Types of Chemical and Mechanical Surface Processing for SRF Cavities

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USPAS Course:

SRF Technology: Practices and Hands-On Measurements

January 2015

Established Procedures

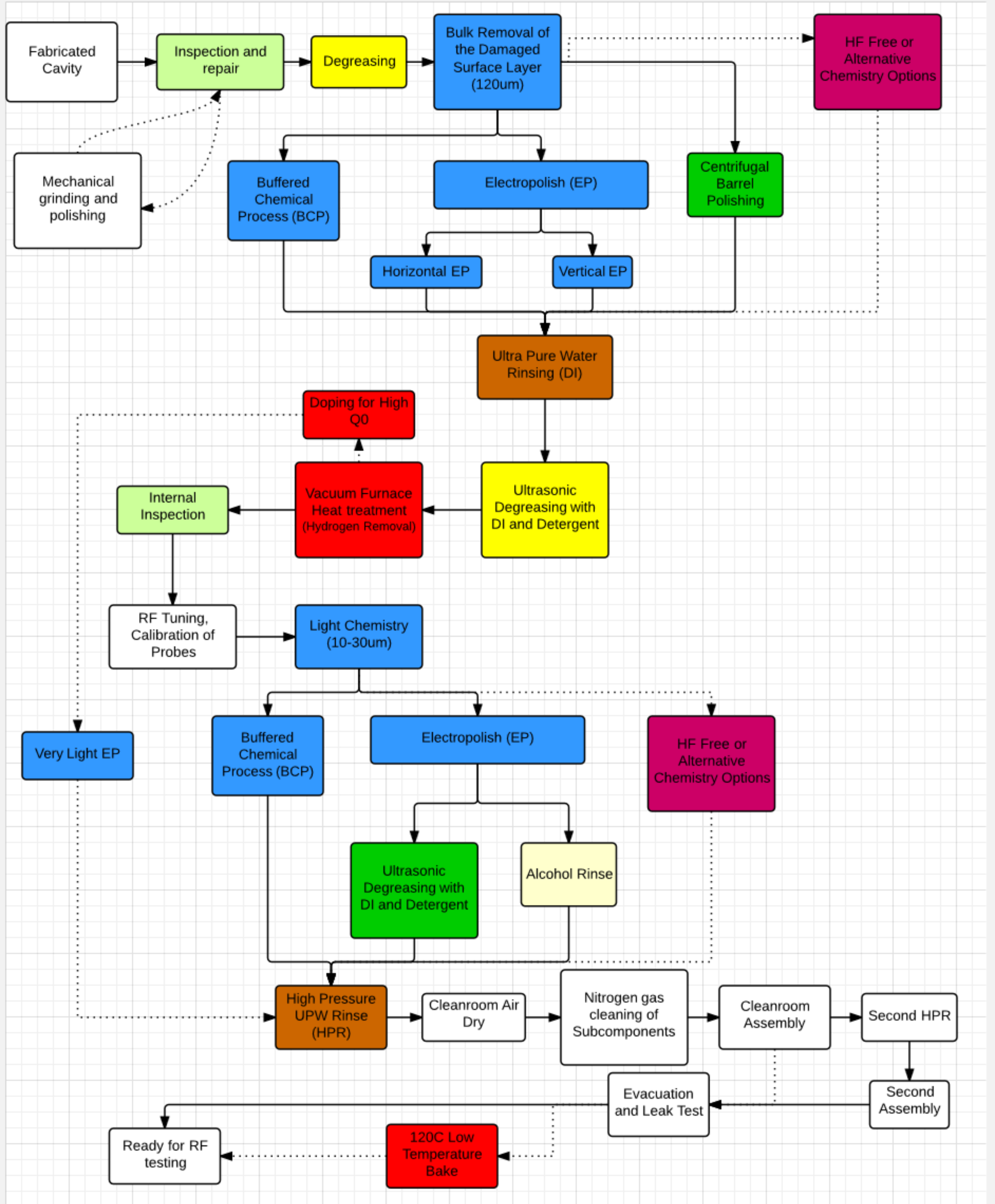
- Preparation of Cavity Surfaces
  - Mechanical Removal
    - Mechanical Grinding
  - Chemical Removal
    - Buffered Chemical Polish (BCP)
    - Electropolish (EP) Horizontal or Vertical
- Surface Cleaning Methods
  - Ultrasonic Degreasing
  - High pressure water (HPR)
  - Nitrogen Gas Cleaning
- Vacuum Treatments
  - Heat Treatment High Temperature
  - Low Temperature Baking

Procedures Under Development

- Centrifugal Barrel Polishing (CBP)
- Nitrogen Doping
- HF Free Chemistry
- Dry Ice Cleaning (DIC)
- Dry Chemistry

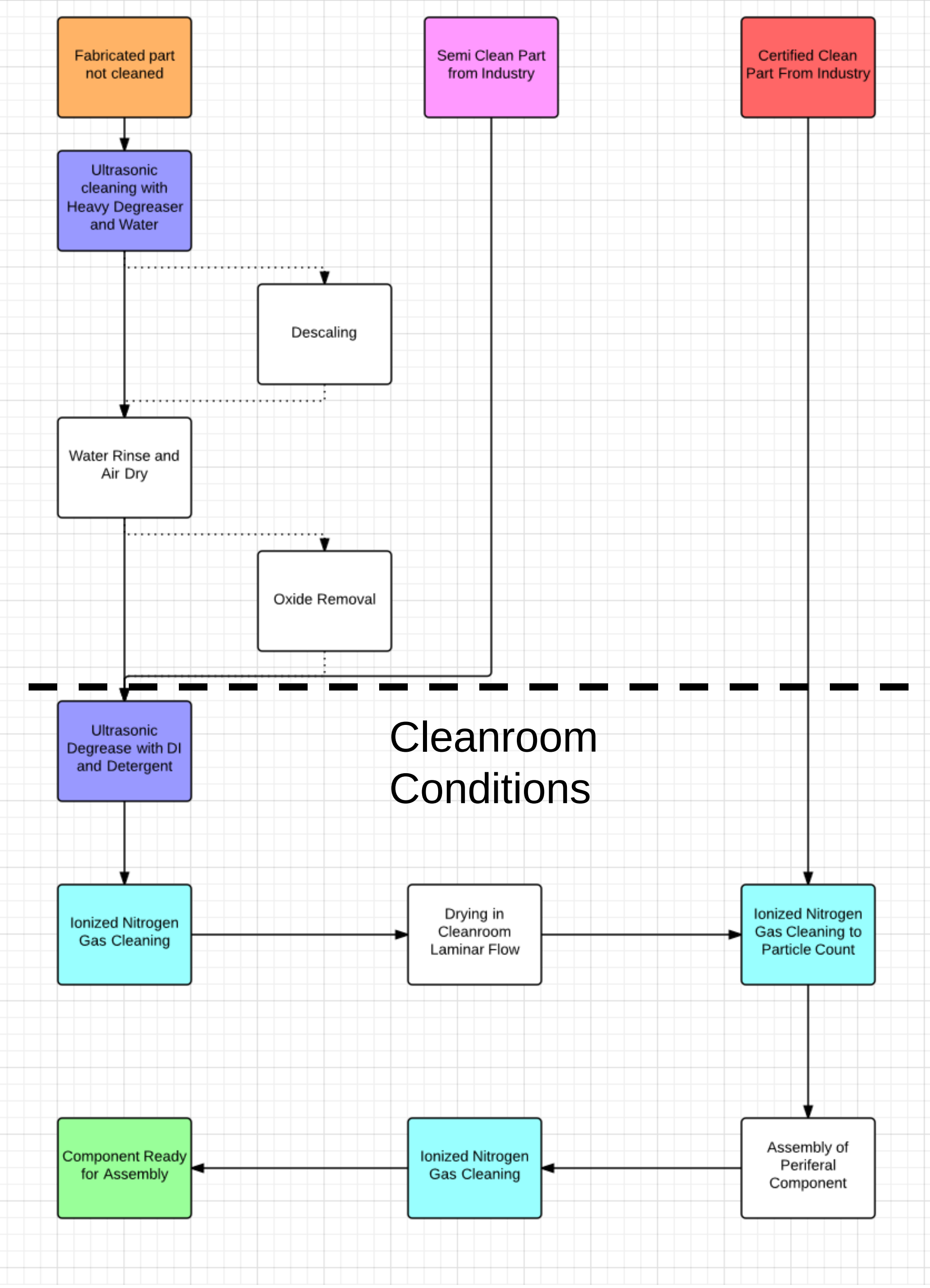
Cavity Preparation Steps for Performance Qualification

- Baseline Processes
  - Typical steps taken for most cavities
- Optional Paths
  - Multiple options are available and depend on the performance requirements
- Alternative Processes
  - New processes underdevelopment that have shown good results and some benefits for the performance or cost

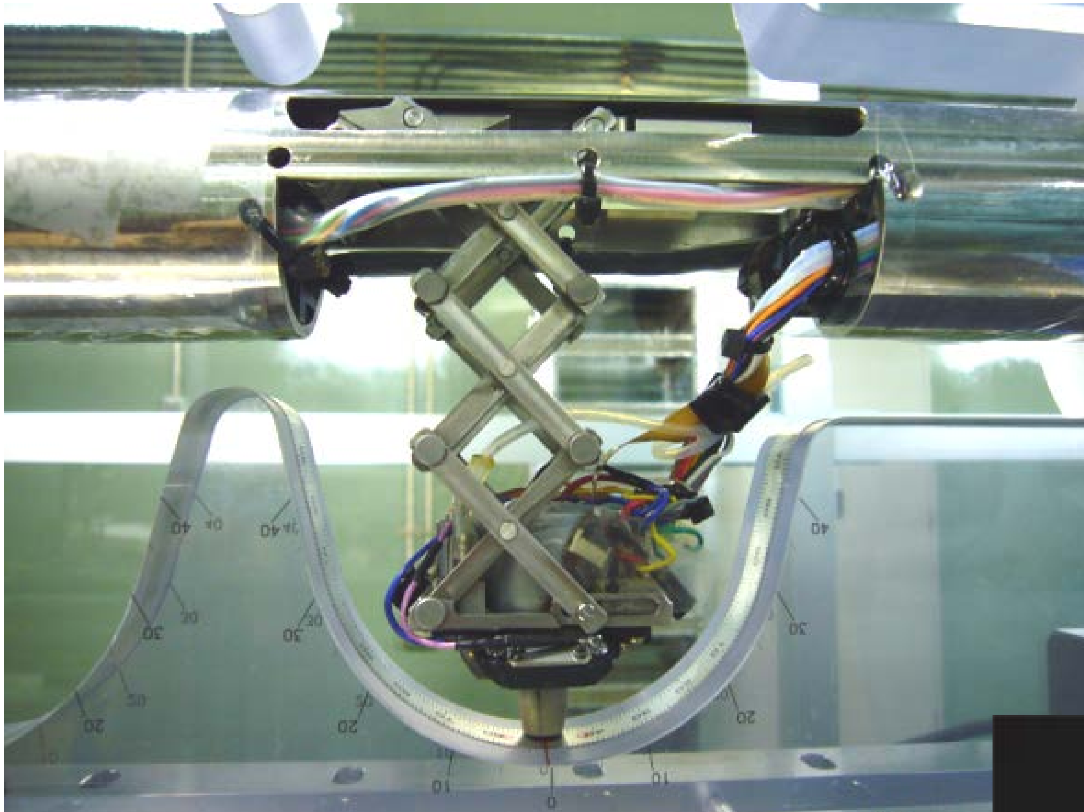


Peripheral Component Cleaning

- Fabricated parts not clean
  - Descaling
  - Degreasing
  - Chip removal
- Semi Clean Parts from Industry
  - Degreaseing
  - Removal of oxides
  - Removal of Particulates
- Certified Clean Parts From Industry
  - Removal of Particulates



Guided Mechanical Grinding



- Articulated Rotary Tool
- Abrasive Material
- Camera and Light

K. Watanabe, KEK

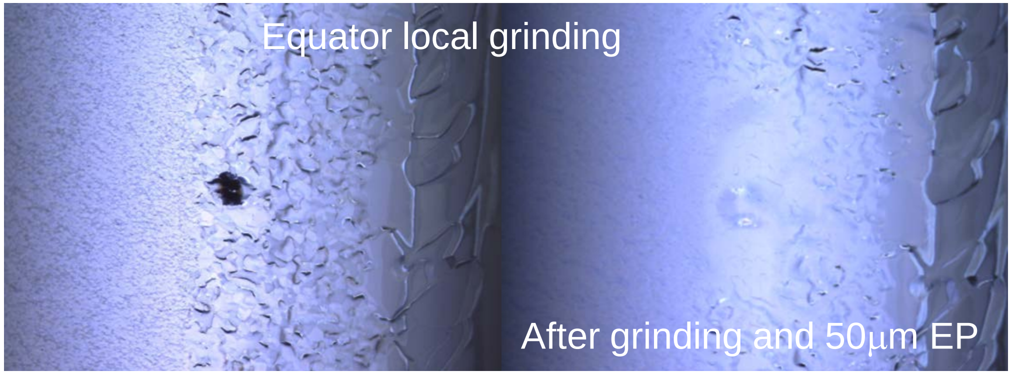
Hand Grinding and Local Repairs

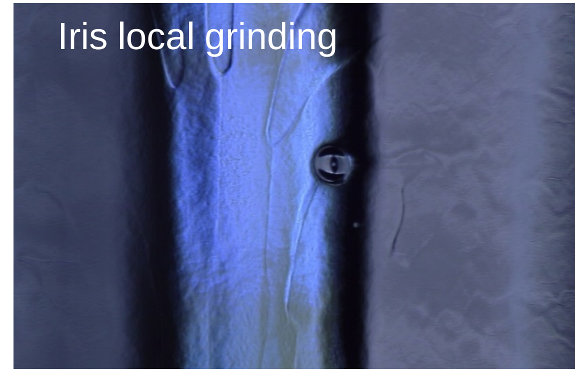
3M Abrasive Wheels (scotch brite)

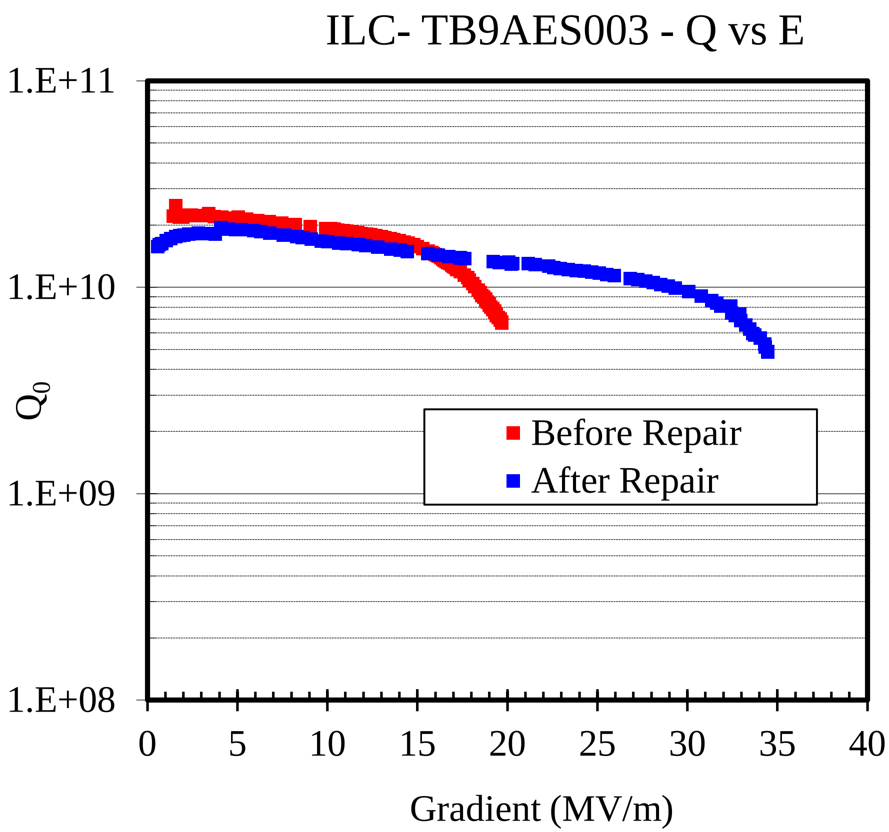
Hand Held Rotary Tool

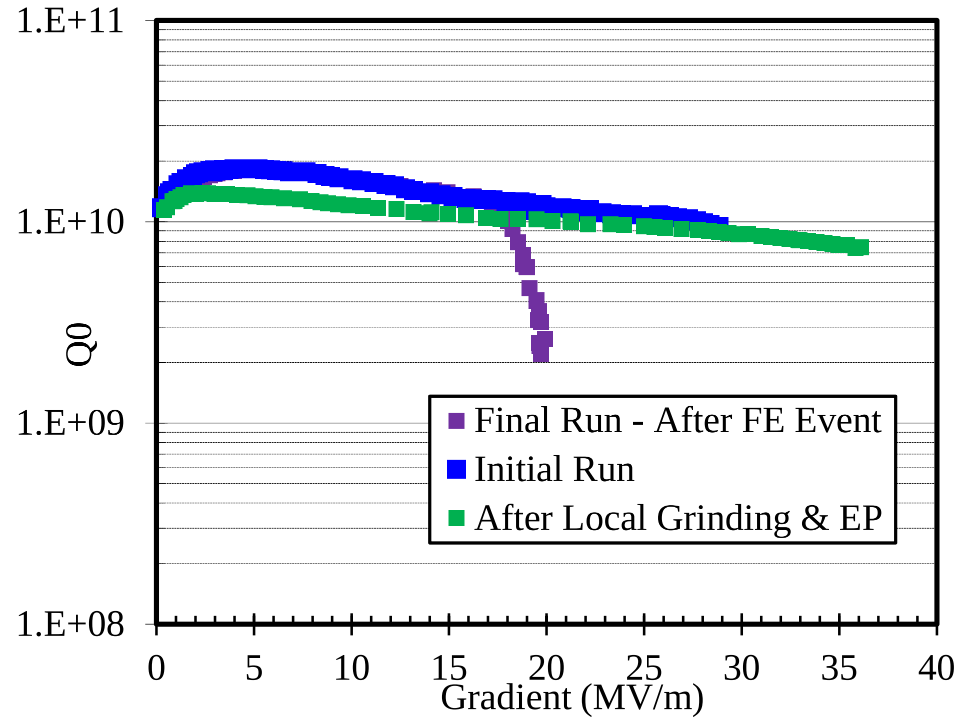
- Compressed air
- Electric
- Part Held in Place
- Light and Camera

Localized Mechanical Grinding is Effective!

