

VARIOUS RINSING EFFECTS TO MITIGATE CONTAMINANTS BROUGHT BY BCP ON NIOBIUM SRF CAVITY SURFACE

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Abstract

Various rinsing processes have been widely used after the surface treatment for niobium cavities. After the chemical treatment of the cavities some residues from the etching acids are left on the surface. To examine rinsing effects in detail, we carried out BCP experiment followed by post-polishing methods on a series of niobium samples at KEK. As a result of the BCP process [1,2] some contaminants like fluorine, carbon, etc. have been detected at the surfaces which may be the prominent cause of limiting the performance of SRF cavities. To remove these contaminants, various rinsing processes such as ultra pure water rinse, ultrasonic pure water rinse, alcoholic rinse, detergent rinse, high pressure water rinse (HPR) have been used after the BCP. In this article, we report the effect of HPR with different high pressures on the contaminants. The surface of the samples treated with HPR was analyzed using X-ray photo-electron spectroscopy.

INTRODUCTION

Niobium is the main material used for superconducting radio frequency (SRF) cavities to achieve high field gradient in particle accelerators. During the fabrication of SRF cavities many contaminants and damages would occur. In order to achieve good performances of SRF cavities these contaminants and defects are necessary to be removed and hence the cavity inner surface is chemically etched to get surface smoother. There are various methods to get the smooth surface by chemical etching such as buffered chemical polishing (BCP), electro-polishing (EP), electro-chemical buffing (ECB). After the surface treatment, some residues such as sulfur, fluorine from the etching acids and dust particles are attached on the surface which are hurdle in achievement of high accelerating gradient (E_{acc}) and high Q_0 value of the SRF cavities therefore post-polishing processes of inner surface has a great role in achievement of good performances of SRF cavities. After Chemical etching, cavities are passed through the rinsing procedure to remove chemical residues and dust particles from the surface.

In this paper, we try to present our efforts towards finding of the optimized pressure for HPR. In this regard, two samples were BCPed and subjected to HPR with different pressures. For the HPR experiment a commercial high pressure washer machine was used for the experiment which can attain the pressure of 15 MPa. After the experiment, sample surfaces were analyzed by

XPS (x-ray photo-electron spectroscopy). A force gauge was also developed to measure the actual pressure at certain nozzle distances.

SURFACE ANALYSIS SYSTEM

The surface analysis system is maintained at extremely high vacuum and connected with three loadlock system. The one of the loadlock chambers has the facility to attach a vacuum suitcase [3] (maintained at UHV) with it and sample can be transferred from the suitcase to the analysis chamber keeping in vacuum. Samples, which are necessary to keep in vacuum conditions always, can be analyzed by using this three loadlock system. A sample storage chamber, which maintains the base pressure of 10^{-8} Pa, is also connected to one of the loadlock chambers to keep remaining samples in carousel in UHV. Additionally, samples can also be mounted to one of the loadlock chambers directly from the atmosphere. An electron energy analyzer, an ion mass spectrometer, a x-ray source, an electron gun, an ion gun for depth profiling, an extractor gauge, and a residual gas analyzer are installed within one analysis chamber. The analysis system is capable of executing Auger electron spectroscopy, secondary ion mass spectrometry SIMS with argon ion etching, and XPS with probing area of 2 mm. Fig. 1 is the 3D diagram of surface analysis system. The XPS was utilized to investigate the BCPed surfaces.

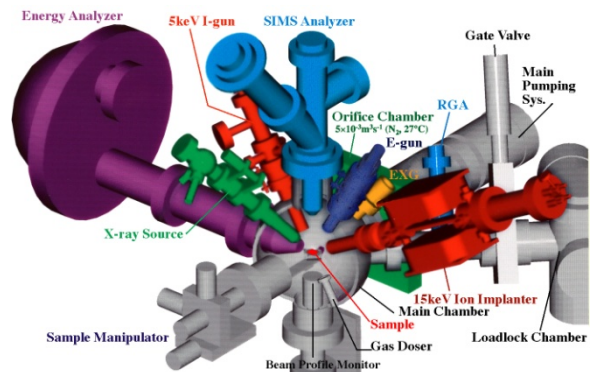


Figure 1: 3D diagram of surface analysis chamber.

EXPERIMENTAL PROCEDURE

We have conducted a BCP experiment followed by HPR with different pressures to explore the effect of different pressures of HPR on chemical contaminants left by the etching acids. In the experiment, two niobium (Nb) samples (rectangular type: 20x14x2.8) were BCPed. After BCP, both samples were subjected to high pressure rinsing at different pressures of 8 MPa and 15 MPa.

