

# **First Cavity Performance Test for Nb<sub>3</sub>Sn Superconducting Cavity by Vapor Diffusion Process at KEK**

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### Outline

- R&D motivation
- KEK coating system
- Sample coating
- Cavity coating
- Cavity performance test
- Summary, Future works

### Nb3Sn R&D at KEK



### Nb3Sn vapor diffusion R&D for High-Q has just started at KEK

- Motivation
  - Development of High-Q Nb3Sn cavity
  - Compact SRF accelerator with cryocooler
- Vapor diffusion system (Furnace, Nb coating chamber, vacuum pump and etc. ) was construction.
- Coating tests were carried out.
- Characteristics of several coating samples were observed.
- A Nb cavity was coated with Nb3Sn then the performance was measured.

# Design of KEK coating system

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







# Sample coating

- Sample coating : Searching parameter for cavity
- Two types of sample are coated
  - Nb foil : RRR~30, 5 × 55 × 0.07 [mm<sup>3</sup>], CP
    - Used for Tc measurement and cross section observation
  - Nb plate : RRR~300, 7 × 7 × 3 [mm<sup>3</sup>], mirror polish
    - Used for surface observation
- Sample holder : made of niobium
- Sample coating parameter
  - Nucleation : 500°C 4.5 h
  - Coating : 1300°C~1500°C 1.5 h (Tin heater) 1050°C~1200°C (Furnace)
  - Annealing : 1100°C 1.5 h or 0 h
  - Nb chamber is evacuated during entire coating process.



### Sample coating result

- Sample was coated several parameters.
  - Coating temperature : 1050°C~1200°C (furnace)
  - Coating time : 1.5 h / 3.0 h
  - Annealing time : 1.5 h / 0 h
  - Nb foil on top of sample holder : Available/ Not available.
  - Nucleation condition and Tin pot temperature Nb was fixed. : Tc I
    - Nucleation :500°C 4.5h
    - Tin pot . : 1400°C
- Samples with annealing have voids on surface.
- Sample coating number 23 was chosen as cavity coating parameter.
  - Coating time/ Annealing time : 3 h/0 h
  - Tin at% is around 24 at%.
  - Tc ~ 18 K
  - There are no voids on surface.

Typical	Sampl	e coating	Result
		0	

Sample Coating Num.	4	9	11	20	21	22	23
Coating Temp[C]	1200	1100	1100	1100	1050	1150	1100
Coating Time[h]	1.5	1.5	1.5	3	3	3	3
Annealing Time[h]	1.5	1.5	0	0	0	0	0
Nb foil	—	_	—	$\checkmark$	$\checkmark$	$\checkmark$	-
Tc [K]	11.1 ±0.1	18.0 ±0.2	17.9 ±0.4	17.6 ±0.2	17.9 ±0.2	18.2 ±0.2	18.1 ±0.1
Thickness [μm]		1.7 ±0.2	1.3 ±0.3	2.7 ±0.4	2.9 ±0.3	3.5 ±1.0	1.5 ±0.3
Tin at%	20.7	25.3	23.6	23.7	24.3	24.3	24.0
Voids	$\checkmark$	$\checkmark$	_	_	_	_	_

With Void (No. 9)

. . . . . . . . . .

5 00um



# **Cavity Coating**

- The shape of coating cavity was a TESLA-like single cell.
- Nb3Sn coating process
  - Nucleation : 500°C 4.5 h
  - Coating : Furnace 1100°C 3 h Tin pot 1400°C 3 h
  - Annealing : 0 h
- Nb sample was hanged on cavity flange.
  - Nb plate
  - Nb foil



## **Cavity Coating Result**

### **Inside of the cavity**

- Before coating : metallic luster
- After coating : matte
  - Whole inside equator was matte.

### **Measurement result of witness sample**

- Grain size :  $1 \sim 3 \mu m$ .
- Thickness :  $1.7\pm0.4~\mu m.$
- Composition ratio of Tin :  $23.1 \pm 0.4at\%$
- Cavity seems to be successfully coated.

	Sample(23 <sup>rd</sup> )	Witness sample
Thickness [µm]	$1.5 \pm 0.3$	$1.7 \pm 0.4$
Tin at%	24.0	$23.1 \pm 0.4$



After Coating

#### Before Coating (EP2)





# Measurement setup (Flux, RF)

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- Flux expulsion when cavity became SC state was measured.
  - Flux expulsion in Fast/Slow cooling
  - External magnetic field : 0 mG,  $\pm$  50 mG
- Sensor position
  - Cernox : Top/ bottom iris, Equator
  - Flux gate : 0 deg. 120 deg. 240 deg.
    - 0 deg. : Ζ, θ, R axis
    - 120 deg. : Ζ, θ, R axis
    - 240 deg. : Ζ, θ axis
- Solenoid coil
- Bottom heater
- Cavity temperature gradient when cavity became SC (ΔT) : Top iris temp. – Bottom iris temp.



### Flux Measurement

- Each magnetic filed change occurred between 17.5 K and 18 K.
- ΔT : Deference top and bottom iris.
  - Values between superconducting transition.
- ΔFG : Deference NC FGs values and SC FGs values.

