

HF-Free Bi-Polar Electropolishing for Application on Multi-Cell Elliptical Cavities

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19th International Conference on RF Superconductivity
Dresden, Germany 30th –July 5th , 2019

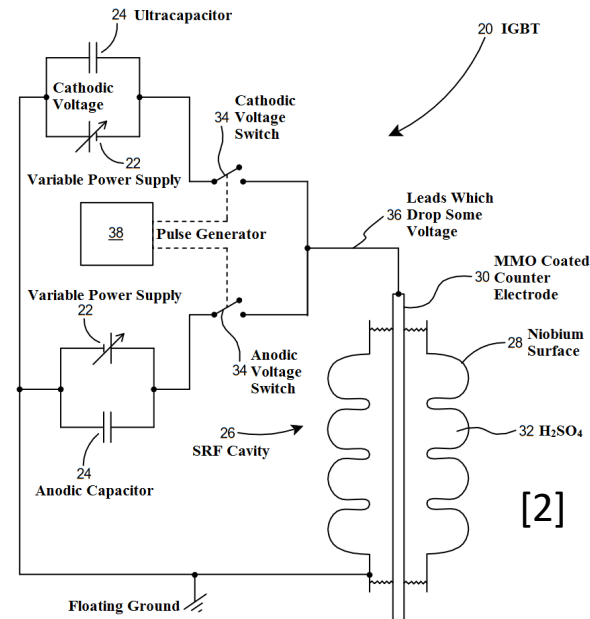
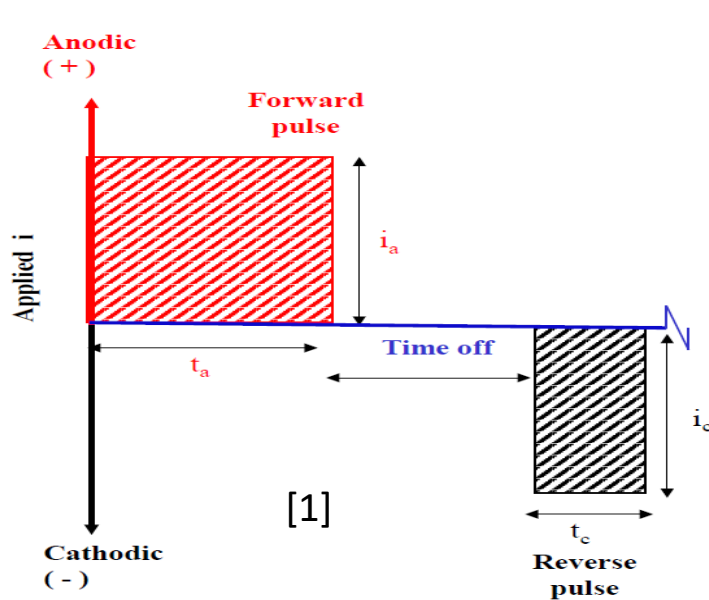
Outline

- 1. Why BPEP?**
- 2. Understanding mechanism**
- 3. Applying to cavities**
- 4. Expanding capacity to deal with multi-cells**

Why bipolar pulsed EP study?

- **So far, Nb SRF cavity work requires the use of hydrofluoric acid (HF)**
- **HF is a dangerous and poisonous acid**
 - Expensive procurement and disposal
 - Expensive infrastructure for safe handling
 - Serious personnel hazard
 - Limited industrial vendor interest
- **Can we find a better alternative?**
 - Cheaper
 - Safer
 - More industrialization options?

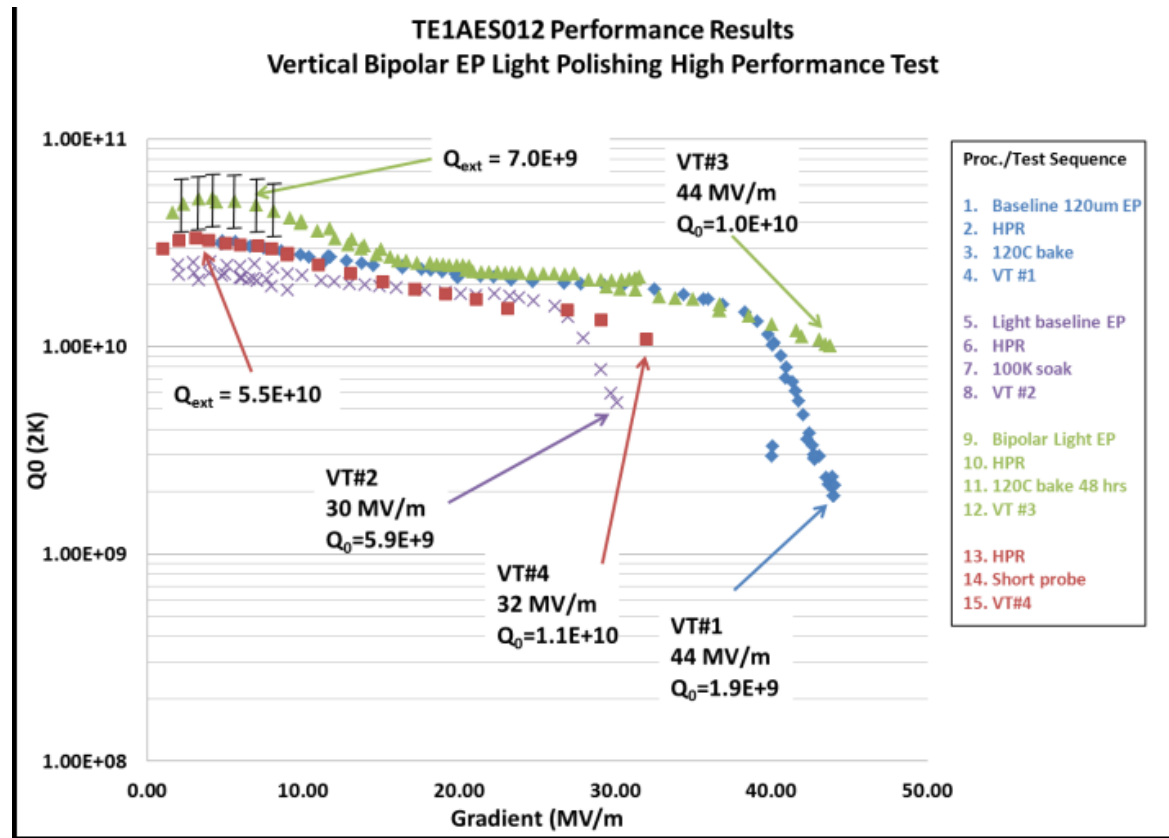
The bipolar pulsed EP (BPEP) promises a Chemistry for Processing Nb components that does not require HF



- ✓ **Niobium cavity** (working electrode), **mixed metal oxide** (MMO) coated Cu rod (counter electrode), and **H₂SO₄** or other non-HF solution (electrolyte).
- ✓ With a high-conductivity electrolyte (w/o HF), uses anodic pulses to repetitively alternately **anodize** the Nb then **erode the oxide** via reverse polarity hydrolysis -- safer and ecologically superior [1,2].
- ✓ No by-product for degrading RF performance (No Sulphur, as compared with traditional EP) .
- ✓ More economic compared with other surface processes, and good for commercialization [2].

1. E. J. Taylor, M. E. Inman, T. D. Hall "Electrochemical system and method for electropolishing superconductive radio frequency cavities", U.S. Pat No. 9 006 147, April 14, 2015; Japanese Pat No. 6 023 323, October 14, 2016; European Pat No. 2 849 908, February 15, 2017.
2. J. Musson, H. Tian, C. Reece, "High current bi-polar pulse system for use in electrochemical metal surface finishing", US patent in application.

Good performance cavity processed by BPEP, Questions remain?



From A.M. Rowe *et al.*,
SRF2013, Paris, TUIOC02

- This single cell was BPEP'ed by Faraday Technology and tested at FNAL has comparable RF performance for Nb cavity with traditional EP process (HF + H₂SO₄ – reported at SRF 2013).
- That was initial positive test, but no understanding about the mechanism, removal rate, removal uniformity, or path to industrialization.

