ETCHING OF NIOBIUM SAMPLE PLACED ON SUPERCONDUCTING RADIO FREQUENCY CAVITY SURFACE IN AR/CL$_2$ PLASMA

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Abstract

First results on plasma etching of single cell SRF cavity are presented. It has been proven with flat samples that the bulk Niobium (Nb) removal rate and the surface roughness after plasma etchings are equal to or better than wet etching processes. The plasma properties inside the single cell SRF cavity depend significantly on frequency, pressure and power. To understand the plasma properties and chemical kinetics of plasma etching process inside the single cell cavity, we are using a single cell cavity with 20 sample holders symmetrically distributed over the cell. These holders are being used for niobium coupon etching as well as diagnostic ports for optical measurements. Multiple optical probes with optical fibers have been utilized for optical emission spectroscopy measurements. A power supply in the radio frequency regime (100 MHz) and another power supply in the microwave frequency regime (2.45 GHz) are used to produce the plasma inside the cavity. The geometry of SRF cavities made of bulk Nb allows two optional discharge configurations (a) long wavelength asymmetric capacitively coupled RF discharge, where the wavelength exceeds the cavity length by an order of magnitude and (b) short wavelength coaxial cavity microwave discharge, where the wavelength corresponds to the similar order as iris to iris distance of the cavity. The plasma parameters at different pressure and power levels in combination with the analysis of the niobium sample etched will be used to determine the adequate frequency regime for plasma etching of Nb cavities.

INTRODUCTION

To achieve theoretically predicted values of accelerating fields, the surface of cavities must be prepared by a process that decreases surface roughness, produces surfaces with less prominent grain boundaries, and does not introduce additional impurities in the bulk of Nb. Plasma-based surface modification provides an excellent opportunity to achieve these goals. It is a crucial technology in the development of semiconductor circuit elements, and it has been applied in preparation of superconducting devices. However, it has not been considered so far as a viable alternative to the existing cost-intensive and environmentally unfriendly (liquid) acid-based technology.

We have shown that etching rates of bulk Nb as high as 1.5 µm/min can be achieved in a barrel electrode type microwave glow discharge using Cl$_2$ as the reactive gas [1]. This is a very promising result indicating that plasma etching could be a viable alternative to the environmentally problematic acid etching of SRF cavities due to the use of hydrofluoric acid in the chemical bath. A suggested process at high pressure (~1 Torr) and moderate input power density (~2 W/cm^3) has proven to be efficient enough to be employed for accelerator cavities.

SINGLE CELL CAVITY

Plasma etching in Nb single cell cavities and the RF performance of this cavity has to be tested by vertical testing method. We used a bell jar vacuum system (See Fig. 1) which has completely covered the SRF Nb single cell cavity. Discharge was generated inside the cell in the regime of capacitively coupled asymmetric RF discharge. A specially designed diagnostic cell is used for preliminary testing of the homogeneity of the plasma and surface processing performance. The cell has a set of 20 sample holder holes that can be used as plasma observation windows or small sample holders for etching tests. After completion of the tests with the diagnostic cell, a set of standard single cells will be prepared for vertical RF performance testing.

Figure 1: Single cell cavity experimental setup.

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Asymmetric RF Discharge

Due to the differences in the surface area of driven and grounded electrodes, there is an asymmetry in the voltage between the plasma and the driven electrode and between the plasma and the grounded electrode. It can be easily shown through the capacitor model [2] (See Fig. 2).

As smaller area has a smaller capacitance so it has a larger voltage drop. On the other hand, the larger area electrode has a large capacitance and a small voltage drop.
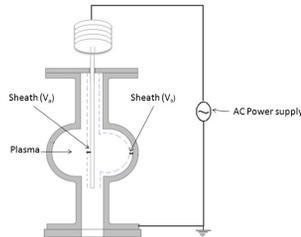

Figure 2: Scheme of asymmetric discharge.

The scaling of the voltage drop in the plasma sheath with the surface area of electrode is

$$\frac{V_a}{V_b} = \left(\frac{A_b}{A_a}\right)^{5/2} \tag{1}$$

As the ion bombarding energy is the sum of total energy gained by the sheath potential and the energy ion already had before entering into the sheath. The ion energy does not just depend on the electron temperature but it also depends on the source geometry and the application of a bias voltage. So the ion kinetic energy lost at the surface is [2]

$$E_i = V_s + \frac{T_e}{2} \tag{2}$$

$$V_s = \frac{T_e}{2} \ln\left(\frac{M}{2\pi m}\right), \tag{3}$$

where T_e is the electron temperature, M and m are the ion and electron mass, respectively. We designed our electrode in such a way that sheath voltage V_s is approximately 10 V at the grounded electrode sheath to ensure an efficient etching of SRF cavity surface, which is grounded in our case.

