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FIRST TRIAL OF THE IN-SITU NITROGEN INFUSION AT KEK*

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Abstract

The nitrogen infusion is the new surface treatment technique for improving the RF loss and the maximum accelerating gradient of superconducting cavity. In this process, it is important to be carried out continuously both the 800 °C annealing in vacuum and 120 °C nitrogen infusion without exposure to the atmosphere. The annealing serves activation process by removing the oxide layer. However, there is a risk of dust particles entrance in to the cavity when the cavity is put in the vacuum furnace. The in-situ nitrogen infusion system was prepared to prevent the dust entrance. The other target is to investigate whether nitrogen infusion effect or something changes happen in the case of applying nitrogen infusion technique without removing the oxide layer. It can only introduce nitrogen into a cavity during 120 °C low temperature baking and transport a cavity to the vertical test system without exposure to the atmosphere. We tried to infuse nitrogen to a single cell by keeping 120 °C and 48 hours with 3 Pa nitrogen. The cavity was annealed in another furnace and applied high pressure rinsing before nitrogen infusion. The vertical test result was same Q as the normal 120 °C baking without nitrogen. It suggests that oxide layer prevents infusion of nitrogen.

NITROGEN TREATMENT

The nitrogen treatment of superconducting niobium cavity is getting a lot of attention as a new technology to improve the performance limit of ILC standard recipe [1]. KEK is studying to apply this technology to ILC. It was applied to 1.3 GHz cavities and confirmed that Q value and accelerating gradient were improved [2,3]. The first step in this procedure at KEK is that the surface is removed by the electropolish to create new niobium surface. The cavity is installed to the vacuum furnace after high pressure rinsing. However, there is a risk of dust particles entrance in to the cavity. We designed the in-situ nitrogen infusion system to prevent the dust particle entrance. The second step is that cavity is annealed at 800 oC for 3 hours, then cooled down to 120 oC and kept in 3.3 Pa nitrogen. It is important not to expose to the atmosphere during heat treatment. The cavity is taken out to the atmosphere and cleaned by high pressure rinsing. Thereafter, cavity is not applied heat treatment such as baking because heat treatment changes the surface condition. The final surface condition is made by 800 oC annealing and 120 oC nitrogen infusion. If additional electropolish is applied to remove defects which limit the maximum field after vertical test, high temperature annealing is required again. We studied whether gradient and Q value change when 120 °C nitrogen infusion applied after exposed the cavity to the atmosphere.

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IN-SITU NITROGEN INFUSION

The system of the in-situ nitrogen infusion is a modified baking system to control the nitrogen pressure during 120 C baking (Fig. 1). Top and bottom flanges of the cavity each have one vacuum port. The system can be baked before injecting nitrogen into the cavity. Main pump is the turbo molecular pump (HiPace700) which pumping speed is 700 L/min. The pressure reached about 3×10^{-7} Pa after baking. The gas flow can be controlled by the automatic gas regulating valve (RME005) during the nitrogen injecting. The pressure is controlled by monitoring the capacitance gauge mounted at pumping side of the cavity. The purity of the nitrogen cylinder is more than 99.99995 vol.%. This system can keep the same purity of nitrogen gas cylinder in the cavity during nitrogen injection because the base pressure is enough low. The cavity can be transported to the vertical test stand without exposing the inner surface of the cavity to the atmosphere.

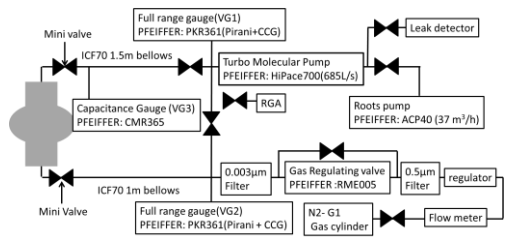

Figure 1: In-situ nitrogen infusion system.

