BUFFERED CHEMICAL POLISHING DEVELOPMENT FOR THE BETA=0.53 HALF-WAVE RESONATOR AT MICHIGAN STATE UNIVERSITY*

L. Popielarski, L. Dubbs, K. Elliott, I. Malloch, R. Oweiss, J. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA

Abstract

The beta=v/c=0.53 half wave resonator (HWR) is being developed for the Facility for Rare Isotope Beams (FRIB). One prototype resonator has been built completely at MSU and four other resonators have been built by industry. The proposed surface treatment is buffered chemical polishing (BCP) and high pressure rinsing with ultra pure water. The BCP process is being optimized to achieve resonator performance goals during certification testing. Research is focused on the improvement of the damaged layer removal (uniformity), the heat exchanger design, and the quality of BCP acid solution. Several etches have been completed on the HWRs. Process data such as: removal rate, temperature profiles, and niobium concentration in solution have been collected. The process data was studied versus the vertical test results; maximum accelerating voltage, quality factor and field emission onset voltage. The chemistry fixture development and process data versus test results will be presented.

INTRODUCTION

The FRIB project requires a total of 341 certified SRF cavities. This includes 144 of the beta=0.53 HWR type. After final fabrication, the internal surface of the cavity is chemically etched to remove a damaged layer of 100-150 microns. The cavity is then degreased, heat treated at 600 °C for 10 hours for hydrogen degassing. After heat treatment the cavity receives a light etch and high pressure rinse and then is assembled for vertical testing.

The finished cavity with helium vessel weighs 230 pounds (104 kg) and has an internal surface area of about 1.11 m^2. The internal volume is 13 gallons (49L). A standard 1:1:2 BCP recipe of 1 part HF (49%), 1 part nitric acid (79%), and 1 part phosphoric acid (85%) is used. The acid temperature is cooled to and maintained at 13-17 °C, by flowing the acid through a storage tank with a submersed Teflon loop heat exchanger with 10 °C chilled water flowing. The acid flow rate is 8-10 gpm (30-38 lpm) and is continuously filtered in the recirculating loop. Cavities without helium vessels are cooled by wrapping with ice packs, and cavities with helium vessels are cooled with cold water flow through helium space

CHEMICAL PROCESS DEVELOPMENT

Before the cavity is processed, a predicted etch rate is determined by way of an etch rate sample test. Eight 2 mm thick niobium samples are etched for 25 minutes. Weight and thickness measurements are taken before and after etching.

First BCP Set-Up

The cavity is oriented horizontally as shown in Figure 1. The RF ports at the top and bottom. The acid is pumped up through the bottom RF port and exits at the top RF port. The beamports and cleaning ports are sealed with Teflon flanges. A niobium in-situ sample is mounted on one of the beam port blanks.

Figure 1: Orientation of HWR for first BCP etching set-up.

In addition to the etch rate sample test, thickness measurements have been performed on various locations of four HWRs before and after etching. The thickness measurement was performed using an ultra sonic thickness measurement (USTM) device and was used to determine the material removal at a given point. The average of these etch rates resulted in an average for the entire cavity. This etch rate was compared to the predicted etch rate (PER) found by the sample test.

Information from both the predicted etch rate and the thickness measurements is used to estimate an etch time required for the desired average material removal. We found that the removal measured by USTM was 30% of the removal estimated from the predicted etch rate.

The material removed (measured by USTM) at different locations on the cavity indicated the etching was non-uniform. The removal along the beam port quadrants (sides) of the cavity was much higher than desired removal. The removal at the top and bottom (acid in/out) RF ports was much lower than desired. The removal near the inner conductor center was also much higher than desired removal. Very little removal was seen at the top and bottom quadrants.

* This material is based upon work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661

The figure below illustrates the non-uniformity of the etching.