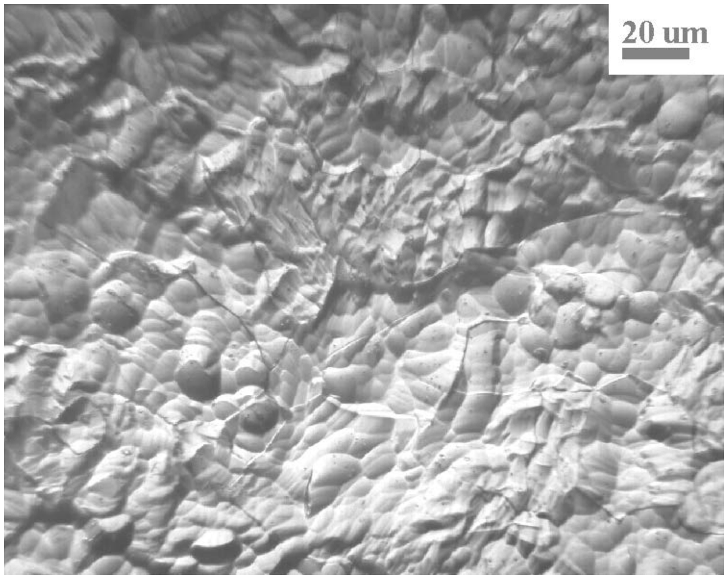


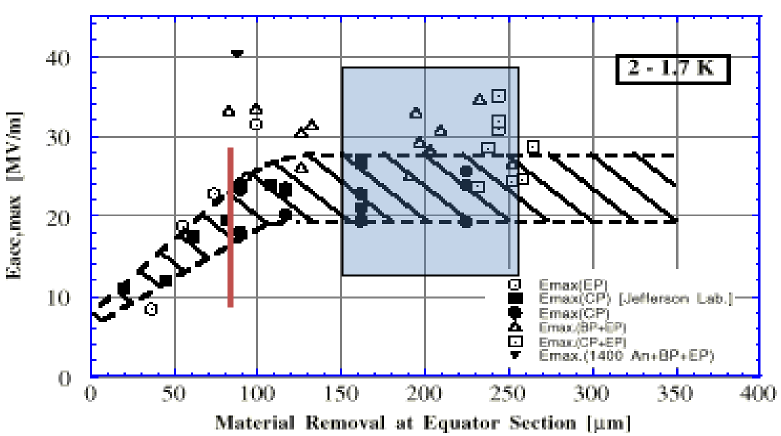




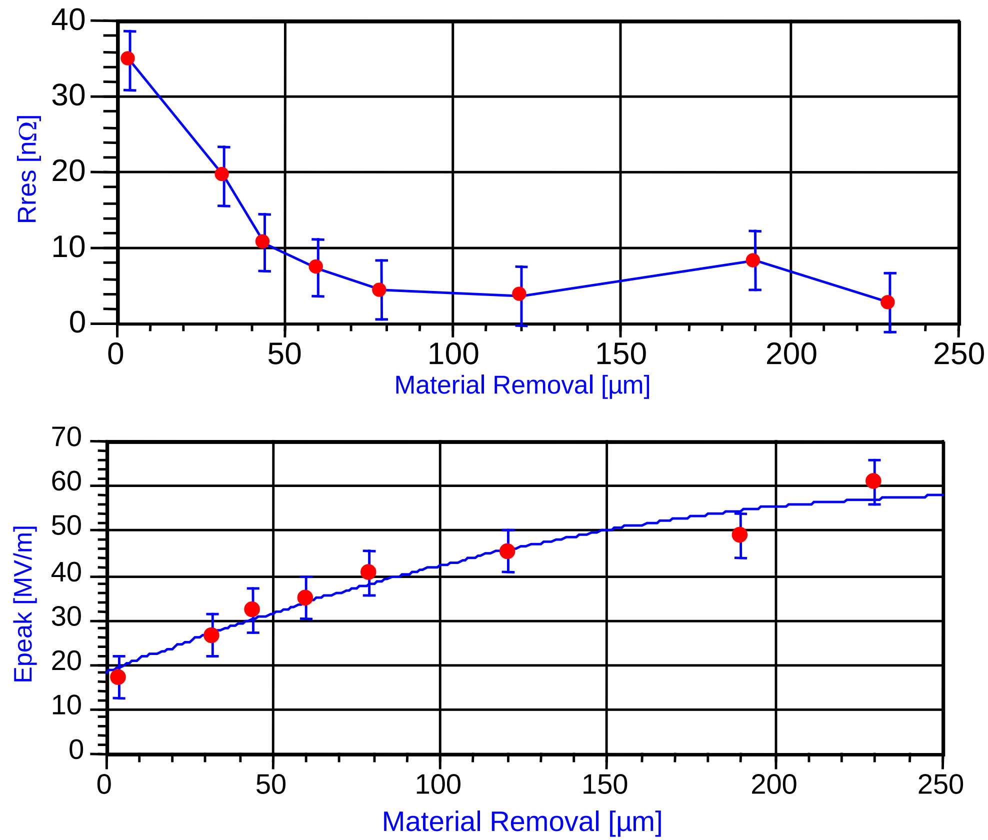


The Need For Material Removal



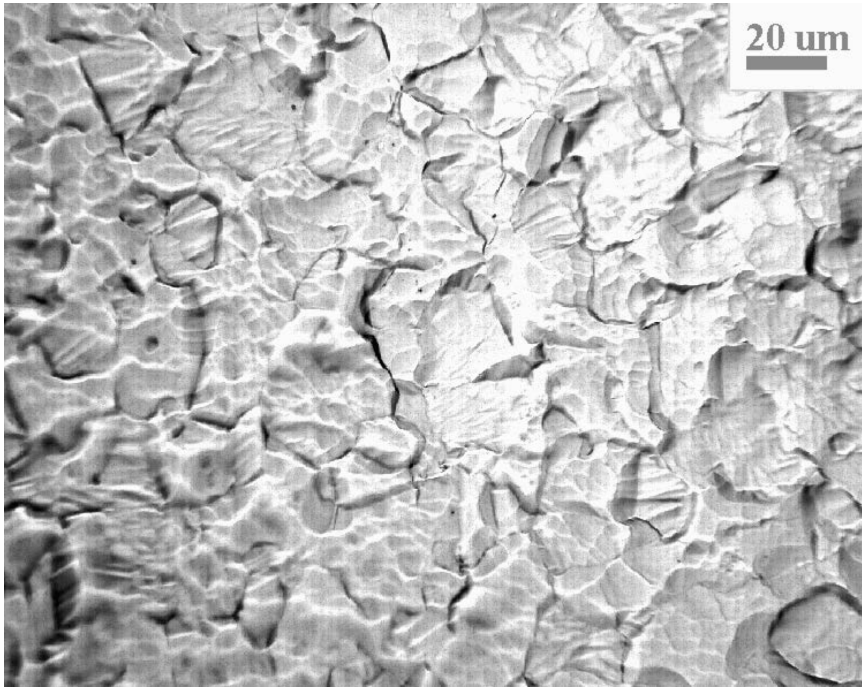


K. Saito

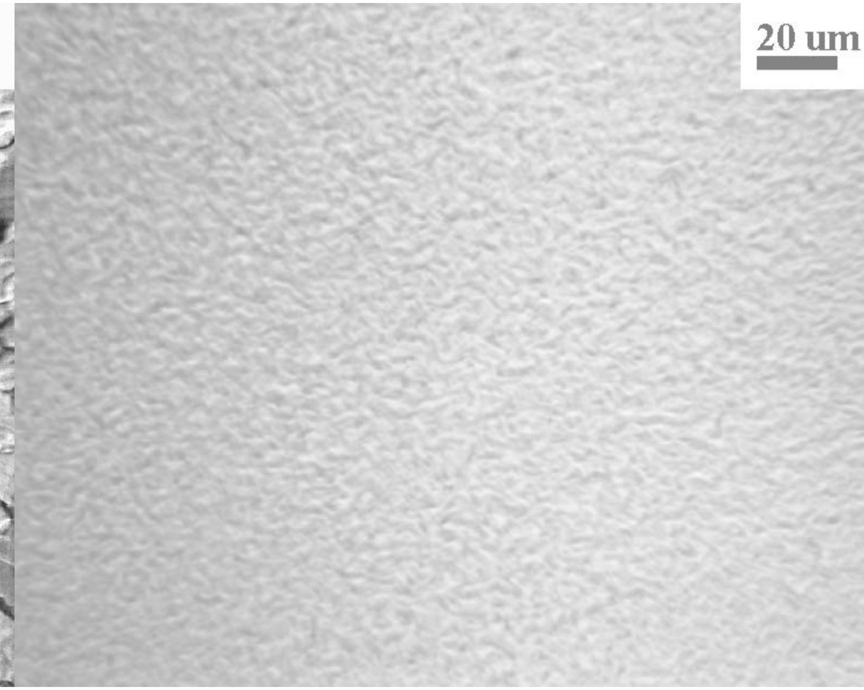


P. Kneisel

Niobium Material Removal by Chemistry



Niobium surface after BCP



Niobium surface after EP

Buffered Chemical Polish (BCP)

Acid (Reagent Grade)

HF (49% w/w),

HNO_3 (65% w/w),

H_3PO_4 (85% w/w)

Typical Mixture
1:1:1 etching subcomponents
or 1:1:2 etching structures

Reaction:

Forms NO_2 , Orange Brown Gas

Oxidation

$$6\ Nb + 10\ HNO_3 \leftrightarrow 3\ Nb_2O_5 + 10\ NO + 5\ H_2O$$

Reduction

$$3\ Nb_2O_5 + 18\ HF \leftrightarrow 3\ H_2NbOF_5 + 3\ NbO_2F + 6\ H_2O$$

Insoluble

$$3\ NbO_2F + 12\ HF \leftrightarrow 3\ H_2NbOF_5 + 3\ H_2O$$

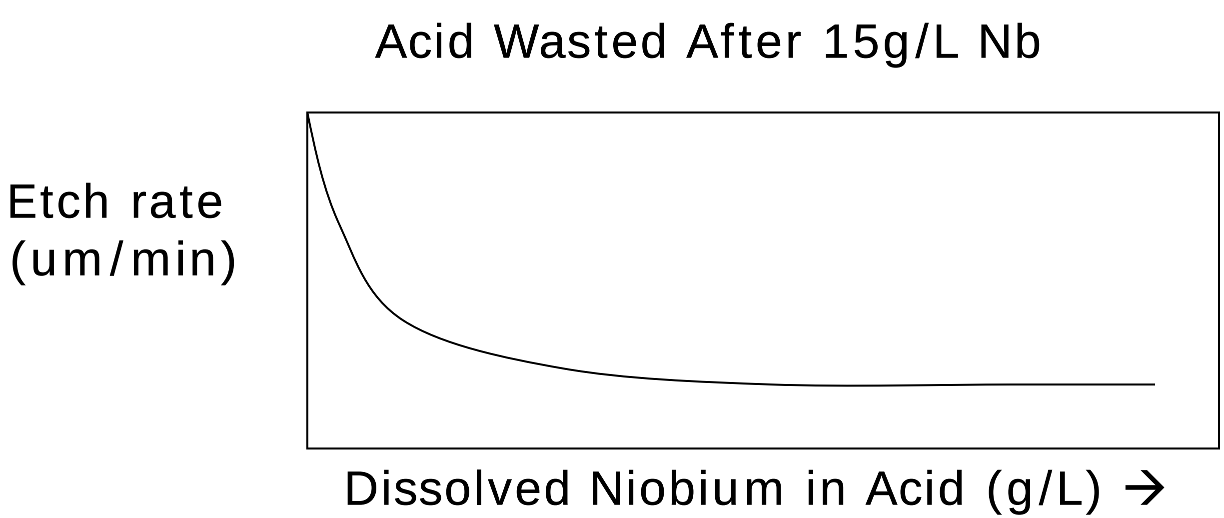
I. Malloch etal. , FRIB

What parameters are Important for Cavity Etching by BCP?

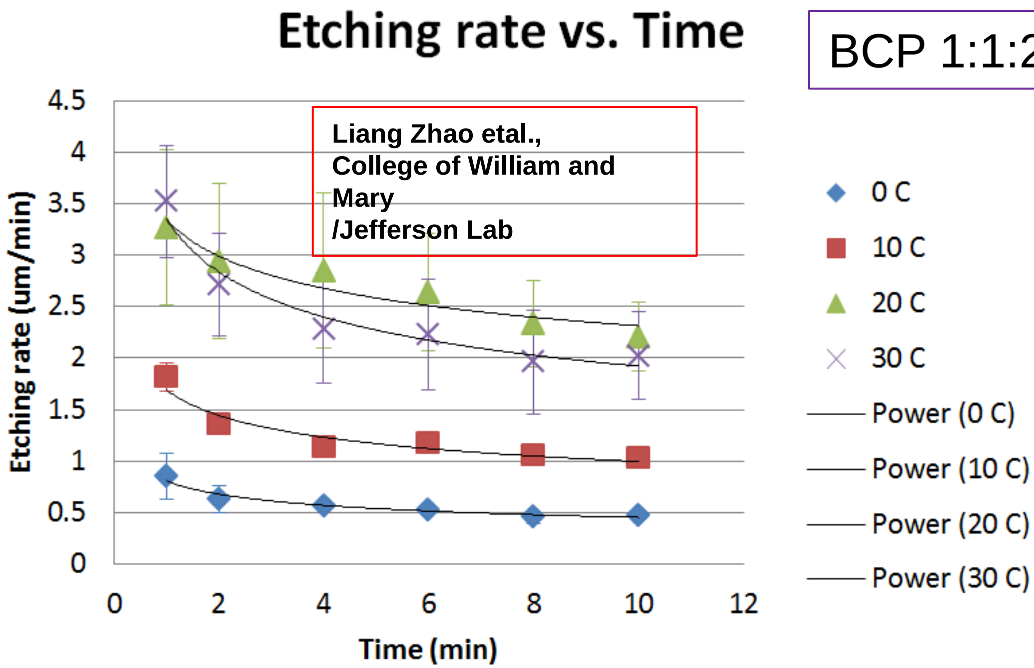
- **Temperature, Time**
- **Acid Velocity and Distance from Inlet,**
- **Grain Size and Grain Orientation**
- **Gas Bubble Evolution and Control**
- **Acid Contamination,**

Use of BCP:

- **1:1:1 still used for etching of subcomponents - Reduces Time** (etch rates of 8-10 um/min)
- **1:1:2 used for most cavity treatments**
  - Mixing necessary → reaction products at surface
  - Acid is usually cooled to 10-15C (1-2um/min) to control the reaction rate and Nb surface temperatures (reduce hydrogen absorption)



Temperature effect - Etching rate



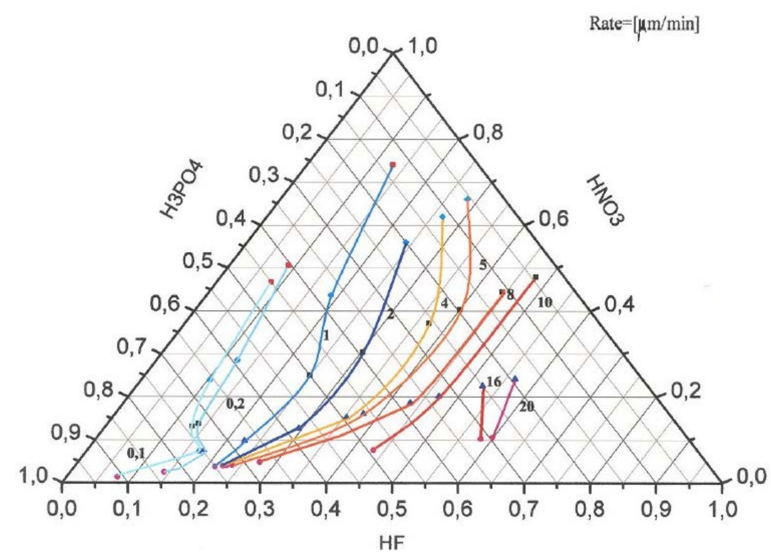
BCP 1:1:2

- ➢Etching rate – etching time curves exhibit power relation.
- ➢Etching rate, $R = kt^{-0.2}$

Temperature C	k
0	0.8
10	1.7
20	3.3
30	3.4

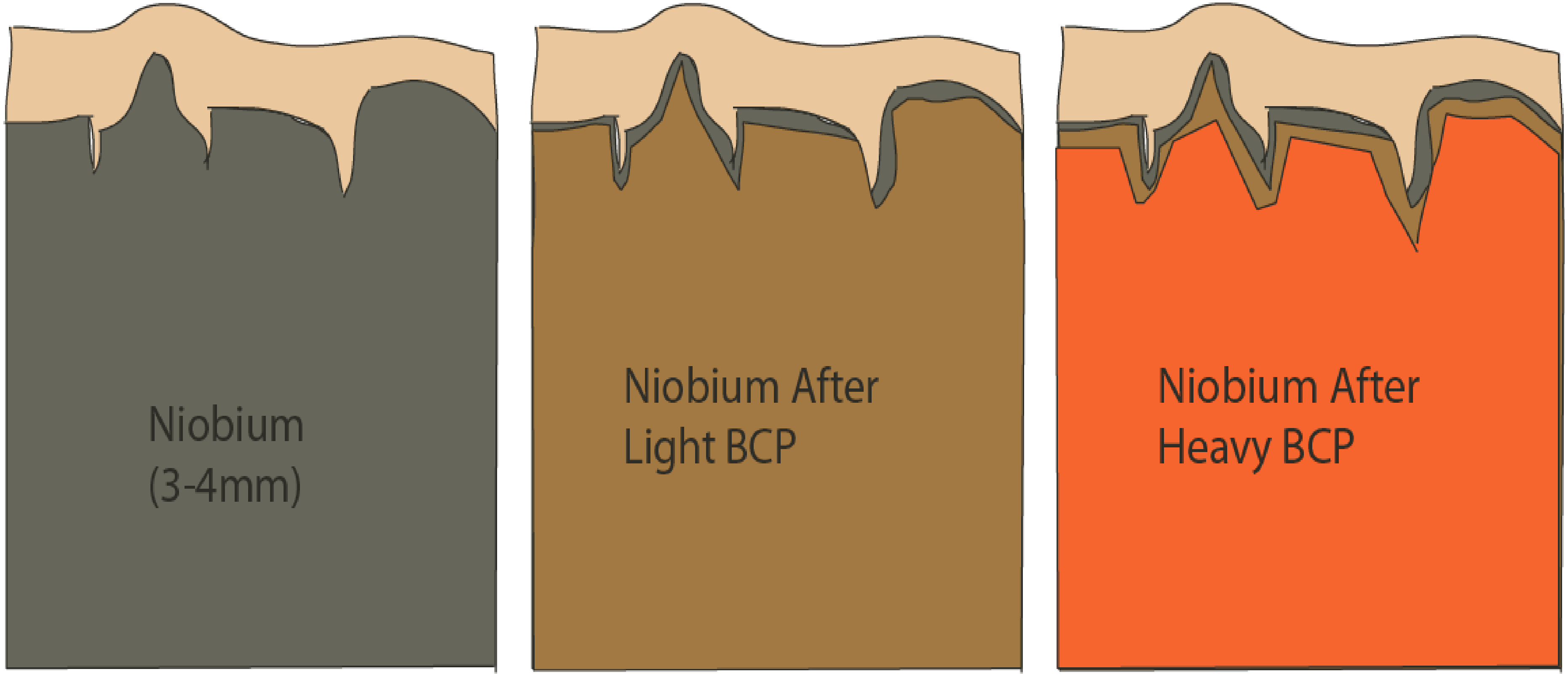
Concentration effect on Etch Rate

Ternary Diagram of Etch Rate vs Percentages of Individual Acids



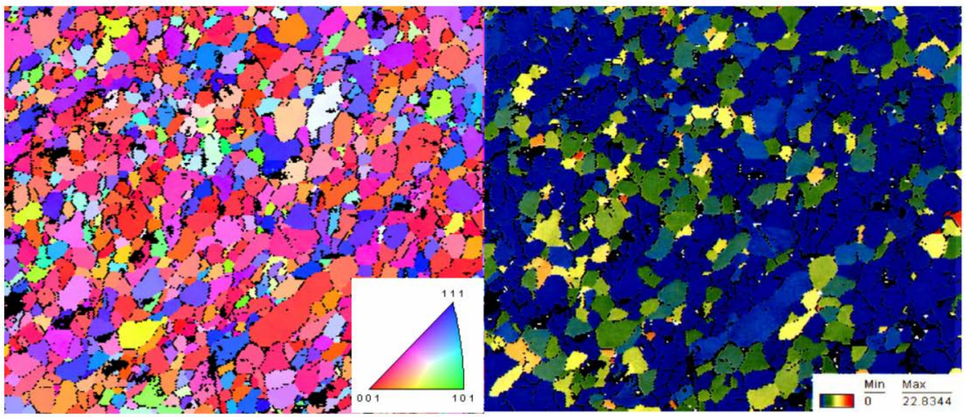
V. Palmieri, INFN

Effects of BCP on The Niobium Surface



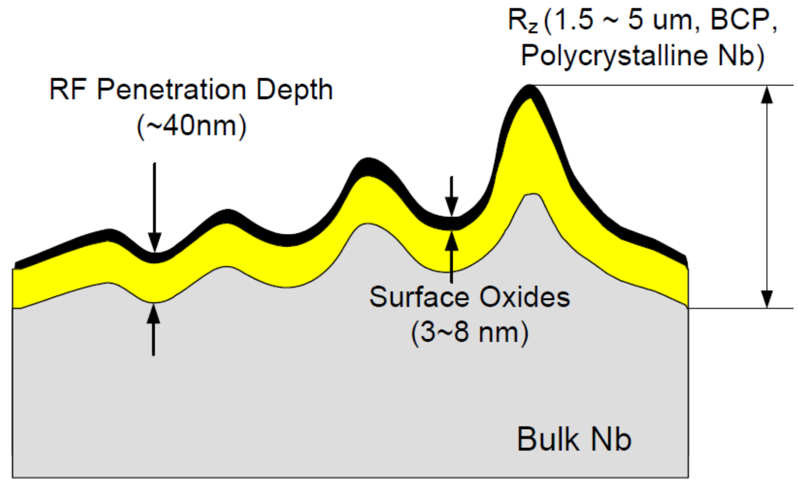
Polycrystalline Niobium Material

Electron Backscattering Diffraction Clearly Revealed Surface Grains Sizes & Orientations aft. BCP



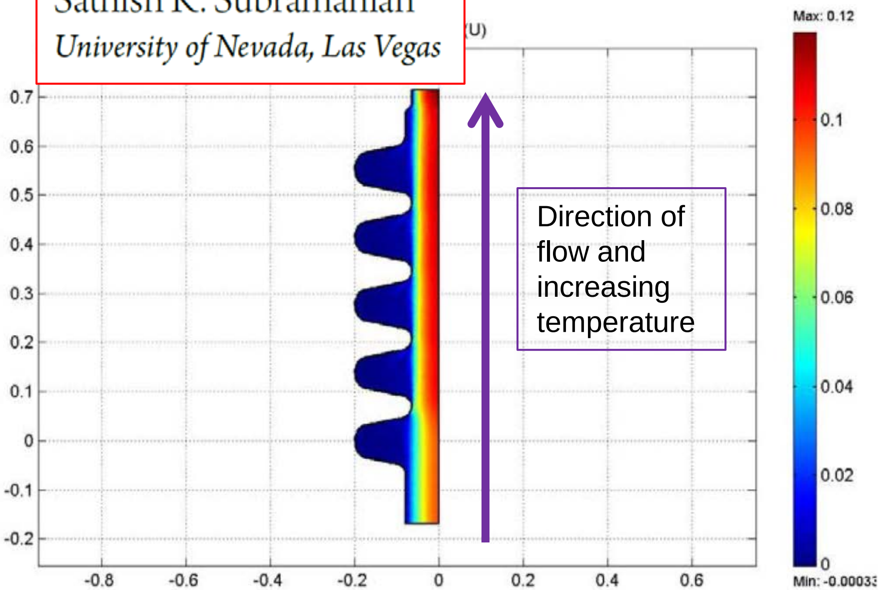
Grain orientation & mis-orientation deviation map(1.5 mm * 1.5 mm)

