OPTIMIZATION OF PLASMA PARAMETERS FOR ETCHING OF SUPERCONDUCTING RADIO FREQUENCY CAVITY SURFACE IN Ar/Cl_2 PLASMA

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Abstract

We are pursuing the development of low cost environmentally friendly dry etching of superconducting radio frequency (SRF) cavities in Ar/Cl_2 discharges. 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 on frequency, pressure and power. To understand the plasma properties and chemical kinetics of the 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 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 improve the RF performance of the SRF niobium cavities, the cavity surface must be prepared by a process that improves surface roughness, removes impurities and creates less sharp grain boundaries. Currently used technologies are buffered chemical polishing or electro polishing. These technologies are based on the use of hydrogen fluoride in liquid acid baths, which poses major environmental 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. We have seen that plasma etching technology has replaced the wet etching process in the semiconductor industry

While the results with flat samples were very encouraging, 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. In every case, however, the surface roughness of plasma etched sample is equal or better than the chemically etched samples [1].

SINGLE CELL CAVITY EXPERIMENT

In order to test the RF performance, plasma processing has to be applied to a single cell cavity. Cavity shape is defined by the resonant low-loss requirement for generating the accelerating gradients. This design does not favor the electric configuration on the surface that is optimal for plasma etching. Therefore, the generic etching configurations are capacitive coupled radio frequency or a coaxial microwave discharge. A specially designed diagnostic cell has been used for preliminary testing on homogeneity of 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 cryogenic RF performance testing.

Figure 1: Single cell cavity with sample holders.

To verify the non-uniformity and other plasma parameters of the plasma in the cavity, a fibre optic diagnostic system was developed. The driven electrode is 2.6 mm in diameter 17.2 cm long niobium rod and the

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cavity is grounded. Five optical fibres of 1 mm diameter are placed with the help of a feedthrough at the 5 different hole positions on the cavity. 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.

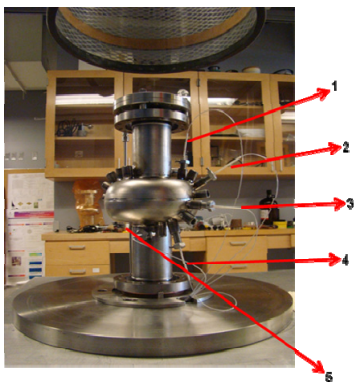

Figure 2: Single cell experimental set up.

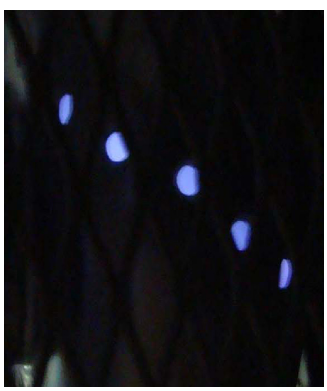

Figure 3: Plasma through the cavity holes.

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.

To achieve this goal we had a choice of two power supplies, one in radio frequency regime at 100 MHz (500 W) and other in the microwave frequency regime at 2.45 GHz (1.2KW).

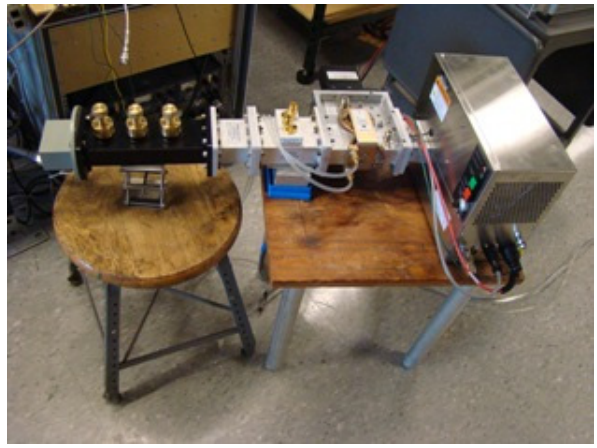

Figure 4: Microwave power supply with circulator, tuner and power meter.

Figure 5: Radio frequency power supply with matching network and power meter.

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.

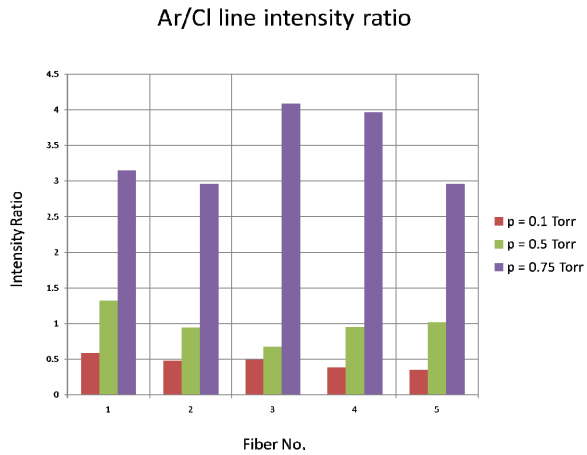

Figure 6: Relative intensity of argon and chlorine lines.

In Figure 6 is shown 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 not huge difference between the line intensity ratios 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.

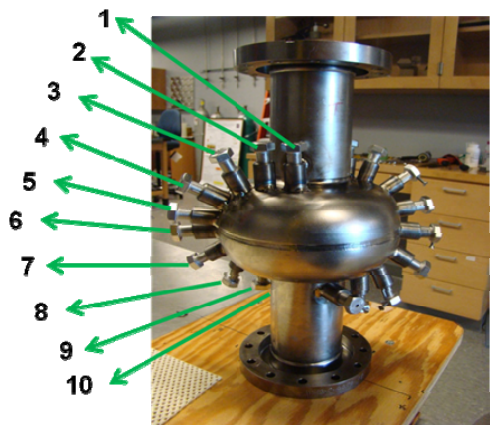

Figure 7: The sample number placed on actual shape of cavity.

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

This diagram in Fig. 8 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. 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.

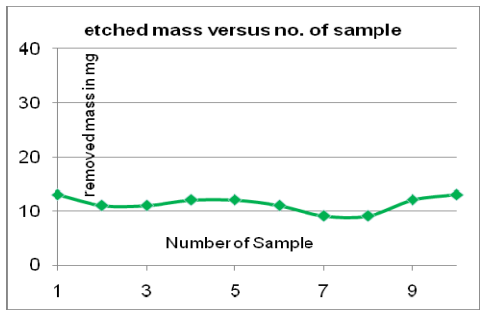

Figure 8: Material removed by plasma etching as a function of sample position on cavity surface.

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.

REFERENCES

[1] M. Rašković, S. Popović, J. Upadhyay, L. Vušković, L. Phillips, and A.-M. Valente-Feliciano, J. Vac. Sci. Technol. A 27 (2) (2009) 301.

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

[3] M. Rašković, L. Vušković, S. Popović, L. Phillips, and A.-M.Valente-Feliciano, S. B. Radovanov, and L. Godet, Nuclear Inst. & Methods in Physics Research A 569 (2006) 663.