STUDY OF ETCHING RATE UNIFORMITY IN SRF CAVITIES

J. Upadhyay, S. Popović, and L. Vušković
Department of Physics - Center for Accelerator Science, Old Dominion University, Norfolk, VA 23529, USA
A.-M. Valente-Feliciano and L. Phillips
Thomas Jefferson National Accelerator Facility, Newport News, VA 23606, USA

Abstract

Plasma based surface modification is a promising alternative to wet etching of superconducting radio frequency (SRF) cavities. The crucial aspect of the technology development is dependence of the etching rate and surface roughness on the frequency of the power supply, pressure, power level, driven electrode shape and chlorine concentration in the gas mixture during plasma processing. To optimize the plasma parameters, we are using a single cell cavity with 20 sample holders symmetrically distributed over the cell. These holders are used as diagnostic ports for the measurement of the plasma parameters and as holders for the samples to be etched. The plasma properties are highly correlated with the shape of the driven electrode and chlorine concentration in the Argon/Chlorine gas mixtures.

INTRODUCTION

To improve the RF performance of the SRF niobium cavities, the cavity surface must be prepared by a process that enhances surface smoothness, removes impurities and create less sharp grain boundaries. Currently used technologies are buffered chemical polishing or electro polishing. These technologies are based on the use of hydrogen fluoride (HF) in liquid acid baths, which poses major environmental and personal safety concern. HF-free plasma-based ("dry") technologies are a viable alternative to wet acid technologies as they are much more controllable, less expensive and more environment-friendly.

As a proof of concept we developed an experimental setup for etching of small niobium samples. The microwave plasma (2.45 GHz) frequency inside a quartz tube was used for this experiment. The gas mixture used was 97% argon and 3% chlorine. While the results with the flat samples were very encouraging [1], with etching rates up to 1.7 µm/min and surface roughness down to below 100 nm, the two parameters could not be achieved with the same treatment. Results are indicative of competitive character of the surface smoothness and etching rate. This is not a problem since the discharge parameters can be switched from one mode to the other during a single process. In every case, however, the surface roughness of plasma etched sample is equal or better than the chemically etched samples. The next step in the development of plasma etching technology for SRF cavity is to perform the etching of a single cell niobium cavity on optimized plasma parameters.

SINGLE CELL CAVITY EXPERIMENT

The cavity envelope is defined by the resonant low-loss requirement for maximizing the cavity Q factor and generating the accelerating gradients at microwave frequency of 1.5 GHz. It is designed to the resonant power coupling, maximum electric field at central axis, and minimum electric field at the cavity walls to avoid field emission. Optimal plasma etching conditions will require an electric field at the walls to produce the needed voltage for radial acceleration of reactive ions. Therefore, power coupling for plasma etching has to generate a TEM mode component. In this work we are studying two cases: (a) EM wavelength much longer than the cell longitudinal dimension, at frequency of 100 MHz, and (b) EM wavelength smaller than the cell length at frequency of 2.45 GHz. A specially designed diagnostic cell has been used for preliminary testing of the plasma uniformity and surface processing performance. The cell has a set of 20 sample holder ports that can be used as plasma observation windows or small sample holders for etching tests. To verify the non-uniformity and other plasma parameters in the cavity, a fiber-optic diagnostic system is developed. Five optical fibers of 1 mm diameter are placed with the help of a feed through at the 5 different hole positions on the cavity, as shown in Fig. 1.

Figure 1: Single cell experimental set up.

As the plasma properties and in turn the etching properties vary substantially with the frequency, pressure and power levels inside the etching reactor, we have to optimize these parameters for the most efficient and uniform surface material removal from the samples placed on the cavity perimeter.

