SURFACE PREPARATION

Jean Delayen

Center for Accelerator Science
Old Dominion University
and
Thomas Jefferson National Accelerator Facility





Required Procedures for Qualifying SRF Cavities

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (~150 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
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- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking

If cavity meets specs after cryo-RF test...



Additional Steps for Cavity String

- Final mechanical tuning
- He-vessel welding
- Degreasing
- Final material removal (10-20 μm)
- Final HPR
- Horizontal assembly into cavity-string
- Evacuation of cavity string





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Degreasing with Ultrasonic Agitation

Why is degreasing needed

- To remove grease, oil and finger prints from cavity surfaces
- To remove surface contamination due to handling, RF measurements and QA inspection

Implementation:



- Usually performed in Hepa filtered air
- Water quality is good, 18 M Ω cm, Filtration > 0.2 μ m
- Manually or semi-automated processes available
- Problem: Parts are wet and vulnerable to particulate contamination





Ultrasonic Cleaning

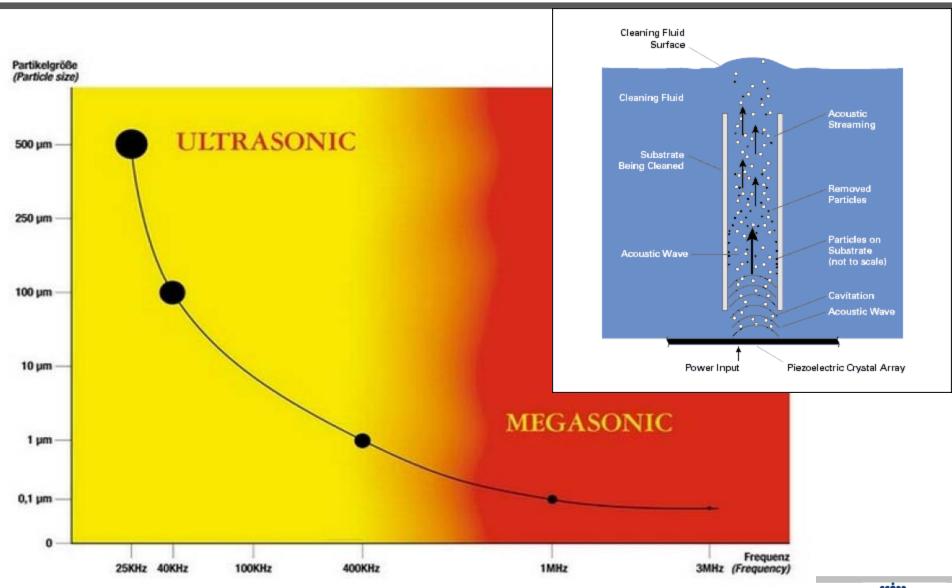
- Immersion of components in DI water and detergent medium
- Wave energy forms microscopic bubbles on component surfaces.
 Bubbles collapse (cavitation) on surface loosening particulate matter.
- Transducer provides high intensity ultrasonic fields that set up standing waves. Higher frequencies lowers the distance between nodes which produce less dead zones with no cavitation.
- Ultrasonic transducers are available in many different wave frequencies from 18 kHz to 120 kHz, the higher the frequency the lower the wave intensity.

Cavities and all hardware components (Flanges, nuts & bolts...) have to be degreased with ultrasonic cleaning





