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HEAT TREATMENT FOR A PROTOTYPE HALF-WAVE RESONATOR CAVITY*

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Abstract

Performance of a superconducting cavity, which is made of niobium, tends to be degraded easily depending on the surface resistance. Surface residual resistance of the cavity can be increased during the cavity fabrication process such as pressing, cutting and welding, because these multiple steps can lower the crystallinity of the cavity surface not only by disordering the lattice atoms but also by supplying lattice atoms with impurity atoms. Among the impurity atoms, hydrogen is a well-known impurity that lowers the cavity performance in the form of Q-disease. High temperature heat treatment was performed to remove impurity gas (mainly hydrogen) from the surface of a cavity, and to anneal the cavity to increase the crystllinity. Optical images of the heat treated niobium sample were investigated. Crystallinity was improved after the heat treatment by investigating X-ray diffraction (XRD), thus it confirmed that the annealing was successfully carried out. The residual gas analysis (RGA) was investigated and it confirmed that hydrogen was removed successfully during the heat treatment.

INTRODUCTION

For a LINAC, called "RAON" adopts four types of superconducting cavities: quarter-wave (81.25 MHz), half-wave (162.5 MHz), and two different single-spoke resonators. All the superconducting cavities operate with an alternating current of high frequency; thus, the surface resistance must be extremely low to maintain the superconductivity. Superconductivity will vanish easily if the temperature of any part in the cavity reaches a critical temperature (Nb_{Tc} is around 9.2K).

The temperature-dependent BCS resistance decreases as the temperature goes down but the residual resistance is independent of the temperature [1]. The residual resistance due to the imperfection of a raw superconducting material is almost fixed during the production. However, multiple steps for fabricating cavities can inevitably increase the residual resistance additionally by creating other sources such as a dislocation, a vacancy and a grain boundary. Therefore, the cavity fabrication must be carefully processed not to have additional surface resistance [2, 3].

One of the surface treatments to improve the surface resistance, the high temperature baking is well known for both annealing the cavity and removing hydrogen from the surface of the cavity. In case of annealing, it is for recovering the crystallinity of the cavity by moving disordered atoms to their original lattice points. In case of removing hydrogen, it is for outgassing impurity gas, which acts as a potential scattering center as a form of hydride at low temperatures.

From the earlier studies, hydrogen, which is easily introduced into the cavity surface during the chemical etching, is well known for deteriorating cavity performance in the form of Q-disease [4]. The residual gas analysis (RGA) and the X-ray analysis were performed after the heat treatment to check the baking effect. In this study, we report how the heat treatment process was carried out. Furthermore, we discuss the results along with the cavity performance in terms of the residual gas and X-ray analyses.

FURNACE SETUP & EXPERIMENTAL

Specifications of a vacuum furnace for the heat treatment are listed Table 1. Figure 1 shows the vacuum furnace.

Table 1: Specifications of Vacuum Furnace

Items	Specification	Unit
Dimension	$1 \times 1 \times 3$ (W×L× H)	Meter
Vacuum	10^{-6}@650°C, 10^{-8}@25°C	Torr
Temperature	Max. 1400°C,	Celsius
Heating element	Molybdenum	Mo

Figure 1: Vacuum furnace for heat treatment:cavity is loaded horizontally.

And Fig. 2 shows the horizontally loaded half-wave resonator cavity (HWR) with a titanium box in the furnace. HWR cavity is in contact with the titanium box through alumina (Al_2O_3) frame in order to avoid the chemical reaction between the cavity and the titanium box, as shown in Fig. 2. Two thermocouple wires are connected to titanium box, and two additional thermocouple wires are connected to cavity flanges, which are made of SUS316L.

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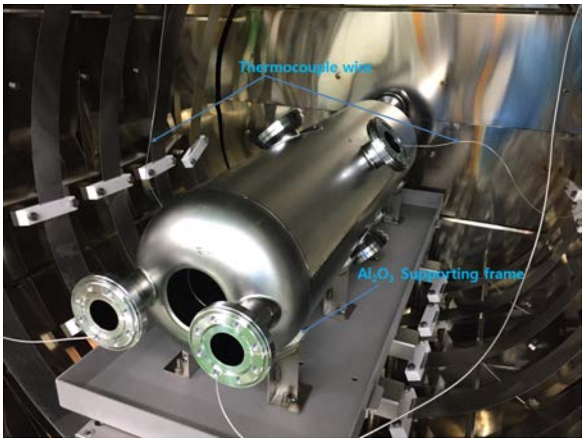

Figure 2: Loaded HWR cavity in the furnace: two thermocouples are connected to the cavtiy's flanges, two additional thermocouples are connected to titanium box, cavity is contact to the titanium box through alumina frame.

