SUMMARY OF THE FRIB ELECTROPOLISHING FACILITY DESIGN AND COMMISSIONING, CAVITY PROCESSING, AND CAVITY TEST RESULTS*

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Abstract

Recently, a new Electropolishing (EP) facility was constructed and commissioned at the Facility for Rare Isotope Beam (FRIB) with the purpose of supporting advanced surface processing techniques for SRF R&D activities. The FRIB production cavities opted for a Buffered Chemical Polish (BCP) method due to its cost effectiveness and was supported by successful outcomes in other facilities with low beta cavities in a similar frequency range. All 324 cavities used in FRIB Linac were processed in-house at MSU using BCP and exhibited satisfactory performance during testing. As part of the FRIB energy upgrade R&D, 5-cell 644 MHz elliptical resonators will be employed, desiring the use of EP and advanced techniques such as nitrogen doping and medium-T baking. The EP facility is designed to accommodate all types of cavities used in FRIB and possesses the capability for performing EP at low temperatures. Here we report the details of design and commissioning of the EP facility, highlights of encountered issues and subsequent improvements, and preliminary results from vertical tests conducted on the cavities.

INTRODUCTION

As the production run of cavities for the FRIB accelerator reached completion, work began on planning for the future. An energy upgrade utilizing 644 MHz elliptical style β =0.65 cavities was already being planned for the FRIB accelerator. R&D work of advanced cavity processing techniques such as nitrogen doping and medium temperature baking were also being considered. These projects require the ability to chemically process cavities by EP.

FRIB worked with an outside contractors to design and build a processing room and accompanying ventilation scrubber similar to the existing chemical etching facility described previously [1]. The processing room and scrubber provide a negative pressure area to house and operate the EP system while mitigating potential hazard exposure outside the EP facility.

With the processing room in place, work began on building out the EP process. Mechanical design work was split into four design packages:

• <u>EP Hood:</u> Fume hood used to contain process, including cavity, acid tanks, and plumbing while providing connections to scrubber ducting to ventilate area

- <u>EP Tool:</u> Included the end groups and support frame used for installing cavities to while providing interfaces to process plumbing, rotation mechanism, DC power supply, cooling water loop etc.
- <u>Cathode Installation and Rinse Area</u>: provided area for cathode and end group installation as well as rinse area to clean and neutralize cavities post-EP processing
- <u>Cooling Shower Enclosure:</u> provided means to connect to cooling water loop and contain spray inside EP hood

All design and fabrication work was performed/coordinated by the FRIB Mechanical Design Department. The primary driver for the design of the mechanical structure was to accommodate the largest planned cavities at the time shown in Table 1.

Table 1: Summary of Physical Cavity Processes FormingMechanical Design Basis

Characteristic	Value	Cavity Type
Length	129 cm	SNS β=0.80 six-cell elliptical
Diameter	84 cm	Jacketed FRIB β=0.65 five-cell elliptical
Weight (plus vol- ume full of acid)	320 kg	Jacketed FRIB β=0.65 five-cell elliptical

Installation was performed by FRIB processing personnel with close support from mechanical designers.

After mechanical infrastructure was installed, work on other sub-systems could take place:

- <u>Process plumbing:</u> storage tanks, pumps, valves, motors, and plumbing used for handling of chemicals, acidic rinse water, and external cavity cooling loop
- <u>Controls system:</u> developed for operator control of system, process and safety monitoring, process interlocks, etc.
- <u>DC Power Supply:</u> infrastructure for supplying direct current to cavities required to drive electropolishing reaction
- <u>Conventional Utilities:</u> connecting system to power, compressed air, dry nitrogen, ultra-pure water, and sanitary sewer

^{*}This material is based on work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661, the State of Michigan, and Michigan State University. †metzgar@frib.msu.edu

With all facility infrastructure in-place as seen in Fig. 1, work proceeded to testing and commissioning.



Figure 1: FRIB electropolishing facility with β =0.80 sixcell elliptical cavity connected to system in preparation for commissioning EP process.

COMMISSIONING

A SNS β =0.80 six-cell elliptical cavity generously provided by Oakridge National Laboratory was used for initial commissioning work. Extensive water testing was performed to verify process control worked as expected and that the system was leak tight. Electrolyte composed of a 10:1 mix of 98% sulfuric and 48% hydrofluoric acids was then transferred into the system and the first electropolishing process was performed on 16 December 2021.

