



FARADAY 
TECHNOLOGY, INC.

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

**Acid-Free Electropolishing of SRF Cavities
NP Phase II Grant # DE-SC0011235**

**Faraday Technology, Inc.
Cornell University**

Maria Inman, PhD
P.I.

Tim Hall, PhD
Project Lead

E. J. Taylor, PhD
Founder & Chief Technology Officer

Faraday Technology, Inc.

August 9, 2016

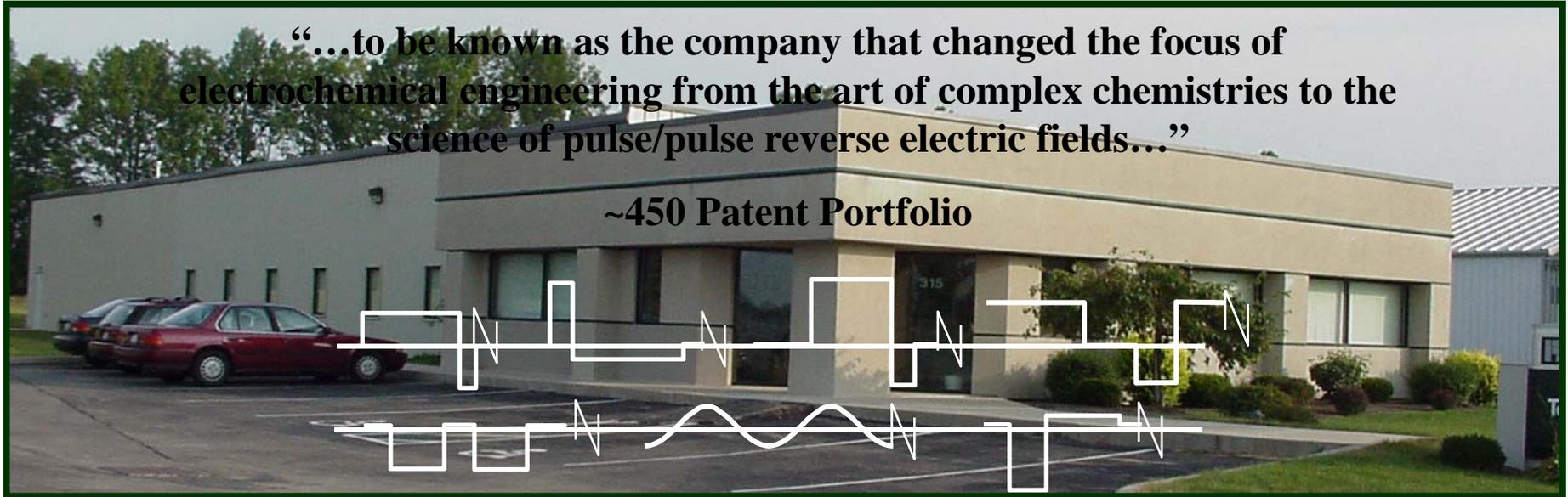


U.S. DEPARTMENT OF
ENERGY

Office of
Science

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TimHall@FaradayTechnology.com
JenningsTaylor@FaradayTechnology.com

Company Overview: FARADAY TECHNOLOGY, INC.



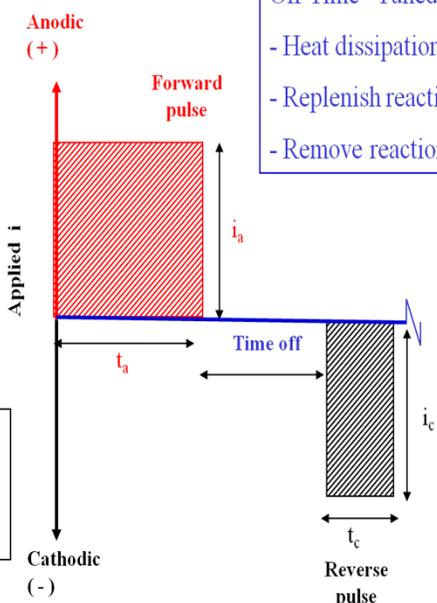
- Electrochemical engineering processes and technologies – founded 1991
 - ~29 Issued Patents and ~15 Pending Patents in this area
 - www.FaradayTechnology.com
- Subsidiary of Physical Sciences, Inc. (Boston, MA) – acquired 2008
 - www.psicorp.com
- Collective employment ~140; ~90 MS/PhD/Annual revenue of ~ \$40 million

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

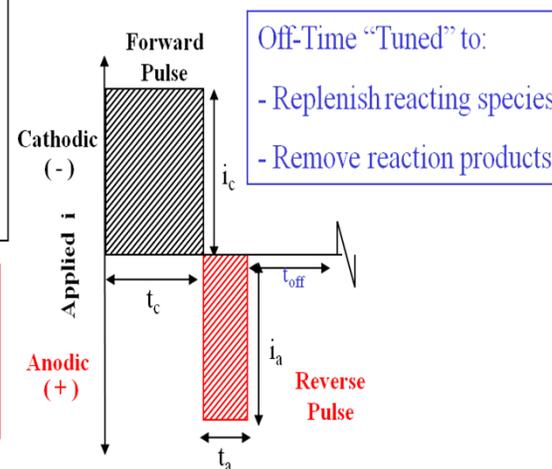
Cathodic Pulse “Tuned” to:
- Reduce oxide/depassivate surface
→ Eliminate need for HF

- 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

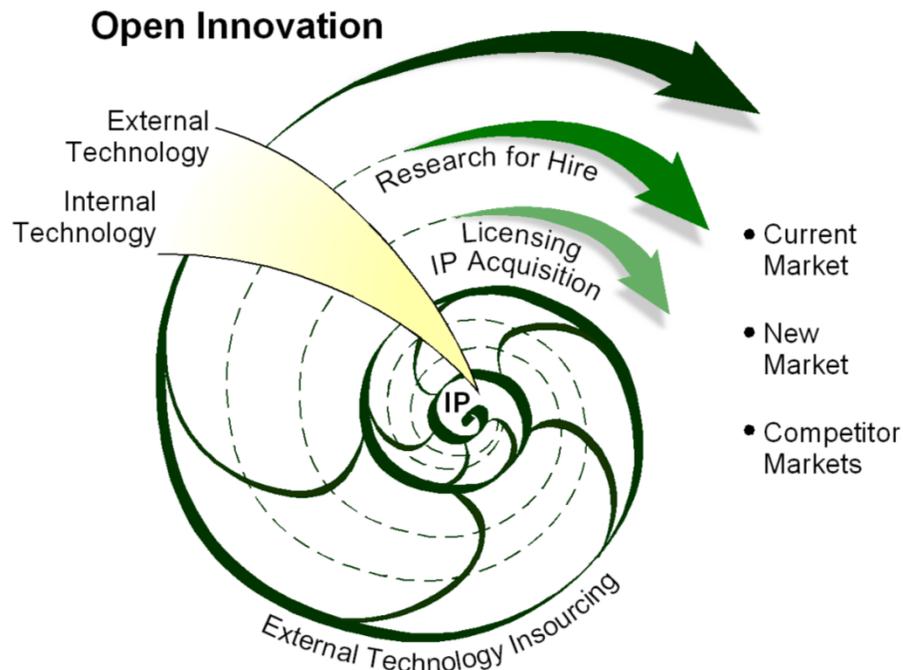
Anodic Pulse “Tuned” to:
- Remove H₂ effects
→ Acidify interface



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

Business Model: Open Innovation



- Establish IP (29 patents issued/know-how)
- Leverage Federal SBIR opportunities as non-equity technology funding
 - Retain IP rights
- Collaborate with universities and government laboratories
- Develop electrochemical engineering solutions based on PC/PRC processes
- Transition technology & competitive advantage to large companies via
 - Field-of-use licenses
 - Patent acquisition (8)

Development of robust process is critical!

