ECONOMICS OF ELECTROPOLISHING NIOBIUM SRF CAVITIES IN **ECO-FRIENDLY AOUEOUS ELECTROLYTES WITHOUT HYDROFLUORIC ACID***

E.J. Taylor, M. Inman, T. Hall, S. Snyder, Faraday Technology, Inc., Englewood, OH 45315, USA A. Rowe, Fermilab, Batavia, IL 60510, USA D. Holmes, Advanced Energy Systems, Inc., USA

Abstract

A major challenge for industrialization of SRF cavity fabrication and processing is developing a supply chain to meet the high production demands of the ILC prior to establishment of a long term market need. Conventional SRF cavity electropolishing is based on hydrofluoricsulphuric acid mixtures. In comparison, FARADAYIC® Bipolar EP applies pulse reverse electrolysis in dilute sulphuric acid-water solutions without hydrofluoric acid and offers substantial savings in operating and capital costs. Based on an economic analysis conducted with AES on the cavity processing requirements associated with the ILC, we project the cost of FARADAYIC® Bipolar EP to be about 27% that of the Baseline EP. In terms of tangible cost savings, the cost per cavity for the FARADAYIC[®] Bipolar EP and Baseline EP are \$1,293 and \$4,828, respectively. A major operating cost saving for Bipolar EP is associated with low sulphuric acid concentration and the absence of hydrofluoric acid. A major capital cost saving for Bipolar EP is associated with vertically oriented stationary cavity. Additional intangible cost savings are expected in terms of worker safety as well as less material degradation and maintenance requirements. Continued development and validation of FARADAYIC[®] Bipolar EP on lengths equivalent to nine cell cavities will contribute greatly to the industrialization of SRF accelerator technology.

INTRODUCTION

Electropolishing (EP) is used for final surface finishing of niobium SRF cavities to achieve high accelerating gradients and quality factors. Conventional EP for SRF cavities is based on anodic electrolytic dissolution under the influence of direct current (DC) electric fields in an electrolyte consisting of a mixture of sulphuric acid (95-98%) and hydrofluoric acid (49%) in a 9:1 volume ratio [1]. The presence of hydrofluoric acid in the conventional EP process presents considerable challenges in terms of worker safety in terms of compliance with Occupational Safety and Health Administration (OSHA) regulations and environmental considerations in terms of compliance with Environmental Protection Agency (EPA) regulations. Cavity processing facilities world-wide are expected to have similar concerns [2]. Hydrofluoric acid is used industrially for chemical milling or through mask etching and cases of serious worker injury and even fatality associated with hydrofluoric acid accidents have been documented [3]. The use of hydrofluoric acid significantly impacts the cost of cavity processing and is an important consideration in terms of "industrialization" of SRF cavity processing.

With funding from the Department of Energy (DOE) Small Business Innovation Research (SBIR) program, Faraday received Phase I funding to demonstrate the feasibility of electropolishing niobium coupons using pulse reverse current (PRC), in contrast to DC, to enable use of eco-friendly electrolytes. The pulse reverse current approach was based on previous successes in enabling simple electrolytes for surface finishing of a wide variety of materials and components [4-7].



Figure 1: Schematic representation of electropolishing of SRF cavity using pulse reverse current in an aqueous electrolyte without hydrofluoric acid.

During this feasibility study, we successfully demonstrated the ability to electropolish niobium coupons in 5 to 10 wt% aqueous sulphuric acid electrolyte using PRC. The electrolyte did not contain hydrofluoric acid and the surface roughness measured at Thomas Jefferson National Accelerator Facility (TJNAF) were equivalent to 20 that obtained using the conventional sulphuric acid/hydrofluoric acid electrolyte with DC (0.5 to 3 nm) ght

respective authors

by the 1

and

^{*}Work supported by DOE Grant Nos. DE-SC0011235 and DE-SC0011342, and DOE Purchase Order No. 594128. # jenningstaylor@faradaytechnology.com

[8]. Although the Phase II SBIR follow-on funding was not awarded, Faraday received funding from the American Recovery and Reinvestment Act (ARRA) through Fermi National Accelerator Laboratory (FNAL) to transition the PRC niobium coupon electropolishing process to 1.3 GHz single cell cavities [9].