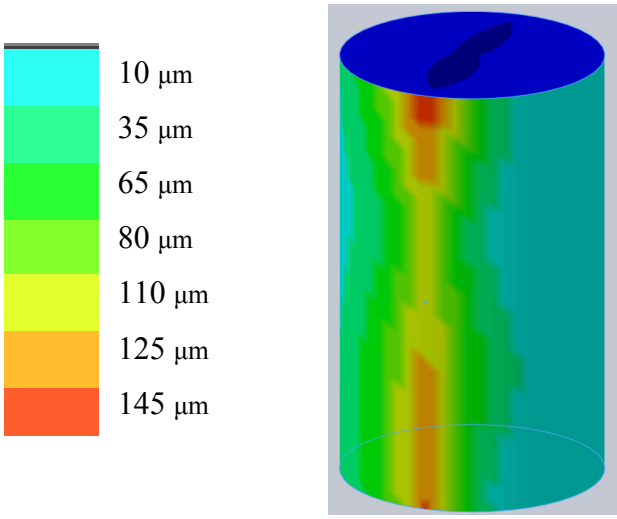

Figure 2: Cavity HWR_003 1st etch. Desired removal: was 100µm.

Notice in Figure 2 that the maximum removal (shown in red) is 14 times greater than the minimum removal. Also, the majority of the cavity receives much less removal than the desired 100 microns.

Flow Simulations

Flow simulations, using SolidWorks FlowXpress Analysis Wizard, were performed to better understand the BCP flow in the cavity.

The flow model indicates that a relatively high velocity flow impinges directly onto the surface of the inner conductor. As expected, USTMs at this location show a much higher etch rate than other locations on the cavity.

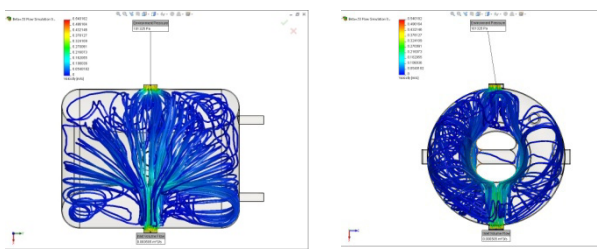

Figure 3: Flow simulation of first chemical etch set-up. BCP enters through bottom port and exits through top RF port.

Second BCP Set-up

Numerous (12) flow studies were completed to determine an etching configuration that would improve the average etch rate and uniformity of the cavity. We arrived at a design which incorporated 4 PTFE dispensing quills 8.75" long and 0.9" in diameter with a 0.375" hole on the side of the quill near the tip. The quills protrude into the cavity through the cleaning ports, and are aligned such that they all face clockwise. The BCP exits the cavity through the top RF port. All of the dispensing quills are pointing clockwise, causing a swirling pattern to develop as shown in Figure 4.

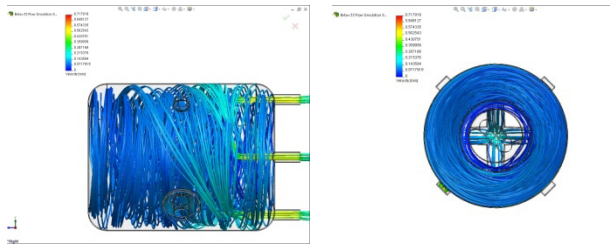

Figure 4: Flow simulation of chosen design. BCP enters through 4 quills and exits through top RF port.

The new dispensing quill fixtures were used for the bulk etch of HWR_004.

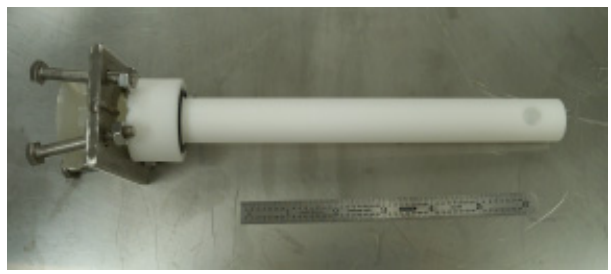

Figure 5: A BCP dispensing quill.

