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

Laura Popielarski, Lindsay Dubbs, Kyle Elliott, Ian Malloch, Rania Oweiss, John Popielarski

Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824 USA

INTRODUCTION

FRIB requires a total of 341 certified SRF cavities
- Number of beta=0.53 HWR type cavities: **144**
- Weight cavity w/ helium vessel: **230 lbs** (104 kg)
- Internal surface area: **1.11 m²** & Internal volume:**13 gal** (49L)

Process Steps
1. Chemical BCP etch to remove damaged layer **100-150 µm**
2. Hydrogen degas heat treatment at **600 °C for 10 hours**
3. Light etch and high pressure rinse
4. Assembled for vertical testing

Original Acids	Recipe for BCP
Phosphoric Acid (85%)	2 parts Phosphoric Acid
Hydrofluoric Acid (49%)	1 part Hydrofluoric Acid
Nitric Acid (70%)	1 part Nitric Acid

- Acid temperature:**13-17 ° C**
- Acid flow rate is **8-10 gpm** (30-38 lpm)
- Cavities w/o helium vessels are cooled by wrapping with ice packs
- Cavities w/ helium vessels are cooled by cold water flowing through helium space

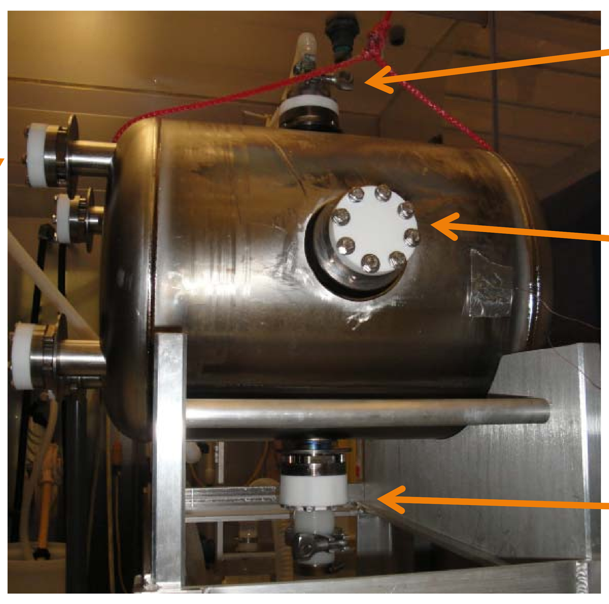

Figure 1. The HWR β = 0.530 Nb cavity without helium vessel shown with acid flowing in the bottom RF port and out the top RF port with cleaning ports and beam ports closed

CHEMICAL PROCESS DEVELOPMENT

First BCP Set-Up
- Predicted etch rate is determined by sample test
- Sample weight and thickness measured before and after etching
- The cavity is oriented horizontally as shown in **Figure 1**.
- Acid is pumped up through the bottom RF port and exits at the top RF port.
- Beam ports and cleaning ports are sealed with Teflon flanges.
- Nb in-situ sample is mounted on one of the beam port blanks
- Ultrasonic thickness measurements (USTM) taken on four preproduction HWRs before and after etching

The average of the 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.

- **Removal measured was 30% of removal estimated from predicted etch rate**
- **Maximum removal was 14x greater than minimum removal**

USTM at different locations indicated the etching was non-uniform **(Figure 2)**
- Removal near the inner conductor center was much higher than desired removal
- Removal along the beam port quadrants (sides) of the cavity was much higher
- Removal at the top and bottom (acid in/out) RF ports was much lower .

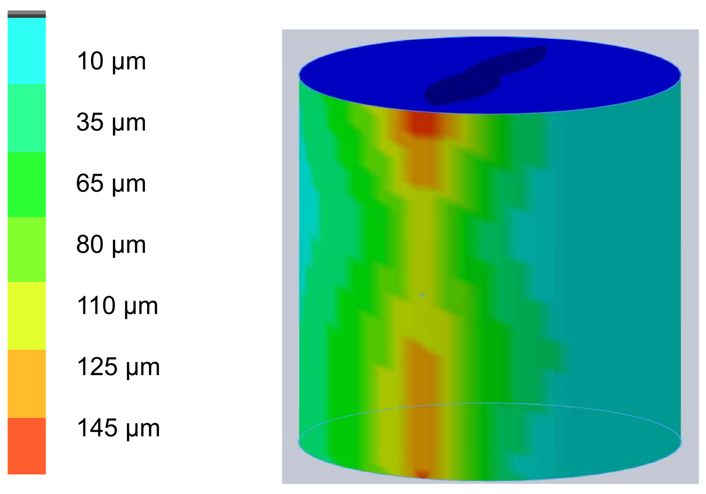

Figure 2. The maximum removal (shown in red) is 14 times greater than the minimum removal. Also, the majority of the cavity receives much less removal than the desired 100 microns.

