

## DEVELOPMENT AND SCALE-UP OF AN HF FREE ELECTROPOLISHING PROCESS IN SINGLE-CELL NIOBIUM SRF CAVITIES\*

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### Abstract

The performance of niobium SRF cavities is strongly dependent on a microscopically smooth and clean surface, achieved using buffered chemical polishing or electropolishing, which require a viscous electrolyte containing hydrofluoric acid to achieve niobium oxide breakdown and current distribution control. An ideal polishing process would include: electrolyte free of hydrofluoric acid; control of surface roughness to less than 0.1 micron; surface free from contamination; current distribution control enabling uniform polishing; removal of at least 100 microns. Additional benefits from the use of a low viscosity could enable cavity electropolishing protocol more compatible with industrial scale processes. Faraday and Fermilab are collaborating to develop and scale-up the FARADAYIC Electropolishing process to validate its performance for single-cell cavity electropolishing. FARADAYIC Electropolishing combines pulse reverse electric fields and low viscosity aqueous electrolytes to control current distribution and oxide formation during metal removal. Recent results on coupon polishing will be presented including polishing rates up to 1 micron/min, control of electrolyte temperature to below 20°C, and surface finishes less than 0.2 microns over 4 mm length scales. Construction of a single-cell cavity electropolishing apparatus at Faraday is discussed.

### INTRODUCTION

This technology addresses the need for eco-friendly polishing technologies for niobium cavities, that are not dependent on the use of hydrofluoric acid. This program is in response to a request for proposal from Fermi National Accelerator Laboratory (Fermilab) seeking vendors capable of performing fluorine-free bulk material removal and surface polishing of niobium superconducting radio frequency (SRF) accelerating cavities. The SRF cavities may be used as part of a proposed Project X facility to be sited at Fermilab and for the International Linear Collider R&D program. The following specifications are minimum requirements for the process:

- Electrolyte free of hydrofluoric acid
- Control of surface roughness to a microscale finish,  $R_a \leq 0.2 \mu\text{m}$
- Surface free from contamination after polishing
- Current distribution control that enables uniform polishing across the entire cavity surface

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- Controlled removal of up to 150  $\mu\text{m}$

### TECHNICAL APPROACH

Faraday has achieved a technical breakthrough with the demonstration of the feasibility of the FARADAYIC Electropolishing process,[1,2] which employs sophisticated electric fields to control oxide film formation during metal removal, to achieve Nb polishing without hydrofluoric acid. The process is generally applicable to electropolishing of other strongly passive materials, such as titanium and nickel-titanium shape memory alloys.[3] FARADAYIC Electropolishing differs from conventional electropolishing through the use of a user defined, asymmetric waveform (in comparison to a constant voltage/current) permitting benefits that include:

- Control of oxide film formation,
- Improved polishing uniformity by producing a more homogenous current distribution through the control of process parameters,
- Increased metal removal rates, if desired, and
- The use of low-acid or neutral, aqueous electrolytes.

Based on the feasibility of electropolishing Nb coupons, this activity is directed towards validating the process for single-cell SRF cavities using existing apparatus developed at Argonne National Laboratory.[4] While this activity is directed towards an eco-friendly process, the use of low viscosity electrolytes could offer added benefits. For example, as recently reported by researchers at Fermilab,[5] non-uniform agitation and temperature effects associated with the current process disrupts the viscous boundary layer and facilitates fluoride anion access to the niobium cavity surface and promotes etching. We presume that the non-viscous electrolyte used in the FARADAYIC ElectroPolishing process would be less susceptible to these process instabilities and could lead to a robust industrial scale process.

A generalized pulse reverse electrolysis waveform is shown in Figure 1, and consists of an anodic peak current density,  $i_a$ , and associated on time,  $t_a$ , a cathodic peak current density,  $i_c$ , and associated on time,  $t_c$ , and an off time,  $t_0$ . (The sum of the on times and off time is the period of the waveform. The anodic,  $D_a$ , and cathodic,  $D_c$ , duty cycles are the ratios of the respective on times to the period. In contrast to conventional DC processes where the control variable is either constant voltage or current, the FARADAYIC Electropolishing process introduces several key process parameters that are user defined: the peak anodic current density, anodic on time, and duty cycle. The proper selection of these variables is critical

for the successful implementation of the process due to the strong influences that the variables have on mass transport rates, current distribution and metal removal rates. Specific to this application, the FARADAYIC Electropolishing process eliminates the need for hydrofluoric acid and uses a relatively low concentration sulfuric acid electrolyte with low viscosity. Although the mechanism for the FARADAYIC ElectroPolishing is unclear, the mechanism is distinct from the current electropolishing process based on a compact oxide (“salt”) film with mass transport limited access by fluoride anion.[6]

