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TRIAL OF NITROGEN INFUSION AND NITROGEN DOPING BY USING J-PARC FURNACE

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Abstract

KEK has been carrying out SRF cavity developments toward higher Q-values and higher accelerating gradients. In the past nitrogen-doping was tested using the KEK furnaces, but it did not succeed. This time nitrogen infusion and nitrogen doping are tested using the J-PARC's furnace, which has an oil-free pumping system and is mainly pumped by a 10000 L/s cryopump and three 3000 L/s turbo pumps. Nitrogen pressure is controlled by a variable leak valve and an additional turbo pump. To avoid performance degradation during heat treatment, flanges of cavities are covered by Nb caps and foils. Nitrogen infusion at 120 degrees was applied to a single cell cavity and cavity performance was measured by vertical tests after HPR and assembly. Nitrogen doping at 800 degrees is also applied to another single cell cavity. After applying EP and HPR, vertical tests were carried out. Nb samples were also installed into the furnace during heat treatment. Surfaces are analysed by SIMS and XPS. In this presentation, we report procedure of nitrogen infusion and doping, vertical test results and results of surface analysis.

INTRODUCTION

Nitrogen treatment of superconducting niobium cavity is getting a lot of attention as a new technology to improve the performance limit of ILC basic recipe. KEK is also researching to apply this technology to accelerators such as ILC and ERL. KEK measured the nitrogen doped cavity treated in KEK small and large furnace [1, 2]. KEK small furnace is used for mulch purpose such as niobium cavity annealing, brazing and so on. The pumping system is consists of a diffusion pump with liquid nitrogen trap, a mechanical booster pump and a roots pump (Osaka Vacuum, Ltd. RD600 500 m^3/h) (Fig. 1(a)). KEK large furnace is used only for superconducting cavity treatment. The pumping system is consists of a diffusion pump (ULVAC Inc. PFL-22 10000 L/sec), a mechanical booster pump (ULVAC Inc. PMB024CM 33300 L/min) and a rotary pump (ULVAC Inc. PKS-070 7000 L/min) (Fig. 1 (b)). Q values and accelerating gradient were lower than ILC recipe. The cause was considered to be oil contamination from the pumps. J-PARC furnace was selected because it consists of oil free pumps and good ultimate vacuum pressure. It is used for degassing of stainless chambers using for J-PRAC accelerators. The pumping system consists of three turbo pumps (SIMADZU Corp. TMP 3202M 300L/sec), three scroll pumps (ANEST IWATA Corp. ISP500 500 L/min) and one cryopump (CANON ANELVA Corp. CAP220 10000L/sec) (Fig. 1(c)).

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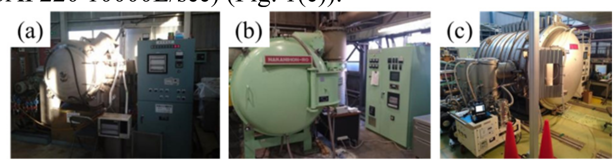

Figure 1: KEK furnaces (a) KEK small furnace. (b) KEK large furnace. (c) J-PRAC furnace.

DETAILS OF J-PARC FUANCE

Nitrogen gas line was added to J-PARC furnace. Figure 2 shows the main pumping systems and the nitrogen line. During the nitrogen introduction, the turbo pumps were stopped and gate valve of the cryopump was closed. There are no gate valves at head of turbo pumps. Purity of the nitrogen source is more than 99.99995 vol%. Nitrogen line was connected with ICF flanges and baked at around 120 ºC. The introduced gas was pumped by the portable pump unit which is consisted with turbo pump and scroll pump. Pressure is controlled by controllable variable leak valve.

Cavity was high pressure rinsed and double packed at class 1000 clean room before heat treatment. The pack was opened just before installing the J-PARC furnace. Figure 3 shows the cavity and samples installed in the furnace. Inner surface of the cavity was covered with niobium cap and foil. These careful treatments shut out the particles and contamination. The cavity was mounted on the Inconel stage and supported by niobium V block plate. Temperature is controlled far from the cavity. Thermoelectric coupler monitor was inserted between Inconel stage and V block. Various size niobium samples are heat treated with niobium cavity at same time. Niobium samples are shaved out from fine grain niobium plate. Samples are also covered with niobium.

