STUDY OF HPR CREATED OXIDE LAYER AT Nb SURFACES

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Abstract

The performance of superconducting radio frequency (SRF) niobium (Nb) cavities mainly depends on final surface condition therefore the surface preparation of these SRF cavities becomes very important. The preparation of surface includes two steps; surface chemistry (in order to get a smooth surface) and cleaning/rinsing (in order to remove contaminants left after the surface chemistry). As high pressure rinsing (HPR) with ultra pure water (UPW) is most commonly used surface cleaning method after the surface chemistry, it's very interesting to characterize the Nb surfaces after HPR. The surface characterizations show the presence of a thicker oxide layer at Nb surface as an outcome of HPR. In this article, we report the production of oxide layer (FWHM thickness) based on different conditions such as high pressure and doses. The surface characterization was done by XPS (x-ray photoelectron spectroscopy) with depth profiling.

INTRODUCTION

Final surface preparations [1] of niobium (Nb) superconducting radio frequency (SRF) cavities play a critical role in order to achieve high performances. Final surface preparation includes mainly two steps 1) surface chemistry in order to make surface smoother and 2) post cleaning processes in order to remove chemical residues left after the surface chemistry. As a step two, the High Pressure Rinsing (HPR) with ultra pure water/deionized water (UPW/DI), is most commonly used surface cleaning procedure worldwide [2,3]. The HPR seems to be a most effective cleaning procedure and HPR treated cavities have shown high field gradient with a high Q value. For the last two decades, HPR has been successfully used on SRF cavity surfaces and became an integral part of the final surface preparation of Nb SRF cavities. In order to make HPR more effectively for removal of the contaminants from cavity surface, the HPR operating parameters such as high pressures and doses should be carefully determined.

In this paper, we report our efforts towards finding of the optimized pressure and doses for HPR. In this regard, three samples were initially buffer chemical polished (BCPed) and subjected to HPR with three different pressures and two doses. For the HPR experiments, a commercial high pressure washer machine was used which can reach to a maximum pressure of 15 MPa. After the experiments, sample surfaces were analyzed by XPS (x-ray photo-electron spectroscopy).

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EXPERIMENTAL DETAILS

Surface Analysis

After the experiments, all the samples surfaces were analysed by XPS and a depth profile can be obtained. Our surface analysis system contains one main chamber along with three loadloack mechanism. The main chamber is equipped with 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. The analysis system is capable of executing Auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS) with argon ion etching, and XPS with probing area of 2 mm. Fig. 1 is the overview of our surface analysis system. The main chamber is maintained at extremely high vacuum [4].

The three loadlock mechanism provides the facility to transfer the samples from vacuum environment to main chamber without exposing them in to air. A vacuum suitcase (maintained at UHV) can be attached to one of the loadloak chambers with it and sample can be transferred from the suitcase to the analysis chamber keeping in vacuum. 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 the carousel in UHV. Also, samples can be mounted to one of the loadlock chambers directly from the atmosphere and can be transferred to main chamber after achieving UHV.
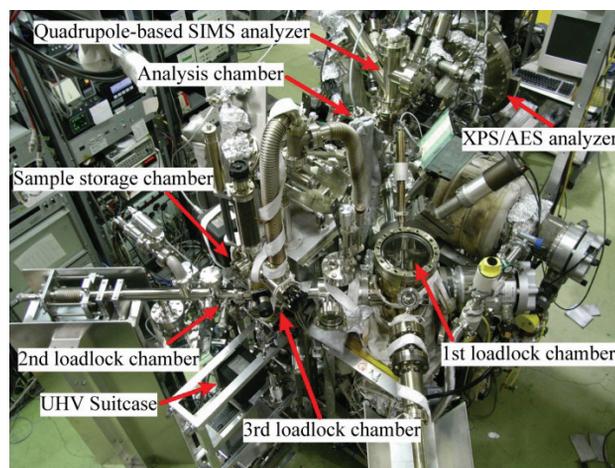
Figure 1: Overview of the surface analysis chamber.

HPR Experiments

We have conducted a series of HPR experiments on three Nb samples with three different pressures and two doses. The dose can be defined as the total injected

quantity of water/area. The aim of the experiment was to explore the effect of different parameters of HPR on contaminants removal after the surface chemistry.

A commercial Kranzle high pressure washer machine was used for the HPR experiments which can attain the pressure up to 15 MPa. The nozzle of the lance was made of ceramic with the diameter of 1 mm. For the experiments, three Nb samples (rectangular type: 20x14x2.8mm^3) were initially BCPed and a depth of 20 µm was removed. The sample (one by one) was mounted on a base plate which was fixed on a slide rail and can be slid during the HPR. 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. Each sample was subjected to two doses at different places for each pressure. In order to keep the same dose as of other HPR facilities, we had calculated the total scanning time of the sample during the HPR was 1 second corresponding to a dose of 0.79l/cm^2. The other dose of 7.9l/cm^2 corresponding to a scanning time of 10 second was selected to compare the effect of different doses. During the HPR, each sample was manually scanned. Totally, six experiments were conducted with two doses for three different pressures. 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 and a depth profile was obtained for each experiment.

EXPERIMENTAL RESULTS

XPS results of the BCPed sample followed by UPW rinsing only, showed that a huge amount of fluorine (up to 7 at. %) was found at top surface.

