

MEASUREMENT OF HYDROGEN ABSORBED IN NIOBIUM

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Abstract

Hydrogen absorbed in niobium was measured using effect of hydrogen Q-degradation. A niobium cavity was designed and manufactured for this experiment. Hydrogen was introduced from outside of the cavity by electrolysis of diluted sulfuric acid on the outer surface of the cavity with an anode made by stainless steel. The Q-factor is one of the most unstable property of superconducting cavities. Especially, the reproducibility of Q-factor cannot be so expected after disassembled and reassembled it. In this experiment, the Q-factor was measured without disassembling, because hydrogen was introduced from outside of the cavity. The Q-degradation was observed successfully. And the Q-factor becomes worse and worse, when hydrogen was introduced more and more.

To estimate the quantity of hydrogen which is absorbed in niobium, small and thin niobium samples were prepared. They were warmed by the energizing heating in vacuum after having introduced hydrogen. The out-gas was analyzed by QMS.

This method can be applied to measure the absorbed hydrogen during electro or chemical polishing of cavities without influence of changing the surface morphology.

MOTIVATION

Superconducting cavities were installed in many accelerators for high energy physics such as KEKB, CEBAF, LHC, etc. And many project such as ILC, some of FEL project, etc have plan to adopt it. The superconducting cavity can provide high electric field with small RF dissipation. It required high smoothness and cleanness to make high performance. If it is cooled down slowly what spend more than 1 hour through between 150 and 60K, to degas hydrogen is also required to avoid Q-degradation [1].

Because the cavity require high cleanness, the Q-factor changes just is disassemble and reassemble [2]. The measured result of cavity performance include contributions of the smoothness, cleanness, absorbed hydrogen, and others. It is difficult to separate them. Previous research suggest relationships between the quantity of absorbed hydrogen and method of surface treatment. But if the inner surface of cavity were treated by each method, all of condition changes simultaneously. It is impossible to compare the quantity of absorbed hydrogen between each surface treatments through the measurement of cavity performance. On the other hand, absorbed hydrogen can be diffused in the metal at normal temperature. The distribution in the niobium which thickness is 5mm becomes almost flat after

a few hours [3]. Therefore, the hydrogen introduced from outer surface affect same as one from inner surface. In this method, it is expected the quantity of absorbed hydrogen can be measured without changing the cleanness, smoothness and some other condition of the cavity.

INTRODUCTION OF HYDROGEN

As a method to introduce hydrogen to niobium, electrolysis of diluted sulfuric acid is known [4]. It was confirmed that the method can introduce hydrogen to niobium. The shape of niobium samples for that experiment is shown in figure 1. The thickness of them were $20\mu\text{m}$. When electro-

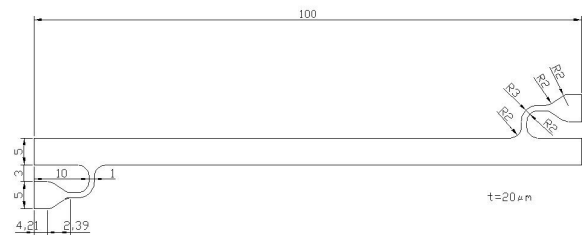


Figure 1: Schematic view of the niobium sample

lysis was done, the sample which works as a anode put on isolator plate. A stainless steel plate which works as a cathode was set up 5mm apart from the niobium sample. The setup was dipped into 0.1 mol/l sulfuric acid. The current of 1.5A was applied to the setup.

The sample which introduced hydrogen heated in vacuum to purge the hydrogen. When the current of 3A was applied to the sample, the temperature becomes $550\text{ }^\circ\text{C}$. Dependency between the peak partial pressure of hydrogen and electrolysis time is shown in figure 2. Hydrogen was obviously introduced by electrolysis.

COAXIAL CAVITY

Design of coaxial cavity

A coaxial type cavity was designed for this experiment. It is shown in figure 3 To introduce hydrogen easily, it have a pool to do the electrolysis or other surface treatment. It use TEM coaxial propagation mode. And that's length is half of wavelength of operation frequency. As shown in figure 3, it is connected at center of cavity. Operation mode have maximum electric field and no magnetic field there. No current across the connection. But Superconducting cavity is very sensitive to small dissipation due to it's high

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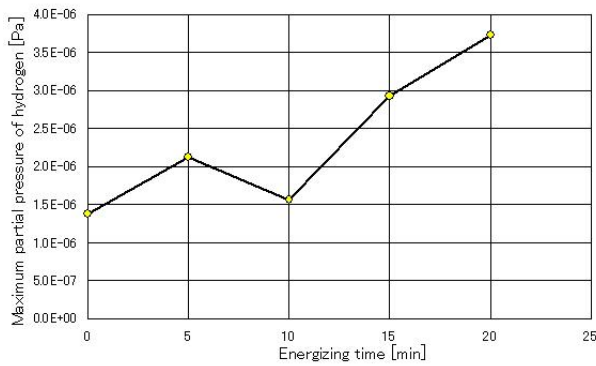


Figure 2: Dependency between the peak partial pressure of hydrogen and electrolysis time. Hydrogen was introduced by electrolysis. The peak hydrogen partial pressure tends to rise by lengthening the processing time.

