

# PLASMA PROCESSING OF Nb SURFACES FOR SRF CAVITIES\*

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## Abstract

Field emission is one of the most critical issues to achieve high performances of niobium (Nb) superconducting radio frequency (SRF) cavities. Field emission is mainly related to contaminants present at top surface of SRF cavities that act as electron emitters at high gradient operation and limit the cavity accelerating gradient. An R&D program at the Spallation Neutron Source (SNS) is in place [1, 2] aiming to develop an in-situ plasma processing technique to remove some of the residual contaminants from inner surfaces of Nb cavities and improve their performance. The plasma processing R&D has first concentrated on removing hydrocarbon contamination from top surface of SRF cavities. Results from the surface studies on plasma processed Nb samples will be presented in this article and showed the removal of hydrocarbons from Nb surfaces as well as improvement of the surface workfunction (WF).

## INTRODUCTION

Plasma cleaning R&D for niobium (Nb) superconducting radio frequency (SRF) cavities is currently being pursued at the Spallation Neutron Source (SNS), Oak Ridge National Laboratory. The first objective of the plasma processing R&D is to develop an in-situ cleaning technology for SNS high beta (HB) cryo-modules [1, 2]. SNS HB cavities are presently limited by electron emission (field emission) during operation at high accelerating gradients and operating below their designed accelerating gradient. Organic contaminants located in different adsorption sites at top surface of SRF cavities can be responsible for the field emission and degradation of the cavity performance. In-situ plasma processing appears to be a promising technology for in-situ removal of such organic contaminants from top surface of SRF cavities. Plasma cleaning of carbonaceous contaminants is already being used extensively in the semiconductor industry and has shown its potential to clean organic contaminants.

Plasma ignition in the plasma reactor is accomplished by applying appropriate RF field level for the desired gas composition and working pressure. The gas discharge creates a plasma containing reactive species in the processing volume. In multicell SRF cavities, plasma ignition can be controlled by exciting a cavity with a combination of different resonant modes of the cavity while a continuous gas flow is maintained [2, 3]. The reactive species formed in the plasma react with the organic contaminants present at top surface, forming volatile compounds which are pumped out through the vacuum system. Plasma processing of SRF cavities at SNS utilizes the gas mixture of neon (Ne) and oxygen (O<sub>2</sub>) where Ne is the primary gas to generate and tune the plasma in a cavity [3] and O<sub>2</sub> is used as a cleaning agent for organic

contaminants. Plasma generated reactive species of O<sub>2</sub> reacts with the organic contaminants and oxidizes them into the volatile compounds which are then pumped out. In order to study plasma chemistry during the plasma processing, residual gas analyzer (RGA) can be utilized and provide direct measurement of the partial pressure of the volatile by-products from the plasma reaction. To assess the effect on the field emission after the plasma processing, we utilized a scanning kelvin probe (SKP) which measures the surface workfunction (WF) [4,5]. The surface WF is particularly important for our application as it is directly related to the field emission via Fowler-Nordheim law (see Eq. 1) [6].

$$j \propto \frac{E^2}{\phi} \exp\left(\frac{-a \times \phi^2}{E}\right) \quad (1)$$

Where,  $j$  is current density,  $E$  is electric field and  $\phi$  is work function. In practice,  $E$  is not the ideal surface electric field but is a surface enhanced electric field,  $E = \beta E_0$  ( $\beta > 1$ ), where  $\beta$  is known as field enhancement factor. It can be seen from Eq. 1 that increasing the work function helps reducing the field emission current. Thus, cleaning contaminants with lower work function than the natural cavity top surface is desirable. SKP is an ideal tool to measure the changes in work-function after the plasma processing. In this article, we present plasma processing studies of Nb samples using RGA and SKP.

## PLASMA PROCESSING

### Plasma Reactor

A barrel type plasma reactor made of Pyrex glass was used for the plasma processing of Nb samples. The glass reactor has an inlet and outlet port for the process gases and to connect vacuum system. The reactor assembly is located inside a microwave system which operates at 2.4 GHz with a maximum power of 500 Watts. The complete plasma reactor was provided from SPI supplies [7]. Fig. 1 shows the picture of the microwave plasma reactor.

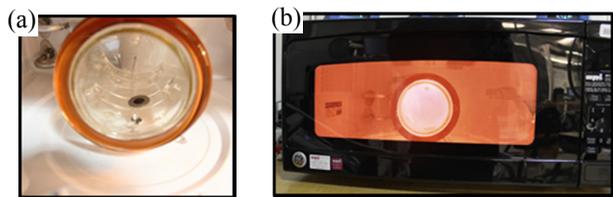


Figure 1: Microwave plasma processing. (a) contaminated Nb sample in the plasma reactor (b) plasma ignited in the microwave plasma reactor.

The process gases were introduced in the reactor via a gas manifold to control the gas flow and mixture. An additional nitrogen (N<sub>2</sub>) line was also available for venting the reactor when exchanging the samples. The outlet line of the plasma reactor was connected to the vacuum system and to an RGA.