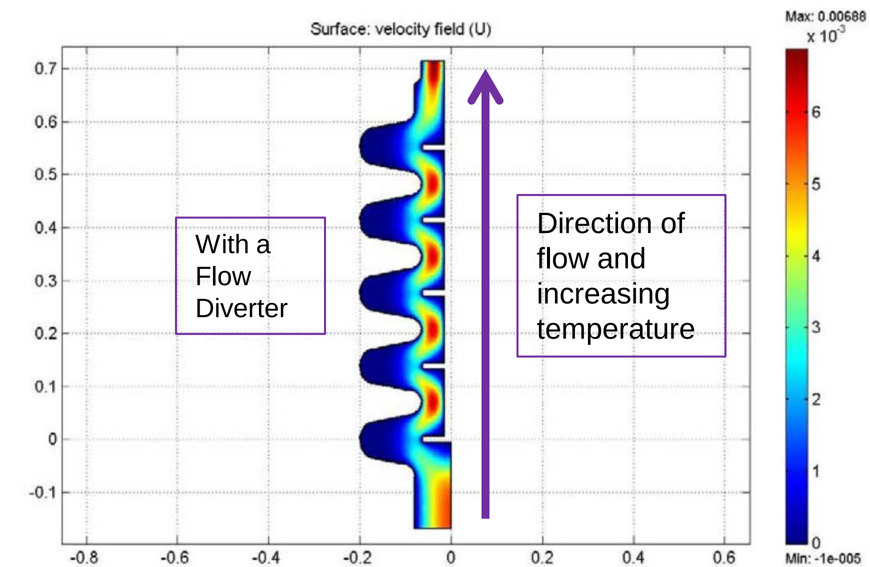
Hui Tian, JLab



Acid Flow and Its Effects

Sathish K. Subramanian
University of Nevada, Las Vegas





Important factors with vertically etching of SRF cavities:

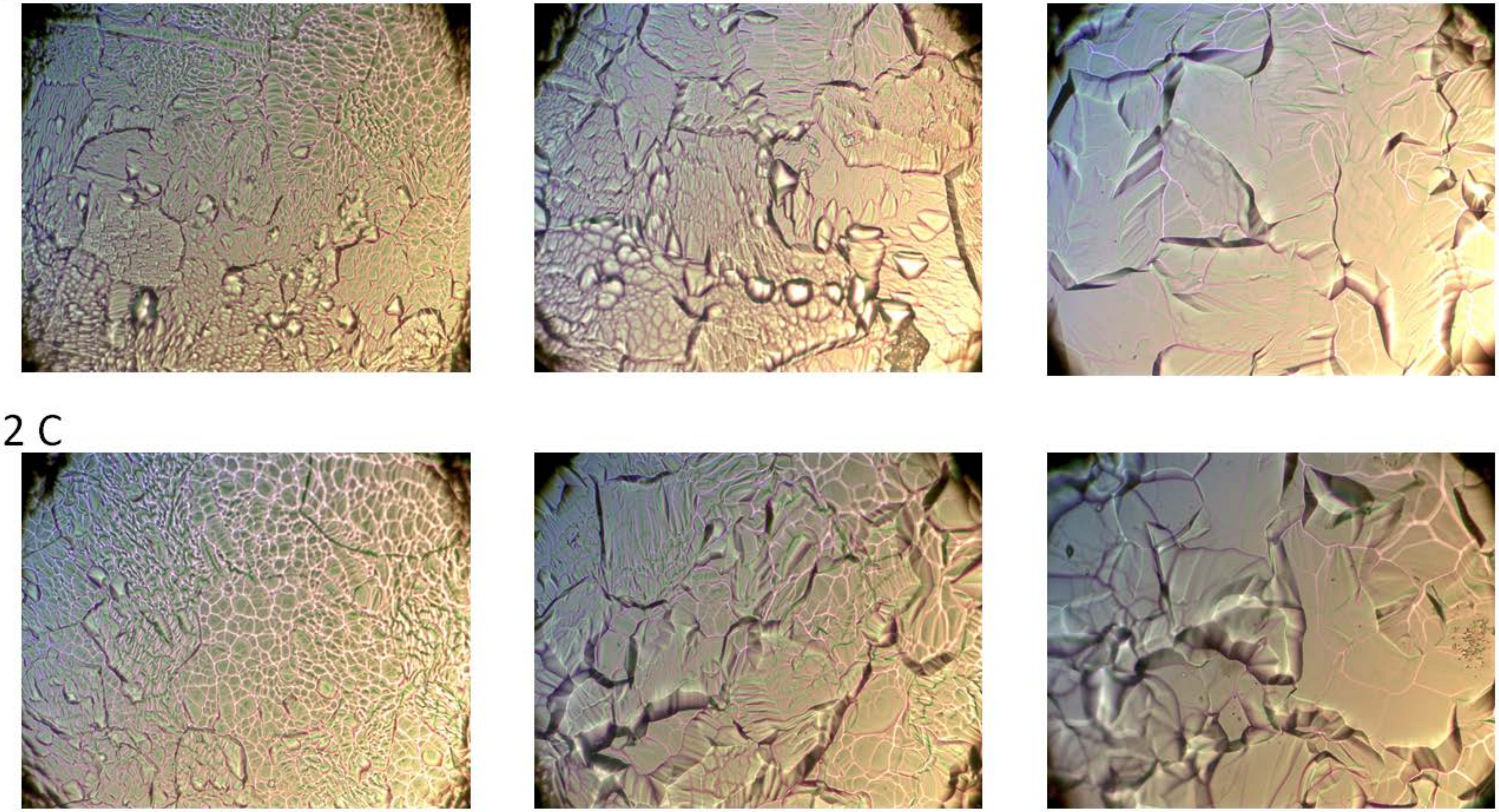
- Temperature gradient forms from bottom to top which gives an increased material removal in the direction of flow
- Higher flow velocities increase etch rate
  - Therefore Iris etches more than equator by a factor of 1.3-2.0 times EQ
  - EQ has very little mixing and therefore increases temperature

BCP Temperature and Etch Time Effect On Surface Topography

400X Magnification

10 C 1min 7min 20min

22 C



Etching Rate is Grain Orientation Dependent

Liang Zhao etal.,
College of William and Mary
/Jefferson Lab

(BCP) Systems for Cavity Etching:

- Bulk & Final chemistry
  - Bulk removal of (100-200um)
  - Final removal of (5-20um) to remove any additional damage from QA steps and produce a fresh surface

Implementation:

- Cavity held vertically
- Closed loop flow through style process, some gravity fed system designs
- Etch rate 2X on iris then equator
- Temperature gradient causes increased etching from one end to the other
- Manually connected to the cavity but process usually automated

Electropolish (EP)

Electrolyte = 1 part HF(49%), 9 parts H_2SO_4 (96%)

Hydrogen Gas

Reaction:

Oxidation

$$2Nb + 5SO_4^{2-} + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^-$$

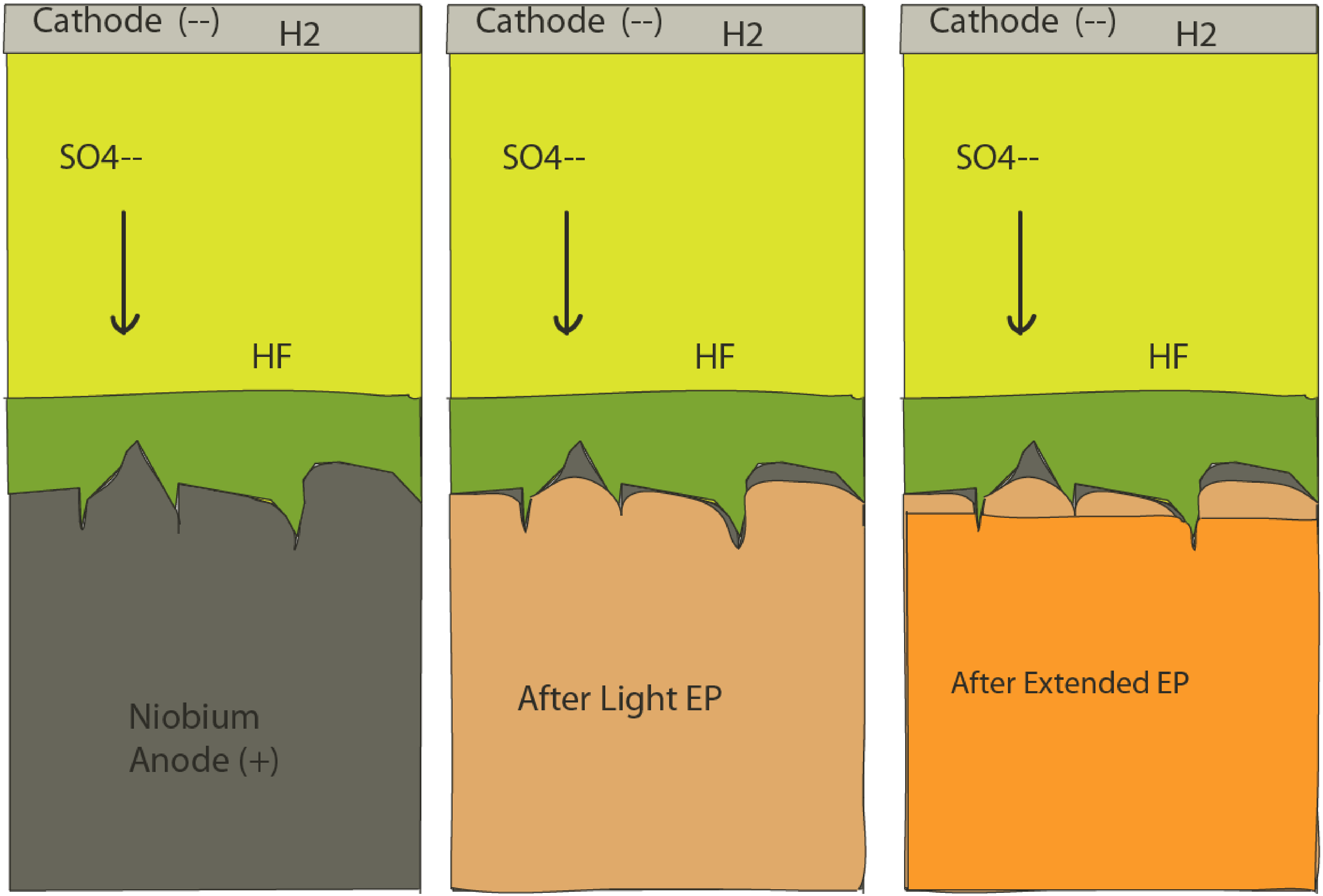
Reduction

$$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F\ 0.5H_2O + 1.5H_2O$$

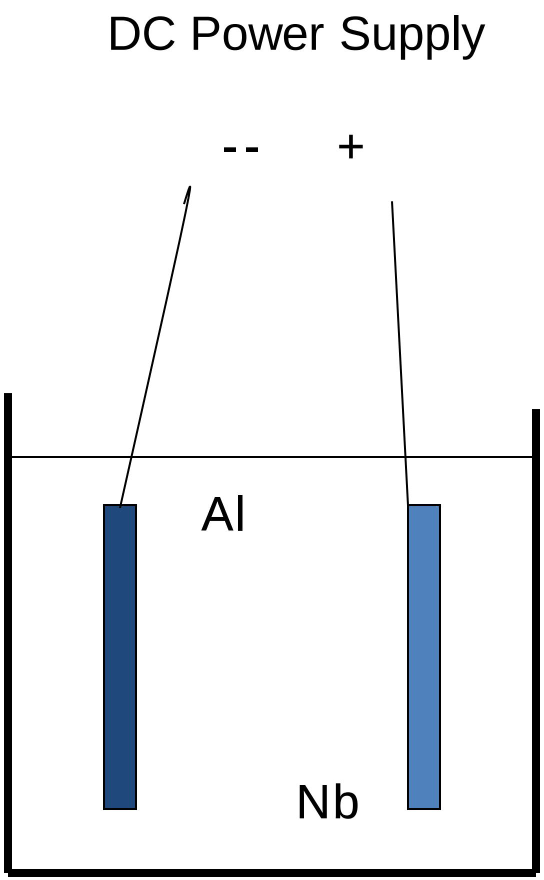
$$NbO_2F\ 0.5H_2O + 4HF \rightarrow H_2NbOF_5 + 1.5H_2O$$

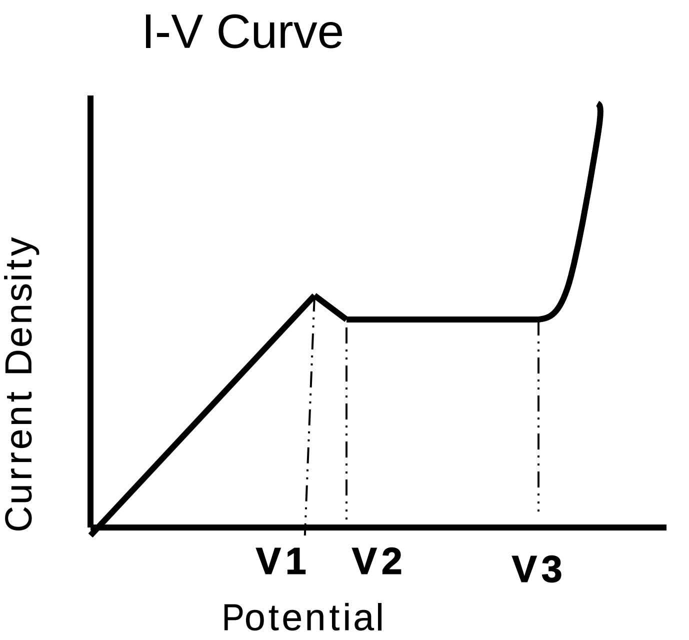
These are not the only reactions that take place!

Nb Surface Effects After EP



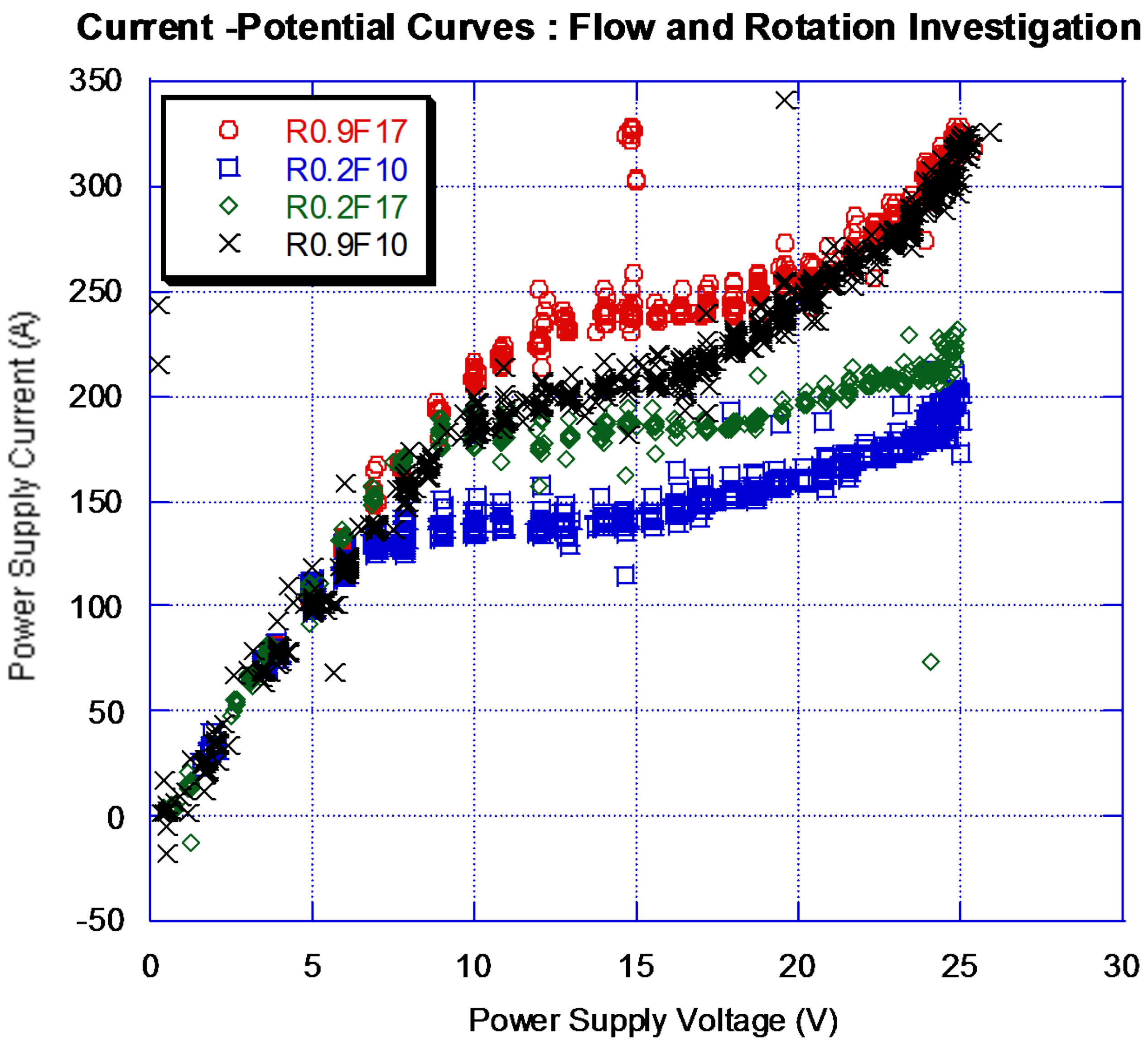
Basic Concepts of EP



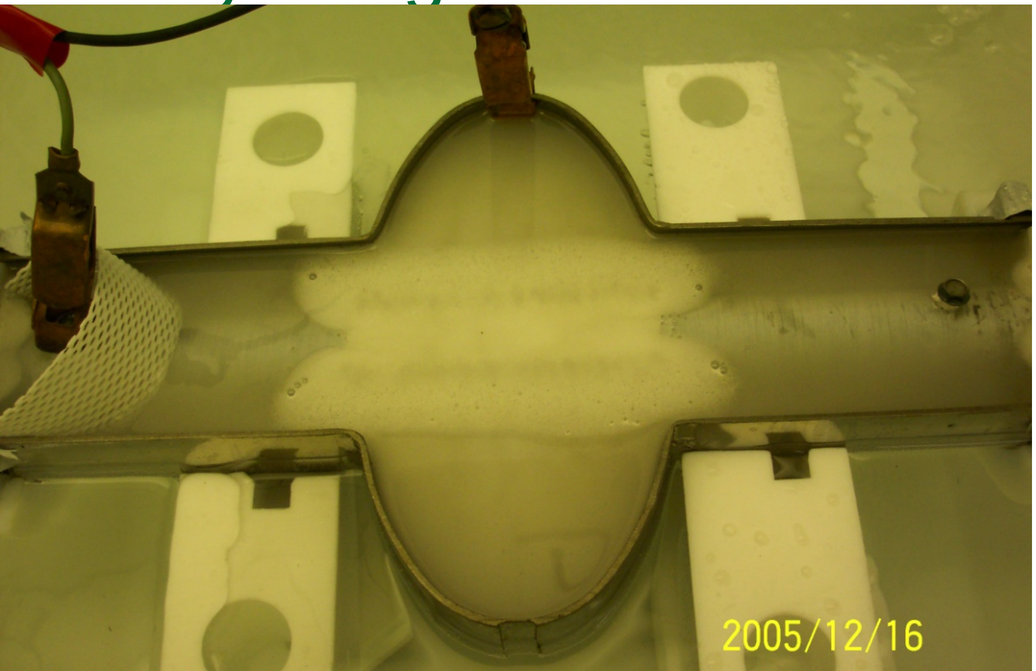


- **0-V1- Concentration Polarization occurs, active dilution of niobium, electrolyte resistance**
- **V2-V3 – Limiting Current Density, viscous layer on niobium surface**
- **>V3 Additional Cathodic Processes Occur, oxygen gas generated**