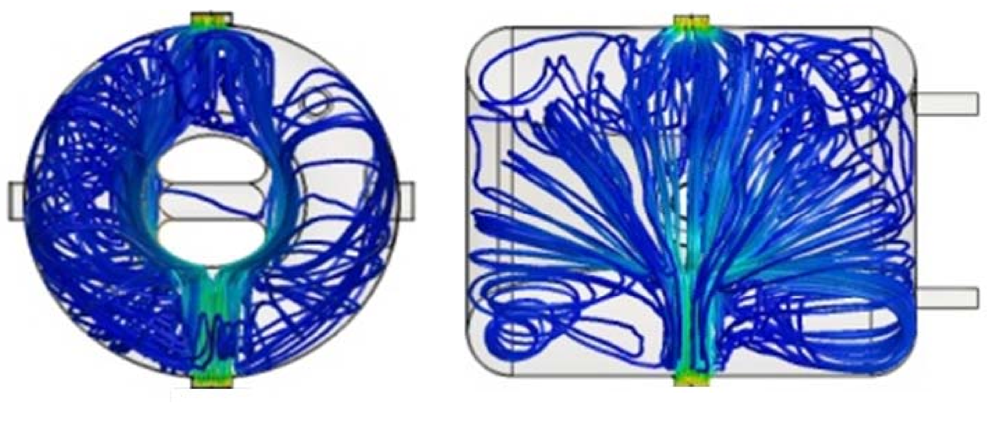

Figure 3. The SolidWorks flow simulation of the first BCP set up that shows low flow in areas of low etch removal and high flow corresponding to high etch removal

SolidWorks FlowXpress Analysis Wizard was used to simulate the BCP flow in the cavity. The flow model **(Figure 3)** indicates a relatively high velocity flow hits directly on the surface of the inner conductor. As expected USTMs at this location show a much higher etch rate than other locations on the cavity. 12 flow studies were completed to determine an etching configuration that would improve the average etch rate and uniformity of the cavity.

*Revised BCP Set-Up (**Figure 4**)*
- 4 PTFE dispensing quills 8.75" long and 0.9" diameter
- 0.375" hole on the side of the quill near the tip
- Quills protrude into cavity through the cleaning ports aligned such that they all face clockwise. This causes a swirling pattern as shown in **Figure 5**
- BCP exits the cavity through the top RF port
- Average etch rate increased to 46% of the PER
- 50% increase in etch rate over the first etching method

