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Successful results of N-infusion in collaboration with KEK/JAEA

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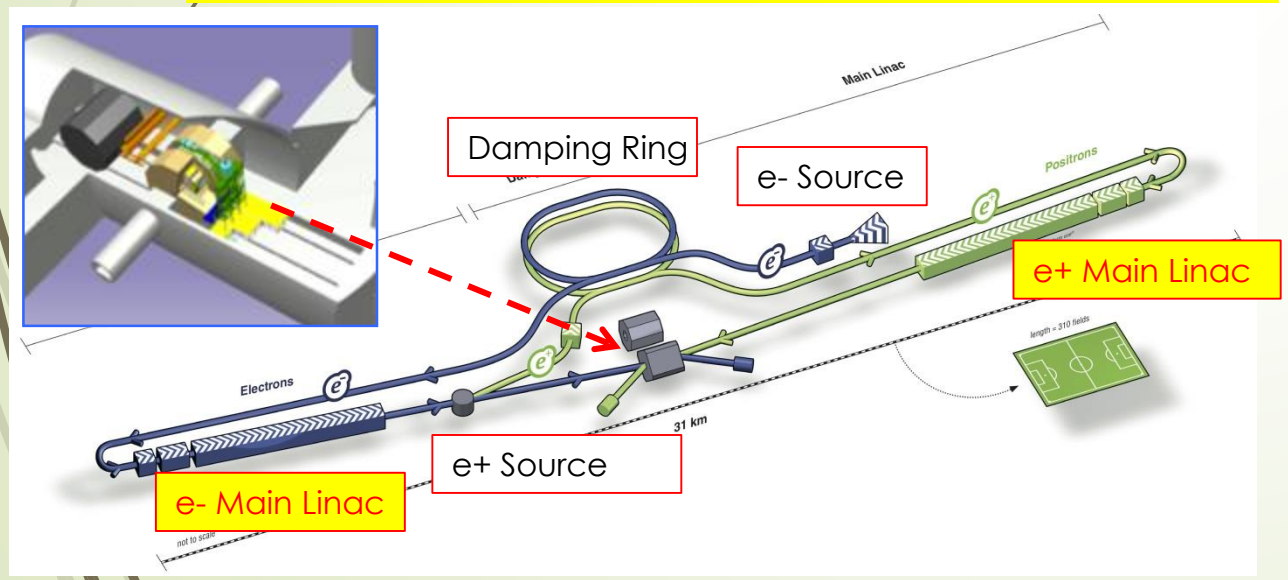
Outline

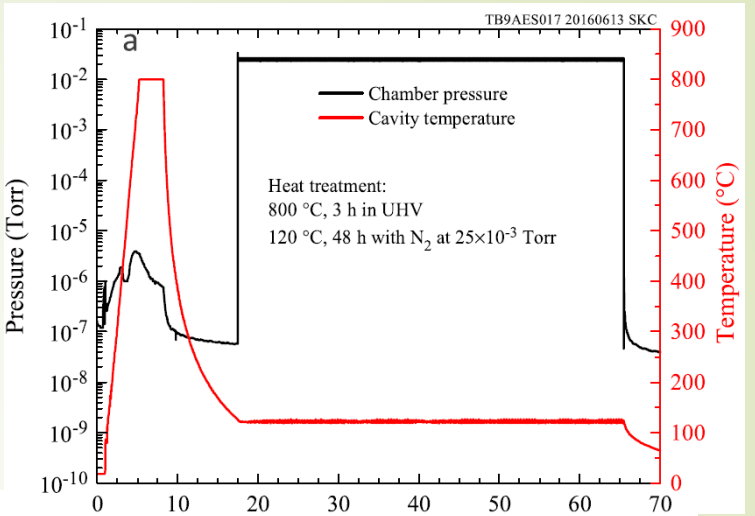
1. Nitrogen infusion
2. J-PARC furnace
3. First trial of N-infusion
4. Second and third trial of N-infusion with improved vacuum system
5. 4^{th} - 6^{th} trial of N-infusion.
6. Sample analysis
7. Summary

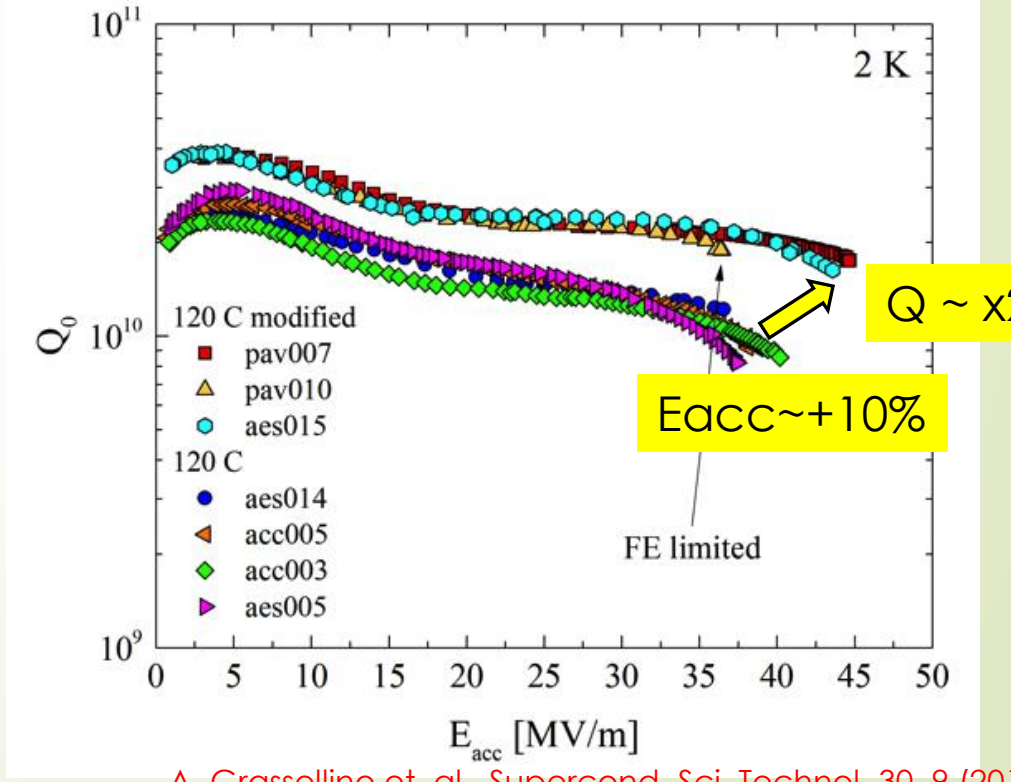
Nitrogen Infusion

- Nitrogen infusion is a recent breakthrough technology for improving superconducting cavity performance.
- We will apply N-infusion technology for ILC main linac and reduce the cost.
- A nitrogen-rich surface layer is formed by introducing nitrogen during the cavity heat treatment without exposed to atmosphere.

It is expected that
- The accelerating gradient is improved by about 10%.
  ⇒ Number of cavities and modules can be reduced.
- The Q value is improved about twice.
  ⇒the load of the refrigerator can be reduced.



