Bipolar EP: Electropolishing Without Fluorine in a Water Based Electrolyte

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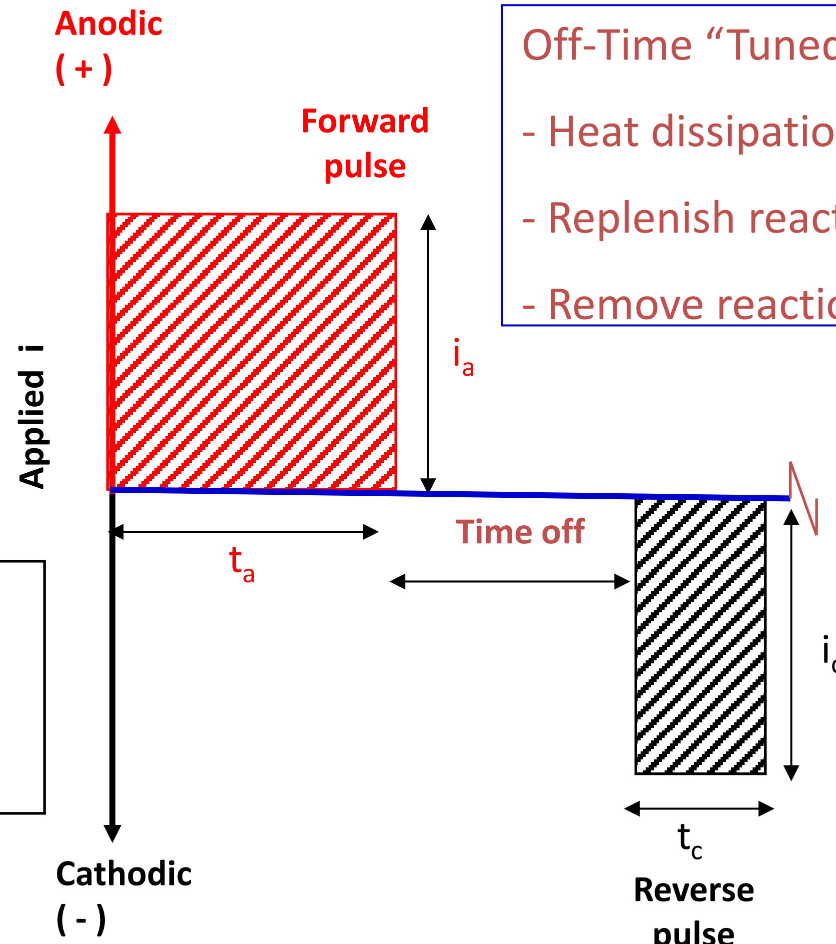
U.S. DEPARTMENT OF ENERGY SRF2013 PARIS FARADAY TECHNOLOGY, INC. Fermilab

What is bipolar EP?

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

Electric Field Control – Not Chemistry ➔ Electrolyte Flexibility

Why pursue bipolar EP?

- Electropolishing without HF
  - Labs strongly dislike HF due to safety issues
  - Ecological footprint can be reduced
- Potential ‘Drop-in’ EP technology that may replace traditional HF-based EP.
- Potential industrial scalability improvement over traditional EP (vertical orientation).
- Electrolyte modification from 9:1 solution of 95% H_2SO_4:49% HF to dilute H_2SO_4. Working concentration is 5-10 wt% H_2SO_4.
  - Relatively safe and ecologically superior
- Potential improvement of EP parameter control.

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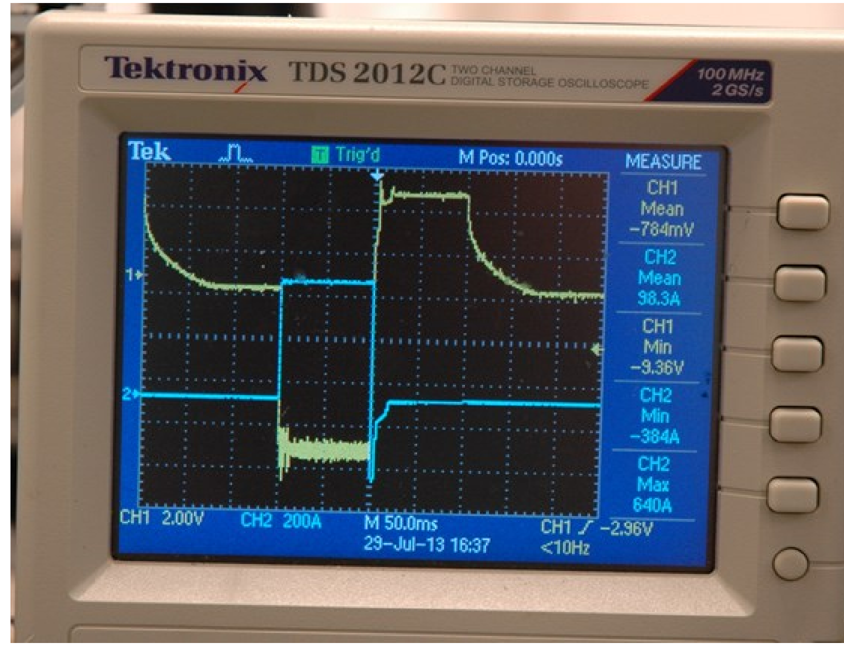
Project Goals