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;

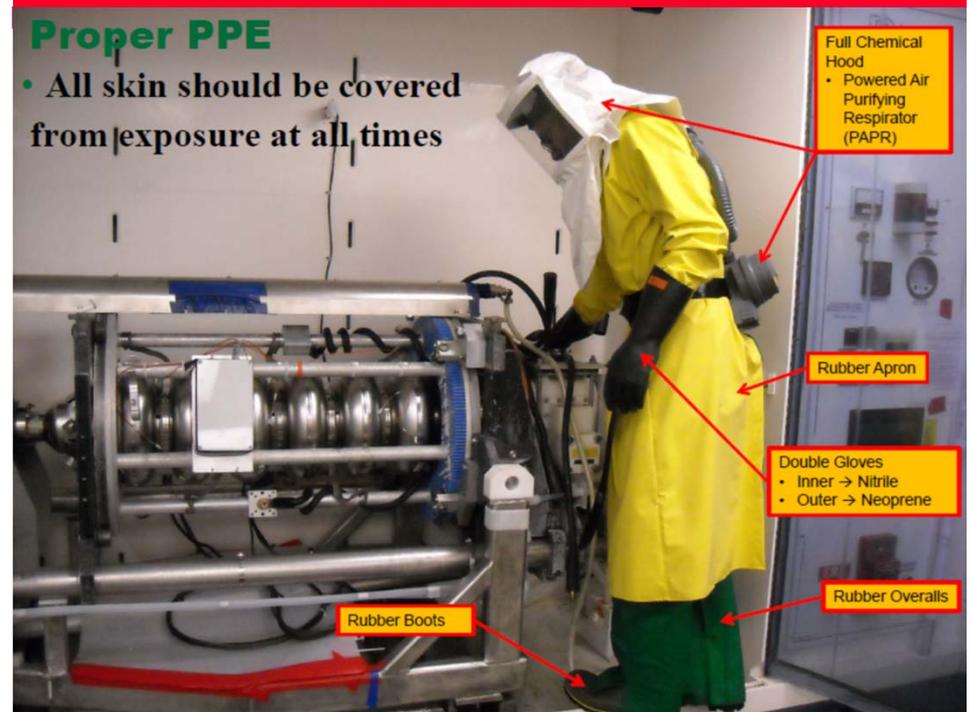
**9:1 H₂SO₄ (98%) : HF(48%)
electrolyte (DC)**

HF → Safety/Cost Burden

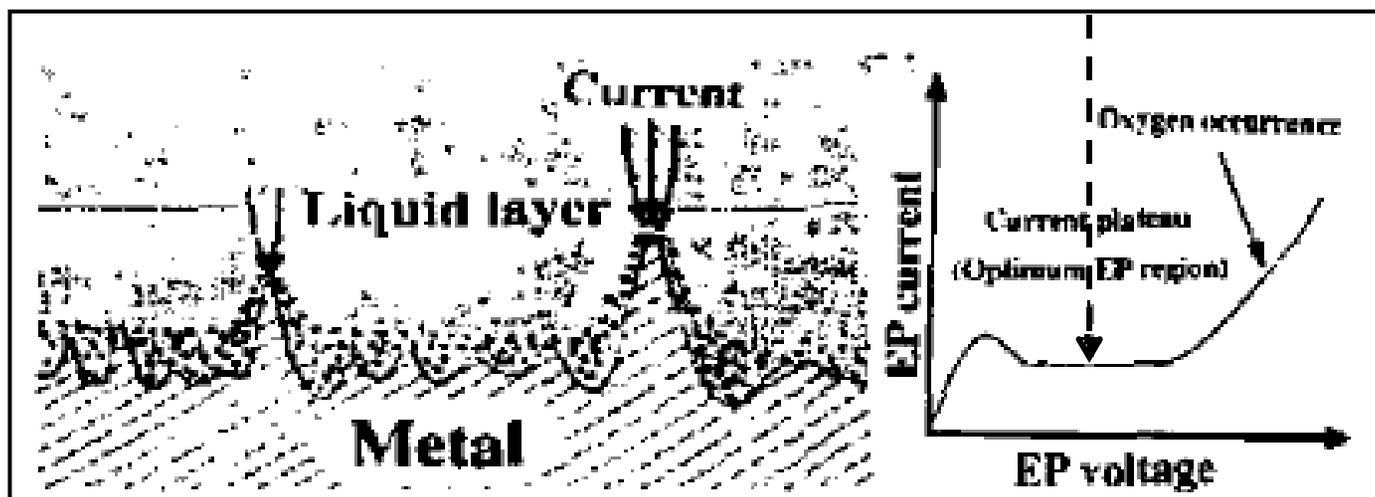
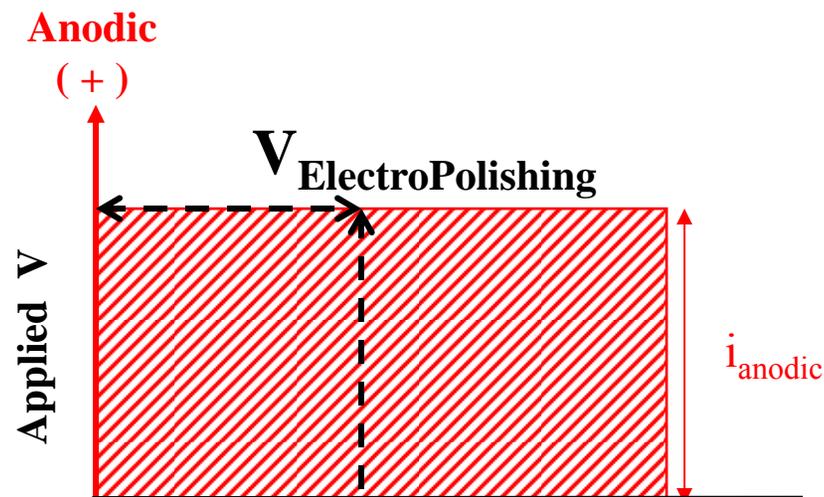
“viscous salt film” paradigm

Personal Protective Equipment (PPE) for “conventional” SRF niobium cavity electropolishing using sulfuric acid – hydrofluoric acid mixture.

John Mammoser, Instructor
“Chemical Safety for SRF Work”
U.S. Particle Accelerator School
January 2015



- Rectification:
 - DC - Constant voltage
 - Viscous salt film per Jacquet
 - Passive (oxide) film formation
- Electrolyte:
 - Concentrated/viscous acid
 - Chilled to increase viscosity
 - Chemical addition to remove oxide



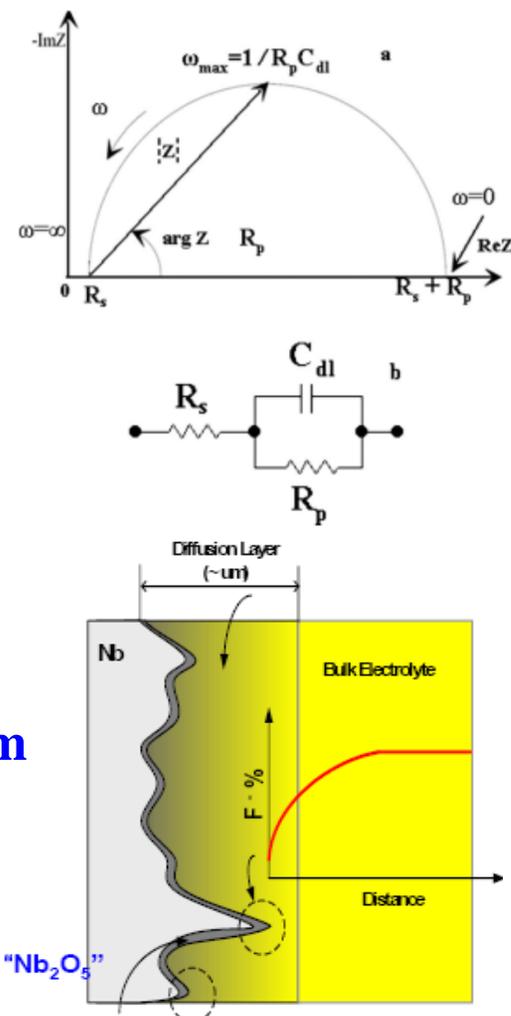
† P.A. Jacquet, *Trans. Electrochem. Soc.*, **69** 629 (1936).

- Nb EP for SRF “Cavities” → Nb_2O_5
- Soluble niobium fluorides and niobium oxyfluorides[†]:



- Recent EIS studies consistent with **EP (Jacquet) paradigm**

→ Diffusion limited F^- to a compact “salt” (oxide) film^{††}

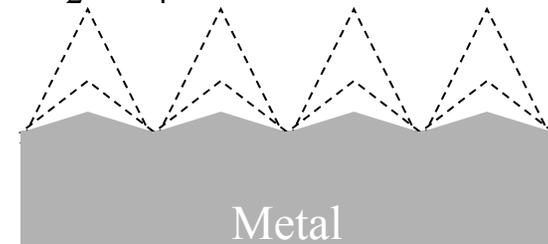


[†]P. Kneisel, “High Gradient Superconducting Niobium Cavities: A Review of the Present Status” *IEEE Trans. Appl. Superconductivity*, 9(2) 1023-1029 (1999).

^{††}H. Tian, C. Corcoran, C. Reece, M. Kelly “The Mechanism of Electropolishing of Niobium in Hydrofluoric-Sulfuric Acid Electrolyte” *J. Electrochem. Soc.* 155(9) D563-568 (2008).