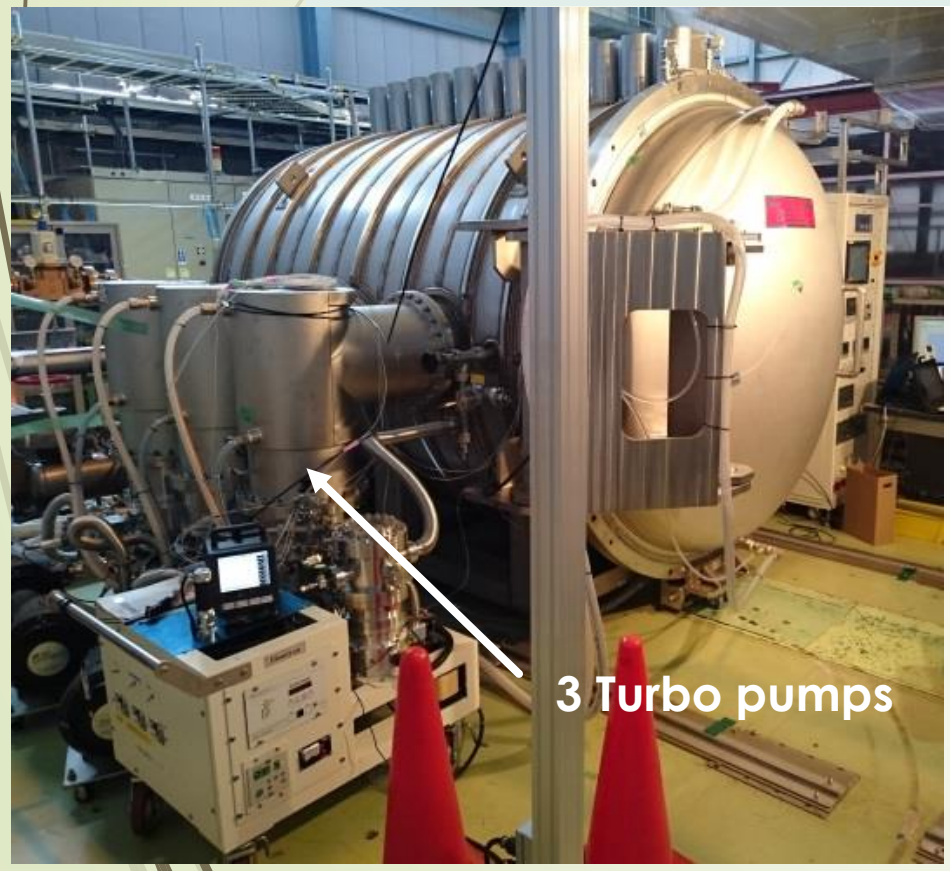


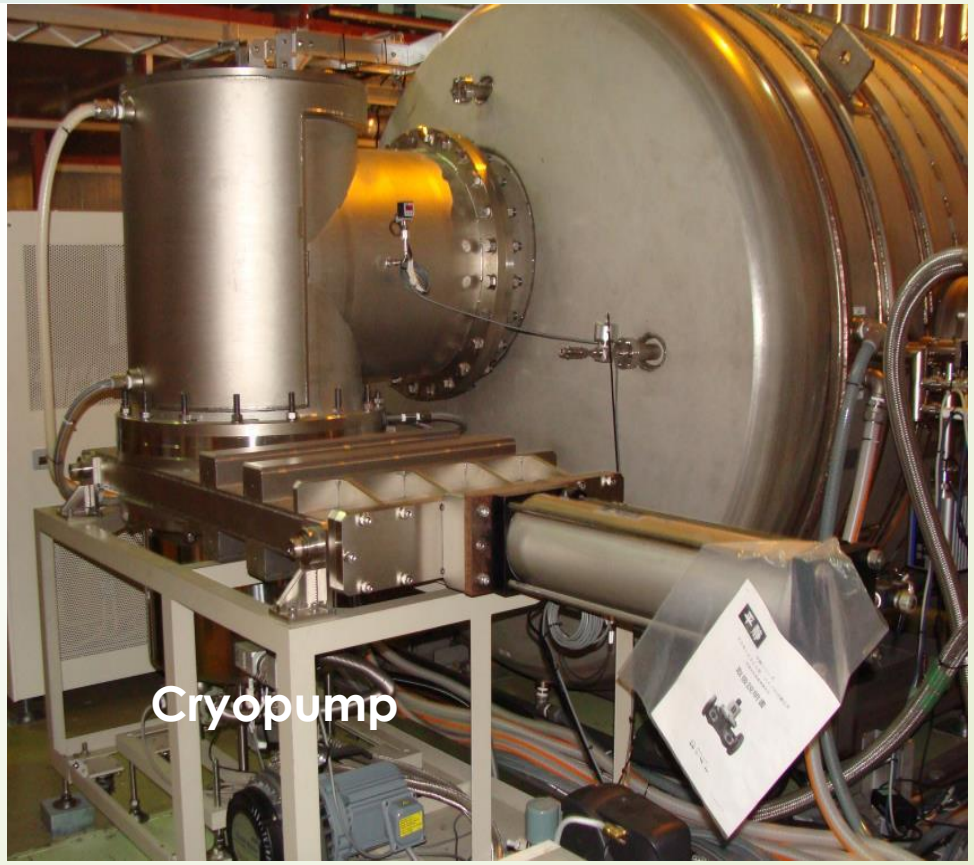


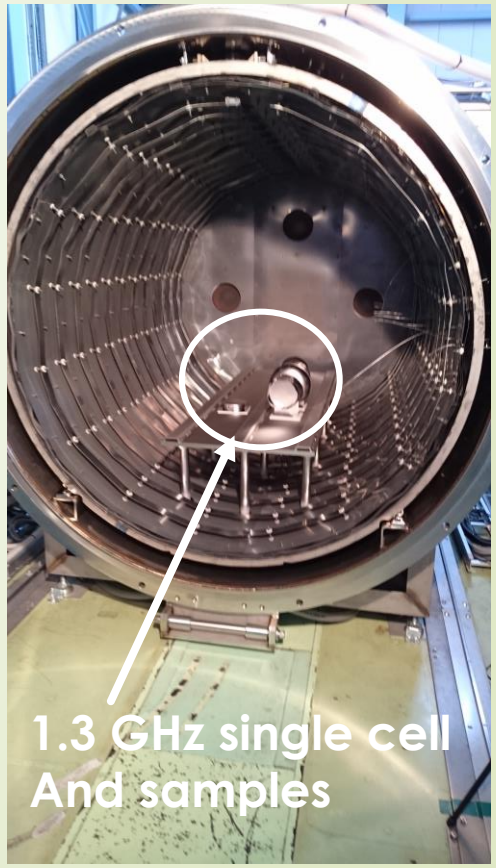
A. Grassellino et. al., Supercond. Sci. Technol. 30, 9 (2017)

J-PARC Furnace

- We have been tested Nitrogen infusion recipe with single or 3 cell cavities.
- J－PARC furnace has been used for SUS and Ti chambers degassing.
- Main pumps are oil free.
  - Turbo pump 3unints: SIMADZU TMP3202M (3000L/sec x3)
    + Scroll pump 3unints: ANEST IWATA ISP500 (500L/min x3)
  - Cryopump 1unint: ANELVA CAP220 (10000L/sec)
- Small samples for surface analysis were set beside with cavity.