Fibre Optics Diagnostic System

To verify the non-uniformity and other plasma parameters of the plasma in the cavity, a fibre optic diagnostic system is developed. The driven electrode is 2.6 mm in diameter 17.2 cm long niobium rod and the cavity is grounded. Five optical fibres 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. 3). The optical emission spectroscopy of the plasma inside the cavity was simultaneously carried out from all the 5 fibres with the help of a spectrometer (Princeton instruments, Acton SP 2750) attached with a CCD camera (Apogee Alta model U1109) with 2048x506 pixel

matrix. For the purpose of simultaneously recording spectra through each of the fibres, the camera was set in the focal plane of the spectrometer so that rows of 2048 pixels were in the vertical direction and columns of 506 pixels in the horizontal direction. Each pixel had an area of 12x12 μm^2. In this arrangement, plasma emission spectra were recorded in the horizontal direction, and the individual fibres were discriminated in the vertical direction. The optical fibre can be recessed inside the ceramic tube to reduce the acceptance angle and protect from the reactive plasma. These optical probes can be moved with the help of these ceramic tubes, which enabled the measurements at different locations inside the cavity.

Figure 3: Single cell experimental setup for optical emission spectroscopy.

The fibres were positioned on the holes indicated in Fig. 4.

Figure 4: Plasma through the cavity holes.

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 surface material removal from the samples placed on the cavity perimeter.

The experimental approach for 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.

PRELIMINARY RESULTS

Optical emission spectroscopy has been done with two gas mixtures, one is 100% Argon and other is 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. The initial results are very encouraging.
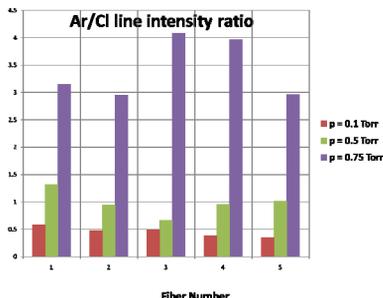

Figure 5: Relative intensity of Argon and Chlorine lines.

Fig. 5 shows the line intensity ratio of Argon and Chlorine at different pressures on different fibers. There are two indications that can be drawn from the diagram. First, there is no huge difference between the line intensity ratio from different fibers at the same pressure, which means that the requirement of plasma uniformity was satisfied. Secondly, with the increase of pressure, the ratio is increasing on each fibre, which means that the production of the chlorine radical is saturating at the higher pressures. During the optical emission spectroscopy measurements of the single cell plasma, we exposed the cavity to different power and pressure for a limited time. To check the uniformity of the etching of the samples placed on the single cell, we measured the weight of the samples before and after the plasma exposure. The positioning of the sample on the cavity is shown in Fig. 6.
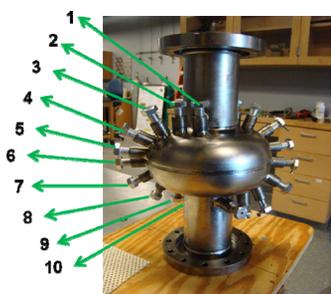

Figure 6: The sample number placed on actual shape of cavity.

The relation between the removed mass and number of the sample which were placed at different places on the cavity perimeter is shown in Fig. 7.

This diagram tells us that there is not too much variation in mass removal dependence on the place where each sample was placed. Detailed experiments would be carried out in future but preliminary results

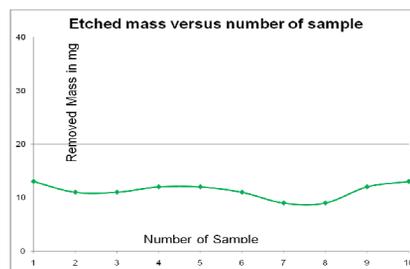indicate that etching is possible with the help of RF power source.

Figure 7: The number of sample placed on cavity versus mass removed due to plasma etching.

As these samples were exposed to different pressure and power for different time we cannot establish firm information on the dependence of etching rate on pressure and power. However, the uniform removal of a layer with thickness measured in tens of micrometers is an encouraging preliminary result.

CONCLUSION

In view of the relatively complex technological challenges facing the development of plasma-assisted surface treatment, we have adopted this 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 the RF performance test compatible with existing standards.

Based on highly encouraging results with flat Nb samples and on a relatively straightforward transition to the cavity wall processing, we can state confidently that efficient plasma etching of Nb superconductive RF cavities can be developed into a low-cost, environment-friendly technology to replace the "wet process", which uses a strong mixture of acids, including HF.

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 superior or comparable to the currently used technologies.

REFERENCESS

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