- 1st Issue: Focusing current on asperities – Viscous H₂SO₄



- 2nd Issue: Polishing oxide forming materials:



→ Chemically dissolve the niobium oxide - HF

Other approaches:

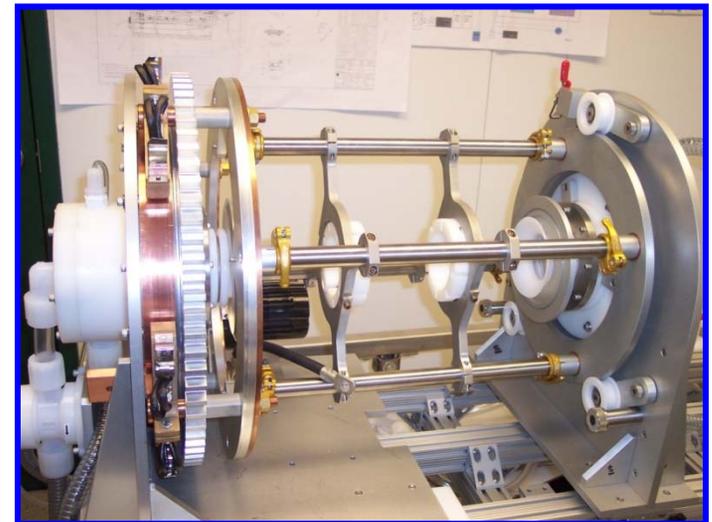
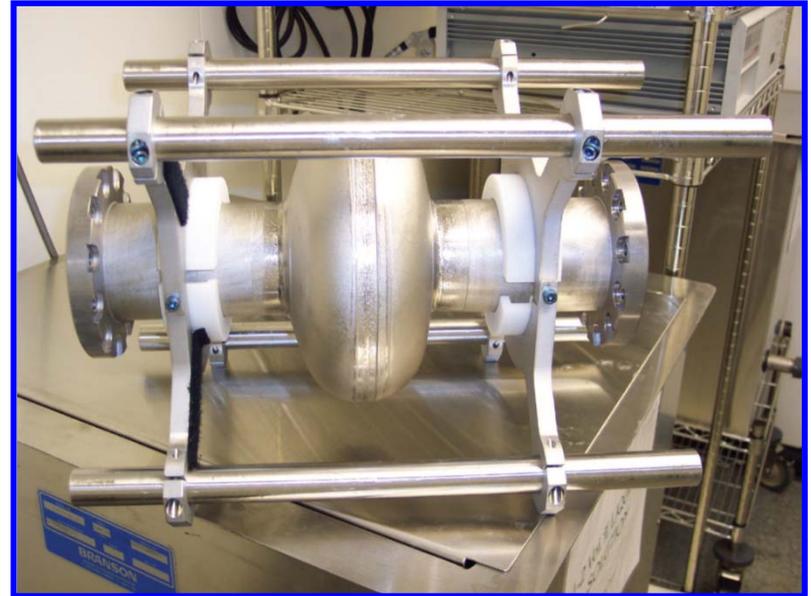
1. Non-aqueous electrolytes[†] with < 5% H₂O
 - Limited industrial implementation (NiTi stents)
2. Ionic Liquids

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

EP is slow, therefore generally two step process:

1. Bulk: Buffered chemical polishing
 - $\sim 100\mu\text{m}$ removal at $0.5\text{-}1\mu\text{m}/\text{min}$
 - $R_a \sim 2\mu\text{m}$
 - 1:1:2 $\text{HNO}_3(69\%):\text{HF}(49%):\text{H}_3\text{PO}_4(85\%)$
 2. Final: Electropolishing
 - 17V (cell voltage)
 - $\sim 25\mu\text{m}$ removal at $\sim 0.1\text{-}0.3\mu\text{m}/\text{min}$
 - $R_a < 0.2\mu\text{m}$
 - 9 parts $\text{H}_2\text{SO}_4(96\%)$ to 1 part $\text{HF}(49\%)$
 - Process conditions
 - ✓ Horizontal orientation
 - ✓ Partially electrolyte filled (60%)
 - ✓ Rotation
- ➔ Stringent safety protocols
- ➔ Reject rate associated in part with EP?

1.3 GHz Single Cell



Anodic Pulse “Tuned” to:

- Focus current distribution

➔ Eliminates need for viscous electrolytes

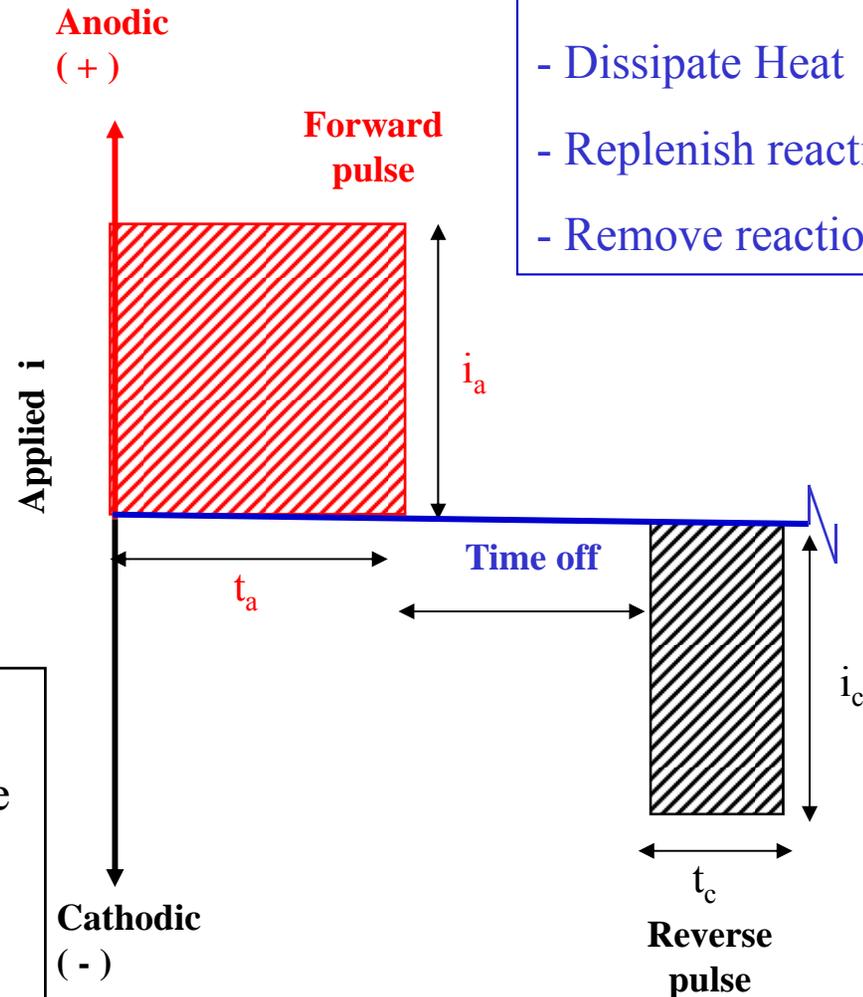
Cathodic Pulse “Tuned” to:

- Depassivate surface/remove oxide

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

Off-Time “Tuned” to:

- Dissipate Heat
- Replenish reacting species
- Remove reaction products

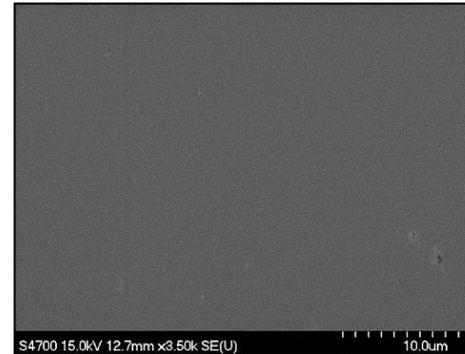


†E.J. Taylor “Adventures in Pulse/Pulse Reverse Electrolytic Processes: Explorations and Applications in Surface Finishing” *J. Appl. Sur. Fin.* 3(4) 178-89 (2008).

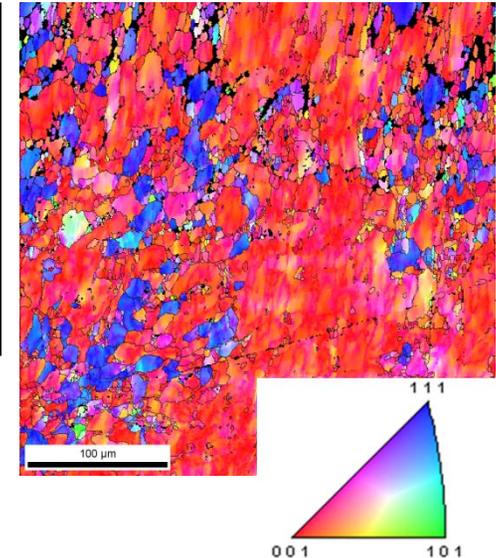


- Pulse/Pulse Reverse - coupons
 - Aqueous H₂SO₄ electrolyte (5 to 30%)
 - Strong Passive film
 - ➔ Forward (anodic) pulses
 - ➔ Reverse (cathodic) pulses
 - Fast Waveforms
 - Bulk removal (100μm)
 - ~0.5-1μm/min
 - Slower Waveforms
 - Final EP (25μm)
 - ~0.03-0.3μm/min
 - R_a < 0.05μm (stylus)
 - Extremely clean surface