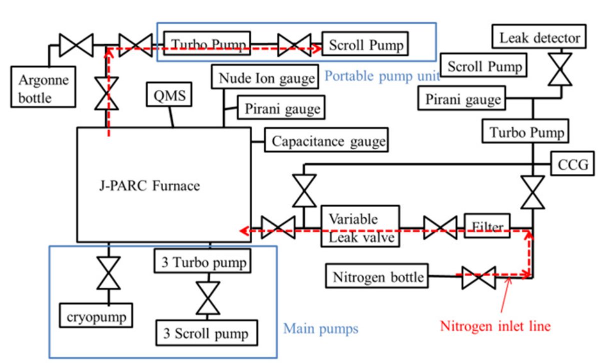

Figure 2: Diagram of J-PARC furnace.

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Figure 3: Pictures of J-PARC furnace.

ANALYSIS OF N-DOPE HEAT TRATED AT KEK LARGE FURNACE

Nitrogen-doping using KEK furnaces were not succeeding. GD-OES and X-ray were used to analyse the using sample surface. Samples and cavity are heat treated separately. The cavity and samples are covered by niobium. Pressure is controlled by controllable variable lead valve. The main pump was pumped the nitrogen during the nitrogen introduction.

Figure 4 shows the nitrogen treatment for the single cell fine grain cavity. The doping parameter is referred FNAL, J-lab, Cornell parameter [3, 4, 5]. The nitrogen was doped 20 minutes at 2.7 Pa after keeping 3 hours at 800 °C. The cavity was kept 30 minutes at 800 °C after doping and naturally cooled down to room temperature. Figure 5 shows the vertical test (VT) results. VT1 was the ILC recipe. After nitrogen doping, the surface was electro polished by 15μm, 15μm and 5μm respectively every in VT2, 3, 4. The surface treatments of the cavity after electro polishing were 15 minutes ultrasonic rinsing and 180 minutes high pressure rinsing. After assembling the cavity, VT2 was applied baking 48 hours at140°C and VT2 and VT3 were not applied baking. There is no x-ray observed in all VT.

Figure 6 is the GD-OES analysis of nitrogen doped sample using KEK large furnace. Nitrogen was detected up to 20 μm and below the detection limit at more depth. Hydrogen was much lower after heat treatment. It is clearly understood the nitrogen doping effect. Carbon was doped after heat treatment. It suggests the contamination from oil pumps was doped deeply and formed poor niobium surface. The carbon also detected by XPS (Fig. 7). XPS was measured at room temperature. X-ray source was Al K-alpha (1486.6eV). The depth direction was measured by spattering. Spattering rate is 9.1nm/min estimated from SiO2 reference sample.

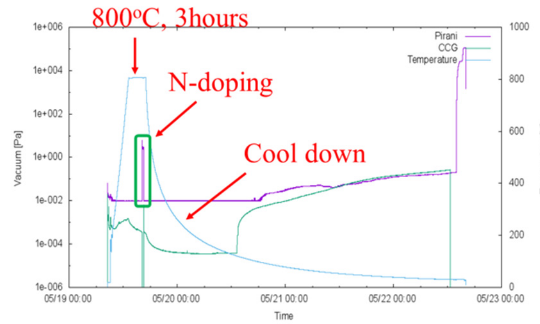

Figure 4: Nitrogen doping condition.

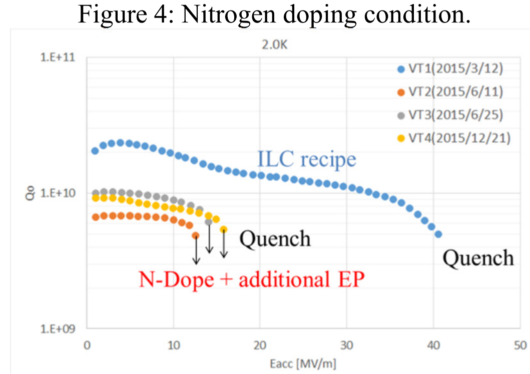

Figure 5: Vertical test results of nitrogen doped cavity treated in the KEK large furnace.

