# Effect of the Annealing Temperature for the Hydrogen Q-degradation on Superconducting Cavities

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### Abstract

Hydrogen Q-degradation was studied in niobium superconducting cavities prepared by barrel polishing, and electropolishing without annealing, though a fast cooling down of cavities. Cavity performance with various annealing temperature were tested using a 1.3GHz singlecell cavity to compare the effects of annealing temperature for hydrogen Q-degradation.

## **1** Introduction

It is known that the fast cool down is one possible procedure to prevent hydrogen Q-degradation for the high purity niobium cavities that exhibit this phenomena with slower cool down; stay relationally long time at 100-150K in the cooling procedure[1,2]. Hydrogen has been concentrated at niobium surfaces and it cause a phase transformation involving hydrogen at low temperature to yield Q-degradation[3].

It has been used buffing and mechanical grinding to remove surface irregularities before electropolishing(EP) at KEK. Recently, a barrel polishing was developed as a simpler method, instead of these mechanical preparations[4]. It is successfully used to 1.3GHz cavities with adopting annealing( $\sim$ 800°C), however, its effect on the Q-degradation isn't well known.

#### 2 Hydrogen Q-degradation

In order to make sure that the fast cool down is still feasible for preventing the Q-degradation to the cavity prepared by barrel polishing, EP without annealing, the following measurements were carried out by using a 1.3GHz single-cell cavity fabricated in our company[5]. This cavity was formed by deep drawing of high purity niobium sheets(thickness=2.5mm, residual resistivity ratio : RRR=200), then trimmed and electron beam welded.



Fig.1 1.3GHz single-cell superconducting cavity

Table 1 Single-cell cavity specifications

Frequency	1.3[GHz]
Rsh/Q	102[Ω]
G	274
Esp/Eacc	1.78
Hsp/Eacc	43.8[Oe/MV/m]

Figure 1 shows the cavity, and the specifications calculated by SUPERFISH code are summarized in table 1.

In the first experiment, the cavity was barrel polished by 30  $\mu$  m at the equator, then electropolished by 10  $\mu$  m on average and was rinsed with high pressure(HPR ; ~80kgf/cm<sup>2</sup>) deionized pure water. No annealing was carried out. After the treatments, it was evacuated to 1x10<sup>-9</sup> Torr and sealed in vacuum. The fast cool down to 4.2K was carried out in one hour. The result is shown in Fig.2 with mark :  $\bigcirc$ . It shows Q<sub>0</sub>(unloaded Q value) as a function of accelerating field gradient(E<sub>acc</sub>). Q<sub>0</sub> were sufficiently high at low field, however Q<sub>0</sub> began to drop at E<sub>acc</sub>=3MV/m. The maximum field( $E_{acc,max}$ ) was limited to 19.5MV/m due to thermal quench. This phenomenon which degrade the  $Q_0$  with  $E_{acc}$  increase was considered as the hydrogen Q-degradation. However it might be caused by surface contamination of the cavity.

In order to remove the surface contamination effectively, the cavity was rinsed with hydrogen peroxide(H<sub>2</sub>O<sub>2</sub>). This H<sub>2</sub>O<sub>2</sub> rinsing method had been tried for TRISTAN cavities at KEK. The result is shown in Fig.2 with mark :  $\Box$ . Q<sub>0</sub> were degraded more seriously and E<sub>acc.max</sub> was limited to 7.0MV/m.

To make sure that this phenomena was the Q-degradation, the cavity was held 2 hours at 100-120K which is most dangerous temperature for hydrogen Q-degradation. As shown in Fig.2 with mark :  $\triangle$ , Q<sub>0</sub> were degraded remarkably and E<sub>acc,max</sub> was limited to very low field due to thermal quench.

In the following test, the cavity was annealed at 770°C for 5 hours in the titanium box to degas hydrogen, and rinsed with high pressure deionized pure water. As shown in Fig.2 with mark : •,  $Q_0$  were  $2x10^{10}$ , and  $E_{acc,max}$  reached to 26.2MV/m.

According to these results, it was found that rinsing with  $H_2O_2$  did not help to cure the Q-degradation in our case.

# 3 Annealing temperature to prevent the Q-degradation

The cavity was treated several annealing temperature to compare the performances using the same cavity. In all experiments, the cavity was barrel polished by  $30 \,\mu$  m to make its surface to the same state. Then the cavity was electropolished by  $10 \,\mu$  m, annealed for 5 hours at temperature from  $400^{\circ}$ C to  $770^{\circ}$ C, and rinsed with high pressure deionized pure water. The cavity was held 1.5 hours at 100-120K before cooling down to 4.2K to make sure the condition of Q-degradation.