Present status of bipolar pulsed EP study in Jefferson Lab

- **Systematic mechanistic and surface studies guide the design of the BPEP system and process optimization.**
- **Significant contributions from DOE-supported summer students (SULI): Cynthia McCord (2015 & 2016), Bradley Straka (2017 & 2018), Jonathan Carroll (2018), Hannah Hu (2019). The work was supported by US DOE.**

References: 2018 SULI student posters:

“Optimizing and Understanding Bipolar Pulsed Electropolishing (BPEP) on Niobium with HF free electrolyte at Jefferson Lab”, Jonathan Carroll

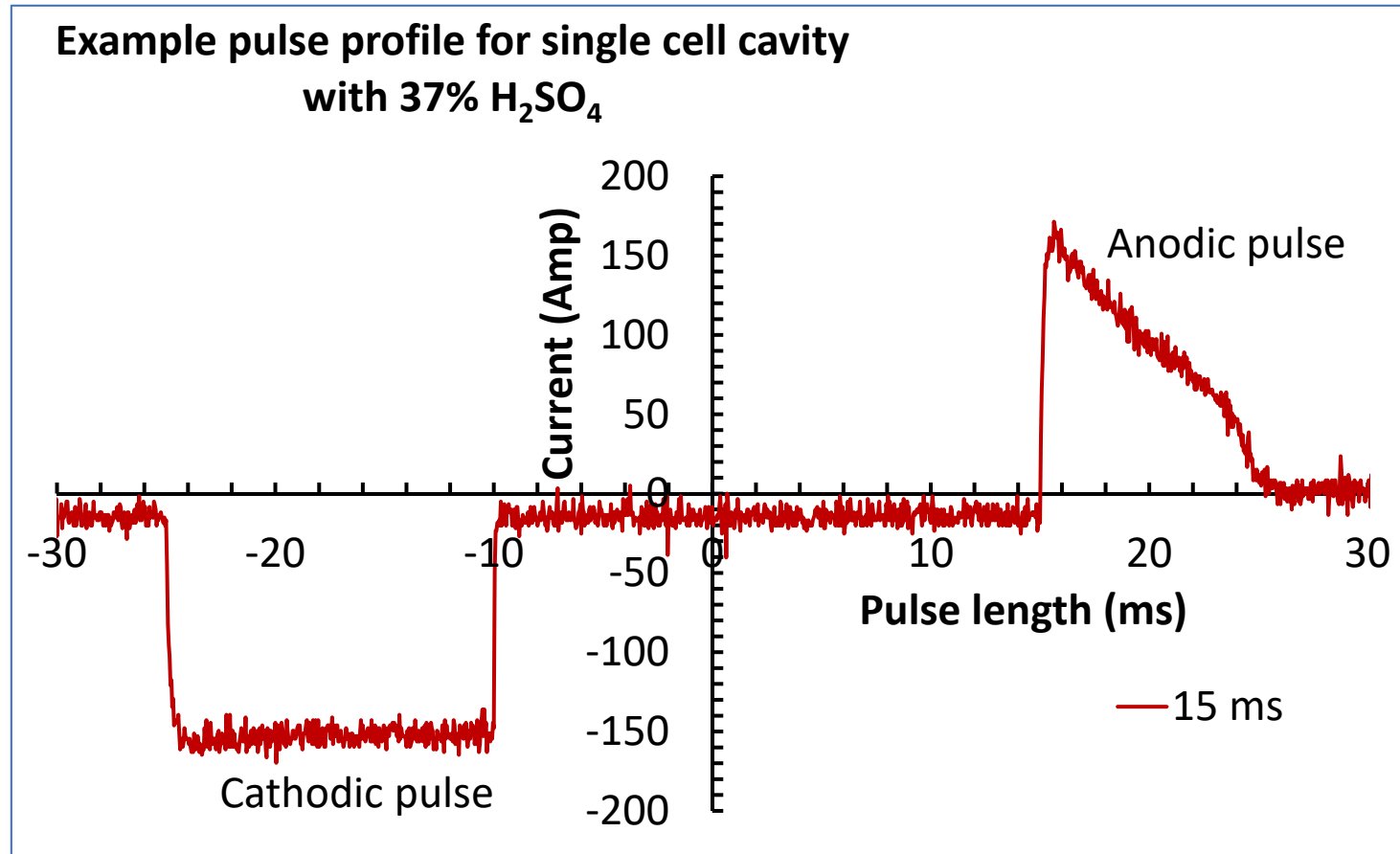
“Bipolar Pulsed Electropolishing of Single and Multi-cell Nb SRF Cavities with HF Free Electrolyte”, Bradley Straka

H. Tian *et al.*, LINAC18, TUPO043.

- **We have implemented a unique bipolar pulsed electropolishing system with upgraded pulse control system for processing single cell and multicell Nb cavities at JLab. [1]**
- **BPEP polishing has been applied to single cells, a 7-cell CEBAF C100 cavity, and to 9-cell TESLA-style cavities as the final process.**
- **On-going collaboration with Faraday Technology Inc.**

[1] J. Musson, H. Tian, C. Reece, *“High current bi-polar pulse system for use in electrochemical metal surface finishing”, US patent in application.*

Bipolar EP pulse structure Illustration



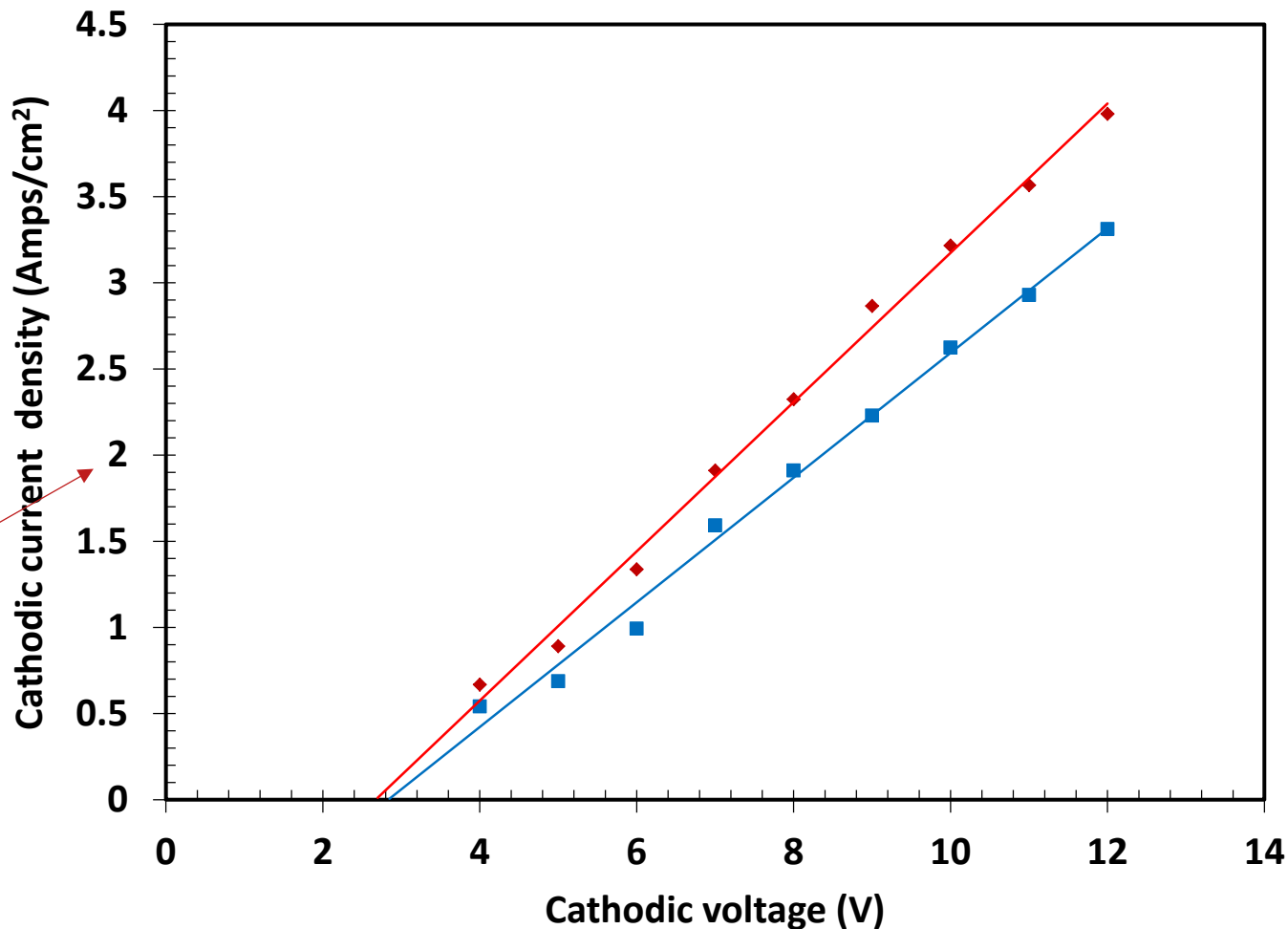
- ✓ Cathodic current is always constant- hydrolysis dominantly.
- ✓ Anodic current has time structure and decays to 0 with long-enough pulse-anodization of Nb.
- ✓ Amplitude of both depends on electrode geometries (cavity & counter electrode) and electrolyte conductivity.

