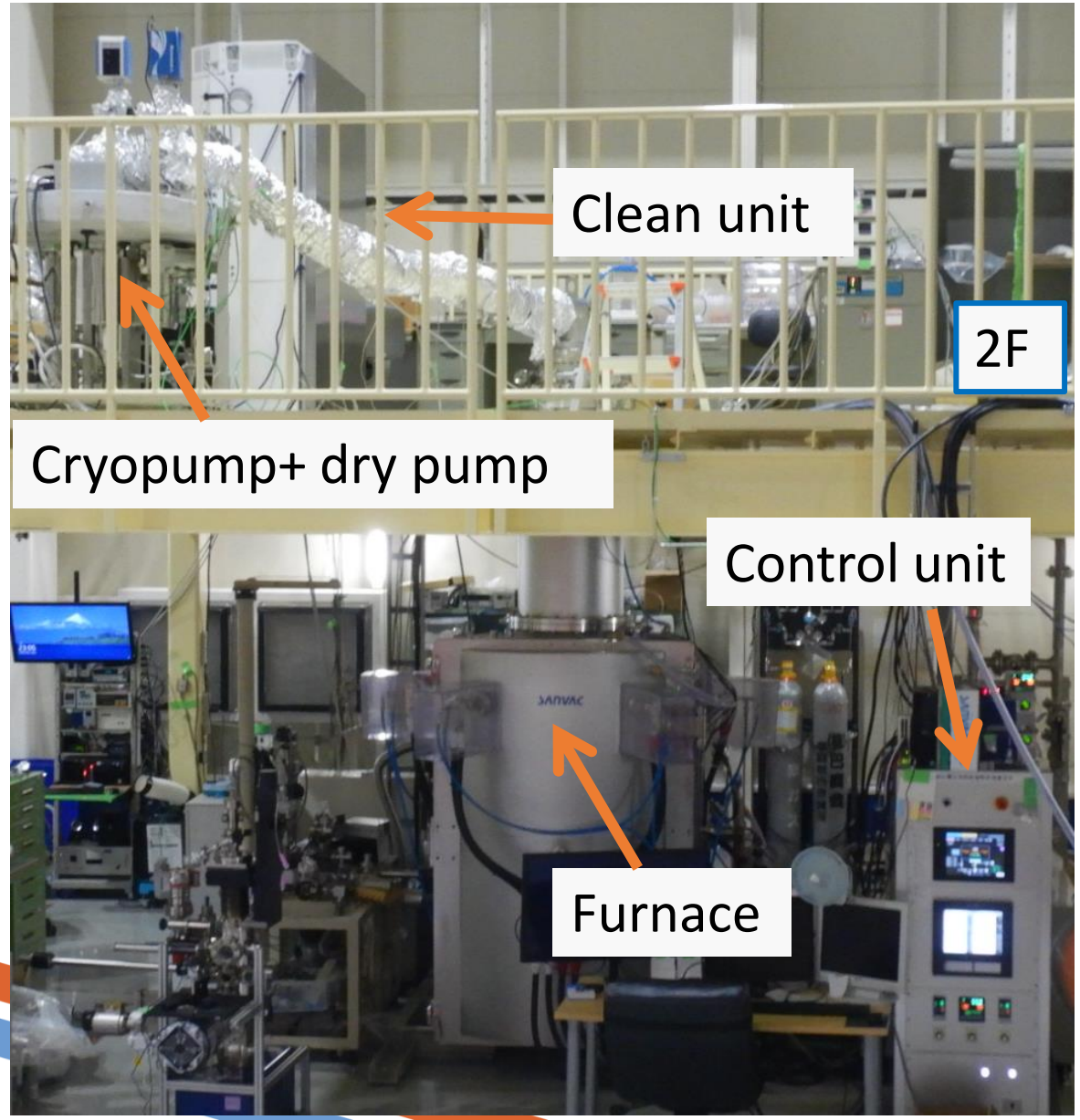


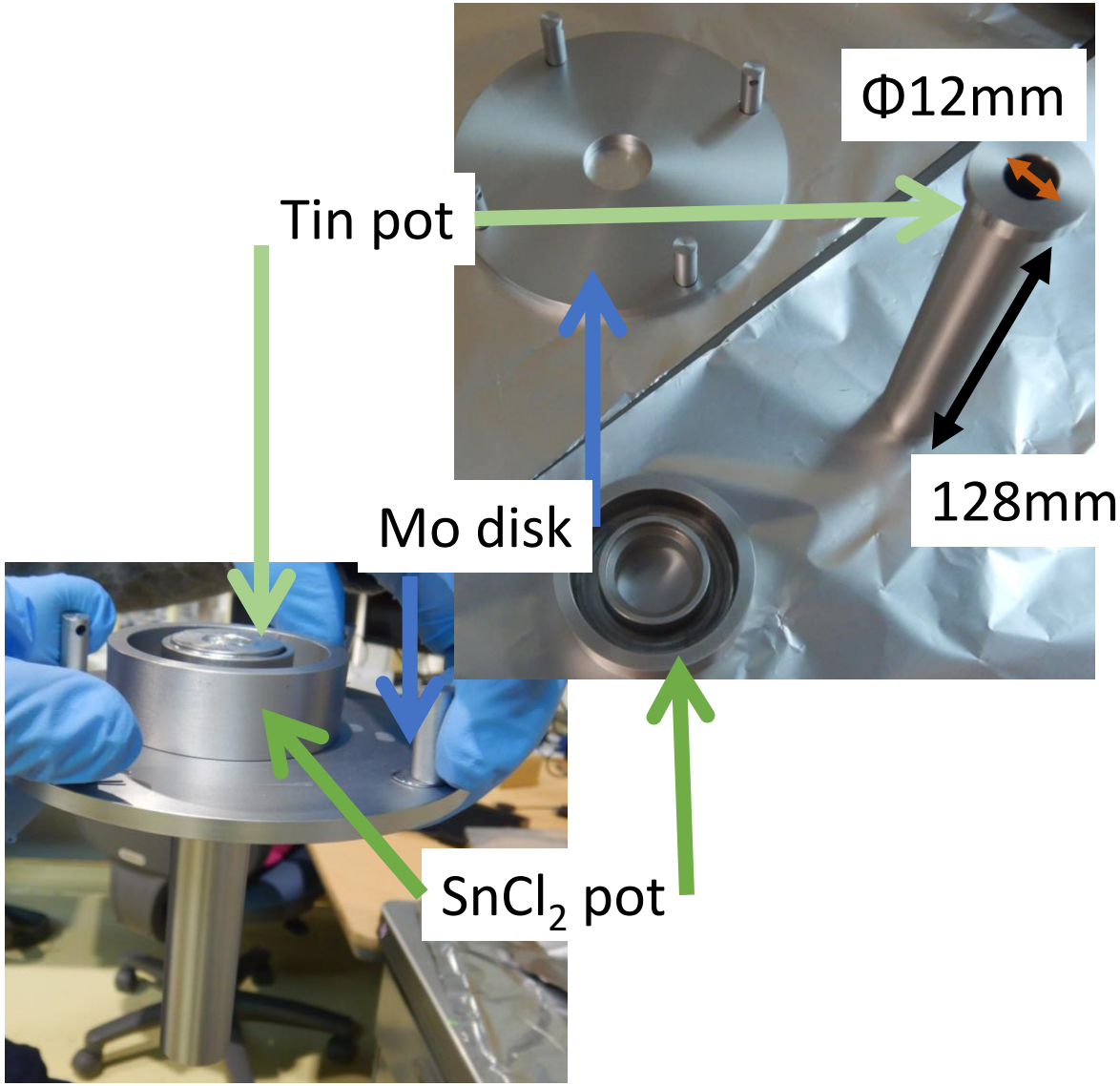
Thermal fixed point

Simulation result



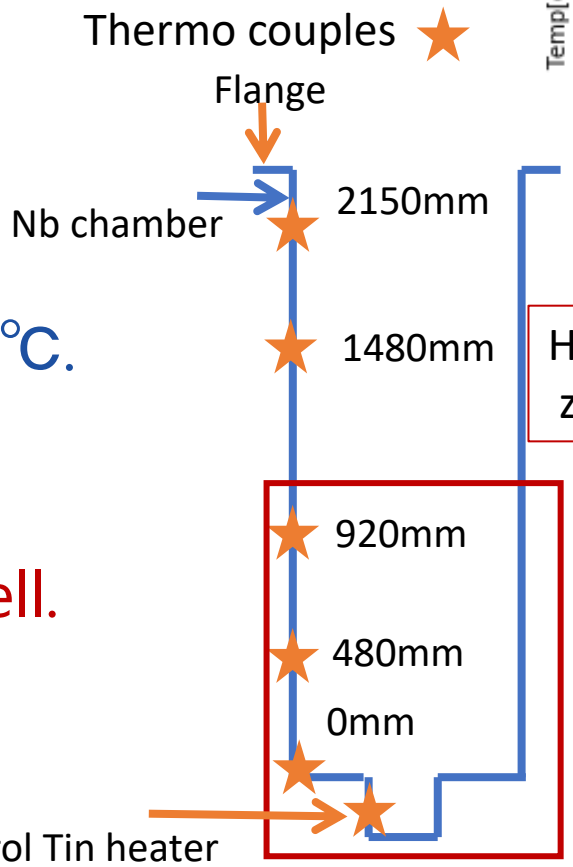
Coating system at KEK

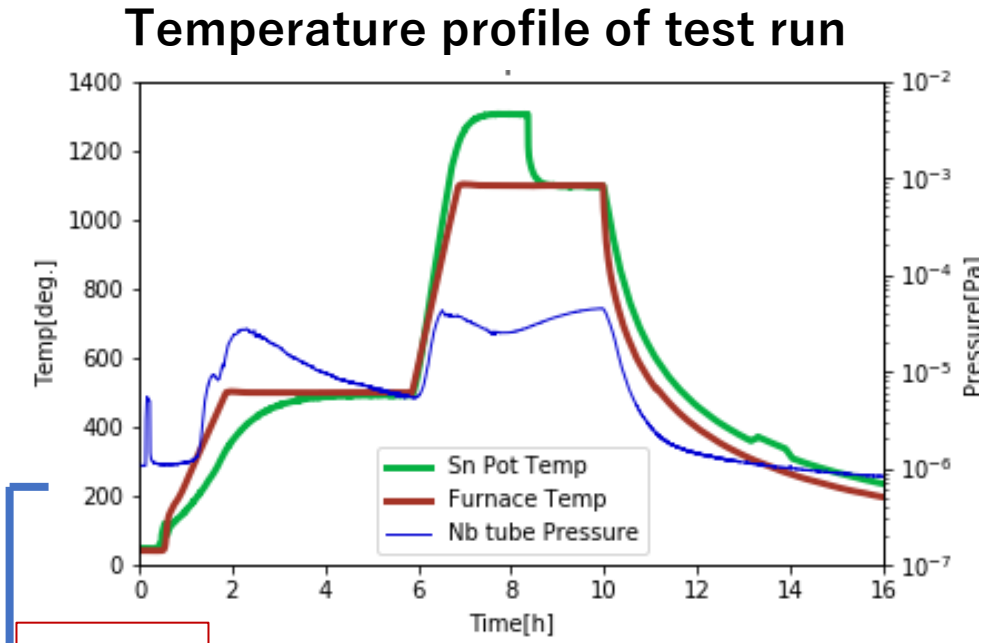




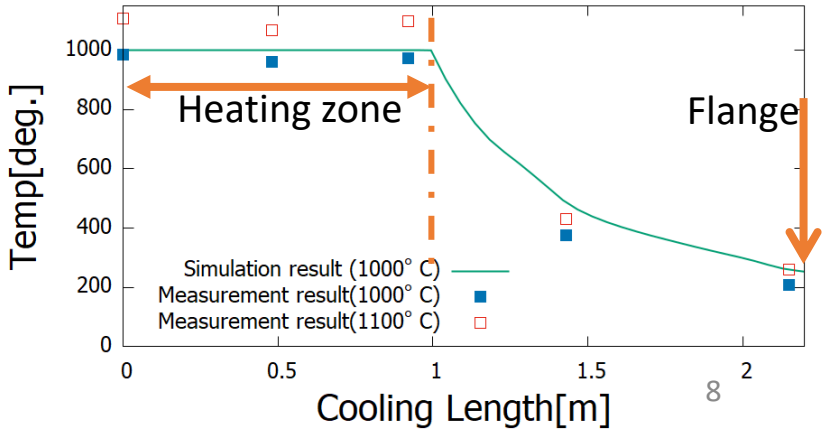
Furnace test run

- Coating system was tested after construction.
- In the test run, temperature distribution of coating system was confirmed.
  - Furnace : 1100℃
  - Tin heater : 1300℃
- Flange temperature was below 300℃.
  - Heating zone 1100℃ :
    Flange temperature < 259℃
- The entire coating system works well.