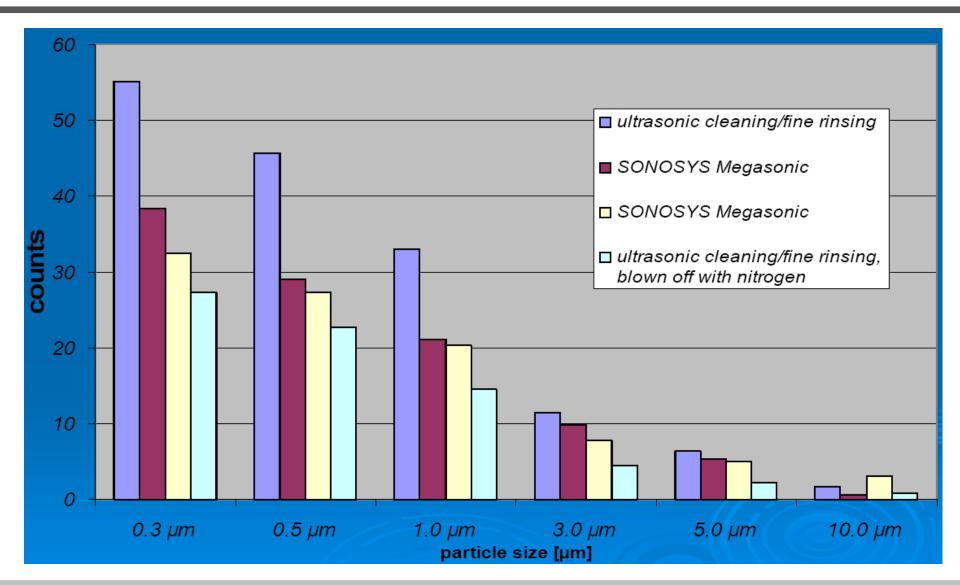
Megasonic Cleaning







Studies on Efficient Cleaning Methods







Example on Nb Sample

Test on cleaning procedure/detergent: Nb sample polluted with grease and oil









Ultrasonic Tanks for Cavity Cleaning







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- Removed of by drogen from bulk Nb Chemical:
- Me
 Buffered Chemical
 - Cr Polishing (BCP)
 Electropolishing (EP)

 al surface

Mechanical
Centrifugal Barrel Polishing
(CBP)

- Additional "cleaning" steps if Electropolishing (EP) is used
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Acid Etching of Sub-components & Cavities:



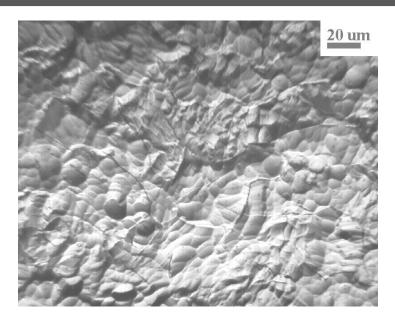
Implementation: (BCP or EP)

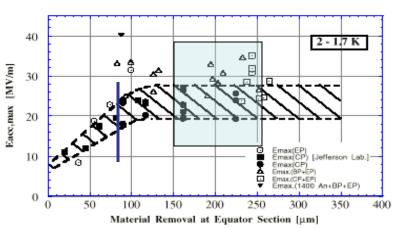
- Sub-components require
 - Removal of oxides which come from fabrication steps → lower losses and improve sealing
- Cavities require:
 - Interior chemistry to remove damaged surface layer incurred in welding and deep drawing (100-200µm)
 - Exterior chemistry to remove surface oxides that occurred in welding (10-30μm)
- Subcomponents usually processed by hand in wet bench
- Acid quality usually electronic grade or better, low in contaminants
- Acid temperature control required to prevent additional absorption of hydrogen (Q-disease)
- Acid mixture difficult to QA

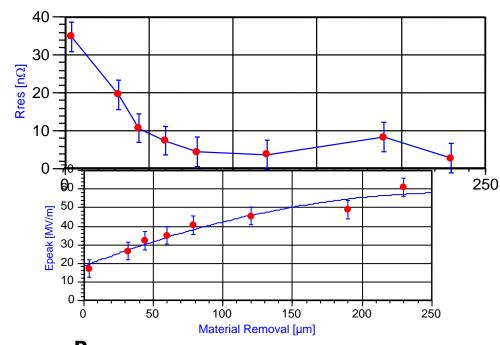




The Need For Material Removal







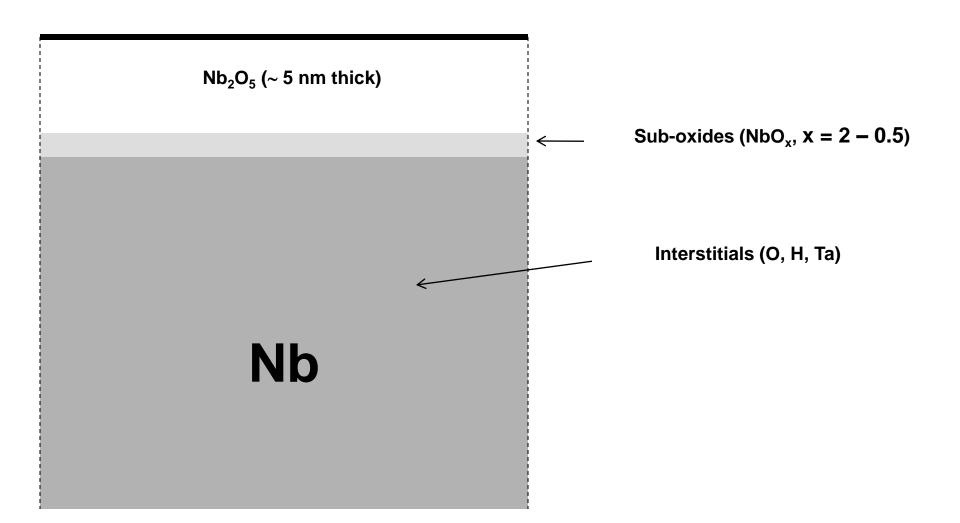
P. Kneisel

K. Saito





Nb Surface







Buffered Chemical Polish (BCP)