During the transition from 3"X3" Nb coupons to single cell Nb cavities, we observed a current transient in the anodic current that we attributed to a transition from oxide film formation/growth to oxygen evolution. We speculated that our polishing mechanism occurred by removal of the niobium oxide during the cathodic pulse, and termed this process "cathodic electropolishing" [10]. We observed that the presence of the anodic current transition is important to effectively electropolish niobium. To adapt PRC EP to single-cell cavities, we adjusted the timing of the waveforms to provide sufficient time for the anodic current transition to occur. We applied the process to the final electropolishing step for singlecell SRF cavities provided by Fermilab, and Fermilab testing demonstrated RF performance characteristics very similar to those achieved using conventional hydrofluoric acid-based processing. "Bipolar EP", a descriptive term suggested by FNAL, achieved accelerating gradients above 40 MV/m and O_0 above 1E+10 at 35 MV/m using an electrolyte "no more hazardous or ecologically unfriendly than a household cleaner" [11,12].

In addition to the worker and environmental concerns, another barrier to "industrialization" associated with the conventional sulphuric/hydrofluoric process is associated with the capital cost of the EP tool. Due to the high viscosity of the conventional EP solution and the presence of elliptical regions within the SRF cavities, conventional EP is conducted with the cavity oriented horizontally and partially filled with electrolyte while rotating in order to avoid streaking due to bubble flow across the elliptical regions of the cavity [13,14]. Since the Bipolar EP process enables the use of low viscosity aqueous electrolytes, we were able to orient the cavities vertically and completely filled with electrolyte without the need for rotation (Figure 1) [15]. Due to the attributes of Bipolar EP, we envision a simple cavity processing tool capable of processing multiple cavities using electrolyte manifolds and electrical bus bars (Figure 2).

With additional DOE SBIR funding and a Purchase Order from Oak Ridge National Laboratory (ORNL), we are extending the Bipolar EP process to nine-cell Nb SRF 1.3 GHz cavities and three-cell Nb SRF 805MHz cavities, respectively [16]. In this paper we present a first order cost comparison of conventional (Baseline) EP and FARADAYIC[®] Bipolar EP.

ECONOMIC COST ESTIMATE OF NIOBIUM CAVITY EP

For the cost comparison the Baseline EP and FARADAYIC[®] Bipolar EP processes, we used the cavity processing experience of Advanced Energy Systems, Inc. (AES) [17]. In order to meet the U.S. six year requirement

of 3,600 cavities for the ILC, AES estimatess that 3,827 cavities will need to be processed. Based on processing 3,827 cavities, AES estimates that five (5) Baseline EP systems are required to meet the ILC requirement. We used this as the basis for the cost estimate associated with the niobium SRF cavity Baseline EP and Bipolar EP processes.



Figure 2: Concept drawing of tool for processing multiple cavities.

The primary differences between the conventional niobium cavity Baseline EP and the Bipolar EP are presented in Table 1. The differences between the Baseline EP and Bipolar EP processes which impact capital, operating and intangible costs are:

- o Electrolyte: operating, capital, intangible
- Cavity Orientation: operating, capital
- Cavity Rotation: operating, capital
- Material Removal Rate: operating, capital
 - Table 1: Comparison of Baseline and Bipolar EP

	Baseline EP	Bipolar EP
Electrolyte	9:1 H ₂ SO ₄ :HF	~5% (wt)
		H ₂ SO ₄ in H ₂ O
Processing	DC: ~17 V	Pulse Reverse:
Voltage		~3V/9V
Processing	25°C	25°C
Temperature		
Cavity	Horizontal	Vertical
Orientation		
Electrolyte	60%	100%
Volume Fill		
Electrolyte	~8 L/min	~8 L/min
Flow Rate		
Cavity Rotation	1 rpm	No Rotation
Cathode	Aluminum/Tube	Mixed-Metal
Material/Shape		Oxide Coated
		Titanium/Rod
Material	0.3 µm/min	0.15 µm/min
Removal Rate		

For our cost comparison and analysis, we have used the conservative material removal rate obtained during SRF single-cell cavity processing during the DOE SBIR Phase I program. The cost comparison is presented below in terms of 1) operating costs for consumable acids and acid waste disposal, 2) operating costs for labor, and 3) capital costs for EP tool, facilities and infrastructure. These

and

estimates are then used to estimate the cost to meet the cavity requirements for the ILC. Finally, a qualitative discussion of the intangible costs is presented.