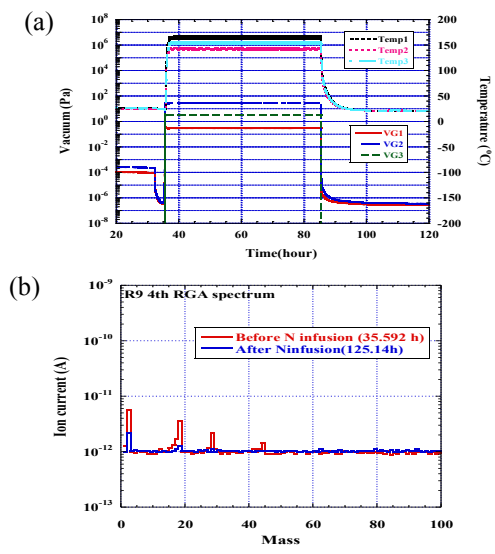

Figure 2: Example of nitrogen infusion. (a) Vacuum and temperature trend. (b) Residual gas spectrum before and after nitrogen infusion.

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Figure. 2 shows the example of nitrogen infusion. Nitrogen gas was injected after pumped the system include the cavity. The vacuum gauges are mounted at head of the turbo molecular pump (VG1), just after nitrogen injection port (VG2) and pumping side of the cavity (VG3). These monitored pressure was much differed from each because vacuum pipes are narrow. Since VG3 was kept 3.3 Pa during nitrogen infusion, the cavity pressure would be higher. All temperature monitors were put on the cavity cell. The temperature of each place in the cavity was controlled within ±15 °C. Figure 2(b) shows the residual gas before and after baking with nitrogen. The main residual gas was hydrogen.

CAVITY AND PROCESS

All tests were carried out by using 1.3 GHz single cell cavity (R-9). Nb sheet was fine grain produced by Tokyo Denkai and cavity was fabricated by MiraPro. Table 1 shows the history of the cavity. The study of the in-situ nitrogen infusion started from the 3rd vertical test. The cavity was annealed at 800 °C for 3 hours before 1st Vertical test and applied 20 µm electropolishing before 2nd vertical test. The baking temperature of the in-situ nitrogen infusion was set 120 °C and 160 °C. The cavity was not opened to air between 3rd and 4th VT. Therefore, 160 °C in-situ nitrogen infusion included the 120 °C in-situ nitrogen infusion effect. The standard recipe which baking temperature set to 120 °C and 160 °C, also carried out in 5th and 7th vertical test for comparison. The 6th vertical test was the original nitrogen infusion which continuously applied 120 °C nitrogen infusion after 800 °C annealing in vacuum furnace. However, the Q value was degraded. The reason has not been understood yet. Ref [3] discussed the detail. We focused on the in-situ infusion and standard recipe results.

RESULTS OF VERTICAL TEST

The surface resistance can be divided to the BCS resistance depends on the temperature and residual resistance which is constant term. The residual resistance is mainly increased by the trapped DC magnetic field. The cryostat for vertical test in KEK was modified to decrease the trapped magnetic flux. The magnetic shield and solenoid coil minimize the geomagnetic field around the cavity. And the temperature gradient of the cavity during cool down is controlled by the heater at top beam pipe for the flux expulsion actively [4]. If the cavity is quenched, the magnetic flux is trapped. Figure 3 shows the vertical test results of the in-situ nitrogen infusion and the standard recipe. Unfortunately, the 2K data of 5th and 7th vertical test was measured after quench. The residual resistance is higher than 3rd and 4th. The vertical test data was taken starting from low temperature. In the 5th vertical test, the multipacting was occurred at 15 and 18 MV/m during 1.5 K data taking and the flux was trapped (Fig. 4 (c)). In the 7th vertical test, the cavity was quenched at 26 MV/m in 1.5 K data taking and the flux was trapped. After quenched the cavity, we take the 1.5 K data again (Fig. 4 (d)). The BCS and residual resistance can be separated by using temperature dependence data shown in Fig. 4. The residual resistance can be compared using low temperature data.

