

# NEW IMPROVED HORIZONTAL ELECTROPOLISHING SYSTEM FOR SRF CAVITIES\*

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## Abstract

The best performance of niobium SRF accelerating cavities is obtained with surfaces smoothed with electropolishing chemical finishing. Jefferson Lab has recently specified, procured, installed, and commissioned a new versatile production electropolishing (EP) tool. Experience with EP research and operations at JLab as well as vendor interactions and experience guided development of the system specification. Detailed design and fabrication was awarded by contract to Semiconductor Process Equipment Corporation (SPEC). The delivered system was integrated into the JLab chemroom infrastructure and commissioned in 2020. The new EP tool provides much improved heat exchange from the circulating H<sub>2</sub>SO<sub>4</sub>/HF electrolyte and also the cavity via variable temperature external cooling water flow, resulting in quite uniform cavity wall temperature control and thus improved removal uniformity. With the JLab infrastructure, stabilized process temperature as low as 5 °C is available. We describe the system and illustrate operational modes.

## BACKGROUND

Bulk Nb SRF cavities are almost always fabricated via sheet metal forming and machining techniques and integrated by electron beam welding. Because clean, crystallographic niobium is essential for best superconducting properties, of order 200 μm of surface material is typically chemically removed from the RF surface to remove any surface damage or fabrication contamination. To support the highest surface magnetic fields, surfaces additionally need to be smooth at the submicron level to avoid local field enhancements approaching critical field amplitudes.

The community has developed the H<sub>2</sub>SO<sub>4</sub>/HF (10:1 using US standard reagent HF) electrolyte based diffusion-limited electropolishing that is now employed generally for at least the final chemical finishing steps [1-4]. As experience has been gained and process refinements realized, the lessons learned are now being integrated into the specifications of new production processing tools. Jefferson Lab has recently completed this process and has commissioned a new production horizontal cavity electropolishing (EP) system. The system is now in regular routine use.

There are inherent limitations if one uses the acid solution as both the chemical agent and the process coolant. The best one can do to establish reproducible process conditions is to regulate the supply acid

temperature and flow rate and take pains to ensure symmetric outflow of acid from the cavity. One may then attempt to reduce smaller diameter surface heating by increasing the distance of these surfaces from the cathode by masking portions of the cathode, but this has the offsetting effects of increasing cathode current density (increasing sulfur precipitation rates) and increasing likelihood of etching roughing in beampipe appendages (HOM cans or coupler structures). For some structures this is doable when the resulting non-uniform removal still meets requirements. Changing process conditions or rates then involves changing the temperature of the supplied acid/coolant, the convenience of which depends strongly on the details of all of the infrastructure systems involved.

The most robust process control solution is to separate the chemical agent role from the process coolant role. This is accomplished via provision of external temperature-controlled water to essentially define the process temperature with flow adequate to remove any heat produced with negligible temperature rise in the cavity and near-surface solution. How well and flexibly this functions in reality depends on the available infrastructure systems.

## SYSTEM SPECIFICATION

As a part of the JLab TEDF Project 2012-13 [5], a flexible support infrastructure was set up to accommodate the evolution of process tooling. This infrastructure provides generous capacity of ultrapure water (UPW) and process cooling water at various temperatures. The project also included a clean cavity chemistry processing room which has been used to house the JLab cavity EP system. The EP system originally set up in the old facility and largely commissioned to support the ILC R&D activity 2008-2012 and then the CEBAF 12 GeV Upgrade was relocated here into the new facility in 2013 and recommissioned. As system components were aging and process improvement lessons had been learned, a specification for a new system was developed.

Several key features were sought from the new system:

- Locate the electrolyte sump below grade and in an adjoining service space for enhanced safety.
- Arrange cavity setup for improved ergonomics and reduced assembly labor.
- Improved external cavity cooling.
- Upgraded and serviceable PLC control with contemporary user interface.
- Increased capacity to handle larger cavities.