The cathodic chemistry is simply hydrolysis

Cathodic Current Density increases with Cathodic voltage
(15% vs 37% H_2SO_4)

Small sample
process
characterization

Note the
implications of
these numbers

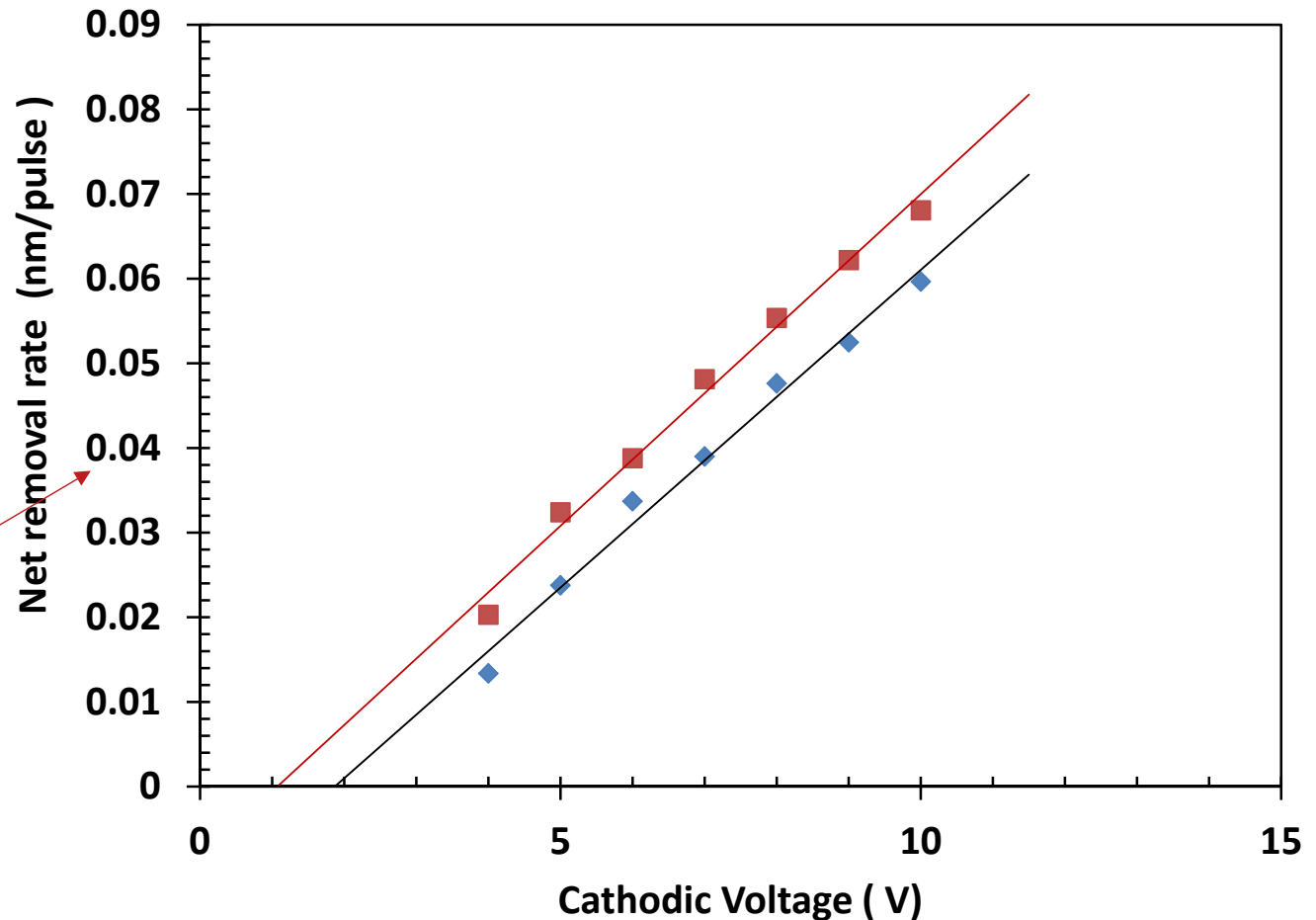


Removal rate increases linearly with cathodic voltage, a function of local current density

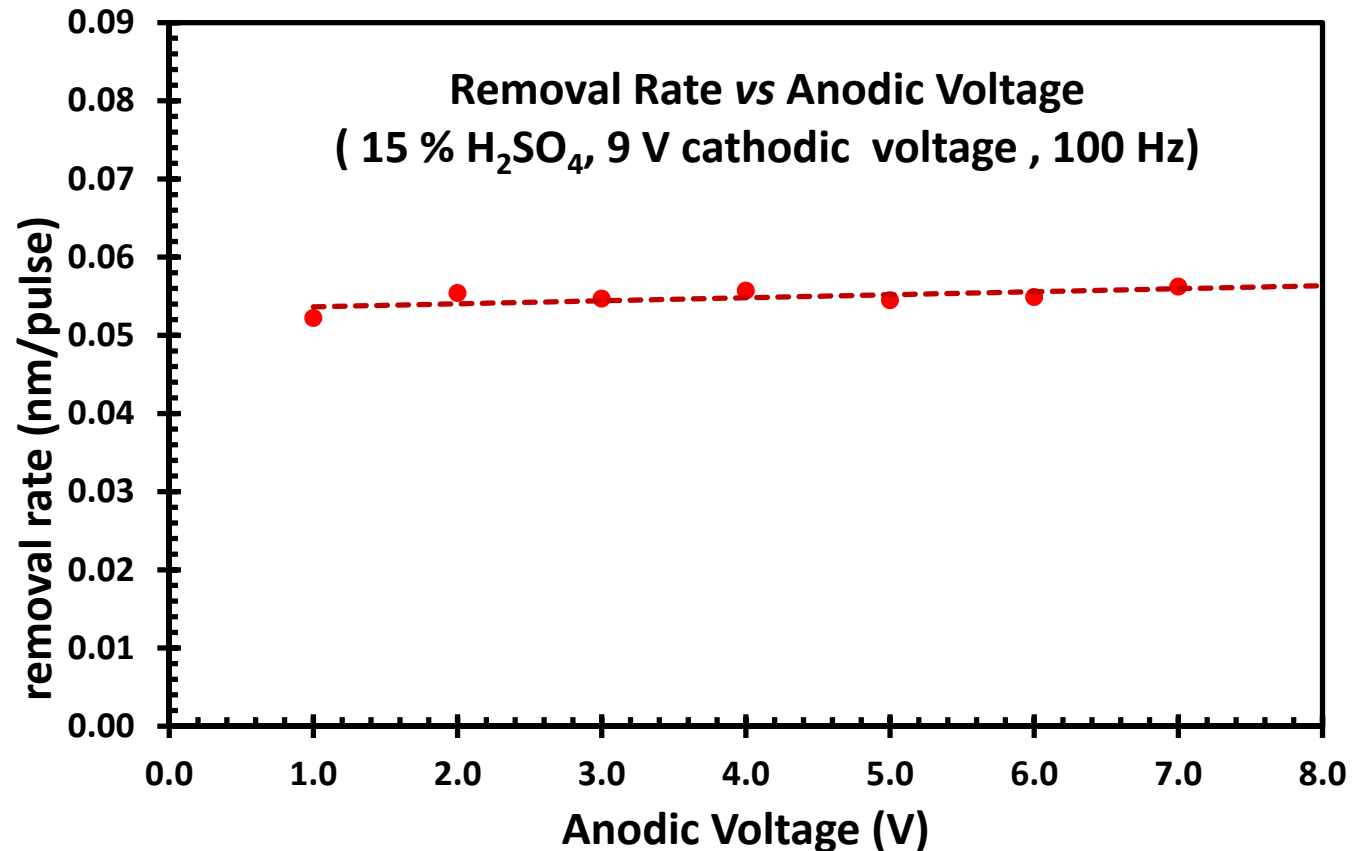
Avg. removal rate (nm/pulse) with different cathodic voltage 15 % vs 37% H_2SO_4 , 4 V anodic, 100 Hz PRF