HF (49%), HNO₃ (65%), H₃PO₄ (85%) Mixture 1:1:1, or 1:1:2 by volume typical

Oxidation

 $2Nb + 5HNO_3 \rightarrow Nb_2O_5 + 5NO_2$

Brown gas

Reduction

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

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

Reaction exothermic! Use H₃PO₄ as "buffer" to slow reaction rate

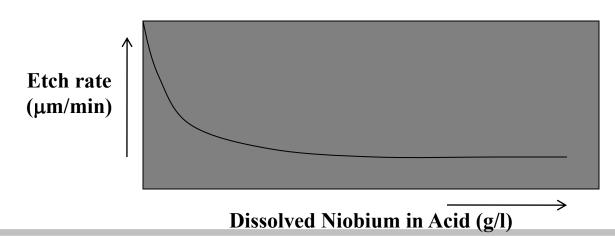




Use of BCP:

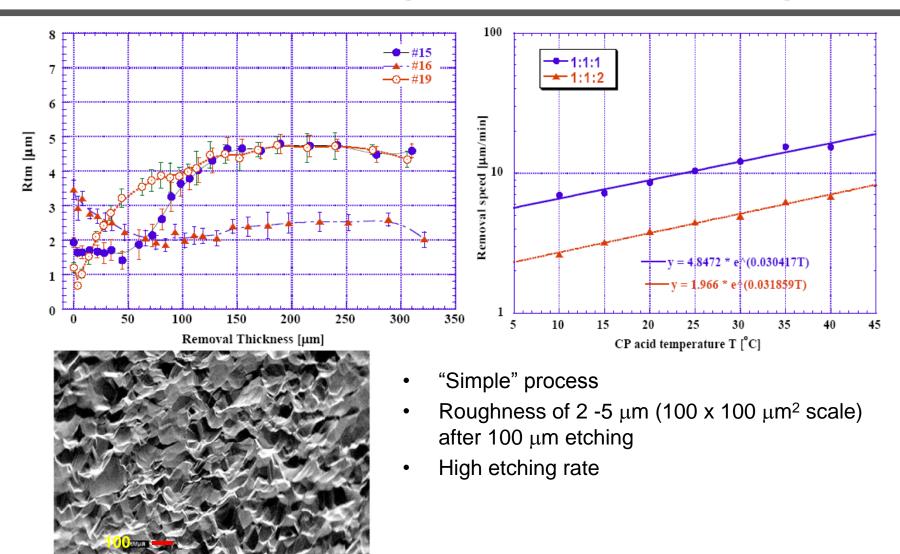
- 1:1:1 still used for etching of subcomponents (etch rates of $\sim 8~\mu m/min)$
- 1:1:2 used for most cavity treatments (etch rates of ~ 3 μm/min)
 - Agitation necessary → reaction products at surface
 - Acid is usually cooled to 10-15 $^{\circ}$ C (1-3 μ m/min) to control the reaction rate and Nb surface temperatures (reduce hydrogen absorption)

Acid wasted after 15 g/l Nb





BCP: Surface Roughness and Etching Rate







BCP Systems for Cavity Etching

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



- Cavity held vertically
- Closed loop flow through style process, some gravity fed system designs
- Etch rate 2x on iris then equator, if no stirring mechanism
- Temperature gradient causes increased etching from one end to the other
- · Manually connected to the cavity but process usually automated



