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PLASMA TREATMENT OF BULK NIOBIUM SURFACES FOR SRF CAVITIES

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Abstract

Two types of electric discharges were used to demonstrate the validity of plasma surface treatment for superconducting radio-frequency (SRF) cavities. The experiments were performed on disc-shaped Nb samples and compared with identical samples treated with buffer chemical polishing (BCP) techniques. Surface analysis indicates comparable or superior properties of plasma-treated samples. These promising results are still preliminary and additional work is in progress.

INTRODUCTION

The preparation of cavity walls has been one of the major challenges in SRF accelerator technology, since accelerator performance depends directly on the physical and chemical characteristics at the cavity surface. The objective of the present research is to minimize the degradation of the cavity RF performance due to the presence of Nb-oxides, by employing various surface modification techniques involving electric discharge plasmas. Images obtained with several surface characterization techniques show increasing etching rates in the regions of enhanced electric field. This smoothing behaviour leads to oblique shapes at the surface and apparent elimination of sharp edges. To understand the complexity of the collision processes involved in removal of niobium oxide layers, plasma modelling is being initiated in addition to the experimental work. We used the *ab initio* approach for obtaining the electronic structure and the Binary-Encounter-Bethe (BEB) method for the cross sections calculations. These results will be included in the plasma-surface interaction model, currently under development.

EXPERIMENT

The bulk Nb used for fabrication of SRF cavities comes in form of metal sheets covered with a thin layer of oxides. Our samples were cut from the same high RRR Nb sheet, prepared in the identical way, and compared after processing at different plasma conditions. The samples in the form of 1” or 2”-diameter disks were degreased before processing and, in the case of large mechanical damage of the surface, mechanically polished.

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Electric Discharge Reactors

The microwave cavity discharge system, shown in Fig. 1 is a typical “barrel” reactor, capable of maintaining plasma treatment with pressures up to 1 Torr. In the microwave discharge, the high energy transfer efficiency from the microwave electric field to the gas results in high electron and high radical densities in the plasma. These plasma conditions are more favourable for plasma etching than for sputtering processes. Also, the higher gas temperature in plasma contributes to a higher rate of chemical reactions and vaporization of chemical reaction products. Biasing a sample can increase the small value of the sheath potential. Nb samples are placed on ceramic holders in the central part of a reaction chamber. Low background gas pressure is obtained using a system of mechanical and turbo molecular pumps, both corrosive gases resistant. The gas in the reaction chamber has high constant flow rates, so that reactive species lost due to chemical reaction can be replenished and products of chemical reaction removed away from the sample. The gas flow control is done through flow meters connected to a controller. For processes that demand more than one reaction gas, a mixing chamber is placed in front of the reaction chamber to facilitate a better mixing of gases. The reactor allows an option of biasing the sample.

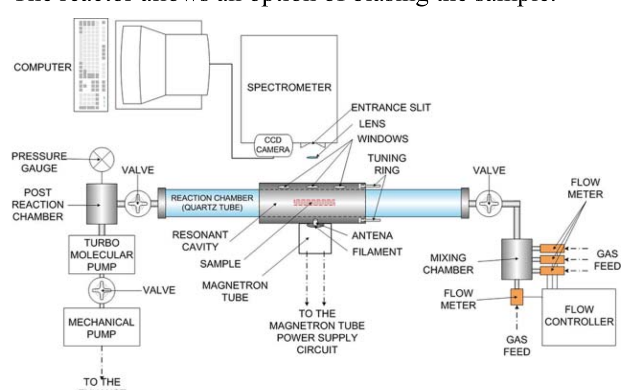

Figure 1: Microwave cavity discharge system.

The repetitively pulsed d.c. diode (PLAD) system operating in the voltage range 100 V – 20 kV at repetition rate between 500 Hz and 10 kHz was used for proof-of principle bulk Nb plasma etching [1]. This reactor is designed and operated by Varian Semiconductor Equipment Associates as a doping system and is described in more details elsewhere [2]. The reactor can be used in two operational modes: in the bulk mode and in the sheath mode. In the bulk mode, the sample is connected to the cathode that is negatively biased to generate the pulsed plasma, while the hollow cathode and the anode are grounded. In the sheath mode, shown in Fig. 2, the sample is also connected to the cathode, but the hollow cathode is charged by connection to the same high-voltage as the cathode.

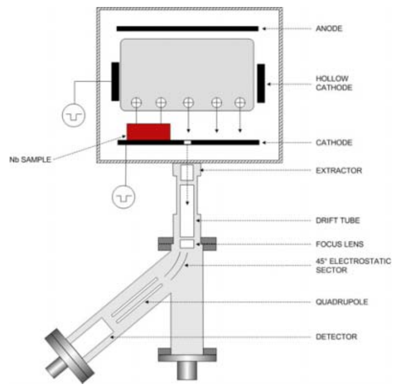

Figure 2: Repetitively pulse DC diode system [2].