Key processing parameters were met and are summarized in Figs. 2 and 3. Figure 2 shows current oscillations were present and electrolyte temperatures were maintained at acceptably low levels. This was done at a low flow rate (1 L/min) so as to not disrupt the electrolyte viscous layer critical for successful EP. The external cooling shower was able to maintain cavity wall temperature in an acceptable range (14-24 °C) during the process as shown in Fig. 3.

In addition to process quality metrics, safety requirements were met. The system was able to handle the strong acids comprising the electrolyte. No hazardous gases were detected at any point during the process confirming scrubber airflow system was well designed. The system was able to effectively neutralize the acidic rinse water generated by rinsing at the end of the process.

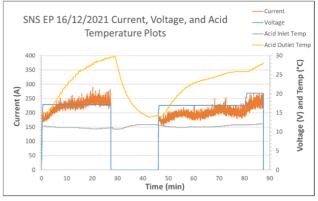


Figure 2: Current, voltage and acid temperature data from commissioning EP process the FRIB EP facility.

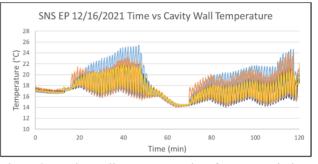


Figure 3: Cavity wall temperature data from commissioning EP process.

Once the cavity was removed from the system, average removal was determined via ultrasonic thickness gauge measurements. A total of 198 points were measured with average removal being 16.9 μ m. This was compared to the removal in μ m as calculated from the total current density in mA/cm² over the duration of the process in hours using Eq. (1).

$$d(\mu m) = 0.811 * j\left(\frac{mA}{cm^2}\right) * t(h)$$
(1)

This number was calculated to be 11.7 μ m which is in fairly close agreement with ultrasonic thickness gauge removal. With processing lasting approximately one hour, the removal rate of 12 μ m/hour is similar to removal rates for bulk processing seen in similar facilities.

One of the most important characteristics of EP is the quality of the surface finish. The SNS β =0.80 six-cell elliptical cavity was previously electropolished so could not be used for visual comparison. The next cavity processed in the facility was a FRIB β =0.53 half-wave resonator (HWR). A comparison of the cavity RF space borescope inspection can be seen below in Fig. 4.

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Figure 4: Comparison of FRIB β =0.53 HWRs chemically processed by chemical etching (a) and EP (b).

Figure 4a on the left shows a cavity that was processed exclusively by BCP chemical etching, for a total of 240 μ m of removal. The cavity in Fig. 4b on the right underwent 60 μ m of removal by BCP etching followed by 60 μ m of EP. The side-by-side result is clear; processing in the FRIB EP system resulted in a smoother, more mirror-like finish than processing exclusively by BCP chemical etching.

Cavity B was also weighed before and after the EP process so comparison between removal based on the measured current density and weight of niobium removed could be made. Average removal calculated via weight difference was 66.2 μ m while removal calculated from Eq. (1) was 62.8 μ m. This close agreement confirms reliability of measured current draw.

SYSTEM PROCESSING SUMMARY

Including the initial commissioning process, at total of 14 processes have been performed in the EP facility. Table 2 below shows a summary of these processes. Serial number with a "-9XX" indicate the cavity is a single cell.

Table 2: Summary of EP Process at the FRIB

#	Cavity	Description
1	SNS	Commissioning EP
2	S53-155	Commissioning HWR EP
3	S53-155	Bulk EP, 60 µm
4	S53-155	Commissioning cold EP
5	S65-905	Commissioning 1-cell EP
6	S65-905	Bulk EP 60 µm + 10 µm cold
7	S65-905	Bulk EP 60 μ m + 10 μ m cold
8	S53-159	Bulk EP, 60 µm
9	S53-159	Bulk EP, 60 µm
10	S53-155	Final EP, 20 µm
11	S53-159	Final EP, 20 µm
12	S53-155	Final EP reprocess, 20 µm
13	S65-904	Bulk EP, 40 µm
14	S65-901	Final EP, 5 µm for N-doping

Table 3 below lists key system parameters of the current electropolishing facility at the FRIB.