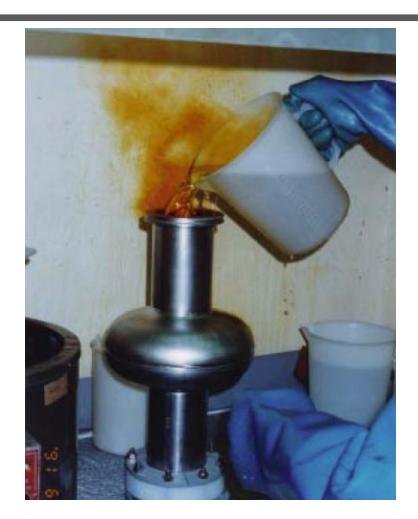




Chemical Etching Setups



Old system for CEBAF cavities



BCP of single cell cavity under chemical flow hood





Chemical Etching of Outer Surface

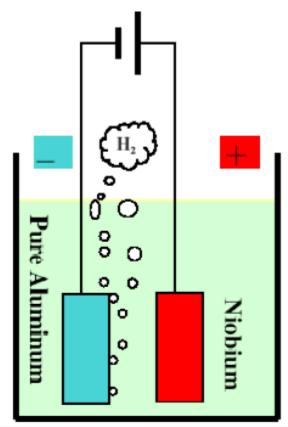
 ~ 20 um are removed from the outer surface of the cavity by BCP to remove "dirty" layer after fabrication in order to improve the heat transfer at the Nb/LHe interface (Kapitza resistance)

- Some labs do this as part of cavity preparation procedure (DESY), some don't (JLab)
- No clear influence on cavity performance





Electropolishing



Acid:

 H_2SO_4 (>93%): HF(46%)=10:1 V/V

Electropolishing (EP) of Niobium:

- Both electrodes are immersed in electrolyte
- A voltage is applied between Nb (anode) and counter electrode (cathode, Al)

Basic reactions:

Oxidation

$$2\text{Nb} + 5\text{SO}_4^{2-} + 5\text{H}_2\text{O} \rightarrow \text{Nb}_2\text{O}_5 + 10\text{H}^+ + 5\text{SO}_4^{2-} + 10\text{e}^-$$

Reduction

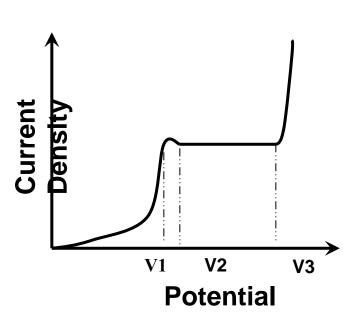
$$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F 0.5H_2O + 1.5H_2O$$

NbO₂F 0.5H₂O + 4HF → H₂NbOF₅ + 1.5H₂O **Hydrogen gas produced at cathode**

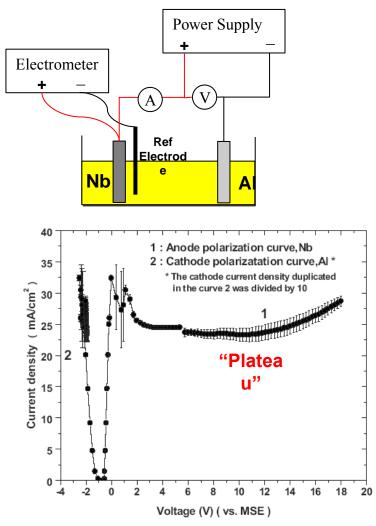




I-V Curve



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



Electrode potentials should be measured wrt a Reference Electrode!

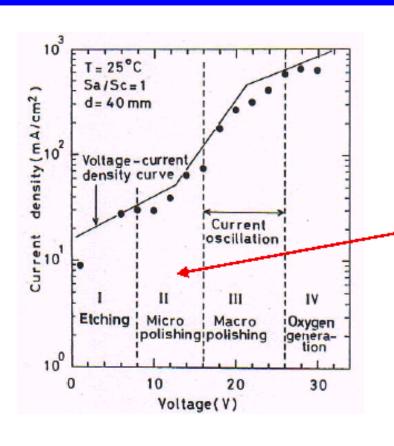


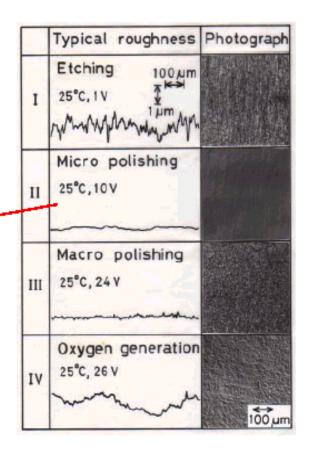


Good surface finish when in right I(V)