- Develop pulse/pulse reverse (bipolar) waveform capable of polishing fine grain niobium samples using 5-10% H_2SO_4.
  - Surface finish and appearance must be equivalent to trad. EP
  - Removal rates—determine what is possible.
  - Power requirements—scale for 1-cell & 9-cell cavities
- Construct small EP facility for 1.3 GHz 1-cell cavities compatible with horizontal EP to prove 'drop-in' technology
  - Horizontal EP tool fabrication
  - US cleaning capability
  - Power supplies sized for 1.3 GHz 1-cell (0.13 m^2)
  - Modest utilities—DI water, power
- Perform iterative polishing on 1.3 GHz 1-cell cavities.
  - Sacrificial cavity for waveform development
  - Performance trial cavities
  - Vertical tests

Sample Studies

- Coupon studies yielded mixed information
  - Electrolytes of 10-30% H_2SO_4 produced similar surface finish, but did affect current density and removal rates.
  - Achieved removal rates in the range of 0.1-10 µm/min. Tunable to applied cycle frequency and duty factor.
  - Evaluated cathodic and anodic waveform voltages + on/off times for heat dissipation and power requirements.
  - Achieved surface finishes of Ra <0.2 µm -- equivalent or better than traditional EP.
  - Determined that power requirements as a function of surface area did not scale with available cavity/cathode area ratios. Polishing edge effects dominated and skewed results.
  - Found waveform shape for niobium, but not necessarily input values compatible with large surface areas.
  - Terminated sample studies due to inability to scale input variables with available cavity parameters.

Bipolar Waveforms



Rectifier Values:

P_{avg} = 199 W

V_{anodic} = +4 V

$V_{cathodic}$ = -10 V

$Current_{avg}$ = 90 A_{osc}, 117 A_{rect}

$Current_{pk}$ = 368 A_{an}, 550 A_{ca}

Typical 1-cell EP:

P_{avg} = 630 W

P_{pk} = 1 kW

Voltage = 18 VDC

$Current_{avg}$ = 35 A

Cavity Polishing Waveform Details:

- 5% H_2SO_4
  - 4 V anodic for 200 ms, off 300 ms, -10V cathodic for 200 ms
- 10% H_2SO_4
  - 4 V anodic for 100 ms, off for 150 ms, -10V cathodic for 100 ms
- Achieved only 1.5 µm/hour removal rates.

*Frequency doubled due to conductivity improvement

SEE TUPO54

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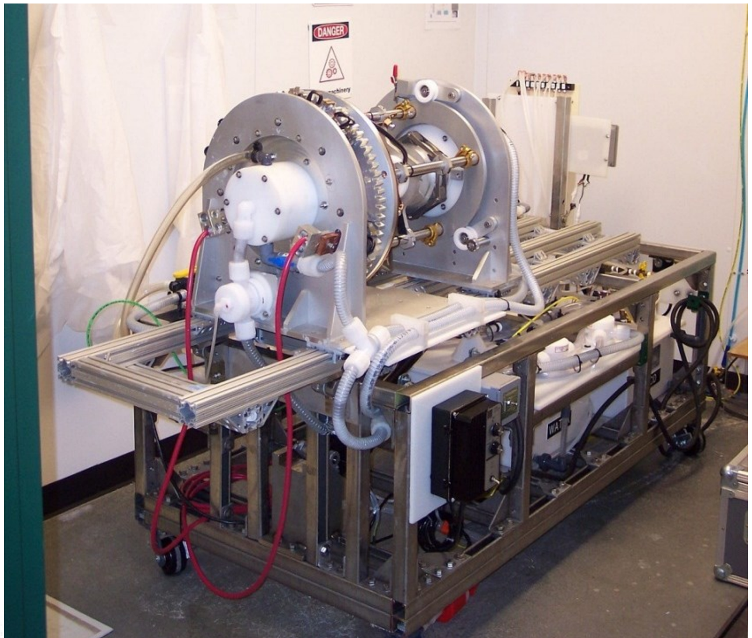
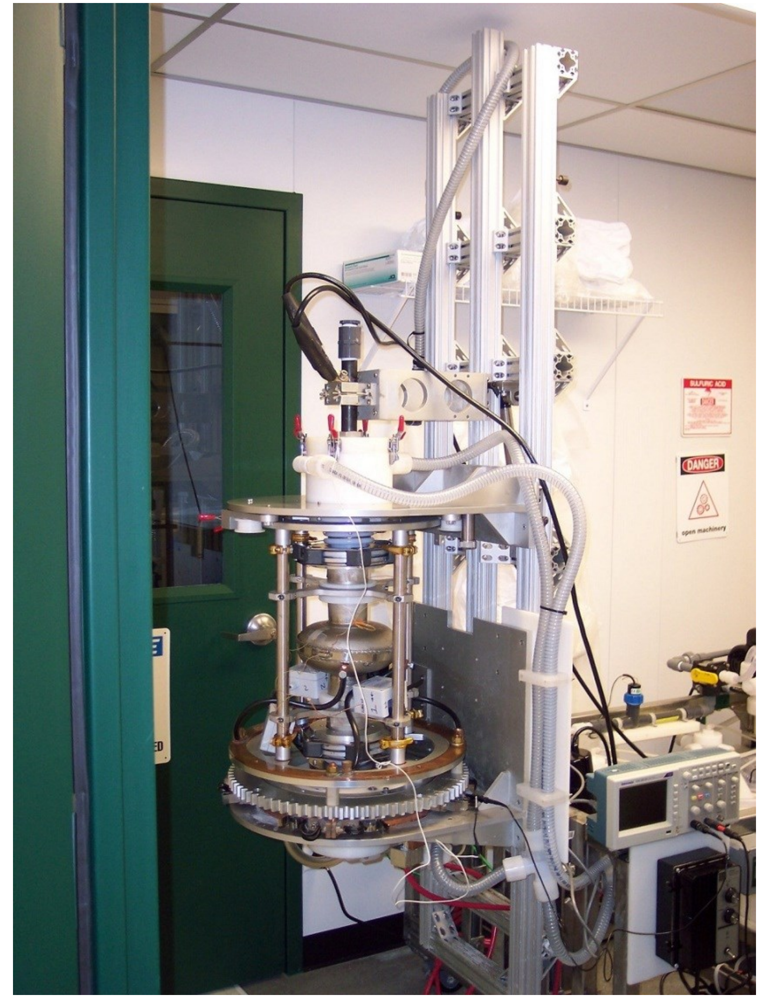
‘Drop-in’ (replacement) EP?

