Activities Directed Towards HF-FREE ElectroPolishing of Niobium SRF Cavities

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

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CRADA No. DE-AC05-06OR23177

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August 9, 2017

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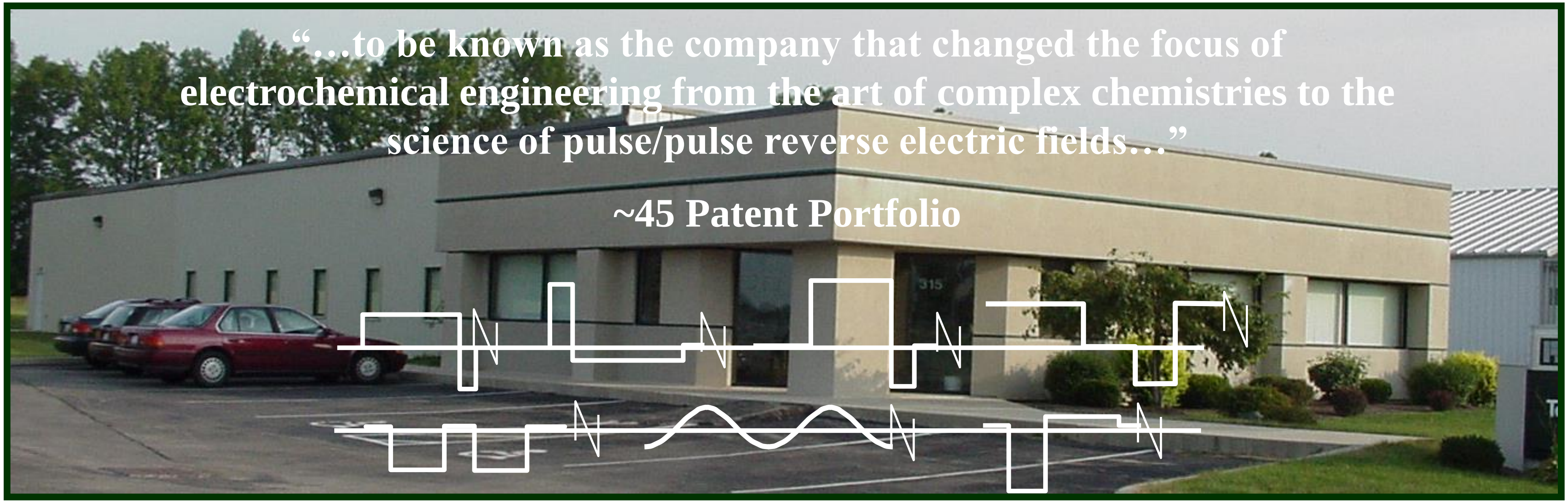
Company Overview: FARADAY TECHNOLOGY, INC.



- Electrochemical engineering processes and technologies – founded 1991
  - In particular, pulse & pulse reverse electrolytic processes
  - ~30 Issued Patents and ~15 Pending Patents in this area
- Perspective
  - PhD in electrochemical kinetics, MS in Technology Strategy, Patent Bar
- www.FaradayTechnology.com

Vision: Pulse Current/Pulse Reverse Current

"…to be known as the company that changed the focus of electrochemical engineering from the art of complex chemistries to the science of pulse/pulse reverse electric fields…"

Electrochemical Machining, Polishing, Deburring, Through-Mask_Etching

Anodic Pulse "Tuned" to:

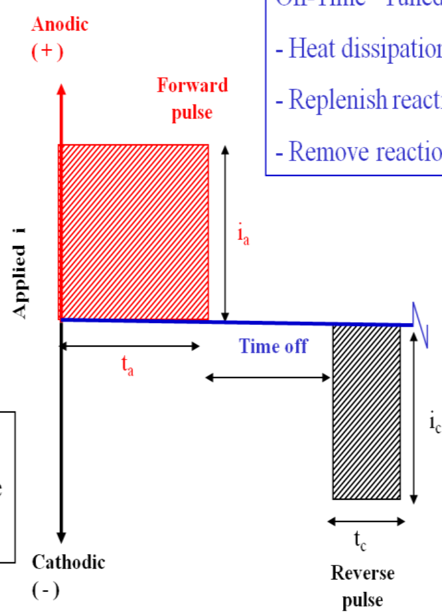
- Control current distribution
  - ➔ Eliminates need for viscous, low water content electrolytes

Off-Time "Tuned" to:

- Heat dissipation
- Replenish reacting species
- Remove reaction products

Cathodic Pulse "Tuned" to:

- Reduce oxide/depassivate surface
  - ➔ Eliminate need for HF



- 2008 Blum Award for Pulse Reverse Finishing
- 2016 R&D 100 Finalist for Nb EP

Electrodeposition/Plating

Cathodic Pulse "Tuned" to:

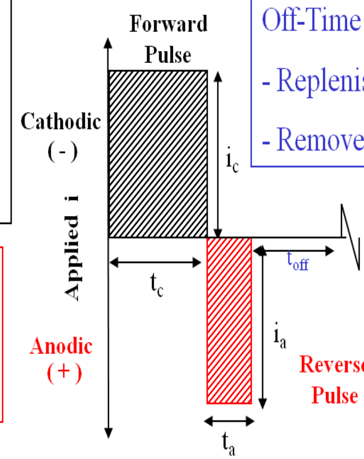
- Enhance mass transfer
- Control current distribution
- ➔ Simplify chemistry

Anodic Pulse "Tuned" to:

- Remove H_2 effects
- ➔ Acidify interface

Off-Time "Tuned" to:

- Replenish reacting species
- Remove reaction products



- 2011 R&D 100 for Co-Mn Alloy Plating
- 2013 Presidential Green Chemistry Challenge award for Cr^{+3} Plating

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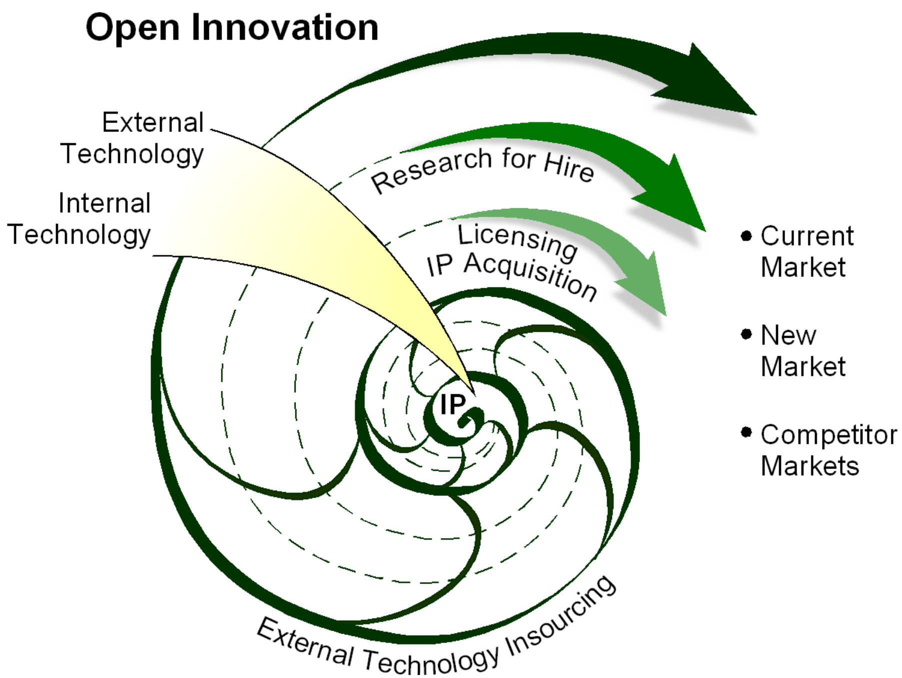
Business Model: Open Innovation



Development of robust process is critical!