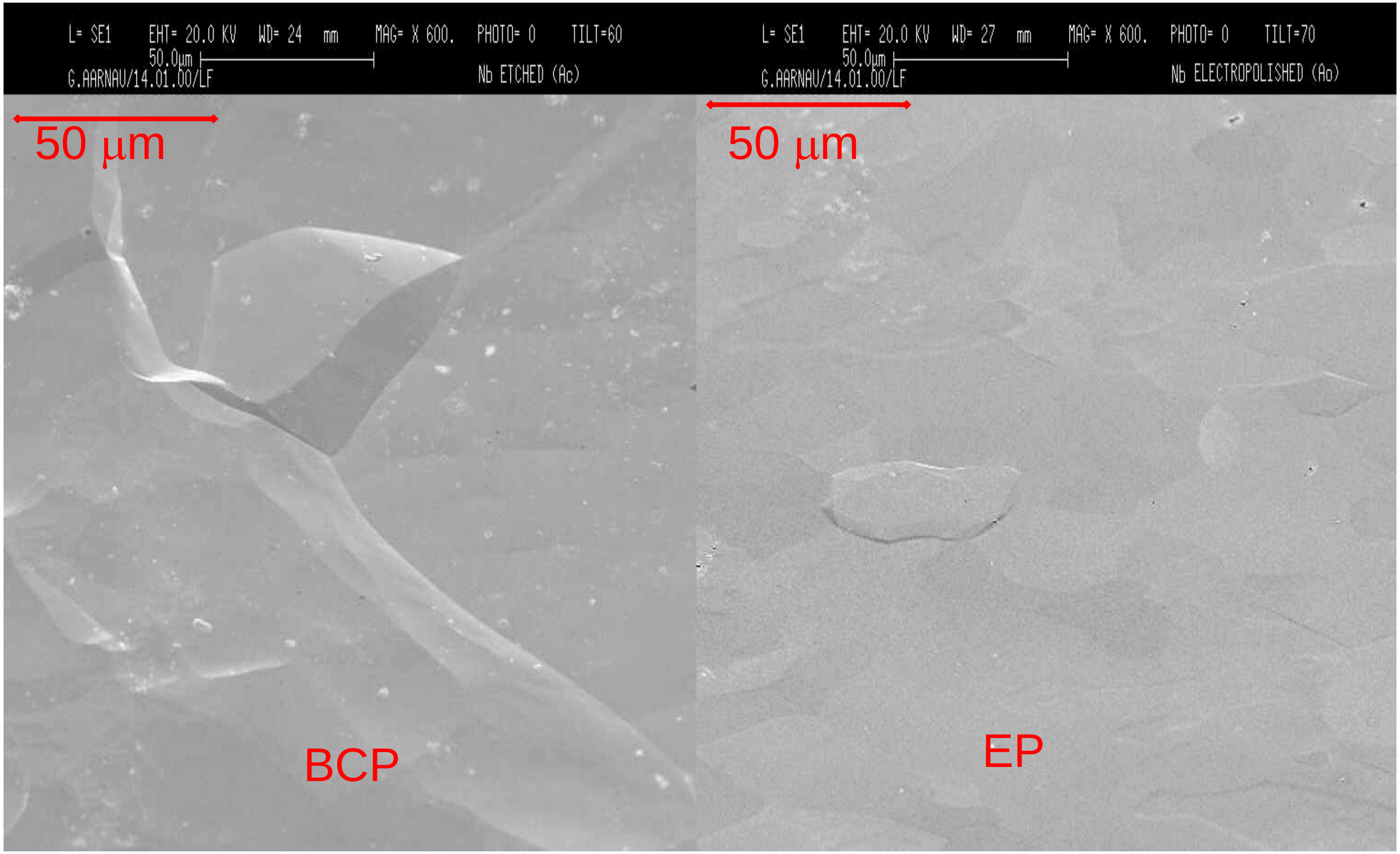
Cavity IV Curve not easy to interpret



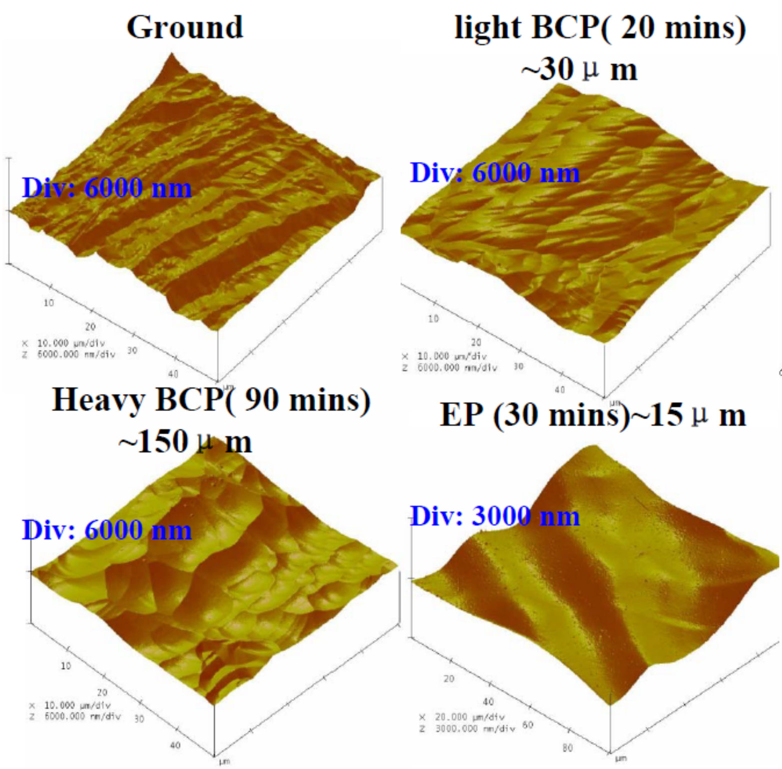
Hydrogen Gas Shielding Experiment

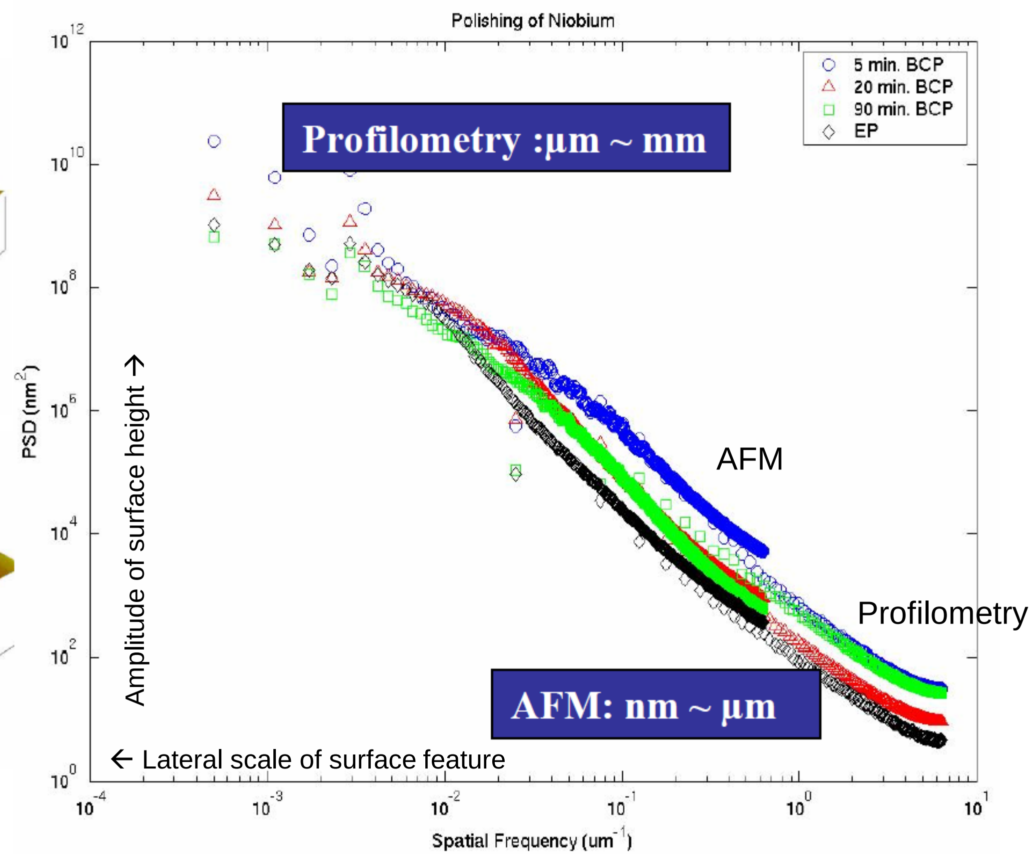


Surface Roughness of Niobium



Surface Topography of Niobium Samples after BCP/EP Treatment

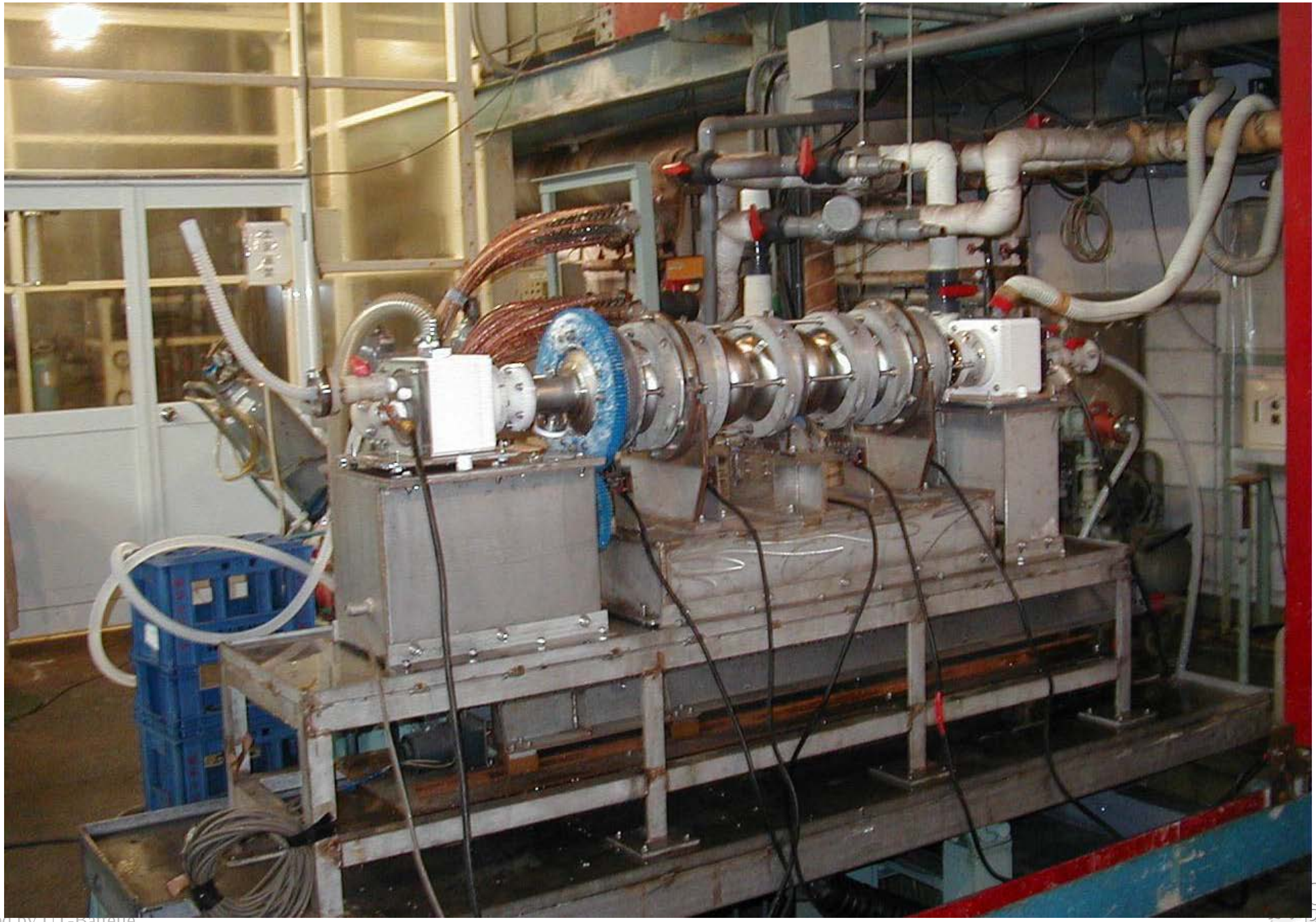




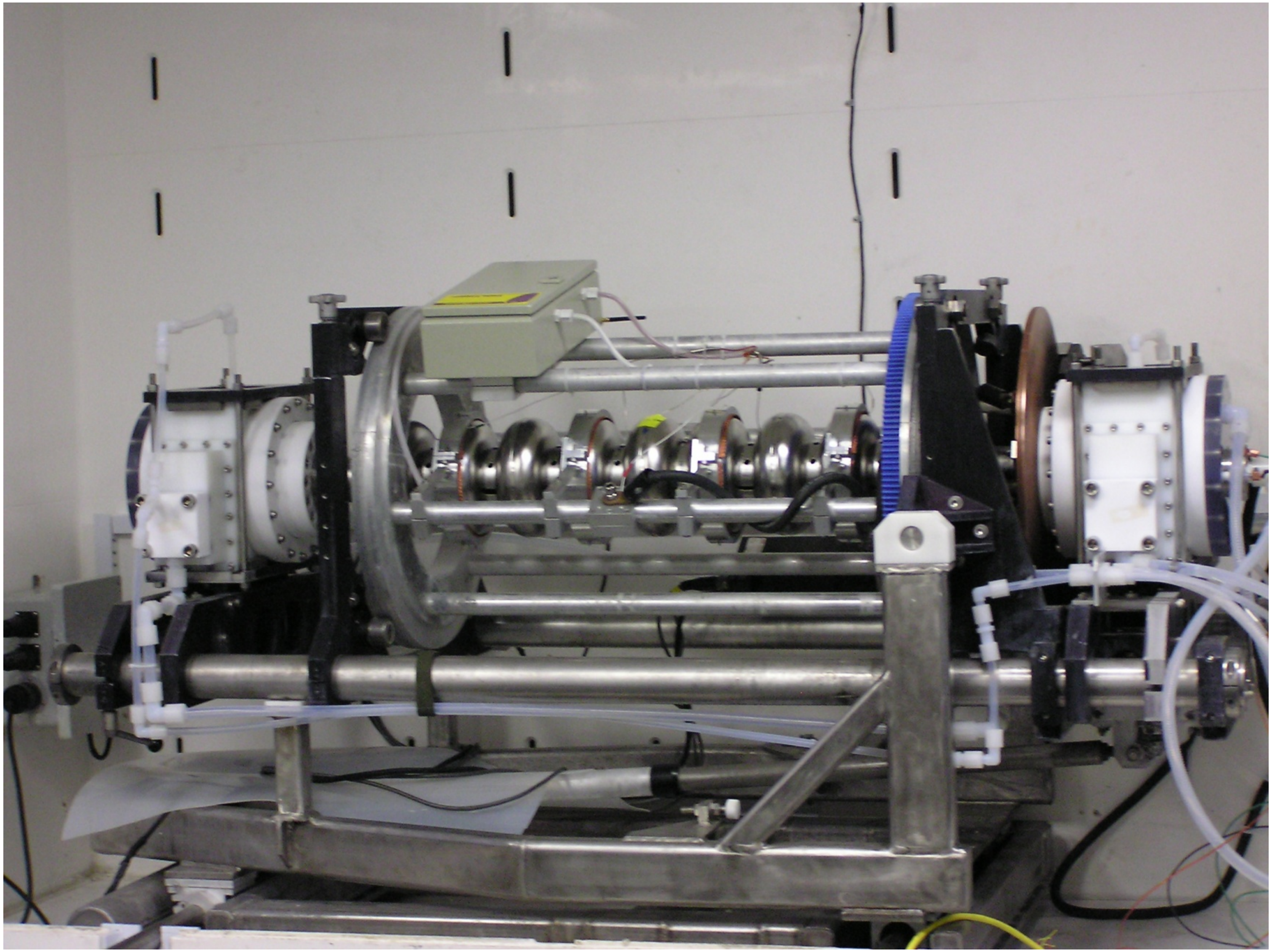
Heavy BCP removes all mechanical damage, but results in a rougher surface.

EP has smaller PSD and the longest correlation length- better macro & micro polish.

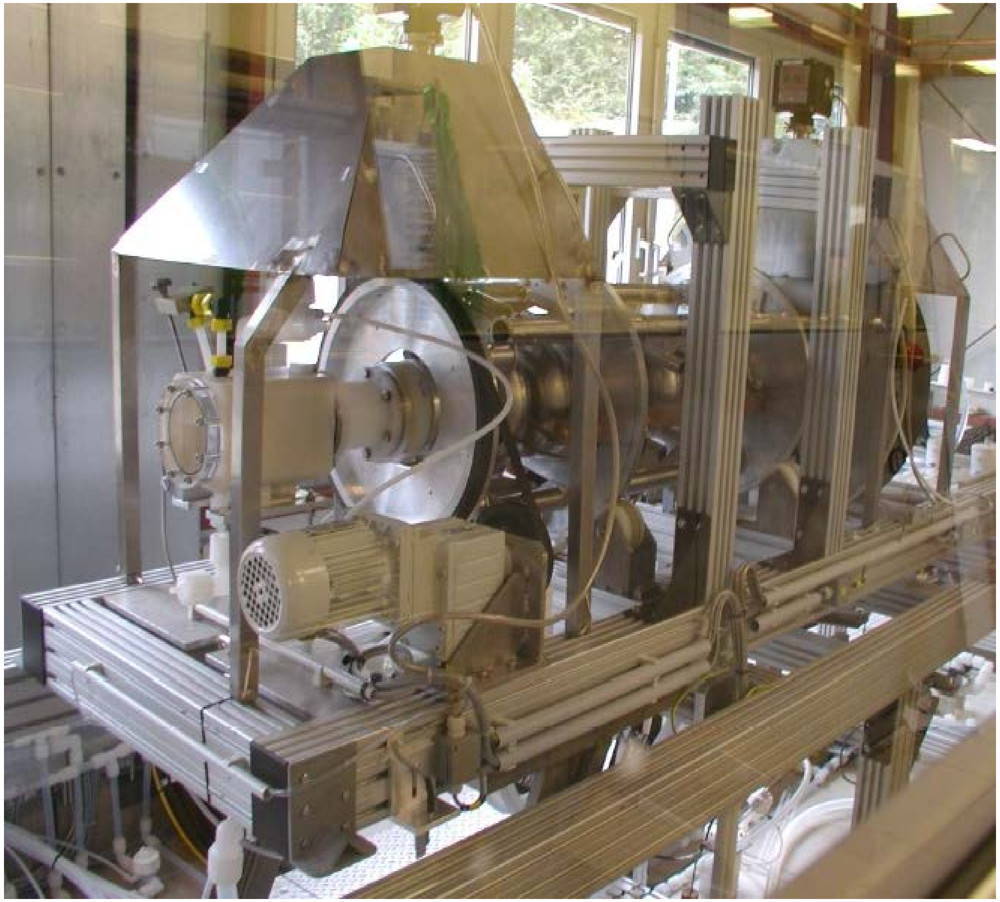
Electropolishing of 9-cell Resonators (Nomura Plating & KEK)



Electropolishing Systems JLAB



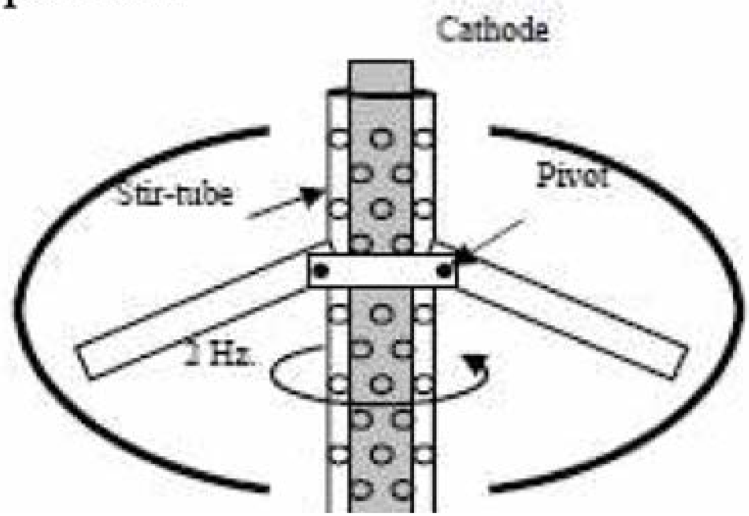
Electropolishing Systems DESY



Cornell University
Laboratory for Elementary-Particle Physics

Vertical Electropolish Proven Effective

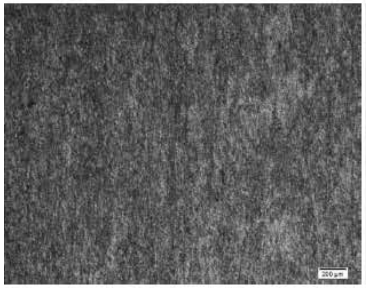
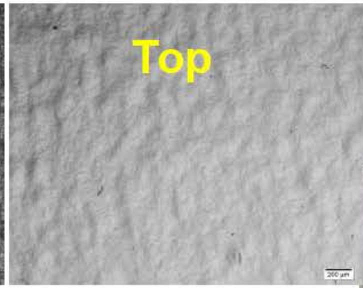
- We have demonstrated gradients >35 MV/m in individual cells of two 9-cell cavities processed with vertical EP.
- In each test the π-mode was limited by quench.



