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THE EFFECT OF PROCESS PARAMETERS ON THE SURFACE PROPERTIES OF NIOBIUM DURING PLASMA ETCHING*

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Abstract

We have shown that plasma etching using an electronegative Ar/Cl_2 discharge can effectively remove surface oxide layers on Nb samples as well as bulk Nb from single cell SRF cavities [1]. With accelerating fields on the order of wet etching processes and a decrease in field emission the use of plasma assisted etching for bulk Nb processing is a worthwhile endeavor. We are presenting work on the surface properties of plasma etched Nb. Cavity grade Nb coupons were made by water jet cutting, and then polished to achieve surface properties equivalent to electropolishing (<1 micron). The coupons were plasma etched while process parameters (rf power, gas pressure, temperature and dc bias voltage) are varied. These samples are placed on the inner surface of the cylindrical cavity to be etched. The experimental setup is similar to the single cell cavity plasma etching setup [2]. Each sample is weighed and scanned before and after plasma processing with an AFM, SEM, and digital optical microscope that provide both atomic composition and surface roughness profiles. Comparing the scans allows us to make conclusions about the effect of each experimental parameter on the surface properties.

INTRODUCTION

Superconducting radio frequency (SRF) Niobium (Nb) cavities require sufficient surface preparation before they can be considered for use in high energy particle accelerators. Improving the cavity's surface smoothness, the removal of surface impurities, and dulling the effect of grain boundaries all have significant impacts on the cavity's performance. Currently, buffered chemical polishing (BCP) or electropolishing (EP) are the most common methods for achieving satisfactory surface characteristics [3]. These processes, while effective, require the use of hydrogen flouride liquid acid baths, of which pose great environmental and human safety concerns. The semiconductor industry has been successful for a number of years in using "dry" plasma etching for the processing of silicon wafers. Using this as inspiration, plasma etching of a 3-D asymmetric Nb surface has proven to be a promising alternative to wet etching techniques [1, 2, 4–6]. This plasma etching alternative has the opportunity to provide a more controllable, cost effective, and safer form of surface modification that can achieve accelerating fields and Q-factors on the order of it's wet etching counterparts. In addition, plasma etching seems to reduce the field emission of SRF Nb cavities significantly, making the endeavor all the more worthwhile [1].

We have shown that plasma etching using an electronegative Ar/Cl_2 discharge can effectively remove surface oxide layers on Nb samples as well as bulk Nb cavities [1, 2, 4–6]. During this work, relationships between experimental parameters and the etching rate of the plasma were explored. These parameters include the rf power, gas pressure, sample temperature, gas composition, electrode geometry, dc bias voltage, and frequency of the power source. While some of these parameters may play a larger role in the etch rate than others, all must be considered when attempting to attain an ideal figure of merit. The etch rate and amount of surface material that can be removed are obviously important when one considers the application of this technology to SRF cavities, but it is also important to address the other qualities that make a proper superconducting cavity, including the surface properties.

As it was important to find the relationship between experimental parameters and the etch rate, it is important to understand the relationship between certain parameters and the resulting surface properties of plasma etched Nb. The parameters of interest are 1) gas pressure, 2) rf power, 3) temperature, and 4) dc bias voltage. These parameters have a great deal of influence on the results, each for their own reasons. In particular, and the main emphasis of this paper, the dc bias on the inner electrode provides one of the mechanisms required for etching to occur at all. Normally, the smaller powered electrode is the etched surface since it naturally develops a large negative self-bias voltage due to current conservation and it's small surface area. This high negative voltage on the powered electrode attracts the free positive ions in the plasma more than the cavity surface, thus making etching of the cavity impossible, as is seen in Fig. 1 (left) [5]. However, if a positive voltage is forced onto the powered electrode, the cavity becomes the surface with the highest negative voltage, and becomes etched. The same effect can be seen if the surface area of the electrode is increased, since this changes the capacitive nature of the plasma. In order to remove surface impurities, the chlorine ions must be accelerated toward the cavity wall with enough kinetic energy to facilitate a chemical reaction. This is a combination of both physical and chemical etching that is called reactive ion etching. With enough kinetic energy when hitting the surface, the chlorine ions react with the surface to make niobium pentachloride; a volatile product

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that can be pumped out of the system. Increasing the dc bias on the powered electrode increases the kinetic energy and number of ions that are accelerated toward the cavity surface, increasing the amount of etching.