- Leverage Federal SBIR opportunities as non-equity technology funding
  - Retain IP rights
- Establish IP (30 US patents issued)
- Collaborate universities/government labs
- Develop electrochemical engineering solutions based on PC/PRC processes
- Transition technology & competitive advantage to large companies via
  - DEM/VAL; α-scale to β-scale
  - Field-of-use licenses
  - Patent acquisition (8)
- Transition of technology for Federal use
  - DEM/VAL; α-scale to β-scale
  - Insertion at Lab or "logistics" center
- Transition of ElectroPolishing technology to physics community
  - DEM/VAL; α-scale to β-scale
  - Geographic License; US, Japan, Europe
  - "Build to Print" market

  Key: β-scale DOE Lab validation

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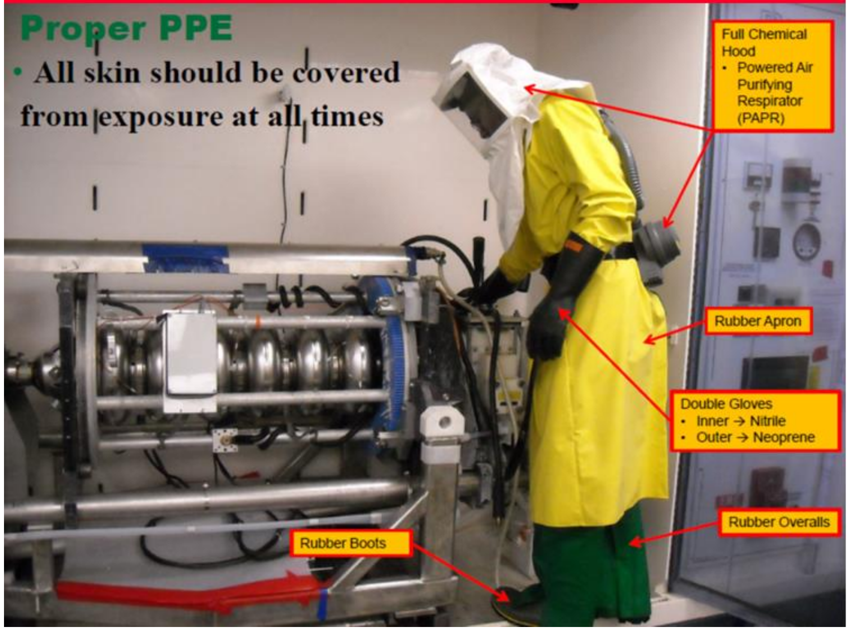
Background: SRF Niobium Cavity Electropolishing (EP)

Nb Superconducting Radio Frequency (SRF) are required for the International Linear Collider as well as other high energy physics projects. To achieve required particle acceleration gradients, electropolishing is the final surface finishing operation;

9:1 H_2SO_4(98%) : HF(48%)
electrolyte (DC)
"High Viscosity"

HF ➔ Safety/Cost Burden

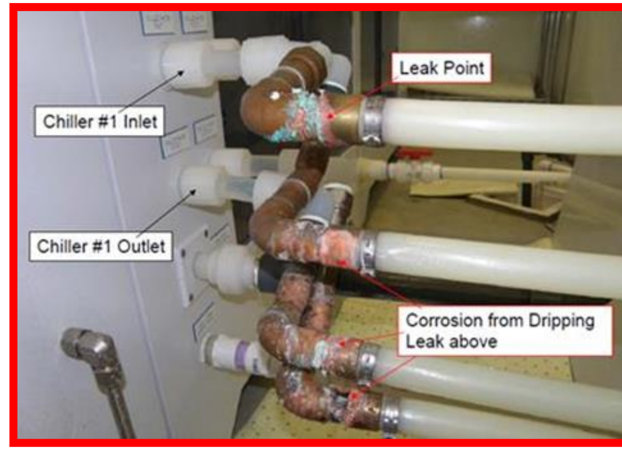
Personal Protective Equipment (PPE) for "conventional" SRF niobium cavity electropolishing using sulfuric acid – hydrofluoric acid mixture.
John Mammosser, Instructor
"Chemical Safety for SRF Work"
U.S. Particle Accelerator School
January 2015



T. Dote, K. Kono(2004), "An Acute Lethal Case of Exposure During A Washing Down Operation of A Hydrogen Fluoride Liquefying Tank", *Japanese Journal of Occupational Medicine and Traumatology*, **52**, 3, pp189-192.

Background: Electropolishing Niobium SRF Cavities

*Nb Superconducting Radio Frequency (SRF) are required for the International Linear Collider as well as other high energy physics projects. To achieve required particle acceleration gradients, **electropolishing** is the final surface finishing operation;*



9:1 H_2SO_4(98%) : HF(48%) electrolyte (DC) "High Viscosity"

HF ➔ Safety/Cost Burden
Corrosive

"…well known… viscous salt film paradigm"

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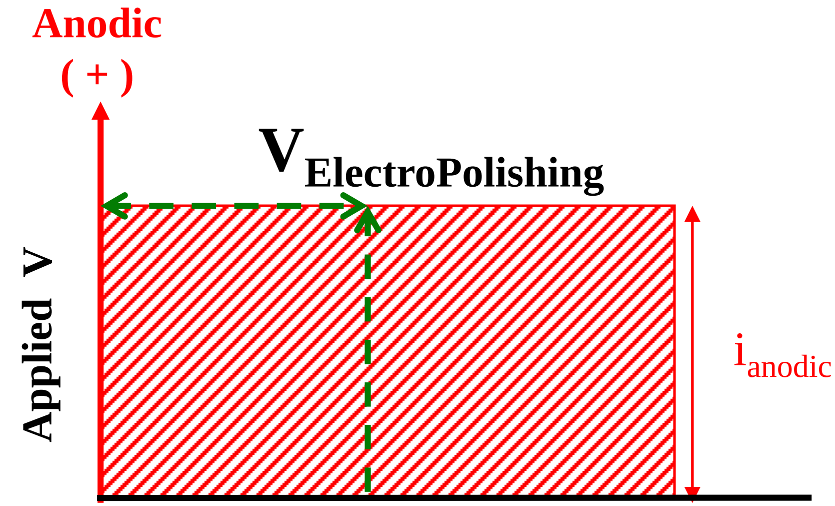
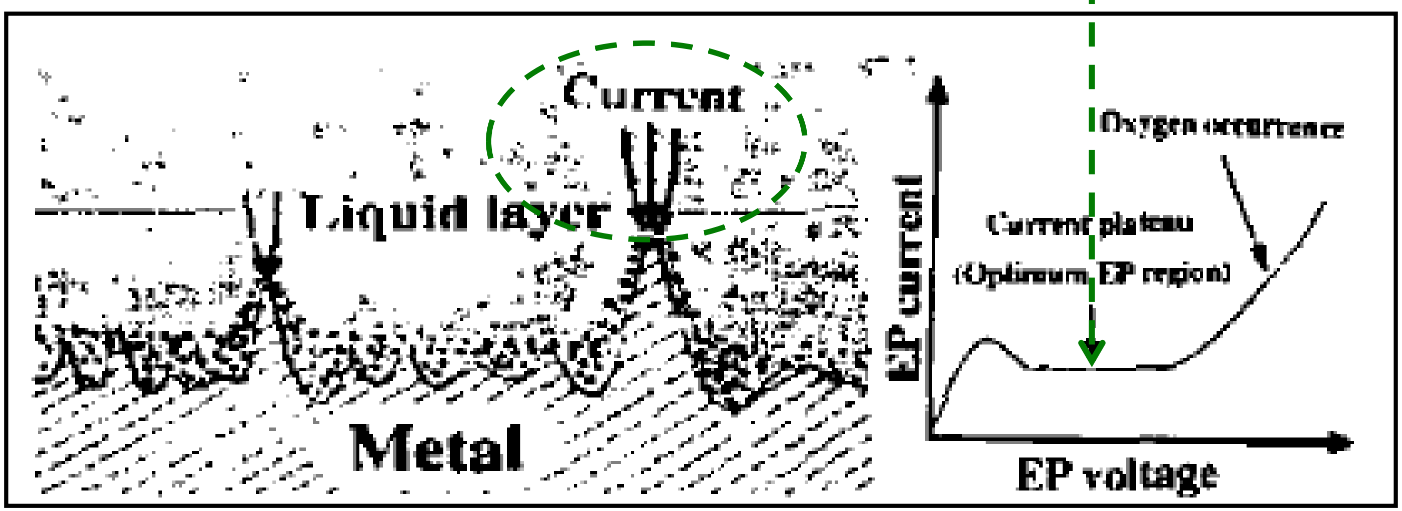
EP Paradigm: Jacquet Viscous Salt Film

- Rectification:
  - DC - Constant voltage
  - Viscous salt film per Jacquet
- Electrolyte:
  - Concentrated/viscous acid
  - Chilled to increase viscosity



➔ *Thick boundary layer*

➔ *Mass transport control to focus current on peaks (tertiary current distribution)*

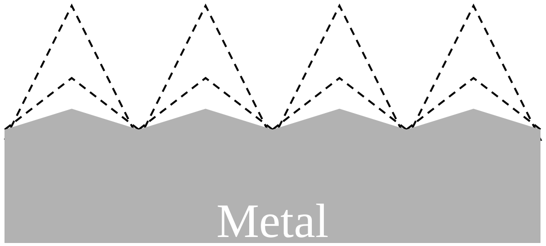


† *P.A. Jacquet, Trans. Electrochem. Soc.,* ***69*** *629 (1936).*

EP Paradigm: Jacquet Viscous Salt Film

- 1st Issue: Viscous/chilled solutions – Jacquet Paradigm:

$$M^0 \rightarrow M^+ + e^-$$



➔ Thick/viscous boundary with concentrated H_2SO_4

- 2nd Issue: Polishing oxide forming materials:

$$2Nb + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 10e^-$$

➔ Oxide removal with fluoride or hydrofluoric acid (HF)

- challenging control/safety issues associated with HF

➔ Non-aqueous electrolytes† < 5% H_2O

- limited industrial implementation (NiTi stents)

P. Kneisel, "High Gradient Superconducting Niobium Cavities: A Review of the Present Status" *IEEE Trans. Appl. Superconductivity*, 9(2) 1023-1029 (1999).