A functional specification was developed in 2015. When enabling funds were identified, a solicitation of corresponding proposals was posted in 2017. After refinement of the specification, a contract was awarded in

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December 2017 to Semiconductor Process Equipment Corporation (SPEC) to design and build the system.

The new EP system hardware was delivered to JLab in November 2018. Because of the necessary service interruption for removal of the old system and installation of the new system, installation start was delayed until December 2019. Installed acceptance testing began February 2020, just before the pandemic-induced facility shutdown. Machine acceptance commissioning resumed in July 2020 (Fig. 1).



Figure 1: New JLab cavity EP system.

## SYSTEM DESCRIPTION

The operational system includes the following characteristics:

- Remote acid sump with improved heat exchanger.
- Safer provisions for adding HF to the sump.
- Reduced labor requirement per cavity cycle.
- Improved safety and ergonomics.
- Robust PLC control.
- Cavity external cooling – uses UPW spray with available chilling. Spray from unobstructed above with sheeting flow over cavity surface.
- Power supply provides 0–20 V DC and up to 250 A.
- Seven wireless readout thermocouple sensors to monitor temperature at several cavity locations.

### Cooling System

The TEDF-provided chilled water (ACW) system circulates throughout the building. Its temperature is set in the Test Lab basement and can be set over the range  $-7.7$ – $4.5$  °C ( $18$ – $40$  °F). This glycol-water is available to flow through the acid sump heat exchanger and the cooling water heat exchanger. A SPEC-provided control loop allows setting a control setpoint for the sump temperature. The only regulation is via ACW cooling flow. There is no offsetting heat source other than ambient conditions and returned acid, if warmer than the sump setpoint.

The cavity cooling provisions of the SPEC system are very nice when coupled to the JLab UPW system. Because the UPW system has a large surge capacity and generous makeup and recirculation rates, its typical supply

temperature is a relatively stable  $20$  °C year round. This water supply is used for external cavity spray cooling via ten spray nozzles (Fig. 2). Although initially requested, there is no temperature control loop on the supplied cavity cooling water. There is a front-panel manually-adjusted flow valve and manual ball valve on the ACW available to the cavity cooling water heat exchanger. The temperature of the supplied water, then, depends on the UPW flow rate through the heat exchanger, the open/closed state of the ACW control valve, and the ACW temperature setpoint (common to the acid sump heat exchanger supply). There is a freeze protection interlock that enforces a bypass of the cooling water heat exchanger when the cooling water temperature is  $2.0$  °C or lower.



Figure 2: 5-cell CEBAF cavity installed for polishing.

### Acid Circulation System

The acid circulation capability of the SPEC system is generous. While the previous system was useful only up to about  $4.5$  lpm ( $1.2$  gpm), the present pump is capable of up to  $56.8$  lpm ( $15$  gpm), programmatically settable. While the pump may be good for this. The balance of loop flow restrictions determine how much of that is useful or beneficial. The basic acid flow path is distributed out of holes in the cathode, mixed by cavity rotation, typically  $1$  rpm, and outward flow both ends of the typical axially symmetric cavity. The acid return flow to the remote sump unit (Fig. 3) is by gravity return, nominally by overflow of a weir. Due to back pressure from flow restrictions, the acid level can rise. That, mixed with waves from cathode inlet orifices set the maximum useful flow as occurring when clear gas space above the acid liquid level is occluded. Then the  $N_2$  purge gas aggravates the instability and spurts of acid may get pushed into the  $N_2$  flow, hydrogen gas purge line. This maximum flow rate will vary with cavity geometry and setting of the variable height weirs. To obtain the same acid volumetric flow out both ends of the cavity requires that the effective weir levels match and any outboard plumbing flow restrictions are negligible.

### Controls

The system also provides for monitor and logging of many parameters, including seven thermocouples attached

to the cavity with wireless connection to the data acquisition system.



Figure 3: Remote electrolyte sump unit.

The controls interface allows input of the cavity surface area and then tracks the accumulated average Nb removal in  $\mu\text{m}$  via the integrated current during operation (five electrons per niobium atom).