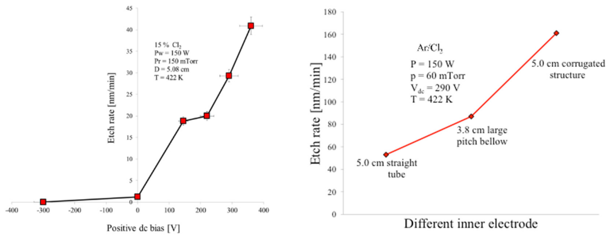

Figure 1: Left: Etch rate vs. dc bias. Right: Etch rate vs. electrode geometry.

The electrode geometry and gas composition have a large influence on the etch rate, but it has been shown that a disc-loaded corrugated structure electrode, along with a 15% of Cl_2 in an Ar plasma produces higher etch rates by reversing the discharge asymmetry [4–6]. The effects of the electrode geometry can be seen in Fig. 1 (right) [6]. The corrugated structure increases the surface area of the electrode, thus decreasing the self-bias on the electrode significantly while the Cl_2 gas concentration provides radicals required for the etch process. These two parameters, electrode geometry and Cl_2 gas concentration, are kept constant throughout the series of experiments.

EXPERIMENTAL PROCEDURE

The design of the surface properties experiment is much like previous experiments conducted to study the etch rate [2]. Niobium disks (coupons) 0.6" in diameter and 0.2" thick are placed in a cylindrical plasma reactor through mini-conflat ports on the side of the grounded cylindrical electrode. The samples are held flush to the surrounding wall of the cavity so that the samples face the plasma at a radial position equal to the cavity radius. Two samples, placed on either side of the electrode, are used for each data point as the experimental parameters are changed independently.

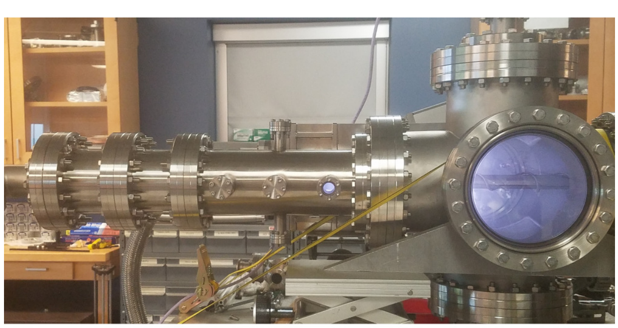

Figure 2: The plasma etching chamber.

In order to draw appropriate conclusions as compared to BCP or EP, the samples are constructed of cavity grade Nb provided by Thomas Jefferson National Laboratory. To avoid the addition of any impurities during the production of the samples, each piece was cut from a solid sheet using a water-jet cutter. The water jet cutting also prevents excessive heating of the samples, of which can cause degradation of the lattice structure. The samples are then hand polished using a Dremel tool and diamond paste to avoid the use of silicon based products. The diamond paste polishing achieves surface roughness on the order of 1 micron or better and allows one to establish a comparative post-processing analysis. Before plasma processing, each sample is weighed and then scanned by three high powered microscopes, an Atomic Force Microscope (AFM), a PHENOM scanning electron microscope, and a HIROX digital optical microscope. Each scan provides a surface roughness profile, while the PHENOM scans also provide the atomic composition measurement. The atomic composition allows us to identify the purity of each sample pre- and post-processing. After plasma processing of each sample, the weight measurement and scans are repeated, leaving no ambiguity as to the effect of plasma etching on the surface properties.

RESULTS AND DISCUSSION

By changing experimental plasma processing parameters independently, each parameter can be identified as having it's own particular effect on the surface characterization of Nb. With these relationships in hand, the parameters can then be fine tuned when etching of a single cell cavity, producing a more desirable surface characterization. Our work is currently ongoing, however some comments about the effect of plasma etching on surface properties can be provided. The sample was plasma etched in an Ar/Cl_2 plasma for 5 hours at a pressure of 30 mTorr, an rf power of 50 W, a temperature of 333 K, and a positive dc bias of 100 V. AFM scans along with roughness profiles and digital photographs of one of the etched samples is provided in Fig. 3. One can see the physical effect that plasma etching had on the sample. While 3.4 microns of material was removed from the surface over the 5 hour etch time, the surface became quite rough, although uniformly so, in comparison to its state before etching. However, the diamond paste polishing proved to be overly effective in smoothing the sample by achieving an rms surface roughness of 22.905 nm in the scanned region. After plasma etching, the rms surface roughness increased over 10x. Even so, the measured surface roughness of 264.51 nm is still well under 1 micron surface roughness. Completion of this work will provide information to conclude relationships between plasma parameters and surface properties.

