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STUDY ON NITROGEN INFUSION USING KEK NEW FURNACE

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Abstract

KEK has been carried out high-Q/high-G R&D, to realize high performance of SRF cavities toward ILC. KEK constructed a new furnace, which is dedicated for N-infusion studies. We performed 10 times of N-infusion trials using 1.3 GHz cavities. Some results showed good Q-values up to high field, however, some results showed degraded Q-E slopes probably due to contaminations. Improvement of accelerating gradient is not observed at moment. We have tried to clean the furnace and Nitrogen injection line to reduce the effect of contaminations. Details of procedures of N-infusion, results of vertical tests, condition of the furnace including RGA spectrum are shown.

KEK NEW FURNACE

N-infusion technique is developed to improve SRF cavity performance for both Eacc (accelerating gradient) and Q-values [1]. KEK also had carried out R&D studies to realize high gradient SRF operations.

Throughout N-doping studies we recognized that cleanness of furnace is most important for Nitrogen treatment [2, 3]. Especially, in case of N-infusion, cleanness of furnace could directly affect to cavity performance, since final EP can not be applied to the cavity.

Figure 1: KEK new vacuum furnace.

Our N-infusion studies started by using the J-PARC furnace, which has oil-free pumping systems. However, the results were not so good. Q-degradation phenomena was sometimes observed [4]. Contamination from the furnace was suspected, since the J-PARC furnace was not dedicated to superconducting cavities.

While studying at the J-PARC furnace, we designed and constructed a new furnace (Figure 1) on KEK cite. Details of the furnace is described in the proceedings [5].

The furnace has oil-free pumping system and main pumping is 10,000 L/sec cryopump. Vacuum pressure can be reached to around 1e-6 Pa. One side of door is surrounded by a clean booth. During injection of Nitrogen, a TMP with 700 L/sec pumping speed was used to reduce background pressure to level of 1e-5 Pa. A mass-flow controller is used to keep Nitrogen pressure. The TMP, RGA and mass-flow controller and Nitrogen injection line are shown in Figure 2.

The furnace was constructed in FY2017, commissioning was carried out for a few months, then it has been using for N-infusion R&D studies.

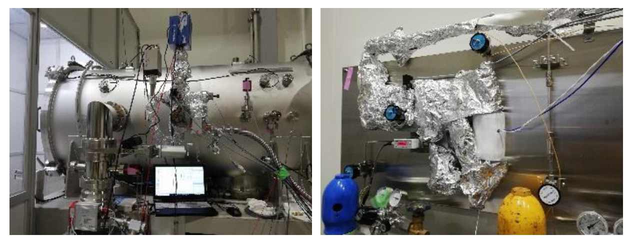

Figure 2: (left) TMP and RGA monitoring system. (right) N-injection system with mass-flow controller.

N-INFUSION PROCEDURE

Table 1 shows history of N-infusion studies performed at KEK new furnace. Total of 10 times N-infusion was carried out.

Before installing to the furnace, cavities were washed by HPR (High Pressure Rinsing), dried in a cleanroom and packed in a clean bag. Then they were transported to the furnace.

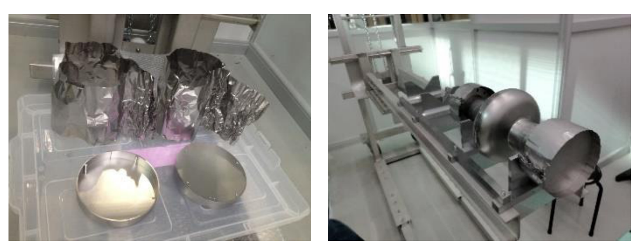

Figure 3: (left) Nb caps and foils to cover flanges. (right) Cavity covered with Nb cap.

As shown in Figure 3, cavity flanges were covered by Nb caps and surrounded by Nb foils. There is enough space, more than several mm, as Nitrogen passage between cavity flanges and Nb caps. These Nb caps and foils are cleaned by CP and ultra-sonic rinsing with detergent, every time. Cavities were supported by Molybdenum jigs and set on Molybdenum table in the furnace.

As shown in Table 1, some N-infusion runs observed Q-degradation, which was also often observed for N-infusion at J-PARC furnace.

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Table 1: Condition of N-Infusion

