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Cryogenic rf test of the first SRF cavity etched in an rf Ar/Cl_2 plasma

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An apparatus and a method for etching of the inner surfaces of superconducting radio frequency (SRF) accelerator cavities are described. The apparatus is based on the reactive ion etching performed in an Ar/Cl_2 cylindrical capacitive discharge with reversed asymmetry. To test the effect of the plasma etching on the cavity rf performance, a 1497 MHz single cell SRF cavity was used. The single cell cavity was mechanically polished and buffer chemically etched and then rf tested at cryogenic temperatures to provide a baseline characterization. The cavity's inner wall was then exposed to the capacitive discharge in a mixture of Argon and Chlorine. The inner wall acted as the grounded electrode, while kept at elevated temperature. The processing was accomplished by axially moving the dc-biased, corrugated inner electrode and the gas flow inlet in a step-wise manner to establish a sequence of longitudinally segmented discharges. The cavity was then tested in a standard vertical test stand at cryogenic temperatures. The rf tests and surface condition results, including the electron field emission elimination, are presented. https://doi.org/10.1063/1.4991888

I. INTRODUCTION

Superconducting radio frequency (SRF) cavities are integral components of accelerators used in nuclear and high energy physics research. A standard material for the SRF cavities is bulk niobium, although other, more complex materials in the form of thin films are currently being explored. At present, the inner surfaces of SRF cavities are chemically treated (etched or electro-polished) to remove impurities, mechanically damaged layers and reduce the surface resistance of the superconducting surface, thus achieve a favorable RF performance. These technologies are based on the use of hydrogen fluoride (HF) in liquid acid baths,[1–6] which poses a major environmental and personal safety concern. A secondary consequence of the acid processing technique is the irregularities in the processed superconductor surface, causing excessive secondary electron activities like secondary electron emission and field emission. A plasma etching method would present a much more controllable, less expensive, and more environment-friendly processing technology. This competitive alternative would also provide the unique opportunity to modify the niobium (Nb) surface for energy efficient superconducting RF properties. In our research, we are developing plasma-processing technology (etching technology, see Refs. 7–9, 11) that has the ability to remove hundreds of microns of defective layers of niobium while the intrinsic properties of the discharge are able to induce unipolar sparks at the local micro protrusions, thus melting and removing them as a source of field emission. This technology will be applied after the cavity fabrication and before inserting the cavities into the cryomodules. Once completely developed, it would be able to replace the existing chemical etching technology. The plasma etching method described here uses a coaxial capacitive radiofrequency discharge of Ar/Cl_2 mixture operating at 13.56 MHz. The Cl_2 gas used in the process forms volatile compounds in reaction with the Nb and its oxides in an rf plasma environment.

Before plasma etching a single cell SRF cavity, ring type Nb samples were used in both a single-diameter and a varied-diameter cylindrical cavity to measure the effect of the process parameters on the Nb etching as reported in Refs. 7, 8. Applying a positive dc bias to the inner electrode and changing the contour of the inner electrode compensated the sheath voltage asymmetry due to a lower surface area of the inner electrode in the coaxial plasma. The etch rate dependence on pressure, rf power, Cl_2 concentration and diameter of the inner electrode was measured and reported in Ref. 7. The etch rate dependence on the temperature, dc bias and understanding of the etch mechanism was reported in Ref. 8. It was found that there is a strong etch rate non-uniformity in the direction of the gas flow. Its dependence on the process parameters is also reported in Ref. 8. The concept of surface enhancement of the inner electrode to partially overcome sheath voltage asymmetry was applied and various structures were tested.[9] The optimum, corrugated structure for reversal of the asymmetry has been determined.[9] A stainless steel pillbox cavity was chosen with the aim of studying the plasma processing effect on varied-diameter structures, as uniform plasma-surface interaction is a challenging task.[10] The lesson learned from the pill box cavity experiment was that the etch rate in the beam tube is extremely high compared to the equatorial locations. The segmented plasma production by moving the inner electrode and gas flow inlet in a stepwise manner was chosen to reduce this problem. The apparatus and method developed for segmented plasma production is reported in Ref. 11. The cryogenic rf test of an actual plasma etched SRF cavity was chosen as the only diagnostic tool left to compare the reactive ion etching in a capacitive RF discharge with the acid chemical etching technologies. In comparing the RF performance the approach was to use an unprocessed single cell SRF cavity, perform the buffer chemical processing (BCP) and apply the cryogenic RF test. The same cavity was then plasma etched and retested at cryogenic temperature.

II. APPARATUS AND METHOD

The apparatus, shown in Fig. 1, was used to plasma etch a single cell SRF cavity. It consisted of an rf power supply, which was equipped with a matching network and connected in series to a dc power supply that provided a positive dc bias to the inner electrode. RF power was coupled to the inner (driven) electrode with a coaxial atmospheric pressure feedthrough. The feedthrough and the inner electrode are attached to a controllable axially moving manipulator, which is shown on the left side of Fig. 1. The cavity, acting as a vacuum vessel, was connected through the antechamber to the pumping system, which consists of a turbo molecular vacuum pump and a roughing pump with vacuum valves and diagnostic gauges. The exhaust gases are collected and processed in a homemade scrubber that is filled with a sodium hydroxide solution in water. Gas is fed to the system through a mixing manifold and a specially designed gas inlet, which disperses the gas mixture. The gas inlet was connected to a second controllable axially moving manipulator, which is synchronized with the first manipulator, as shown in the right side of the image. The gas inlet is a double conical shaped structure as described in Ref. 11. The gas flow inlet was a part of the experimental setup, and it was electrically grounded. The inner electrode was corrugated and made of stainless steel. It was 9.0 cm long, which is smaller than the length of the cell structure.