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### Sample Preparation

Two samples of 25 mm diameter and 1 mm thickness were prepared for the studies. One sample was mechanically polished and the other was buffered chemical polished (BCPed). The mechanically polished sample was first subjected to light  $\text{NeO}_2$  plasma processing for basic cleaning then intentionally contaminated by a black permanent marker (sharpie) to have a thin layer of long hydrocarbon chains such as n propanol and n-butanol. The weight of contaminant's film was measured  $\sim 242 \mu\text{g}$  ( $\sim 1860 \text{ nm}$ ) using a micro-balance. Fig. 2 a shows the Nb sample with the thin film of marker's contaminant before the plasma processing.

### Plasma Processing

In order to ignite plasma, a  $\text{NeO}_2$  (2% $\text{O}_2$ ) gas mixture at a pressure range of 130-140 mtorr was established in the reactor. Total gas pressure and oxygen concentration of 2% was chosen similar to standard conditions for plasma processing in SNS cavities [3]. The heating of the plasma reactor during microwave operation can lead to surface oxidation by the oxygen present in the process gas mixture. To mitigate this issue, we processed the samples in one minute cycles and waited for several minutes between cycles to keep the reactor temperature very close to room temperature. A total of 25 minutes of plasma processing with 40% ( $\sim 200 \text{ W}$ ) of microwave power was performed.

It can be seen in Fig. 2(b) that a significant amount of the marker's contaminant had disappeared after 25 min of  $\text{NeO}_2$  plasma processing. There were still some residues left on the surface and can be cleaned with additional processing time. Based on the weight measurement, the removal rate of contaminant film was found to be  $\sim 8.4 \mu\text{g}/\text{min}$  ( $\sim 65 \text{ nm}/\text{min}$ ). In case of Nb BCPed sample, we didn't find a significant difference in the weight (within the measurement limit) after 25 min of plasma processing.

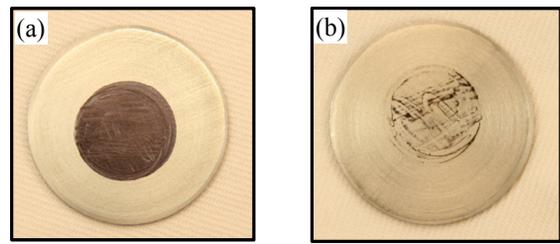


Figure 2: Nb sample with contaminant's film (a) before plasma processing (b) after 25 min of plasma processing.

### Hydrocarbon Removal

RGA spectrum during the plasma process of Nb sample contaminated by the black permanent marker is shown in Fig. 3. A significant increase in the masses 12, 18, 28 and 44 was observed when the plasma was ignited while mass 32 was consumed during the plasma processing. Masses 12, 18, 28, 44 and 32 relate to carbon, water ( $\text{H}_2\text{O}$ ), carbon monoxide ( $\text{CO}$ )/nitrogen ( $\text{N}_2$ ), carbon dioxide ( $\text{CO}_2$ ) and oxygen ( $\text{O}_2$ ) respectively. Some of the RGA signal of  $\text{CO}$  and  $\text{H}_2\text{O}$  coincides with the background signals of residual  $\text{N}_2$  and  $\text{H}_2\text{O}$  in the reactor. However, the variation in the signal intensity during the plasma processing is mostly related to the plasma chemistry.

It is concluded from the RGA spectrum (see fig 3) that the plasma processing is mostly chemical in nature. The reactive  $\text{O}_2$  species generated in the plasma react with the hydrocarbon contaminants present at the top surface and release  $\text{H}_2\text{O}$ ,  $\text{CO}$  and  $\text{CO}_2$  as by-products of chemical reactions. The RGA spectrum of as received Nb BCPed sample was very similar with reduced signal intensities for  $\text{H}_2\text{O}$ ,  $\text{CO}$ ,  $\text{CO}_2$ . Also, similar RGA signals were measured during plasma processing of SNS HB cavities [2, 3]. From Fig 4, it can be seen that the amount of volatile products generated during the plasma processing decreased with time, probably because the top surface is progressively depleted from hydrocarbon contaminants as shown in Fig. 2.