## COMMISSIONING

In August 2020, four days of operation of the JLab horizontal EP system was run to exercise the various operating parameters, observe couplings between these parameters, and to identify convenient operating modes having desirable robustness for routine use.

For this test, the 952 MHz EIC-5 cavity was used. In comparison with most other processed cavities, this design has a larger clear beampipe diameter and larger cell diameter. The total surface area is  $0.972 \text{ m}^2$ .

The cathode used was a “typical” 3.3 cm diameter Al pipe with upward-pointing electrolyte-supplying holes with spacing that matches the cell pitch in 1.3 GHz cavities. For this test, the cathode was completely “unmasked” – no Teflon wrap within the cavity length. This naturally reduces the resistance between cathode and beampipe surfaces, increasing the polarization voltage experienced by the smaller diameter surfaces of the cavity (producing a thicker anodization layer). The maximized cathode surface area also decreases the local current density for a constant total current, lowering the polarization voltage at the cathode – reducing sulfur precipitation reactions and reserving a greater fraction of the applied voltage for cavity anodization potential [6].

To observe the system responses, several sets of combinations of acid sump temperature, acid flow rate, ACW temperature, spray cooling water flow, and spray cooling water temperature were exercised while all parameters were logged. The data from the two-days of operation were then analysed for patterns.

## General Observations

1. The supplied ACW temperature was well regulated, with  $\sim 1.2 \text{ }^\circ\text{C}$  variations around the setpoints used:  $4 \text{ }^\circ\text{C}$ ,  $-4 \text{ }^\circ\text{C}$ .
2. With the physical configuration used, acid flow rate of 28.4 lpm produced acid overflow into the  $\text{N}_2$ /hydrogen purge line. Consider 26.5 lpm (7.0 gpm) to be the maximum stable acid flowrate.
3. With no water cooling, 14 V applied, and 24.6 lpm (6.5 gpm) acid flow, the estimated real acid temperature rise was  $2.5 \text{ }^\circ\text{C}$ .
4. With no water cooling, 14 V, 24.6 lpm acid flow, the average cavity temperature was  $8 \text{ }^\circ\text{C}$  higher than the supplied acid temperature.
5. With no water cooling, the left beamtube was much hotter ( $8\text{--}9 \text{ }^\circ\text{C}$ ) than everywhere else on the cavity. This indicated significantly less flow out the left side of the cavity – due either to effectively higher weir level or increase flow restriction to gravity drain. [Subsequent follow-up releveling attention of the same cavity/cathode configuration reduced this temperature differential by at least 70%.]
6. The sump temperature does not regulate well above  $15 \text{ }^\circ\text{C}$ . Since ACW is used to cool the sump volume as a whole, and there is no forced mixing within the sump, there develops a significant temperature gradient within the sump which significantly complicates regulation of the acid supply temperature when the acid is also serving as the primary process coolant (no cavity cooling water in use).
7. With 11.4 lpm (3.0 gpm),  $\sim 10 \text{ }^\circ\text{C}$  acid, ACW @  $4 \text{ }^\circ\text{C}$ , 14 V, 11.7 lpm (3.1 gpm) water flow provided  $9.4 \text{ }^\circ\text{C}$  cooling water, resulting in  $\sim 12 \text{ }^\circ\text{C}$  cavity temperatures.
8. With 11.4 lpm,  $\sim 10 \text{ }^\circ\text{C}$  acid, ACW @  $4 \text{ }^\circ\text{C}$ , 14 V, 4.5 lpm water flow provided  $6.4 \text{ }^\circ\text{C}$  cooling water, resulting in  $\sim 12 \text{ }^\circ\text{C}$  cavity temperatures, but significantly larger temperature variation, especially warmer beampipes.
9. An exploration of I-V response with different chilled cooling water flow rates was run with ACW of  $4 \text{ }^\circ\text{C}$  and sump temperature of  $10 \text{ }^\circ\text{C}$ . This dataset allowed observation of the current dependence of the average cavity temperature. An essentially linear response is observed between  $8 \text{ }^\circ\text{C}$  and  $25 \text{ }^\circ\text{C}$ , with slope  $4.0 \text{ A}/^\circ\text{C}$ .
10. Always ensure that whenever there is acid in the cavity, the power supply circuit is either at the intended operating voltage or power supply “OFF.” This is not the same thing as power supply ON and 0.0 V. The cavity is vulnerable to gaseous hydrogen uptake into the bulk whenever exposed to HF unless fully anodized. Ensuring there is no current flow is the best way to arrest all  $\text{H}_2$  production. Low voltage operation generates hydrogen gas, but does not shield the cavity surface with an oxide barrier.