Table 3: Key Specifications of the FRIB EP System

Characteristic	Description
Power Supply	60 volts, 500 amps
Chiller Specifications	Two 12 kW chillers at 10 °C
Minimum Acid Tem- perature	9 °C
Acid Pump	1.0-7.5 liters/min
Cavity Rotation Speed	1 RPM (adjustable)

DESIGN OF ELECTROPOLISHING TOOL FOR B=0.53 HWR

Electropolishing of elliptical style cavities is well developed and in some sense more straightforward due to the single cathode installed through beam ports of the cavity. Cavities such as the FRIB β =0.53 HWRs have more complicated geometry so require a different approach. A summary of this cavity design can be found in [2]. A cavity installed in the FRIB electropolishing tool is shown in Fig. 5 below.



Figure 5: FRIB β =0.53 HWR cavity installed in elec tropolishing tool.

One fundamental difference with FRIB β =0.53 HWR EP tooling is that there are four cathodes installed to the cavity rather than a single cathode. One cathode is installed to each rinse port (Figure 6).

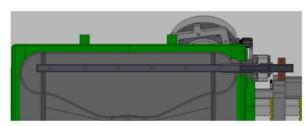


Figure 6: Section view of 3D model showing cathode installed to one rinse port of a FRIB β =0.53 HWR.

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EP removal is heavily dependent on the proximity of the cathode to the cavity surface. If only one cathode were to be installed to the cavity beam port, there would be very little removal around the short plates in particular, but also regions of the outer and inner conductors with no line-of-sight to the cathode. Using four cathodes distributes the cathode surface area through the cavity RF space.

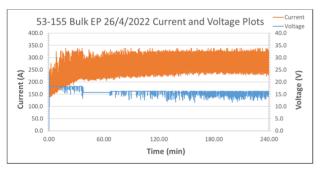
Another key difference with FRIB β =0.53 HWRs is that they have a helium jacket during EP processing. Spraying the helium jacket exterior would result in limited cooling of the niobium cavity surface. To directly cool the niobium surfaces, a cooling water loop is fed through the center of both end groups and connects to the helium fill and return ports. This provision adds complexity to the tooling design because an additional pair of rotary seals are required to accommodate the cooling loop plumbing which is fixed.

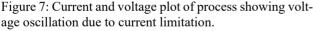
PROCESS IMPROVEMENTS

With any new process, the need for changes and improvements to the system can only be identified through experience. This section summarizes some of the issues encountered and subsequent improvements.

DC Power Supply Unit

Having a supply of direct-current power that is of sufficient capacity and stable is critical to the EP process. After successfully commissioning with a 50 volt, 330 amp unit, limitations manifested during later processes. The first issue is illustrated below in Fig. 7.





The larger surface area of a FRIB β =0.53 HWR compared to the SNS β =0.80 six-cell elliptical cavity exceed the current limitation of a single power supply. Current oscillations are a characteristic of the EP reaction due to the changing resistance of the system as the reaction progresses. The power supply unit is operated in constant-voltage control mode, but when current oscillations reached the 330 amp limit of the unit, voltage needed to drop to compensate resulting in voltage oscillations.

To remedy this issue, an identical power supply unit was placed in parallel bringing total capacity to 660 amps. This solution had its own issue as shown below in Fig. 8.

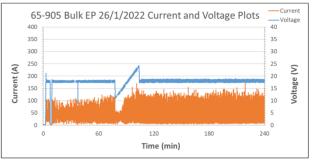


Figure 8: Current and voltage plot of process showing excessive current oscillation and voltage noise.

While current oscillations are expected, the amplitude of these oscillations should be around 20-30% of the average. The current oscillations during a bulk EP process of a β =0.65 single cell cavity were essentially 100%, going down to zero amps. After extensive troubleshooting, it was determined that this extreme current oscillation was caused by excessive noise of the measured current (and voltage) and output signal being fed to the secondary power supply unit. Rather than expending effort to reduce noise in the system, it was decided to change to a single power supply unit rated for 60 volts and 500 amps. As seen below in Fig. 9, the issues with oscillating voltage and extreme current oscillations are resolved.

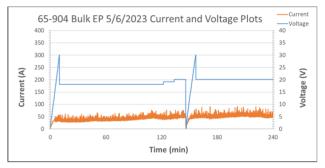


Figure 9: Current and voltage plot of process showing recorded data from current 60V, 500 A power supply unit.