Small sample process characterization

Note the implications of these numbers



Removal rate does not depend on anodic voltage once anodization is finished

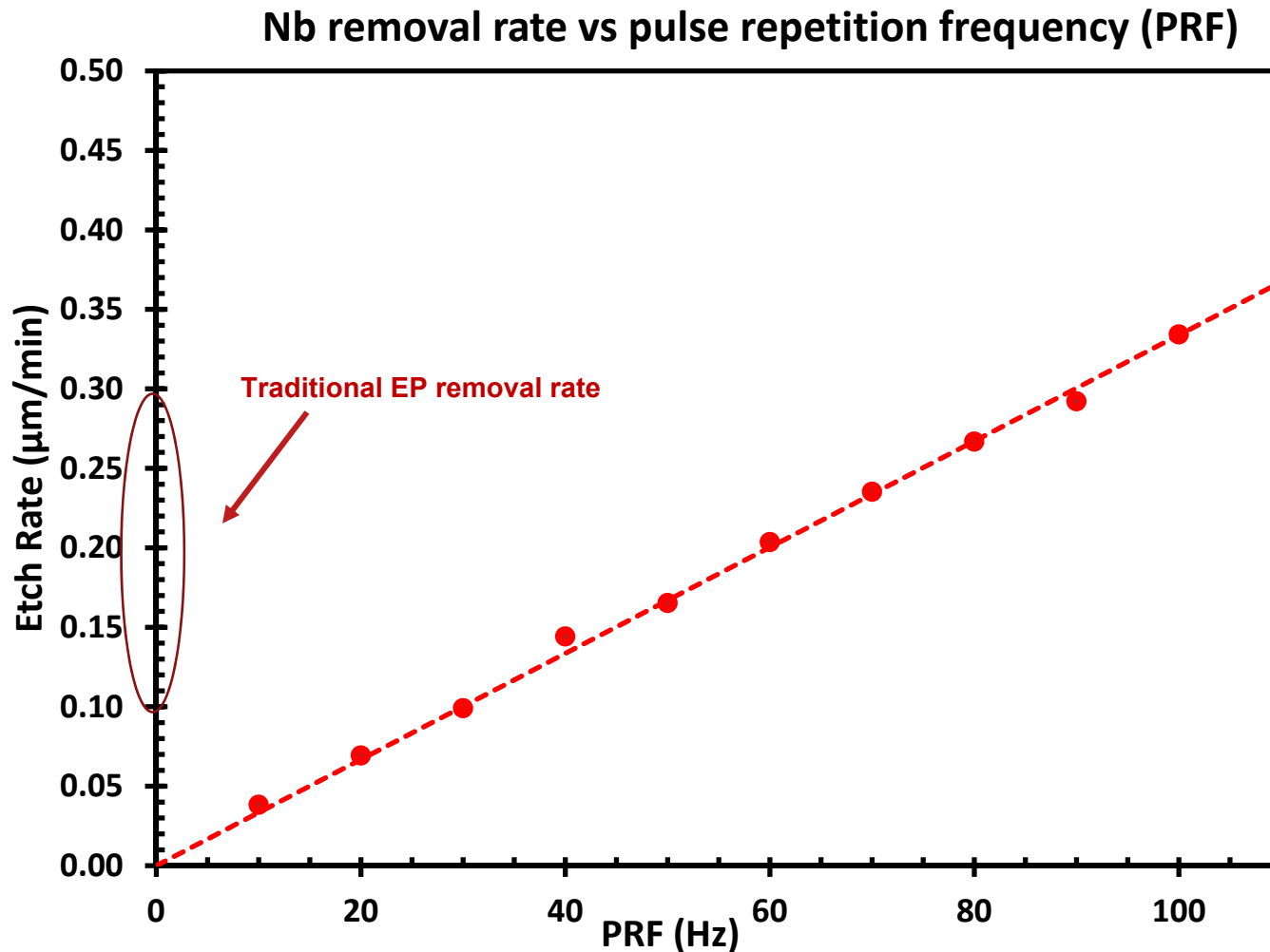


Removal amount per cycle is independent of oxide thickness!

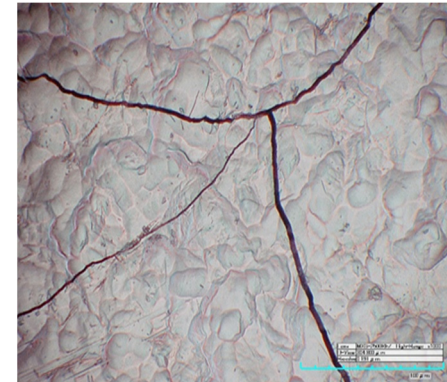
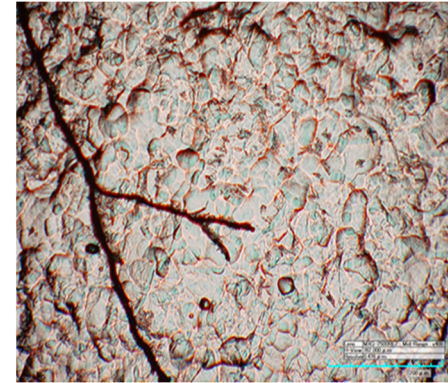
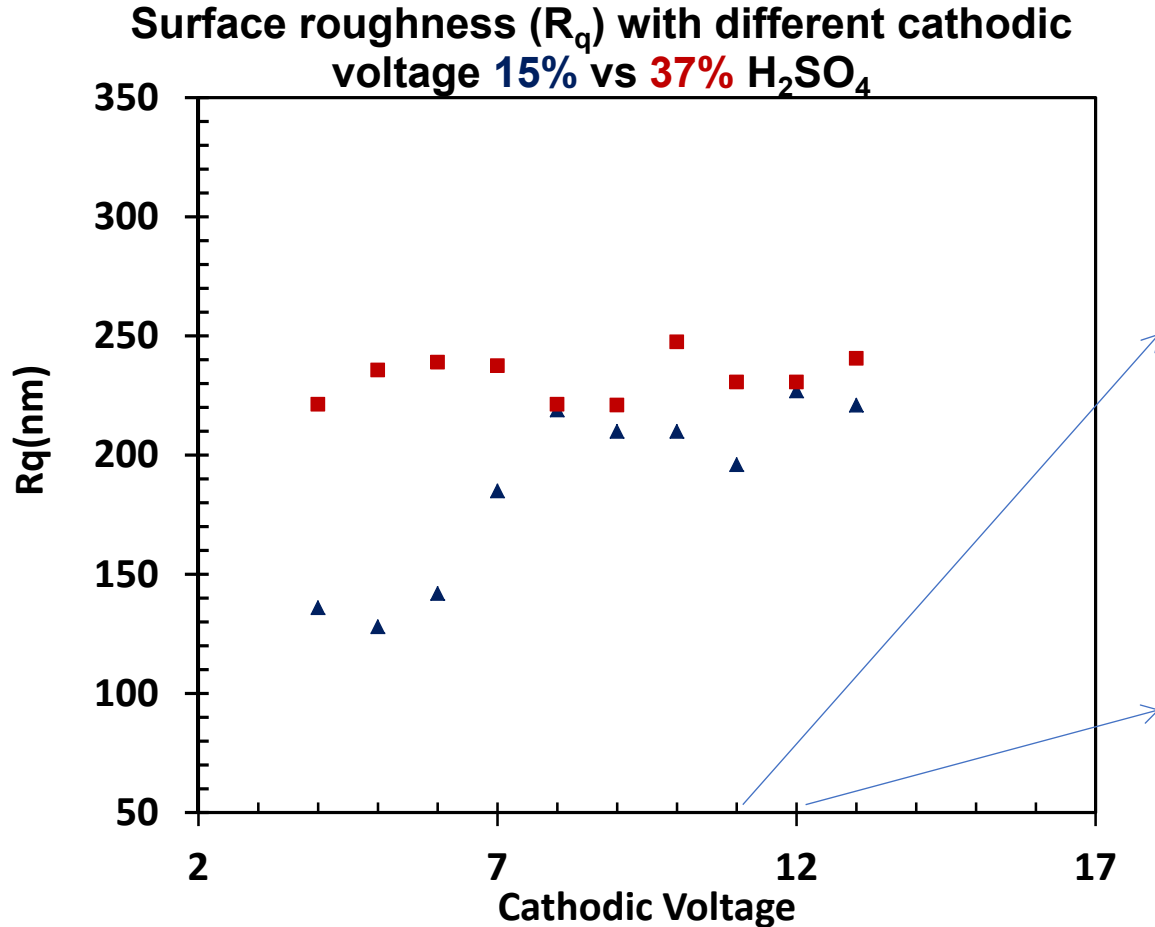
Our theory: hydrogen nano-bubble formation physically erodes the oxide locally in a random way -- *it is not chemistry.*