SEM Image



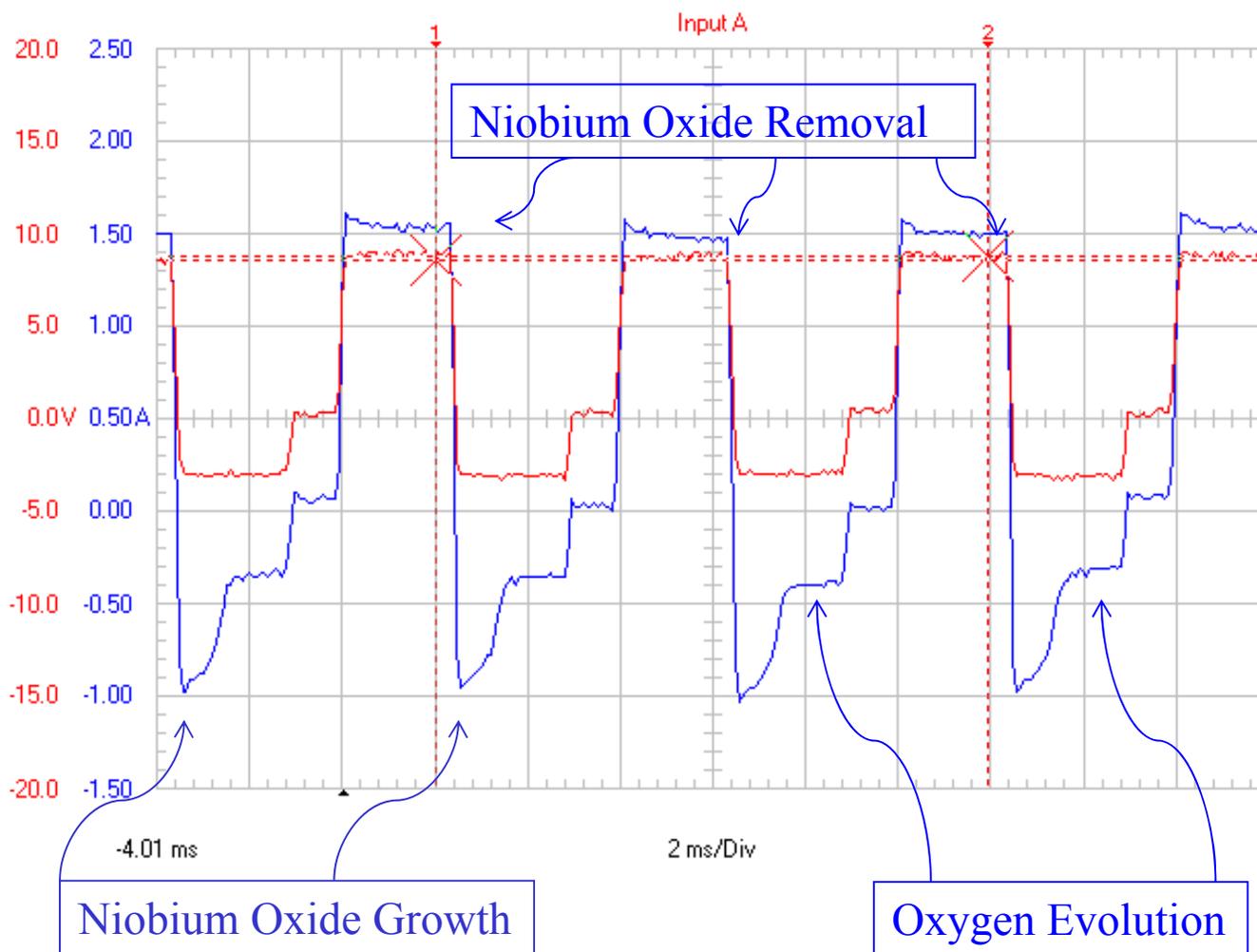
EBSD Profile



Dr. C. Reece, T. Jefferson lab

“...comparable to standard HF EP...”

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



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



- ➔ **Transition from oxide formation to oxygen evolution?**
- ➔ **Anodic current transition correlated with effective EP**

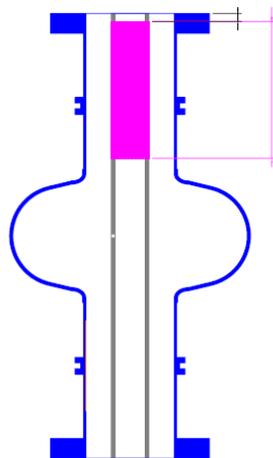
M. Inman, E.J. Taylor, T.D. Hall "Electropolishing of Passive Materials in HF-Free Low Viscosity Aqueous Electrolytes" *J. Electrochem. Soc.*, **160**(9) E94-E98 (2013).



Increased Waveform Timing to Observe Anodic Current Transition†

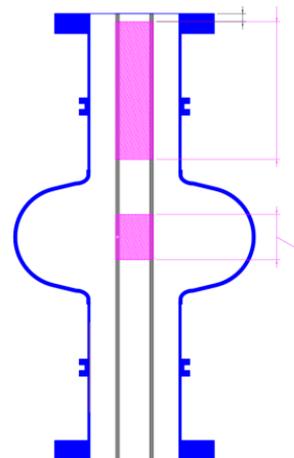


Trial 12



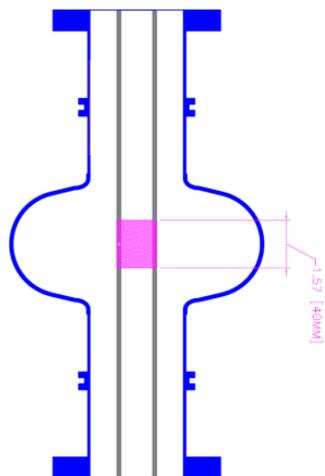
Waveform:
Anodic: 4 V 20 ms
Off: 30 ms
Cathodic: 10 V 10 ms

Trial 14



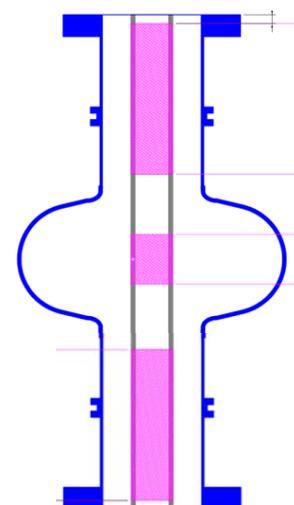
Waveform:
Anodic: 4 V 140 ms
Off: 175 ms
Cathodic: 10 V 100 ms

Trial 13



Waveform:
Anodic: 4 V 140 ms
Off: 175 ms
Cathodic: 10 V 100 ms

Trial 15



Waveform:
Anodic: 4 V 140 ms
Off: 175 ms
Cathodic: 10 V 100 ms

†T. Hall, M. Inman, E.J. Taylor “Electropolishing of Passive Materials in HF-Free Low Viscosity Aqueous Electrolytes: Part II From Coupons to Cavities” *J. Electrochem. Soc.*, in preparation (2016).