Within the boundaries of the generic pulse/pulse reverse waveform, a number of trade-offs, in addition to the  $R_a < 0.2 \mu\text{m}$ , are currently being explored. These include 1) off-times to reduce the average electropolishing current to avoid excessive heat build-up, 2) limit material removal to less than  $200 \mu\text{m}$ , and 3) rectification equipment considerations. Regarding rectification power levels for electropolishing single-cell as well as nine-cell cavities, the fastest timed event (i.e. off-time, anodic on-time, or cathodic on-time) must be greater than or equal to 2.5 msec. A pulse/pulse reverse rectifier able to achieve the power levels required for single-cell/nine-cell electropolishing is currently being developed and is scheduled to be available by the end of 2011.[7]

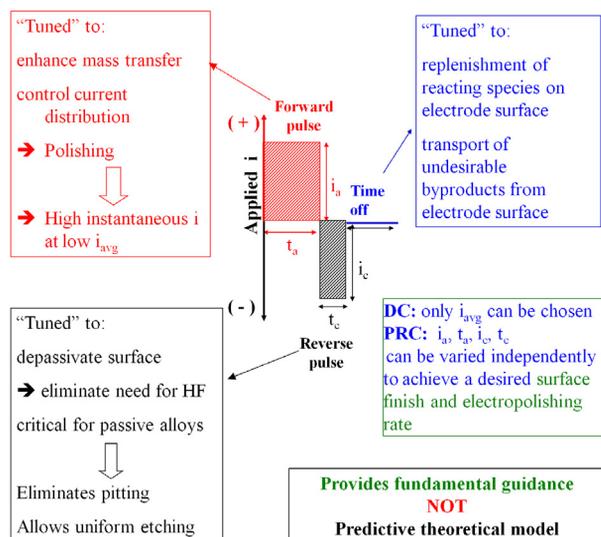


Figure 1: Generic FARADAYIC ElectroPolishing waveform.

## COUPON STUDIES

While the single cavity apparatus is being constructed, Faraday is working on development of the process using 1 inch square and 3 inch square Nb coupons. The coupons are electropolished using the FARADAYIC process in a simple electrolyte of 15 to 50 wt%  $\text{H}_2\text{SO}_4$ . The coupons are polished in a bench-scale flow cell (Figure 2) that

enables electrolyte circulation at low rates throughout the cell and across the surface of the coupon.



Figure 2: Bench-scale apparatus for electropolishing niobium coupons for process development activities.

Electrolyte temperature control is achieved through the insertion of long forward off-times (between anodic and cathodic voltage pulses) in the waveform, in conjunction with cooling coils in the cell, that enables chiller to mitigate heat build up from the polishing process. In these coupon studies, the temperature in the bath has been controlled to as low as  $13^\circ\text{C}$  over several hours of electropolishing, and is typically on the order of  $15\text{-}17^\circ\text{C}$ .

To date, we have investigated anodic voltages ranging from 4 to 30V, cathodic voltages ranging from 4 to 40V, anodic on-times of 0.060 ms to 2.5 ms, cathodic on-times of 0.10 ms to 2.5 ms and greater, and off-times of 0.5 ms to 2.5 ms and greater.

ElectroPolishing rates of up to  $5 \mu\text{m}/\text{min}$  have been achieved to replace BCP ( $10 \mu\text{m}/\text{min}$ ), and  $< 0.5 \mu\text{m}/\text{min}$  have been achieved to replace EP ( $0.5 \mu\text{m}/\text{min}$ ). Polishing depths have ranged from a few microns to over 400 microns. We have achieved  $R_a$ 's as low as  $0.18 \mu\text{m}$ , measured using a stylus profilometer over a 4 mm distance. In addition, coupons were provided for AFM analysis and average  $R_s$  for scan areas of  $50 \times 50 \mu\text{m}$ ,  $10 \times 10 \mu\text{m}$  and  $2 \times 2 \mu\text{m}$  were 3.3 nm, 1.33 nm, and 0.36 nm, respectively.[8]

## SINGLE CELL CAVITY ELECTROPOLISHING APPARATUS DESIGN AND BUILD

Figure 3 shows the apparatus that is being built for single-cell cavity electropolishing, per the design specifications of Argonne National Laboratory/Fermilab.<sup>4</sup> This apparatus will be capable of accommodating electropolishing of nine-cell cavities, although that is not the focus of the current program. Figure 4 shows the exterior of the Class 1000 cleanroom built at Faraday to house the apparatus and polishing and cleaning activities.