>> Not really electropolishing, it is **hydrolytic erosion of Nb oxide**

Removal rate is determined by repetition frequency under same pulse structure

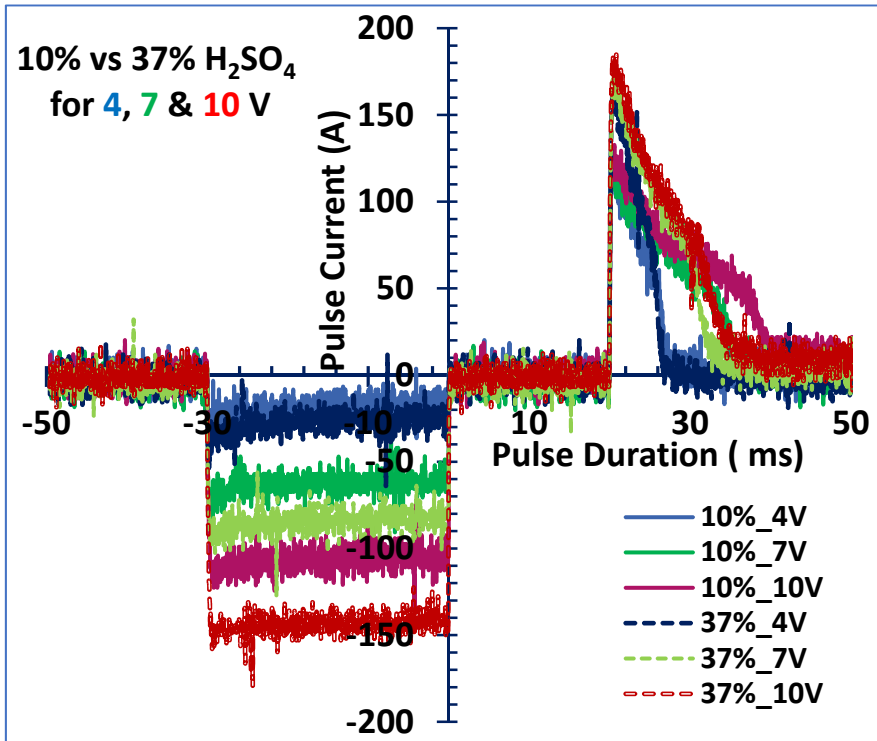


The surface roughness does not depend on cathodic voltage

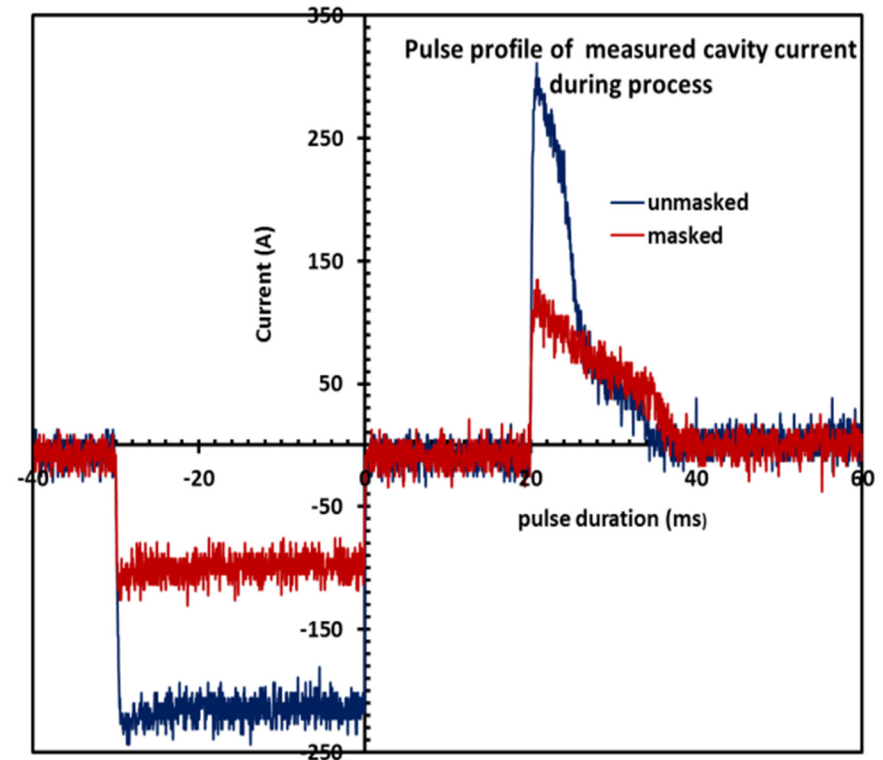


- ✓ With same removal, surface roughness does not depend on cathodic voltage.
- ✓ Too-long, too-high cathodic pulse risks driving hydrogen into the Nb surface >> stress corrosion cracking.