Talk by K.Saito in JLAB on Oct. 2003

Micro and macro electropolishing in niobium EP





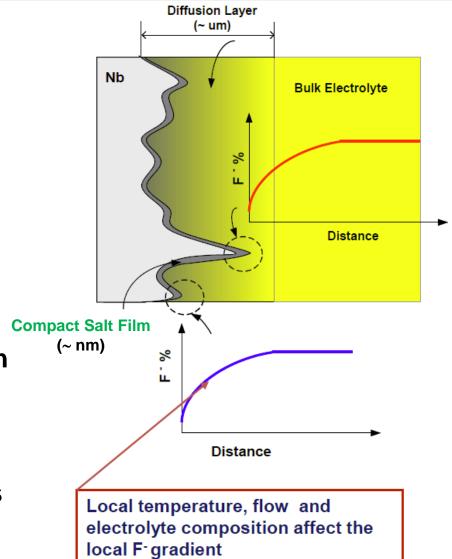
Kenji Saito, KEK, 1989





Basic Mechanism for EP

- Anodization of Nb in H₂SO₄ forces growth of Nb₂O₅
- F⁻ dissolves Nb₂O₅
- These competing processes result in current flow and material removal
- Above a certain anodization potential, the reaction rate plateaus, limited by how fast fresh F- can arrive at the surface (diffusion-limited)
- The diffusion coefficient sets a scale for optimum leveling effects







EP: tricky process

- The current density (30-100 mA/cm²) in the plateau region:
 - decreases linearly with lower HF/H₂SO₄ ratio
 - increases with increasing temperature
- Temperature during the process is maintained between 25 – 35 °C
- Current oscillations often observed during polishing (dynamic balance between oxide formation and dissolution). It's not a necessary condition for good surface finishing but indication of good processing parameters (temperature, voltage, agitation, HF concentration)

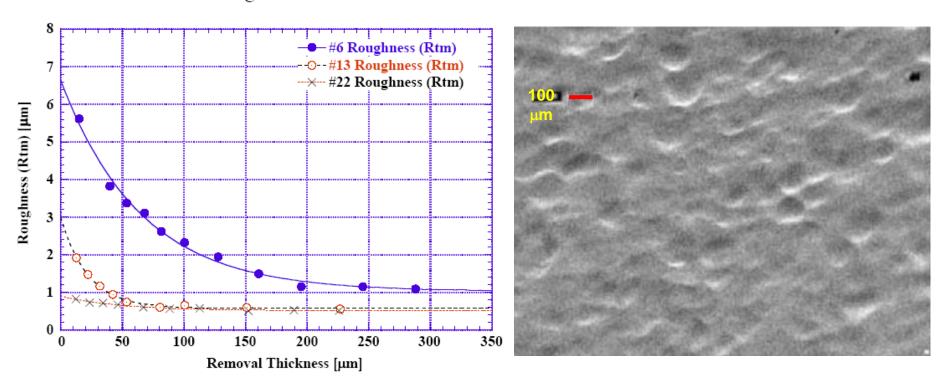
Finding the right balance among the processing parameters becomes complicated when polishing multi-cell cavities!





EP: Smooth Surface

Surface roughness with EP

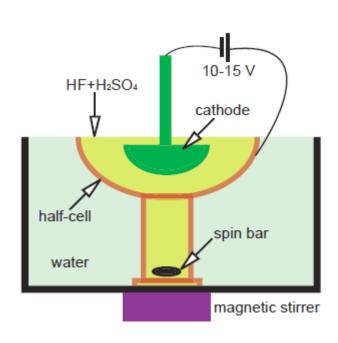


Typical roughness of ~ 1 μ m (100 x 100 μ m² scale)





EP Setups: Half-Cells



R.L. Geng, Cornell Univ.

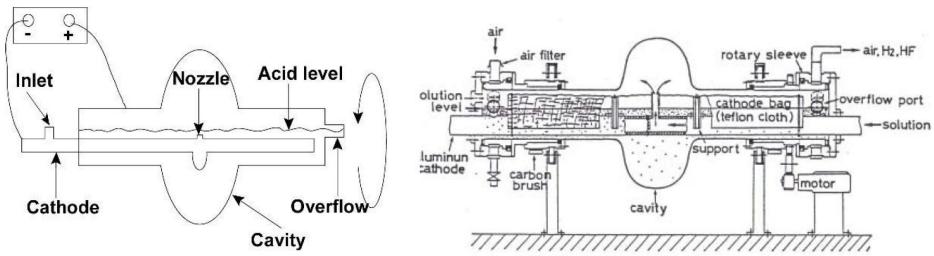


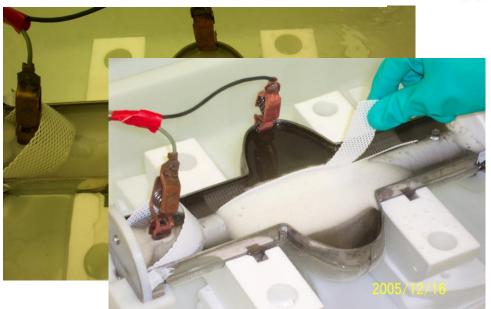
Material removal prior to final equatorial EBW





