Activities Directed Towards *Industrialization* of HF-FREE ElectroPolishing of Niobium SRF Cavities

Acid-Free Electropolishing of SRF Cavities NP Phase II Grant No. DE-SC0011235

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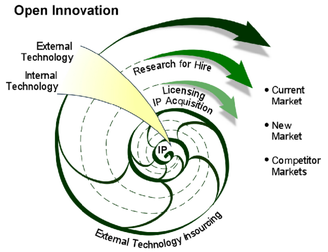
Abstract

Under prior and on-going funding from the DOE (SBIR, ARRA, and ORNL P.O.), Faraday Technology has demonstrated the ability to electropolish single-cell, stacked single cells, and nine-cell niobium SRF cavities in low concentration acid electrolytes (5-10% sulfuric acid) using pulse reverse current electrolysis . In contrast to conventional direct current electropolishing in concentrated sulfuric-hydrofluoric acid electrolytes, the FARADAYIC® ElectroPolishing process enables vertically orientated cavity polishing without the need for cavity rotation. This is inherently more scalable and industrially compatible than the oft-used horizontal orientation. This presentation will review the previous work and present the current status of FARADAYIC® ElectroPolishing activities.

Commercialization Model: Open Innovation



Development of robust process is critical!

- **Leverage Federal SBIR opportunities as non-equity technology funding**
  - Establish IP rights (34 US patents issued)
  - 2 US, Japanese, European patents specific to Nb electropolishing
- **Collaborate with universities/government laboratories**
  - TJNAF, KEK, Cornell, FNAF
- **Develop electrochemical engineering solutions based on PC/PRC processes**
  - FARADAYIC® HF-FREE ElectroPolishing
- **Industrialization: Transition of EP technology**
  - DEM/VAL
    - α-scale at Faraday
    - β-scale at TJNAF, KEK, metal finishing company
  - Become defined "Build to Print & Process"
  - Geographic "jump start" license
    - Japan metal finishing (Marui, Nomura, Mitsubishi)
    - Europe metal finishing industry
  - Faraday US production – then license for volume
    - US metal finishing (TechMetals, ABLE EP)

Key: TJNAF (FNAF) define "Build to Print & Process"

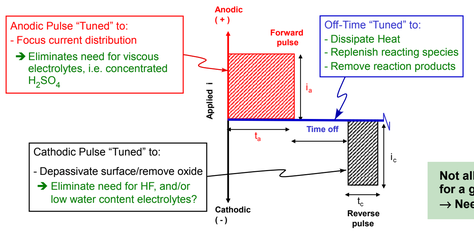
Opportunity: SRF Niobium Cavity Electropolishing (EP)

Current Process	FARADAYIC® Process
9:1 H_2SO_4 (98%) : HF(48%) electrolyte	5 to 30 w/w% H_2SO_4 or 200 g/L $NaNO_3$

T. Dote, K. Kono(2004), Japanese Journal of Occupational Medicine and Traumatology, 52, 3, pp189-192.

- **FARADAYIC® HF-FREE EP**
  - U.S. Pat. No. 9,006,147 4/14/2015
  - Jap Pat. No. 6,023,323 10/14/ 2016
  - EP Pat. No. 2,849,908 1/15/2017
    France, Germany, Italy,
    Switzerland, United Kingdom
  - U.S. Pat. No. 9,987,699 6/5/2018

Pulse Reverse EP Process



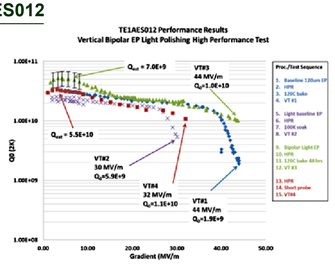
Not all waveforms work for a given application!
→ Need guidance!

Single Cell Cavity – TE1AES012

"BiPolar EP" (PRC)
- Vertical
- 100% Volume Fill
- No Rotation
- 10 wt% H_2SO_4 in H_2O

25 μm removed "light EP"

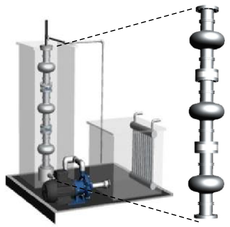
"…Cavity achieved a maximum gradient of ~44 MV/m with a Q_0 of 1 X 10^{10}, the highest gradient observed at Fermilab in any cavity regardless of processing technique…"

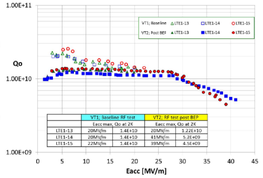


†E.J. Taylor, T.D. Hall, M. Inman, S. Snyder "Electropolishing of Niobium SRF Cavities in Low Viscosity Aqueous Electrolytes without Hydrofluoric Acid" Paper No. TUP054, Presented SRF2013, Paris, FRANCE Sept. 2013.