No.	Date	Cavity name	No. of cells	Nb material	N-infusion process	Q-degradation	Max. Eacc
1	2018.6	R-6	Single	FG	800C, 3h + 120C, 48h w/ 3.3Pa N_2	No	35
2	2018.6	R-9b	Single	FG	800C, 3h + 120C, 48h w/ 3.3Pa N_2	No	26
3	2018.6	R-10	3-cell	LG	800C, 3h + 120C, 48h w/ 3.3Pa N_2	No	27
Summer shutdown							
4	2018.9	R-2	Single	FG	800C, 3h + 160C, 48h w/ 3.3Pa N_2	Yes (strong)	19
5	2018.10	R-6	Single	FG	800C, 3h + 120C, 48h w/o N_2	Yes	32
Apply dedicated burning run after this period							
6	2018.11	R-8	Single	FG	800C, 3h + 800C, 2h + 120C, 48h w/ 3.3Pa N_2	No	36
Improve cooling of cryo-pump by adding cooling-water type shielding plate							
7	2018.12	R-9b	Single	FG	800C, 3h + 800C, 2h + 160C, 48h w/ 3.3Pa N_2	Yes	24
8	2019.1	AES18	Single	FG	800C, 3h + 800C, 2h + 120C, 48h w/ 3.3Pa N_2	No	38
Modify N_2 injection line							
9	2019.4	R-4	Single	FG	800C, 3h + 120C, 48h w/ 3.3Pa N_2	Yes (Weak)	39
10	2019.5	AES18	Single	FG	800C, 3h + 120C, 48h w/ 3.3Pa N_2	Yes	31

First three trials just after commissioning were fine, even without burning run. After summer shutdown, however, Q-degradation started to be observed. We apply burning runs with around 1100 degree C, from 6th N-infusion run.

Figure 4 shows N-infusion process applied from 6th to 8th N-infusion. For these runs, two times of 800 degree C heat treatments were applied for cavities, before injection of Nitrogen at 120 degree C, 48h. Aim of this procedure is to make cleaner cavity surface before N-infusion. Vacuum pressure at second 800 degree C cycle is about one order smaller than first heat cycle. We also expect that hydrogen absorbed in Niobium may be smaller.

After 8th N-infusion run, we added a gas filter on N_2 injection line. It can remove some gas components, including CO_2. Nitrogen injection line is leak tight and baked. We try to make the furnace and Nitrogen injection line as clean as possible.

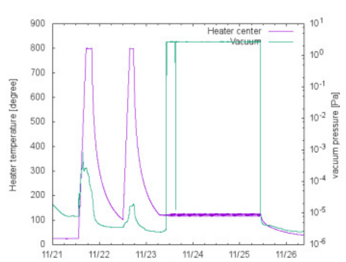

Figure 4: (left) Temperature and vacuum history of typical N-infusion process, which was applied from 6th to 8th N-infusion runs.

Vertical tests were carried out at KEK-STF. Magnetic field inside dewar was cancelled by a solenoid coil. Typically, remaining field is around 1 mG or less. Thermal gradient on the cavity surface was made by a heater during cool-down for better flux expulsions [6].

VERTICAL TEST RESULTS

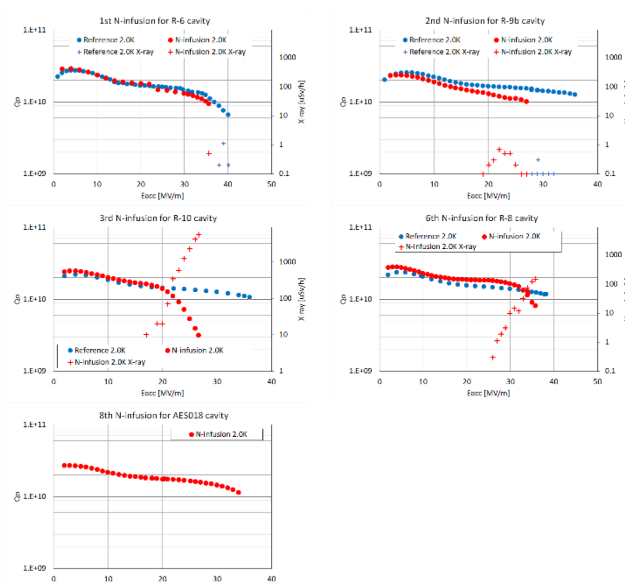

Figure 5: Comparison of vertical test results between N-infusion(red) and reference(blue) for (left-top) 1st, (right-top) 2nd, (left-centre) 3rd, (right-centre) 6th and (bottom-left) 8th N-infusion. No reference data for 8th N-infusion.

Among 10 N-infusion trials, 5 runs were without Q-degradation and remaining 5 runs had Q-degradation. Figure 5 shows Q-E curves taken at 2.0 K for no Q-degradation runs, 1st, 2nd, 3rd, 6th and 8th N-infusion runs.

Vertical test results are compared with reference measurements, EP + 120 degree C 48h baking, except 8th run. Only 6th N-infusion run for R-8 cavity shows slightly better Q-values compared with reference. Unfortunately, Q-value at high field can not be compared because of field emission. Other vertical test results do not show improvement of Q-values for N-infusion cavity. No N-

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infusion cavities at KEK could not exceed accelerating gradient, compared with reference.

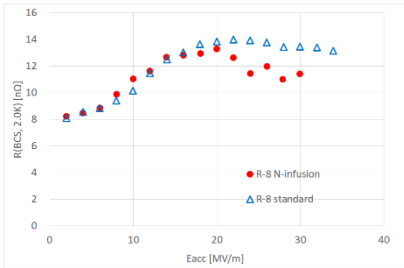

Figure 6: Comparison of deconvoluted BCS resistance at 2.0 K of R-8 cavity for 6th N-infusion and reference.