In Table 2 we present the acid consumption and associated waste disposal costs. AES estimates the unburdened acid costs including waste disposal for the Baseline EP process at \$2,445. Based on an electrolyte of 5% H_2SO_4 in water for the Bipolar EP process, we estimate the acid purchase and waste disposal at 10% of the AES cost, or \$245. These costs are then burdened at 20% and multiplied by the number of cavities required for the ILC.

Table 2: Comparison of EP Acid Costs

	Baseline EP	Bipolar EP
Unburdened	\$2,445	\$245
Burdened (20%)	<u>\$2,934</u>	<u>\$294</u>
ILC (3,827)	\$11,228,418	\$1,125,138

In Table 3 we present the labor hours (20% burden) and costs associated cavity processing based on AES estimates for the Baseline EP process and estimates for the simpler Bipolar EP process. Note the labor estimates do not include time for support staff or management. The major area where we estimate less labor hours for the Bipolar EP process include acid handling, cavity set-up and installation, bulk EP and removal from EP cabinet and rinse. The rationales for the decreased labor hours for these steps are the worker safe electrolyte and the simpler tool (no rotation/vertical orientation) associated with the Bipolar EP process.

Table 3: Comparison of Labor (hour) Requirement

	Baseline	Bipolar
Acid Handling	2.0	0.5
Inspection	1.0	1.0
Surface Clean	0.5	0.5
Ultrasonic Cleaning	0.5	0.5
Weight/Thickness	0.5	0.5
Set-up/Installation	1.0	0.25
Bulk EP	4.0	1.0
Removal and Rinse	1.0	0.25
Inspect/Clean/Weight	2.0	2.0
Packaging	1.0	1.0
Documentation	<u>2.0</u>	<u>2.0</u>
TOTAL Hours	15.5	9.5
\$50/hr (Burdened)	<u>\$775</u>	<u>\$475</u>
ILC (3,827)	\$2,965,925	\$1,817,825

Capital cost estimates for the Baseline EP tool and infrastructure are derived from the actual costs incurred by AES for the current Baseline EP facility. These costs are presented in Table 4. The total unburdened costs associated with one Baseline EP tool, including facilities and infrastructure, is \$1,060,389.

As noted above, to meet the ILC cavity requirement AES determined they would require five (5) EP processing tools. Consequently, the items from the single Baseline EP tool/facility cost estimate were multiplied by five (5) after assuming a discount for elimination of nonrecurring design charges or a quantity discount. The discounts were based on our "best guesses" based on similar costs. A similar approach was used to estimate capital elements for Bipolar EP as appropriate. The major cost differences associated with the Bipolar EP were associated with the tool cost, negative pressure processing room and scrubber and exhaust system. Recall that we conservatively estimate the material removal rate for the Bipolar EP to be approximately 50% that of the conventional Baseline EP process. While we demonstrated faster material removal rates on coupons and are confident we can demonstrate faster removal rates in the Phase II program, we use the 50% material removal rate for this first order estimate.

Table 4: Comparison of Tool/Facilities Costs

	Baseline EP
EP Tool (Design, Fabrication,	\$758,312
Installation)	
EP Tool Shipping	\$18,828
Negative Pressure Room	\$132,000
Vapor Scrubber/Exhaust	\$52,349
Facility Modifications:	
10 Ton HVAC	\$19,500
Electrical Service	\$20,150
Electrical Distribution	\$38,900
Plumbing (Waste, Shower, Water,	\$12,750
Chillers)	
Fire Sprinklers	<u>\$7,600</u>
Total Cost (Unburdened)	\$1,060,389

Consequently, we require ten (10) Bipolar EP processing tools. We estimated the Bipolar EP tool cost from a FARADAYIC[®] ElectroCell that Faraday is installing at Viasystems' quick-turn printed circuit board (PCB) fabrication facility in Anaheim, CA. While the PCB installation is for plating, it contains bus bars, solution flow with associated pumps and plumbing, all of which will be required for the cavity processing tool. We assume the PCB plating cell to be of a similar complexity to the Bipolar EP processing tool. Specifically, the ElectroCell for PCB fabrication processes twelve (12) 450x600 mm PCB panels. The twelve PCB panels are processed in six pairs in six ElectroCell compartments.