Vertical EP

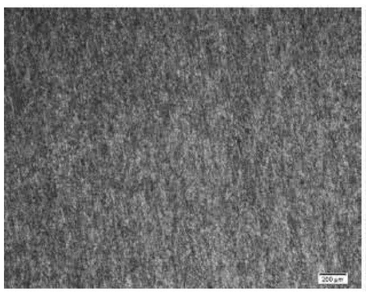
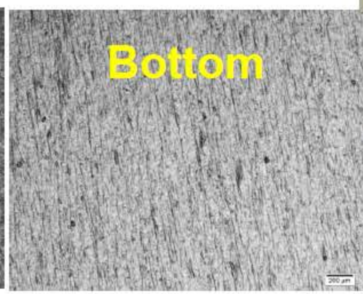
S. Kato, KEK

Surfaces after EP without Stirring

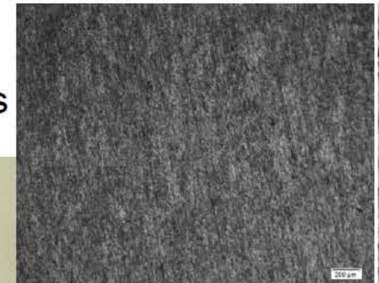
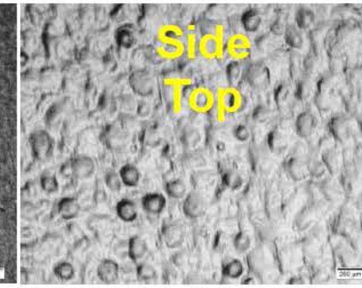
before

RT = 77.8 µm
Ra/Rz
=0.25/1.3 µm

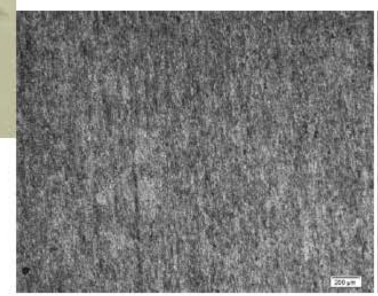
RT = 40.7 µm
Ra/Rz
=0.36/2.5 µm

RT : Removal Thickness

before

RT = 47.5 µm
Ra/Rz
=0.80/4.6 µm

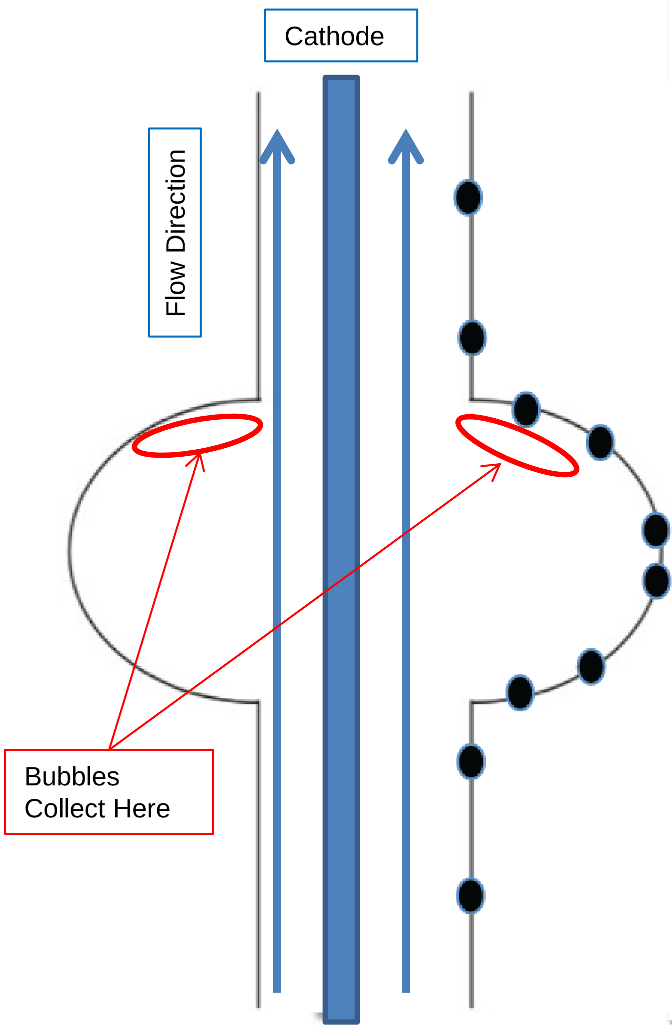


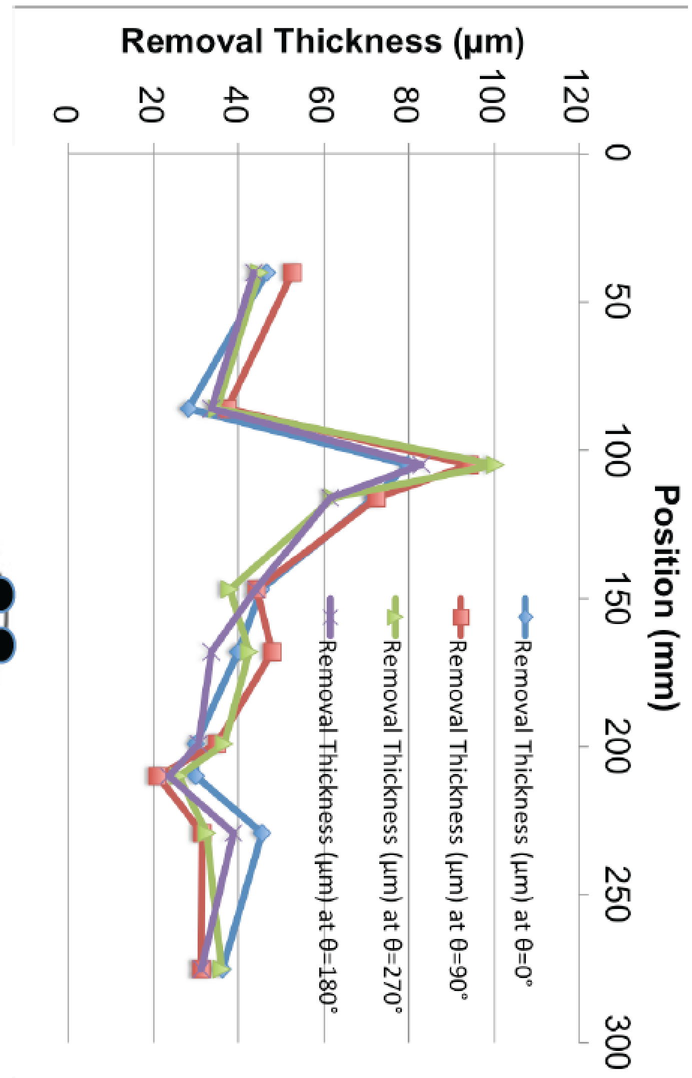

RT = 39.0 µm
Ra/Rz
=0.29/2.2 µm

- H_2 bubbles directly hit and remove sticking bubbles quickly from the top sample surface to make the surface smoother.
- Bubbles might remain at surface of the side top sample with a longer residence time to make it rougher. This was also observed after VEP done with rod cathode.

Issues With Vertical EP

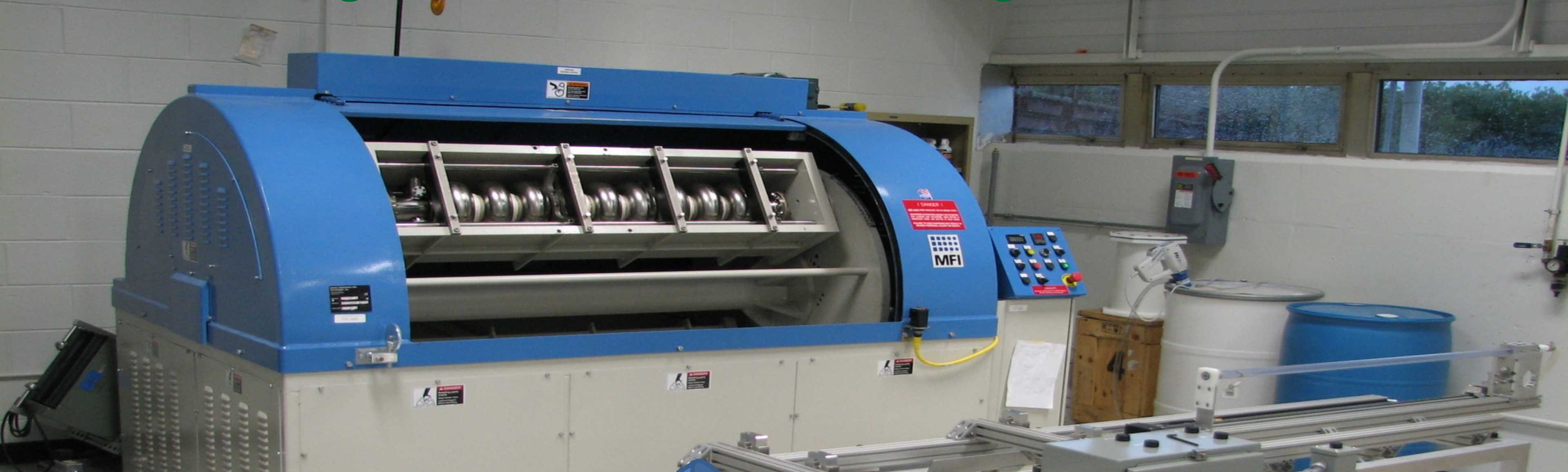
S. Kato, KEK

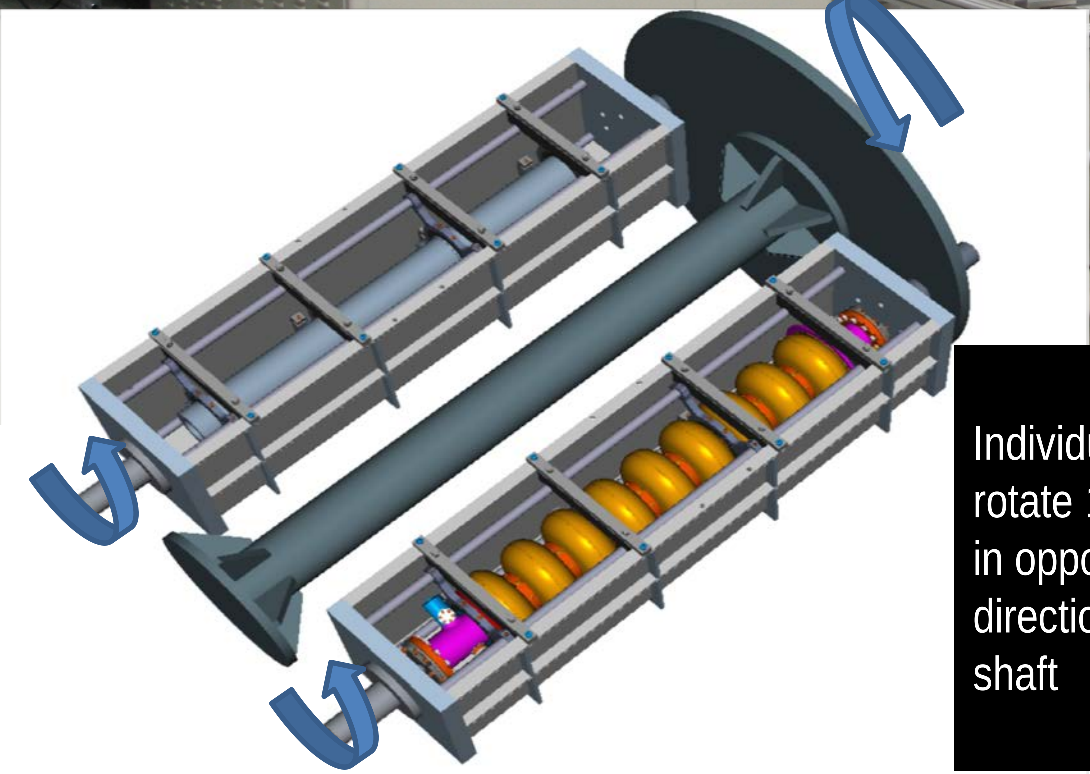




Vertical EP on a HB Dressed Cavity JLab

Centrifugal Barrel Polishing





Individual Barrels rotate 115 RPM in opposite direction to main shaft

C. Cooper, FNAL

Centrifugal Barrel Polishing

Polishing media and processing time

based on Fermilab/JLAB recipe

Step 1

8 hours

ceramics
TS compound
water

Step 2

15 hours

plastic media
TS compound
water

Step 3

30 hours

alumina 15 μm
wooden blocks
water

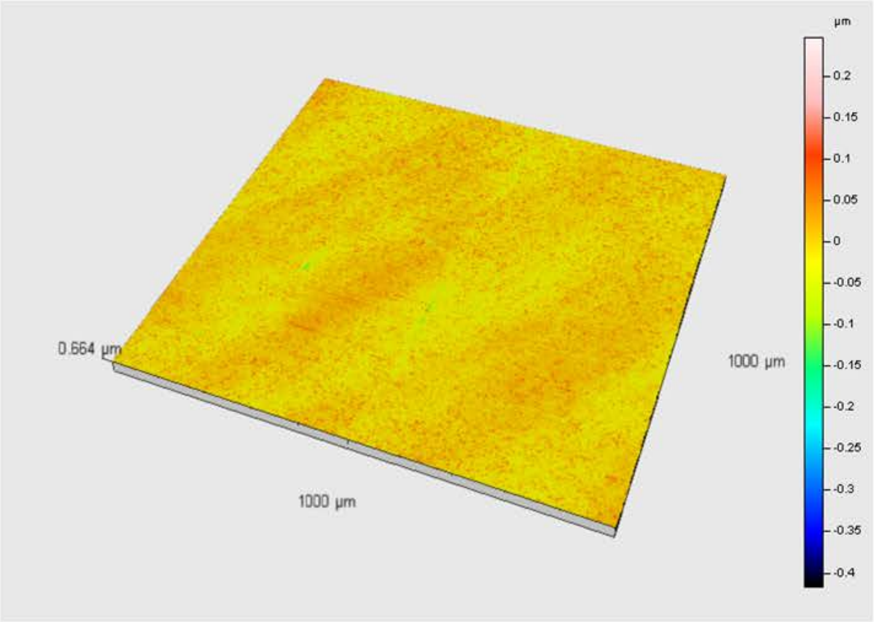
Step 4

40 hours

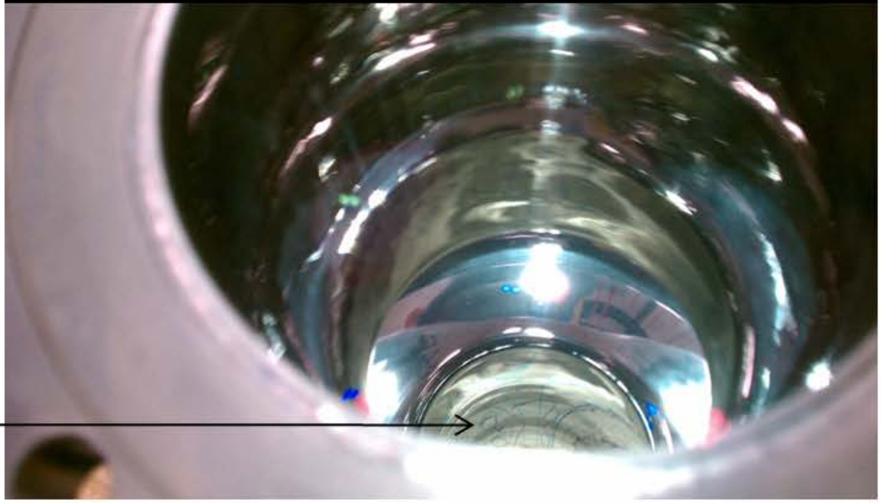
colloidal silica 40 nm
wooden blocks
water

A. Prudnikava, Uni Hamburg

3. CPB of a Cavity to a Mirror Like Surface



Single Cell Polished to Mirror Finish

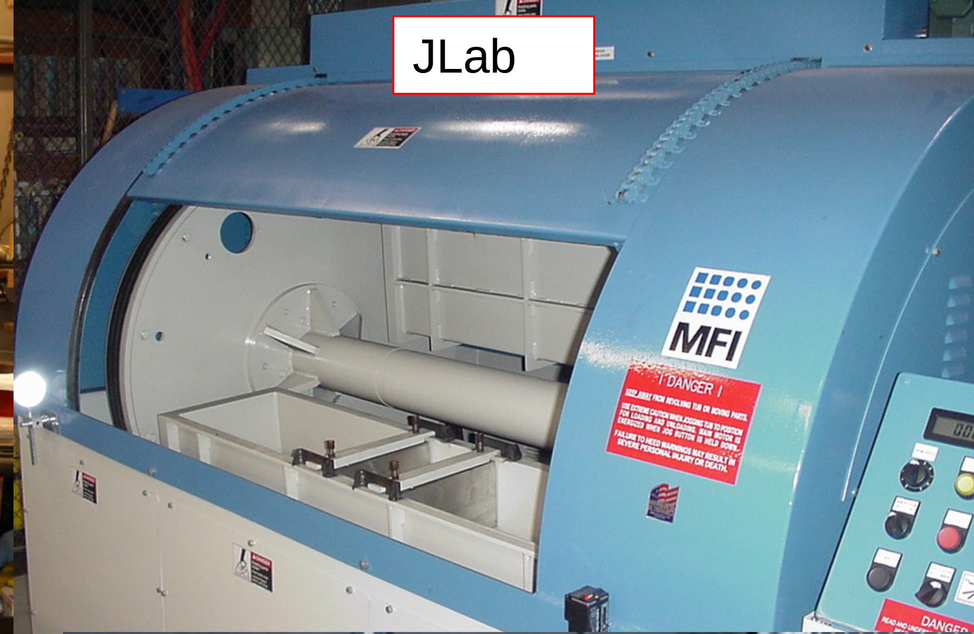


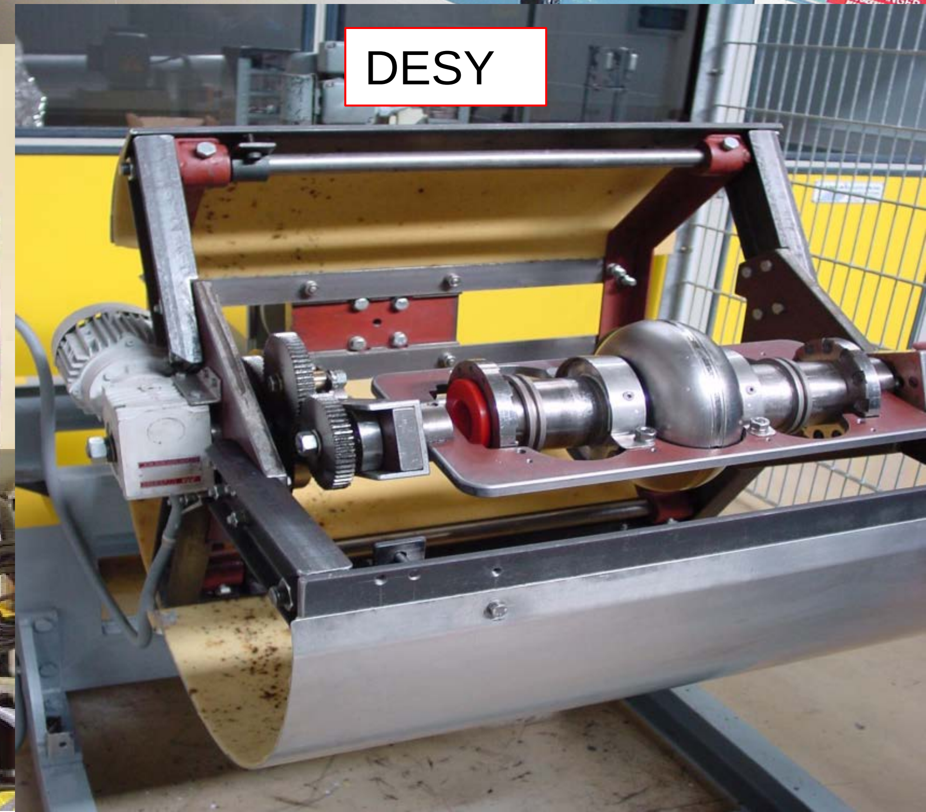
C. Cooper, FNAL

Ra = 0.0139 µm +/- 0.00216 µm
Rz = 0.139 µm +/- 0.0242 µm

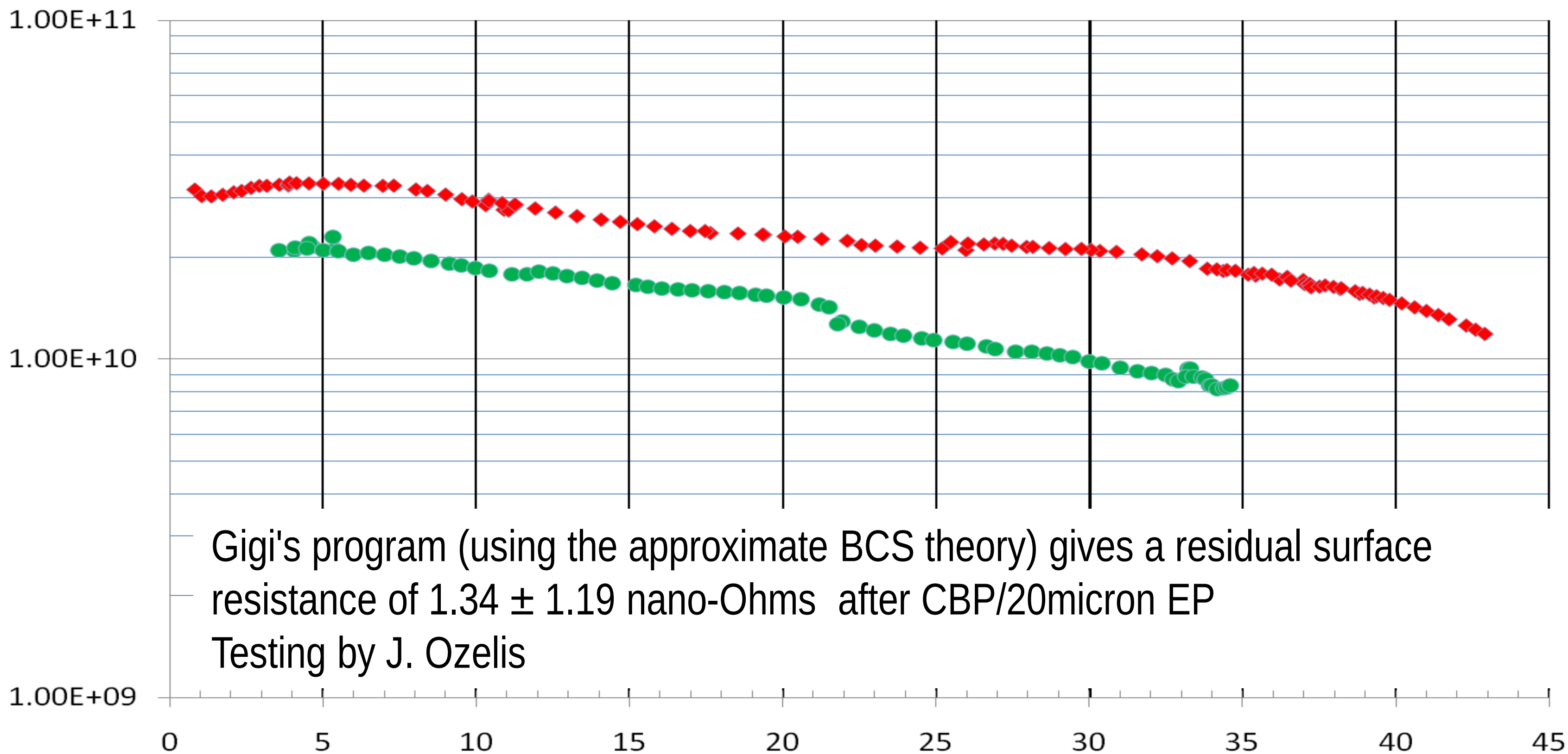
Typical finish achieved by fine polishing.