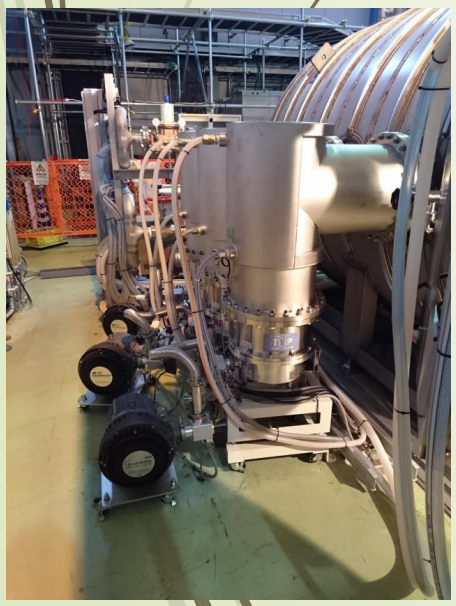


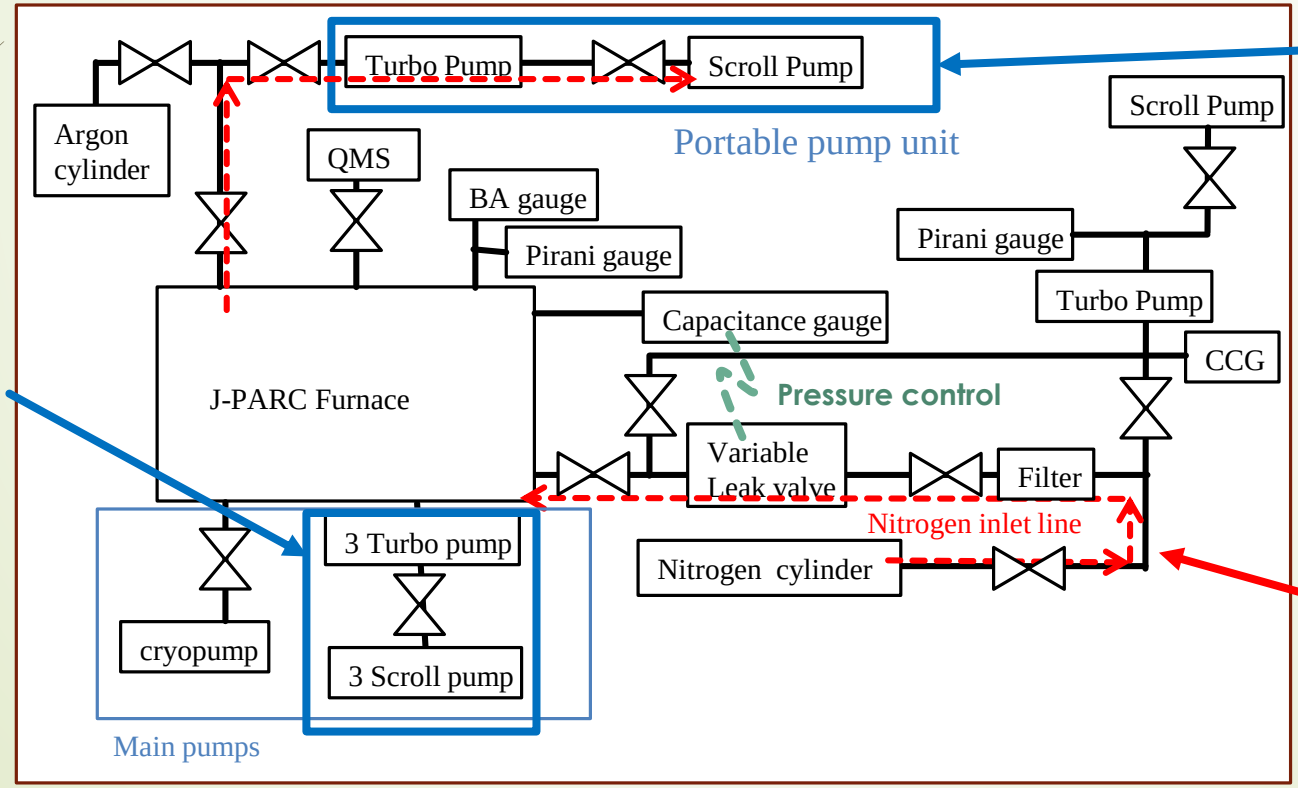


N-infusion system

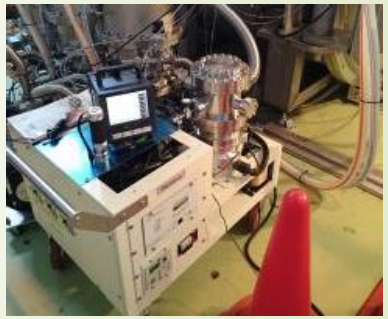
- A nitrogen introduction line was added for nitrogen infusion.
- For high temperature annealing at 800 ° C, turbo pump and cryopump were used.
- Two patterns of pumping system were used during nitrogen infusion. At first, considering the load of pumps, the main pumps were stopped and the portable pump was used. However, the reached pressure was poor. Therefor, three turbo pumps of the main pumps were used. The cryopump was shut out by the valve.
- The chamber pressure during N-infusion was monitored with a capacitance gauge and adjusted with a variable valve controller. A nitrogen bottle with a purity of 6 N was used.

Main 3 TMPs





Portable pump unit



Nitrogen inlet line

History of N-infusion at J-PARC

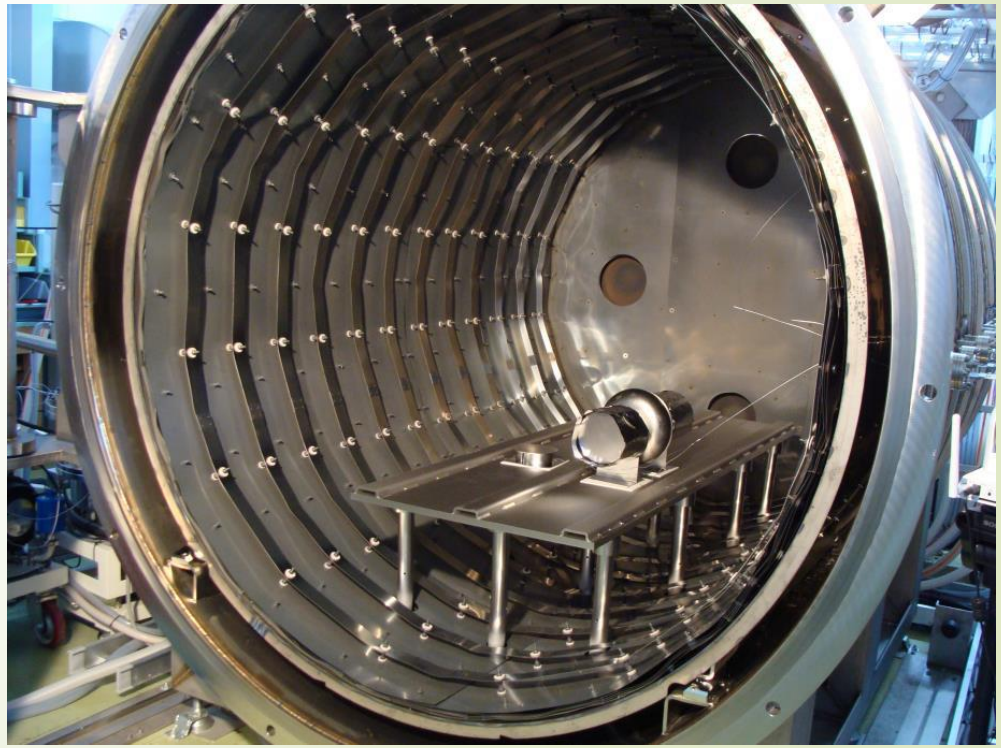
- The 1st nitrogen infusion trial was failed in which the Q value degraded from low gradient.
- We thought that base pressure during N-infusion was too high because the portable pump unit was used. Therefore we switched to the main pumps.
- After that, we succeeded twice. However, from the 4th trial, a phenomenon similar to the 1st trial has occurred. We have not elucidated this cause.

#	Month	Result	# of Cells	Series	Treatment	N2 pumping unit	Cavity material
0	2017.3	Success	Single	R-6	800C Anneal		FG (TD)
1	2017.4	Fail	Single	R-2	N-Infusion(800x3h+120x48h)	Portable pump unit	FG(TD)
1'	2017.6	Fail	Single	R-2	N-Infusion(800x3h+120x48h) w/o N2	Portable pump unit	FG(TD)
2	2017.11	Success	Single	R-8c	N-Infusion(800x3h+120x48h)	TMP50%	FG(TD)
	First successful case! Q value and gradient were improved.						
3	2018.1	Success	Single	R-9b	N-Infusion(800x3h+125x48h)	TMP50%	FG(TD)
	Q value was improved. Q value at high gradient was degraded by Field Emission.						
4	2018.2	Fail	Single	R-2	N-Infusion(800x3h+160x48h)	TMP50%	FG(TD)
5	2018.3	Fail	Single	R-9	N-Infusion(800x3h+120x48h)	TMP50%	FG(TD)
5	2018.3	Success	Three	R-10b			LG(CBMM)
	First successful case of LG! Q value was improved. Q value at high gradient was degraded.						
6	2018.4	Fail	Single	R-2	N-Infusion(800x3h+120x48h)	TMP50%	FG(TD)
6	2018.4	Fail	Single	R-9b			FG(TD)

Cavity preparation for heat treatment

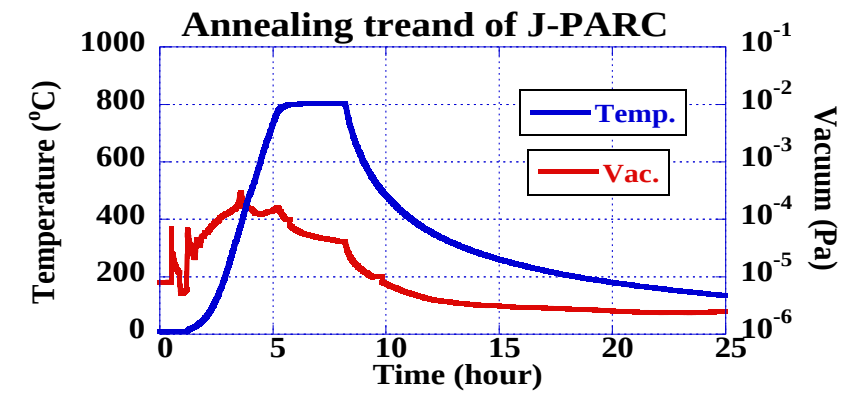
- Cavity was applied refresh EP, HPR(flange open) 2 hours, dried one night and double-packed inside class-1000.
- Nb cap and foil were polished by CP and ultrasonic cleaned with degreasing, dried inside class-10, packed inside class-1000.
- These were prepared at KEK-Tsukuba campus. And Transport to J-PARC. Setup into J-PARC furnace.