The results of these measurements are shown in Fig.3 and summarized in table 2. In these results, no Q-degradation were observed for annealing temperature higher than 500°C, and higher accelerating fields were obtained for higher annealing temperature. When  $E_{acc,max}$  were limited, normal conductivity exist locally on the cavity surface and its local heat was balanced with cooling of the cavity by superfluid helium. In these condition, no X-ray was observed at the maximum field.

On the other hand, in the case of  $400^{\circ}$ C annealing,



Fig.2 Performance of the cavity barrel polished





 
 Table 2 Summary of the cavity performance with annealing temperature

Temp.[°C]	$R_{res}[n\Omega]$	E <sub>acc,max</sub> [MV/m]	Q-degradation
770	6	26.2	no
600	9	24.8	no
500	7	17.4	no
400	4000	3	yes

 $Q_0$  were degraded remarkably about 4  $\mu$   $\Omega~$  and  $E_{acc,max}$  was very low. Serious Q-degradation appeared in the case of 400°C annealing.

According to these results, it was found that  $500^{\circ}$ C is lowest temperature to prevent hydrogen Q-degradation. And it was found that  $E_{acc,max}$  increase with annealing temperature increasing.

#### **4 Discussion**

The maximum field ( $E_{acc,max}$ ) was limited by the thermal quench due to the heating of the cavity surface. In the

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experiments, no X-ray were detected at the maximum field. It is considered that heat on the cavity surface is caused by niobium hydride, surface defects by electron beam welding and residual chemicals.

First, in case of niobium hydride, hydrogen absorbed in niobium bulk during EP are degassed by annealing, and amount of degassed hydrogen increase with temperature. The residual hydrogen in niobium bulk combines with niobium to form niobium hydride(NbH<sub>x</sub>). Power loss on the cavity surface increase with NbH<sub>x</sub>, so  $E_{acc,max}$  was different by the annealing temperature. Fig.4 shows hydrogen pressure during annealing procedure as a function of time. Characteristic diffusion time, t is estimated by formula(1) with diffusion constant, D and average length, R.

$$=\frac{R^2}{D}$$
 ...(1)

The degassed hydrogen amount were estimated by integrating hydrogen pressure above  $6 \times 10^{-6}$ Torr and normalized by the value of  $770^{\circ}$ C annealing. Table 3 shows degassed H<sub>2</sub>, assumed D as a function of temperature and calculated t for R=1mm. In the case of  $770^{\circ}$ C annealing, t is 1.4 hours, and it is much shorter than annealing time(5 hours). So it can be considered that hydrogen were sufficiently degassed at  $770^{\circ}$ C annealing, compared with other temperature.

Second, in case of surface defects, it was observed that Vickers hardness of cold-worked niobium recovered at about 600  $^{\circ}$ C according to the experiments by Tokyo Denkai Co. Ltd.. Therefore, it is considered that defects on the cavity surface are possible to recover by annealing.

Third, in case of residual chemicals, the cavity was rinsed with high pressure deionized pure water after annealing, so it is considered almost residual chemicals were removed[6]. If residual chemicals exist on the cavity surface, field emitted electrons were generated and X-ray were detected. However no X-ray was observed at the maximum field. Therefore it is considered that heat on the cavity surface is not caused by residual chemicals in these measurements.

#### **5** Conclusion

The characteristics of niobium superconducting cavity prepared by barrel polishing, and electropolishing without annealing was measured, and it was observed a light Qdegradation on the cavity, though a fast cooling down of the cavity. And it was found that rinsing with  $H_2O_2$  did not



Fig.4 Hydrogen pressure during annealing procedure

 
 Table 3 Summary of degassed hydrogen amount and characteristic diffusion time

Temp.[°C]	Degassed H <sub>2</sub>	t[hour]	D[cm <sup>2</sup> /s]
770	1.00	1.4	2.0x10 <sup>-6</sup>
600	0.30	6	4.5x10 <sup>-7</sup>
500	0.37	30	9.0x10 <sup>-8</sup>
400	0.16	280	1.0x10 <sup>-8</sup>

help to cure the Q-degradation. The effect of annealing temperature was studied, and it was found that the annealing procedure is necessary for the present surface treatment. It was suggested that 500  $^{\circ}$ C is lowest temperature to prevent hydrogen Q-degradation.

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