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For the HPR [4] experiment with different pressures, a commercial Kranzle high pressure washer machine was used which can reach the pressure up to 15 MPa. The nozzle of the lance was made of ceramic with the diameter of 1 mm. To measure and compare the actual pressure at certain distance from the nozzle with the pressure shown by pressure gauge of HPR machine, a force gauge was also developed. Force gauge contains a stain-less-steel needle of the diameter of 1.5 mm which was fixed on a movable cylinder and movable cylinder was attached from the base plate by four springs. A linear scale was attached and the movement of knob of the cylinder can be read from scale which can be converted to force exerted by high pressure water beam as well as in pressure.

For HPR experiment with different pressures, a sample base plate was constructed which was fixed on a slide rail and can be slide to scan the sample during HPR. To keep the total injected quantity of water/area (dose) same as other HPR facilities, we calculated total scanning time of the sample is 1 second for the experiment. We also scan the sample for 10 seconds to see the time dependence of HPR.

Both the samples were subjected to HPR for 1 second (Dose = 0.786 l/cm²) and 10 seconds (Dose = 7.86 l/cm²) at different places and each sample was scanned manually for 1 second on the one side and 10 seconds on the another side. We used one separator which separates the sample in two parts, to avoid the effect of rinsing of another position by side-streams of the water beam. The distance between sample and nozzle was kept 50 mm to get highest pressure and to avoid the spreading of water beam. The experiment was conducted in class 1000 clean room.

After the HPR experiment, all the samples were analyzed by XPS to get the variation in fluorine atomic percentage as a function of sample length.

EXPERIMENTAL RESULTS

The XPS results of the experiment show that fluorine was drastically reduced on the surfaces and shows the pressure dependence as well as scanning time dependence. Fig. 2 shows the variation of atomic percentage of fluorine with the sample length. Left side in the plot shows the F concentration after scanning for 1 second and right side shows the F concentration after scanning for 10 seconds.

Perusal of Fig. 2, it can be concluded that the pressure as well as time duration of HPR are the key parameters to remove such chemical contaminants. According to the XPS analyses results, the fluorine was reduced mostly (<0.2 at. %) in case of sample surface rinsed with the pressure of 15 MPa for 10 seconds.

Additionally, we found Cu (0.1 %) and Zn (0.4%) on the surface which probably came from the brass components of the high pressure machine.

Chemical analysis of the water sample shows that Cu and Zn were found in the water from the machine.

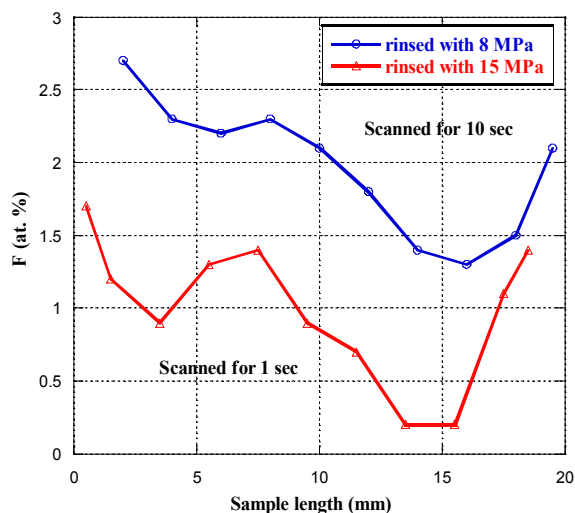


Figure 2: variation of fluorine concentration with sample length.

Following Table 1 shows the F concentration on the different sample surfaces.

Table 1: Atomic Percentage Present On the Sample Surface.

Pressure (MPa)	F (at. %)	F (at. %)
	Dose = 0.786 l/cm ²	Dose = 7.86 l/cm ²
8	2.2	1.3
15	0.9	<0.2

DISCUSSIONS

The surface analyses results of the HPR experiment confirms that HPR is the effective rinsing procedure to remove chemical residues left by surface treatment process for which pressure and time duration are the key parameters. After the chemical treatment of the surface some residues left by the process such as fluorine and sulfur are expected to be the measure source of field emission during the operation of Nb SRF cavity. If HPR with optimum parameters [5] is able to remove such contaminants then the performance of Nb SRF cavities might increased and even BCP treated Nb cavities can achieve high E-field gradient. Our force gauge measurements showed that the actual pressure at cavity surface is quite lower than the pressure shown by the gauge which may limits the HPR performance unexpectedly and not able to remove such contaminations. Therefore, a detailed study to find the optimum parameters for HPR is indeed. We are going to continue our study to find such parameters and to see the effect of different pressures on chemical contaminants left by BCP, EP, ECB, etc.

CONCLUSIONS

We have conducted HPR experiment with different pressure and time. The experimental results showed that fluorine was reduced drastically after the HPR with 15 MPa for 10 seconds which confirms that HPR has a great potential to remove the chemical contaminants after the process. A force gauge was also developed to measure the actual pressure at sample surface. The measurements show that actual pressure on the sample surface was reduced about four time at the distance of 50 mm which cause the insufficient rinsing to remove such chemical contaminants.

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