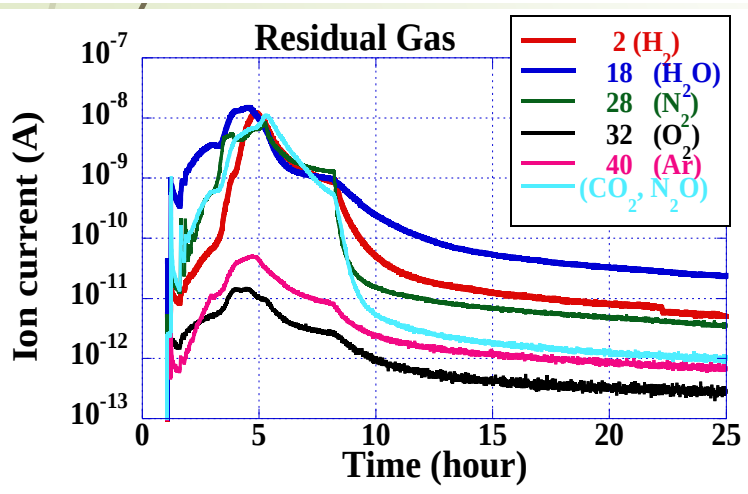


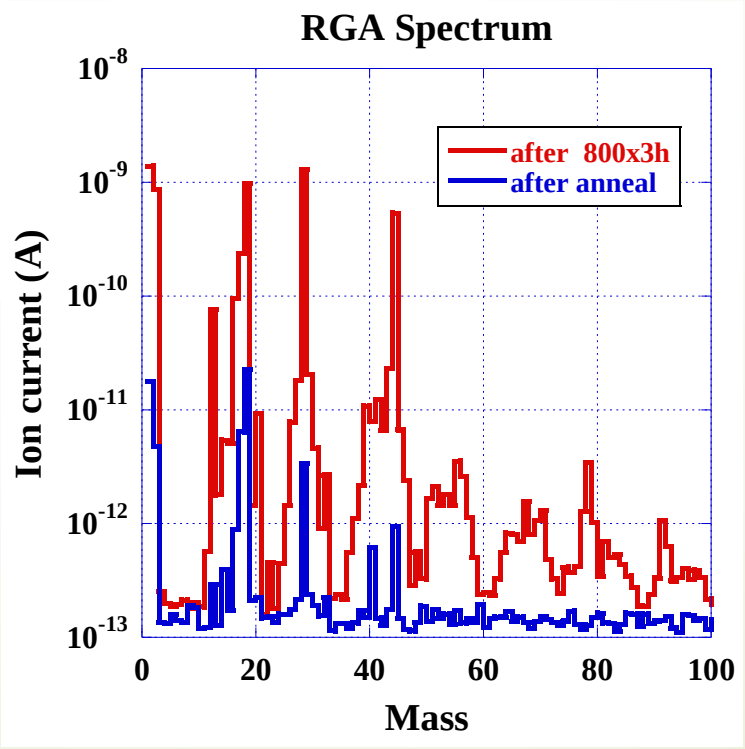



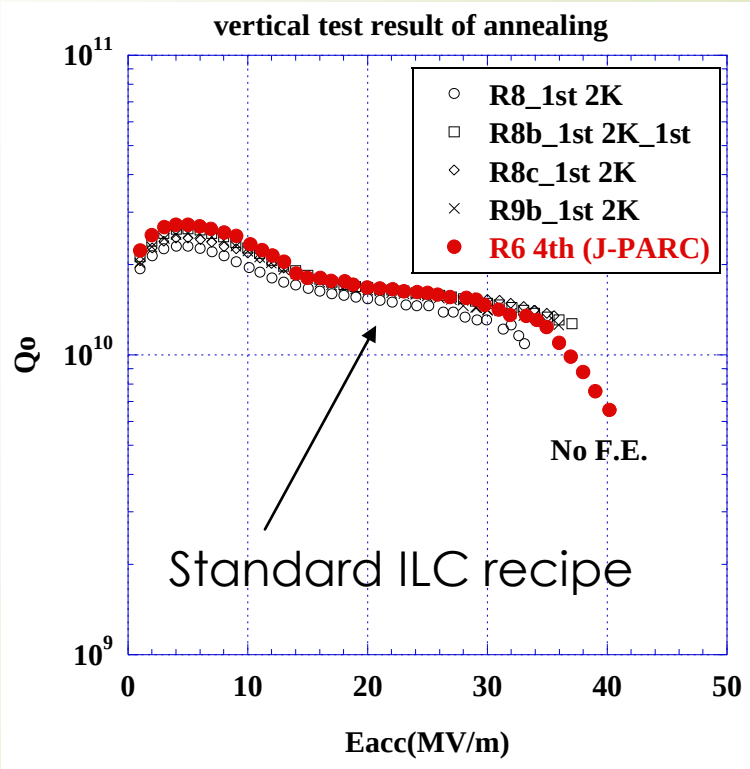
Performance of Furnace after annealing

- We checked furnace cleanness by normal annealing at 800 °C.
- Furnace pressure after cooling reached 2.4E-6 Pa. Residual gas are mainly H2, H2O, N2, CO2, (N2O). Hydrocarbons seems enough low.
- Cavity was measured without EP after annealing and with baking 120 °C x48 h.
- The performance was the same as the standard ILC recipe including final EP.