J.B. Mathieu, D. Landolt "Electropolishing of Titanium in Perchloric Acid-Acetic Acid Solutions" *J. Electrochem. Soc.* 125(7) 1044 (1978).

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Consequence of High Viscosity EP

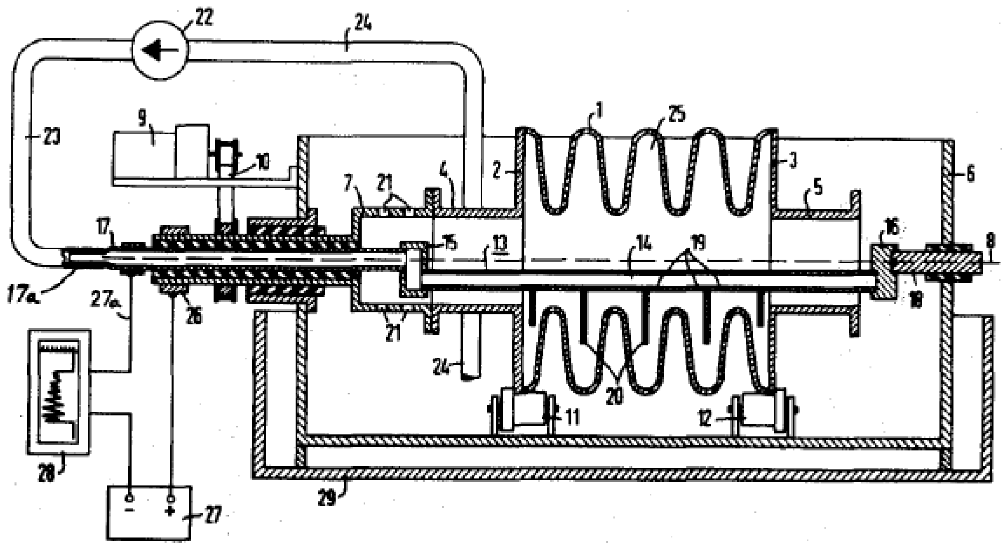
US Patent No. 4,014,765 (Siemens)

Problem of …

*"…electrolytic polishing hollow niobium bodies of a **complicated geometrical structure**…where development of gases…rise from the cathode…forming **gas pockets**…resulting in portions of the **inside surface not polished**…"*

…is solved by…

*"…**horizontally orienting** the hollow niobium **body…partially filling** said hollow body with polishing solution and slowly **rotating** said hollow body…"*



Note;
electrode "fins" to level current distribution disclosed – only recently considered by the particle physics community?

➔ *Horizontal operation adds significant capital cost in cavity processing tools and hinders process industrialization!*

Pulse Reverse EP Process

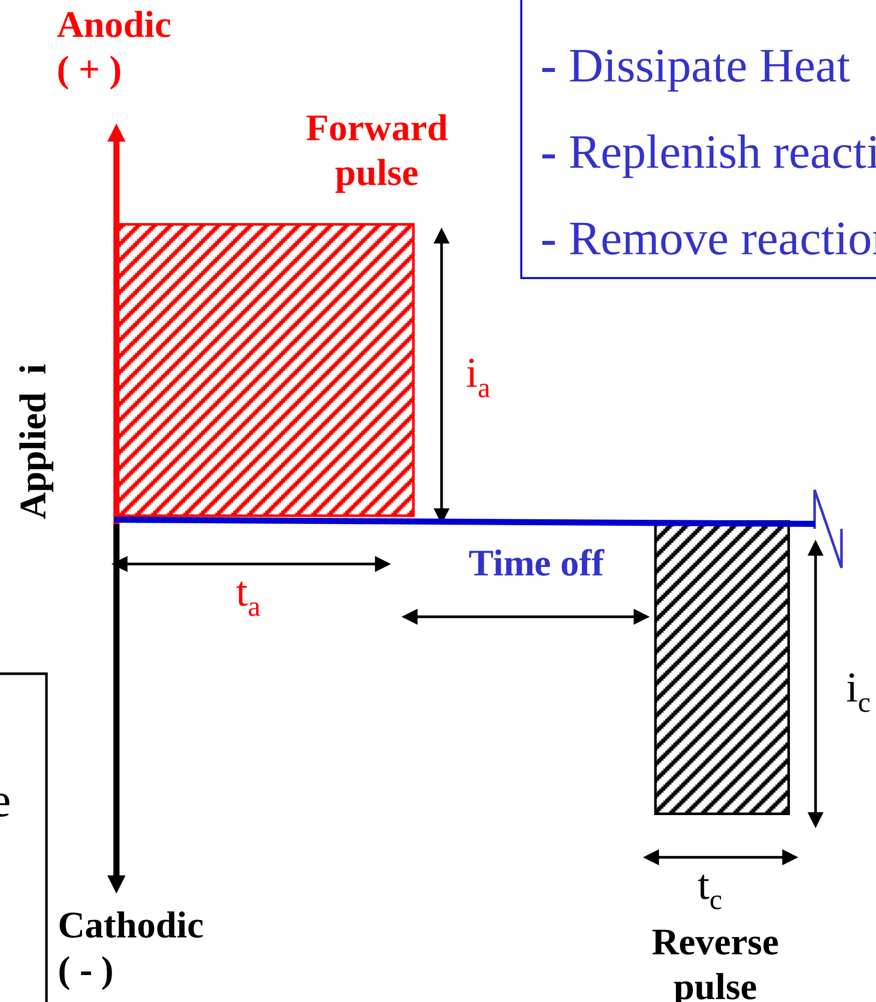
Anodic Pulse "Tuned" to:

- Focus current distribution

  ➔ Eliminates need for viscous electrolytes

Off-Time "Tuned" to:

- Dissipate Heat
- Replenish reacting species
- Remove reaction products



Cathodic Pulse "Tuned" to:

- Depassivate surface/remove oxide

  ➔ Eliminate need for HF, and/or low water content electrolytes?

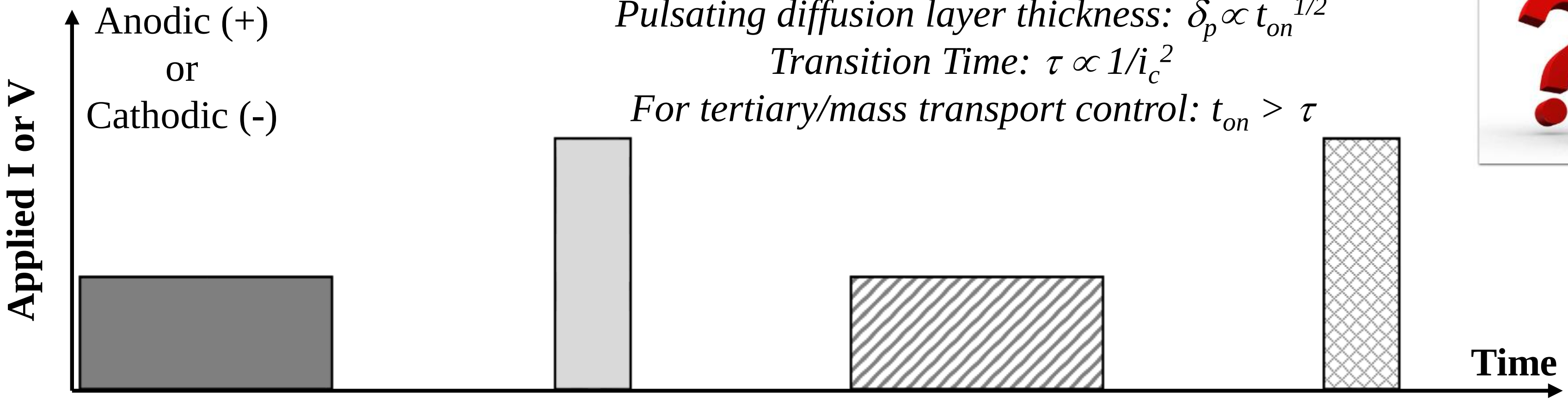
Guidance for Pulse Waveform Parameters†

KEY RELATIONSHIPS

Pulsating diffusion layer thickness: $\delta_p \propto t_{on}^{1/2}$

Transition Time: $\tau \propto 1/i_c^2$

For tertiary/mass transport control: $t_{on} > \tau$



MACROPROFILE	MACROPROFILE	MICROPROFILE	MICROPROFILE
Long $t_{on} \rightarrow \delta_p < \delta$	Short $t_{on} \rightarrow \delta_p << \delta$	Long $t_{on} \rightarrow \delta_p < \delta$	Short $t_{on} \rightarrow \delta_p << \delta$
Larger pulsating diffusion layer maintains macroprofile	Smaller pulsating diffusion layer maintains macroprofile	Larger pulsating diffusion layer maintains microprofile	Smaller pulsating diffusion layer converts microprofile to macroprofile
Low $i_{peak} \rightarrow$ Higher τ Design $t_{on} >> \tau$	High $i_{peak} \rightarrow$ Smaller τ Design $t_{on} << \tau$	Low $i_{peak} \rightarrow$ Higher τ Design $t_{on} >> \tau$	High $i_{peak} \rightarrow$ Smaller τ Design $t_{on} >> \tau$
Favors more tertiary control	Favors more primary or secondary control	Favors more tertiary control	Favors more tertiary control
SLIGHTLY MORE **NON-UNIFORM** THAN DC	*SIGNIFICANTLY MORE* **NON-UNIFORM** THAN DC	*SLIGHTLY MORE* **UNIFORM** THAN DC	*SIGNIFICANTLY MORE* **UNIFORM** THAN DC

Adv in Electrochemical Science and Engineering XVIII: "Breaking the Chemical Paradigm in Electrochemical Engineering: Case Studies and Lessons Learning from Plating to Polishing" publication scheduled Fall 2017.