†A.M. Rowe, A. Grassellino, T.D. Hall, M.E. Inman, S.T. Snyder, E.J. Taylor "Bipolar EP: Electropolishing without Flourine in a Water Based Electrolyte" Paper No. TUIOC02, Presented SRF2013, Paris, FRANCE, 2013.

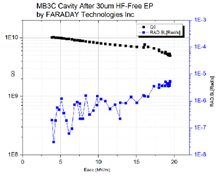
3 Stacked 1-Cell/3-Cell Cavities



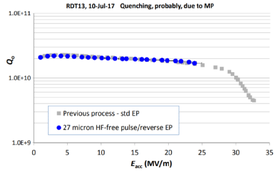


	VT1, Baseline RF test	VT2, RF test post BCP
	Eacc max, Q0 at 2K	Eacc max, Q0 at 2K
LTE1-13	20MV/m	1.4E+10	31MV/m	1.21E+10
LTE1-14	20MV/m	1.4E+10	41MV/m	1.1E+09
LTE1-15	22MV/m	1.4E+10	39MV/m	4.5E+09

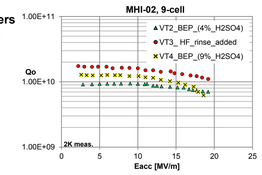
"…very good results." J. Mammosser
Prototype Medium Beta 0.61 805MHz for SNS



1-Cell/N_2 1-Cell/9-Cell Cavities



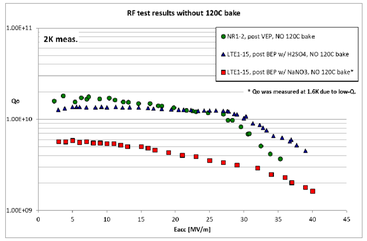
- **1-Cell Cavity β-Test at JLAB using Faraday Parameters**
  - JLAB home grown rectifier (L. Phillips/C. Reece)
  - Equivalent performance (C. Reece)
- **1-Cell Cavity N_2 "Doped" processed at Faraday**
  - Equivalent performance (Not Shown)(C. Reece)
- **9-Cell Cavity processed at Faraday**
  - Highest performance for this cavity 32 MV/m
  - Radiation limited due to contaminant (C. Reece)



VT2; post Bipolar EP with 4% of H_2SO_4, but NO 120C bake
VT3; HF rinse added, but NO 120C bake
VT4; post Bipolar EP with 9% of H_2SO_4, but NO 120C bake

The 9-cell, MHI-02, has NO std. baseline data since the cavity was used for the high-Q R&D, the first VT (VT1) included the high-Q surface treatment (low temp. bake with N_2).

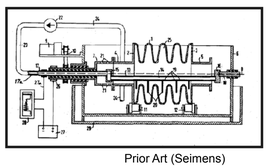
Acid Free 1-Cell Cavity Processing



	Baseline EP	Acid-Free Bipolar EP	Bipolar EP
Electrolyte	9:1 H_2SO_4:HF	200 g/L $NaNO_3$ + 19.5 g/L Tris Acid + 1 g/L Tris Base mixture in H_2O	~5% (wt) H_2SO_4 in H_2O
Processing Voltage	DC: ~17 V	Pulse Reverse: ~5V/20V	Pulse Reverse: ~3V/9V
Processing Temperature	25 C	40 C	25 C
Cavity Orientation	Horizontal	Vertical	Vertical
Electrolyte Volume Fill	60%	100%	100%
Electrolyte Flow Rate	~8 L/min	~8 L/min	~8 L/min
Cavity Rotation	1 rpm	No Rotation	No Rotation
Cathode Material/Shape	Aluminum/Tube	Titanium/Rod	Titanium/Rod
Material Removal Rate	0.3 μm/min	0.4 μm/min	0.15 μm/min

Overall This Study
- Achieved material removal rate 2 times greater than what has been achieved with our patented Bipolar EP (BEP) by utilizing the Acid-Free BEP approach.
- Demonstrate that Acid-Free BEP diminish performance
  - But can be recovered to outperform the conventional VEP by applying a post treatment with our light acid water BEP process.

Commercialization: IP Strategy



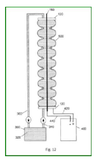
Prior Art (Seimens)

"Method for the electrolytic polishing of the inside surface hollow niobium bodies" U.S. Patent No. 4,014,765 issued March 29, 1977.