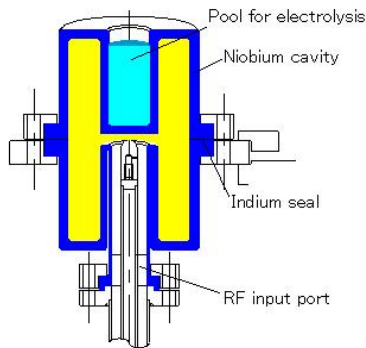


Figure 3: Schematic view of niobium cavity. It have

Q-factor. The cavity was not opened after achieved sufficiently high Q-factor in series of experiment. An RF input port is attached in the inner conductor of that. The gap length of inner conductor is 6mm. And that's center is shifted 0.5mm to increase the coupling.

Result of measurement

Hydrogen was introduced three times. To introduce the hydrogen, electrolysis was applied 30min every operation. The current density on the niobium surface was adjusted to 0.5mA/mm^2 at the reaction region. As shown in figure 4, the Q-degradation was observed. The Q-degradation was find to compare just after rapid cooldown and cooldown after maintain at 77K during 2days. Because the cavity was annealed to eliminate hydrogen, the Q-degradation was not occurred before introduce the hydrogen. After introduce the hydrogen, Q-degradation was observed when cavity was cooled after maintain 77K 2days. After first introduction, No Q-degradation was observed whether it was cooled rapidly. After second introduction, even the cavity cooled rapidly showed Q-degradation.

The Q-factor deteriorate by hydrogen introduction were achieved, anyway. The surface resistance was estimated as

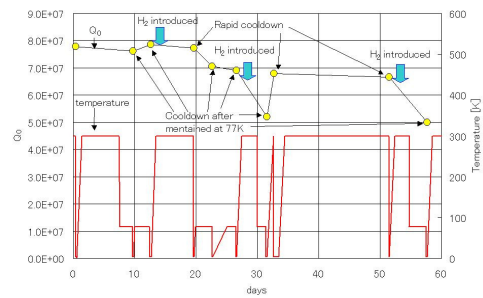


Figure 4: The transition of Q-factor is shown. The Q-factor after maintain 77K was deteriorated by hydrogen introduction. Difference of the Q-factor between rapid cooldown and cooldown after maintain 77K shows that the Q-degradation was caused by hydrogen.

shown in figure 5. Following assumption was applied to estimate surface resistance.

- The surface resistance in the cavity was uniform.
- The surface resistance of inner conductor attached the acid pool was changed by hydrogen introduction.

The surface resistance tends to increase every time hydrogen was introduced.

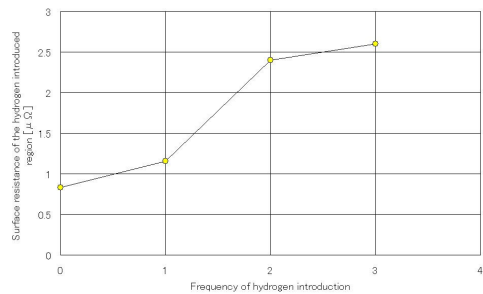


Figure 5: The transition of deteriorated Q-factor is shown. The results of a measurement when cooling after it was maintained at 77K were picked. The Q-factor shows the tendency to deteriorate by the hydrogen introduction.

CONCLUSION

It was confirmed that electrolysis of diluted sulfuric acid can be apply to introduce hydrogen into niobium. To calculate the quantity of introduced hydrogen, the vacuum system should be calibrated.

A coaxial cavity was designed to estimate quantity of absorbed hydrogen. Hydrogen was introduced into a part of the cavity wall from outside. The Q-factor was measured without cavity reassembling. And the surface resistance tends to increase by increasing the introduced hydrogen. It was confirmed that hydrogen introduced from outside of cavity can make Q-degradation.

It must experiments to confirm reproducibility. And the level of degradation must be normalized by the quantity of introduced hydrogen.

The electro or chemical polishing method should be applied to compare each other as a next step.

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