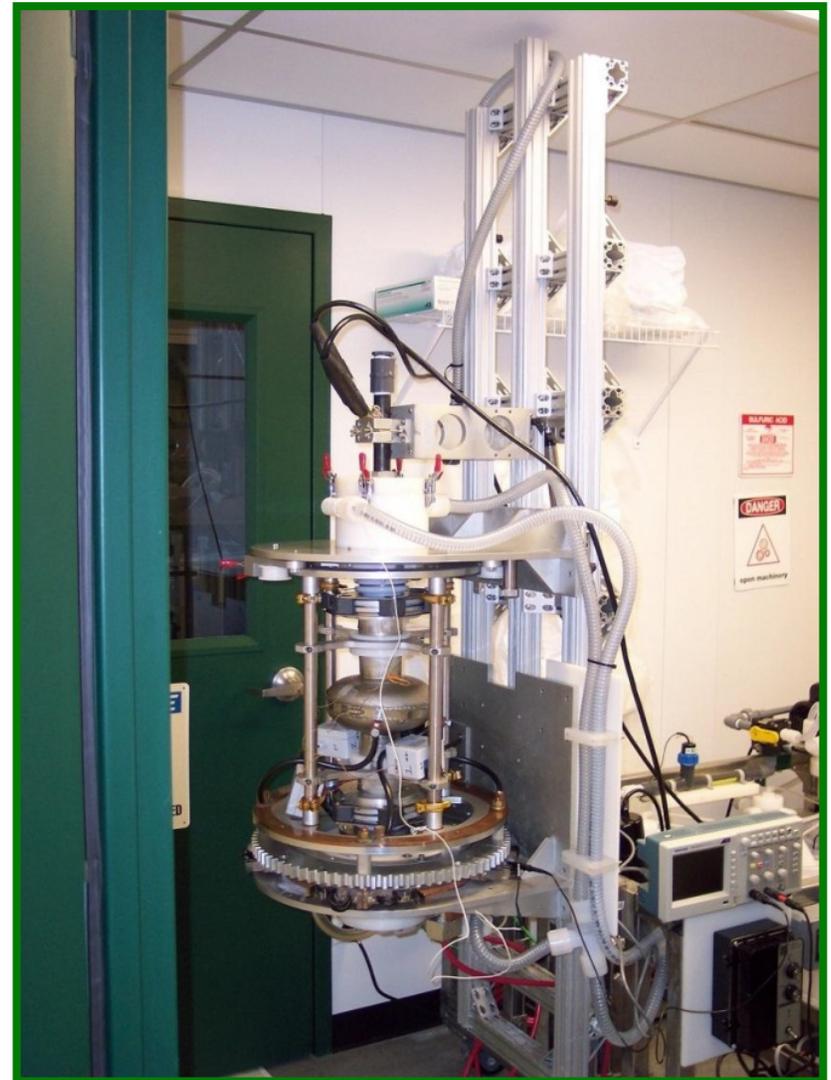


1.3 GHz Single Cell

Subsequent 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



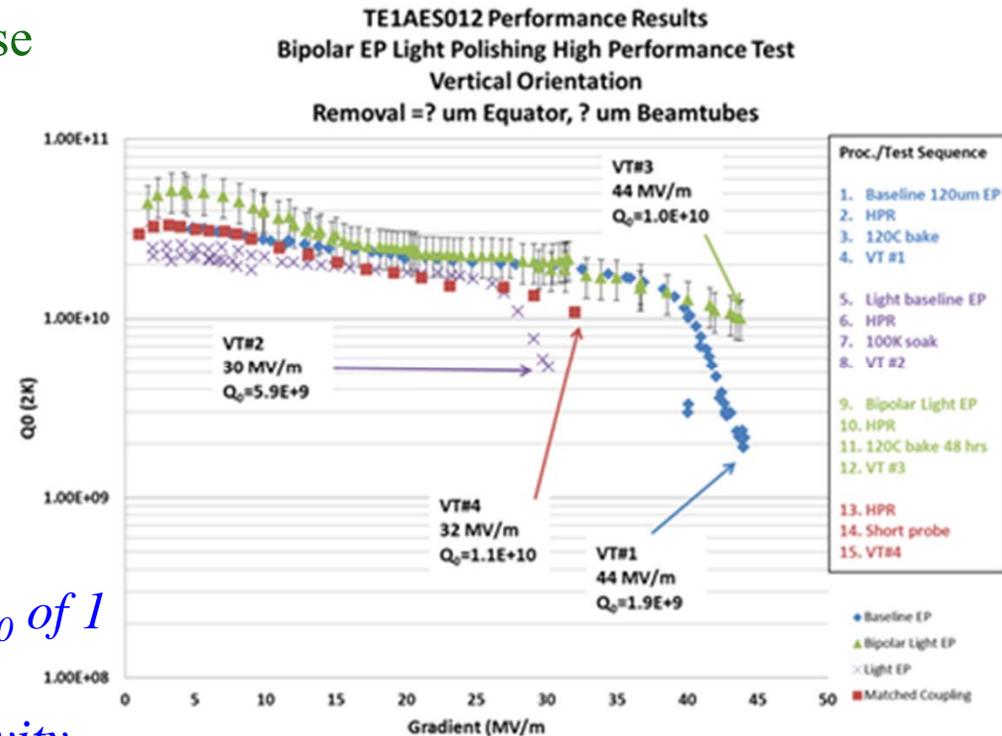


1.3 GHz Single Cell

FARADAYIC[®] HF-FREE EP (Pulse Reverse Current)

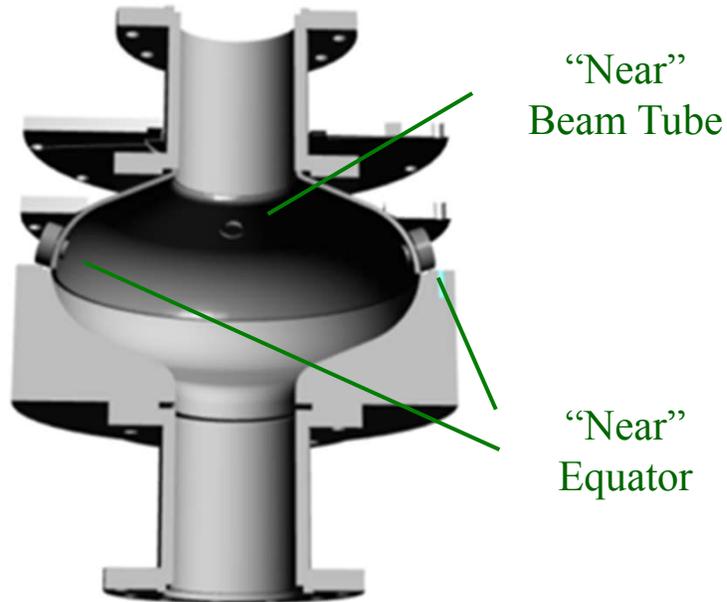
- Vertical
- 100% Volume Fill
- No Rotation
- 10 wt% H₂SO₄ in H₂O

➔ 25 μm removed “light EP”
 ➔ Cavity achieved a maximum gradient of ~44 MV/m with a Q₀ of 1 X 10¹⁰, 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 Fluorine in a Water Based Electrolyte” Paper No. TUIOC02, Presented SRF2013, Paris, FRANCE, 2013.



Removal Rate:
Cavity half-cell facing downward
"Near" Beam Tube 1.79 $\mu\text{m}/\text{h}$
"Near" Equator 1.25 $\mu\text{m}/\text{h}$
Ratio: 1.43

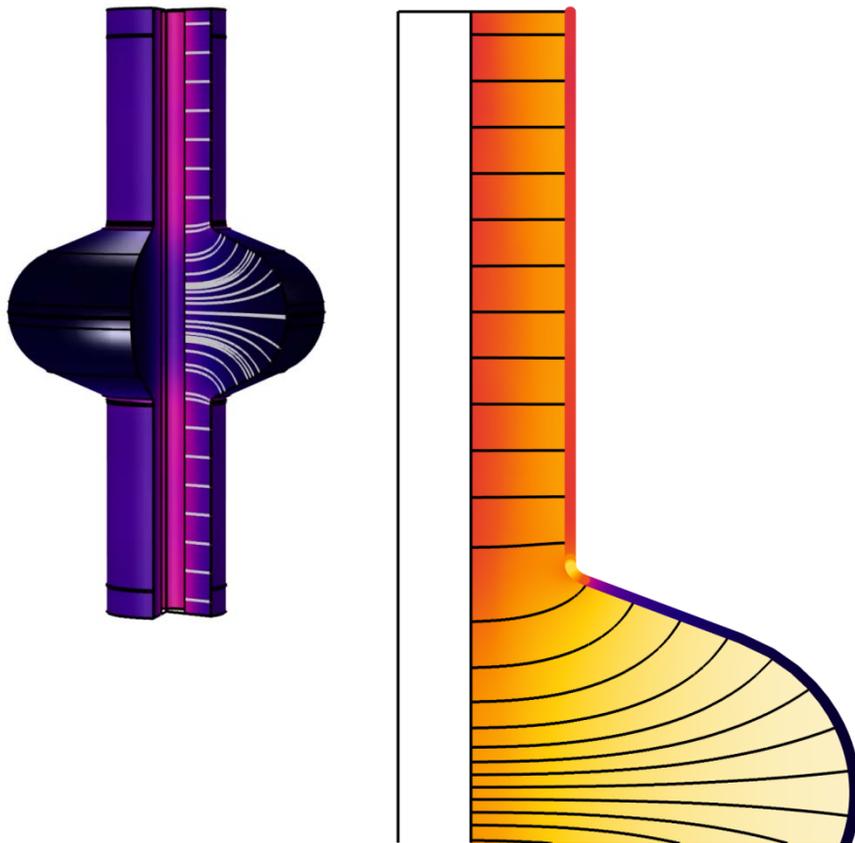
Conventional $\text{H}_2\text{SO}_4/\text{HF}$ EP
Beam Tube to Equator
Ratio: 1.5



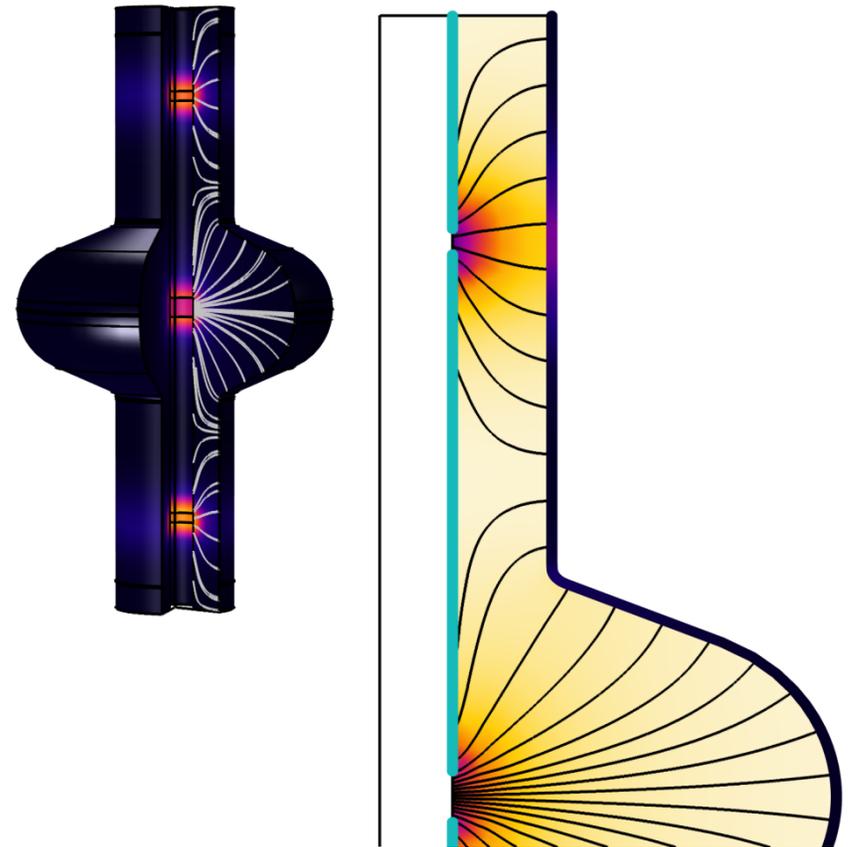
Removal Rate:
Cavity half-cell facing upward
"Near" Beam Tube 1.39 $\mu\text{m}/\text{h}$
"Near" Equator 1.09 $\mu\text{m}/\text{h}$
Ratio: 1.28