The titanium box is shown in Fig. 3, it is useful for removing hydrogen during the heat treatment since titanium has a relatively low vapor pressure compared to the niobium at a temperature of 650°C. The temperature for heating cavity is 650°C, and the ramping-up speed is 10°C per minute. Once the temperature reaches to 650°C, heating lasts for 10 h under the vacuum of about 10^{-6} Torr. The vacuum furnace is cooled naturally once the heat treatment is over. Two days are required for the cavity to cool down to 50°C naturally. For measuring the outgassed gases from the cavity, we monitored the partial pressures during the entire heat treatment by using a RGA analyzer (QME220, Pfeiffer Vacuum Co.). For investigating the effect of the heat treatment on the microstructure of the cavity, we prepared niobium samples having sizes of around 1×1 cm^2. All samples were cut from the beam port cup part by using the electrical discharge method (EDM). Samples were put on an alumina plate having the size of 10×10 cm^2 so as to avoid the chemical bond between the samples and the titanium carrier.

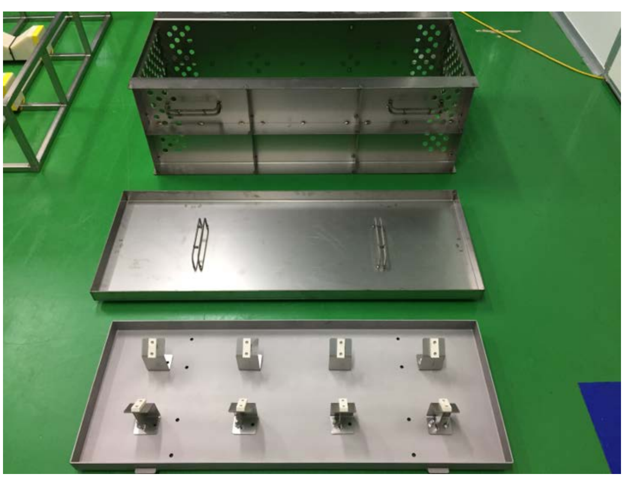

Figure 3: Titanium box: body, top, and bottom part.

Figure 4 shows the Nb samples in the furnace for XRD analysis. Optical images were taken after heat treatment by using an optical microscope (Xi-Cam, VesticVison Co.). The grain sizes of the samples were also measured by using an optical microscope. X-ray diffraction analysis was performed to investigate the effect of the heat treatment on the sample's crystallinity.

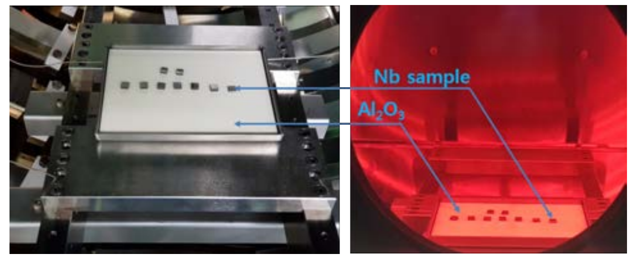

Figure 4: Niobium sample setup for XRD analysis: samples are mounted on the alumina plate (left), samples are heated in the furnace (right).

XRD AND RGA

Crystallinity means how much atoms are ordered in the microstructure, thus, the more the atoms are ordered, the more the crystallinity increased. High crytallinity also means that low resistance because carriers can travel long distance without colliding with scattering centers. Thermodynamically, heat energy lowers an activation energy of disordered atoms to return to their lattice points. However, at the same time, a grain growth must be suppressed. which means grains must not be grown to a great extent during the heat treatment. A mechanical strength strongly depends on the microstructure. The microstructure of fine grains have a high yield strength than that of the large grains. Therefore, annealing cavity must be carefully performed to satisfy two purposes simultaneously, one is to increase crystallinity (decrease residual resistance), the other is to suppress the grain growth of the cavity. It is known that hydrogens in the cavity can easily form hydrides on the cavity surface unless the cavity is cooled down quickly, these hydrides increase surface resistance only to deteriorate cavity performance. This is reported as Q-disease. Thus, removing hydrogen from the surface of niobium cavity must be performed in order to avoid the Q-disease phenomenon.

