

HYDROGEN Q-DISEASE AND ELECTROPOLISHING

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Abstract

To find out the mechanism of the hydrogen absorption during electropolishing (EP), degree of hydrogen Q-disease of an L-band niobium (RRR (Residual Resistivity Ratio) = 200) single-cell cavity was compared between 1) continuous EP, 2) intermittent EP and 3) dipping into EP solution. We found that hydrogen Q-disease did not occur in case 1) and occurred in case 2) and 3). We also found that with the niobium (RRR = 200) samples treated by the same way as above 1), 2), 3), hydrogen absorption was not observed in case 1) and was observed in case 2) and 3).

During the measurement of the cavity performance, we noticed that a baking (130°C, 48hr) was helpful to ease the seriousness of the hydrogen Q-disease.

Concentration of the hydrogen was measured on the niobium samples that was treated with EP removing different amount of surface material (after hydrogen absorption through dipping into acid solution) and the results suggested that the absorbed hydrogen was localised in the surface layer of the samples.

1 INTRODUCTION

If the cooling-down speed is slow, the precipitation of the dissolved hydrogen occurs around 100 K and this increases the surface resistance of a niobium cavity. This phenomena is called as “hydrogen Q-disease” and well studied around 10 years ago [1] (An increase of the surface resistance is also observed with fast cool-down (about 30 min from room temperature to 4.2 K) if an amount of above-threshold hydrogen exists). But the detail of the mechanism is not yet clear.

At the time of TRISTAN R&D, continuous horizontal electropolishing of niobium superconducting cavities was developed in KEK [2]. The EP requires extra tools such as an electrode etc. and takes longer time when compared with chemical polishing. Probably because of that drawback, EP had not been applied to niobium cavities as the standard treatment except at KEK. But recently, superiority of EP over chemical polishing on high gradient field was observed [3] and EP attracted the attention. Now, EP is becoming to be a standard surface treatment all over the world and some new EP systems were introduced during this workshop.

It had been recognised that during old intermittent EP process, hydrogen is absorbed into the niobium cavity

and this hydrogen causes the hydrogen Q-disease. In the TRISTAN project, there was not sufficient time to check if the hydrogen absorption occurs for the new continuous horizontal EP process and annealing was done for the all niobium cavities.

After the TRISTAN, we started looking into better alternatives for surface treatment. If the hydrogen absorption does not occur during surface treatments, annealing can be omitted and the cost and the time will be reduced. At first, we examined a relationship between the hydrogen Q-disease and the EP. Using an L-band niobium single-cell untreated cavity (no barrel polishing as a pre-treatment) of RRR = 200, we checked if the hydrogen Q-disease occurs with present continuous EP. And the hydrogen Q-disease did not occur after having removed approximately 200 microns from the surface (Figure 1, [4]). On the other hand, hydrogen absorption was observed in a niobium sample that had been put into the cavity during the centrifugal barrel polishing (a pre-treatment of EP) for 8 hours [5]. But if absorbed hydrogen is localised within the surface layer of the cavity and the mechanism of the hydrogen absorption is found out, the hydrogen Q-disease may be cured by a surface treatment.

So we started to find out the mechanism of hydrogen absorption and the location of absorbed hydrogen. In this paper, we present what we found experimentally about the hydrogen Q-disease of a niobium cavity and the hydrogen absorption with the niobium samples that was treated with EP.

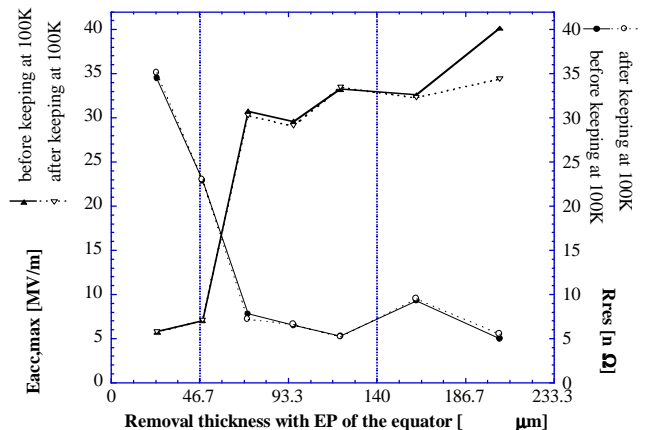


Figure 1: Hydrogen Q-disease did not occur with continuous EP of 200 microns.

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2 EXPERIMENTAL

2.1 Evaluation on the Hydrogen Q-disease

We evaluated the level of the hydrogen Q-disease as the increase of a residual surface resistance (R_{res}) caused by a process of keeping a cavity at 100K for 16 hours. We also evaluated the effect of a surface treatment as the increase of the level of the hydrogen Q-disease before and after that treatment. Hence, 4 different measurements of the R_{res} are required to evaluate one treatment.

2.2 Measurement of hydrogen concentration in niobium samples

Hydrogen concentration in test samples was measured by melting niobium samples and emitted gas was analysed using a gas chromatography. In this method, we can not separate chemically bonded hydrogen from absorbed hydrogen. We can see only the average of the hydrogen concentration in a bulk sample.