Un-masked Cathode Tool



Masked Cathode Tool

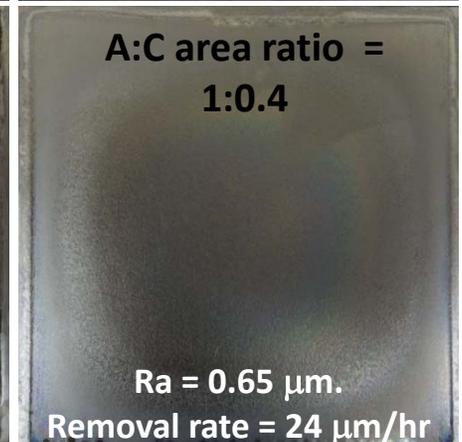
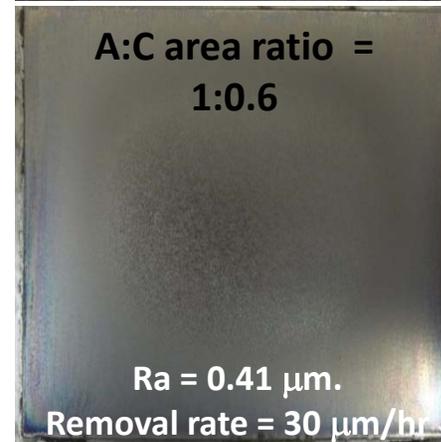
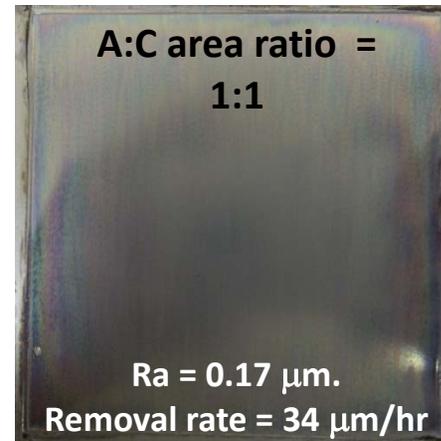
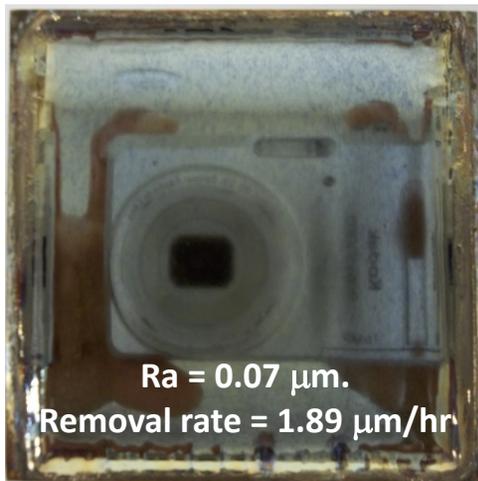


→ improvement in “primary” current distribution; ~1.1:1 ←



KCl/KH₂PO₄ pH ~7

Buffered NaNO₃ pH ~6



Potential for

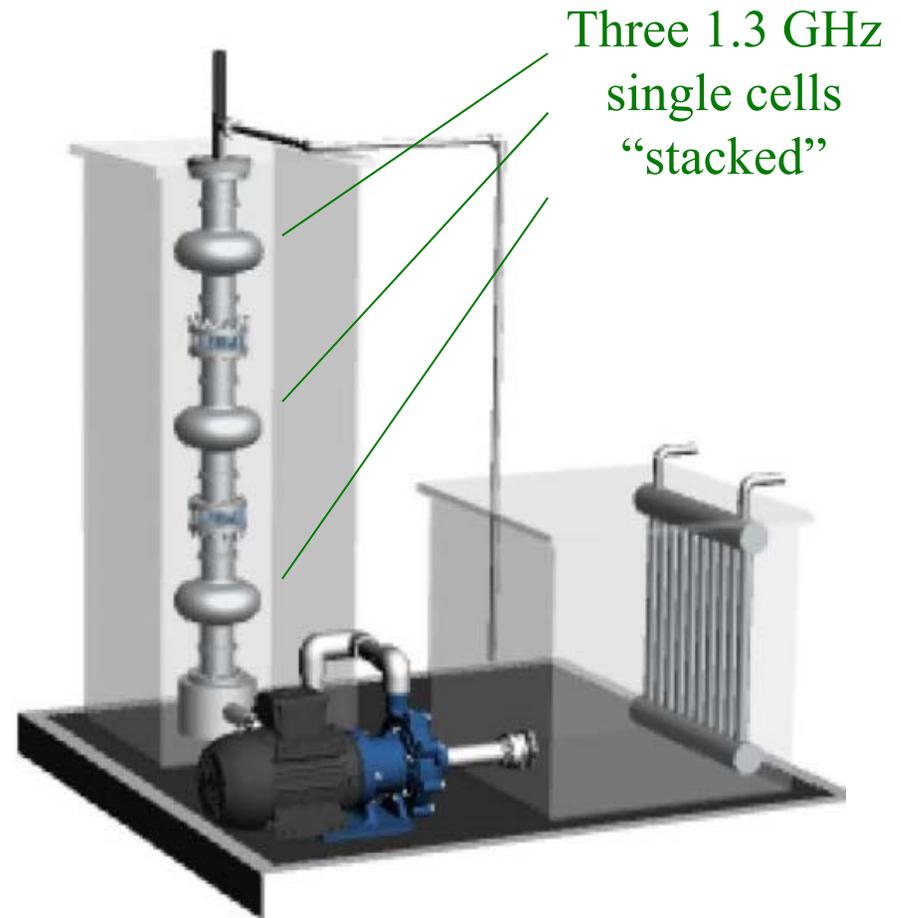
1. COMPLETELY Acid-Free FARADAYIC[®] EP
2. High rate EP front-end bulk processing

Next steps: EP in “button” cell cavity and single-cell SRF cavity



- Three single-cell Nb SRF cavities
 - Characterized/supplied by Cornell
 - Stacked with “spacers” to simulate nine-cell cavity height/flow effects
 - After processing by Faraday, returned to Cornell for characterization
 - Anticipated receipt of cavities by September 15th 2016

Note: processing of three-cell Nb cavity from ORNL provides indication of success?