EP Systems: Single-Cell



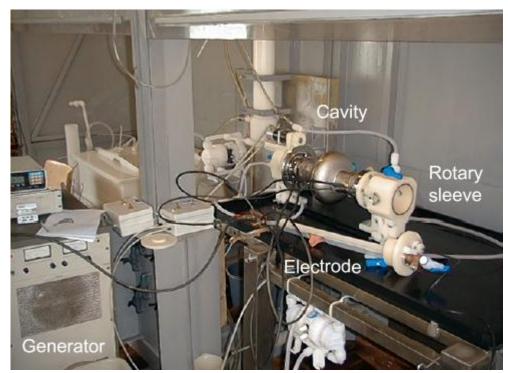


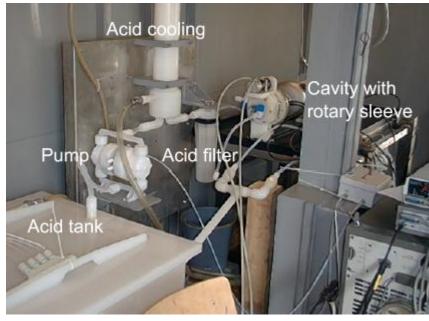






EP Systems: Single-Cell





Single-Cell setup at CERN Horizontal continuous electropolishing, polishing rate \sim 0.3 μ m/min