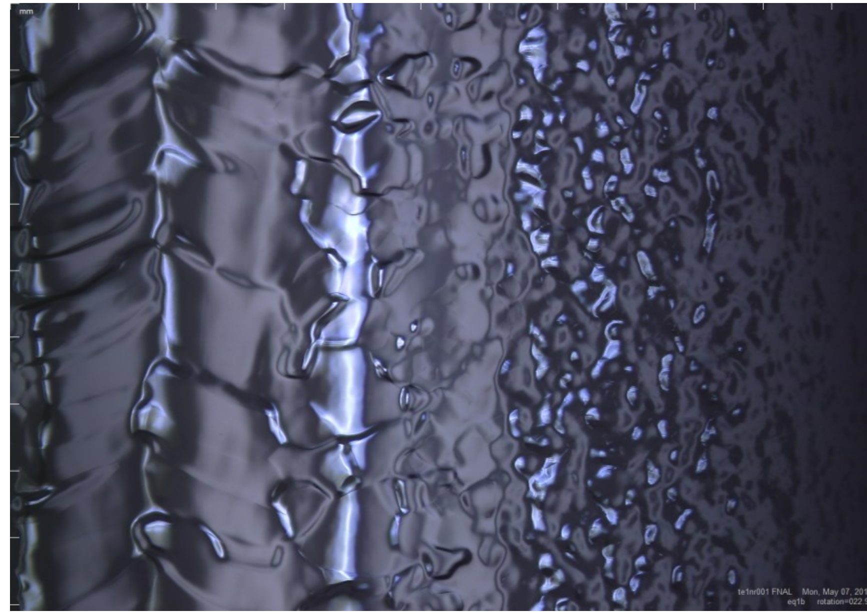


Horizontal EP tool at Faraday Technology, Inc. Initial trials were performed horizontally, but transitioned to vertical with dramatically improved performance.



Bipolar EP Surface Details

TE1DESYB5—20 um bipolar EP.



TE1NR001 >120 um bipolar EP.

Unmasked cathode:

- 3:1 removal ratio beamtube/iris:equator

Partially masked cathode:

- 2.5:1 removal ratio beamtube/iris:equator

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Performance Trial #1

- Goal: Verify vertical test performance of bipolar EP on a single-cell 1.3 GHz cavity (TE1DESYB5).
  - Process & Test Sequence:
    - Vertical test #1 for baseline performance previously treated cavity.
    - Light Vertical Bipolar EP - > 10μm removal at equator.
    - High pressure rinse and vertical test preparation
    - Vertical test #2.