IV Curve

IV curves are plots that are recorded to establish the "polishing regions" of a system. It is important that the electropolishing reaction occurs in the polishing region of an IV curve [3]. This region is identified at the voltage where current does not increase with increasing voltage. This is known as the "plateau region". Work by Chouhan et al [4] shows performing EP under the plateau region conditions leads to superior cavity surface finish. Many factors such as cavity surface area, acid temperature, and niobium concentration in electrolyte can affect where this plateau region occurs so it is critical to perform this IV curve test at steady state conditions for every process. The larger current unit provides the excess capacity to perform this test.

53 Rinse Port Surface Issues

After the first round of bulk EP on a FRIB beta=0.53 HWR, surface pitting was observed in multiple rinse ports. This can be seen below in Fig. 10.



Figure 10: Pitting from EP process of FRIB b=0.53 HWR.

The likely cause to this was the tight fit of the flanges used to insert the cathodes into the rinse ports. The cathodes were designed to be as large a diameter as possible in order to maintain a sufficient anode-to-cathode surface area ratio. The flanges used to interface with the cavity rinse port flanges included a guide that served the purpose of preventing scratching during cathode installation and to mask the port to prevent excessive removal due to close proximity to the cathode. This tight fit likely cause poor electrolyte draining so that upon mixing with rinse water, the excessive heat of mixing resulted in pitting. The cathode/flange assembly was altered by removing the guide, extending the weep hole in the cathode to improve draining, and adding a thin PFA plastic sleeve to maintain masking in the port. Subsequent EP processes have shown improved surface finish inside rinse ports.

VERTICAL TEST RESULT

According to Saito et al [5], the primary benefit of electropolishing, when paired with low-temperature bake (LTB), is a flat Q-slope at high field (HFQS). Figure 11 below shows a graph of the range of performance data for cavities processed solely by BCP chemical etching vs the cavity (S53-159) that was processed by EP and LTB.

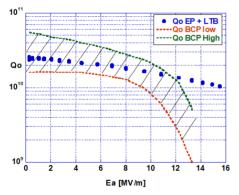


Figure 11: Comparison of the general range of performance data for cavities processed BCP etching with cavity processed by EP and LTB.

The flat HFQS shows clear improvement of the cavity performance. Ultimately, this is the most critical parameter to consider when validating a new process. These data show that the FRIB EP facility successfully improved cavity performance.

SUMMARY

A new electropolishing process facility has recently been designed and built at the FRIB. The facility was designed to be flexible to accommodate processing of many cavity types and is housed in a ventilated, dedicated processing room. The processing facility includes mechanical infrastructure to handle and contain the process components. Process components consist of a plumbing, pumps, valves, and motors all operated by a control system. A DC power supply drives the EP reaction and the facility is supported by other conventional utilities such as compressed air, sanitary sewer, and ultra-pure water.

Operational and water testing were executed using a SNS β =0.80 six-cell elliptical cavity. After testing completion, the first EP process was performed on this cavity on 16 December 2021. Since then, thirteen EP processes have been conducted. Of particular near-term importance is the development of tooling to electropolish FRIB β =0.53 HWR cavities. New end groups were designed to accommodate multiple cathodes and an interface with the helium jacket to facilitate direct cooling of the niobium cavity surface.

As more processes occur in the facility, issues and opportunities for improvement present themselves. One such significant improvement was upgrading the DC power supply unit so it met the needs of future process. Making sure the current supplied to the process was sufficient in quantity and stability will be critical to successful EP. Conducting IV curve test to ensure conditions are in the correct polishing region and altering flange design to facilitate draining and rinsing are other ways to improve cavity surface finish

With regard to future work, HWR cavity S53-159 showed minimal HFQS as a result of EP processing plus LTB. Cavities for a spare FRIB β =0.53 HWR will be processed in this manner. The EP facility will also be critical for future plans at the FRIB including energy upgrades utilizing 644 MHz elliptical cavities and R&D work involving advanced processing techniques incorporating nitrogen-doping and medium-temperature baking.

ACKNOWLEDGEMENTS

Many different groups assisted with development of the facility and the FRIB. Firstly, Thomas Reid, Ben Guilfoyle, and Mike Kelly at Argonne National Laboratory collaborated with FRIB personnel by giving a tour of their electropolishing facility and observing processes. Many different groups at FRIB were involved with the facility from Mechanical Design, Controls Engineering, Power Supply, Conventional Facilities and Infrastructure, and numerous subject matter experts. Lastly, John Mammosser and Matt Howell generously sent a spare SNS β =0.80 six-cell elliptical cavity to be used for the commissioning EP process.

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