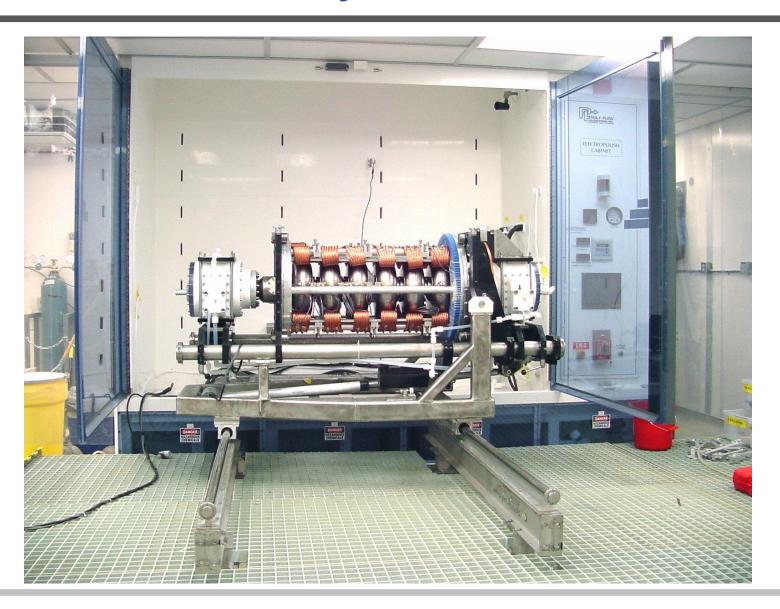




DESY



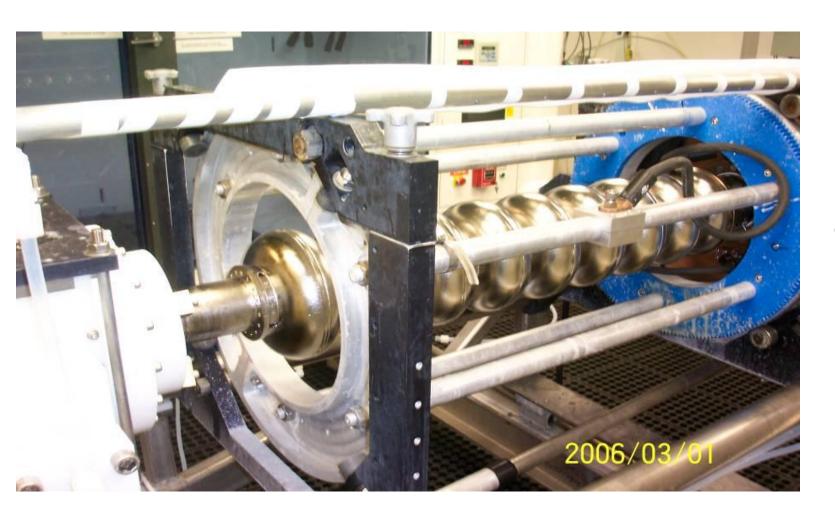




JLAB



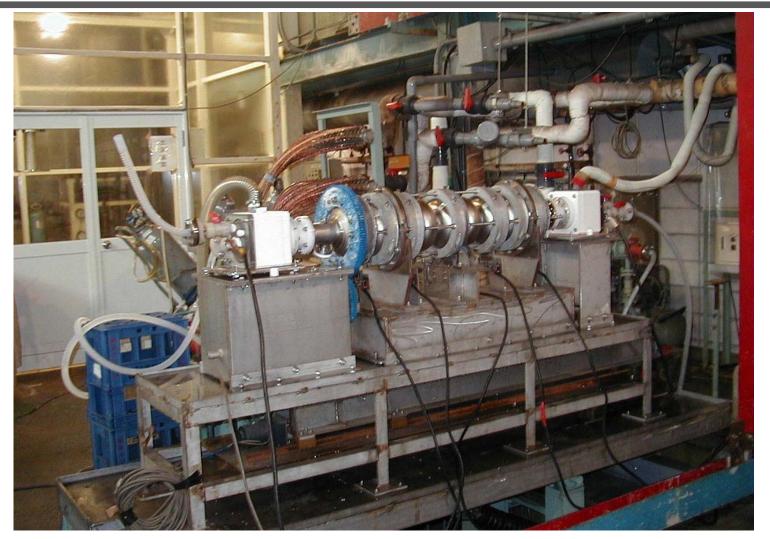




JLAB







Nomura Plating and KEK





EP Issues

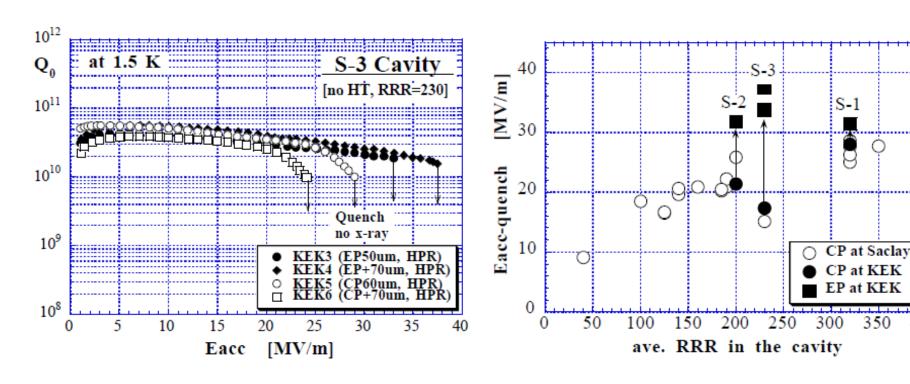
- HF disappears quickly from electrolyte due to surface temperature and evaporation and must be added routinely
- Difficult to add HF to the Sulfuric, reaction looses HF plus adds water to electrolyte which causes matt finishes
- Sulfur precipitates found on niobium surfaces (insoluble) and in system piping (monoclinic), impossible to add meaningful filtration
- Removal of sulfuric from surfaces difficult and requires significant amounts of DI water, hydrogen peroxide or alcohol rinses
- Typically cavity processed horizontally, slowly rotated
- Etch rate 2x on iris then equator

Why bother with EP?