It is interesting that there is not field emission in 4th vertical test. The cavity was just applied in-situ nitrogen infusion at 160 C without opening the cavity to the air. High pressure rinsing or other surface treatment were not applied after 3rd vertical test. The nitrogen gas injected to the cavity was as low as 3 Pa, it didn't have force to blow off the field emission source. It is considered that baking suppressed the field emission by changing the surface condition.

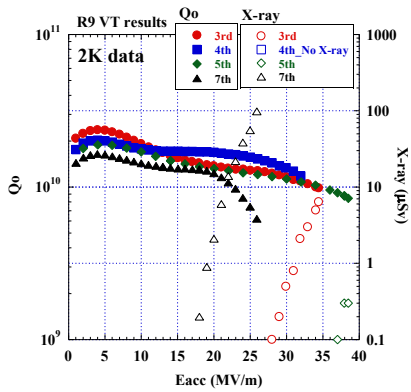

Figure 3: Vertical test results.

Table 1: History of the R-9 Cavity

#	Surface treatment	Detail
1st	800 °C annealing without final EP (EP2)	As received +EP 100 µm +Anneal 800 °C x 3hr +USR 15 min + HPR 3 hr + Baking 120 °C x48 hr, w/o N2
2nd	Standard recipe without baking	+ EP 20 µm + USR 15 min + HPR 3 hr
3rd	120 °C Baking with Nitrogen	+ (without opening to the air) +Baking 120 °C x 48 hr, w/ N2 3Pa
4th	160 °C Baking with Nitrogen	+ (without opening to the air) +Baking 160 °C x 48 hr, w/ N2 3Pa
5th	Standard recipe (120 °C baking without Nitrogen)	+ EP 20 µm + USR 15 min +HPR 3 hr + Baking 120 °C x 48 hr, w/o N2
6th	Nitrogen infusion (120 °C x 48h)	+ N-infusion (800 °C x 3 hr + 120 °C x 48 h, w/N2 3Pa) +USR 15 min + HPR 3 hr
7th	160 °C Baking without Nitrogen	+ EP 20 µm + USR 15 min + HPR 3 hr + Baking 160 °C x 48 hr, w/o N2

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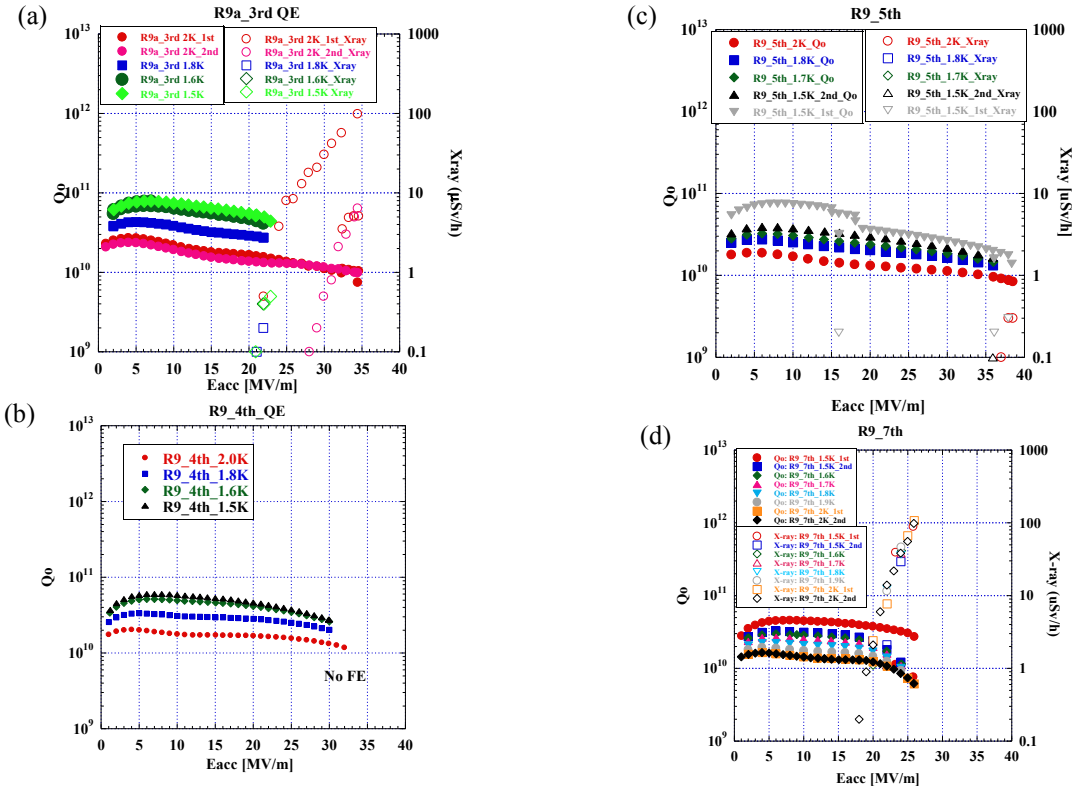