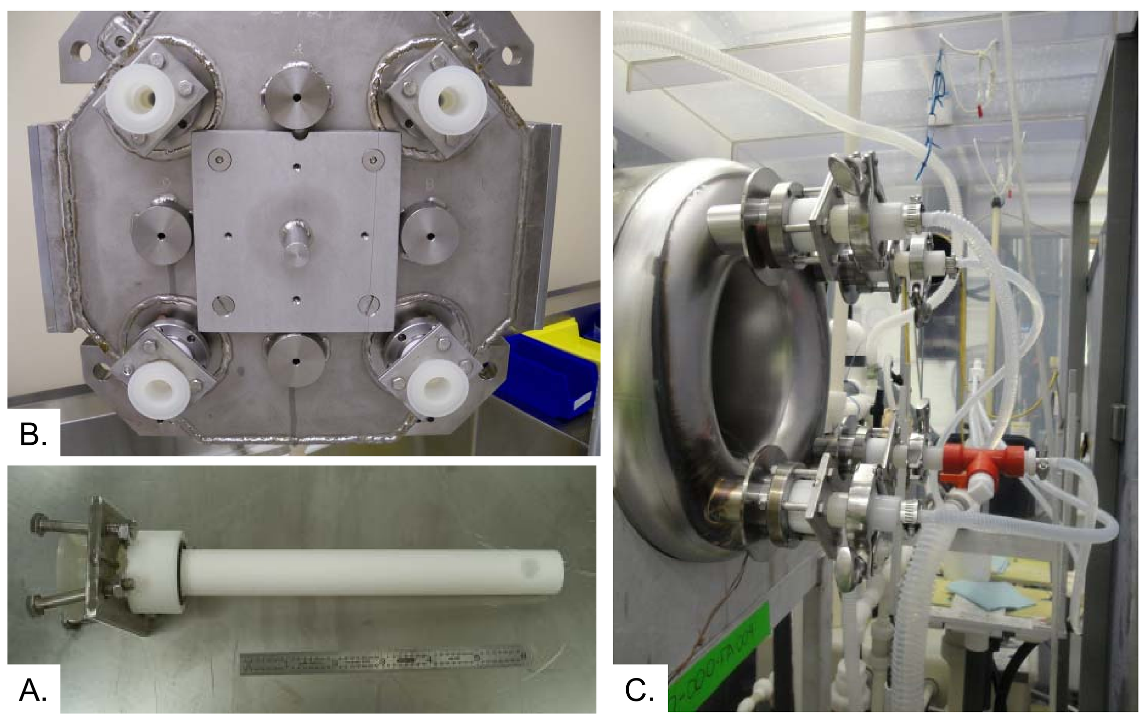

Figure 4. Revised BCP Set-up. A.) Etching quill. B.) 4 etching quills installed to the cleaning ports of a HWR cavity with helium vessel. C.) Cavity installed to chemistry tool with BCP going in through quills and exiting through top RF port. Drained through bottom RF port.

*USTM material removal with revised BCP setup **(Figure 5)***
- Etch rate at all points closer to the PER & increased uniformity
- Peak removal where BCP exits dispensing wand directly on conductor
- Maximum removal on HWR_004 1st etch is **3.5 times** greater than minimum removal

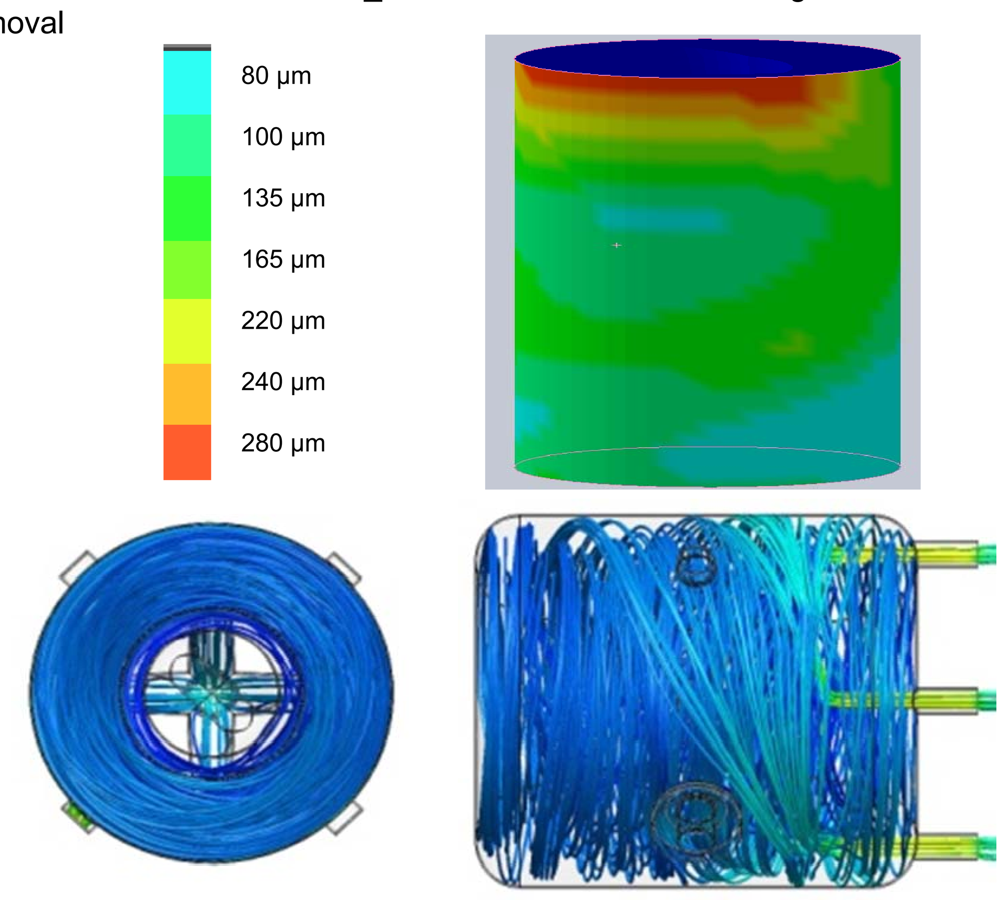

Figure 5. Etch removal and flow model of the revised BCP setup.