Basically, Q-E curves are taken for different temperatures between 1.5 K to 2.0 K. Vertical test results for R-8 cavity, right-centre plot of Figure 5, were analysed and BCS resistance at 2.0K were deconvoluted. Figure 6 shows deconvoluted BCS resistance of R-8 cavity for N-infusion and reference. No significant difference is shown between them. Slight better Q-value for N-infusion comes from reduced residual resistance.

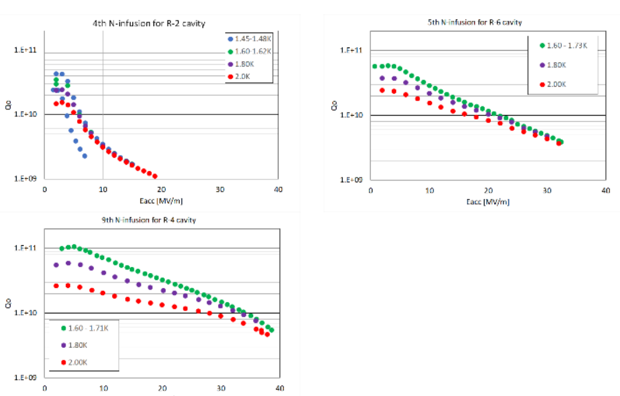

Figure 7: Vertical test results for failed N-infusion: (left-top) 4th, (right-top) 5th and (left-bottom) 9th N-infusion runs.

Although we care about cleanness of the furnace and N-infusion procedure itself, about half of N-infusion runs show Q-degradations. Three Q-degradation slopes are shown in Figure 7. 4th N-infusion run, just after the summer shutdown, had strongest Q-degradation. Right-top of Figure 7 is typical Q-degradation slope, which is very similar to the slopes for 7th and 10th N-infusion runs. This slope is also very similar to the one, which we observed for N-infusion at J-PARC furnace. Slope for 9th N-infusion is weak.

RGA SPECTRUM

In order to monitor cleanness of the furnace, an RGA is mounted on the furnace. Bypass line is prepared to monitor gas components even during Nitrogen injection, by using a variable leak valve. Figure 8 is RGA spectrum for N-infusions. RGA spectrum for good (6th N-infusion) and bad N-infusion (7th N-infusion) are compared. Observed main gas contributions are H_2, H_2O, N_2 and CO_2. RGA spectrum are very similar for both cases. No clear difference of no hint for Q-degradation mechanism are observed.

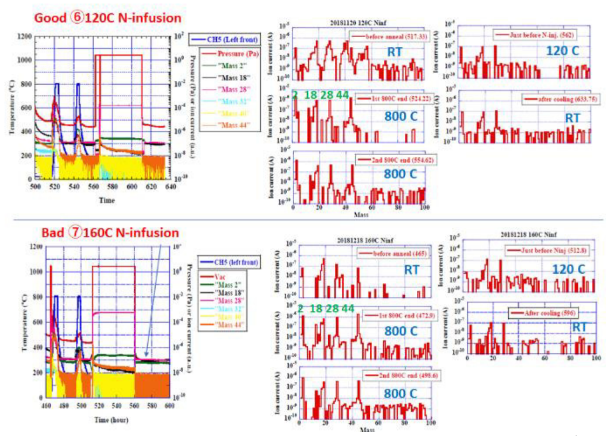

Figure 8: RGA spectrum for good (6th) and bad (7th) N-infusion runs.

Figure 9 shows RGA spectrum just after start of Nitrogen injection. Some amount of CO_2 contribution, around 3 order lower than N_2, are observed. Flushing like phenomena is also observed.

We added a gas filter in the Nitrogen injection line. Gas purity was improved, but situation did not change. Later we recognized that when furnace door is open, air come into the furnace and even during heat treatment these components remains on the furnace surface, then when Nitrogen is fed to the furnace, they are detached from the surface. Therefore, to eliminate these contaminations is difficult.

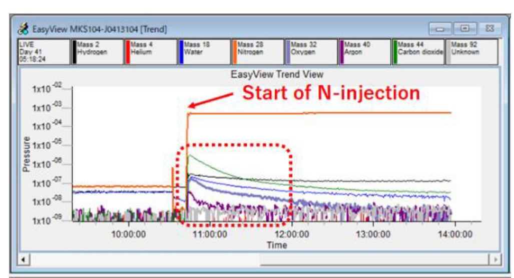

Figure 9: RGA spectrum during Nitrogen injection.

SUMMARY

KEK had constructed new oil-free furnace for N-infusion studies. We had carried out total of 10 times N-infusion runs. We tried to make the furnace and surrounding components as clean as possible, however, still Q-degradation remains. Half on N-infusion was successful, without Q-degradation, but other half was failed with Q-degradations. Even for the successful case, improvement of Q-value is just a little and no cavities show improved accelerating gradient.

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