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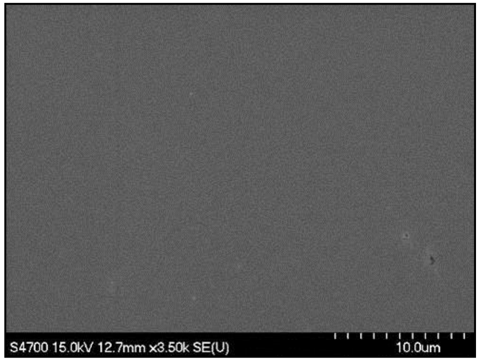
Pulse Reverse Niobium EP: Coupons

- Pulse/Pulse Reverse - coupons
  - Aqueous H_2SO_4 electrolyte (5 to 30%)
  - Strong Passive film
    - ➔ Forward (anodic) pulses
    - ➔ Reverse (cathodic) pulses
  - Fast Waveforms
    - Bulk removal (100µm)
    - ~0.5-1µm/min
  - Slower Waveforms
    - Final EP (25µm)
    - ~0.03-0.3µm/min
    - R_a <0.05µm (stylus)
  - Extremely clean surface

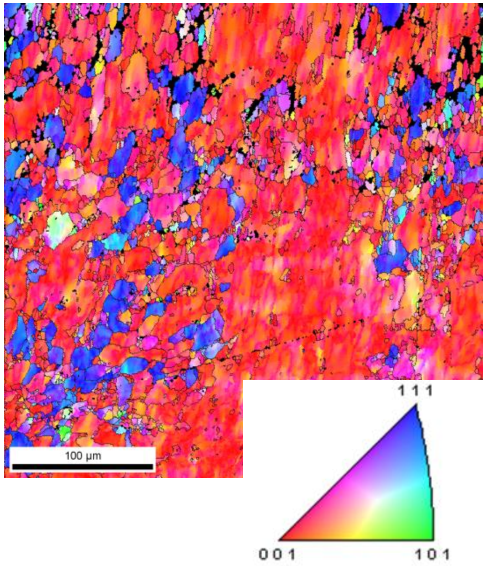
Dr. C. Reece, T. Jefferson lab

"…comparable to standard HF EP…"

SEM Image

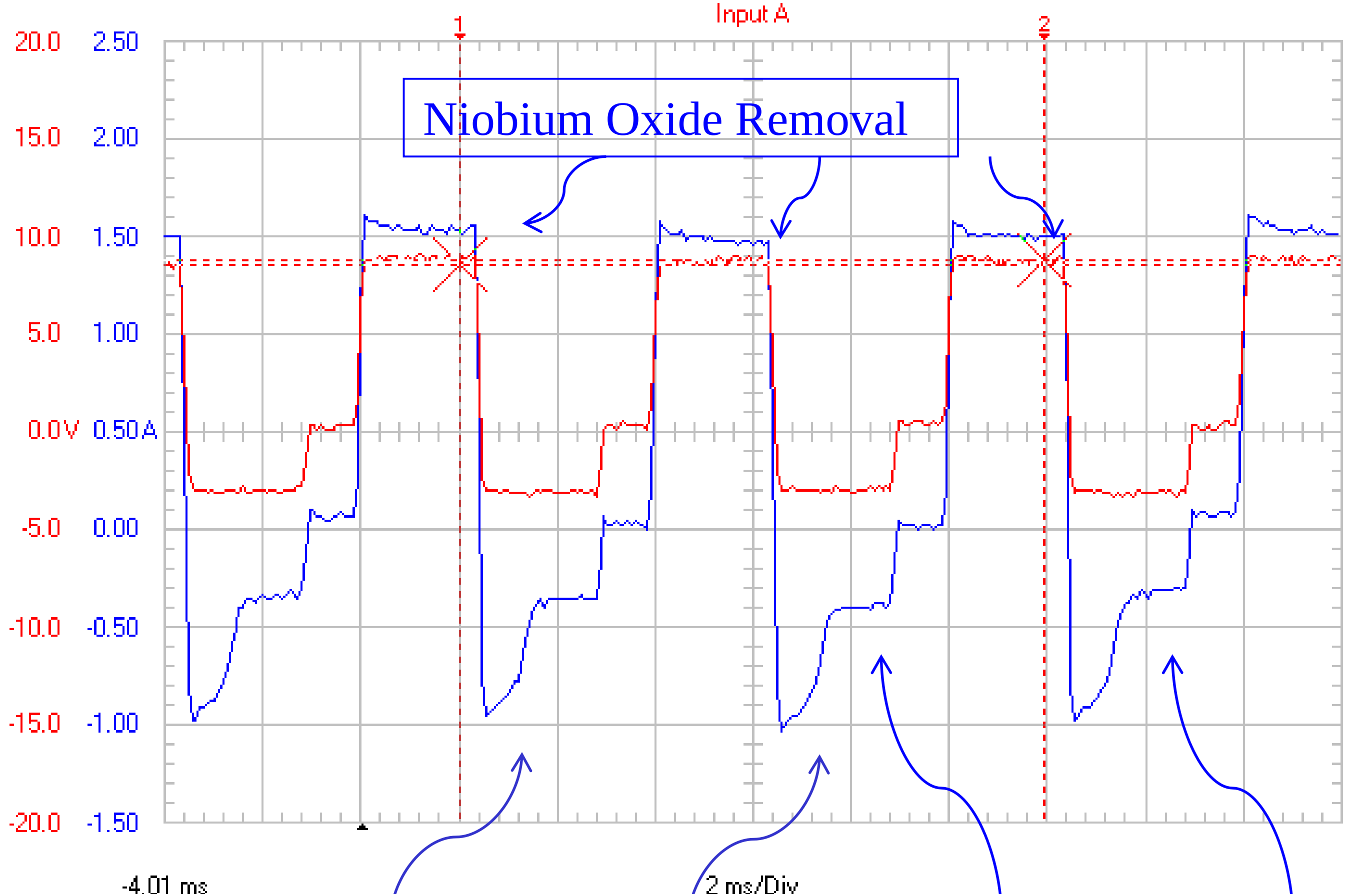


EBSD Profile



Scan size **µm**	Scan No.	R_{max} **nm**	R_a **nm**	RMS **nm**
50x50	1	35.00	2.71	3.34
	2	37.30	3.54	4.73
	3	69.66	3.74	4.69
10x10	1	22.59	2.25	2.87
	2	16.16	0.41	0.54
2x2	1	9.42	0.36	0.46

Anodic Current Response† *Cathodic EP?*



Datablock

Name	= Input A	Input B
Date	= 8/15/2011	8/15/2011
Time	= 9:14:18 AM	9:14:18 AM
Y Scale	= 5 V/Div	500 mA/Div
Y At 50%	= 0.0 V	0.50 A
X Scale	= 2 ms/Div	2 ms/Div
X At 0%	= -4.01 ms	-4.01 ms
X Size	= 300 (300)	300 (300)
Maximum	= 9.6 V	1.70 A
Minimum	= -3.8 V	-1.08 A

Cursor Values

X1:	1.99 ms
X2:	13.91 ms
dX:	11.92 ms
Y1:	8.6 V
Y2:	8.8 V
dY:	0.2 V

Anodic: 3V 2.5 ms
off: 1.0 ms
Cathodic: 9V 2.5 ms

$5Nb + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 10e^-$

$H_2O \rightarrow 1/2O_2 + 2H^+ + 2e^-$

Transition from oxide formation to oxygen evolution?

Anodic current transition correlates with effective electropolishing!

†M. Inman, E.J. Taylor, T.D. Hall "Electropolishing of Passive Materials in HF-Free Low Viscosity Aqueous Electrolytes" *J. Electrochem. Soc.*, **160**(9) E94-E98 (2013).