Future Plans
- Further optimize fixtures to reduce the peak removals (300 µm)
- Adjust angle that BCP exits the dispensing wand
- Reduce differential etching while still providing the desired swirling effect.

HEAT EXCHANGE & AVERAGE ETCH RATE: THERMODYNAMIC APPROACH

To remove heat generated during etching **10-13 °C** water flows through the helium vessel at a rate of **1-2 gpm**.

The heat exchanger has proven to be effective at maintaining cavity surface temperatures below **20 °C (Figure 6**).

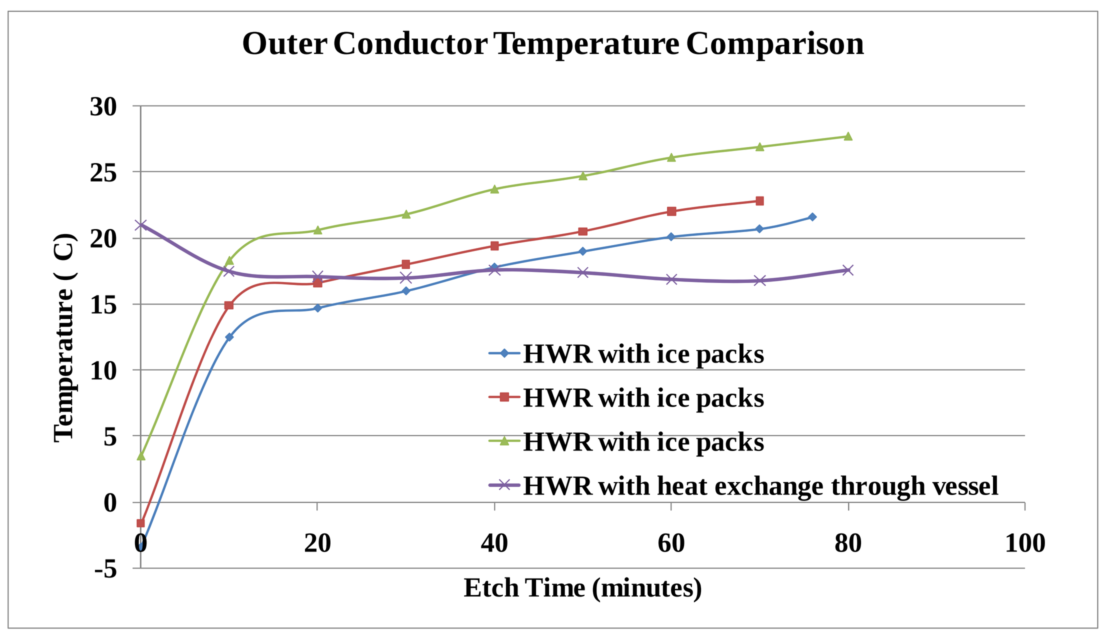

Figure 6. Plot of outer conductor surface temperature during etching

The heat exchanger allows us to measure the cooling water's flow rate and inlet to outlet temperature change. Thermocouples have been installed in the system to measure the temperature at the inlet and outlet of the cavity for BCP and heat exchanger. 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):

$$E.R = \frac{m_{H_2O}\, c_p\,(T_{out} - T_{in}) + \dot{m}_{BCP}\, c_p\,(T_{out} - T_{in})}{1183.8 \dfrac{W}{m^2 \dfrac{\mu m}{\min}} (S.A.)} \quad (1)$$

Integrating the average etch rate over a given time interval will give a total etch removal in µm. This method has been used 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
- Construct test structure to confirm amount of Nb removal calculated by model
- Develop a program to take temperature and flow measurements at regular intervals and convert them into cumulative removal

CAVITY VERTICAL TEST RESULTS

Based on the data set collected so far, there is little relation between the Nb concentration in the BCP solution and field emission onset or the maximum x-ray levels **(Figure 7 A-D).**