Studies of single cell for optimization of BPEP process

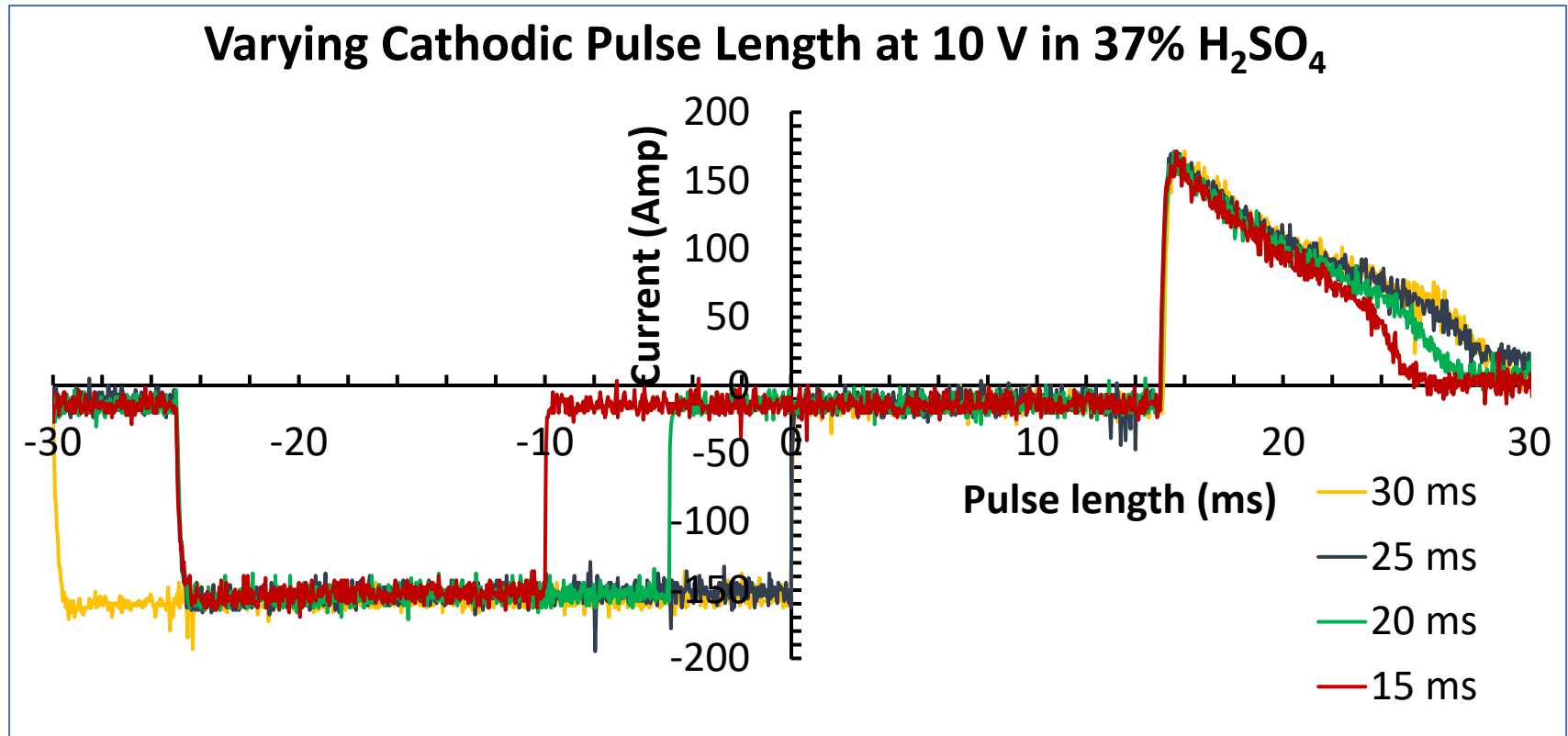


Electrolyte conductivity and cathodic pulse voltage affect both cathodic current and anodic pulse current shape.



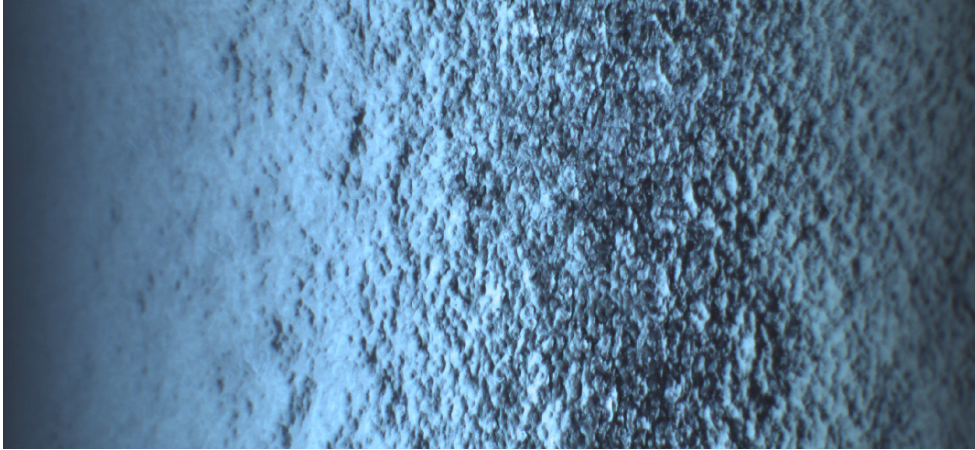
Geometry of cavity and center electrode affect anodic current pulse shape and cathodic pulse current-affect the removal rate .

Working to optimize pulse lengths vs. repetition rate for maximum removal rate



- ✓ Cathodic pulse duration may affect the efficiency of oxide removal at each pulse.
- ✓ The optimum repetition frequency will be influenced by cavity shape and the reactive surface area of the counter electrode. **Need to maximum counter electrode reactive surface area.**
- ✓ Maximizing cathodic current density **demands higher current** and performance from pulse generator and control system.

The internal cavity surface processed by BPEP



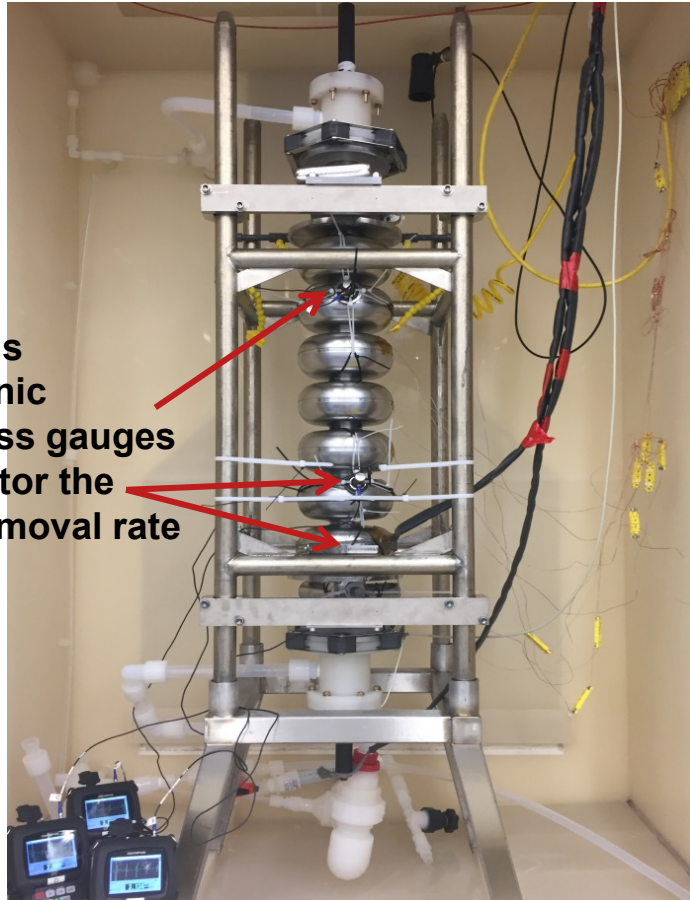
At beam tube



At the equator

The optical images (taken by the Kyoto camera) of the inside of the C100-1 cell indicates that BPEP smooths the sharp edges of grains, yielding a comparable surface finishing to traditional EP.

Improving removal uniformity and maximizing removal rate is needed for BPEP process