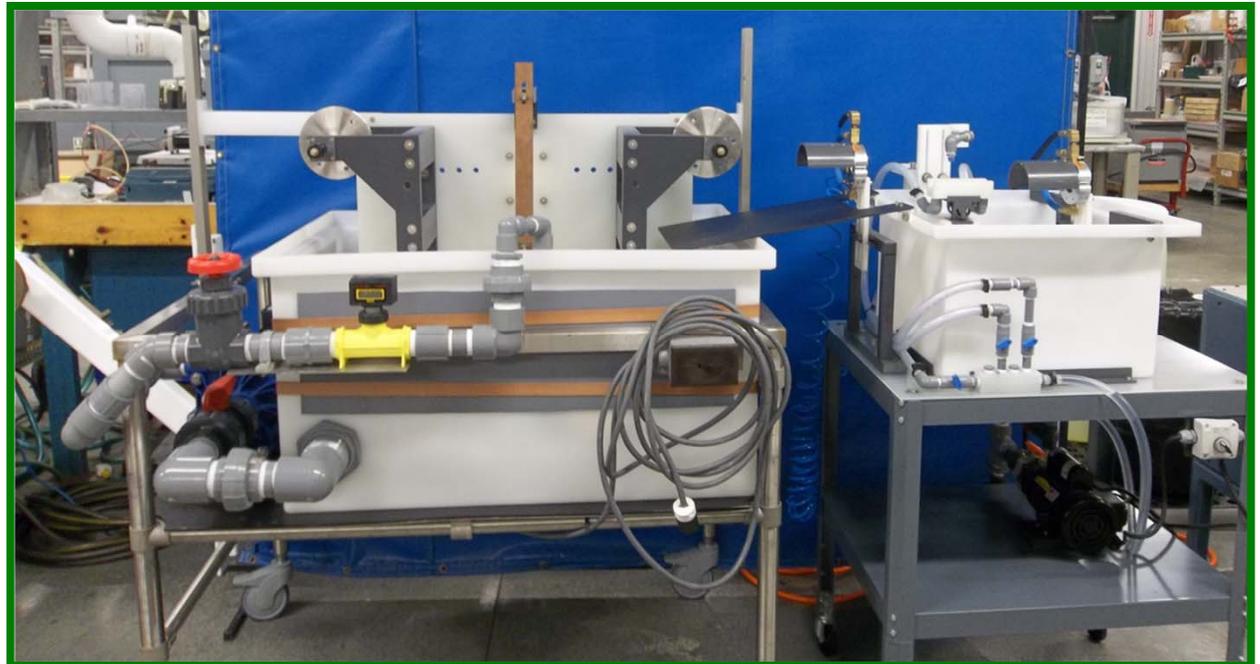
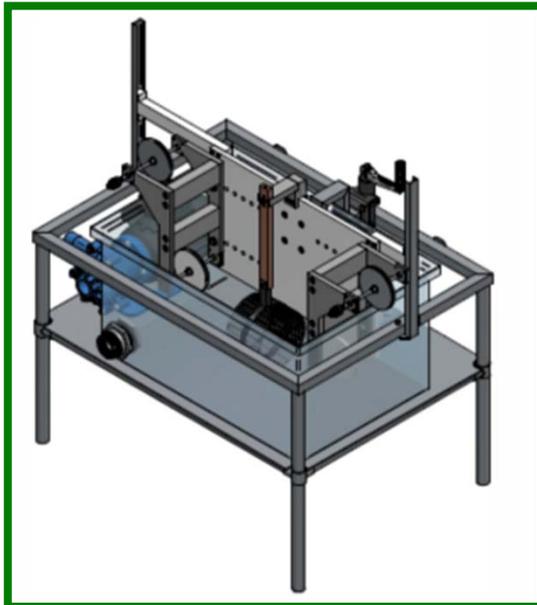
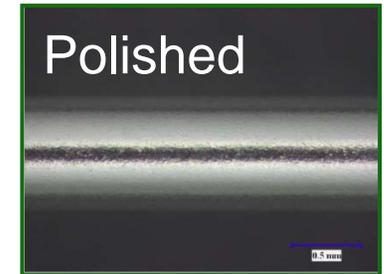
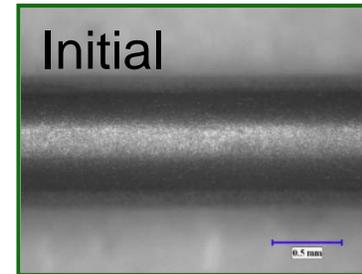
Objective: Demonstrate FARADAYIC[®] HF-FREE EP applicability to nine-cell cavities.

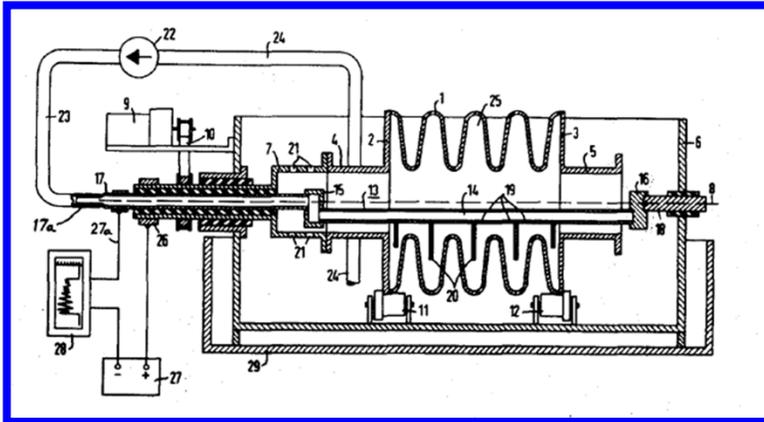
Next step: Process optimization

1. Uniformity
2. Speed of material removal

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





Prior Art (Siemens)

Roth, O. Schmidt “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

- Horizontal orientation
- Partially filled
- Rotation

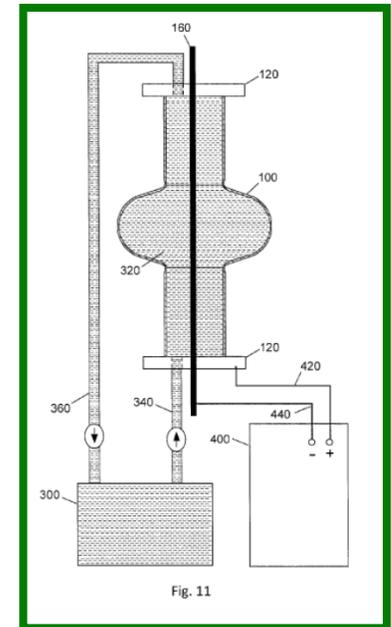
➔ Challenge for industrialization

1. Electrolyte safety
2. High capital and operating costs

Intellectual Property Portfolio

- 1st U.S. patent issued
- U.S. and foreign patents pending
 - Japan and Europe
- FARADAYIC registered Trademark
- FARADAYIC registered Service mark

(CTO admitted to “Patent Bar”)

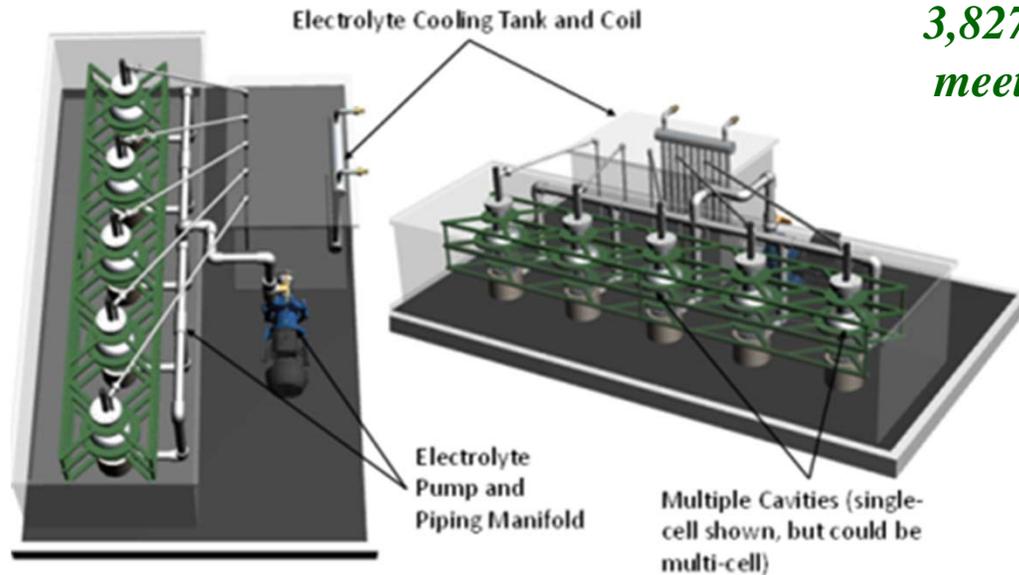


FARADAYIC® HF-FREE Electropolishing

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 issued April 14, 2015.

Low viscosity – aqueous electrolyte (~5% H₂SO₄)

- Vertical orientation
- Completely filled
- No rotation
- ➔ Industrially compatible



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

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

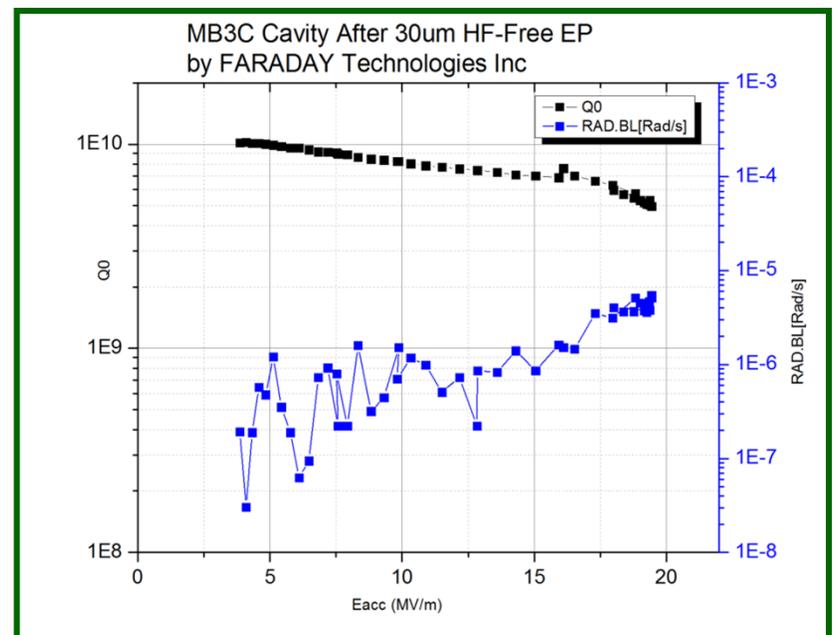
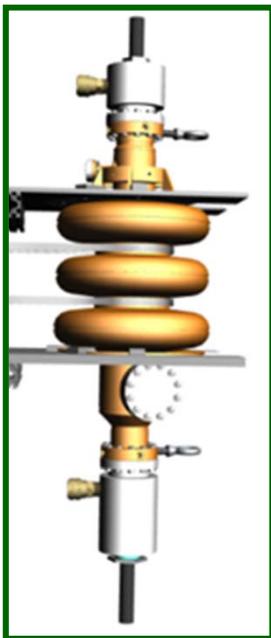
	Baseline EP	FARADAYIC EP
Electrolyte	9:1 H ₂ SO ₄ :HF	~5% (wt) H ₂ SO ₄ in H ₂ O
Processing Voltage	DC: ~17 V	Pulse Reverse: ~3 V / 9 V
Processing Temperature	25°C	25°C
Cavity Orientation	Horizontal	Vertical
Electrolyte Volume Fill	60%	100%
Electrolyte Flow Rate	~8 L/min	~8 L/min
Cavity Rotation	1 rpm	No Rotation
Cathode Material/Shape	Aluminum/Tube	Mixed-Metal Oxide Coated Titanium/Rod
Material Removal Rate	0.2 μm/min	0.05 μm/min