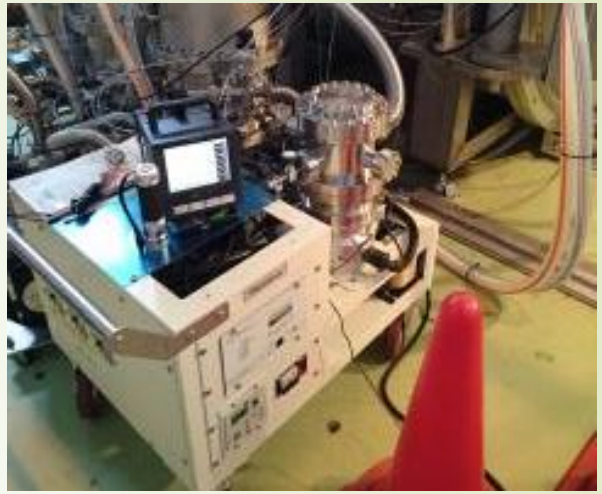


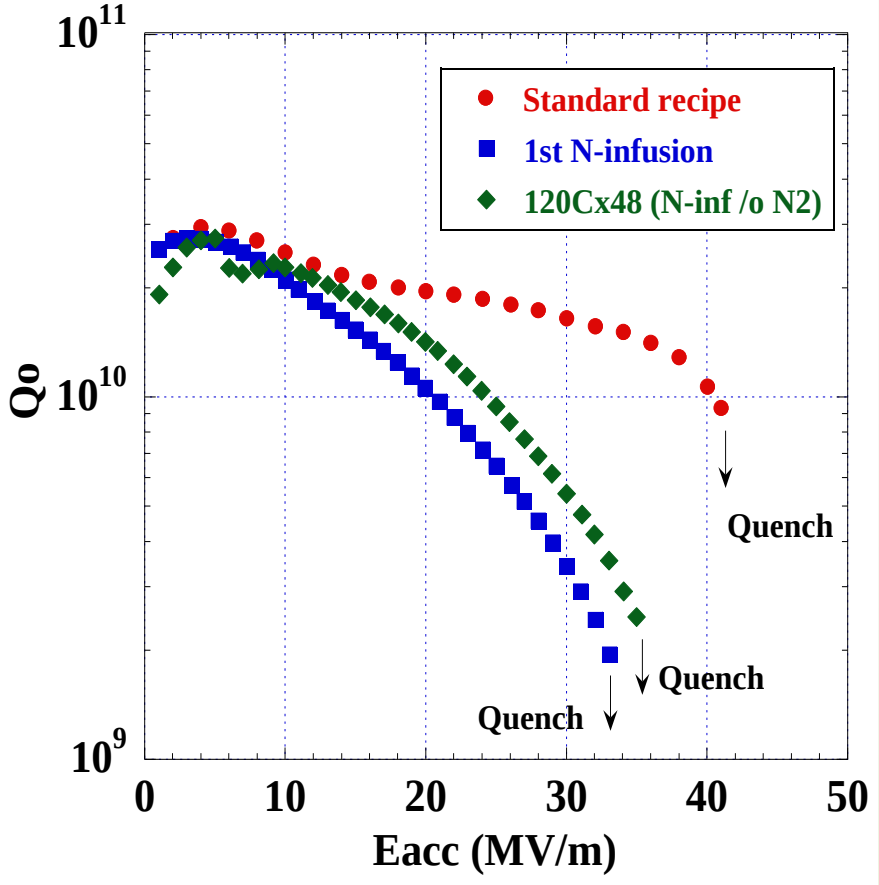


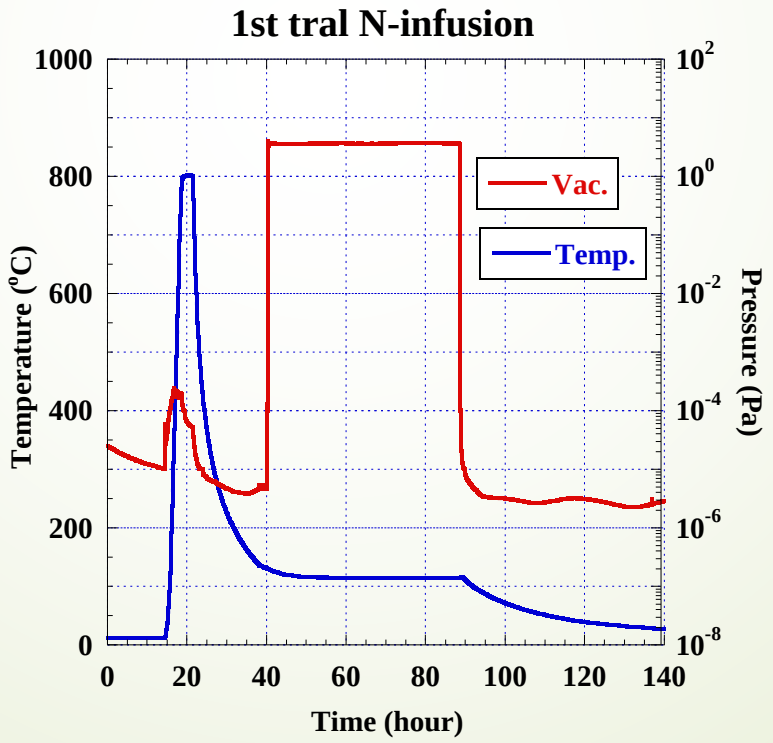


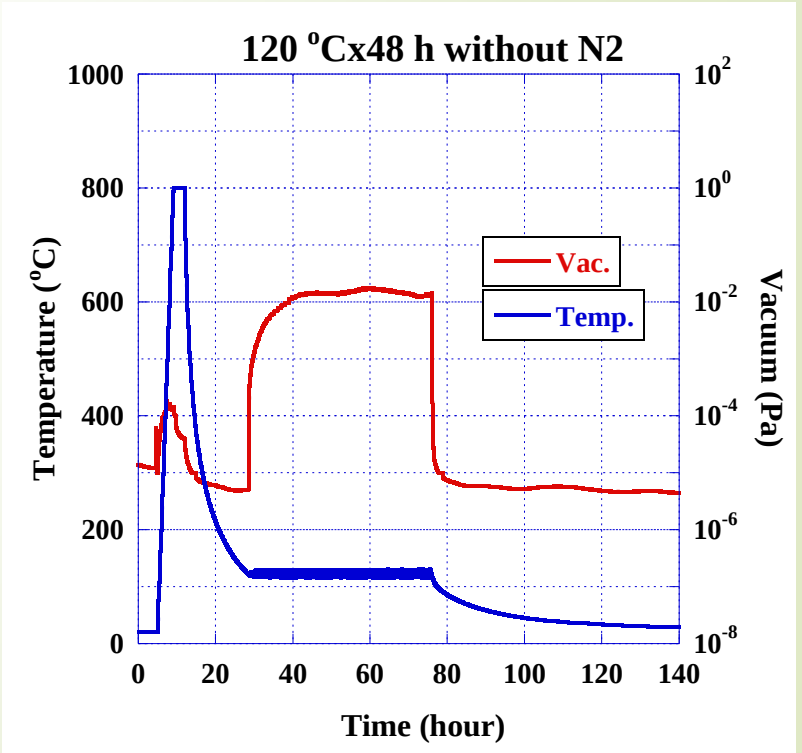
1st trial N-infusion

- 1st trial of N-infusion was failed.
- We suspected that residual gas infused to the cavity because the pumping speed of the portable pump unit was too low.
- The result of dummy N-infusion without nitrogen was same as 1st trial. From this data, the base pressure of 1st N-infusion was ~1.4 E-2 Pa.