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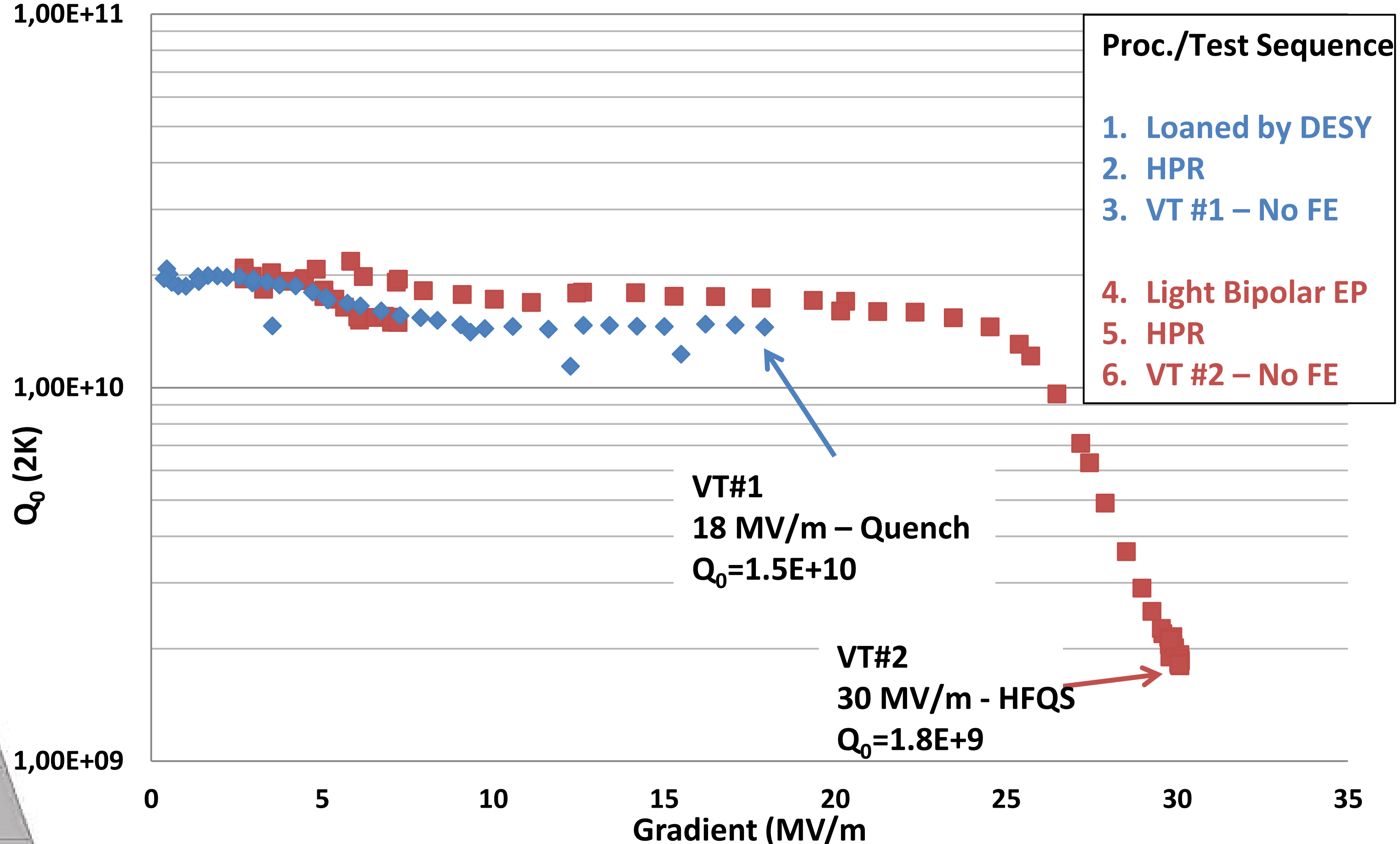
TE1DESYB5 Performance Results
Bipolar EP Light (~20 um) Polishing Recipe Test
Vertical Orientation - Unmasked Cathode



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Performance Trial #2

- Goal: Perform bulk electropolish on a **poor** performing cavity (TE1AES007) using Bipolar EP to determine process compatibility.
  - Process & Test Sequence:
    - Vertical test #1
    - Bulk Vertical Bipolar EP - > 50µm removal at equator.
    - High pressure rinse and vertical test preparation
    - Vertical test #2.