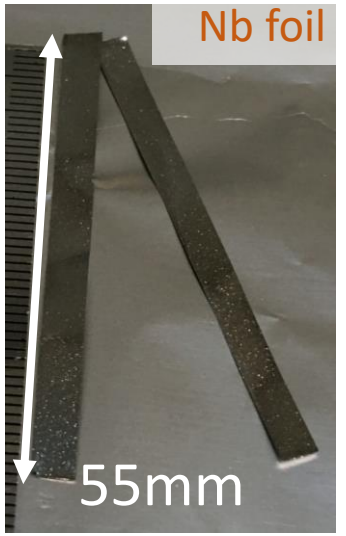


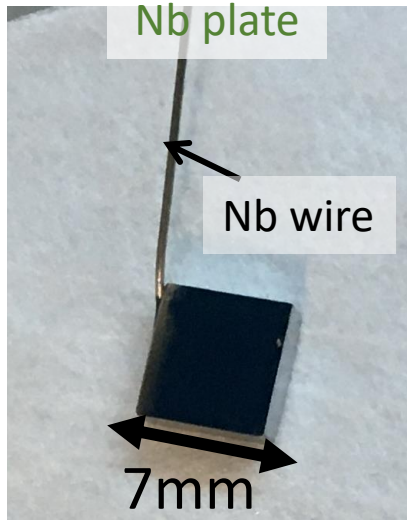
Nb chamber cooling part



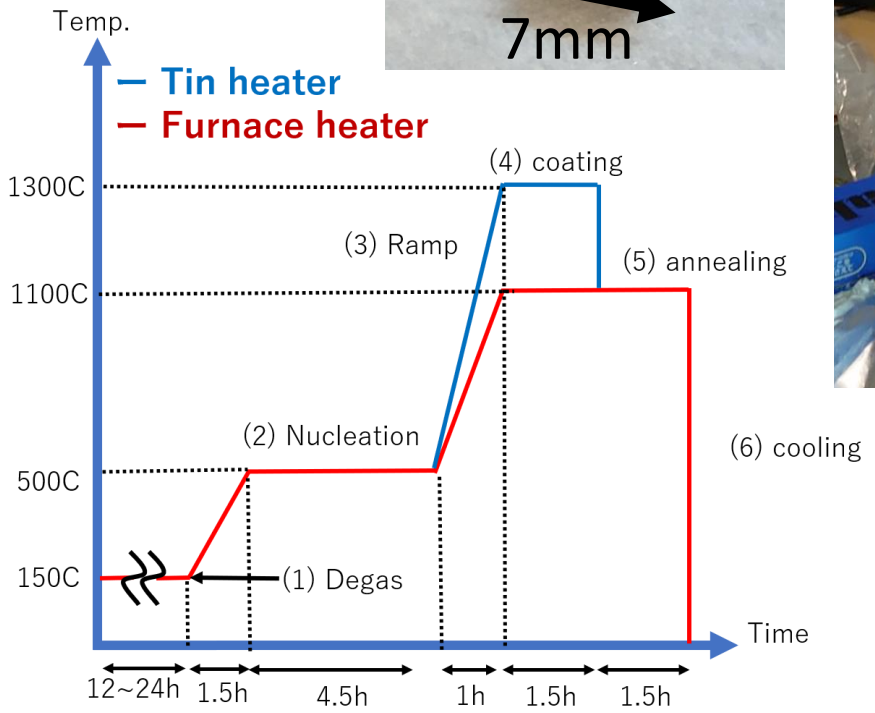
Sample coating

- Two types of sample are coated
  - Nb foil : RRR~30, 5 × 55 × 0.07 [mm^3], CP
    - Used for Tc measurement and cross section observation
  - Nb plate : RRR~300, 7 × 7 × 3 [mm^3], mirror polish
    - Used for surface observation
- Sample holder : made of Nb
  - Samples were hanged with Nb wire.
- Typical sample coating parameter
  - Nucleation : 500°C 4.5h
  - Coating : 1300°C~1500°C 1.5h (Tin heater)
    1100°C (Furnace)
  - Annealing : 1100°C 1.5h
  - Nb chamber is evacuated during entire coating process.





Sample holder with Tin pot



Sample measurement

	Model name	Observation parameter
SEM / EDS	HITACHI 3030	Composition ratio of tin
SEM	Hitachi SU3500	Surface and cross section
MPMS	Quantum Design MPMS-7	Hc2, Tc measurement
Laser microscopy	Keyence VK-X3000	Surface observation and surface roughness measurement

SEM / EDS (TM3030)

SEM (SU3500)

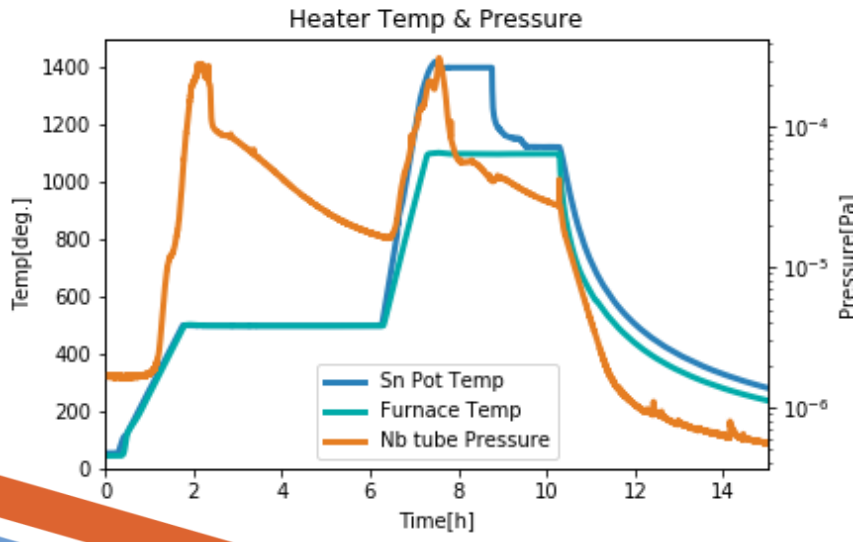
MPMS (MPMS-7)

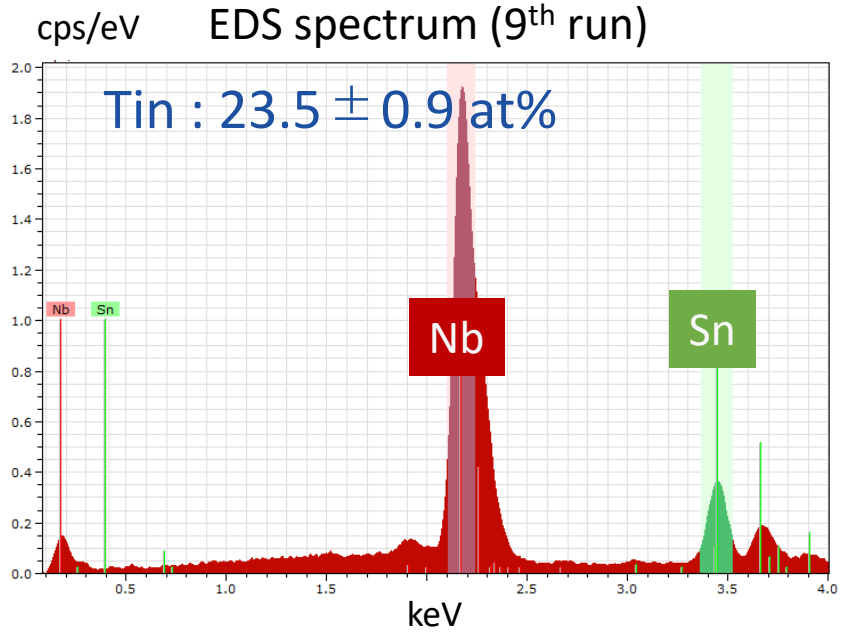
Laser microscopy (VK-X3000)

