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Improving the work function of the niobium surface of SRF cavities by plasma processing



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ABSTRACT

An in situ plasma processing technique using chemically reactive oxygen plasma to remove hydrocarbons from superconducting radio frequency cavity surfaces at room temperature has been developed at the spallation neutron source, at Oak Ridge National Laboratory. To understand better the interaction between the plasma and niobium surface, surface studies on small samples were performed. In this article, we report the results from those surface studies. The results show that plasma processing removes hydrocarbons from top surface and improves the surface work function by 0.5–1.0 eV. Improving the work function of RF surface of cavities can help to improve their operational performance.

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1. Introduction

Field emission is one of the critical issues in high gradient superconducting radio frequency (SRF) cavities that limits the cavity performance [1–3]. Previous studies on field emission in SRF cavities have concentrated on protrusions and particulate contaminations at the RF surface [4–6]. During surface studies at spallation neutron source (SNS), shallow coverage of hydrocarbons at the niobium surface has been systematically observed even after standard surface preparation such as chemical polishing followed by ultra pure water rinsing. Evidence of hydrocarbons on the RF surface has also been found in cavities [7].

Evidence shows that the hydrocarbon contaminations can enhance the field emission and may limit the cavity accelerating gradients. Plasma cleaning of SRF cavities is currently being pursued at the SNS, Oak Ridge National Laboratory (ORNL) [7]. Plasma cleaning of hydrocarbon contaminants is a well-established process in the semiconductor industries for cleaning of semiconductor wafers [8,9]. One of the attractive features of plasma processing for

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http://dx.doi.org/10.1016/j.apsusc.2016.02.030 0169-4332/Published by Elsevier B.V. SRF cavities is that it can be applied in situ when cryomodules have been finally assembled and installed in accelerator tunnels. Other plasma processing for etching of niobium SRF cavities is currently being explored at J-lab [10,11].

Operating gradients of SNS six-cell high beta SRF cavities are presently limited by field emission and below the design value. An in situ plasma processing technique at room temperature for these multicell cavities has been developed to remove hydrocarbon contamination [12–14]. Plasma in multicell SRF cavities is generated and tuned by using fundamental passband modes while a continuous gas flow is maintained. Neon is the primary gas for plasma ignition and tuning in a desired cell of the cavities. Once the plasma is ignited and tuned in a desired cell, oxygen is introduced into the cavity to oxidize hydrocarbon contaminants. The volatile byproducts from the oxidation process are continuously pumped out. Plasma processed cavities have been tested at cold and showed significant reduction in the level of electron activities and improvement in their accelerating gradients [13, 14].

In order to better understand the characteristics of the niobium surface after plasma processing, detailed surface studies on small samples were performed. This paper will discuss the results from those studies. Surface characterizations of niobium samples were done using secondary ion mass spectrometry (SIMS) for chemical analysis and with scanning kelvin probe technique for work function measurements.

2. Importance of work function for SRF cavities

Field emission is one of the main factors that limits the accelerating gradients of SRF cavities. The field emitted current density is defined by the Fowler–Nordheim theory (Eq. (1)) [15].

$$j = a \frac{(\beta E)}{\varphi} \exp\left(\frac{-b\varphi^{3/2}}{\beta E} + \frac{c}{\varphi^{1/2}}\right)$$
(1)

where, *j* is the current density, φ is the surface work function, *E* is the surface electric field, β is known as the field enhancement factor and parameters $a = 1.54 \times 10^6$, $b = 6.53 \times 10^3$ and c = 10.4 are constants.

Particulate contamination on RF surface can increase the field enhancement factor β thus the field emitted current. To mitigate particle contamination, cryomodule assembly is for example done in clean room. It is believed that shallow coverage of hydrocarbons can lower the work function φ of the niobium surface and also increase the field emitted current. In situ plasma processing aims at removing hydrocarbon contamination and improving the work function of the cavity surface.

In the present work, a scanning kelvin probe system was systematically used to measure the work function of sample's surface [16,17]. This system operates on Lord Kelvin's principle and measures contact potential difference (CPD) between two metallic surfaces. During the measurements, the probe with known work function (gold plated and 2 mm diameter) vibrates and measures the CPD between sample's surface and probe's surface as a function of applied backing potential. The absolute work function of the sample's surface can then be calculated using following equation

$$\phi_{\text{sample}} = \phi_{\text{probe}} + \text{CPD} \tag{2}$$

The accuracy level of the work function measurements were in the order of ± 0.1 eV.

3. Work function sensitivity to top surface composition of niobium

In this article, top surface refers to the first monolayer of a material and near surface refers to several monolayers below the top surface. The work function for a surface is directly related to its composition. The reported work function of polycrystalline niobium is 4.3 eV [18]. However, niobium is typically covered with its natural oxide of Nb₂O₅ with reported work function of 5.2 eV [19]. Other factors such as crystal structure, oxide states and surface contaminations can significantly affect the work function of niobium surface [19-21]. Because work function is sensitive to surface conditions, the work function was measured systematically in the studies presented hereafter. In addition, chemical analysis of top surface and near surface was done by SIMS [22]. SIMS uses ion sputtering of the top surface and mass analysis of the secondary ions generated by the sputtering process. In the work presented here, the SIMS equipment [23] utilizes a primary beam of Ga⁺ ions for sputtering of sample surface and a quadrupole mass filter for mass analysis. Chemical analysis by SIMS is very informative method especially for characterization of top surface composition but sensitivity of the signal measured for different species depends on various factors such as sputtering rate of different elements and ion escape probability etc. In the following, the signal of each species in each SIMS spectrum is normalized by the total intensity collected during the measurement.



Fig. 1. Negative SIMS spectrum of chemically polished niobium sample surface.



Fig. 2. SIMS negative spectrum of mechanically polished niobium sample surface. Similar to chemically polished niobium surface, hydrocarbon peaks were observed on the mechanical polished niobium surface. Small amount of Cl at top surface might have originated from sample preparations, grinding/polishing and handling.

3.1. Work function and composition of a chemically polished niobium surface

A niobium sample was buffered-chemically polished using standard acid mixture for cavity processing followed by rinsing in deionized water and drying under nitrogen flow in a clean room. The work function of chemically polished surface was measured at 4.5 eV which is significantly lower than the work function of Nb₂O₅ (niobium pentoxide). Chemical analysis by SIMS was performed to understand the lower work function. SIMS negative mass spectrum obtained for chemically polished niobium surface (Fig. 1) shows mass peaks related to water, hydrocarbons, fluorine, and niobium oxides. The peaks at mass 12 (C⁻) and 13 (CH⁻) are fragments of hydrocarbon contaminants on the surface which can lower the work function.

3.2. Work function and composition of a mechanically polished niobium surface

Niobium samples were prepared by EDM wire cut out from a high purity niobium sheet and mechanically polished using a sample grinder/polisher [24]. Silicon-carbide abrasive paper was used for grinding and polishing. The samples were subsequently cleaned with isopropanol solvent and ultrasonically cleaned in deionized water for 15 min. The rms surface roughness of the mechanically polished samples was usually less than 1 μ m. The work function of mechanically polished surface was measured typically at 4.8 eV which is lower than the work function of Nb₂O₅. Similar to chemically polished niobium samples shown in Fig. 2 also revealed the presence of hydrocarbons at the top surface.

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