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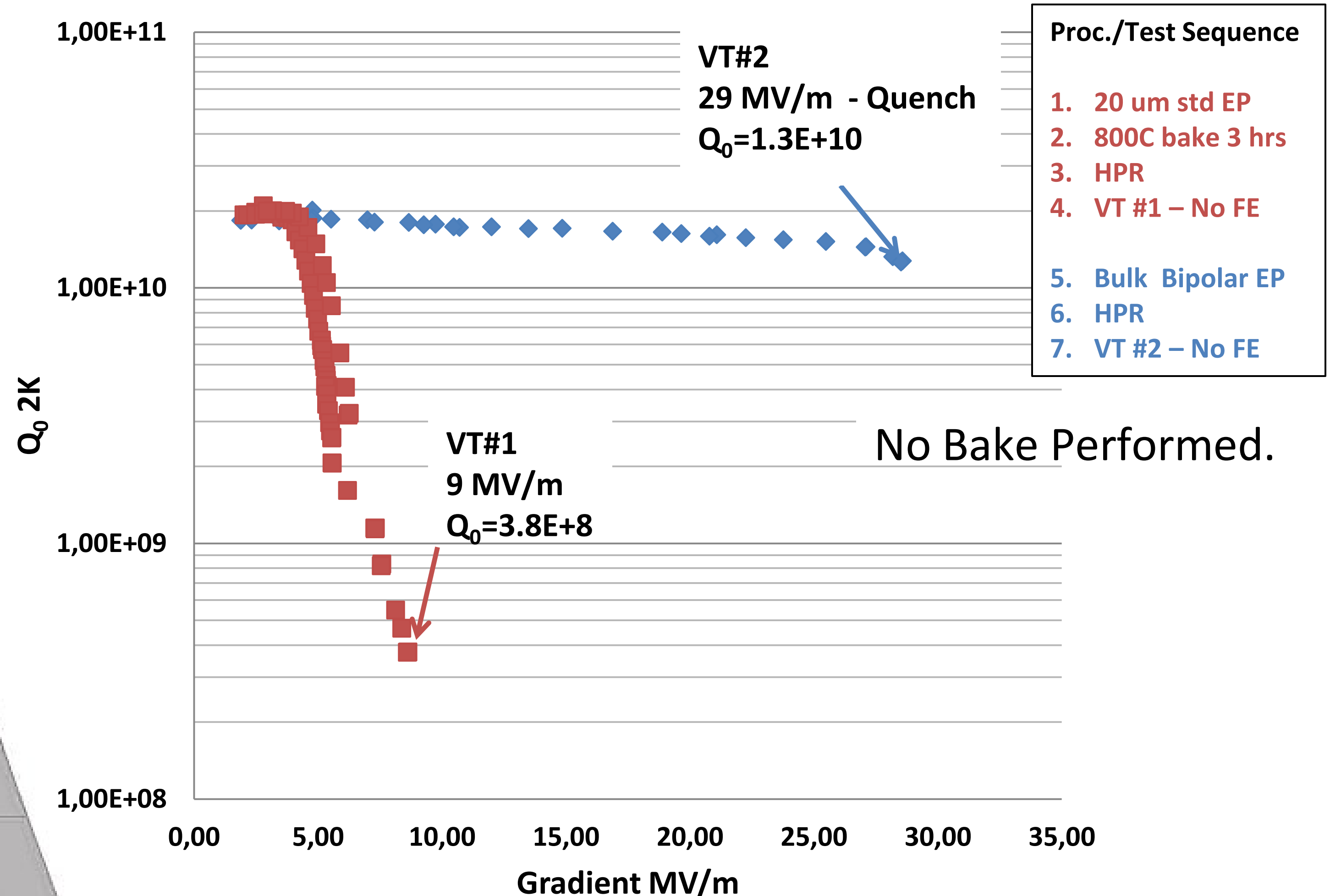
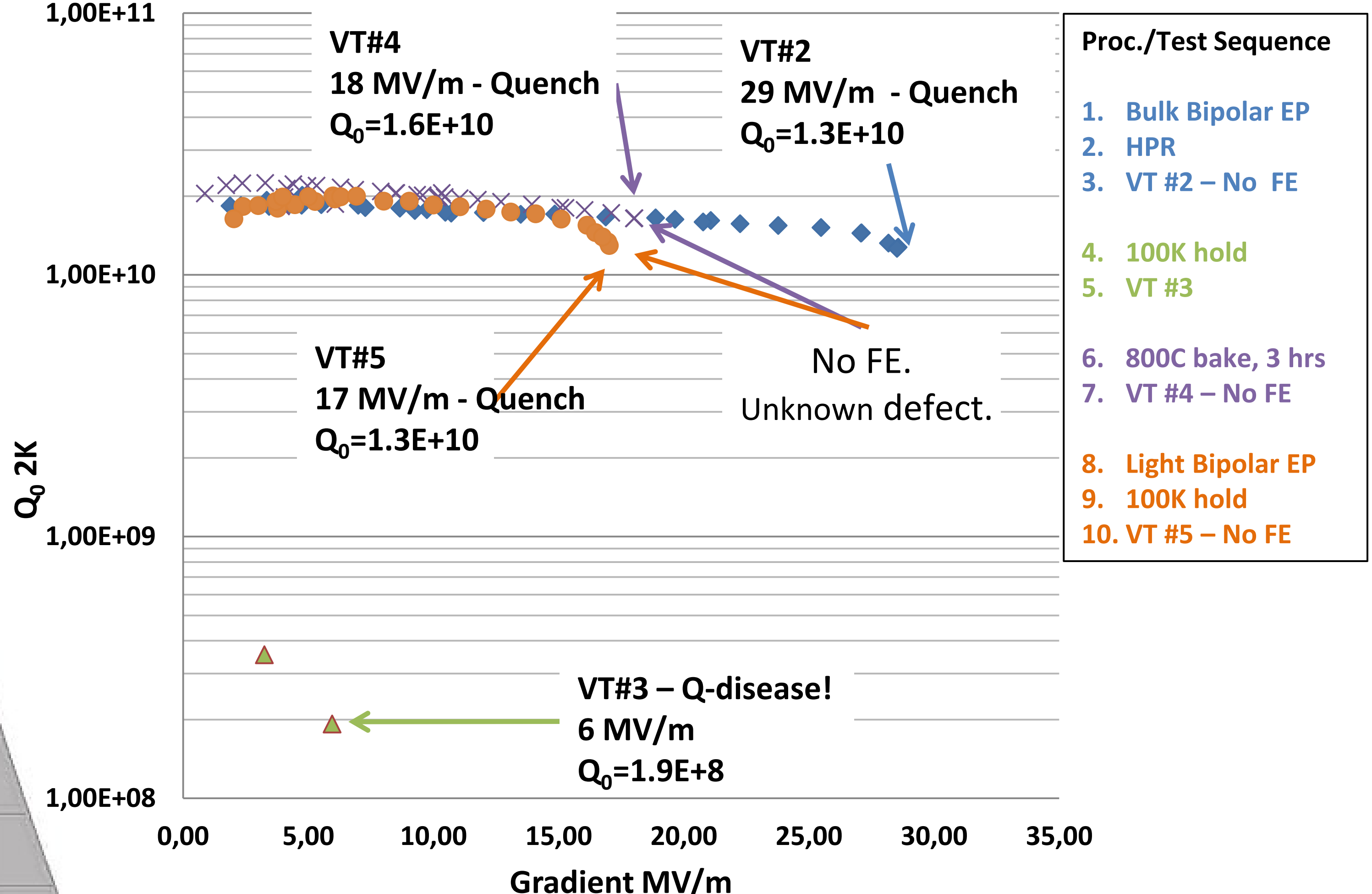
TE1AES007 Performance Results
Bipolar EP Bulk (~100 um) Polishing - Vertical Orientation
Partially Masked Cathode



