IMPROVING THE WORK FUNCTION OF NITROGEN-DOPED NIOBIUM SURFACES FOR SRF CAVITIES BY PLASMA PROCESSING*

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Abstract

Work function and surface chemistries of SiC-polished, electropolished, and nitrogen-doped niobium coupons were analyzed before and after plasma processing using a neon-oxygen gas mixture. These studies represent an initial enquiry into the feasibility of applying the plasma processing technique designed at ORNL for the Spallation Neutron Source (SNS) to the nitrogen-doped Nb cavities for the Linac Coherent Light Source (LCLS-II) upgrade. Work function of all measured samples was increased after plasma processing, which indicates the strong potential of the plasma processing technique as a tool for increasing the accelerating gradient of nitrogen-doped cavities.

INTRODUCTION

Nitrogen-doped superconducting radio-frequency cavities are being used in the upgrade to the Linac Coherent Light Source (LCLS-II) as Fermi National Accelerator Laboratory (FNAL) has shown that N-doping leads to an increase in the quality factor of niobium (Nb) cavities [1-8]. Oak Ridge National Laboratory (ORNL) has found that oxygen plasma cleans hydrocarbons from the surface of niobium (Nb). Removal of these hydrocarbons from the Nb surface leads to an increase in work function (WF), which in turn leads to an increase in stable accelerating gradient for superconducting radio-frequency (SRF) cavities such as those in use in the Spallation Neutron Source (SNS) at ORNL [9], [10]. Work function is essentially the ease with which electrons can be stripped from the surface to a point immediately outside that surface. By the Fowler-Nordheim equation, Eq. (1), an increased work function corresponds to a decrease in field emission and thus an increase in achievable stable accelerating gradient [11] for cavities limited by field emission.

$$J = a\frac{\beta E}{\phi}e^{-b\frac{\phi^{3/2}}{\beta E}+\frac{c}{\phi^{1/2}}} \qquad (1)$$

where a = 1.54E6, b=6.53E3, and c=10.4 are constants for niobium, E is the electric field at the cavity surface in MV/m, β is the field enhancement factor, ϕ is the surface work function in eV, and J is the current density in A/m^2.

A collaboration between ORNL, FNAL and the Stanford Linear Accelerator Laboratory (SLAC) to investigate recipes to combine Nitrogen doping and plasma processing for application to the LCLS-II cavities. The present study is motivated by determining whether the oxygen plasma processing technique can benefit the nitrogen-doped LCLS-II cavity surfaces.

MATERIALS

Materials were procured from Tokyo Denkai, Wah Chang, and Ningxia in order to compare the effects inherent with material sourcing with the effects of post-processing. Preparation was performed at ORNL and FNAL sites, as noted in the text. Plasma processing and final testing was performed at the ORNL facility. Samples were taken from each process stage for measurements of work function and surface chemistry, through the use of kelvin probe and secondary ion mass spectroscopy (SIMS) instruments, respectively.

Sample Preparation

Niobium coupons were manufactured at ORNL to diameters of 10 mm and 25.4 mm from 4 mm thick Nb sheets purchased from Tokyo Denkai. Nb coupons machined at FNAL had the same diameters but a thickness of 3 mm. FNAL material was purchased from Tokyo Denkai, Wah Chang, and Ningxia. Materials received no post-processing except that noted explicitly in the text.

ORNL samples were polished to 100 μm, finished with 1200 grit SiC, and placed in an ultrasonic ethanol bath and rinsed afterwards; these samples are hereafter referred to as "SiC." Note that SiC is not an rf surface. All samples were electropolished to 140 μm in an HF/HCl solution at FNAL and ultrasonically cleaned. Samples sent back to ORNL for analysis in this condition are designated "EP" samples, for "electropolish only."