Vertical EP

Pulse Reverse EP Studies

- Vertical (electrolyte "dump" mode)
- 100% Volume Fill
- No Rotation
- 5-10 wt% H_2SO_4 in H_2O

➔ Analogous to plating of IDs

➔ Simpler/Industrial Compatible

➔ *Enabled by low viscosity electrolyte!*

Single Cell Cavity – TE1AES012

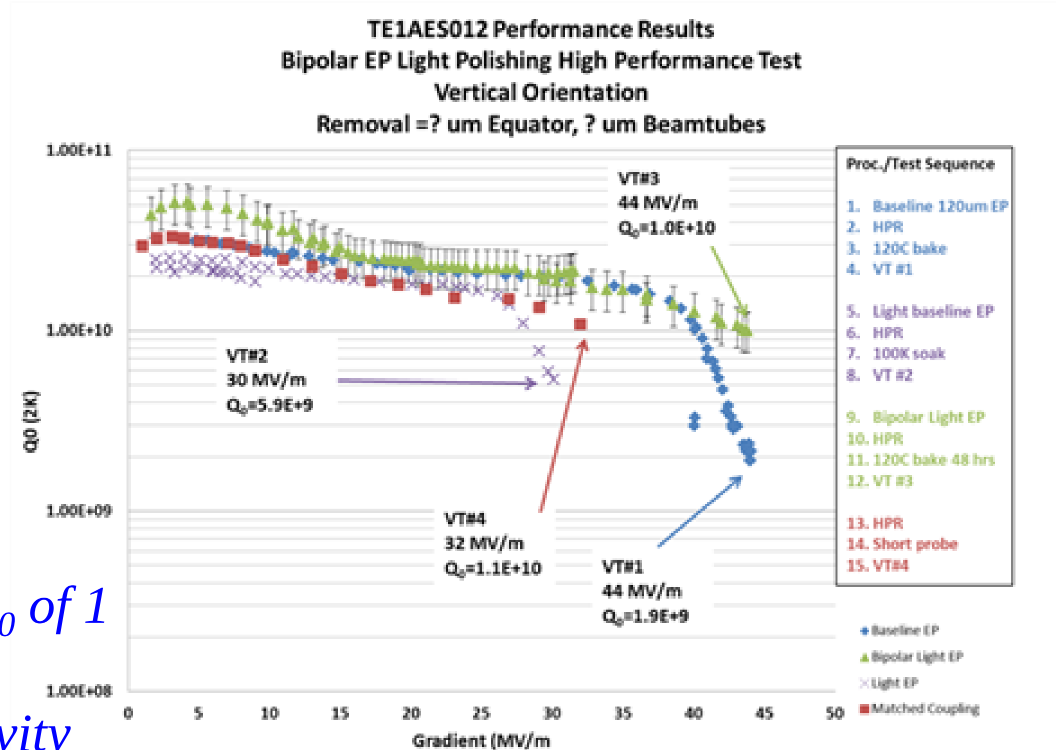
FARADAY TECHNOLOGY, INC.

"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 Hydroflouric 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.

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Commercialization: “Market Acceptance”

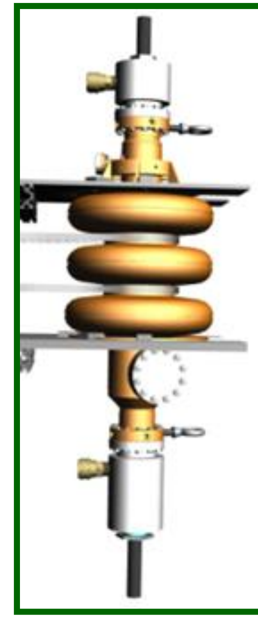
ORNL

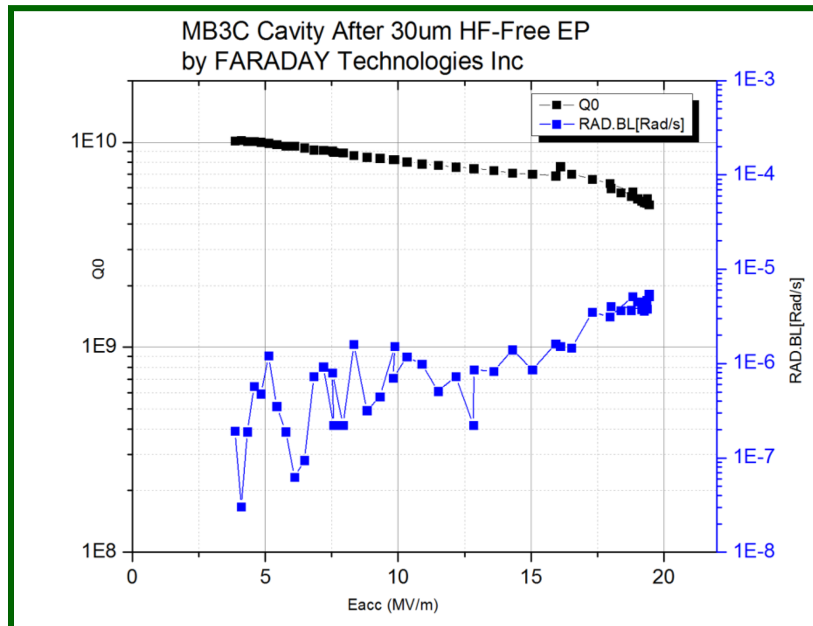
- P.O. for Nb coupons ($5K)
- P.O. for 3-cell Nb cavity electropolishing

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

- P.O. for EP apparatus ($80K)

Approximate height of 9-cell cavity



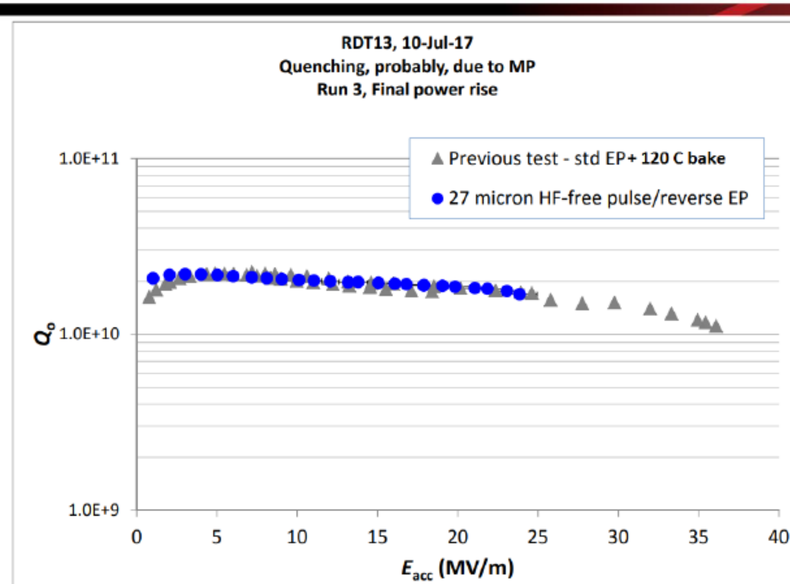


Commercialization: Jefferson Lab Activities

Jefferson National Accelerator Facility CRADA No. DE-AC05-06OR23177

- α-Scale (at Faraday) FARADAYIC® EP of “N_2 doped” single cell Nb Cavity
  - ➔ SUCCESS!
- β-Scale (at Jlab) FARADAYIC® EP of single cell Nb Cavity
  - ➔ SUCCESS!
- β-Scale (at Jlab) FARADAYIC® EP of single cell N_2 doped Nb Cavity
  - ➔ UNDERWAY -