Cernox 1

Cernox 2

Cernox 3



#### R-9 VT17th FluxGate0.0 mG 0.3 K



## Flux Measurement

- Compared  $\Delta$ FGs when  $\Delta$ T ~ 0 K, 1 K and each mag. Field.
  - (): ΔT ~ 0 K
  - ◆:△T ~ 1 K
- mag. Field 0 mG : Effect of Thermal current
  - Several mG decreased.
- mag. Field ± 50 mG : Effect of thermal current + flux expulsion
  - Similar value as 0 mG
  - No flux expulsion was measured.
  - e.g.)Nb cavity : Z axis field was enhanced 1.56 ~1.7 times. [1][2]

### External magnetic field vs Magnetic field change (Z axis)



0 deg(FG1),  $\Delta T \sim 1 K$ 

120 deg(FG2), ΔT~1 K

240 deg(FG4), ΔT~1 K

[1] Kensei Umemori, TTC 2018
[2] S.Posen et.al., JOURNAL OF APPLIED PHYSICS 119, 213903 (2016)

### Flux measurement

- Compared  $\Delta$ FGs when  $\Delta$ T ~ 0 K, 1 K and each mag. Field.
  - (): ΔT ~ 0 K
  - ◆ : △T ~ 1 K
- mag. Field 0 mG : Effect of Thermal current
- mag. Field  $\pm$  50 mG : Effect of thermal current + flux expulsion
- $\Delta$ FG of 0 mG and  $\pm$  50 mG was similar in each axis.
  - Flux expulsion was not measured.
  - Effect of external magnetic field was small.



0.0

-2.5

-5.0

-7.5

-10.0

-12.5

-15.0

∆FG[mG]



Z axis

20

0 deg(FG1), ΔT~0 K 120 deg(FG2), ΔT~0 K

240 deg(FG4), ΔT~0 K

0 deg(FG1), ΔT~1 K

120 deg(FG2), ΔT~1 K

240 deg(FG4), ΔT~1 K

External magnetic field vs Magnetic field change (Z axis)

-20

## Discussion (Flux expulsion)

- No flux change from ext. mag. field was measured.
  - Nb cavity : Z axis field was enhanced 1.56 ~1.7 times. (Full expulsion)[1][2]
  - Nb3Sn cavity : ΔFG was not dependent on Ext. mag. field.
- Would behavior of ΔFG be different if outside CP was applied ?
  - Outside of the cavity was Nb3Sn. : Outside is also matte.
  - It was possible  $\Delta$ FG was from thermocurrent between outside Nb<sub>3</sub>Sn and Nb substrate.

Kensei Umemori, TTC 2018
S.Posen et.al., JOURNAL OF APPLIED PHYSICS 119, 213903 (2016)









- Cavity performance was measured in each Temperature with several temperature gradient (ΔT).
  - Measurement temperature : 1.5 K ~ 4.3 K
  - ΔT : 0.01 K, 0.2 K, 0.25 K, 1.0 K
- Qo at 4.2 K 1 MV/m,  $\Delta T = 0.01$  K : 3.9  $\times 10^9$
- Qo at 4.2 K 1 MV/m,  $\Delta T = 1.0$  K : 2.5  $\times 10^9$
- Maximum Eacc was around 11.5 MV/m in each measurement.



- Rs was decomposed into  $\mathrm{R}_{\mathrm{BCS}}$  and  $\mathrm{R}_{\mathrm{res}}$ .
- $R_{BCS}$  @ 4.2 K( $\Delta T$  =0.01 K) : 62 n $\Omega$  ~ 132 n $\Omega$
- $R_{res} (\Delta T = 0.01 \text{ K})$  :  $5 \text{ n}\Omega \sim 61 \text{ n}\Omega$





- Rs was decomposed into  $R_{BCS}$  and  $R_{res}$
- $R_{BCS}$  : Week dependent on  $\Delta T$
- $R_{res}$  : Strong dependent on  $\Delta T$





- Rs was decomposed into  $R_{BCS}$  and  $R_{res}$
- $R_{BCS}$  : Week dependent on  $\Delta T$
- $R_{res}$  : Strong dependent on  $\Delta T$ 
  - Rres increased between 40 n $\Omega$  ~ 65 n $\Omega$
  - Flux from thermal current was trapped





### Discussion (RF measurement)

### • Qo was lower than $1 \times 10^{10}$ at 4.2 K.

- R<sub>BCS at 4.2K</sub> was much higher than expected (e.g. compared to results at other labs).
  - $\Delta$  of the film was not good ?
  - Contamination of Carbon or Tin ?
- Rs increased when Eacc increased.
- ΔT dependent Rres was strong.
  - Rres increased depending on  $\Delta T$ .
  - Flux from thermal current was trapped.



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## Summary



- A vapor diffusion system was constructed at KEK.
  - Total coating system works well.
- Several samples were coated, and single cell cavity was coated.
- First Nb3Sn coating for cavity succeeded.
  - Tc of the cavity was between 17.5 K and 18 K.
  - Qo was  $3.9 \times 10^9$  at 1 MV/m and 4.2 K ( $\Delta T = 0.01$  K).
  - Maximum Eacc was 11.5 MV/m.
- Flux change was measured in 0 mG /  $\pm$  50 mG.
  - No flux expulsion was measured.
  - Flux change was dominant on  $\Delta T$ .
- R<sub>BCS</sub> was higher than expected.
- Rres has strong dependent on  $\Delta T$
- Rres also has strong dependent on Eacc

### Future work

- To realize high-Q of Nb3Sn cavity above  $1\times10^{10}$  at 4.2 K.
  - Furnace environment: We just built clean booth to improve clean coating environment.
  - Nb substrate: Try anodized and research other treatment before coating
  - Optimize coating parameter: Nucleation time/temp, coating time, etc.
  - Coupon cavity will be made and search the parameters.
- Details of flux change and effect of thermal current on Qo will be investigated.



Thank you for your attention !