Performance Trial #3

- Goal: Evaluate Q-disease behavior due to bulk and light bipolar EP.
  - Process & Test Sequence:
    - 100 K hold
    - Vertical test #3
    - 800C bake 3 hrs with end caps (no-foils)
    - High pressure rinse and vertical test prep
    - Vertical test #4
    - Light Bipolar EP – 20 um at equator
    - High pressure rinse and vertical test prep
    - 100 K hold
    - Vertical test #5

TE1AES007 Performance Results
Bipolar EP Q-disease Studies



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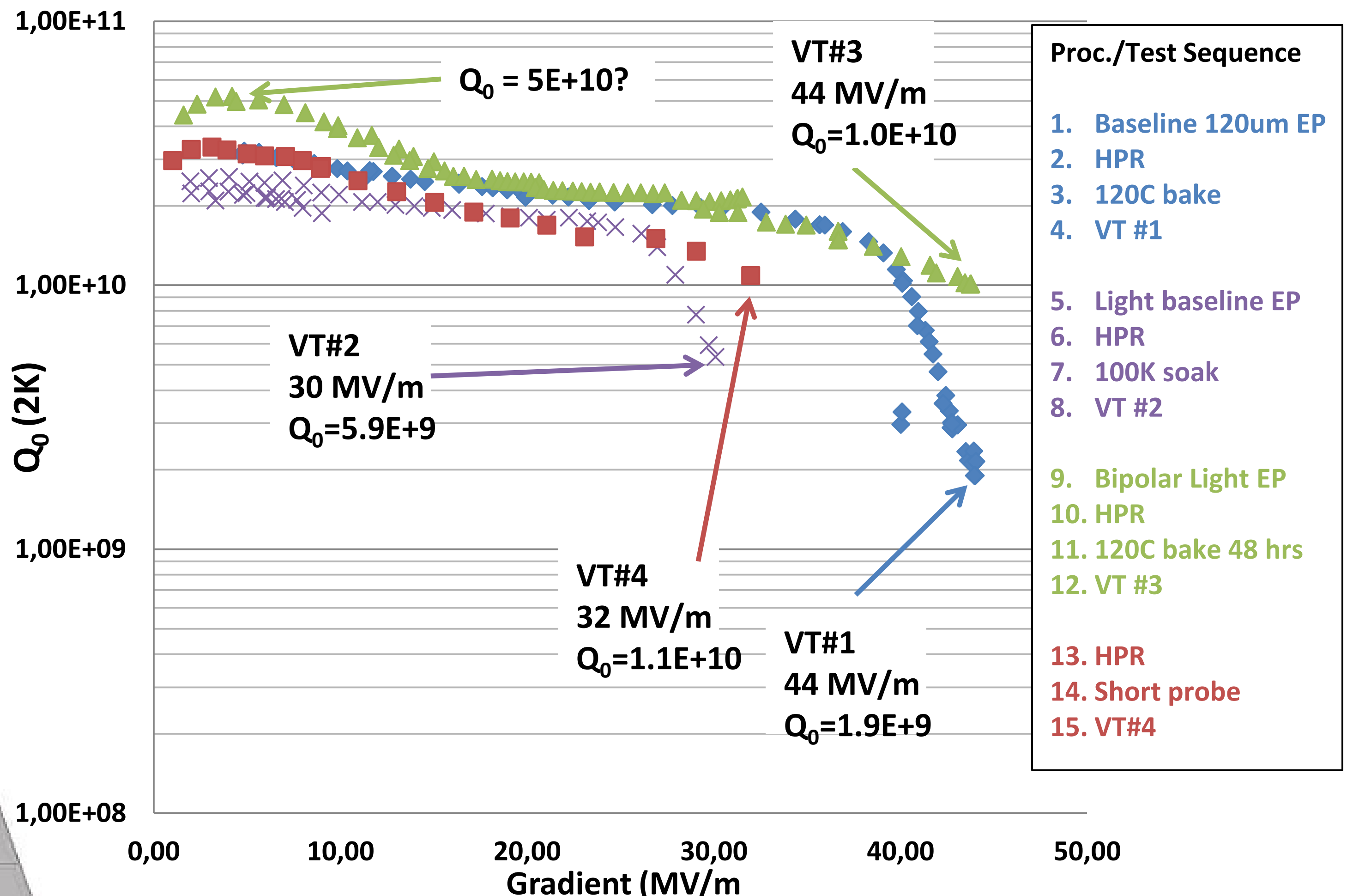
Performance Trial #4