Figure 5 shows the general layout within the cleanroom facility. The cleanroom is split into two rooms: one for electropolishing and the other for cleaning, drying and packaging of the single-cell cavities for shipment to Fermilab for evaluation. We anticipate electropolishing experiments of single-cell cavities to commence in the Fall of 2011.

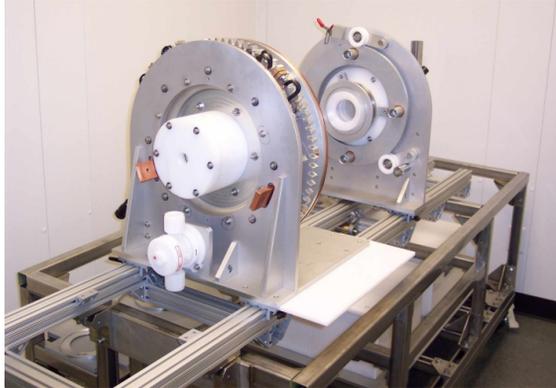


Figure 3: Apparatus for electropolishing single-cell cavities under construction at Faraday. The design is based upon the facility currently in use at Fermilab.



Figure 4: Cleanroom facility at Faraday Technology, Inc.

### SUMMARY

A novel, eco-friendly electropolishing technology for niobium is being scaled up for validation at the single cavity level, with the potential to expand to nine-cell cavities. This technology utilizes a pulse reverse electric field to control oxide film formation and current distribution, enabling uniform electropolishing to a very low Ra, in a low viscosity aqueous sulphuric acid electrolyte that does not contain hydrofluoric acid.

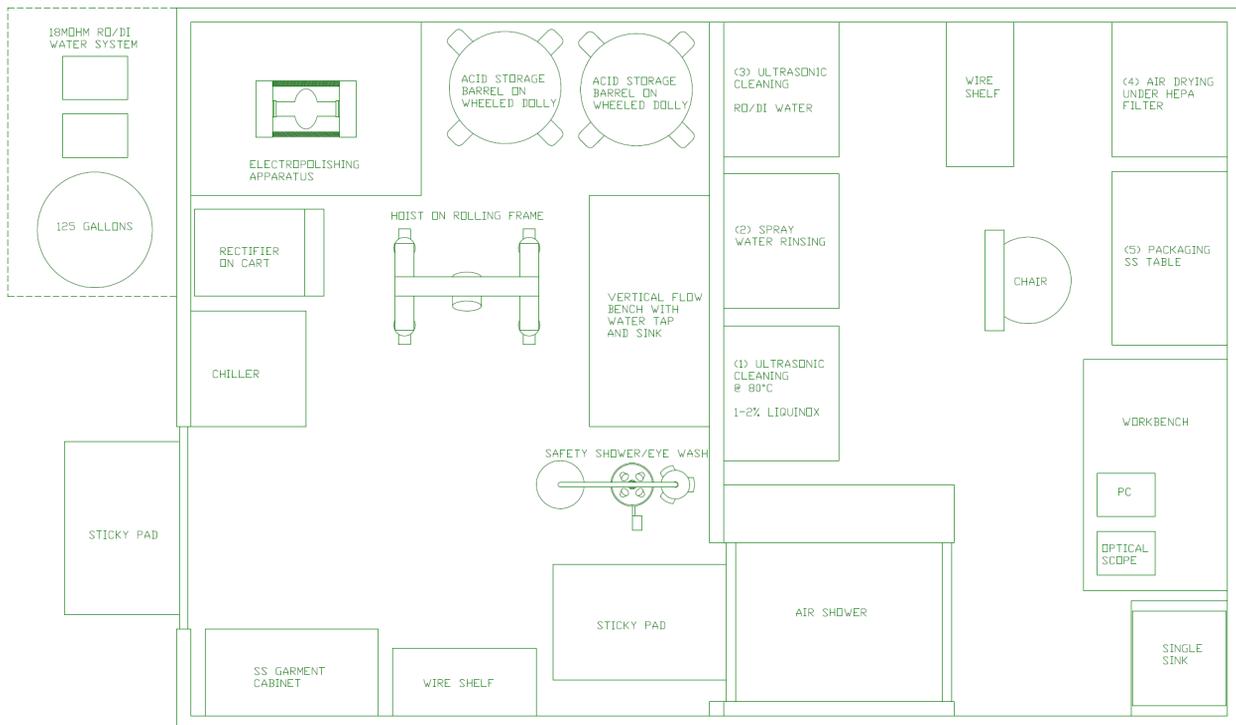


Figure 5: Layout of the cleanroom facility at Faraday Technology, Inc.

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