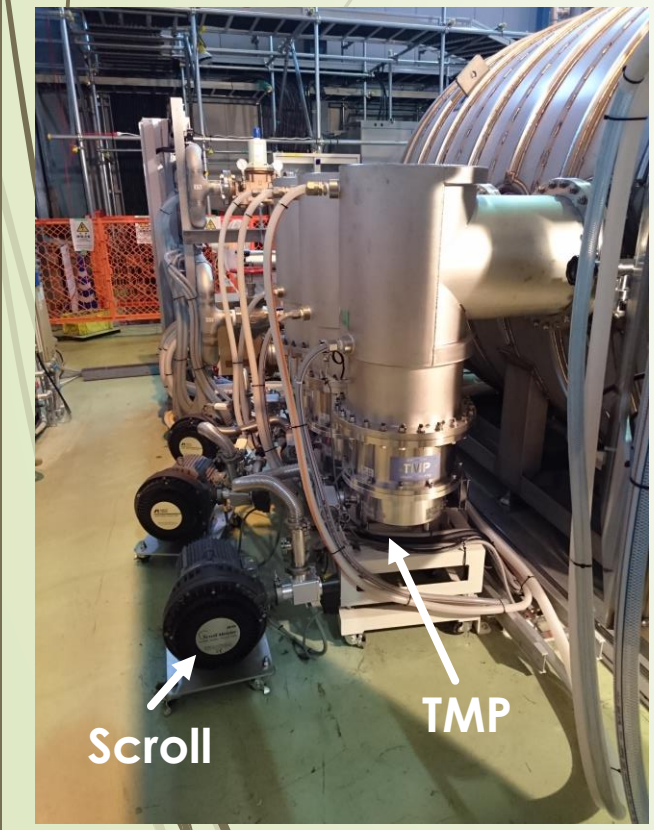


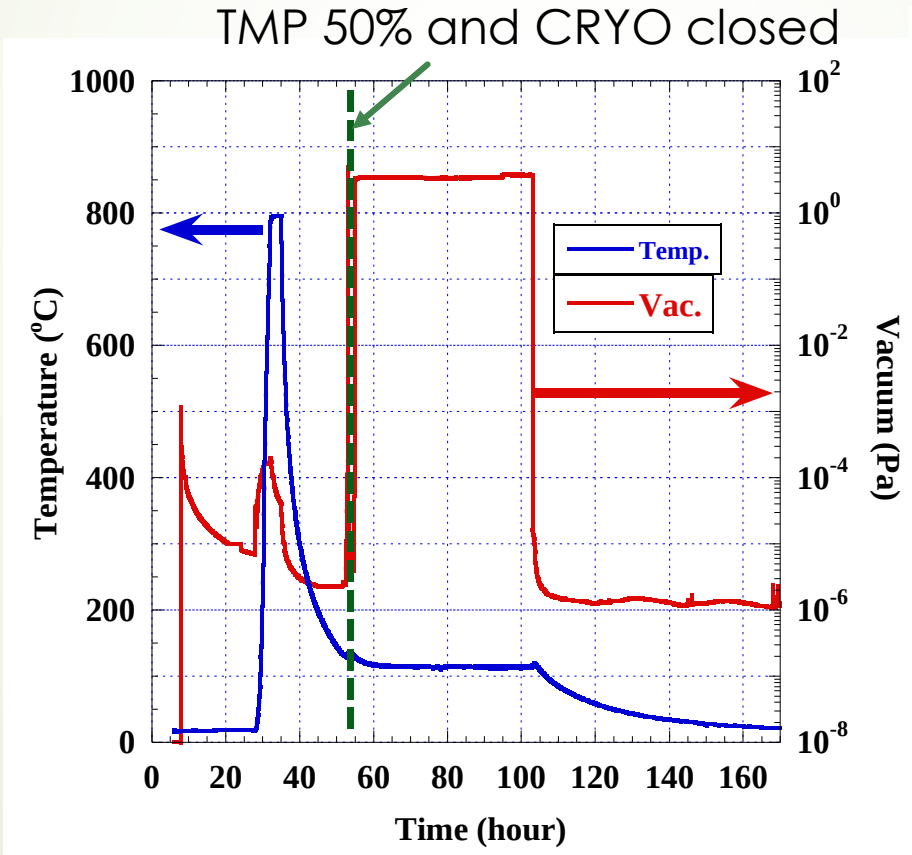


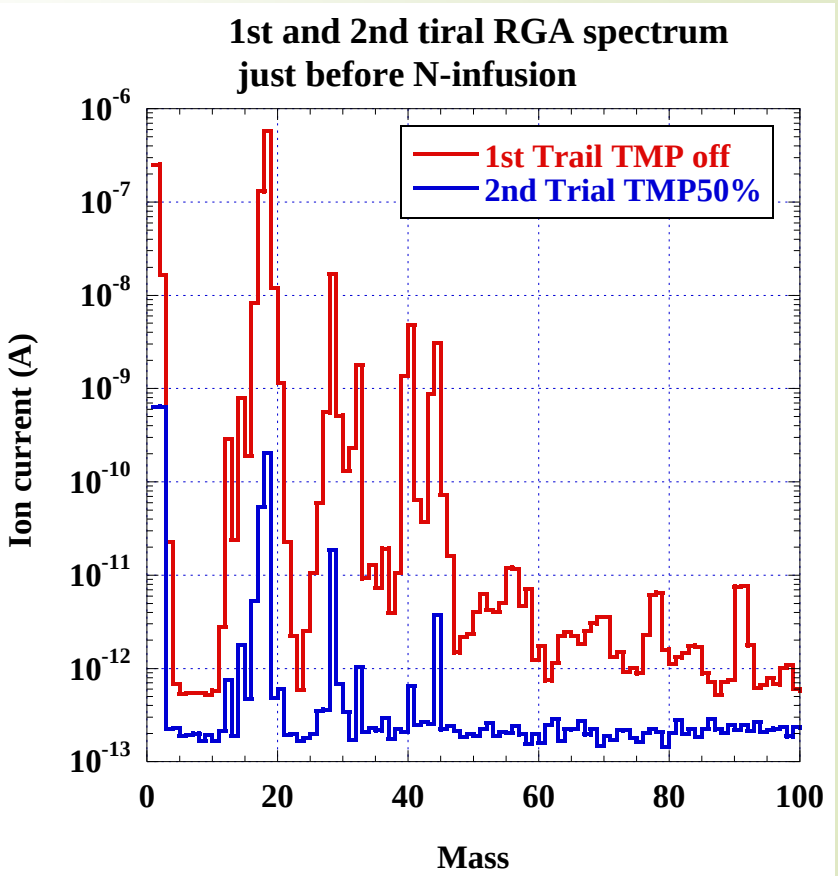
2nd trial N-infusion

- From 2nd Trial, we used main 3 TMPs during N-infusion.
- Rotation speed was slow down to 50 % because of the load of TMP blade.
- RGA spectrum just before N-infusion improved about 3 digits and base pressure was about ~1E-5 Pa.