Typical coating result at KEK

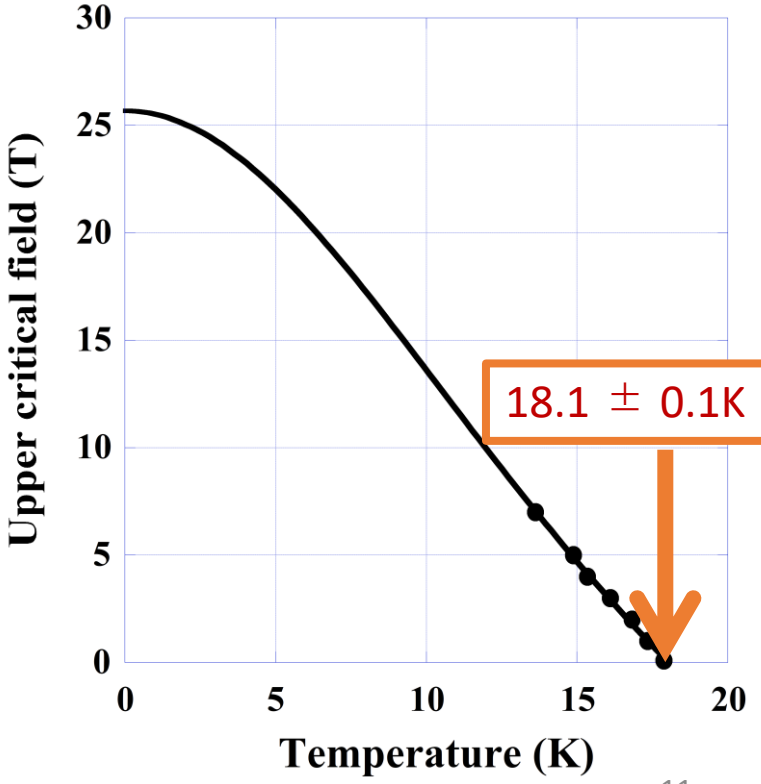
- Surface composition ratio of Tin is 23.5 ± 0.9 at%.
- Tc ~ 18 K

Coating parameter(9th run)
Nucleation : 500℃ 4.5h
Coating : 1400℃ 1.5h
Annealing : 1100℃ 1.5h





Temperature vs
Critical magnetic field (9th run)

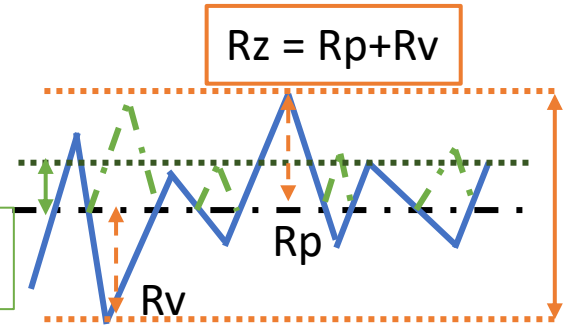


Typical coating result at KEK

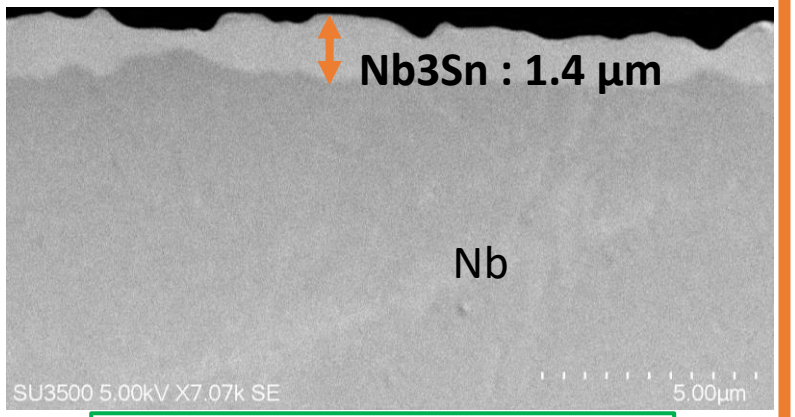
Coating parameter(3rd run)
Nucleation : 500℃ 4.5h
Coating : 1300℃ 6.5h Annealing : 1100℃ 1.0h

- Thickness is around 1.4 μm.
  - Run 9th is not observed.
- Surface roughness
  - Ra : 0.11 μm
  - Rz : 0.72 μm