RESONANT MICROWAVE BREAKDOWN

The breakdown electric field at any given frequency depends on the size of the cavity due to the diffusion loss of the electrons to the walls. Although microwave breakdown is essentially a time-dependent initial-value problem, we can use the stationary diffusion equation to evaluate the diffusion length and calculate the breakdown conditions. The stationary diffusion equation is obtained from the electron balance equation [2],

$$\frac{\partial n_e}{\partial t} - D\nabla^2 n_e = q \qquad (1)$$

where n_e is the electron density, D is the electron diffusion coefficient, and q is the gain (or loss) of electron density,

$$q = (\nu_i - \nu_a)n_e , \qquad (2)$$

Where ν_i is the ionization rate, and ν_a is the rate of electron attachment to chlorine atoms and molecules.

$$D\nabla^2 n_e + (\nu_i - \nu_a)n_e = 0. \qquad (3)$$

The breakdown condition is achieved when the electron density gain equals the loss by diffusion

$$\nu_i - \nu_a = \nu_{diff} = \frac{D}{\Lambda^2} \qquad (4)$$

where ν_{diff} is the rate of electron loss by diffusion, Λ is the effective diffusion length. Assuming that D, Λ, ν_i, and ν_a are uniform in the whole volume and that electron density is zero at the walls, the Eq. (3) is reduced to an eigenvalue problem. In the absence of magnetic field, the solution [3] for the cylinder of radius R and height L has the form

$$n_e(r,z) = n_{e0} J_0\left(\frac{2.405}{R}r\right)\cos\left(\frac{\pi z}{L}\right) \qquad (5)$$

where J_o is the zero-order Bessel function of the first kind and the eigenvalue is

$$\frac{1}{\Lambda^2} = \left(\frac{2.405}{R}\right)^2 + \left(\frac{\pi}{L}\right)^2 . \qquad (6)$$

Electron diffusion coefficient is given [2] by

$$D = \left\langle v^2 / 3\nu_m \right\rangle \approx \frac{\ell \tilde{v}}{3} \quad \Rightarrow \quad D \sim \frac{1}{p} \qquad (7)$$

where v and v are the velocity and the average velocity of electrons, respectively, ν_m is the effective collision frequency for electron momentum transfer, and ℓ is the corresponding mean free path. In order to continue the analysis of the resonant microwave breakdown we will approximate the elliptical cavity with equivalent pillbox geometry. The geometry of a single cell of the resonant pillbox is cylindrical with

$$R = \frac{c}{2.61\,\nu_{res}} \quad and \quad L = \frac{c}{2\nu_{res}} \qquad (8)$$

where c is the speed of light, and ν_{res} is the resonant frequency of the microwave field, which is 1.5 GHz in the present case. Then, the effective diffusion length in the absence of magnetic field is

$$\frac{1}{\Lambda} = \frac{\nu_{res}}{c}\sqrt{(2.405 \times 2.61)^2 + 4\pi^2} \cong 9\frac{\nu_{res}}{c} \Big\} \qquad (9)$$

Note that the diffusion length is five times smaller than the pillbox length L. Therefore, it is to be expected that the bulk plasma ionization process will cover the whole longitudinal dimension of the cavity. In the radial dimension, the effective diffusion length is smaller by 3.5 times than the effective pillbox radius. We are expecting plasma uniformity in the single cell at low pressure conditions (less than 5 Torr, approximately).

NON-RESONANT MICROWAVE BREAKDOWN

Since in our case the volume is large, additional electron loss in attachment and formation of negative ions as well as complex geometry lead to non linearity of Eq. (3), which often cannot be solved analytically without adequate approximation. Plasma etching is performed at two frequencies that are far from resonance for the particular SRF cavity with the purpose to generate radial electric field at the walls. The approach results in the so-called asymmetric RF discharge at 100 MHz, where the powered electrode is positioned along the axis [4], and the cavity walls acts as the grounded electrode. At 2.45 GHz the cavity is detuned by a rod antenna to obtain the optimum power coupling

To understand the effect of the length and diameter of the antenna we did CST Microwave simulation were performed on 1.5 GHz resonance cavity with 2.45 GHz frequency power supply. We varied the length of the antenna and look for the most uniform distribution of the electric field inside the cavity.

We found that a rod with small diameter and full length of the cavity would give the most uniform distribution of the field inside the cavity (See Fig. 2).

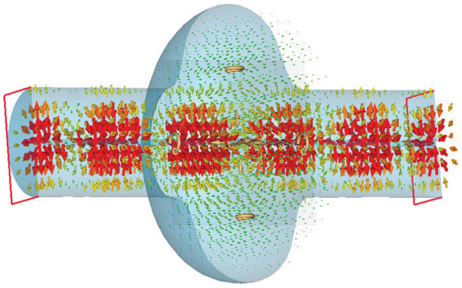

Figure 2: Electric field distribution in the detuned single cell cavity.