Figure 4: Vertical test results at each temperature. (a) 3rd vertical test; nitrogen infusion at 120 °C, (b) 4th vertical test result; nitrogen infusion at 160 °C, (c) 5th vertical test result; standard recipe at 120 °C, (d)6th vertical test; standard recipe at 160 °C

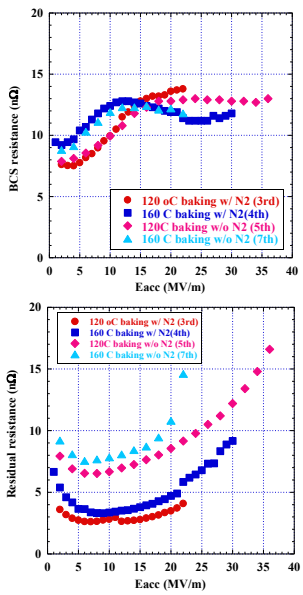

Figure 5: Separated BCS resistance and residual resistance.

Figure 5 shows the BCS resistance part and residual resistance part. The residual resistance is affected by the magnetic flux by the external magnetic field and field emission. It is difficult to discuss the in-situ infusion effect on the residual resistance. The BCS resistance show that the baking temperature is much more influence for the Q value than in-situ nitrogen infusion. These results suggested that high temperature annealing for removing oxidation layer was important for infusing nitrogen into niobium.

SUMMARY

KEK prepared in-situ infusion system that can introduce nitrogen to the cavity during baking and transport to vertical test without exposing to the air. The effect of the in-situ nitrogen infusion was much smaller than baking temperature. It was suggested that the nitrogen was not infused by oxidation layer or nitrogen effect was covered by the other effect. In order to improve the in-situ infusion system, it is necessary to add a system for removing oxidation layer.

REFERENCES

[1] A. Grassellino *et al.*, "Unprecedented quality factors at accelerating gradients up to 45 MV/m in Niobium superconducting resonators via low temperature nitrogen infusion", Supercond. Sci. Technol., vol. 30, p.094004, Aug. 2017, doi:10.1088/1361-6668/aa7afe

[2] T. Okada *et al.*, "Improvement of Cavity Performance by Nitrogen Doping at KEK", presented at the 29th Linear Accelerator Conf. (LINAC18), Beijing, China, Sep. 2018, paper TUPO065, this conference.

[3] K. Umemori *et al.*, "Study on Nitrogen Infusion for 1.3 GHz SRF Cavities Using J-PARC Furnace", presented at the 29th Linear Accelerator Conf. (LINAC18), Beijing, China, Sep. 2018, paper TUPO071, this conference.

[4] K. Umemori *et al.*, "Improvement of magnetic condition for KEK-STF vertical test facility toward high-Q study", in *Proc. SRF'17*, Lanzhou, China, Jul. 2017, pp. 444-447, doi:10.18429/JACoW-SRF2017-TUPB028