Base pressure during N-infusion
1st : ~1 E-2 Pa
2nd : ~1 E-5 Pa

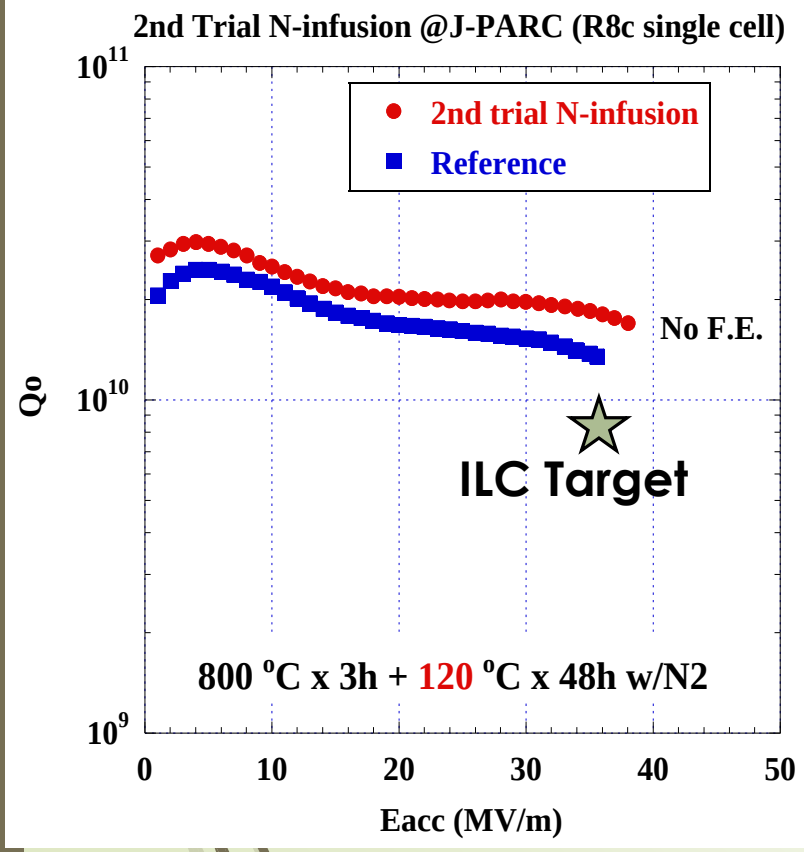


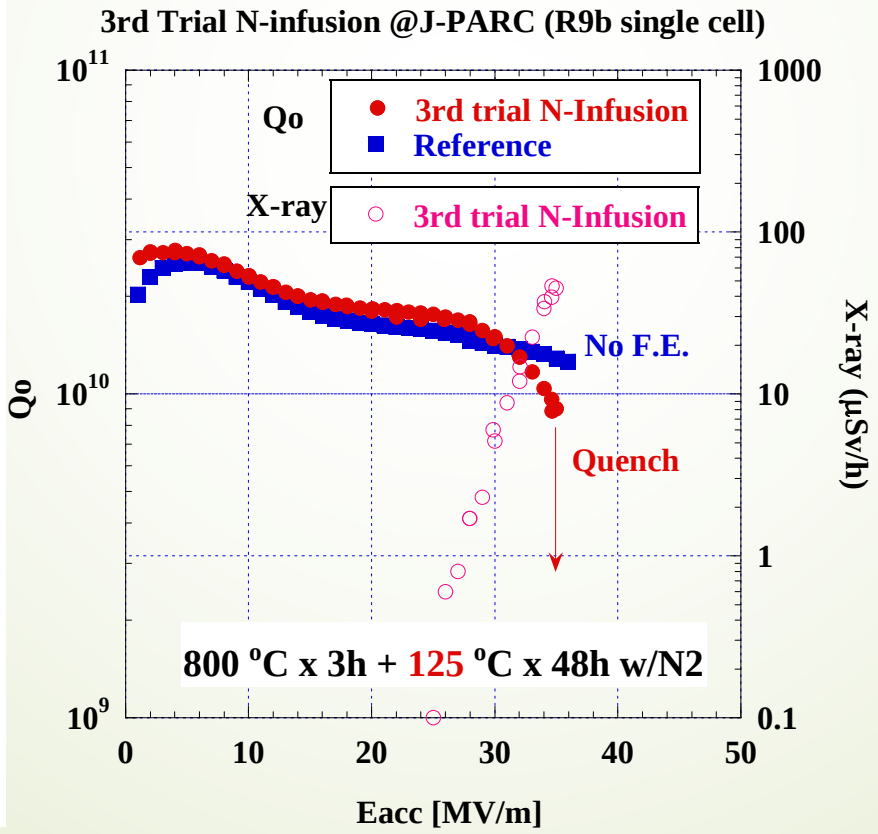


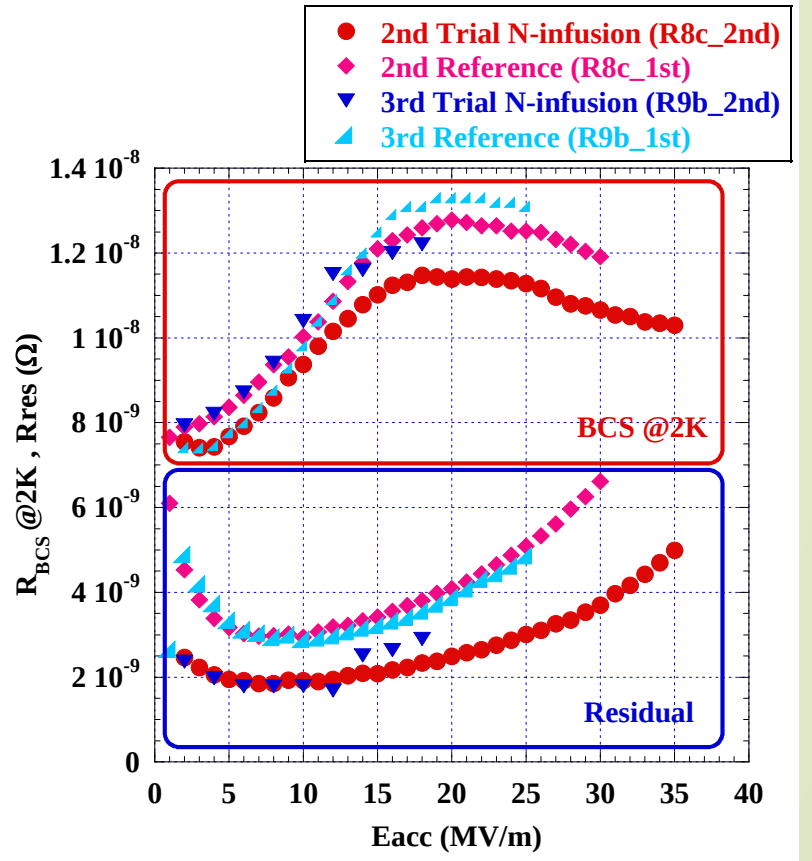


Success of N-infuison

- Both 2nd and 3rd trial N-infusion was succeeded.
- 2nd trial (120 °C N-infusion): Gradient was improved 5% and Q_o was improved 30%.
- 3rd trial (125 °C N-infusion): Unfortunately Q value at high gradient was degrader by field emission.
- Both residual resistance were lowered than reference and BCS resistance of 2nd trial were lower than reference.



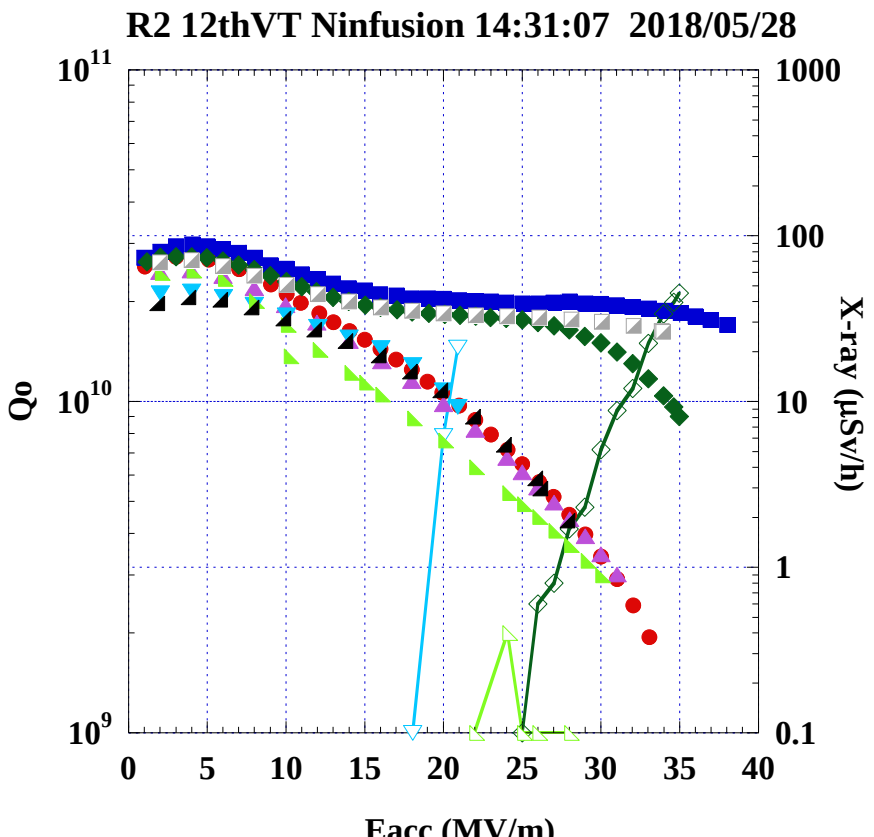




4th -6th trial N-infusion

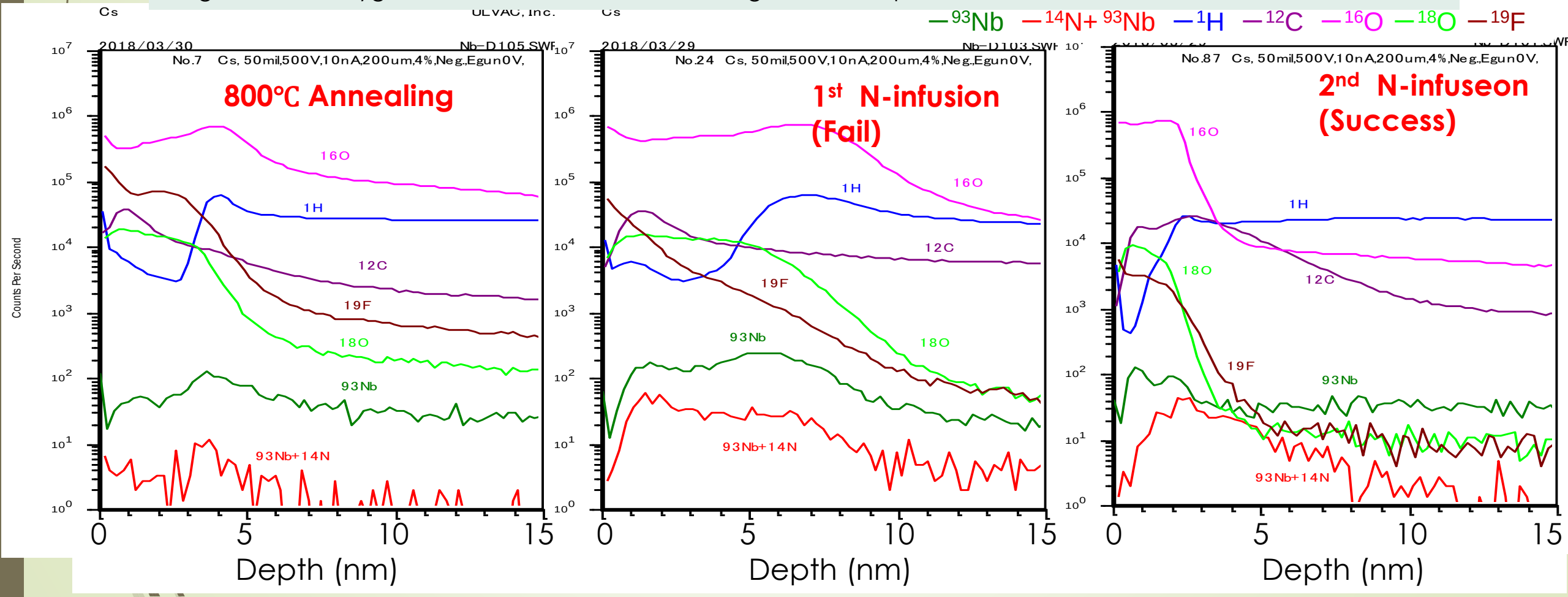
- Q drop from low gradient similar to the 1st trial was recurred from 4th trial.
- Events between the 3rd and 4th trial were the replacement of nitrogen cylinder and degassing of 2 titanium bellows.
- We suspected the lack of baking of the nitrogen inlet line and polishing of niobium cap and foil. We tried baking again and cover cavity more carefully. But these were ineffective.