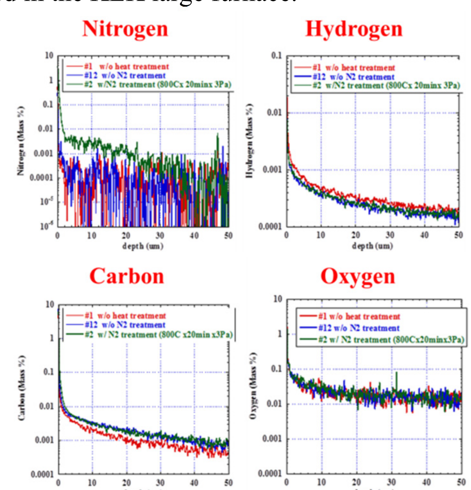

Figure 6: GD-OES analysis of nitrogen doped samples treated in KEK large furnace.

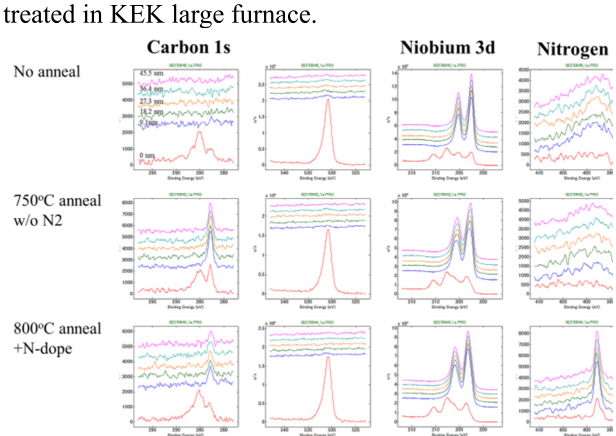

Figure 7: XPS of nitrogen doped sample treated in KEK large furnace.

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N-DOPE AND N-INFUSION AT J-PARC FUNACE

Nitrogen doping was succeeded by using the J-PARC furnace. Figure 8 shows the temperature and pressure trend of the J-PARC furnace. The ultimate pressure after 800 °C annealing reached to 1×10^{-6} Level. This is one order lower than KEK furnaces. We think the base pressure and oil free pumps are critical element in the success of the nitrogen doping. The doping parameter is also J-lab parameter. The nitrogen was doped 20 minutes at 2.7 Pa after keeping 3 hours at 800 °C. The cavity was kept 30 minutes at 800 °C after doping and naturally cooled down to room temperature. However, nitrogen infusion was not succeeded. Nitrogen infusion parameter is also FNAL parameter [6]. Nitrogen was infused after normal annealing 3 hours at 800 °C. Temperature was kept 120 °C during infusion. Cavity temperature was controlled ±5 K. The base pressure during the nitrogen infusion is at around 1.5×10^{-2} Pa. There is no valve between main turbo pump and furnace chamber. Pumping speed of the portable pump unit was not enough. These are reasons why the base pressure increases during nitrogen treatment. Nitrogen was infused 3.3 Pa, 48 hours at 120 °C. In order to evaluate base pressure effect during the nitrogen infusion in the J-PARC furnace, the cavity was heat treated shown in 8(a) and (b) and evaluated in vertical tests.