In order to process ten (10) SRF cavities using the Bipolar EP process, we assume an equivalent cost of two (2) FARADAYIC[®] ElectroCells. Excluding rectifiers, the cost of the two (2) FARADAYIC[®] ElectroCells, based on the Viasystems' installation, is \$79,536 (2 x \$39,768). We assume we will require ten (10) pulse reverse rectifiers, one for each SRF cavity at a conservative cost estimate of \$100,000 each with a 20% quantity discount. In addition, based on discussions with AES, we assume the costs of the negative pressure processing room and the Vapor scrubber and exhaust system are about 50% of that required for the Baseline EP tool. Finally, we assume the facilities modifications for the FARADAYIC[®] Bipolar EP process are two times that of the Baseline process based on the requirement for ten (10) versus five (5) tools. The

capital cost estimates are tabulated in Table 5. The total capital cost estimate to meet the anticipated demand of ILC cavity processing indicates that the FARADAYIC[®] Bipolar EP is ~50% that of the Baseline EP process.

Table 5: Capital Cost Comparison

	Baseline EP	Bipolar EP
	Qty (5)	Qty (10)
EP Tool (Design,	\$2,274,936	\$79,536
Fabrication,		
Installation): (40%)		
Rectifier (20%)	Included	\$800,000
EP Tool Shipping:	\$65,898	\$10,000
(30%)		
Processing Room	\$396,000	\$198,000
(Negative Pressure):		
(40%)		
Vapor Scrubber and	\$183,222	\$91,611
Exhaust System:		
(30%)		
Facility		
Modifications:		
10 Ton HVAC:	\$68,250	\$136,500
(30%)		
Electrical Service	\$50,375	\$100,750
Upgrade: (50%)		
Electrical	\$97,250	\$194,500
Distribution: (50%)		
Plumbing (Waste,	\$31,875	\$63,750
Shower, Water,		
Chillers): 50%		
Discount		
Fire Sprinklers,	<u>\$19,000</u>	<u>\$38,000</u>
Roof Penetrators		
Total Cost	\$3,186,806	\$1,712,647
(Unburdened)		

The final cost comparison addressed materials degradation (corrosion) and maintenance issues. AES has observed considerable materials degradation attributed to the corrosive environment associated with the concentrated sulfuric-hydrofluoric acid mixture used in the Baseline EP process. Corroded chiller fittings are depicted in Figure 3 and Figure 4. These issues could be addressed in future Baseline EP tools by using plastic or TEFLON[®] fittings. Figure 5 depicts corrosion of the rotary seals on the Baseline EP tool leading to leaks.

Recall that the FARADAYIC[®] Bipolar EP cavity is not rotated and does not require rotary seals. Due to the limited experience of AES with the Baseline EP Tool it is not possible to quantify the costs associated with materials corrosion and maintenance at this time. However, we anticipate less materials degradation and maintenance with the less corrosive electrolyte associated with the Bipolar EP process.



Figure 3: Corroded chiller fittings in AES Baseline EP tool.



Figure 4: Corroded chiller fittings on AES Baseline tool.



Figure 5: Corroded rotary seals on AES Baseline EP tool.

The final advantage associated with the FARADAYIC[®] Bipolar EP process is the worker and environmental friendly nature of the low concentration sulfuric acid-water electrolyte that does not contain hydrofluoric acid. This attribute was in fact the rationale for the DOE SBIR and ARRA programs. This advantage is considered intangible and not easily quantified at this time. In Table 6, we summarize the operating, capital, maintenance and intangible costs associated with the Baseline EP and FARADAYIC[®] Bipolar EP processes to meet the anticipated ILC SRF demand of 3,827 cavities.