Niobium sample after the 10-h heat treatment was investigated by using an optical microscope, and the Fig. 5 shows the optical images. Also, XRD analysis is also shown in Fig. 6. The average grain sizes in Fig. 5 lie in the range of between 30 ~70 µm. Since the average grain size of the niobium was less than 50 µm from the certificate supplied by niobium company (ATI, USA), Thus, it was confirmed that the heat treatment was successfully carried out in terms of microstructure because grains did not grow to a great extent during the heat treatment.

The X-ray diffraction analysis was performed to confirm the effect of the heat treatment on the crystallinity of the niobium. The measured XRD patterns of the sample before

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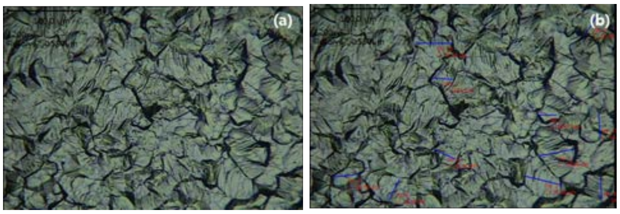

Figure 5: (a) Optical images of the surface of the heat treated sample and (b) grain sizes are drawn in the figure (a) with optical microscope's software. The optical resolution is X1200.

and after heat treatment are shown in Fig. 6. It shows that intensity counts from all 2θ angles were increased after heat treatment, which means that the degree of the crystallinity increased. Also, the FWHM decreased after heat treatment, which means the XRD peak was sharpened. In addition, the relative intensity of the 55° planes increased more than those of the other planes did.

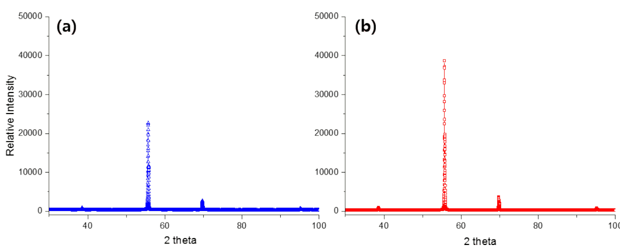

Figure 6: X-ray diffraction analysis: (a) XRD results with no heat treatment and (b) XRD results with a 650°C for 10 hours.

Partial pressures of a few typical gases in the furnace were monitored during the heat treatment by using a residual gas detector. Figure 7 shows the RGA results. Hydrogen showed the highest partial pressure peak about 10^{-4} Torr, consequently, the hydrogen determined the total pressure of the furnace. RGA result shows qualitatively that large amounts of hydrogen were outgassed during the heat treatment.

SUMMARY

Heat treatment of the prototype half-wave resonator cavity was carried out for 10 hrs at 650°C. Nb samples were prepared to investigate the effect of the heat treatment on the niobium material. XRD and RGA analyses confirmed that the heat treatment was successfully carried out in a way

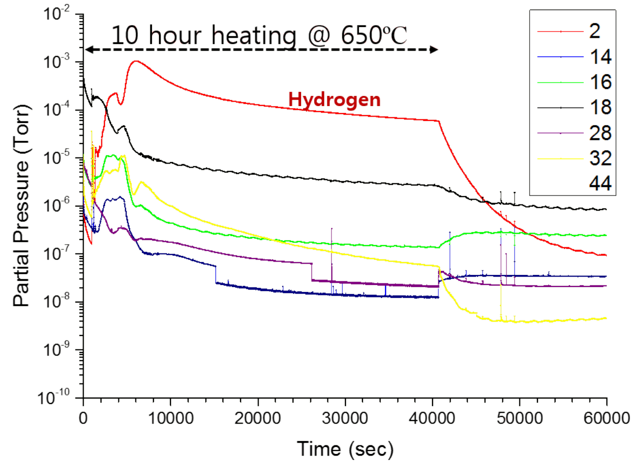

Figure 7: Residual gas analysis: partial pressures of the typical gases. A red graph shows the partial pressure of the outgassed hydrogen from the cavity.

that the cavity was well-annealed and hydrogen impurity was removed from the surface of the niobium cavity. The quality factor of the HWR cavity has been ready to tested in SRF test facility. The effect of the heat treatment on the dimension change of the cavity and the performance of the cavity will be reported.

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