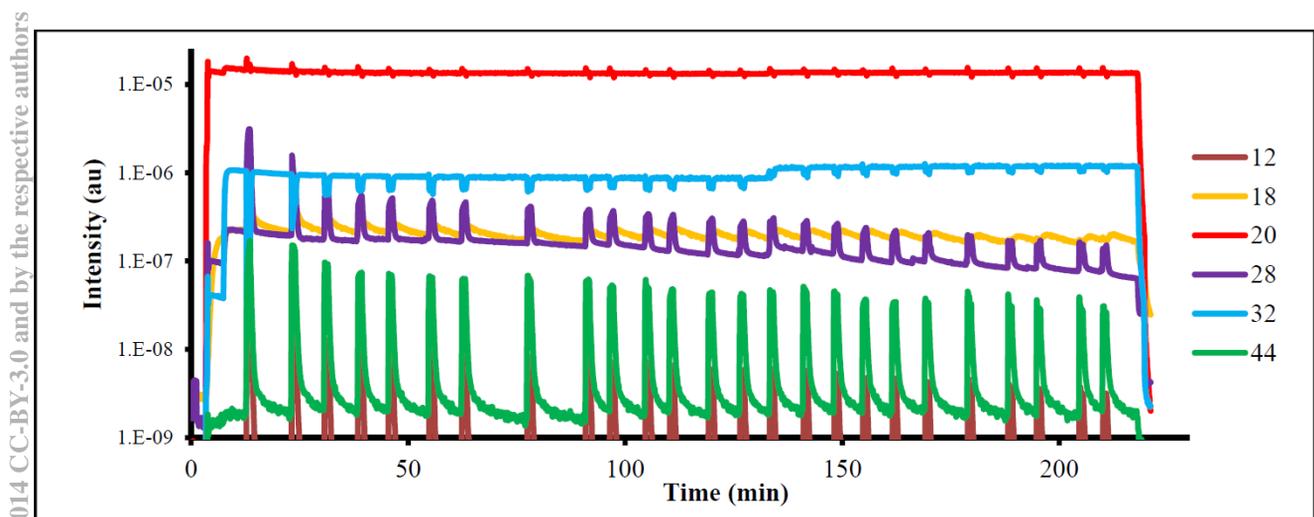


Figure 3: RGA spectrum of Nb contaminated sample during plasma processing.

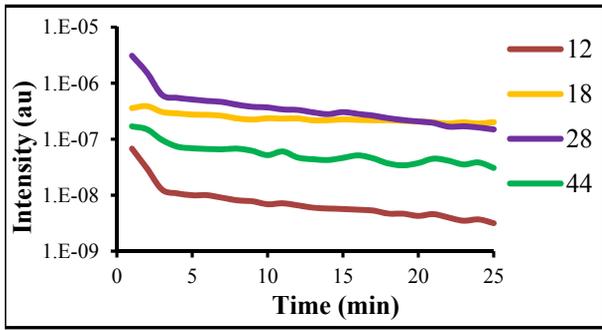


Figure 4: Variation in RGA signal of volatile reaction products as a function of plasma processing duration.

*SKP Measurements*

SKP utilizes Lord Kelvin’s principle to measure the surface WF. The SKP instrument mainly consists of a vibrating probe which is connected to computer via a digital oscillator and data acquisition system. During the measurements, the probe (made of gold and 2 mm in diameter in our case) is scanned above the sample surface at a constant distance and measures the contact potential difference (CPD) between sample’s surface and probe’s surface as a function of applied backing potential [4, 5]. The absolute WF of the sample’s surface can be calculated using Eq. 2.

$$\phi_{sample} = WF_{tip} + CPD_{sample} \quad (2)$$

SKP mapping (fig 5a) of Nb sample with contamination before plasma processing shows a lower WF at area covered with contamination than non-contaminated area. The surface WF was significantly increased after the plasma processing (see fig 5b).

We found that the WF of plasma processed Nb BCPed sample had also increased after plasma processing (see Table 1).

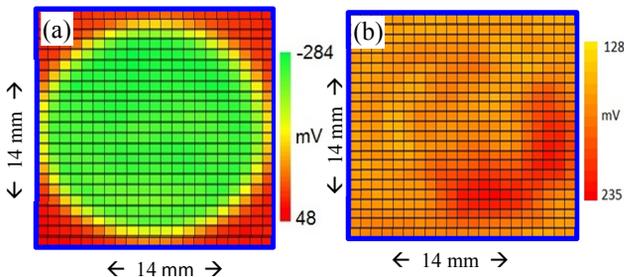


Figure 5: SKP mapping of surface CPD of contaminated Nb sample. (a) before plasma processing (b) after 25 min of plasma processing.

Table 1: Single point surface WF of Nb samples before and after plasma processing

Sample	Work Function (eV)		$\Delta$ (eV)
	Before plasma processing	After plasma processing	
Nb Contaminated sample	4.72	5.10	0.36
Nb BCPed sample	4.53	4.72	0.2

**SUMMARY**

We have presented plasma chemistry and surface WF studies using a microwave plasma reactor using a similar gas pressure and mixture than the one used for plasma processing of SNS cavities. Our experimental results showed that the plasma processing with the NeO<sub>2</sub> (2%O<sub>2</sub>) gas mixtures was very effective to remove hydrocarbon contaminants from the Nb surface. RGA data during plasma processing showed the formation of H<sub>2</sub>O, CO<sub>2</sub> and CO as volatile by-products with the reactive O<sub>2</sub> species in the plasma. SKP measurements showed an increase in the WF after the plasma processing. The increase in the WF is particularly important for SRF cavities as it is directly related to field emission process. Recent cold test result of plasma processed SNS HB cavity had shown more than 25 % improvement in the stable accelerating gradient after plasma processing of hydrocarbon contaminants with reduced field emissions and vacuum activities [3].

Further studies to investigate surface compositions after the plasma processing and surface WF under different plasma processing conditions will be performed.

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