Surface Characterization

We employed several standard surface characterization methods. For optical microscopy analysis, we used a Hirox KH-3000VD high resolution video microscope with magnification of 10×350 to obtain the qualitative and, to a certain extent, quantitative comparison of details from the surface of the samples. Waviness and roughness of the samples were analysed using a surface profilometer KLA TENCOR P-15. The analysis and comparison of samples surface morphology and composition was made with an AMRAY 1800 scanning electronic microscope combined with EDAX 9800 x-ray system. Tri-dimensional imaging and surface roughness analysis were performed using a scanning probe microscope (multimode SPM, Veeco) operating in the tapping mode. The scanned area was 10x10 μm^2. A comparative analysis was made with a sample treated by BCP.

RESULTS

Process Characterization

In-situ mass spectroscopy of the reactor gas, residual ions, and afterglow ion composition, combined with emission spectroscopy of plasma treatment products were used for process characterization. A series of low mass spectral peaks were observed in the presence of Nb samples, indicating rapid removal of impurities and minor etching of pure metal. The corresponding optical emission spectra show strong radiation from atomic oxygen, which was not observed in the absence of samples. The details on process characterization are given in Ref. [1].

Surface Characterization

Figures 3(a, b, c, d) show the micrographs of the surface for samples: (a) untreated, (b) treated in the microwave discharge system containing only Ar, (c) treated in PLAD system containing mixture of Ar and BF_3, and (d) treated by BCP technique. In all cases, the black line represents a distance of 10 µm. The untreated sample (Fig. 3a) shows large features (more than 50 µm long) up to 10 µm high. The plasma treated samples (Figs. 3b,c) show that the features are smaller by an order of magnitude and more oblique, with an average height in the order of 1-2 µm. Individual solitary features, more than 2 µm high, were also observed.

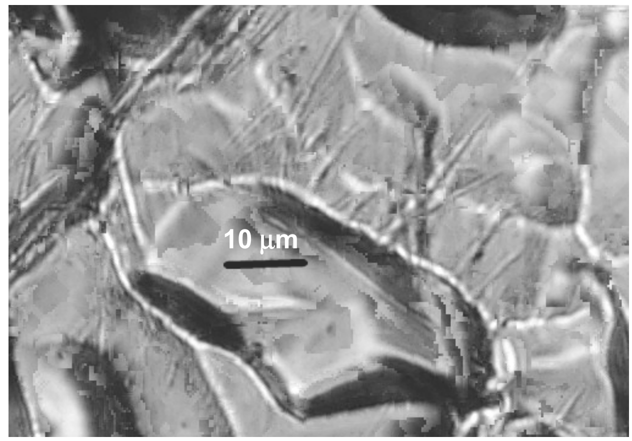

Figure 3a: The untreated sample surface micrograph.

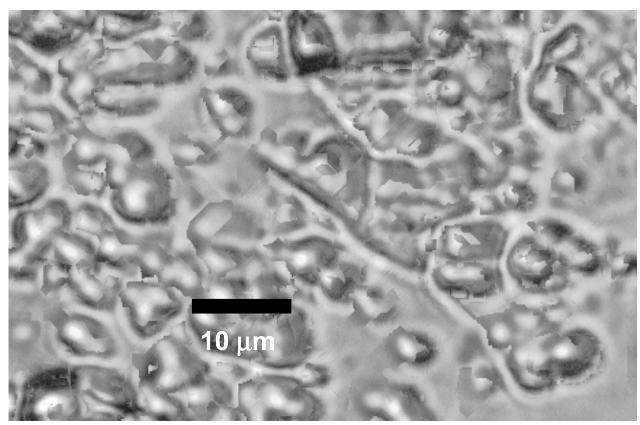

Figure 3b: The microwave plasma treated sample surface micrograph.

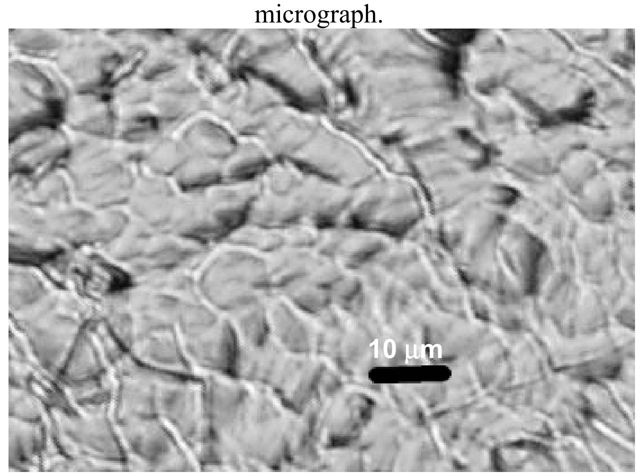

Figure 3c: The PLAD plasma treated sample surface micrograph.