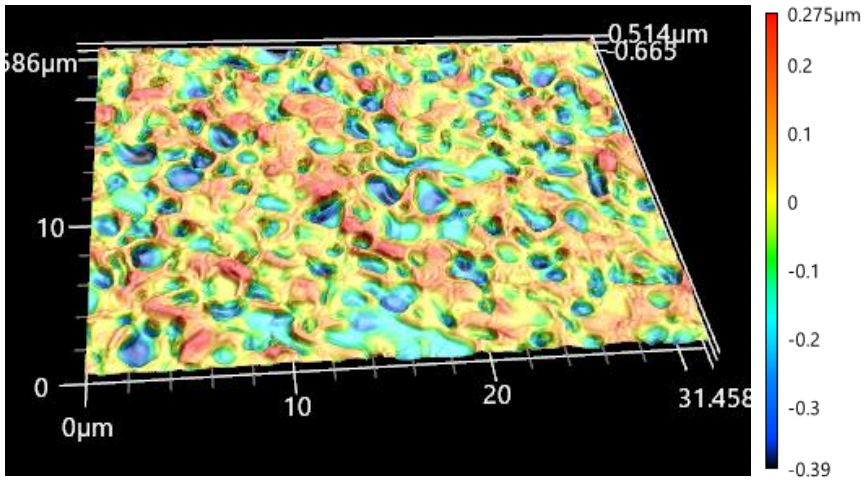
$$Ra = \frac{1}{N}\sum_{n-1}^{N} |Zn|$$



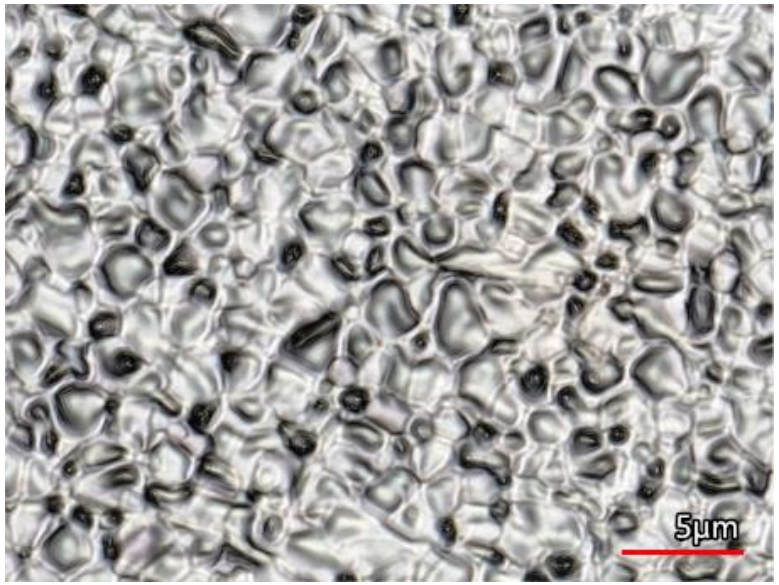
Cross section SEM image (3rd run)



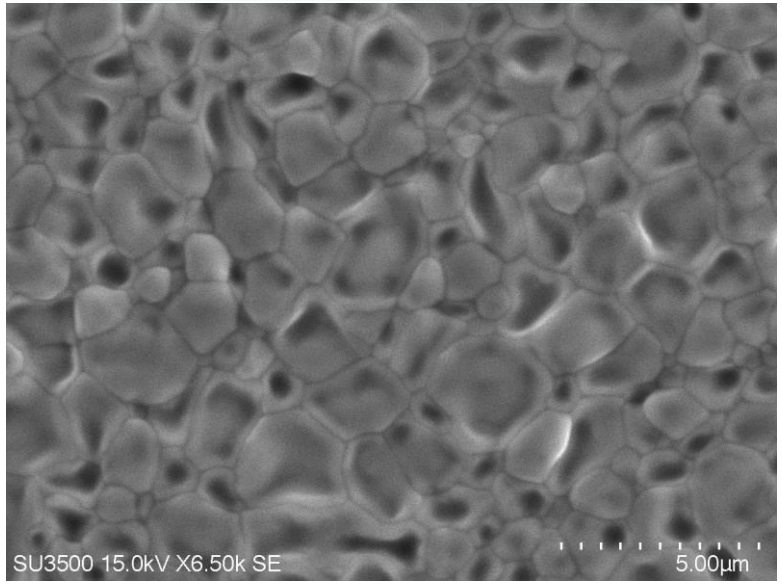
Surface 3D image (9th run)



Surface photomicrograph (9th run)