Notice reflection of graph paper and writing


Cornell
MFI

JLab
MFI
! DANGER !

RRCAT
DANGER
GALA
CENTRIFUGAL FINISHING MACHINE
2010

DESY

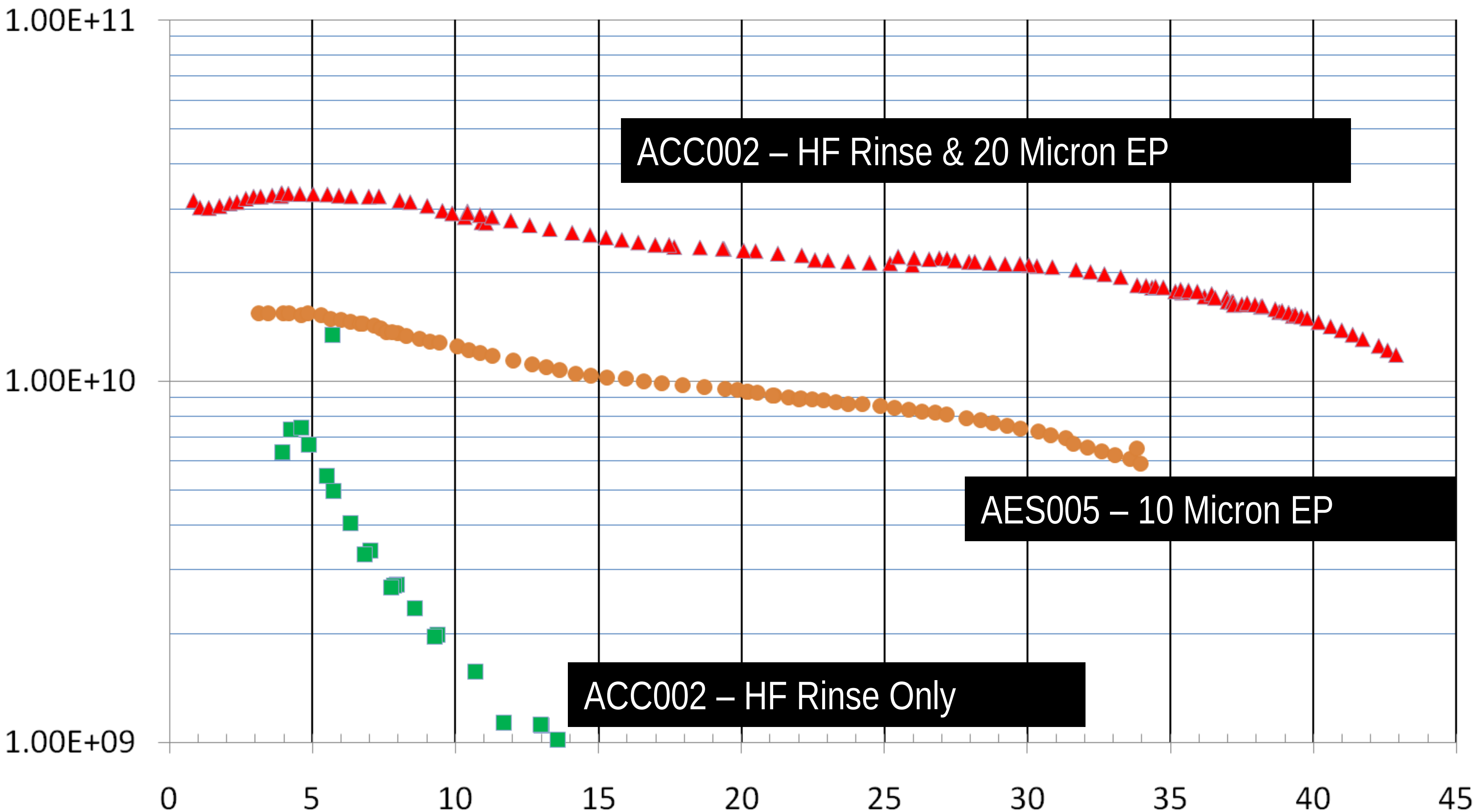
Cavity Improved Results after CBP compared to EP – ACC002



Cavity tested many times after baseline EP processing and reprocessing techniques. Best baseline results shown. Cavity improved greatly after CBP.

C. Cooper, FNAL

How much chemistry needed to remove artifacts of CBP with Mirror Finish?



C. Cooper, FNAL

A. Prudnikava, Uni Hamburg

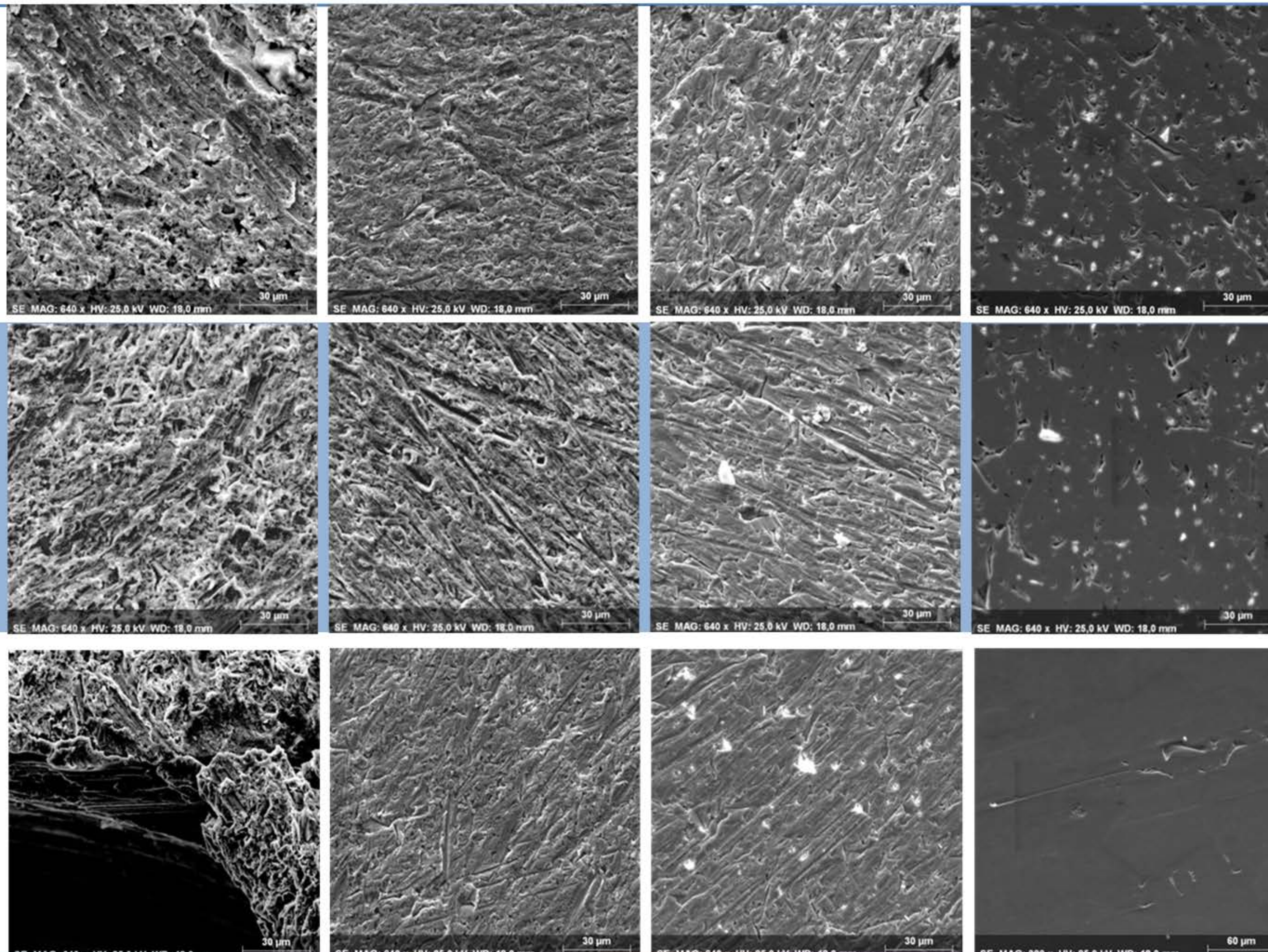
Coupon surface after different steps

Step 1 → Step 2 → Step 3 → Step 4

Tube

Cell

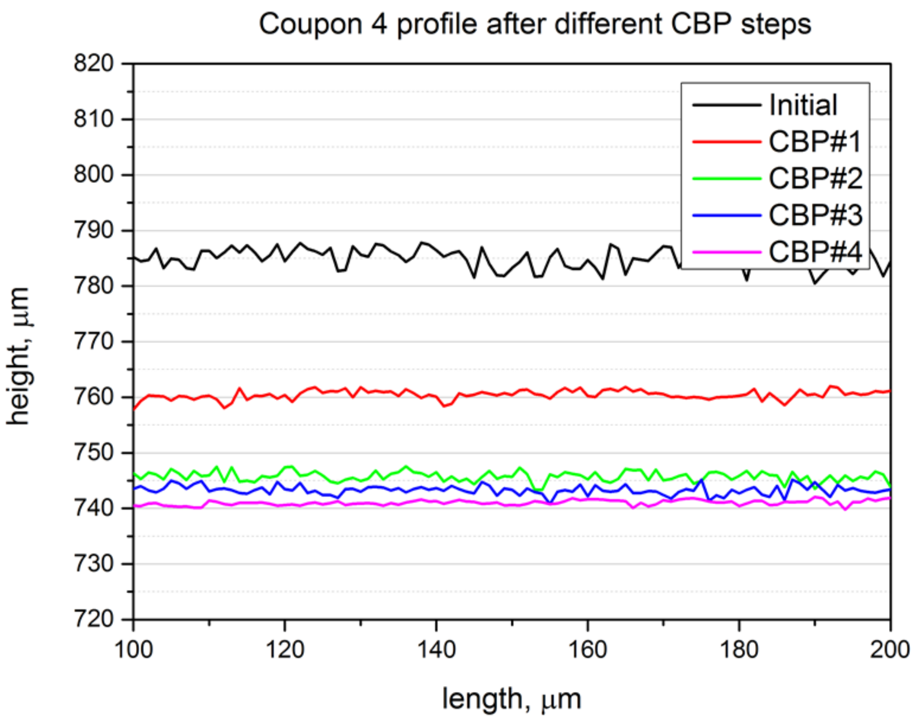
Equator



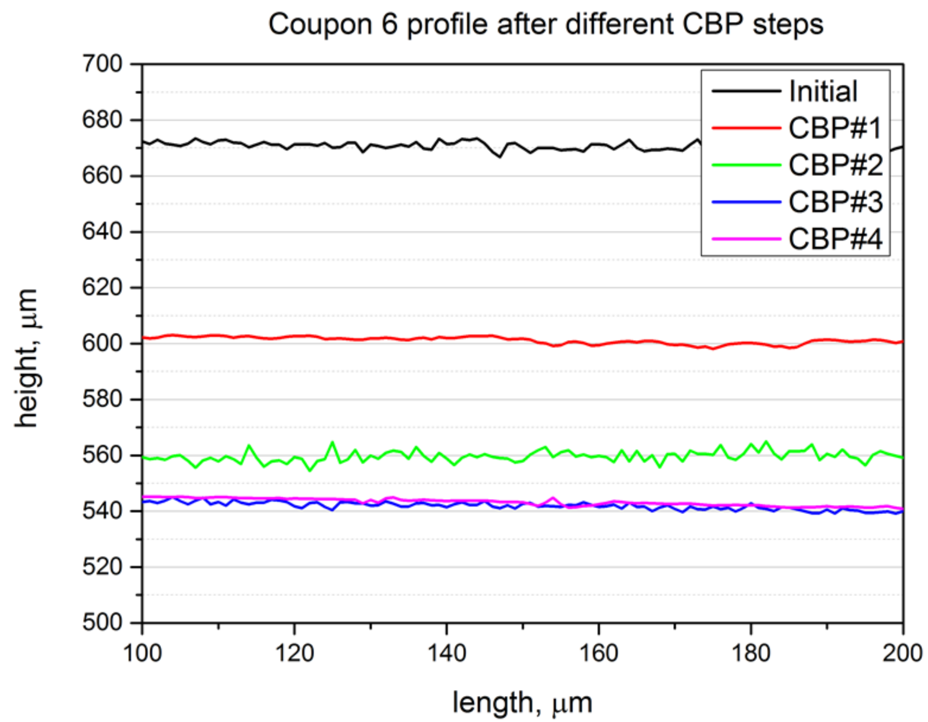
Removal rates

A. Prudnikava, Uni Hamburg

Tube

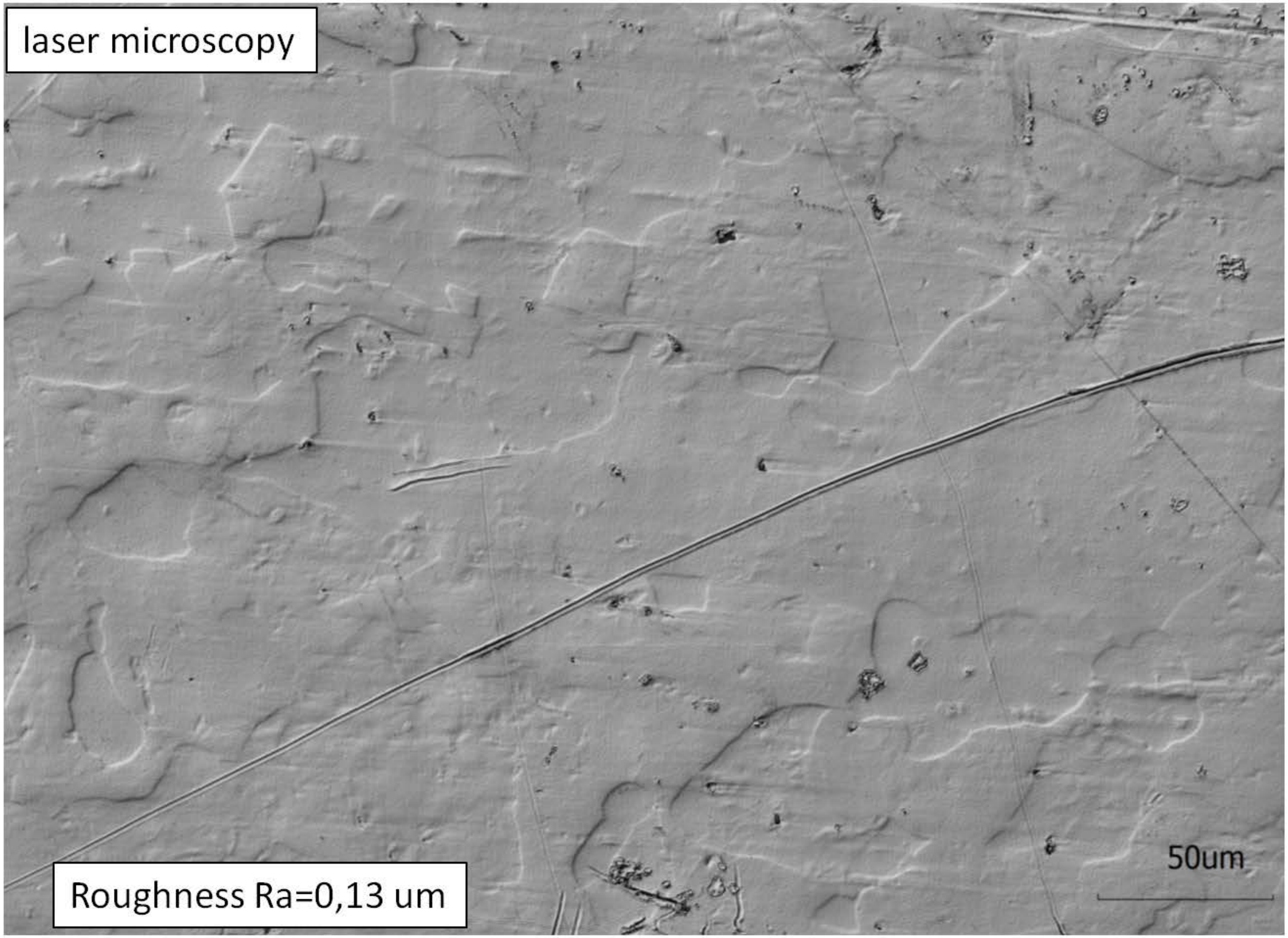


Equator

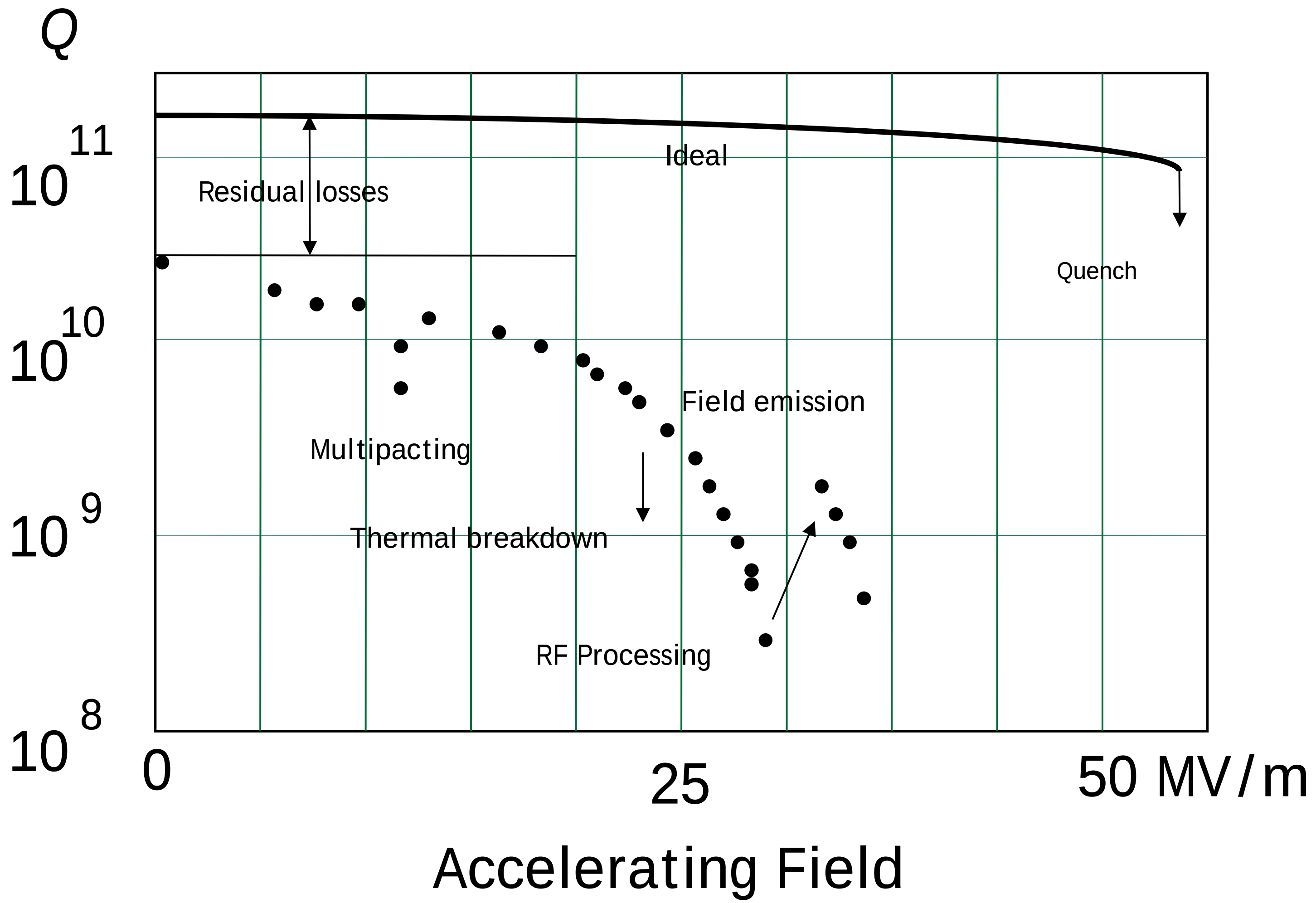


Step	Tube	Equator
1	3 um/h	9 um/h
2	1 um/h	2.5 um/h
3	0.1 um/h	0.7 um/h
4	<0.1 um/h	<0.1 um/h

Step 4
mirror polishing



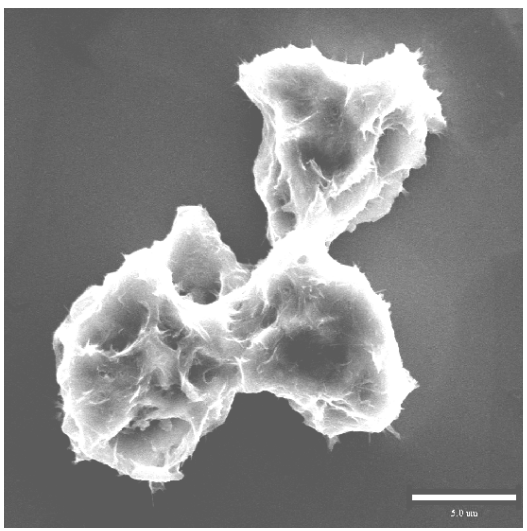
What are we trying to accomplish?