- Goal: Evaluate whether light bipolar EP is capable of producing gradients > 35 MV/m at a Q_0 of 1E+10.
  - Process Sequence:
    - Baseline EP vertical test #1
    - Baseline light EP vertical test #2 (light surface damage repair + Q disease test)
    - Bipolar light EP
    - High pressure rinse and vertical test prep
    - 120 C bake
    - Vertical test #3 (standard probe) (Q_{ext} = 7.0E+09)
    - High pressure rinse and vertical test prep
    - Vertical test #4 (shortened probe) (Q_{ext} = 5.0E+10)

TE1AES012 Performance Results
Vertical Bipolar EP Light Polishing High Performance Test



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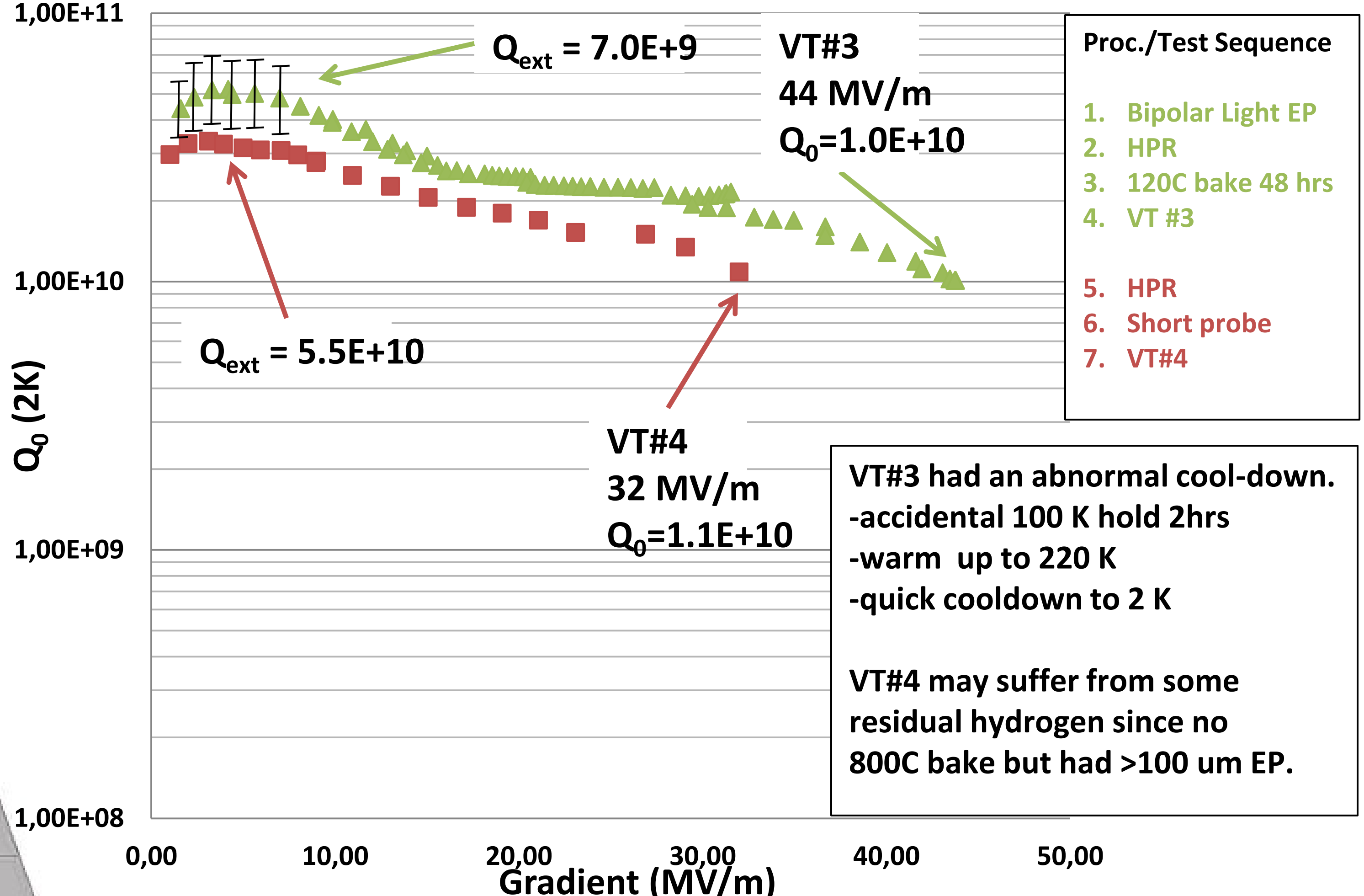
TE1AES012 Performance Results
Vertical Bipolar EP Light Polishing High Performance Test