- 3 HWRs processed and tested
- 9 Total vertical tests
- Cumulative etch removal ranges **98 – 215 µm**
- Nb concentration ranges **0.1 - 35 grams per liter** in BCP at the start of etch

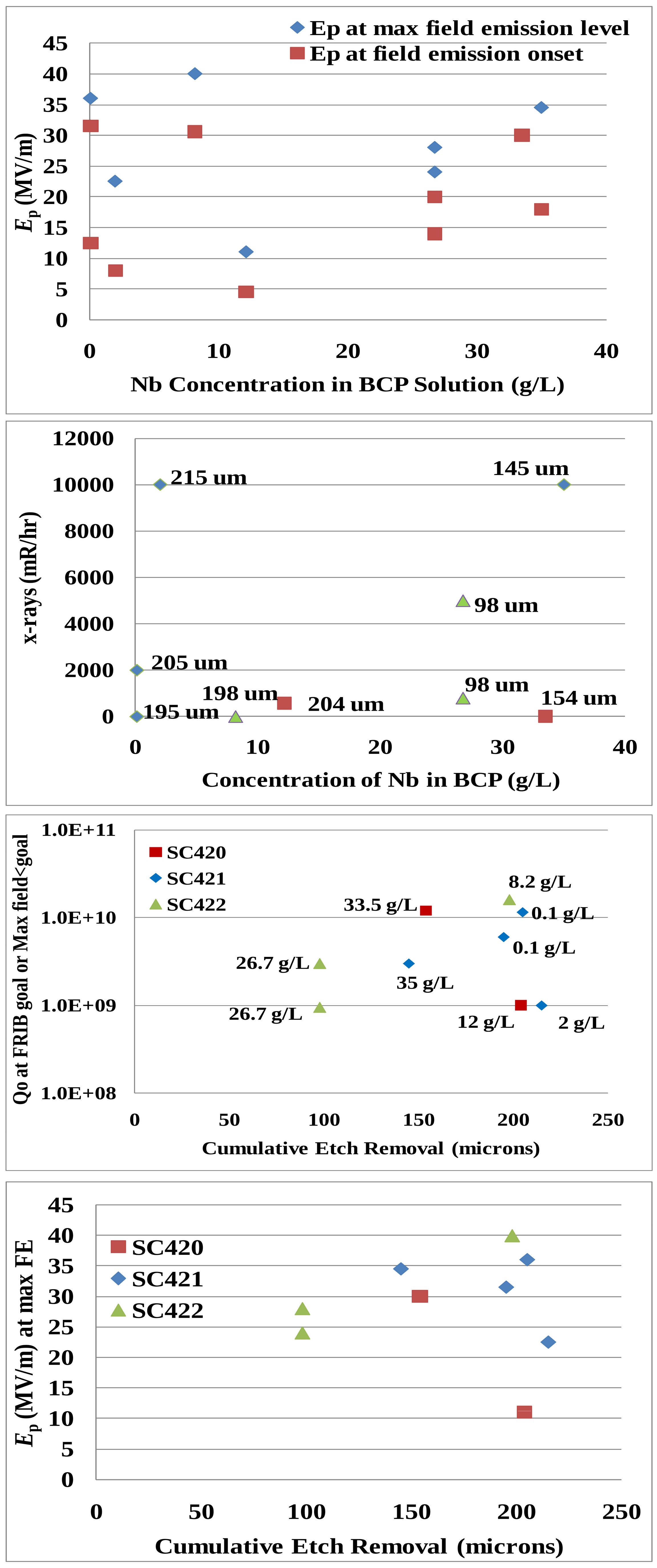

Figure 7. A-D For all of the HWR processes the total cumulative etch removal and the niobium concentration in solution at the start of the etch cycle is recorded. This data is compared with vertical test data such as quality factor (Q_0), peak surface electric field (E_p) at field emission onset (x-rays > 1mR/hr) and E_p at the maximum field emission level.

Additional information on the test data can be found in these proceedings [2]. 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.

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.

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

AKNOWLEDGEMENTS: 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.

FRIB Facility for Rare Isotope Beams
U.S. Department of Energy Office of Science
Michigan State University

This material is based upon work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661. Michigan State University designs and establishes FRIB as a DOE Office of Science National User Facility in support of the mission of the Office of Nuclear Physics.