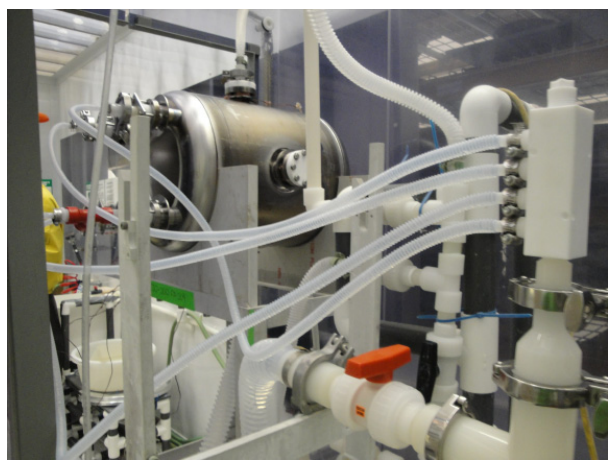

Figure 6: BCP etching configuration for the second set of fixtures using dispensing quills.

The new fixtures were effective in bringing the etch rate at all points closer to the PER and increasing uniformity. However, they caused a peak removal of material at the location where the BCP exits the dispensing wand, striking more directly on the outer conductor.

The maximum removal on HWR_004 1st etch (Figure 7) is 3.5 times greater than the minimum removal. Though this is an improvement over the 14x ratio seen in HWR_003 1st etch (Figure 2) it does leave room for improvement. This maximum removal is seen as the red area in Figure 7 below.

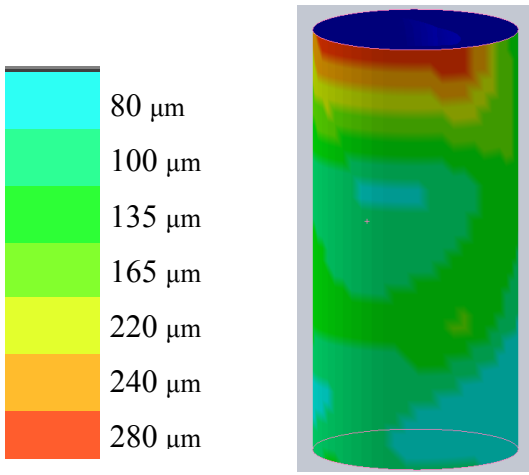

Figure 7: Cavity HWR_004 1st etch. Desired removal was 135 µm.

The average etch rate increased to 46% of the PER. This represents a 50% increase in etch rate over the first etching method. Areas that experienced minimal removal with the first set-up now experience removal closer the desired amount.

The fixtures will be further optimized to reduce the peak removals (300 µm) by adjusting the angle that the BCP exits the dispensing wand. This will reduce differential etching caused by direct impingement while still providing the desired swirling effect.

HEAT EXCHANGE & AVERAGE ETCH RATE: A THERMODYNAMIC APPROACH

Cavities which have helium vessels employ a cool water heat exchanger to remove heat generated during etching. Cool (10-13 °C) water flows through the helium vessel at a rate of 1-2 gpm.

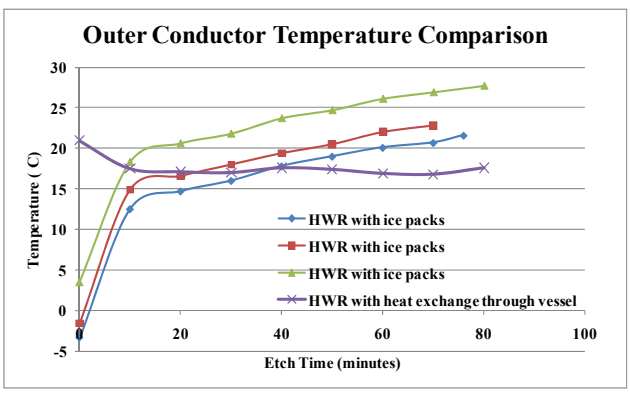

Figure 8: Plot of Outer Conductor surface temperature during etching.