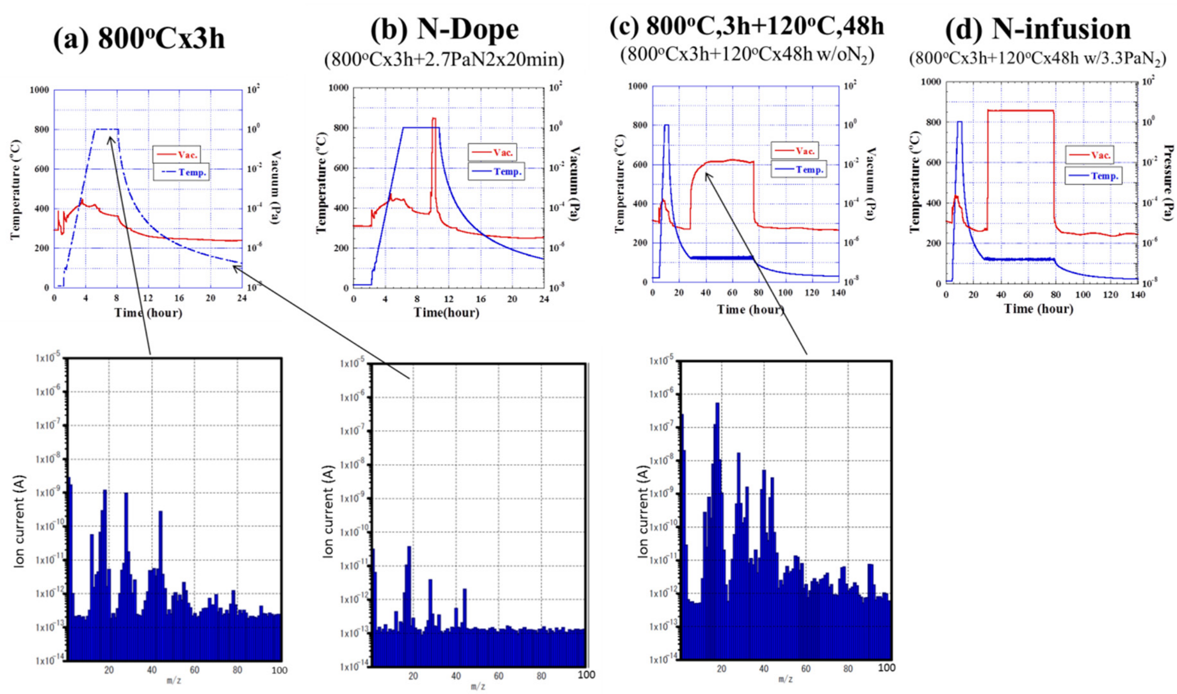

Figure 8: Temperature and pressure trend of J-PARC furnace. (a) Normal annealing 800oC × 3 hours. (b) N-dope. (c) Simulated nitrogen infusion heat treatment without nitrogen introduction. (d) Nitrogen infusion.

Surface treatment after nitrogen doping was 15 µm electro polishing, 3 hours high pressure rinsing. Cavity was dried in class-10 clean room and assembled after rinsing. 120 °C baking was not applied after assembly. In the VT cryostat, magnetic field cancelled with solenoid coil (<1mG) [7]. The cavity was cooled down with thermal gradient by heater. Figure 9 shows the VT results. Q value was 1.5 times higher than ILC recipe at 15 MV/m. The nitrogen doped cavity was quenched at 15 MV/m in 2 K. Other temperature measurements are stopped to avoid the quench. X-ray was not detected in all VT. This is the first success in Japan.

Figure 10 shows the VT results of nitrogen infusion and without nitrogen infusion. These measurements can compare the nitrogen infusion effects in J-PARC furnace. Heat treatment condition is shown in Fig. 8 (c) and (d). Only high pressure rinsing was applied after heat treatment. The cavity was dried 18 hours and assembled in class-10 clean room. Baking also not applied after assembly. In the vertical cryostat, magnetic field and temperature was controlled same as nitrogen doped VT. Both nitrogen infusion and simulated nitrogen infusion without nitrogen show lower results for Q value and gradient. Nitrogen infusion cavity was quenched at 33 MV/m. Simulated nitrogen infusion was quenched at 35 MV/m. There is no x-ray observed in all VTs. We suspect the impurities included the base pressure during the nitrogen infusion caused the Q slope.

Sample surfaces were analysed by SIMS (Fig.11). It includes no heat treatment, normal ILC recipe (800 °C × 3 hours annealing) and N-infusion. Simulated nitrogen infusion sample is not included. Nitrogen seems same in all samples. Hydrogen was degassed in heat treated sample. The nitrogen infused sample shows a higher nitrogen concentration up to the depth 50 µm than the other samples.