Viscous electrolyte (9:1 H_2SO_4 : HF)
- Horizontal orientation
- Partially filled
- Rotation

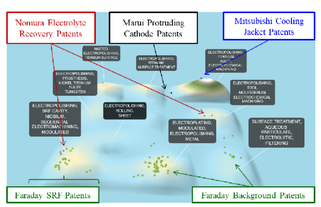
Challenge for industrialization
- Electrolyte safety
- High CapEx and OpEx

Intellectual Property Portfolio
- FARADAYIC® registered
  - Trademark Reg. No. 3,178,757
  - Service mark Reg. No. 3,423,999
- FARADAYIC® HF-FREE EP
  - U.S. Pat. No. 9,006,147 4/14/2015
  - Japan Pat. No. 6,023,323 10/14/ 2016
  - EP Pat. No. 2,849,908 1/15/2017
    France, Germany, Italy, Switzerland, United Kingdom
  - U.S. Pat. No. 9,987,699 6/5/2018



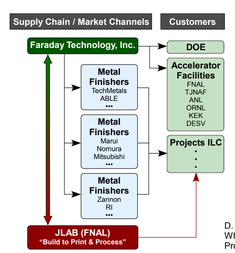
Independent claim directed towards low viscosity!!
- Low concentration – aqueous electrolyte (acid/alkaline)
  - Vertical orientation
  - Completely filled
  - No rotation
- Industrially compatible – metal finishing value chain
  - Safe
  - Low CapEx and OpEx

Commercialization: Synergistic IP?



- Independent claim directed towards low viscosity!!
- Strong patent cluster
- Synergistic with recent art (Marui/Mitsubishi)

Commercialization: Value Chain



- Low concentration – aqueous electrolyte (acid/alkaline)
  - Vertical orientation
  - Completely filled
  - No rotation
- Industrially compatible – metal finishing value chain
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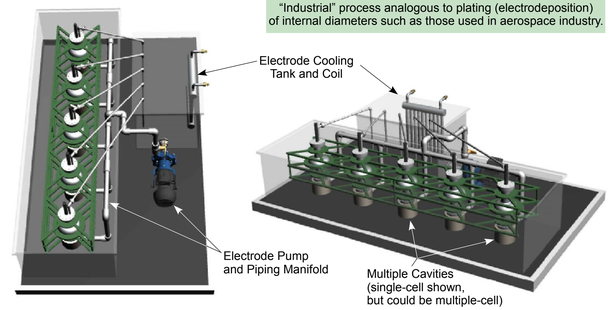
  - Low CapEx and OpEx

D. Sutter, B. Strauss "Technology Transfer – When, Why, Issues and Advantages" Proc. PAC07, MOZAC01 Albuquerque, NM 2007.

Industrialization: BiPolar HF-FREE EP

"Industrial" process analogous to plating (electrodeposition) of internal diameters such as those used in aerospace industry.



Commercialization: Economics

3,827 cavities over six years (U.S. portion) to meet the 3,600 cavities required for the ILC

	Baseline VEP	FARADAYIC® BEP	Acid-Free FARADAYIC® BEP	FARADAYIC® BEP	Combined TOTAL
OPERATING:					
Electrolyte	$11,228,418	$1,125,138	$1,816,436	$1,125,138	$2,941,574
Labor	$2,965,925	$1,817,825	$917,825	$617,825	$1,535,650
CAPITAL:	$3,186,806	$1,712,647		$1,027,588	$1,027,588
TOTAL COST	**$17,381,149**	**$4,655,610**		**Combined:**	**$5,504,812**
INTANGIBLE:					
Materials Degradation	×	✔	✔	✔	✔
Worker/Environment	×	✔	✔	✔	✔

E.J. Taylor, M. Inman, T. Hall, S. Snyder, A. Rowe, D. Holmes "Economics of Electropolishing Niobium SRF cavities in Eco-Friendly Aqueous Electrolytes without Hydrofluoric Acid" Proceedings of SRF2015 MOPB092 pp. 1-5 Whistler, CANADA (2015).