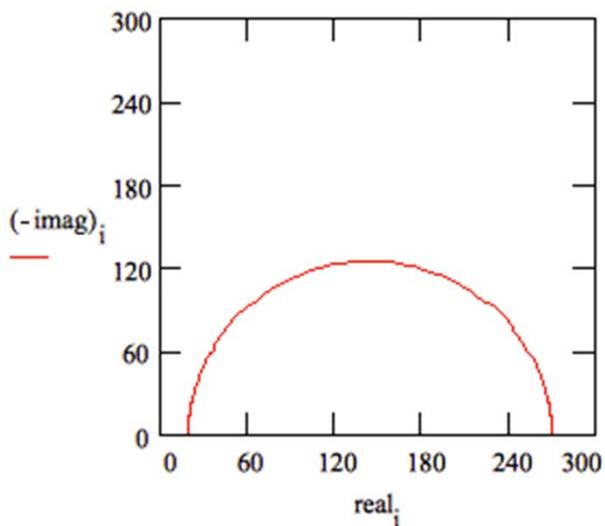
	Baseline EP	FARADAYIC EP
Operating:		
Acid	\$11,228,418	\$1,125,138
Labor	\$2,965,925	\$1,817,825
Capital:	\$3,186,806	\$1,712,647
TOTAL COST	\$17,381,149	\$4,655,610
Intangible:		
Materials	✗	✓
Environment	✗	✓



FARADAY TECHNOLOGY, INC. Commercialization: “Market Acceptance”

- Cavity processing by industry: “Build to Print”
 - Build to print parameters defined by DOE Laboratories (SRF2015)
 - ✓ Thomas Jefferson National Accelerator Facility (Jefferson Lab)
 - ✓ Fermi National Accelerator Laboratory (Fermi Lab)
- Executed (CRADA) with Jefferson Lab 5-17-2016
 - Faraday transfer knowledge of FARADAYIC[®] HF-FREE ElectroPolishing
 - Jefferson Lab provide SRF cavities and FARADAYIC[®] HF-FREE ElectroPolishing β -Scale “user” validation
- P.O. from Oak Ridge National Laboratory for FARADAYIC[®] HF-FREE ElectroPolishing services
“...very good results.” J. Mammosser Prototype Medium Beta 0.61 805MHz for SNS (ORNL)

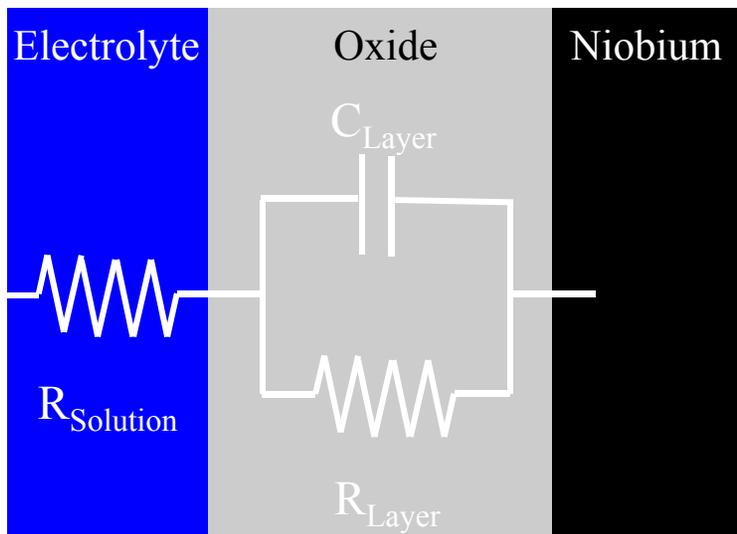
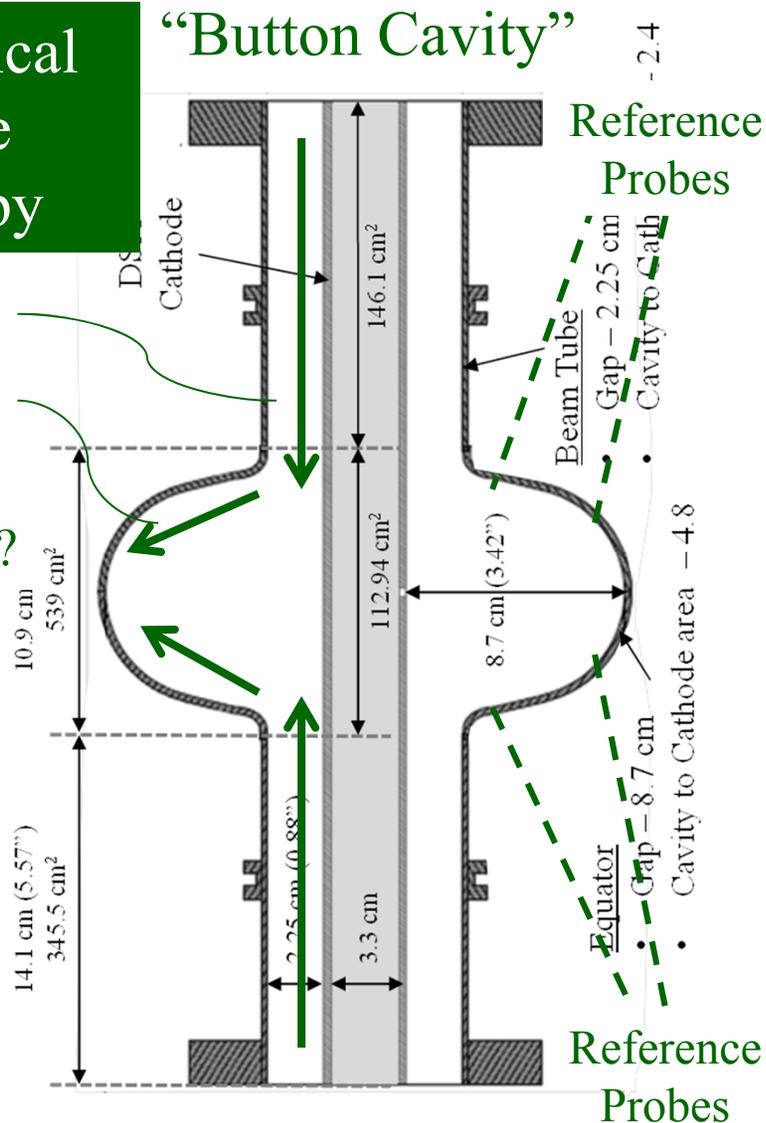




Electrochemical Impedance Spectroscopy

“Button Cavity”

Assumed Oxide Growth Propagation?



Mechanistic understanding will permit process optimization leading to faster processing and enhanced economics.

FARADAYIC® HF-FREE ElectroPolishing of Niobium SRF Cavities

Primary Developer:

1. Faraday Technology, Inc.

Dr. E.J. Taylor, Dr. Maria Inman, Dr. Timothy Hall and Mr. Stephen Snyder

Co-Developing Organizations:

2. Thomas Jefferson National Accelerator Facility
Dr. Charles Reece and Dr. Olga Trofimova
3. Fermi National Accelerator Laboratory
Mr. Allan Rowe and Dr. Anna Grassellino
4. Advanced Energy Systems, Inc.
Mr. Doug Holmes

Product or Service Introduction:

5. Oak Ridge National Laboratory
Dr. John Mammosser and Dr. Jeff Saunders





Department of Energy (DOE) Funding:

- 1) SBIR Phase I Grant No. DE-SC0004588 (Dr. Manouchehr Farkhondeh),
- 2) SBIR Phase I Grant No. DE-FG02-08ER85053 (Dr. L.K. Ken),
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- 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 “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-Viscosity, 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 Electroquímica, 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 scheduled Spring (2017).



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