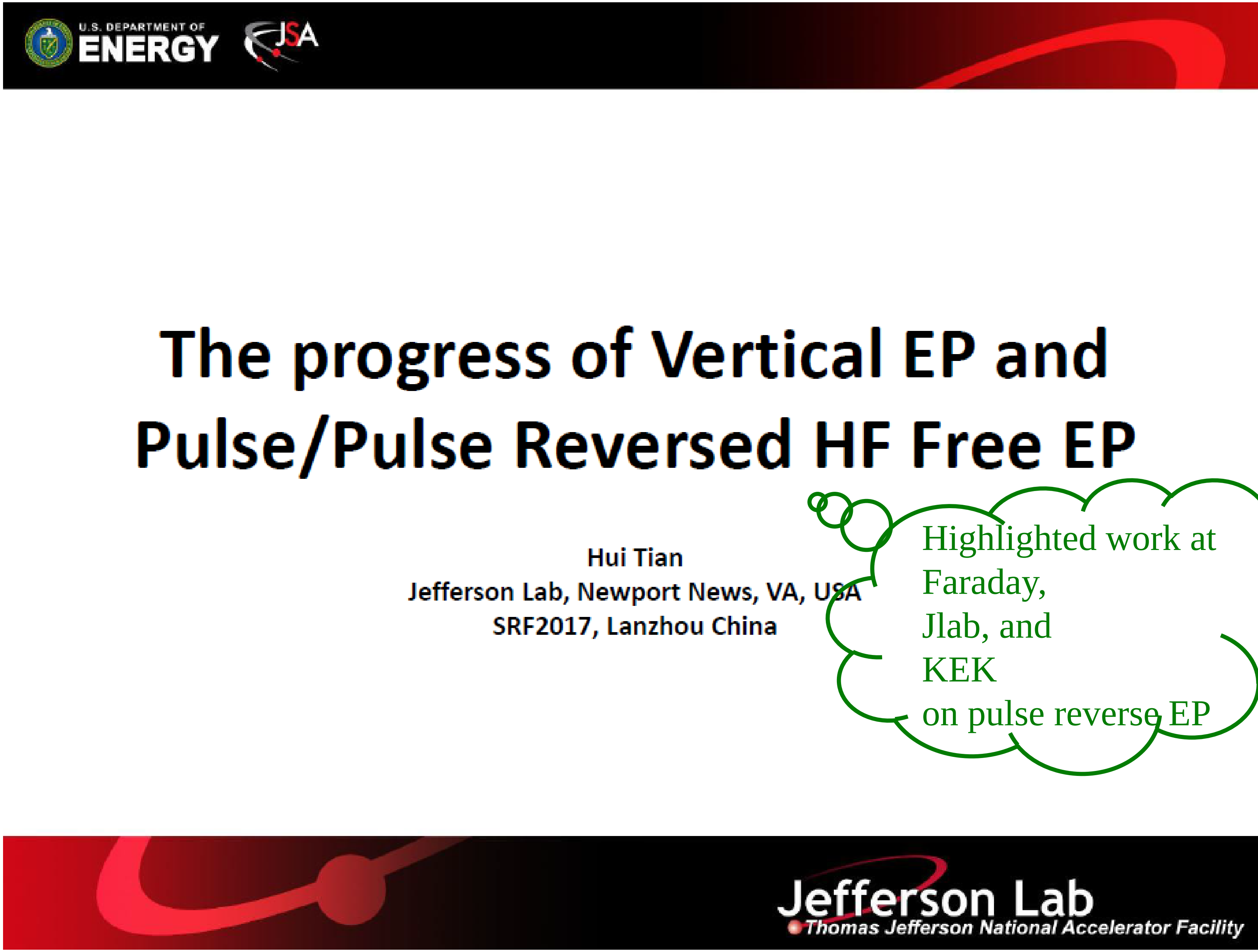
Nb SRF cavity process with pulse/pulse reversed EP at Jlab



The process o f N doped single cell and multi cell Nb cavity is underway for pulse/pulse reversed EP.
The parametric optimization is needed to explore ways towards higher Q_0 and higher yield with pulse/pulse reversed EP.

Jefferson Lab

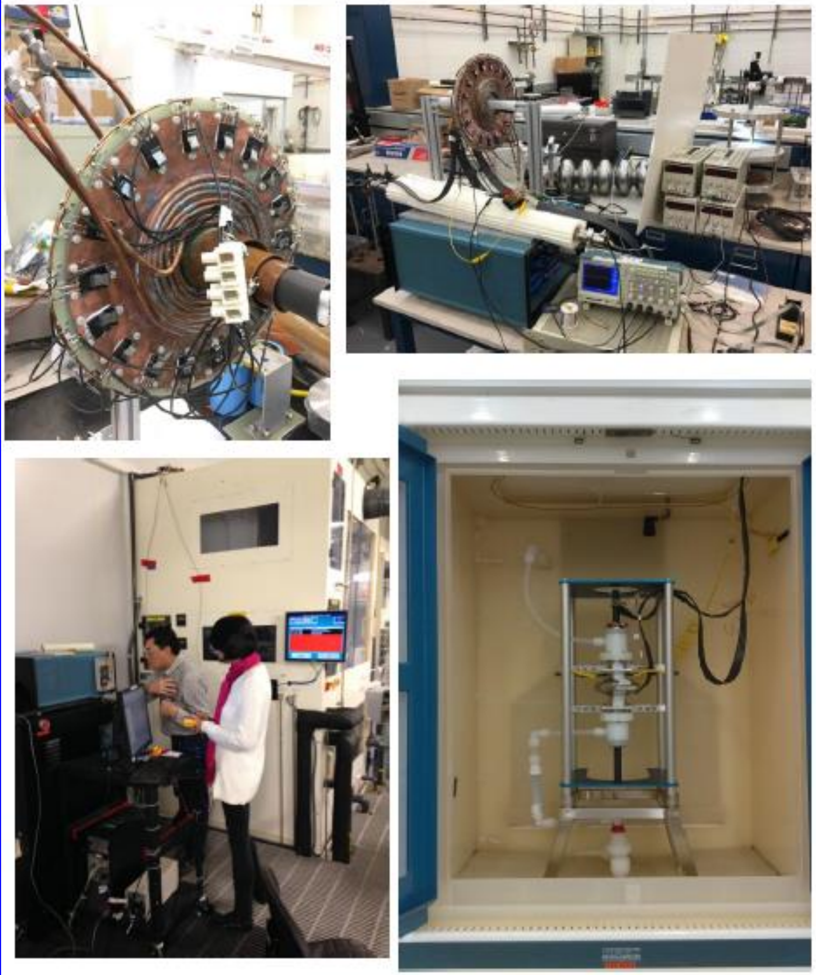
Commercialization: Jefferson Lab CRADA



Highlighted work at
Faraday,
Jlab, and
KEK
on pulse reverse EP

Commercialization: Jefferson Lab Activities

Jlab HF-free pulse/pulse reversed EP development



The pulse reversed EP process uses a custom designed IGBT driver pulse controller to drive two DC power supplies (AMETEK 40V, 250A) to provide the positive and negative pulses. Pulse/pulse reversed electropolishing for single/multicell Nb cavities conducts in the closed chemistry VEP cabinet .

The in-house pulse reversed EP process system demonstrates its capability for higher pulse current, longer pulse duration – multi-cell & different structure SRF Nb cavity .

A CEBAF shape single cell and a ILC shape single cell cavity have been processed by pulse/pulse reversed EP for VTA test.

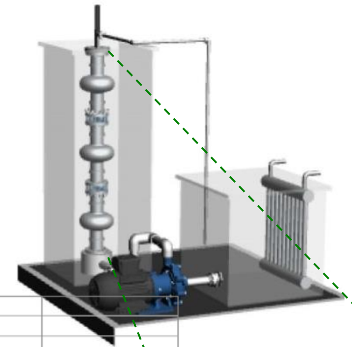
Focused on low-cost rectifier development