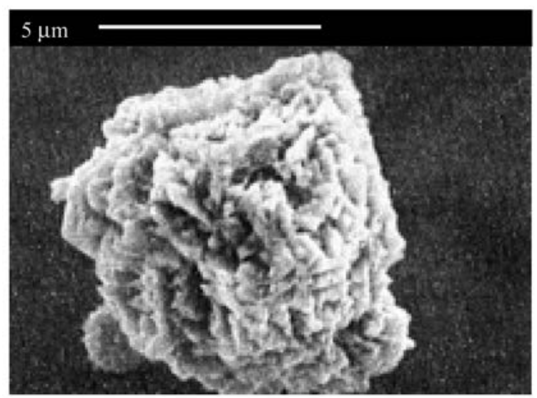


Particulates are still the limitation in the form of field emission

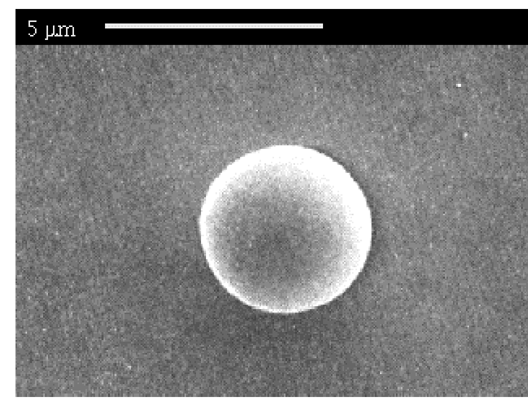
Fig. 11 Example of a scratch and a particle on a niobium surface.

V



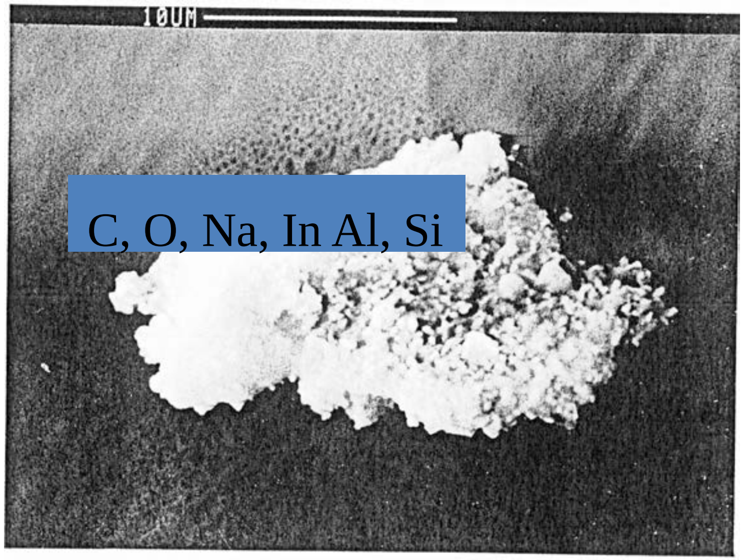


Ni

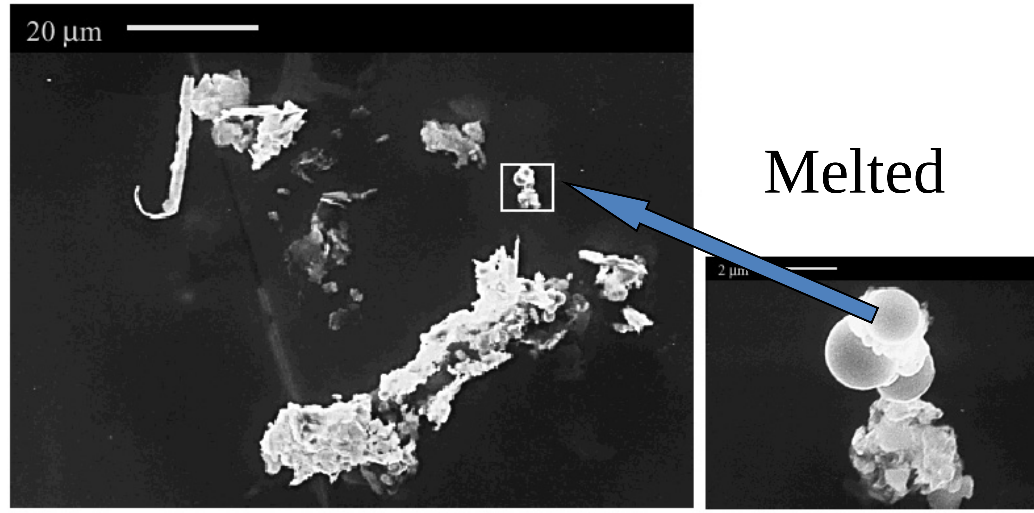


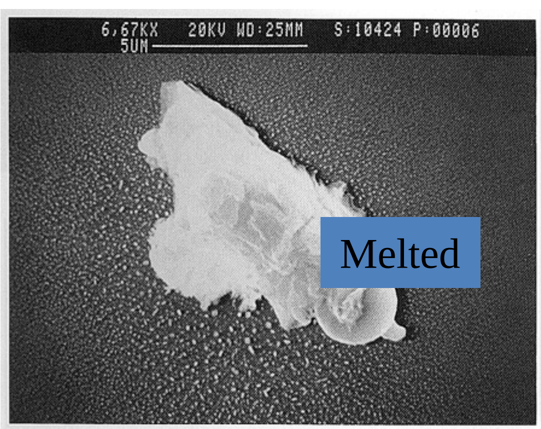
Ni

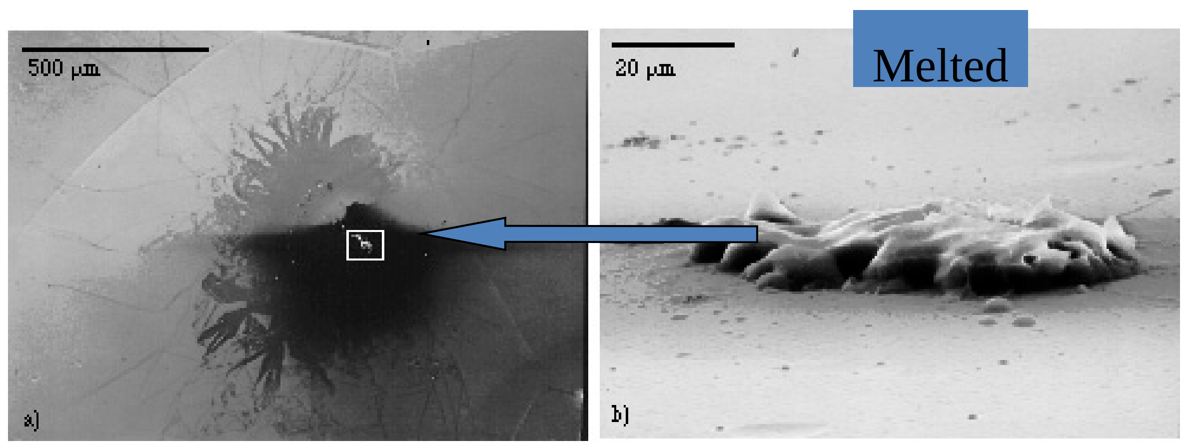
Example of Field Emitters



Stainless steel







Ultrasonic Cleaning

- **Process of generating ultrasound (high frequency pressure) in a cleaning solvent to remove contamination by cavitation bubbles**
  – Typically 20-400kHz
  – Solvent is DI water
  – Detergents: Micro-90 or Liqui-nox, reduce surface tension (1%)
  – Solvent mixture is heated to increase removal
  – The higher the frequency the smaller the nodes between cavitation points
- **Important factors**
  – Where the part contacts the tank surface, will strongly reduce the effectiveness there (no cavitation)
  – Forces on the surface can be a high as 20kpsi, one must be careful of coatings and thin films such as copper plating

Liquinox

Physical Data - Typical value
pH (as is) - 8.5
Specific gravity (g/ml) - 1.07
Density (lbs./gal.) - 8.9
Vapor pressure (mm Hg) - 10.5
Flash Point (degrees F) - None
Phosphate Content (as Phosphorus) - 0%
Organic Carbon (% calculated w/w) - 21%
Fragrance Content - 0%
Surface Tension 1% Sol'n (Dyne/cm) - 32
Color: Pale Yellow
Form: Liquid
Solubility in Water: Completely soluble in all proportions
Hard Water Effectiveness: Highly Effective
Biodegradability: Biodegradable
Foam Tendency: High Foaming
Shelf Life: Two years from the date of manufacture

Chemical Description:
Liquinox consists primarily of a homogeneous blend of sodium linear alkylaryl sulfonate, sodium xylene sulfonate, alkanolamide, and ethoxylated alcohol. Liquinox is anionic in nature.

Small Part Ultrasonic Cleaning Stations

- Rinse tank out
- Fill with DI water
- Add Liquid Detergent
  - Liquinox
  - Micro-90
  - Few percent by volume
- Ultrasonic agitation
  - 15-60 minutes
- Remove and rinse parts with DI water
- Blow dry ionized N2

Cavity and Large Component Ultrasonic cleaning

High Pressure Rinsing:

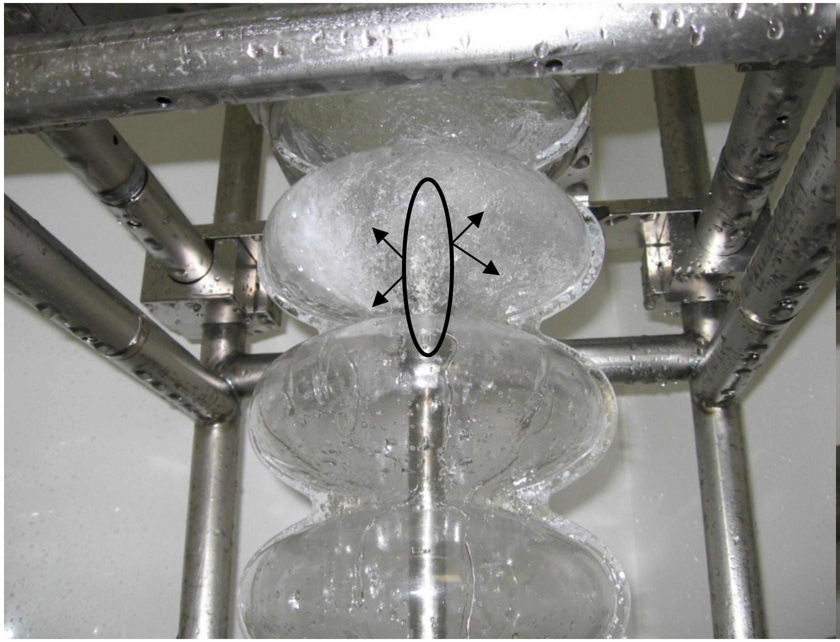
• This is still the best cleaning method against field emission!

- The need for HPR surface cleaning:
  - Entire surface contaminated after chemistry, early field emission will result if not performed
  - Effective at removing particulates on the surface after assembly steps

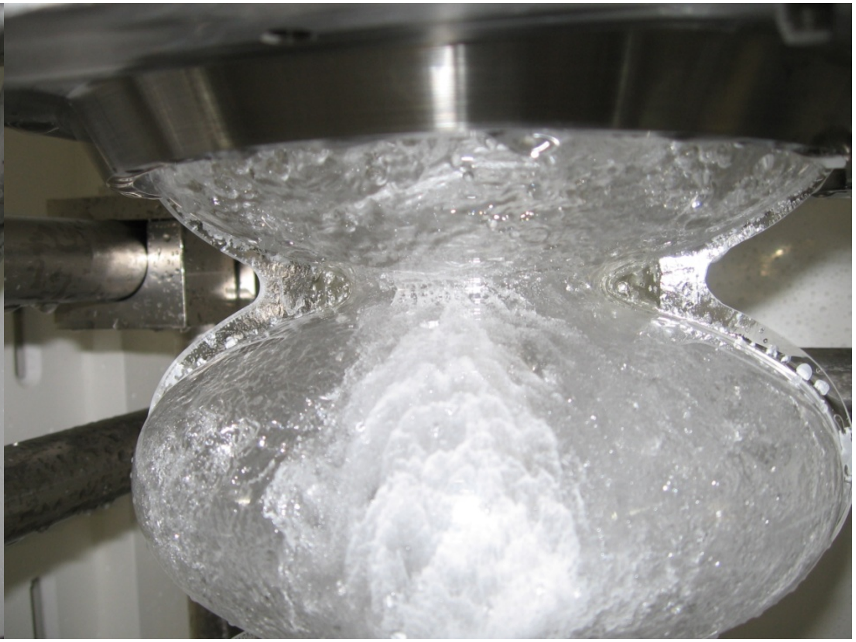
ISSUES:

- HPR systems are still not optimized for the best surface cleaning performance , must be optimized for each cavity shape and HPR system
- **Surface left in a vulnerable state, wet**

HPR spray heads needs to be optimized for a particular geometry!



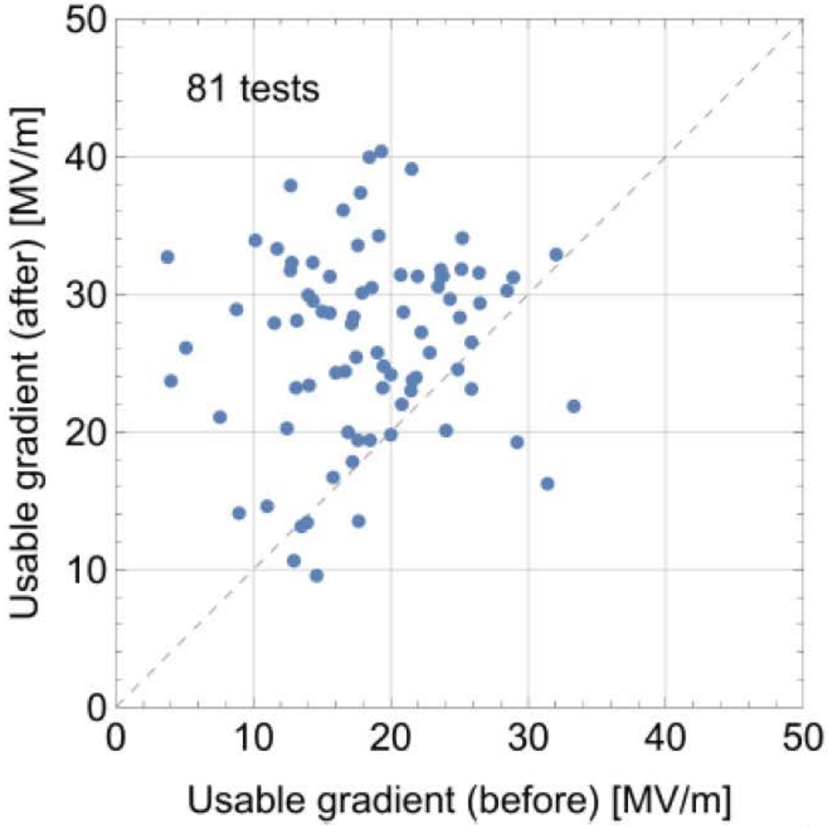
Very effective on irises

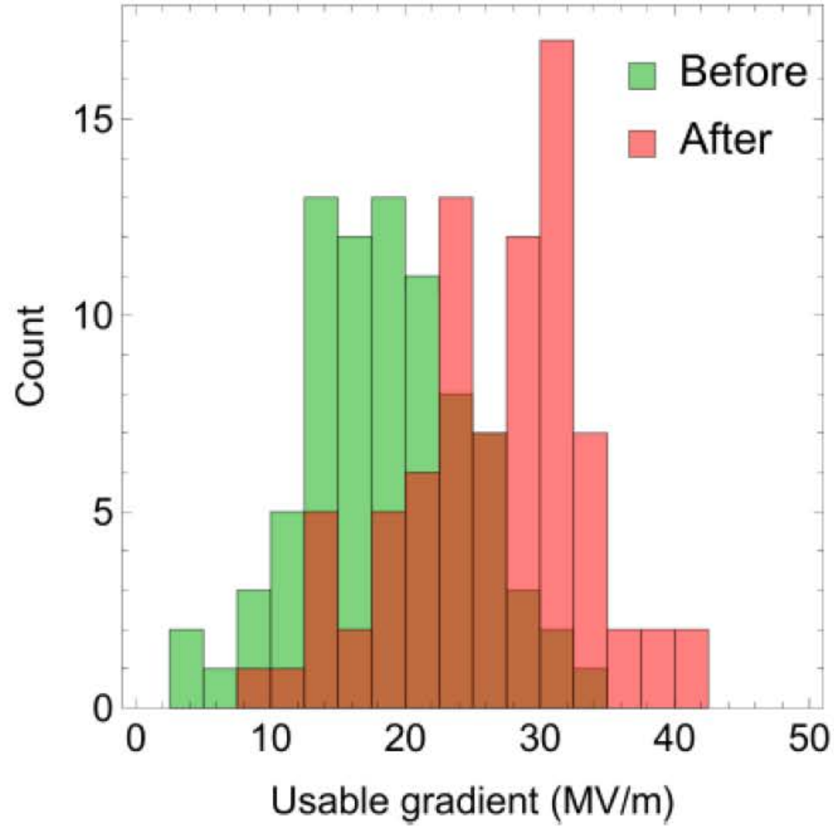


Equator fill with water → too high flow rate

For a given pump displacement the nozzle opening diameter and number of nozzles sets the system pressure and flow rate

DESY/XFEL Retreatments of Cavities

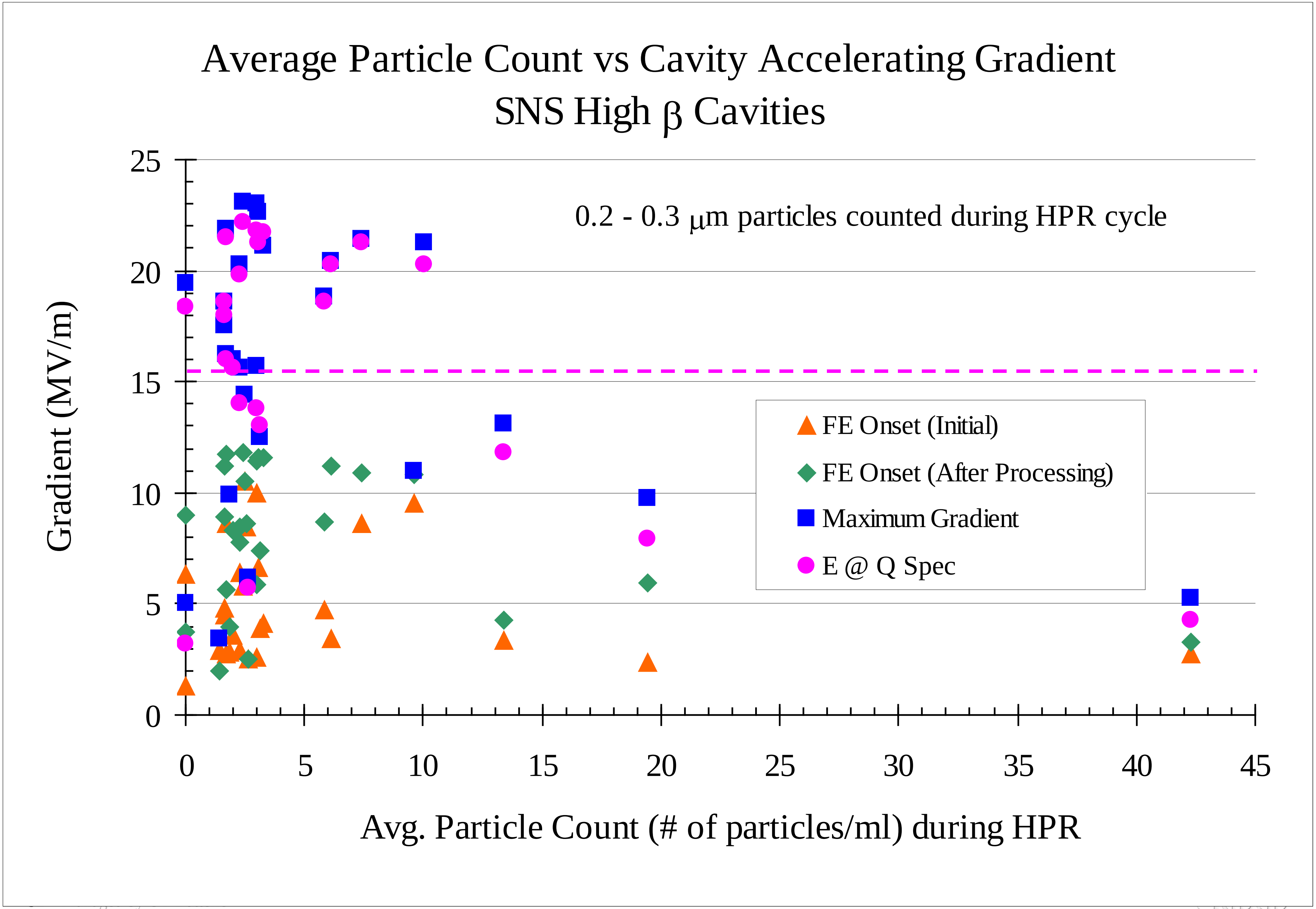




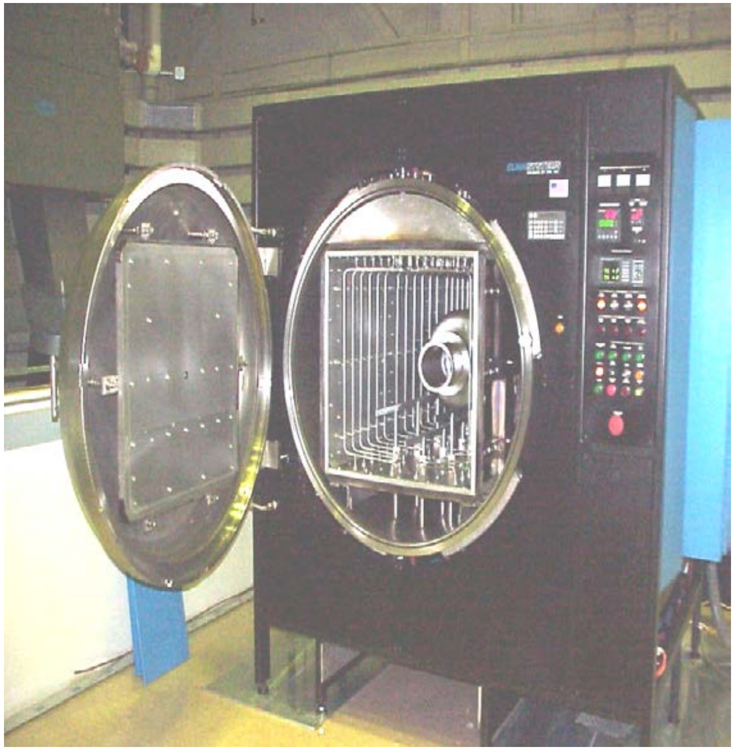
- Analysis of ~80 cavities after **first** re-treatment => **typically HPR**
- Reasons for re-treatment:
  - mostly **field emission** (61 cavities)
  - quench at "low" gradient (7 cavities)
  - low Q-value at low gradient (6 cavities)
  - leak (2 cavities)
  - other (6 cavities)