The remainder of samples were further subjected to a nitrogen-doping procedure designed by FNAL [8]. This doping procedure ("2/6," detailed in Fig. 1) involved heating these samples to 800 °C and holding at this temperature for 3 hours, while the cavity was degassed. A pressure of 26 mTorr of nitrogen was then injected for 2 minutes, then stopped. The cavity remained at 800 °C for additional 6 minutes and was then allowed to furnace cool. A light electropolishing step then removed an additional 5 μm from the surface, such as to eliminate nitride contamination at the surface, leaving instead only 100-200 ppm of interstitial nitrogen in the top surface of the Nb [8]. These samples are
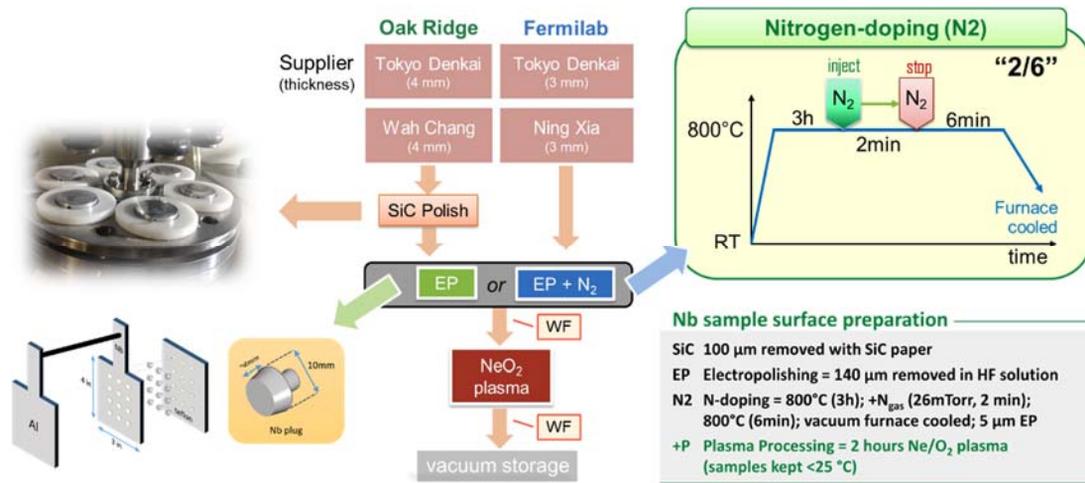
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Figure 1: Process flowchart for samples prepared in EP *vs.* N2 manner. ORNL-prepared samples were subjected to an additional initial SiC-polish as compared to FNAL-prepared samples. "WF" denotes work function measurement.

henceforth referred to as "N2" for [electropolishing and] "nitrogen-doping." Refer to Fig. 1 for details. Note that "EP [only]" samples did not receive any additional heat treatments.

Plasma Processing

The plasma processing station, pictured in Fig. 2, consists of a gas manifold to control gas flow to the plasma processing microwave, or, alternatively, to the single-cell cavity (far-right). A neon oxygen mixture was flowed into the microwave at 450 mTorr and ignited at 200 W for a period of 2 cumulative hours. A residual gas analyzer at the gas outlet allowed for continuous monitoring of outgassing. In the single-cell cavity, samples were likewise ignited for 2 hours.
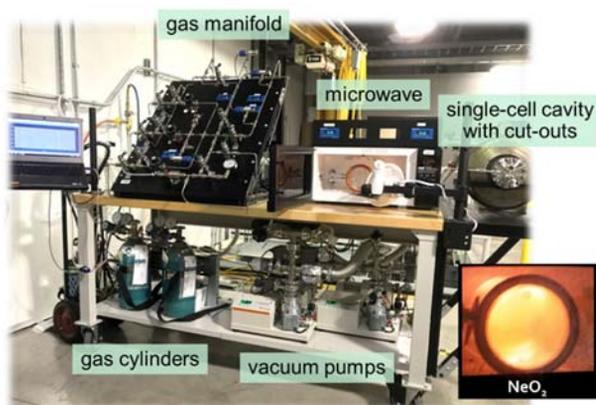

Figure 2: Gas manifold and microwave plasma processor are pictured as utilized. The single-cell cavity is visible far right. The computer screen visualizes the residual gas analyzer results. Inset is an image showing NeO_2 plasma ignited within the microwave.