CONCLUSION

The use of reactive ion plasma etching for the surface modification of SRF Nb cavities has proven to be a potential alternative or beneficial addition to current wet chemical etch processes.We have accomplished a study of the particular plasma parameters required for surface impurity removal, relationships between these parameters and their effect on

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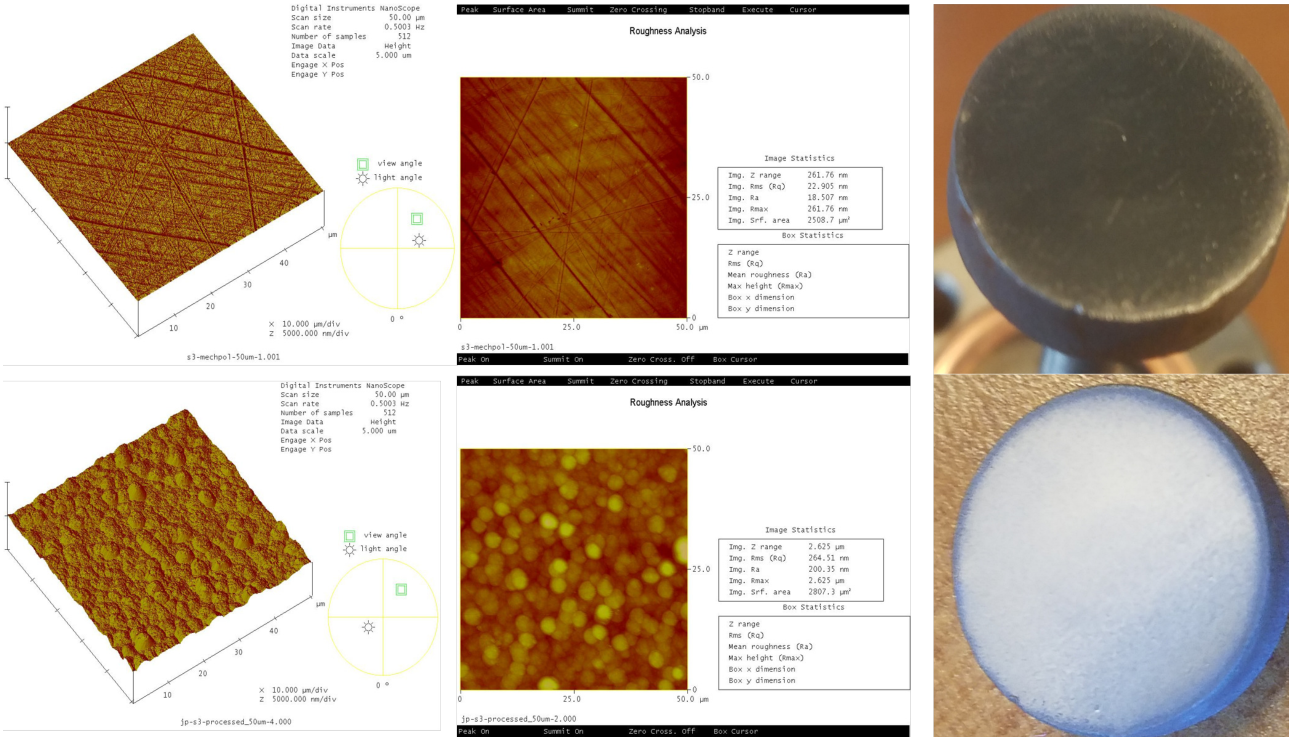

Figure 3: Top: AFM scan, roughness profile, and photo before plasma etching. Bottom: AFM scan, roughness profile, and photo after plasma etching.

the surface properties are being developed. The experimental setup and procedure is presented and an example of the effect of plasma etching on the surface properties of a cavity grade Nb sample is shown. While the plasma etch process did increase the rms surface roughness, the sample started with a surface roughness rms in the tens of nanometers, while ending in the hundreds of nanometers; still well under 1 micron. The presented example only shows results from one particular configuration of experimental parameters and the variation of these parameters could be beneficial or detrimental to the surface properties. The knowledge gained from the conclusion of this series of experiments will be used to fine tune a single cell cavity etching experiment for both etch rate and surface properties.

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