Table 1: The atomic composition present at top surface of the samples after HPR with different pressures as a function of dose

Elements	Atomic Composition Present at Samples Surface (Atomic %)						
	Dose=0.79l/ cm^2			Dose=7.9l/ cm^2			UPW rinsing
	8 MPa	10 MPa	15 MPa	8 MPa	10 MPa	15 MPa	
Nb	19	20	16	18	22	19	24
C	17	17	28	17	21	19	23
O	62	54	55	63	57	62	45
F	1.5	1.2	1	1.2	0.3	0.3	7

As it can also be encountered from the table 1 that the HPR was quite effective to mitigate the fluorine from the sample surfaces. The lowest fluorine was found on the surfaces treated with the higher dose. According to XPS

results, it is evident that not only the higher pressure is effective in order to mitigate the chemical contaminants from the surface but higher dose is more advantageous over the high pressure.
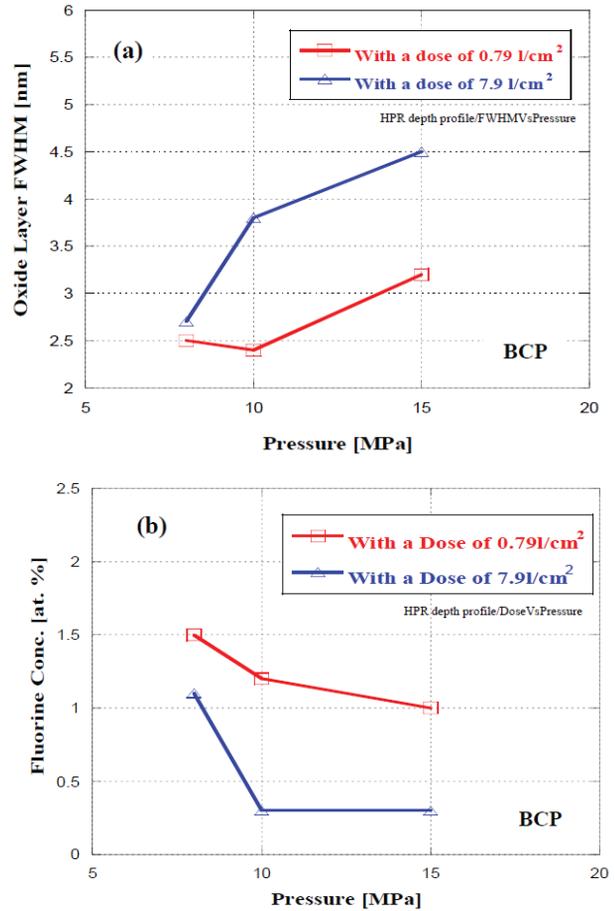
Figure 2: (a) Variation of oxide layer FWHM as a function of pressure and dose. (b) Reduction in fluorine concentration based on pressure and dose.

The depth profiles of all the samples showed a great increment in the thickness of oxide layer after the HPR. It can be seen from the fig. 2 (a) and (b) that the fluorine was reduced to same amount (up to 0.3 at.%) in case of the HPR with 10 MPa and 15 MPa with a high dose but a thicker oxide layer was produced by HPR with 15 MPa and a high dose. The FWHM oxide layer was found 0.7 nm thicker in case of 15 MPa than 10 MPa with a high dose (see table 2).

Table 2: The FWHM oxide layer thickness of after the HPR.

Pressure (MPa)	FWHM oxide layer thickness (nm)	
	Dose=0.79l/ cm^2	Dose=7.9l/ cm^2
8	2.5	2.7
10	2.4	3.8
15	3.2	4.5

It is also noticed that the amount of fluorine was increased at 1 nm depth and was similar in all the cases which shows that the HPR is effective to mitigate the contaminants from top layer of surface (<0.5 nm) only.

DISCUSSIONS

After the surface chemistry of Nb SRF cavities, the chemical residues such as fluorine and sulfur left after the process are expected to be the measure source of field emission during the operation of Nb SRF cavity. The HPR with optimum parameters will provide the better performance of SRF cavities ultimately a significant reduction in the production cost of Nb SRF cavities. Our surface analysis results of the HPR experiments showed that the HPR with high pressure and high dose was quite effective to remove contaminants after the surface chemistry. The depth profiles of the samples showed that the HPR with high pressure and dose produces a thicker oxide layer which might be a concern from SRF point of view. As it can be seen from our results, one side HPR with high pressure and dose seems to be very effective in order to mitigate the contaminants, another side it produces a thicker oxide layer. Therefore, in order to use HPR effectively, the balance in all the operating parameters should be carefully determined.

CONCLUSIONS

We have conducted a series of HPR experiments with three different pressures of 8, 10 15 MPa and two doses of 0.79l/cm^2 and 7.9l/cm^2. The experimental results showed that the fluorine was reduced to same amount after the HPR with high pressures of 10 and 15 MPa and a dose of 7.9l/cm^2 while a thicker oxide layer was created in case of the sample treated with 15 MPa and 7.9l/cm^2. Therefore it can be concluded from our experiments that the HPR with high pressures and doses is very effective to mitigate the contaminants after the surface chemistry but produces a thicker oxide layer.

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