The BCP treated sample (Fig. 3d) has large, but flattened features with sharp edges, and up to 3 µm height. All plasma-treated samples show qualitatively similar surface effects, but the sizes and height of the oblique wavy features differ substantially depending on plasma conditions. Similar surface effects were observed on scanning electron micrographs and scanning probe micrographs. Details are given in Ref. [1].

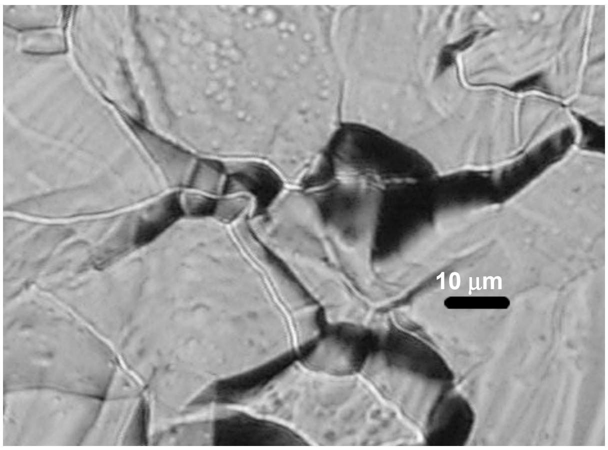

Figure 3d: The BCP treated sample surface micrograph.

KINETIC MODEL FOR PLASMA TREATMENT

We have initiated the development of a comprehensive kinetic model for plasma-surface interaction in order to complement process diagnostics and enhance its interpretation. Challenges facing this activity are multifold. The necessary cross sections and rate coefficients for electron-impact ionization of Nb-based molecules are not available in the literature. We calculated these data by employing the *ab initio* quantum chemistry models. Even in this approach, one had to rely on semi-empirical approximations, such as BEB [3], for cross-section calculations. Validation of the obtained data is limited by the lack of experiments in this area, and by the limited number of basis sets available for description of transition metal atoms. The best results were obtained by applying the core effective potential (CEP-31G) basis set. The highest occupied molecular orbital (HOMO) for stable Nb oxides, shown on the Fig. 4, were calculated using the CEP-31G basis set and the unrestricted Hartree-Fock method.

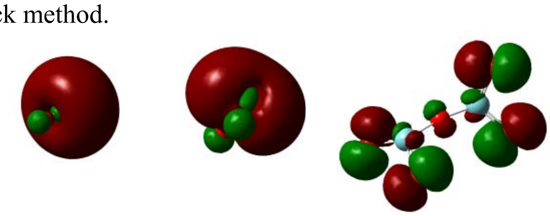

Figure 4: Schematic presentation of HOMO: NbO (left), NbO_2 (middle), and Nb_2O_5 (right).

Total cross sections for electron-impact ionization of NbO, NbO_2, and Nb_2O_5 are given in Fig. 5. Using the value of the total cross sections the rate coefficients were calculated for three different plasma modes: thermal, bulk, and sheath mode. All calculated data for elementary collision processes are currently being implemented in the plasma kinetic model. Details will be given in an upcoming paper.

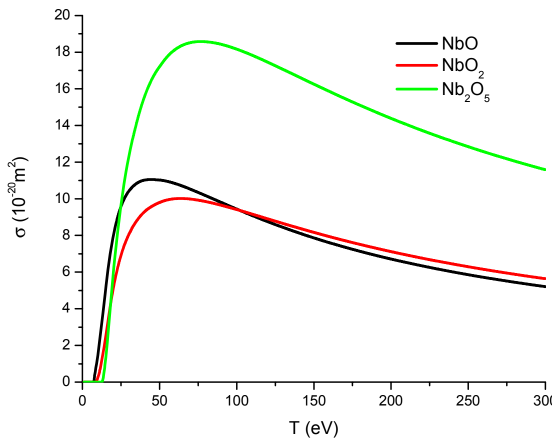

Figure 5: Total cross section for electron-impact ionization of stable Nb oxides.

CONCLUSION

The preliminary results show that plasma-treated samples are comparable or superior to a BCP sample, both in the size of features and sharpness of the boundaries between individual features at the surface. Images obtained with the optical microscope, the scanning probe microscope, and the scanning electron microscope show consistently the tendency of electric discharge plasmas to increase etching rates in the regions of enhanced electric field. This natural smoothing behaviour leads to oblique shapes of the grains and elimination of sharp edges.

ACKNOWLEDGEMENTS

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