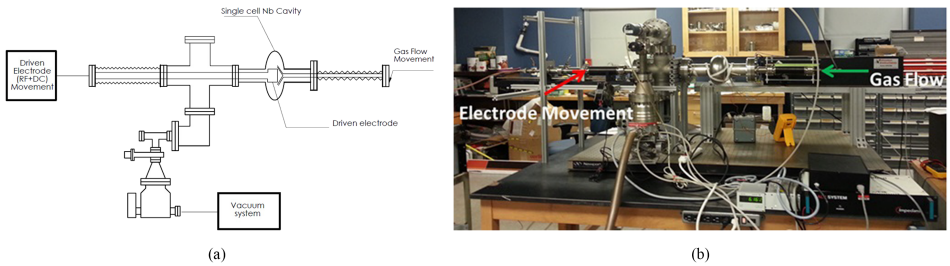

FIG. 1. (a) Schematic diagram of experimental setup of single cell cavity plasma etching system. (b) The photo of experimental setup to plasma etch single cell SRF cavity.

The cavity wall was electrically grounded and served as the outer electrode of the cylindrical rf discharge. Heating tape was wrapped around the cavity and it was attached to a variable-voltage transformer to control the surface temperature during the etching process. The surface temperature was monitored using a thermocouple and a multimeter.

III. RESULTS AND DISCUSSION

A freshly made single cell 1497 MHz SRF cavity was mechanically polished to mirror finishes (details of mechanical polishing procedures are published in ref. 6) and then buffer chemically etched for an additional 60 microns of material removal. The single cell cavity was then heat treated in a vacuum furnace at 600^0 C for 10 hours, degreased and high-pressure water rinsed. The cavity was tested at cryogenic temperatures at the Jefferson Lab VTA facility. The Q_0 vs. E_{acc} test results are shown later in the figure 3. The details about test methods can be found in Ref. 12. This BCP cavity shows field emission around 15 MV/m as reported by the curve with the BCP.

The cavity was then plasma etched for 24 hours. The conditions during plasma etching were as follows: pressure 50 mTorr, rf power 160 W, dc bias 320 V, temperature 231 ^{0}C and dc current 0.930 A. The gas flow rate was 0.43 l/min and the gas mixture used was 15% Cl_2 mixed with Argon. The uncertainty of Cl_2 concentration was 2%, in rf power 10 W, in pressure 4 mTorr, in dc bias 10 V and in dc current 10 mA. The Nb removed from the cell structure was on the order of 10 μm, while the material removed from the beam tube was on the order of 100 μm. The separation between inner electrode and gas flow inlet during the plasma etching process was kept constant at 5 cm. The powered electrode was positioned at the beginning of the etching at one end of the cell and moved sequentially towards the other end of the cell. The beam tube plasma etching occurred due to the expansion of the plasma produced during cell etching.

After 24 hours of plasma etching, the cavity was kept at the same temperature for 10 additional hours and the vacuum pumping system was active for an additional 8 hours. The cavity was then opened to atmospheric pressure. A thick black residue (approximately 0.5 mm thick) was found on the cavity surface. This residue was collected and analyzed.

Surface analysis of the residue shows it to be Fe, Ni, Cr and Cl_2, which suggest that the inner electrode and gas flow inlet, which were made of stainless steel were plasma etched and fell on the SRF cavity surface. The visual inspection of gas flow inlet and the elemental analysis of the residue confirms this. The images from the surface analysis are shown in Fig. 2.

The plasma etched cavity was water rinsed, ultrasonically cleaned and high pressure water rinsed at Jefferson Lab. The cavity was then rf tested at cryogenic temperatures. The results of the test indicated that the quality factor was reduced by an order of magnitude compared to BCP cavity and quenching at around 20 MV/m though field emission shows significant reduction as shown by the curve (BCP+PE) in Fig. 3. There were two strong possibilities for this degradation in the quality factor; one could be the deposition of non-superconducting material on the cavity surface, the other would be a large amount of hydrogen present in the bulk, which decreases the quality factor. This is known as a "Q-disease."