CONCLUSIONS

- Bipolar EP, using a dilute aqueous H_2SO_4 electrolyte without HF yields equivalent RF performance with traditional EP.
- Bipolar EP behaves similarly to traditional EP in that bulk bipolar EP causes Q-disease, but light bipolar EP does not.
- Ecologically friendly and relatively benign electrolyte options for cavity processing exist.
- Bipolar EP needs to be developed for multi-cell cavities.
- Bipolar EP waveform trials need to be resumed to improve material removal rates.
- Industrial partners can provide new ideas and technologies that advance the field of SRF.

ACKNOWLEDGMENTS

- Co-authors

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REFERENCES

- [1] R. Geng, "Cavity String/Cryomodule Cold Test Experience at JLab", TTC Meeting, Beijing, China, December, 2011.
- [2] W. Singer, et al., "Superconducting 1.3 GHz Cavities for European XFEL", THAOR04, RuPAC2012 September 2012.
- [3] S. Aderhold, et al., "Cavity Process," ILC HiGrade Report 2010-005-1, April 2010.
- [4] H. Diepers et al., "A New Method of Electropolishing Niobium", Physics Letters, 37A number 2, (1971), 139-140.
- [5] S. Gerbick, et al., "A New Electropolishing System for Low-Beta SC Cavities," WEIOA03, SRF2011, September 2011.
- [6] C. Cooper, et al., "Centrifugal Barrel Polishing (CBP) of SRF Cavities Worldwide," WEIOA02, SRF2011, September 2011.
- [7] A. Palczewski et al., "Optimizing Centrifugal Barrel Polishing for Mirror Finish SRF Cavity and RF Tests at Jefferson Lab," WEPPC094, IPAC2012, July 2012.
- [8] Honeywell, Inc., "Honeywell Hydrofluoric Acid Medical Brochure 2012", Honeywell Document Database, http://www51.honeywell.com/sm/hfacid/common/documents/Honeywell-HF-Medical-Brochure-2012-final.pdf
- [9] V. Palmieri, et al., "Niobium Electropolishing by Ionic Liquids: What are the Naked Facts?", THOAAU03, SRF2009, September 2009.
- [10] M. Pekeler, et al., "Development and RF Test Results of a New HF and H_2SO_4 Free Electropolishing Method for Superconducting Niobium Cavities," TUP44, SRF2007, September 2007.
- [11] M. Inman, E. J. Taylor, A. Lozano-Morales, T. D. Hall, and H. M. Garich, Electrochemical System and Method for Machining Strongly Passivating Metals U.S. Patent Appl. No. 13/153,874 Jun. 11, 2010.
- [12] E. Taylor, "Electropolishing of Niobium SRF Cavities in Low Viscosity Electrolytes without Hydrofluoric Acid", TUP054, SRF2013, September 2013.
- [13] M. Inman, et al., "Electropolishing of Passive Materials in HF-Free Low Viscosity Aqueous Electrolytes", Journal of the Electrochemical Society, 160 (9), E94-E98 (2013).
- [14] Fermilab Request for Proposal, "Eco-Friendly Polishing", ID Number: 212864RE Rev #2, January 2010.
- [15] M. Kelly, et al., "Surface Processing Facilities for Superconducting RF Cavities at ANL," THP026, LINAC08, November 2009.
- [16] J. Knobloch, et al., "Enhanced Susceptibility of Nb Cavity Equator Welds to the Hydrogen Related Q-virus", Eighth Workshop on RF Superconductivity, October 1997.