3 RESULTS AND DISCUSSION

3.1 Level of Hydrogen Q-disease through continuous EP, intermittent EP or dipping into EP solution

Table 1: Level of hydrogen Q-disease through various EP

Surface treatment	R_{res} and ΔR_{res} [n Ω] after and before keeping at 100 K for 16 hr				Δ (ΔR_{res}) Difference of level of hydrogen Q-disease
	before the surface treatment		after the surface treatment		
	R_{res} before keeping at 100 K	ΔR_{res}	R_{res} before keeping at 100 K	ΔR_{res}	
	R_{res} after keeping at 100 K		R_{res} after keeping at 100 K		
dipping in 30 °C EP solution for 3 hr	5.8	0.3	5.4	0.9	+0.6 (increased a little)
	6.1		6.3		
dipping in 45 °C EP solution for 3 hr	5.4	0.9	4.5	6.8	+5.9 (increased)
	6.3		11.3		
continuous EP 30 °C for ~1.5 hr (33 μ m)	4.5	6.8	5.7	2.5	-4.3 (decreased)
	11.3		8.2		
intermittent EP 30 °C for 6hr (82 μ m)	5.7	2.5	8.7	15.1	+12.6 (increased)
	8.2		23.8		
continuous EP 30 °C for ~2.5 hr (55 μ m)	8.5	11.6	5.3	10.9	-0.7 (decreased a little)
	20.1		16.2		
continuous EP 30 °C for ~5 hr without cathode bag (110 μ m)	5.3	10.9	8.1	14.4	+3.5 (increased)
	16.2		22.5		

To find out a mechanism of hydrogen Q-disease in EP, an effect on a level of the hydrogen was measured for 3 kinds of treatments (continuous EP, intermittent EP and dipping into EP solution). To evaluate the effect of each treatment on Hydrogen Q-disease, we measured R_{res} before and after keeping the sample at 100K (for 16 hours). Results are shown in table 1. These experiments were done using the same cavity (RRR = 200) over and over.

In table 1, two data on continuous EP were when Teflon cloth cover (cathode bag) was applied on cathode (for etching), and the bottom data was also continuous EP but without the cover. (The cathode bag was found to reduce irregular etching due to hydrogen bubbles formed around the cathode). With the continuous EP (with the bag), the level of hydrogen Q-disease did not increase. With the cases of intermittent EP and dipping into EP solution, however, the level increased. It seems that hydrogen was not absorbed if voltage was applied between the cathode and the cavity since the cavity was kept at positive potential and hydrogen ions are also positively charged. Comparing these three continuous EP data, the cathode bag seems to be effective to reduce the hydrogen absorption.

3.2 Hydrogen Absorption with continuous EP, intermittent EP and dipping into EP solution

Figure 2 below shows hydrogen absorption with various EP.

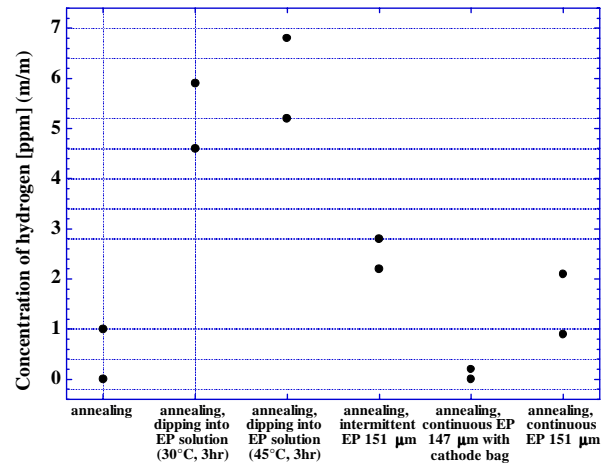


Figure 2: Hydrogen absorption with various EP

To check the relation between the hydrogen Q-disease and hydrogen absorption, we measured the concentration level of hydrogen in the samples that was treated with continuous EP, intermittent EP or dipping into EP solution. (Figure 2) Here, all samples were annealed at 750 °C to remove hydrogen before the treatment. The precision of this measurement was about 2 ppm.

Hydrogen absorption was not observed in the case of continuous EP with cathode bag. Hydrogen absorption was observed in the case of intermittent EP and of dipping into EP solution. No cathode bag was used except for the 5th case in Figure 2 (Cathode bag was used for usual EP of a cavity. The last data of Figure 2 corresponds to the last data of Table 1.), a good agreement was observed between the hydrogen Q-disease of a cavity and hydrogen absorption of niobium samples.

3.3 Degree of Hydrogen Q-disease and Baking

During the experiment shown in Table 1, we noticed that a baking of about 130 °C seems to be effective to reduce the degree of hydrogen Q-disease as shown in Table 2.