Industrialization: Post-Phase II Work

- High rate NO-ACID process (vis-à-vis 5% H_2SO_4)
  - Higher throughput → less systems for given demand: CapEx
- Apparatus/cell design for industrialization
  - Racking/fixturing for metal finishing industry: Compatibility
- Waste recycle, recovery and disposal
  - Protocol for metal finishing industry: Compatibility
- Scale-Up of low cost rectifier breadboard (TJNAF; L. Phillips, C. Reece pat. Appl.)
  - ~$10K versus $80K used in AES economic study: CapEx/Strategic
- Waveform optimization
  - Higher throughput → less systems for given demand: CapEx
- COMSOL modeling (confirming experiments) impact of electrode shape/shielding on current distribution, including "Ninja Electrode (Marui)
  - Improved material removal uniformity: OpEx/Strategic
- COMSOL modeling (confirming experiments) impact of electrode shape on electrolyte distribution, including "Ninja Electrode" (Marui)
  - Improved material removal uniformity: OpEx/Strategic
- Synergies with emerging cavity processing, cooling jackets (Mitsubishi)
  - Improved material removal uniformity: OpEx/Strategic

β-scale testing; TJNAF, ORNL, FNAF, TechMetals, ABLE, KEK, Marui, Mitsubishi, Zannon, Research Instruments → "Build to Print & Process"

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Collaborators:

1. Dr. Fumio Furuta and Dr. Geoff Hoffstaetter; Cornell University, Fermi Laboratory
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3. Dr. John Mammosser and Dr. Jeff Saunders; Oak Ridge National Laboratory
4. Dr. Takayuki Saeki; KEK High Energy Accelerator Research Organization
5. Mr. Allan Rowe and Dr. Anna Grassellino; Fermi Laboratory

Technical Papers, Presentations and Patents

1) M. Inman, T. Hall, E.J. Taylor, C.E. Reece, O. Trofimova "Niobium Electropolishing in Aqueous, Non-viscous, HF-FREE Electrolyte: A New Polishing Mechanism" Proceedings of SRF2011 TUPO012 pp. 277-381 Chicago, IL (2011).
2) E.J. Taylor, M.E. Inman, T. D. Hall "Electrochemical System and Method for Electropolishing Superconductive Radio Frequency Cavities" U.S. Patent No. 9,006,147 filed July 11, 2012 issued Apr. 14, 2015. (Foreign counterparts pending)
3) M. Inman, E.J. Taylor T.D. Hall "Electropolishing of Passive Materials in HF-Free Low Viscosity Aqueous Electrolytes" J. Electrochemical Society 160 (9) E94-E98 (2013).
4) A.M. Rowe, A. Grassellino, T.D. Hall, M.E. Inman, S.T. Snyder, E.J. Taylor "Bipolar EP: Electropolishing without Flourine in a Water Based Electrolyte" Proceedings of SRF2013 TUIOC02 pp. 401-406 Paris, FRANCE (2013).
5) E.J. Taylor, M. Inman "Electrochemical Surface Finishing" Interface 23(3) pp. 57-61 Fall 2014.
6) E.J. Taylor, T. Hall, M. Inman, S. Snyder, A. Rowe "Electropolishing of Niobium SRF Cavities in Low Viscosity Aqueous Electrolytes without Hydrofluoric Acid" Proceedings of SRF2013 TUP054 pp. 534-7 Paris, FRANCE (2015).
7) E.J. Taylor, T.D. Hall, S. Snyder, M.E. Inman "Electropolishing of Niobium SRF Cavities in Low-Viscosirt, Water-Based, HF-Free Electrolyte: From Coupons to Cavities" Invited Talk 226th Meeting of the Electrochemical Society and XIX Congreso de la Sociedad Mexicana de Electroquimica, MEXICO (2014)
8) E.J. Taylor, M.E. Inman, T. D. Hall "Electrochemical System and Method for Electropolishing Superconductive Radio Frequency Cavities" U.S. Patent Appl. No. 14/585,897 filed December 30, 2014.
9) E.J. Taylor, M. Inman, T. Hall, S. Snyder, A. Rowe, D. Holmes "Economics of Electropolishing Niobium SRF cavities in Eco-Friendly Aqueous Electrolytes without Hydrofluoric Acid" Proceedings of SRF2015 MOPB092 pp. 1-5 Whistler, CANADA (2015).
10) E.J. Taylor, M. Inman "Vertical Electropolishing Studies at Cornell" Proceedings of SRF2015 MOPB093 pp. 364-7, Whistler, CANADA (2015).
11) M. Inman, E.J. Taylor, T. Hall, S. Snyder, S. Lucatero, A. Rowe, F. Furuta, G. Hoffstaetter, J. Mammosser "Electropolishing Niobium SRF cavities in Eco-Friendly Aqueous Electrolytes without Hydrofluoric Acid" Proceedings of SRF2015 MOPB101 pp. 390-3 Whistler, CANADA (2015).
12) E.J. Taylor, M.E. Inman, H.M. Garich, H.A. McCrabb, S.T. Snyder, T.D. Hall "Breaking the Chemical Paradigm in Electrochemical Engineering: Case Studies and Lessons Learned from Plating to Polishing" in Advances in Electrochemical Science and Engineering Vol 18 R.C. Alkire (ed) Wiley-VCH, Fall (2018).