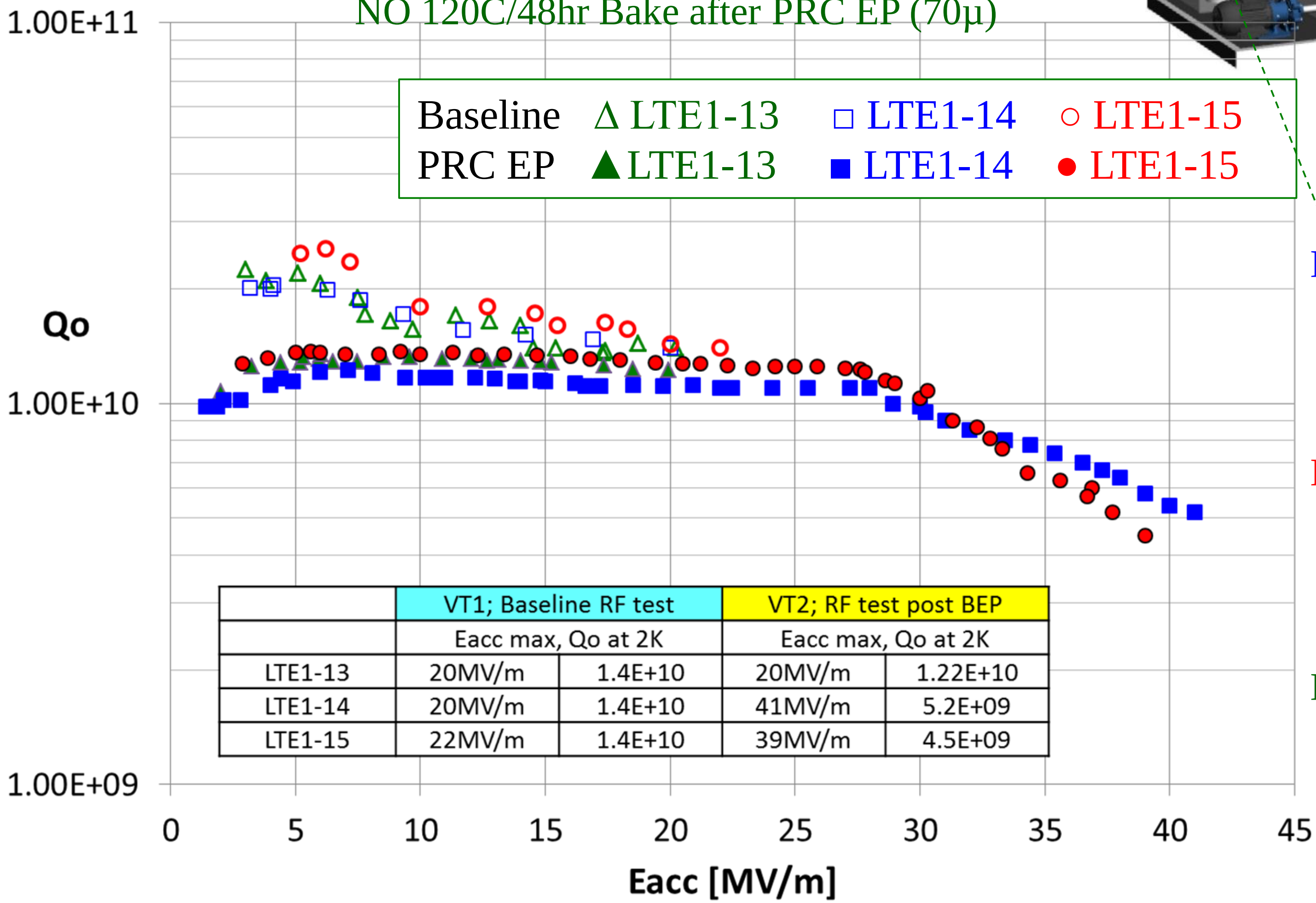
Simulate 9-Cell: 3 Stacked Cavities

FARADAY TECHNOLOGY, INC.

Three single-cell Nb SRF cavities

- Stacked with "spacers" to simulate nine-cell cavity height/flow effects: 9-cell 1250mm; stacked-cell 1200mm





	VT1; Baseline RF test		VT2; RF test post BEP	
	Eacc max, Qo at 2K		Eacc max, Qo at 2K	
LTE1-13	20MV/m	1.4E+10	20MV/m	1.22E+10
LTE1-14	20MV/m	1.4E+10	41MV/m	5.2E+09
LTE1-15	22MV/m	1.4E+10	39MV/m	4.5E+09

Commercialization: KEK Activities

Based on 3-cell stack result, KEK provided

- α-Scale (at Faraday) FARADAYIC® EP of Nine cell Nb Cavity
  - ➔ RESULTS PENDING

Study on Electro-Polishing of NB Surface By Periodic Reverse Current Method with Sodium Hydroxide Solution for Particle Accelerator Application

Highlighted work at Jlab, and KEK on pulse reverse EP

T. Saeki, H. Hayano (KEK),
J. Taguchi, K. Ishida, M. Umehara (Nomura Plating Co, Ltd),
C. E. Reece, and H. Tian (Jefferson National Lab)

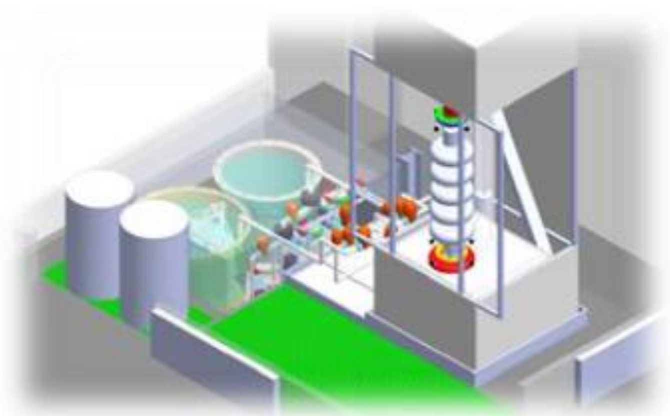
31 May 2017 / 231st ECS Meeting
Symposium: F05: Pulse and Pulse Reverse Electrolytic Processes
Hilton New Orleans Riverside in New Orleans / U.S.A.

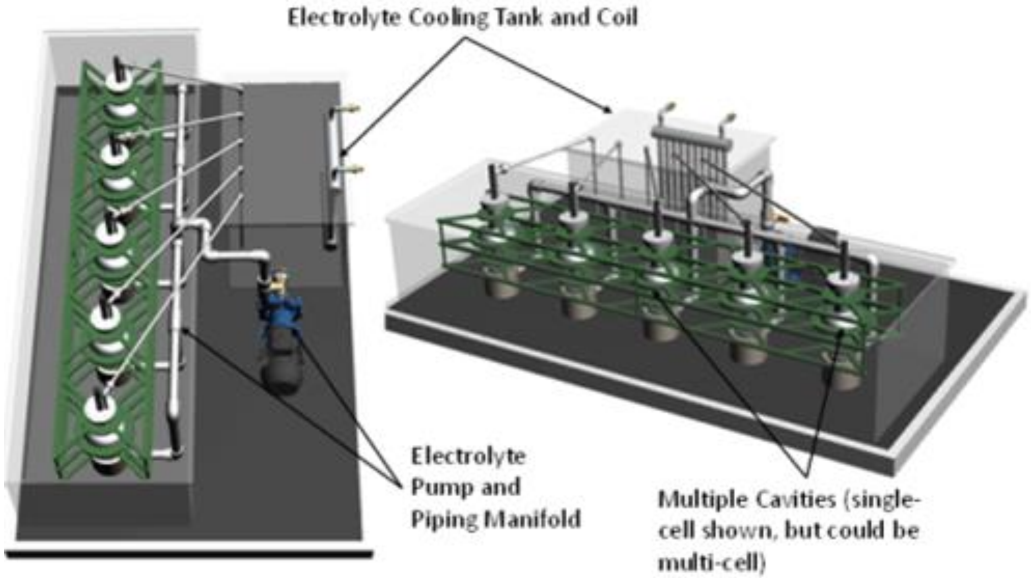
The Electrochemical Society
Advancing solid state & electrochemical science & technology

Commercialization: Economics

FARADAY TECHNOLOGY, INC.

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





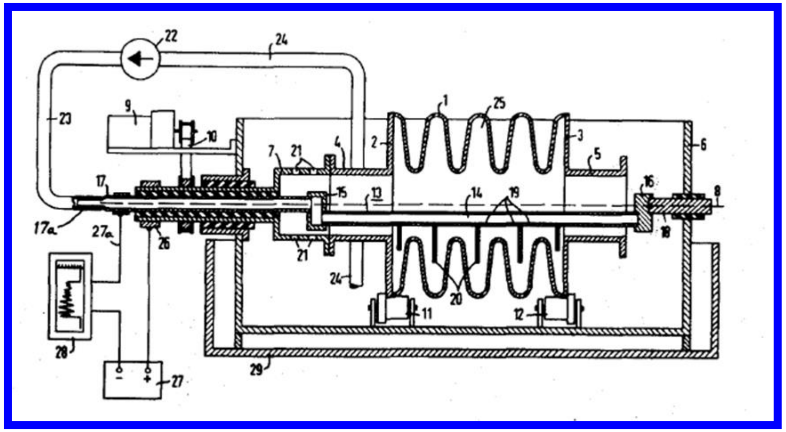
	Baseline EP	FARADAYIC EP
Electrolyte	9:1 H_2SO_4:HF	~5% (wt) H_2SO_4 in H_2O
Processing Voltage	DC: ~17 V	Pulse Reverse: ~3 V / 9 V
Processing Temperature	25°C	25°C
Cavity Orientation	Horizontal	Vertical
Electrolyte Volume Fill	60%	100%
Electrolyte Flow Rate	~8 L/min	~8 L/min
Cavity Rotation	1 rpm	No Rotation
Cathode Material/Shape	Aluminum/Tube	Mixed-Metal Oxide Coated Titanium/Rod
Material Removal Rate	0.2 µm/min	0.04 µm/min

	Baseline EP	FARADAYIC 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	✗	✓

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

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

Commercialization: IP Strategy



Prior Art (Seimens)

"Method for the electrolytic polishing of the insdie 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

- 1st U.S. patent issued, 2nd pending
- Japan & Europe patents issued
- FARADAYIC® registered
  - Trademark
  - Service mark

➔ Independent claim directed towards low viscosity!!