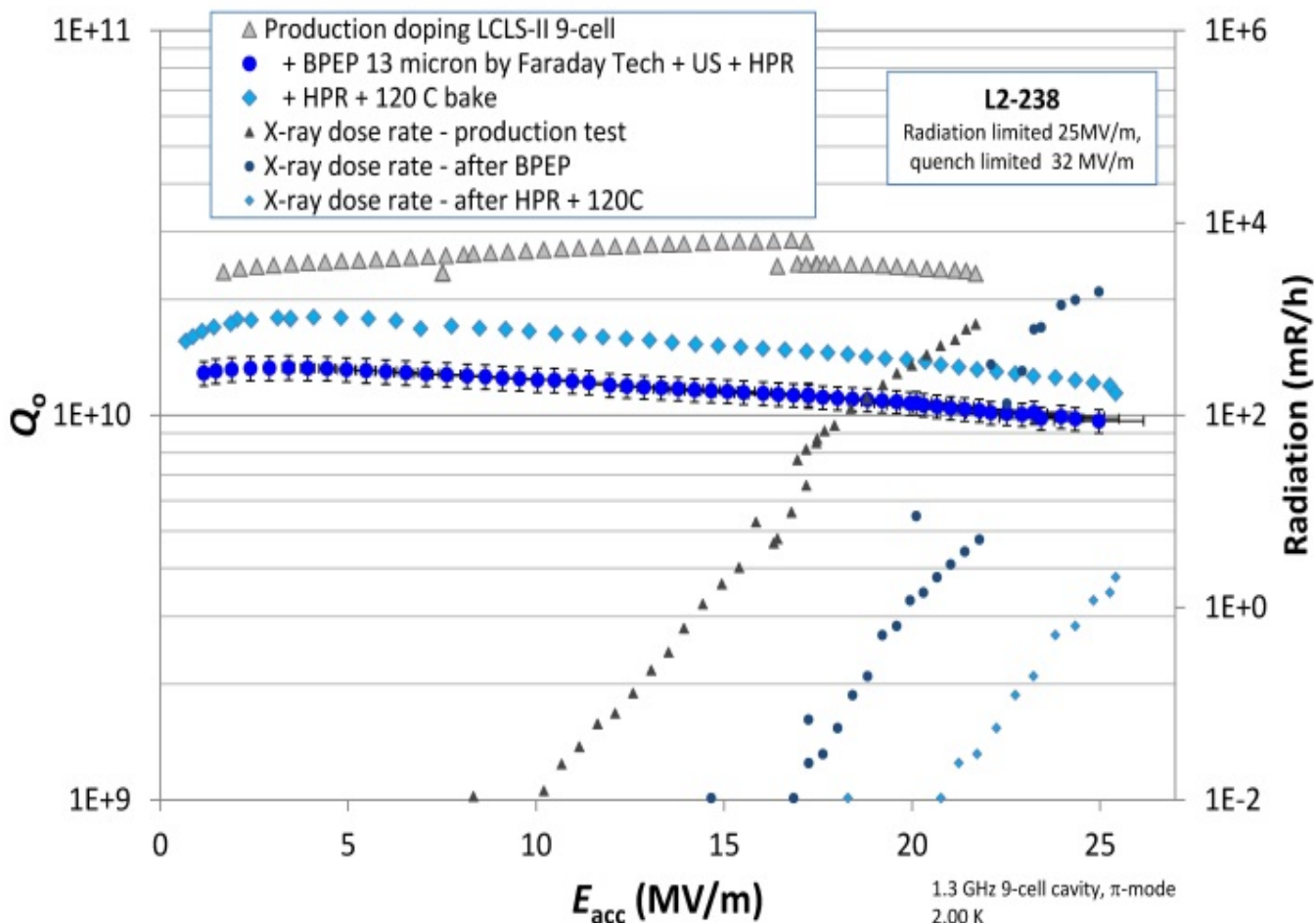


**JLab BPEP setup with
C100-1 7-cell cavity**

- 1.3 GHz **single cell cavity** with 37% sulfuric 15 ms vs. 25 ms cathodic pulse maintaining 11 Hz repetition frequency.
 - 15 ms pulse polished with removal rate at the equator of **0.01 $\mu\text{m}/\text{min}$** and 0.007 $\mu\text{m}/\text{min}$ at the beam tubes.
 - 25 ms pulse polished at the equator with the removal rate of **0.013 $\mu\text{m}/\text{min}$** , and polished the top beam tube with the removal rate of 0.2 $\mu\text{m}/\text{hr}$, and the bottom beam tube with the removal rate 0.6 $\mu\text{m}/\text{hr}$.
- C100 7-cell with 10% sulfuric acid at 6 Hz with a “cathodic pulse” of 8 V, with masked counter electrode, 7 cell was polished at a removal rate of 0.001 $\mu\text{m}/\text{min}$ (more study is underway) – **too slow!**
 - Discovered bad alignment of counter electrode.

The repetition frequency and current capability was limited by past pulse control system. This significantly reduced the removal rate from that on samples.