The set-up for the asymmetric RF (100 MHz) discharge with all details on the driven electrode was described in Ref. [4]. Similar power coupling has been applied to the microwave (2.45 GHz) discharge. The optimization for the electrode length and the uniformity of plasma parameters has to be done. The experimental approach for the optimization of these parameters was to perform the optical emission spectroscopy at different pressure and power at both the frequencies and compare the plasma parameters deduced from the spectroscopy results, etch the samples placed on the cavity perimeter and establish the relation between the plasma parameters and etching rates and surface roughness of the sample.

EXPERIMENTAL RESULTS

During the optical emission spectroscopy measurements of the single cell plasma, the cavity was exposed different powers and pressures for a limited time. Then measurement has been performed with gas mixture of 97% argon and 3% chlorine. Experiments are done at 0.05, 0.1, 0.5, 0.75 Torr pressure. Power is varied with the help of an attenuator. Photo of the discharge seen through the cavity holes is the first evidence of plasma uniformity (see Fig. 3).

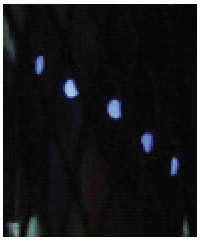

Figure 3: Plasma through the cavity holes.

It is very encouraging as the nonuniformity in optical intensity which directly correlates to nonuniformity of radicals is within 25%.

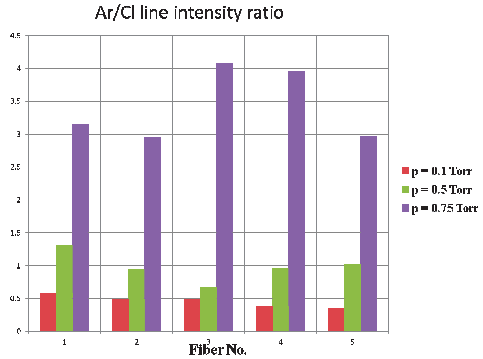

Figure 4: Relative intensity of argon and chlorine lines as a function of pressure in the cell.

Figure 4 shows the line intensity ratio of argon and chlorine at different pressures on different fibers. We draw two significant indications. First, there is no difference between the line intensity ratios from different fibers at the same pressure, which means that the requirement of plasma uniformity was satisfied. Second, with the increase of pressure, the ratio is increasing on each fiber, which means that the production of chlorine radicals is saturating at the higher pressures.

CONCLUSION

In view of the complex technological challenges facing the development of plasma-assisted surface treatment, we have adopted the following experimental approach: (a) to determine and optimize the plasma condition suitable for the uniform mass removal and optimum surface smoothness for the samples placed on the single cell cavity perimeter, (b) to etch a single-cell cavity at established optimum conditions for discharge in the cavity geometry, and (c) to perform RF performance tests compatible with existing standards. In both experiments, the central electrode (100 MHz) and antenna (2.45 GHz) is used to control the breakdown of the large-volume plasma and to assure its uniformity. The operating pressure range is set to the electron diffusion length comparable in size with the cavity dimensions, which has ensured the smoothening of the discharge into a homogeneous, diffuse mode, suitable for uniform etching. The RF performance is the single feature that remains to be compared to the "wet" process, since all other characteristics of the "dry" technology, such as etching rates, surface roughness, low cost, and non-HF feature, have been demonstrated as comparable to the currently used technologies.

AKNOWLEDGMENT

This work is supported by the Office of High Energy Physics, Office of Science, Department of Energy under Grant No. DE-SC0007879. Thomas Jefferson National Accelerator Facility, Accelerator Division supports J. Upadhyay through fellowship.

REFERENCES

[1] M. Rašković, J. Upadhyay, L. Vušković, S. Popović, A.- M. Valente-Feliciano, and L. Phillips, Phys. Rev. ST. Accel. Beams. 13 (2010) 112001.

[2] Yu. P. Raizer, *Gas Discharge Physics, 2nd printing,* Springer (1997).

[3] S. C. Brown, *Introduction to Electrical Discharges in Gases,* John Willey and Sons, Inc., (1966).

[4] J. Upadhyay, M. Nikolić, S. Popović, L. Vušković, L. Phillips, and A-M. Valente-Feliciano, Proceedings of SRF 2011, July 2011, Chicago, USA.