RESULTS

The work function of Nb is highly sensitive to the topmost surface condition and affects the achievable accelerating gradient. Figure 3 shows the work function of samples prepared by silicon carbide polish (SiC), electropolish (EP), and nitrogen-doping (N2), and these groups all combined when treated with neon oxygen plasma in a microwave. EP and N2 increased work function to a statistically noticeable extent. Plasma processing increased the work function of all tested samples, by 0.8 to 1.0 eV, in all instances.

While manufacturer was considered as a possible source of variation, differences between Nb materials sourced from Wah Chang, Tokyo Denkai, and Ningxia were found to be statistically insignificant with respect to work function for the conditions assessed.
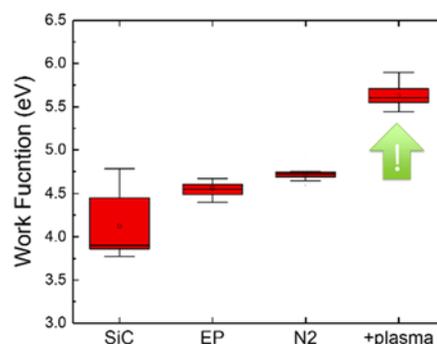

Figure 3: Work function is plotted against final processing step, e.g. SiC polishing, electropolishing, nitrogen-doping, and plasma processing. Plasma dramatically improved the work function of all tested samples.

Secondary ion mass spectroscopy surface spectra for a nitrogen-doped Nb sample before and after 2 h plasma cleaning are presented in Fig. 4. Hydrocarbon concentration at the surface is significantly reduced after processing with Ne/O2 plasma.
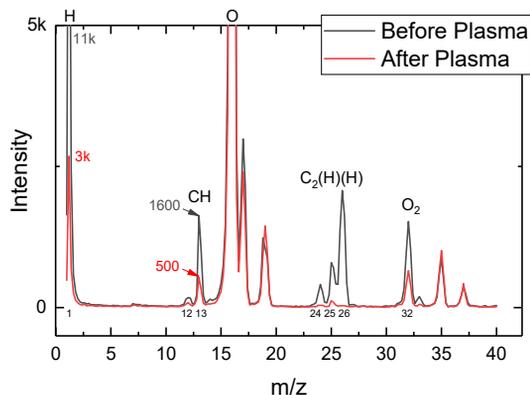
Figure 4: Secondary ion mass spectroscopy surface spectra for N2 sample before and after 2 h Ne/O$_2$ plasma.

Plasma was also ignited for witness samples placed at equator, wall, and iris locations in an 805 MHz single-cell cavity. The effect of plasma on work function at these locations is presented in Fig. 5. These samples experienced a similar increase in work function regardless of location.
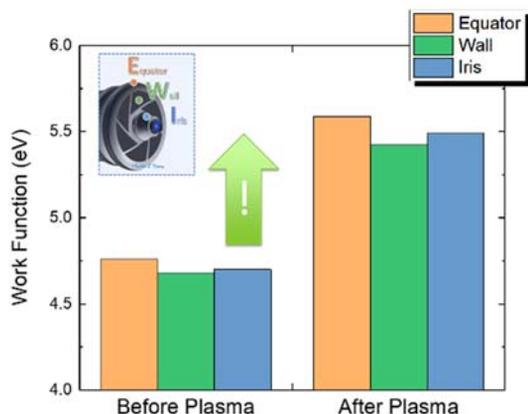

Figure 5: Work function of Nb samples as polished vs. after 2 h of Ne/O$_2$ plasma, for witness samples located at equator, wall, or iris.

CONCLUSIONS

Plasma processing improved work function of all tested samples. This correlated to a decrease in surface contaminants. Plasma processing of a nitrogen-doped 1.3 GHz 9-cell cavity at FNAL is pending [12].

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