#	Result	Series	Treatment	Cavity material
1	Fail	R-2	N-Infusion(800x3h+120x48h	FG (TD)
2	Success	R-8c	N-Infusion(800x3h+120x48h)	FG(TD)
• Pumping unit was changed from portable pump to main TMP 50%				
3	Success	R-9b	N-Infusion(800x3h+125x48h)	FG(TD)
• N2 bottle was changed. • 2 Titanium bellows were annealed at 650 °C x10 hours				
4	Fail	R-2	N-Infusion(800x3h+160x48h)	FG(TD)
5	Fail	R-9	N-Infusion(800x3h+120x48h)	FG(TD)
5	Success	R-10b		LG(CBMM)
• N2 inlet line was baked again.				
6	Fail	R-2	N-Infusion(800x3h+120x48h)	FG(TD)
6	Fail	R-9b		FG(TD)



Sample analysis by D-SIMS

- Samples of 800℃ Annealing and 1st and 2nd N-infusion were measured by D-SIMS and XPS (measured by ULVAC Inc.)
- The deposition of oxygen is deep in the order of 2nd , 800C annealing and 1st.
- Since the Nitrogen pressure, temperature, and time during nitrogen infusion were same for 1st and 2nd, impurity gas such as oxygen seems to accelerate the nitrogen infusion speed.

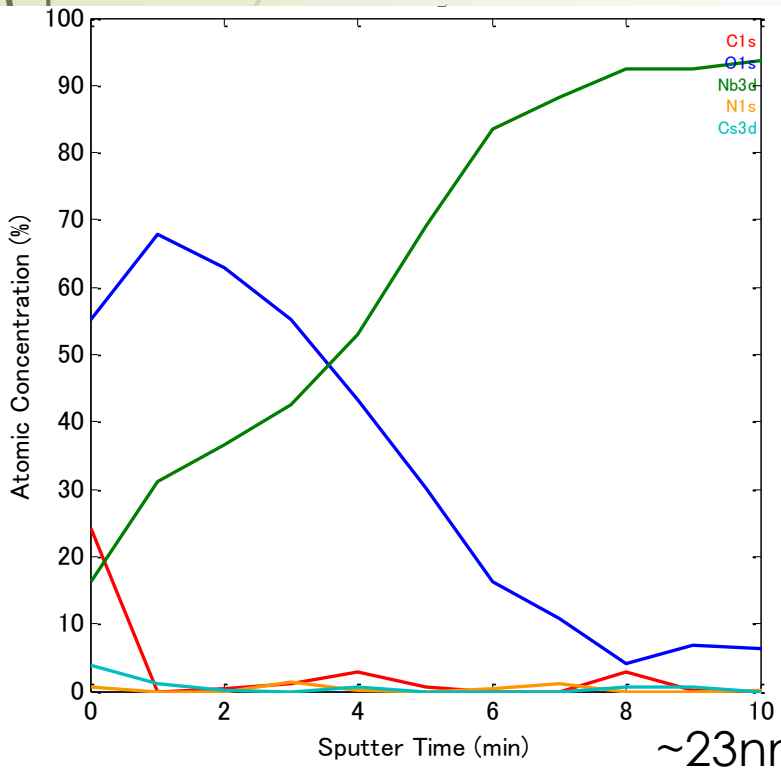


Sample analysis by XPS

- Samples of 800°C annealing, 1st and 2nd N-infusion were measured by XPS (ULVAC Inc.)
- The depth profile of atomic concentration was measure by sputtering (sputter rate was 2.3 nm/min)
- Oxygen depth profile is agree with the SIMS data.

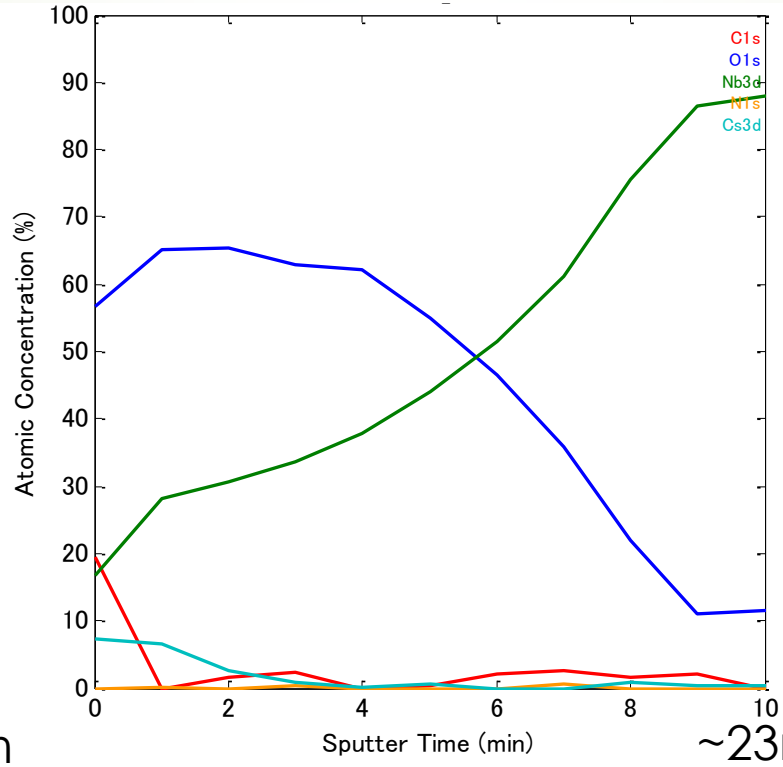
C1s O1s Nb3d N1s Cs3d

800 °C annealing



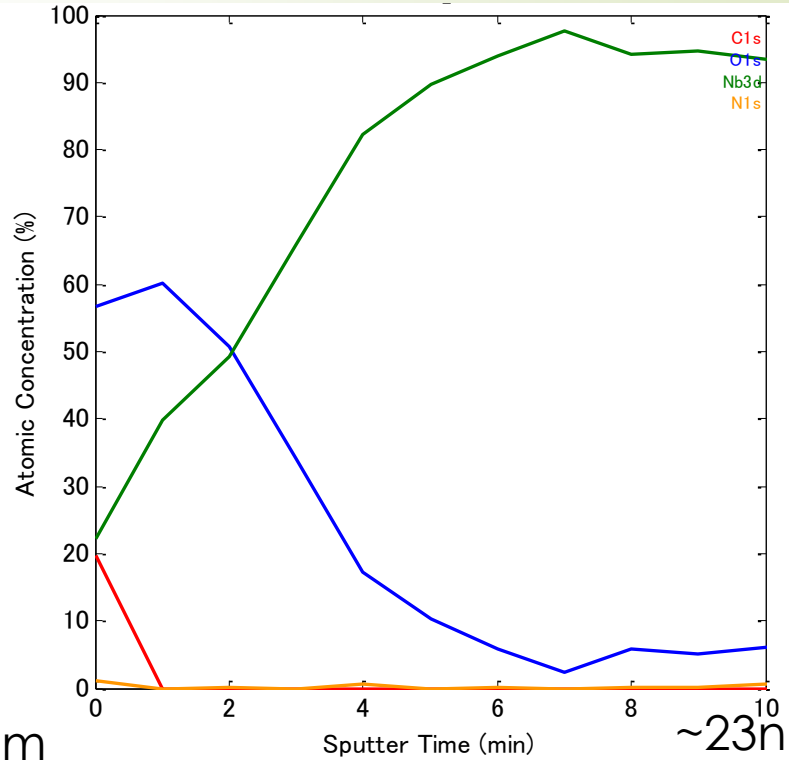
~23nm

1st trial N-infusion



~23nm

2nd trial N-infusion



~23nm

Summary

- Nitrogen infusion is recent breakthrough technology to improve ILC cost performance.
- KEK studied N-infusion recipe by using J-PARC furnace.
- 3 cases were succeeded (including 1 large grain cavity) after improving the base pressure to ~1E-5Pa during infusion.
- However 4 cases were failed. We started to investigate the reason.
- From SIMS and XPS data, oxygen depth profile appears to have a big difference between successful and failed N-infusion.

Backup

Purity of Nitrogen after 3rd trial (Successful case)