The heat exchanger has proven to be effective at maintaining cavity surface temperatures below 20 °C (Figure 8).

In addition to cooling the cavity, the heat exchanger allows us to measure the cooling water's flow rate, as well as the inlet to outlet temperature change. Thermocouples have been installed in the BCP system to measure the BCP temperature at the inlet and outlet of the cavity. Measuring the BCP flow rate completes the thermodynamic model [1] for average etch rate.

The average etch rate at any time during an etch can be calculated using the following equation (1):

$$E.R. = \frac{\dot{m}_{H2O} c_p (T_{out} - T_{in}) + \dot{m}_{BCP} c_p (T_{out} - T_{in})}{1183.8 \frac{W}{m^2 \frac{\mu m}{min}} (S.A.)}$$

Integrating the average etch rate over a given time interval will give a total etch removal in microns.

This method of determining etch rate has been used experimentally on 5 prototype cavity etches. The average etch rate (by thermodynamic model) of the 5 etches was 43.6% of the PER, with a standard deviation of 2.2%. This correlates well to the 46% of the PER calculated by USTMs, and the 40% of the PER calculated by mass change analysis.

Future Plans

The use of the thermodynamic model to eliminate the etch rate test from our standard processing work schedule is desired. More work is required to validate the accuracy of the thermodynamic model before it can be used for production processing.

A test structure should be used to confirm that the amount of niobium removal calculated with the thermodynamic model is validated by a change in mass of the test structure. A program can be developed that will take temperature and flow measurements at regular intervals and convert them into a cumulative removal.

By completing these tasks the etch rate test can be eliminated from the standard processing work schedule.

CAVITY VERTICAL TEST RESULTS

For all of the HWR processes, the total cumulative etch removal and the niobium concentration in solution at the start of the etch cycle is recorded. This data is compared with vertical test data such as quality factor (Q_0), peak surface electric field (E_p) at field emission onset (x-rays > 1mR/hr) and E_p at the maximum field emission level. Additional information on the test data can be found in these proceedings [2]. Three HWRs have been processed and tested, for a total of nine vertical tests. The cumulative etch removal ranges from 98 to 215 microns, and the niobium concentration ranges from 0.1 to 35 grams per liter in BCP at the start of etch.

It is premature to make conclusive correlations with such a small data set of 9 points. Also, other factors may have had more of an effect on the test results as different procedures were also being optimized. Based on the data collected so far, there is little relation between the niobium concentration in the BCP solution and field emission onset or the maximum x-ray levels (Figure 9 and 10).

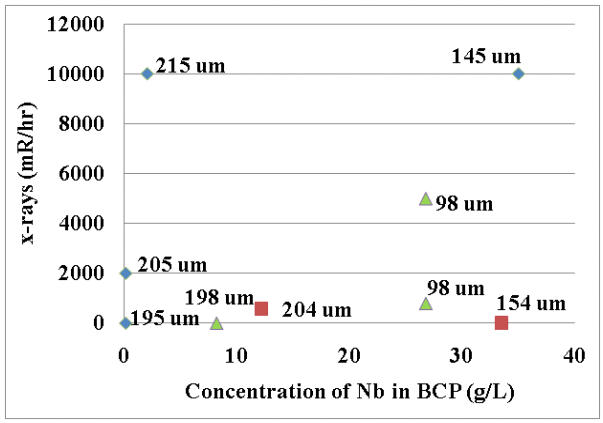

Figure 9: The maximum field emission seen during vertical testing for three different cavities vs. concentration of Nb in BCP at start of etch prior to test.