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### Limitations Noted

The power supply incorporated into the system can provide 0–20 V with up to 250 A. With a maximum advisable average current density of 25 mA/cm<sup>2</sup>, this would constrain the use of this power supply to cavities with surface area of 1 m<sup>2</sup> or less at temperatures yielding this current density.

The acid return flow is all gravity feed back to the sump. The electrolyte has significant, temperature dependent viscosity and some of the return piping size is not overly generous, so that fluid tends to not back up and not allow liquid level regulation with acid flow rates above ~27 lpm, even though the specified pump capacity is about double that.

Spray cooling water flow was best in the range of 11–15 lpm for optimum ACW heat exchanger operation. This seems adequate for all anticipated cavity structures that will fit in the system.

While some cavity electropolishing applications can tolerate significantly different removal rates between smaller and larger radius locations, the JLab system prioritizes material removal uniformity via active temperature control via flood cooling. This greatly simplifies the acid temperature control loop and also creates an upper bound on process temperature (~21 °C), and thus removal rate and cavity throughput rate for bulk removal operations.

### Plateau Curve

As explained elsewhere, the desirable polishing condition is to realize local diffusion-limited reaction rate at all cavity surfaces, thus avoiding any crystallographic etching [1-4]. Such a condition is evidenced by a “plateau” in the system I-V response when temperatures are stabilized. This was illustrated during this test cavity, showing stable current density of 7.0 and 10.7 mA/cm<sup>2</sup> with cavity temperatures at 5 °C and 15 °C, respectively (Fig. 4).

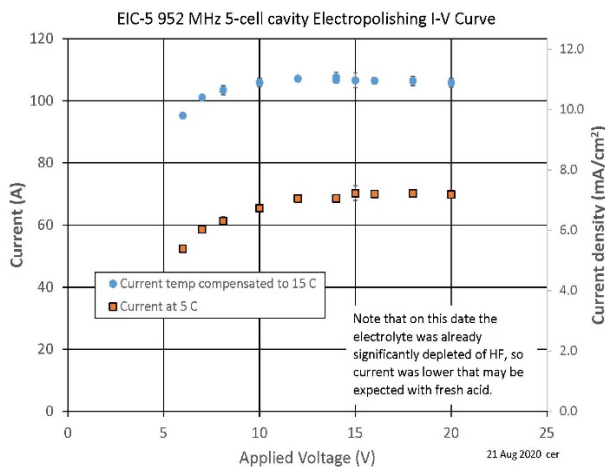


Figure 4: Illustration of I-V “plateau” condition for optimum surface polishing at two different temperatures.

## CAPABILITIES

One of the objectives of the commissioning tests was to converge on a set of operating conditions which appear to be robust and conveniently reproducible for a variety of cavity types that the operations staff may be asked to polish. There is a preference for lower temperatures for the final surface removal when maximum levelling is sought to support maximum surface magnetic fields. This however, significantly slows the process. A convenient solution was found with this system that provides uniform removal at 20 °C for time efficiency, followed by a quick and easy transition to 6 °C polishing effected by only opening the single ACW supply valve on the cooling water heat exchanger. This slows removal rate by about 40% for the final few micron removal. This implementation is now in routine use.

## CONCLUSION

A new system for electropolishing niobium accelerator cavities has been developed, installed, and commissioned at Jefferson Lab. Its design takes advantage of the community’s lessons learned and is now providing reliable well-controlled processing conditions for several construction and research projects.

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