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However there is a doubtful in depth direction analysis of surface roughness. Further study of the surface analysis is necessary to confirm reliability of the surface analysis.

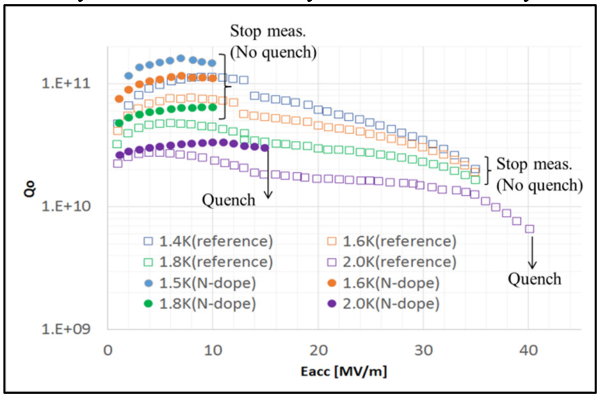

Figure 9: Vertical test results of nitrogen doped cavity at J-PARC furnace.

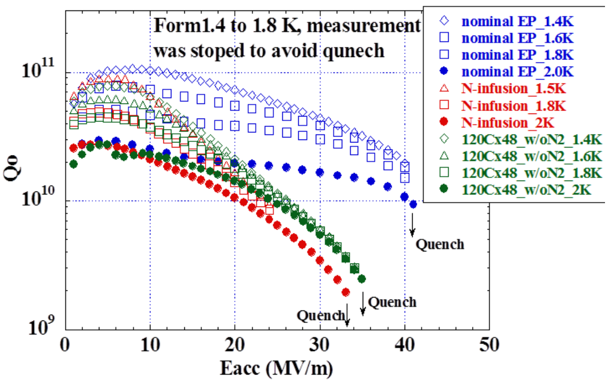

Figure 10: Vertical test results of nitrogen infusion cavity at J-ARC furnace.

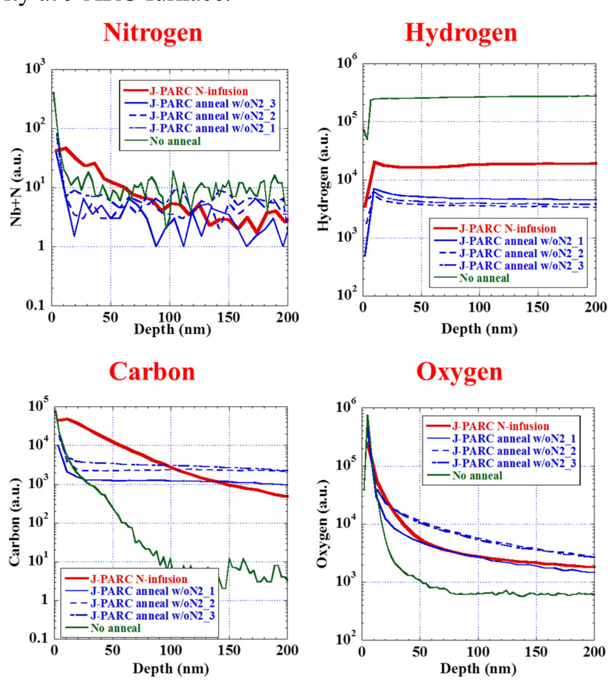

Figure 11: SISM analysis of heat treated samples.

SUMMARY

N-dope and N-infusion study is on-going at KEK to realize high performance of SRF cavities. N-dope was not succeeded by using KEK furnace. We suspect the cavity surface was contaminated with hydro-carbon from oil pumps. Sample analysis also supports this. Using J-PARC furnace, N-doping was successful. High-Q was obtained for 10-15 MV/m. N-infusion was carried out. Degradation occurred at more than 5 MV/m. We suspect the problem is high base pressure during infusion. We will try to push to realize N-infusion technique for high performance SRF accelerators.

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