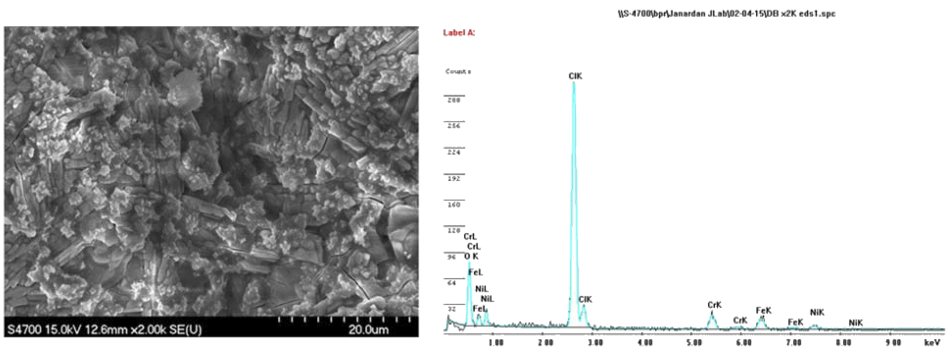

FIG. 2. The images of surface analysis (left) and elemental composition (right) of the residue from the plasma etched cavity.

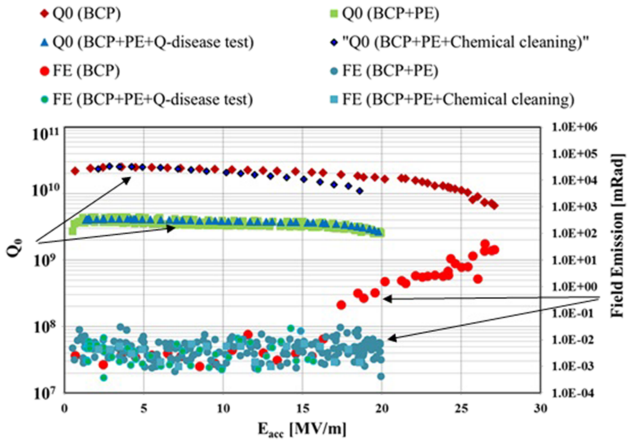

FIG. 3. The rf performance test results of the plasma etched elliptical SRF cavity at 1.8 K. The top portion of the graph presents Q_0 and the bottom portion presents field emission as shown by the arrows.

To test for Q-disease, the cavity was kept at 90K to 140 K over 14 hours and tested. The test result shows no hydrogen disease, as the Q curve looked exactly the same as the rapidly cooled cavity Q curve as shown in Fig.3.

To test the possibility of the stainless steel residue being the cause of the Q degradation, the cavity was chemically cleaned with an aqua regia solution, and phosphoric acid. The use of HF was avoided in order not to disturb the Nb oxide surface and protect the Nb from any etching. During the phosphoric acid cleaning the temperature of the acid was raised to 100^0 C for 60 minute. The cavity was degreased and high-pressure water rinsed and tested again. The removal of stainless steel residue helped and the quality factor of the cavity returned to the BCP cavity level.

Metal particulates are a usual artifact of the cavity manufacturing process when exposed to external fields. The particulate tend to enhance the electric field and emit field electrons. The field emission electrons have been a subject of many analyses and are a phenomenon that is included in the SRF cavity design. When these electrons hit the wall, they generate radiation, which is detected and recorded as shown in Fig. 3 as the integral radiation rate from the cavity, measured in coincidence with Q_0 during the cryogenic test. The onset of radiation coincides with the drop (or variation) of Q_0. The rate varies exponentially with the field according to the modified Fowler-Nordheim relation. The slope of the exponential increase with accelerating field is related to the average size of particulates. A quite similar phenomenon in the presence of plasma discharge is related to the sheath modification around the particulate, field enhancement in the sheath, and subsequent field electron current that develops into a spark, which heats the particulate, melts and destroys it. As a result, there was no sign of field emission during this chemically cleaned plasma etched cavity test as shown in Fig. 3 (BCP+PE+Chemical cleaning). A similar observation on reduction of field emission due to plasma surface interaction was observed and reported for flat samples in Ref. 13.

Therefore, the earlier degradation of Q factor was due to stainless steel residue deposition on the surface of the SRF cavity. The conclusion is that all components (inner electrode, gas flow inlet) used in future plasma etching apparatus should be made of Nb and electrically biased to prevent these components from plasma etching. All the etched Nb was not removed from the system due to the huge amount of material etched and partial condensation on some surfaces. Raising the temperature and using high pumping speed could improve the purity of the cavity surface.

IV. CONCLUSION

We presented the experimental setup and procedure to etch a single cell SRF cavity in an rf plasma discharge and the rf test results of the first plasma etched SRF cavity at cryogenic temperature. The test results suggest that there is a possibility that the plasma-etched cavity would perform as good as a chemically etched cavity if the components (driven electrode, gas supply system, electrical

feedthrough etc...) used during the processing are made of Nb or electrically isolated, so that the processing plasma does not etch these components. As the stainless steel electrode used during this experiments got plasma etched and fell on the cavity surface.

The plasma etched cavity has shown no field emission. The field emission comparison was made after the cell was BCP+HPR (high pressure rinsing) treated and PE+HPR treated. If the simple HPR does remove the particulates as field emission sources, then field emission would not be revealed after the BCP+HPR treatment. Since a reduction in the field emission appeared only after PE+HPR, this test suggests that a mechanism related to PE is the agent for the reduction in field emission. Field emission in the plasma processed cavity did not increase even after multiple chemical cleanings and testing.

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