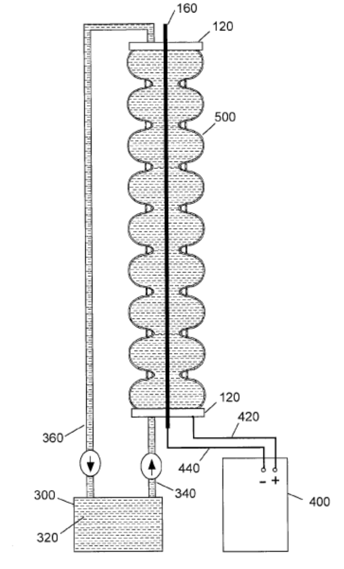


Fig. 12

"Electrochemical system and method for electropolishing superconductive radio frequency cavities"

- U.S. Patent No. 9,006,147 issued April 14, 2015
- Japan Patent No. 6,023,323 issued October 14th 2016
- Europe Patent No. 2,849,908 issued January 15, 2017

Low concentration – aqueous electrolyte (acid/alkaline)

- Vertical orientation
- Completely filled
- No rotation

➔ Industrially compatible

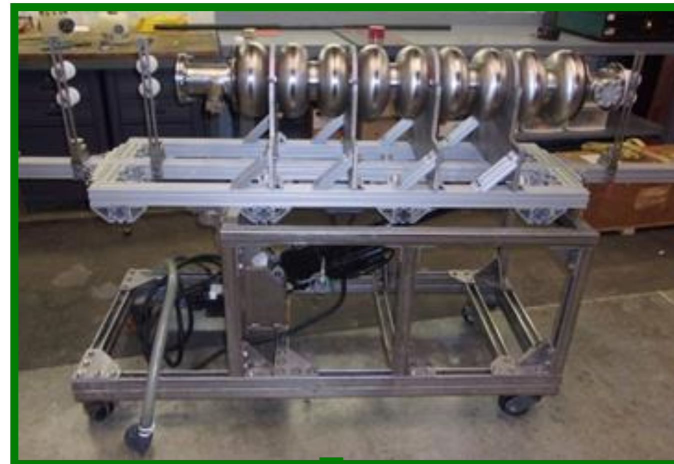
- Safe, Low CapEx and OpEx

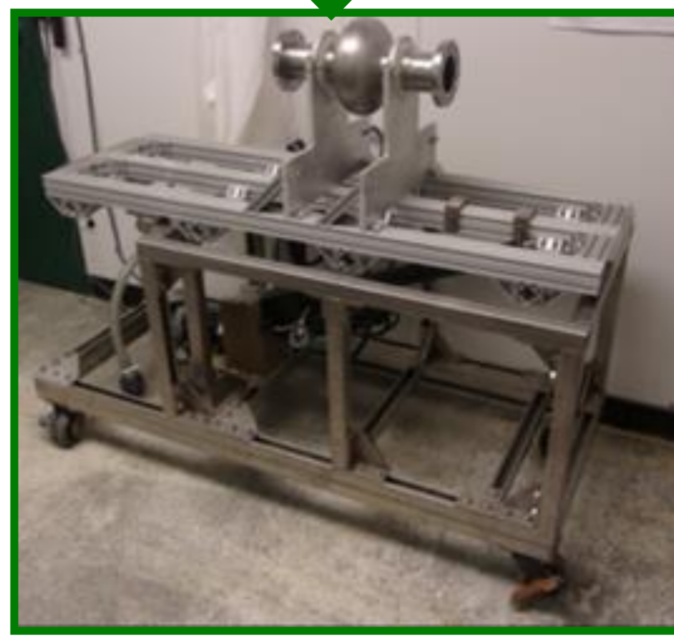
On-Going: Completely Acid-Free EP

1. COMPLETELY Acid-Free FARADAYIC® EP
2. High rate EP front-end bulk processing?

Next steps:

- EP in "button" cell cavity and single-cell SRF cavity
- Recommission apparatus from nine cell to single cell
    - Modify bracketing system
    - Drain & neutralize acid
    - Flush supply lines with D.I water
- Construct new external tank for salt solution





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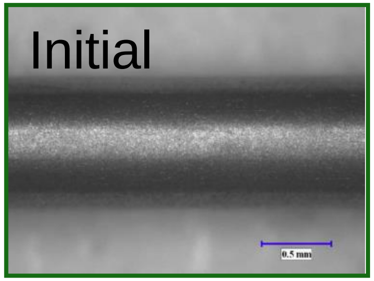
Collaborators:

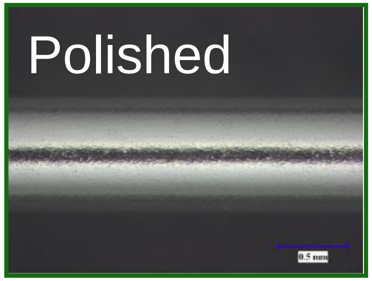
1) Dr. Fumio Furuta and Dr. Geoff Hoffstaetter; Cornell University
2) Dr. Hui Tian, Dr. Charles Reece and Dr. Larry Phillips; Jefferson Lab
3) Dr. John Mammosser and Dr. Jeff Saunders; Oak Ridge National Laboratory
4) Dr. Takayuki Saeki; KEK High Energy Accelerator Research Organization
5) Mr. Allan Rowe and Dr. Anna Grassellino; Fermi Laboratory

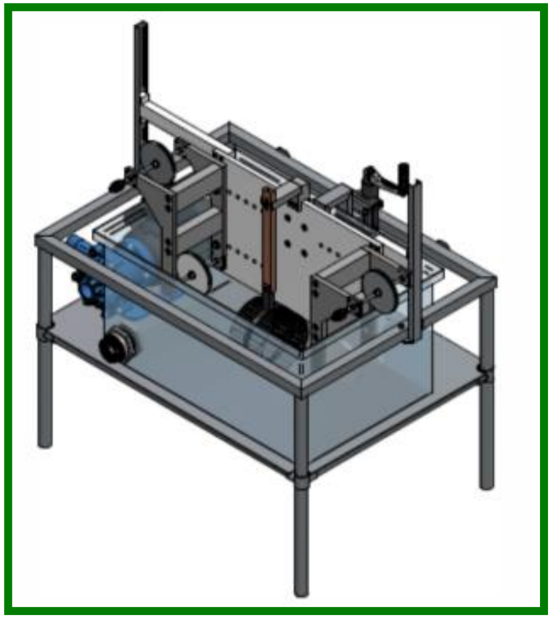
Commercialization: Spin-off

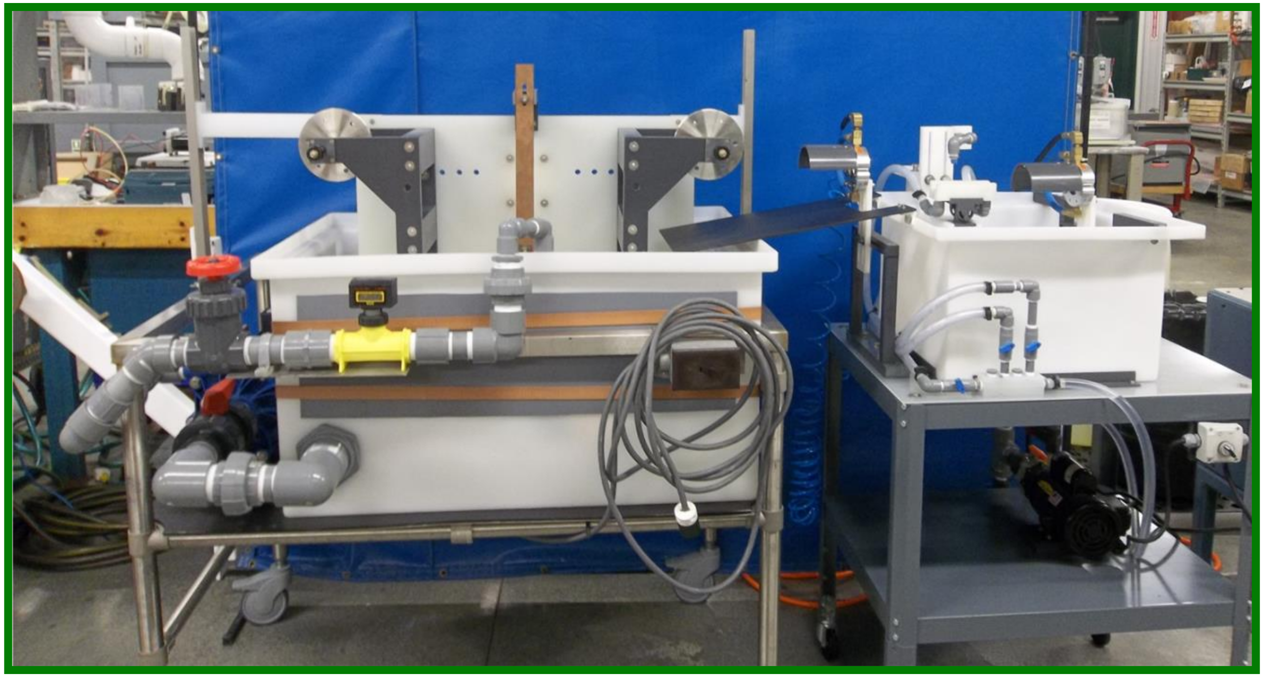
FARADAYIC® ElectroPolishing of Nitinol medical stents (similarities to Nb)

- Received Phase I & II funding from NIH ➔ process validation
- Project funding from OEM for adaptation to wire
  - α-scale reel-to-reel 300 foot spool trials
- TERM SHEET completed for FoU license
  - Market – Medical
  - Product – Wire based stent/shape sets
  - Material - Nitinol
- LICENSED 4-12-2016









FARADAY TECHNOLOGY, INC.

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 April 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 Fluorine 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 “Elecctropolishing 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 Elecctropolishing 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 Eecctropolishing 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 “Elecctropolishing 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 scheduled Fall (2017).