RF test result of nine-cell processed by BPEP at Faraday

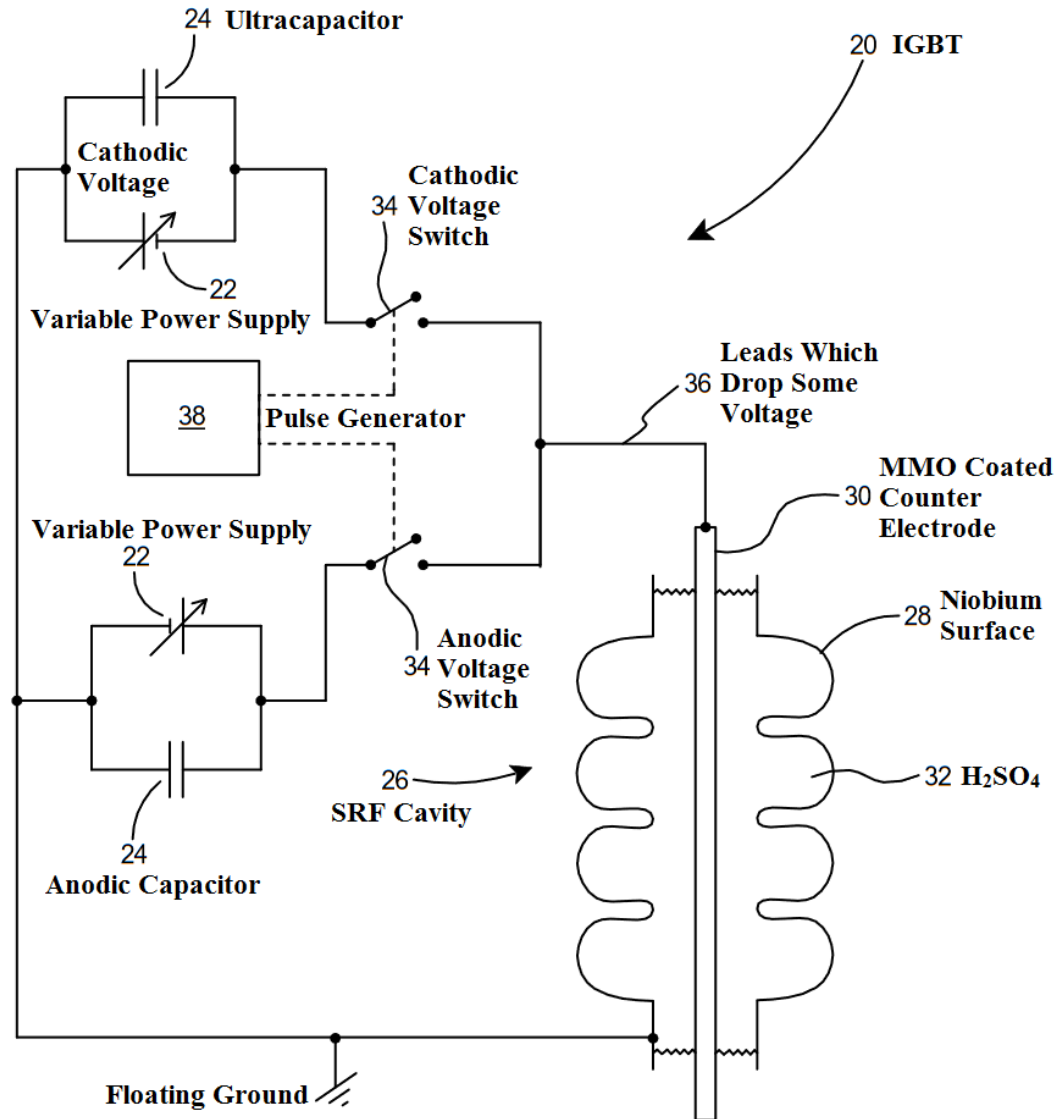


Faraday
removed
13 microns
by weight
- ~90 hrs

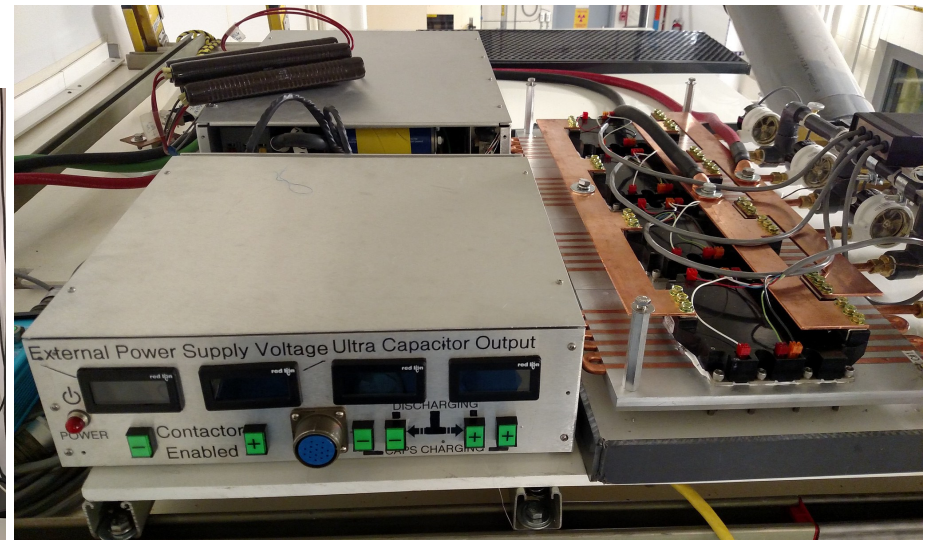
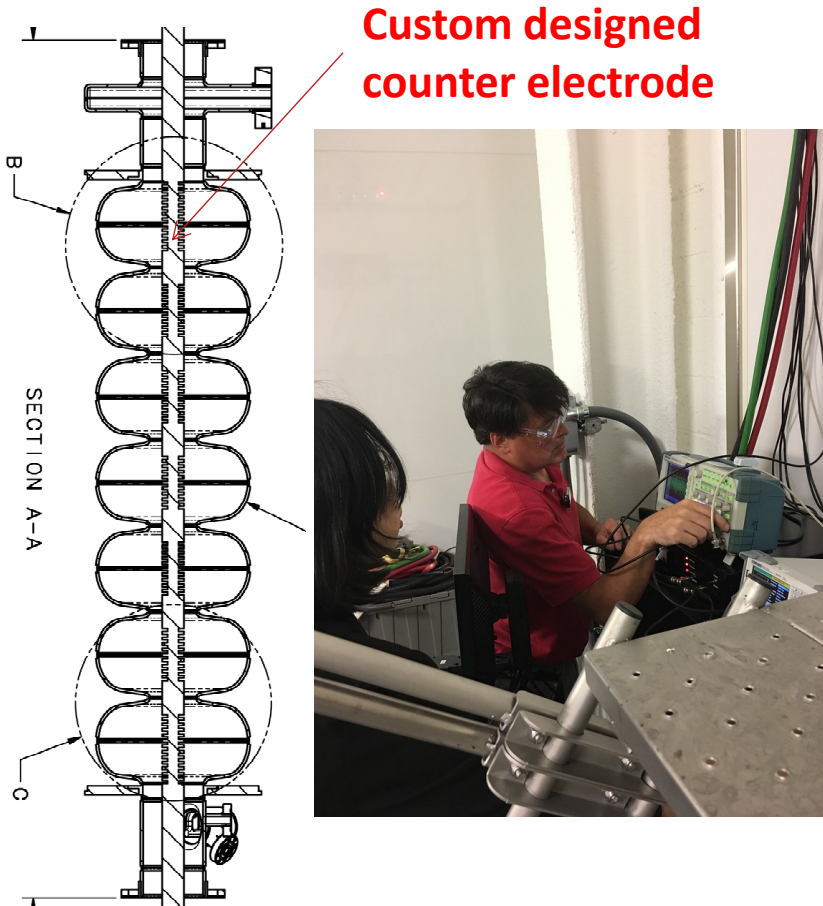
Work with this
cavity stopped
when LCLS-II
Project reclaimed
the cavity.

- This result suggests that BEPE has no fundamental limit in obtaining a gradient in multi-cell cavities up to 32 MV/m.
- Just the beginning...

Upgrade of BPEP at JLab for Multicell Processing



Upgrade of BPEP at JLab for Multicell Processing



Upgrade of high-current pulse control system with water cooling

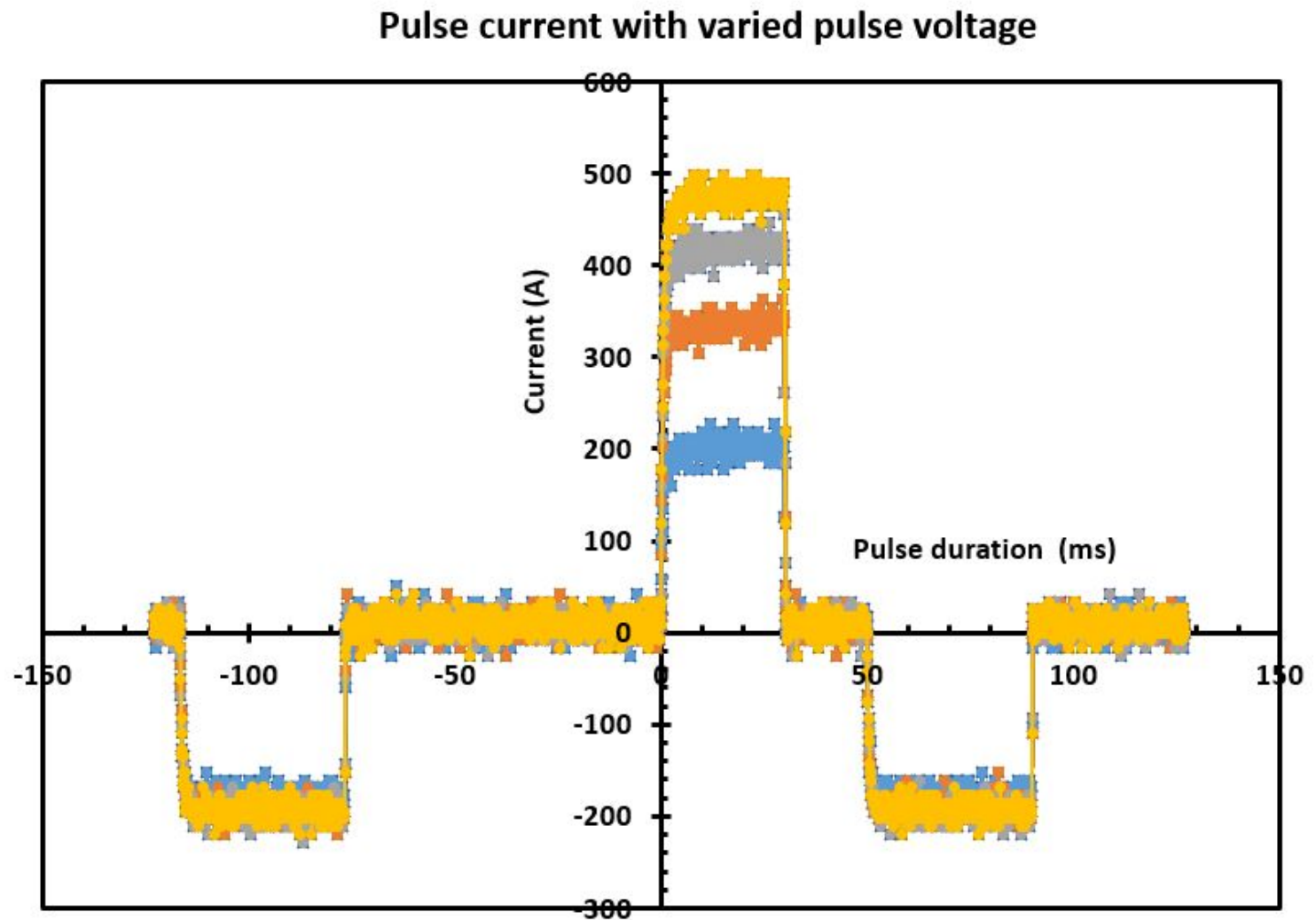
- Two 500 Farad ultracapacitor banks
- Parallel IGBT high-current switches

Net removal rate strongly depends on the cathodic pulse current density and repetition frequency.

- ✓ Increase reactive surface area of the counter electrode.
- ✓ Upgrade pulse control system for optimum removal rate and uniformity on multi-cell cavities (~ 2000 A).

Preliminary test of upgraded pulse system

System test
into resistive
load



Present and future BPEP work

- ❑ Refine process parameters for 7-cell and 9-cell cavities, controlling removal uniformity and maximizing removal rate, process reproducible good performance SRF cavities.
- ❑ Process N-doped cavities to check for any performance differences.
- ❑ Process different structure SRF cavities.
- ❑ Fabricate and process “completely HF-free” single cell Nb cavity.
- ❑ Need (free) good multi-cell cavities to work with..., and the support of community.

Acknowledgments

➤ Jefferson lab :

Gary Cheng, Phil Denny, Derrick Dail, Mark Lester, John Musson, Michael Morrone, Joshua Spradlin, Christiana Wilson and Larry Philips.

➤ Faraday Technology, Inc.,

Timothy Hall, Maria Inman, Rajeswaran Radhakrishnan, E. Jennings Taylor

➤ Summer SULI and REU* students:

Cynthia McCord (2015 & 2016), Bradley Straka (2017 & 2018), Jonathan Carroll (2018), Hannah Hu* (2019).

➤ US DOE Office of Science, Office of Nuclear Physics under contract no. DEAC0506OR23177.

Thank you for your attention!