Table 2: Effect of baking

Surface treatment of the cavity	Rres and ΔRres [mΩ] after and before keeping at 100 K for 16 hr				Δ (ΔRres) Change of degree of hydrogen Q-disease
	before the surface treatment		after the surface treatment		
	Rres before keeping at 100 K	ΔRre	Rres before keeping at 100 K	ΔRre	
	Rres after keeping at 100 K		Rres after keeping at 100 K		
HPR, baking 64h40m			8.5	11.6	
			20.1		
continuous EP 55μm 2.5hr, baking 26h30m	8.5	11.6	4.6	28.5	+16.9 increased
	20.1		33.2		
baking 38h10m (total 64h40m)	4.6	28.5	5.3	10.9	-17.6 decreased
	33.2		16.2		

3.4 Hydrogen Absorption with EDM

Samples of various thickness (from 0.1 mm to 2.5 mm) were cut out from a niobium sheet of RRR = 200 and its hydrogen concentration was measured. The measurement was done twice about different niobium sheets. Here, the area of EDM is almost the same for all samples. Assuming that the amount of absorbed hydrogen is determined from the area of EDM and the absorbed hydrogen does not go out of the samples, the relation of thickness of the samples and hydrogen concentration becomes to be as follows:

$$H = C/t,$$

where “H” is the hydrogen concentration by weight, “C” is the constant and “t” is the thickness of the samples.

However, the real data shows an equation to be as follows (Figure 3):

$$H = 11/t + 4.5,$$

Here, the two constants “11 [ppm•mm]” and “4.5 [ppm]” were obtained by averaging parameters of the fits of the two measurements. If we can assume that the absorbed hydrogen is concentrated in the surface layer of 30 microns [6], the hydrogen concentration was about 400 ppm.

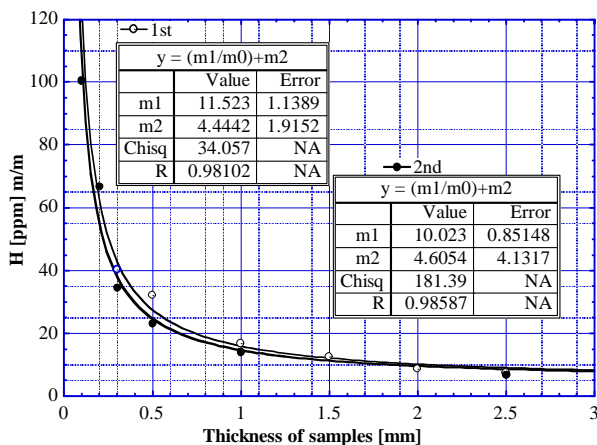


Figure 3: Hydrogen concentration of niobium samples that were cut out with various thicknesses with EDM

3.5 Depth of the hydrogen Absorption

If the absorbed hydrogen is located in the surface layer, we may be able to cure the hydrogen Q-disease through surface treatment. To find out if the absorbed hydrogen is localised, we prepared niobium samples (RRR = 200) of different thicknesses so that final concentration measurements were done on the same thickness (0.5 mm) after removal of different thickness from the surface using continuous EP. Hydrogen absorption on the samples had been achieved through dipping into EP solution (30 °C, 3 hr).

The result is shown in Figure 4. Hydrogen seems to be localized in the surface area of the samples. To find out the depth of the hydrogen absorbing layer, we are preparing the next measurement about some more details.

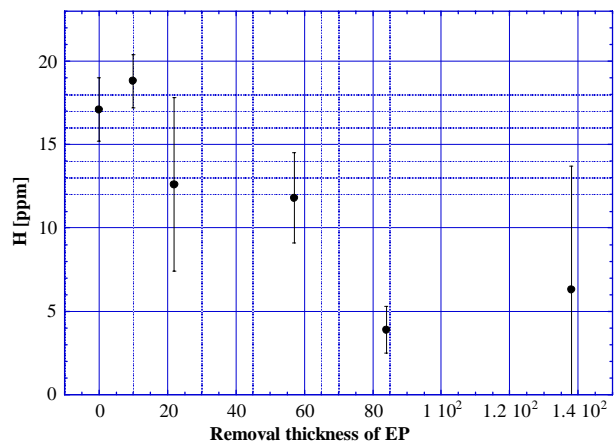


Figure 4: Hydrogen concentration in niobium samples that were polished by EP with various thickness removal.

4 CONCLUSION

With a niobium cavity of RRR = 200, hydrogen Q-disease was not observed after continuous EP that had removed approximately 200 microns from the surface. And a level of the hydrogen Q-disease was increased after intermittent EP or after dipping into EP solution. On the other hand, with niobium samples of RRR = 200, hydrogen absorption was not observed for 150 microns of continuous EP with cathode bag and hydrogen absorption was observed for intermittent EP and dipping into EP solution. This shows a good agreement.

Baking at 130 °C and using a cathode bag during EP seem to be effective in reducing the level of the hydrogen Q-disease.

The absorbed hydrogen after dipping into EP solution seems to be localised within 100 microns from the surface. We are preparing the next measurement about some more details.

5 ACKNOWLEDGEMENT

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