	Before	After
Tests	81	82
G_{AVG} (MV/m)	18.5	26.6
G_{RMS} (MV/m)	6.3	6.8
yield @ 20MV/m	40%	83%
yield @ 26MV/m	10%	56%
yield @ 28MV/m	7%	50%

Preliminary data; results are not published

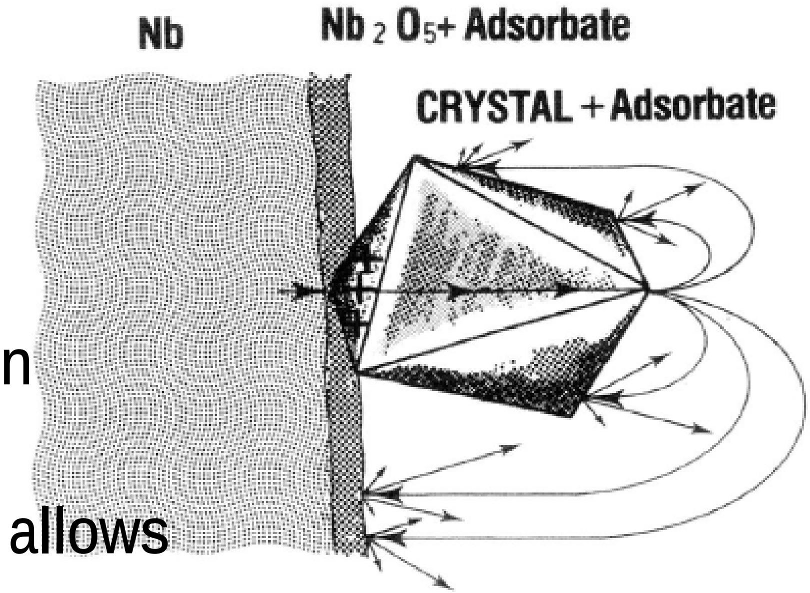

Average Particle Count vs Cavity Accelerating Gradient
SNS High β Cavities
0.2 - 0.3 μm particles counted during HPR cycle
Gradient (MV/m)
25
20
15
10
5
0
0
5
10
15
20
25
30
35
40
45
Avg. Particle Count (# of particles/ml) during HPR
FE Onset (Initial)
FE Onset (After Processing)
Maximum Gradient
E @ Q Spec

Heat treatment (600-800C)



Details		
Temperature of hot zone	Low end 600C	Typical 800C
Vacuum	Start 1e-7 Torr	End 1e-5 Torr
Cavity cleaning	Typically - degreasing	Sometimes- Chemistry and HPR
Support structure	Moly rails or rods	
Automated controls	RGA, PLC	
Process time	6-12 hrs or more	

Helium Processing



- Variation of RF processing
- Keep pressure below discharge condition
- Run cavity in the field emission regime
- Push the gradient as high as the system allows
- The process in details is unknown
  - Electron spraying from FE → bombard surface → ionization of helium at around surface → destroy field emitter???
  - Controlled processing is difficult
    - Relying on field emitter locations and responses
      - Uniformity??

HF Free Electropolish is on the way

- **Several Recipes Currently Under Development**

- **Sulfuric and Water with Bipolar Waveform (Faraday Technology)**
  - **Results very promising on single cells**

- **Choline Chloride, at High Temperature (INFN)**
  - **Small samples successful now working on cavities**

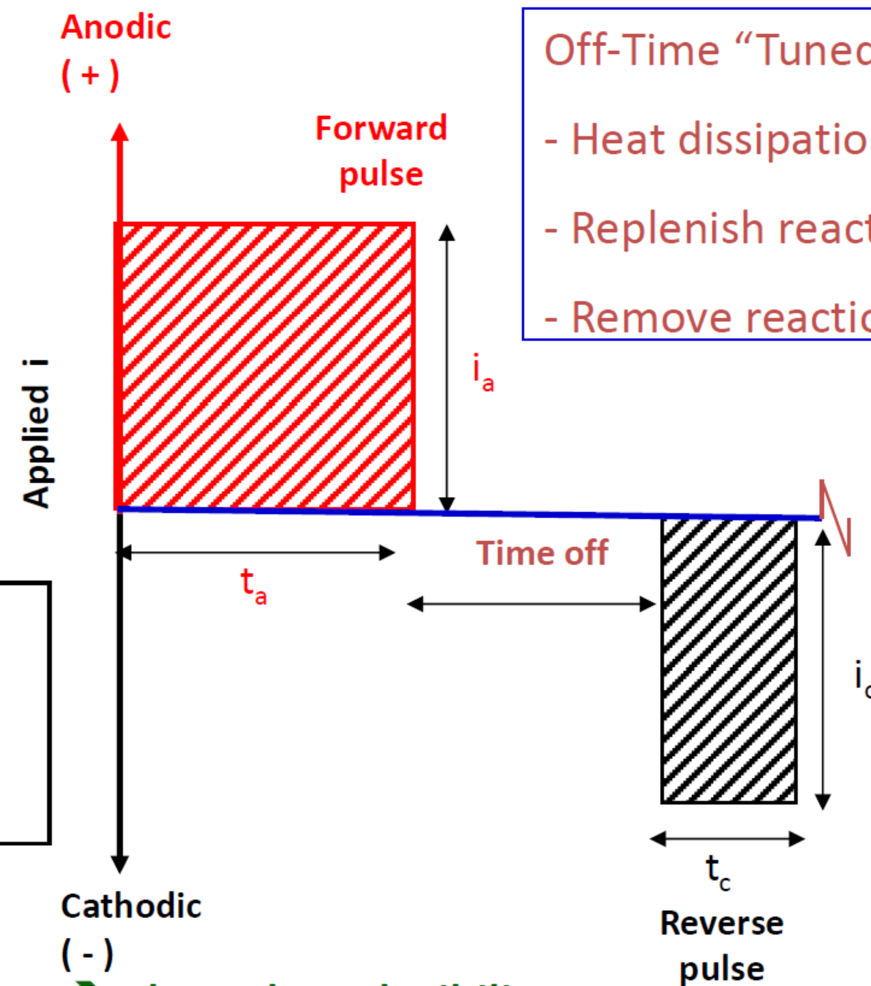
- **Other recipes tried with some success**

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

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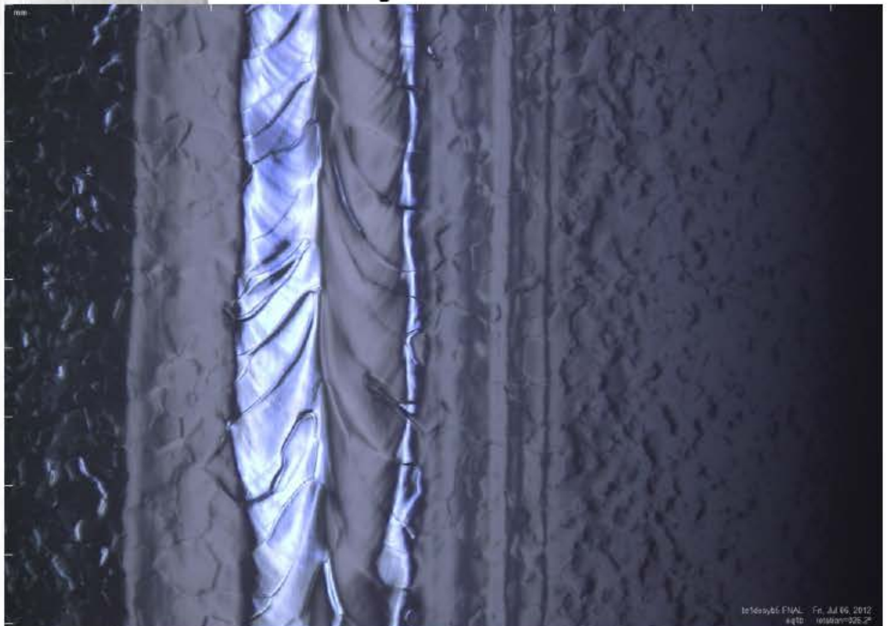
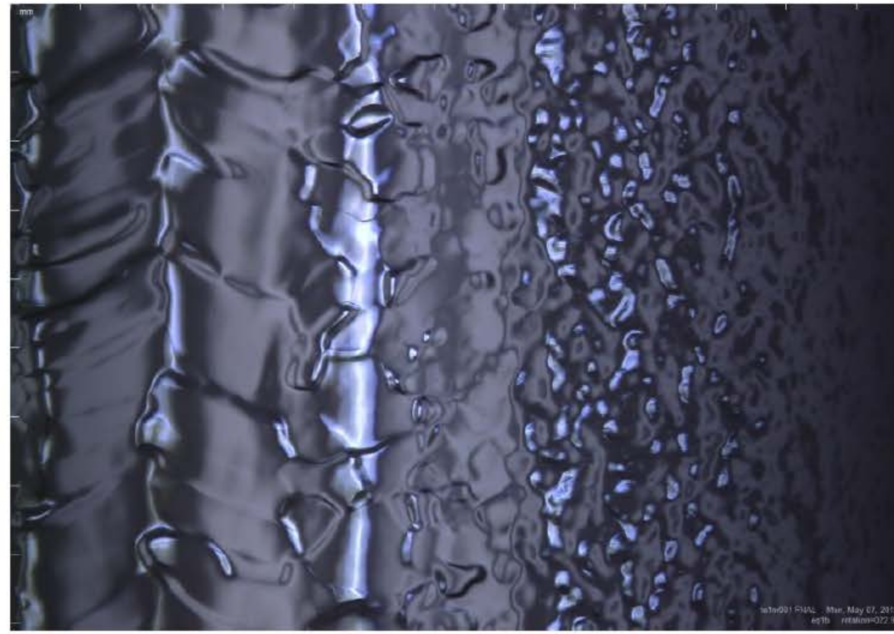
Bipolar EP Surface Details



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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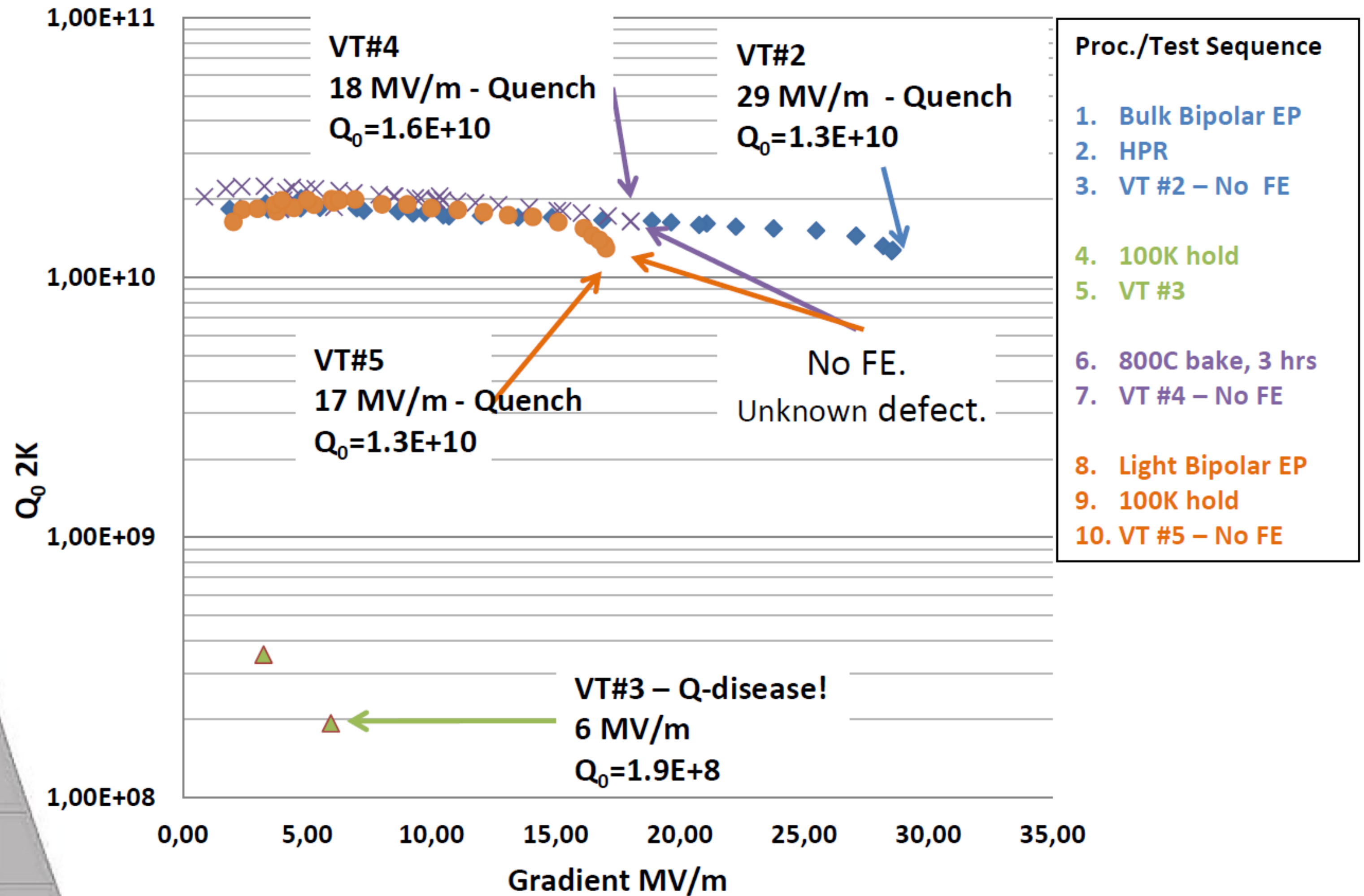
TE1AES007 Performance Results
Bipolar EP Q-disease Studies



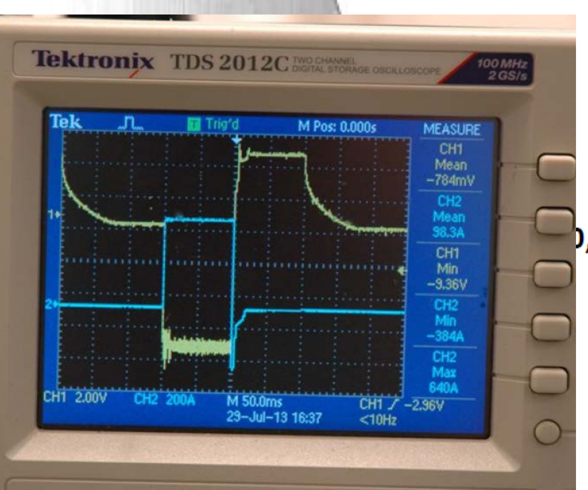
Typical 1-cell EP:

P_{avg} = 630 W

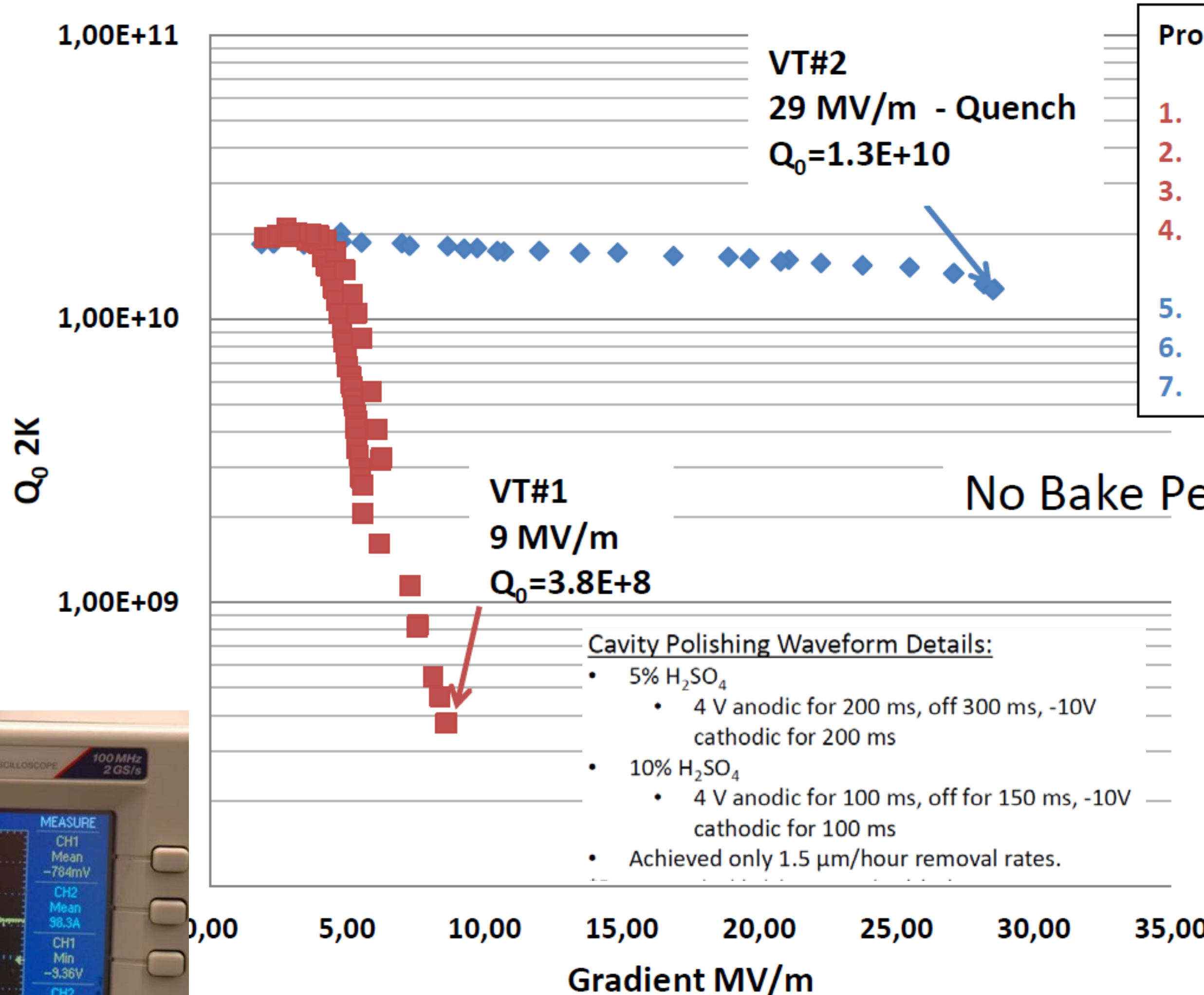
P_{pk} = 1 kW

Voltage = 18 VDC

$Current_{avg}$ = 35 A



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



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.

Proc./Test Sequence

1. 20 um std EP
2. 800C bake 3 hrs
3. HPR
4. VT #1 – No FE

5. Bulk Bipolar EP
6. HPR
7. VT #2 – No FE

No Bake Performed.

Fermilab

Allan Rowe, SRF2013, TUIOC02, Paris, France.

Plasma processing for SRF cavities

- Plasma is a rich and reactive environment
  - Ions, electrons, neutrals, excited neutrals, molecules, radicals, UV…
- Plasma processing is a versatile technique used in many industries for various purposes
  - Cleaning, activation, deposition, crosslinking, etching….
- In-situ plasma processing developed at the SNS to increase accelerating gradients
  - Reactive plasma used to clean hydrocarbons from top surface
  - Increase work function and reduce field emission