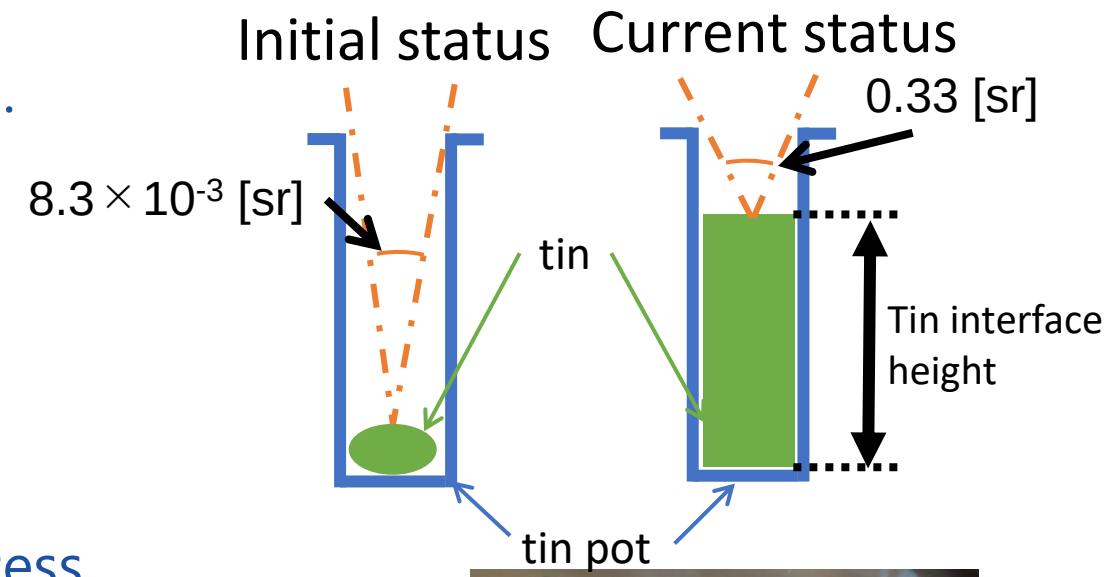


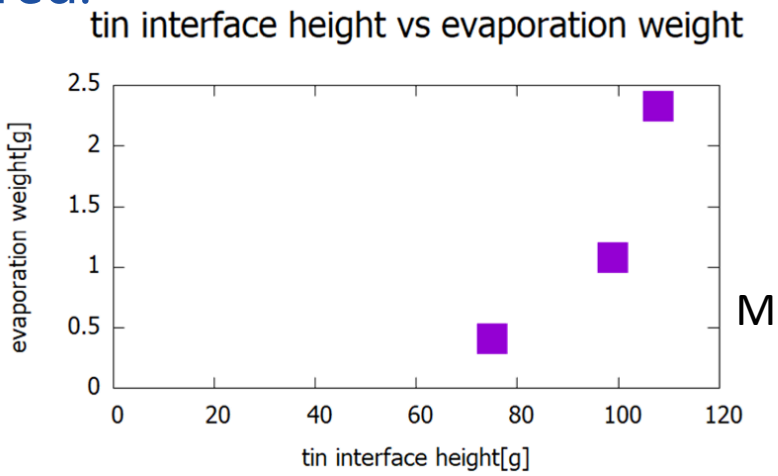
Surface SEM image (9th run)

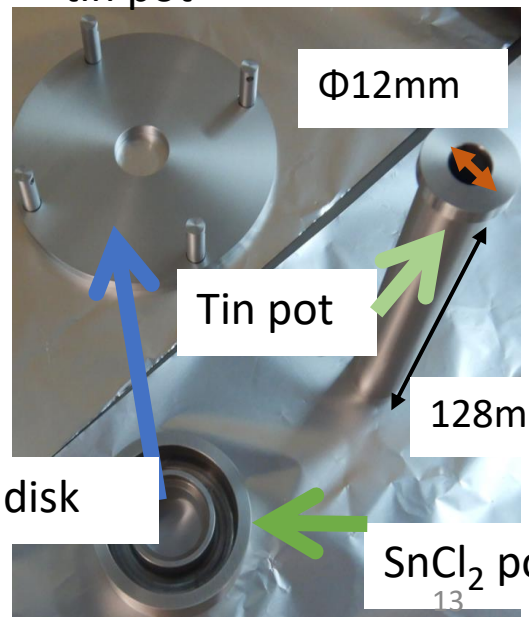


Discussion 1 : Evaporated weight

- Initial: Evaporated weight is less than expected.
  - 1st Run : 0.17g for a coating time of 1.5h
- Currently: Increased amount of tin to improve solid angle of evaporation.
  - Maximum evaporated weight : 2.33g
- But the evaporated weight is uncontrollable.
  - Continue to evaporate during coating process
  - Difficult to optimize.
- New design tin pot will be prepared.