	Baseline EP	Bipolar EP
Operating:		
Acid	\$11,228,418	\$1,125,138
Labor	\$2,965,925	\$1,817,825
Capital:	\$3,186,806	\$1,712,647
TOTAL COST	\$17,381,149	\$4,655,610
Intangible:		
Materials	×	✓
Environment	×	✓

Table 6: Cost Summary for Anticipated ILC Demand

SUMMARY

Based on this first order economic analysis, we conclude that the FARADAYIC[®] Bipolar EP process offers substantial savings relative to the Baseline EP process in terms of operating, capital and intangible costs. A major operating cost saving for Bipolar EP is associated with low sulphuric acid concentration and the absence of hydrofluoric acid. A major capital cost saving for Bipolar EP is associated with vertically oriented stationary cavity processing. Additional intangible cost savings are expected in terms of worker safety as well as less material degradation and maintenance requirements. In terms of tangible cost savings, the cost per cavity for the FARADAYIC[®] Bipolar EP and Baseline EP are \$1,293 and \$4,828 per cavity (3600), respectively. The continued development and validation of the FARADAYIC[®] Bipolar EP process in nine cell cavities will contribute greatly to the industrialization of SRF accelerator technology.

REFERENCES

- F. Eozenou, A. Aspart, C. Antoine, B. Malki, CARE Report 06-10-SRF, EU contract number RII3-CT-2003-506396 (2006).
- [2] V. Palmieri, G. Mondin, V. Rampazzo, D. Rizzetto, V. Rupp, S. Stivanello, S. Deambrosis, A.A. Rossi, "Niobium Electropolishing by Ionic Liquids: What are the Naked Facts?" THOAAU03, Proc. SRF2009, http://jacow.org/.
- [3] D. Blodget, American J. Industrial Medicine **40**, 215 (2001).
- [4] J. Sun, E.J. Taylor, R. Srinivasan, J. Materials Processing Technology 108(3), 356 (2001).
- [5] C. Zhou, E.J. Taylor, J. Sun, L. Gebhart, R. Renz, U.S. Patent No. 6,402,931, June 11, 2002.
- [6] E.J. Taylor, U.S. Patent No. 6,558,231 May 6, 2003.
- [7] E.J. Taylor, J. Appl. Sur. Fin., 3(4), 178 (2008);
 Plating & Surface Finishing 95(12), 24 (2008).
- [8] M. Inman, T. Hall, E.J. Taylor, C. Reece, O. Trofimova, "Niobium Electropolishing in an Aqueous, Non-Viscous HF-Free Electrolyte: A New Polishing Mechanism," TUPO012, Proc. SRF2011, http://jacow.org/.
- [9] M. Inman, H. Garich, S. Snyder, E.J. Taylor, L. Cooley, C.A. Cooper, A. Rowe, "Development and Scale-Up of an HF Free Electropolishing Process in

SRF Technology - Processing

F02-Surface treatments

Single-Cell Niobium SRF Cavities," TUPO017, Proc. SRF2011, http://jacow.org/.

- [10] M. Inman, E.J. Taylor, T.D. Hall, J. Electrochem. Soc., 160(9), E94-8 (2013).
- [11] E.J. Taylor T. Hall, S. Snyder, M. Inman, A. Rowe, "Electropolishing of Niobium SRF Cavities in Low Viscosity Aqueous Electrolytes Without Hydrofluoric Acid," TUP054, Proc. SRF2013, http://jacow.org/.
- [12] A. Rowe, A. Grassellino, T. Hall, M. Inman, S. Snyder, E. Taylor, "Bipolar EP: Electropolishing without Fluorine in a Water Based Electrolyte," TUIOC02, Proc. SRF2013, http://jacow.org/.
- [13] H. Diepers, O. Schmidt, H. Martens, F.S. Sun, Physics Letters 37A(2), 139 (1971).
- [14] H. Diepers, O. Schmidt, US Patent No. 3,939,053, February 17, 1972.
- [15] E.J. Taylor, M. Inman, T. Hall, U.S. Patent No. 9,006,147, April 14, 2015; PCT/US13/45056 filed June 11, 2013.
- [16] M. Inman, E.J. Taylor, T. Hall, S. Snyder, S. Lucatero, F. Furuta, G. Hoffstaetter, J. Mammosser "Electropolishing of Niobium SRF Cavities in Eco-Friendly Aqueous Electrolytes without Hydrofluoric Acid," MOPB101, Proc. SRF2015, http://jacow.org/.
- [17] J. Rathke, M. Cole, A. Favale, E. Peterson, "Fabrication of ILC Prototype Cavities at Advanced Energy Systems, Inc.," MOPD002, Proc. EPAC08, http://jacow.org/.