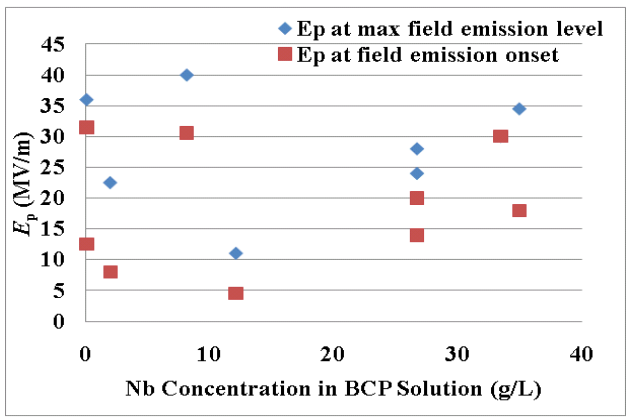

Figure 10: The E_p at field emission onset and at max field emission level vs. the concentration of Nb in BCP solution at start of etch prior to test.

The cumulative etch removal data versus the quality factor and E_p indicates there may be an optimal material removal range between 150-200 microns (Figure 11 & 12).

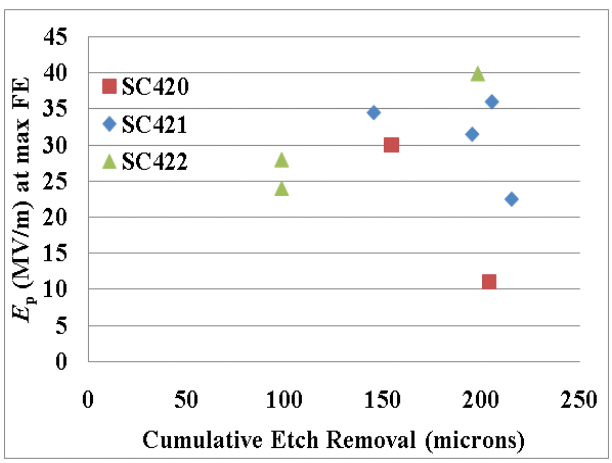

Figure 11: E_p at maximum field emission levels vs. the cumulative etch removal for three different HWRs.

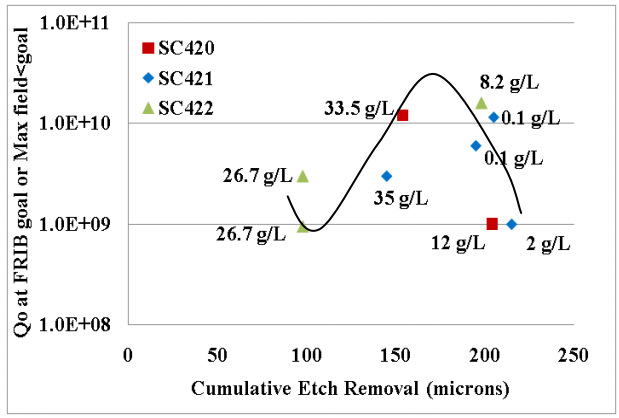

Figure 12: Q_o at E_p =31.5 MV/m or maximum field vs. cumulative etch removal for 9 processes.

CONCLUSION

The chemical processing procedures have been improved since the prototype cavity. The processes are being optimized for FRIB production processing, to improve the surface quality, repeatability, and reduce process work schedule time. Many more cavity processes and tests are necessary to build a statistical base, with which we can better define the acid quality and total removal necessary to achieve the cavity requirements.

ACKNOWLEDGMENTS

The authors would like to thank all the members of the NSCL & FRIB staff whose dedicated effort has made a significant contribution to the progress. In particular, Steve Bricker, Chris Compton, Alberto Facco, Gerald Gentz, Matt Hodek, Tim Kole, Doug Miller, Sam Miller, Dave Norton, Katie Pomaville, Nicholas Putman, Greg Velianoff, Ken Witgen and Jon Wlodarczak. We also would like to acknowledge the impact of our weekly SRF teleconference on the overall success of our program, with special thanks to Curtis Crawford, Walter Hartung, Bob Laxdal, and Peter Kneisel for their continued contributions.

REFERENCES

[1] Heat of Reaction of Niobium in Buffered Chemical Polish: Casey Preston and Chris Compton, 2000.
[2] Dewar Testing of Beta = 0.53 Half Wave Resonators at MSU TUPO056.