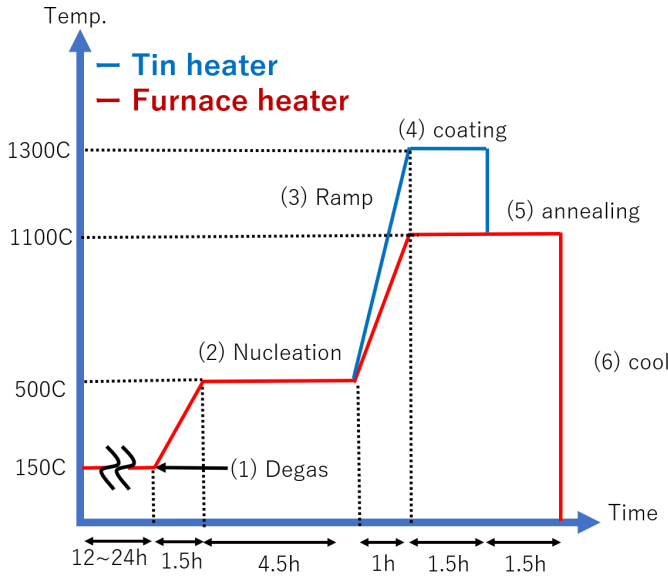




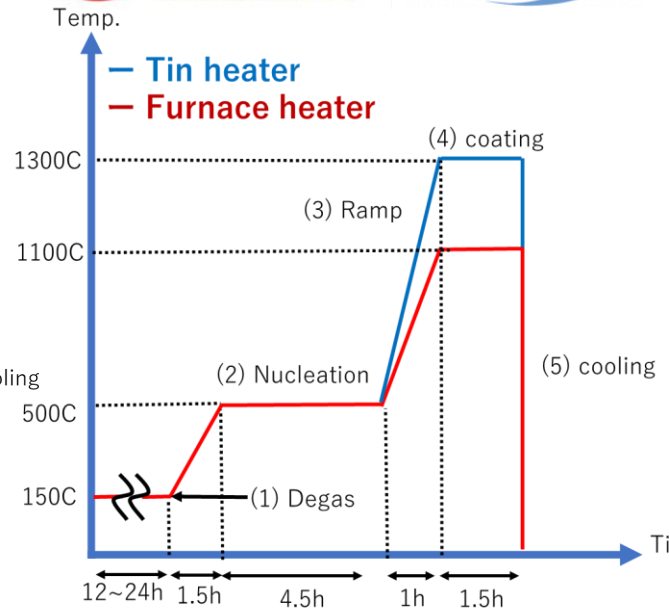


Discussion 2 : Voids

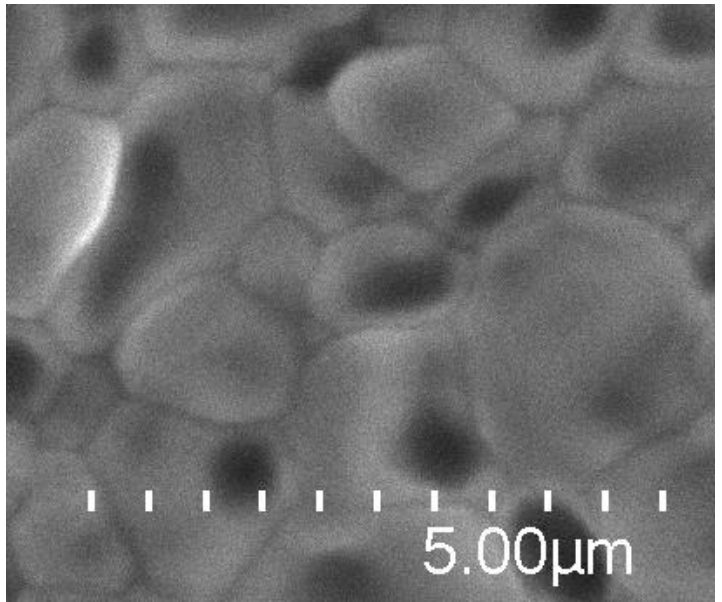
- Voids on coated surfaces were created with typical coating parameter.
- Trying without annealing.
  - Coating : 1.5h.
- Tc is almost the same.
  - Typical coating : 17.96 ± 0.23K
  - Without annealing : 18.05 ± 0.06K
- Composition ratio of tin of both samples is around 24 at%.
- How to make a good surface (without voids)?
- Do voids affect the RF performance?

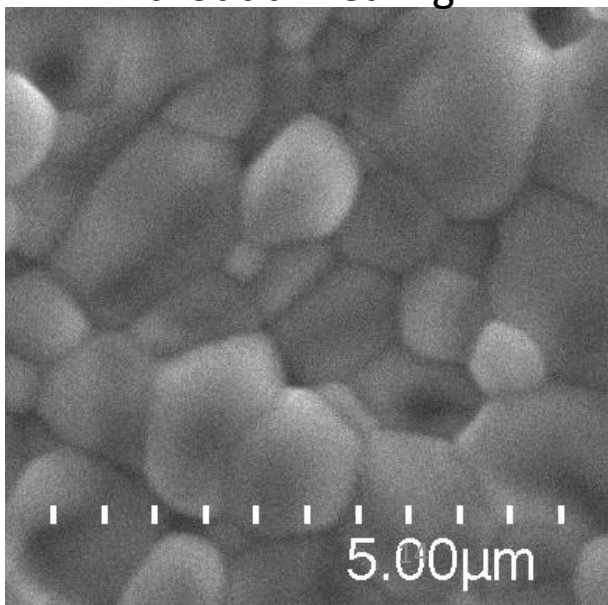


Typical coating surface



Without annealing



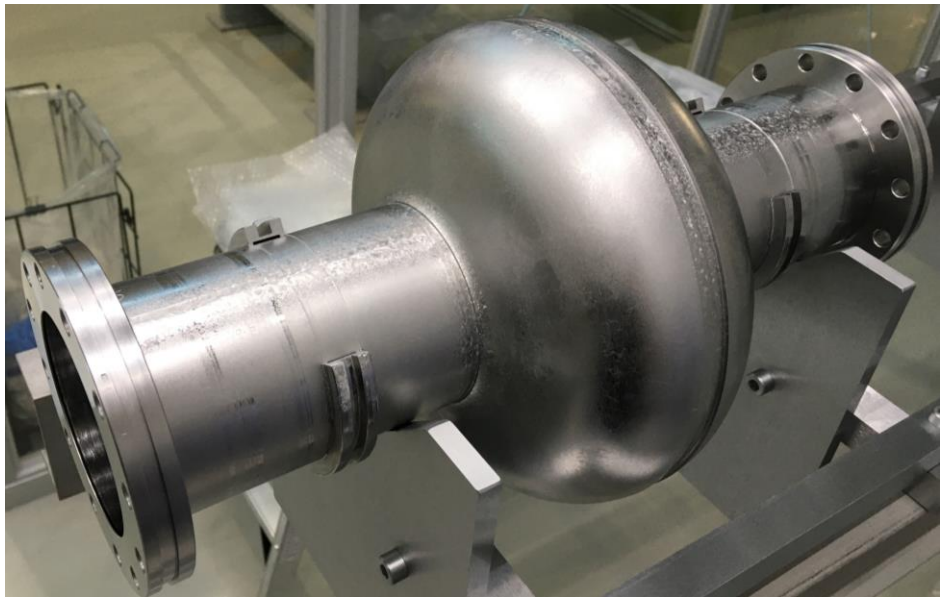


Summary

- A vapor diffusion system was constructed at KEK.
  - Entire coating system works well.
- Typical coating result is:
  - Tc : around 18K
  - Composition ratio of tin : around 24 at%
- Nb_3Sn was successfully coated on Nb samples.
- Discussion topics:
  - Evaporated weight
  - Voids on surface

Outlook

- Cavity vertical test
  - Nb3Sn coating of single cell cavity at the end of November
  - VT in the beginning of December
- Furnace environment
  - Build clean booth to improve clean coating environment
- Optimize coating parameters by taking sample data for systematic evaluation
  - Cross section (Thickness)
  - Tc
  - Grain size
  - Surface roughness



Thank you for your attention !