EP: achieving high accelerating field

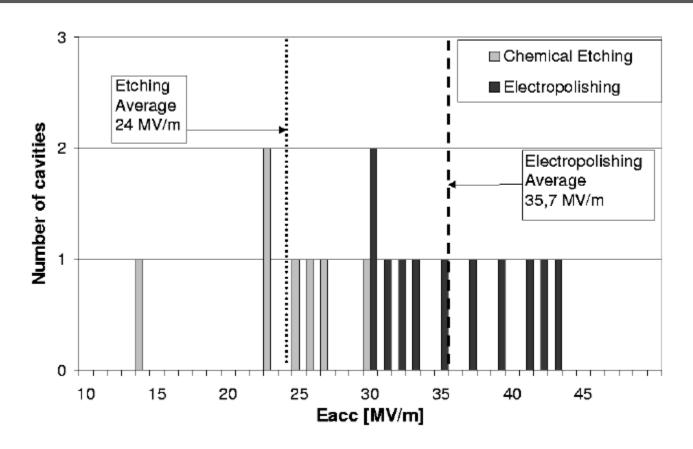


1999, KEK





EP: achieving high accelerating field

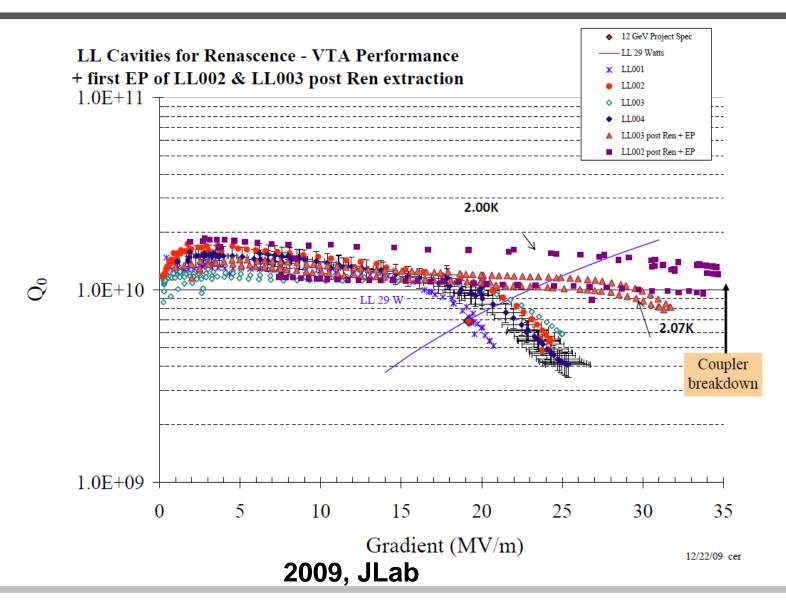


2001, CERN-CEA-DESY Collaboration





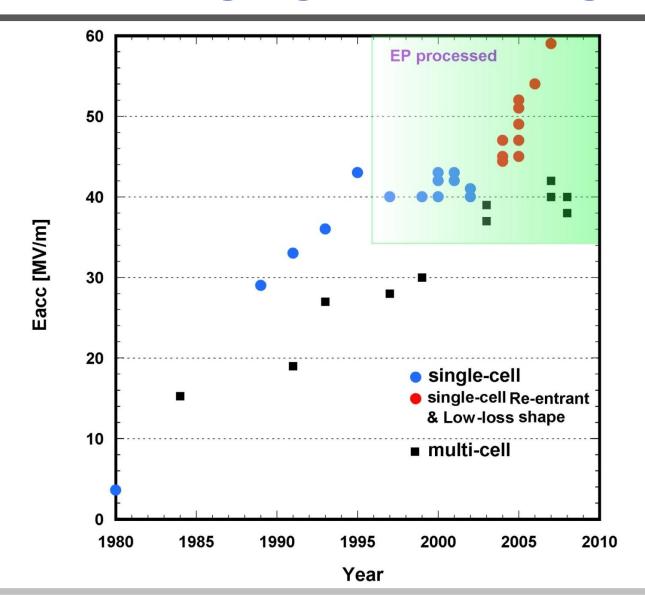
EP: achieving high accelerating field







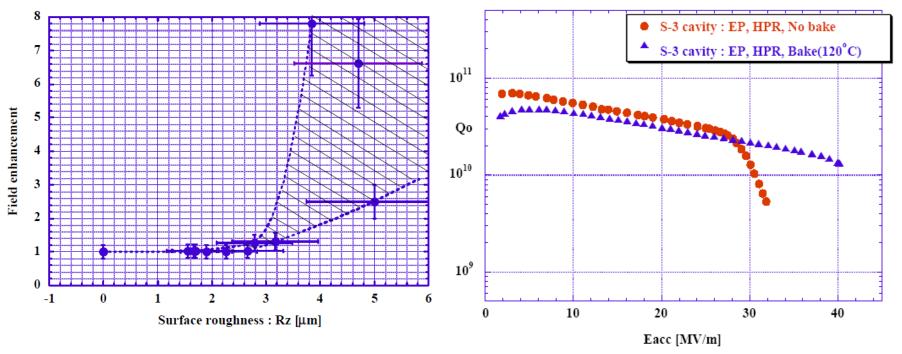
EP: achieving high accelerating field





Why Is EP Better?

- Q-drop recovers after baking
- Smoother surface



$$E_{\rm acc,max} \propto \frac{r \; H_{\rm c,RF}}{\beta}$$

 β = magnetic field enhancement factor r = reduction of critical magnetic field due to "polluted" surface layer





EP: used also in low- β cavities for heavy ion accelerator





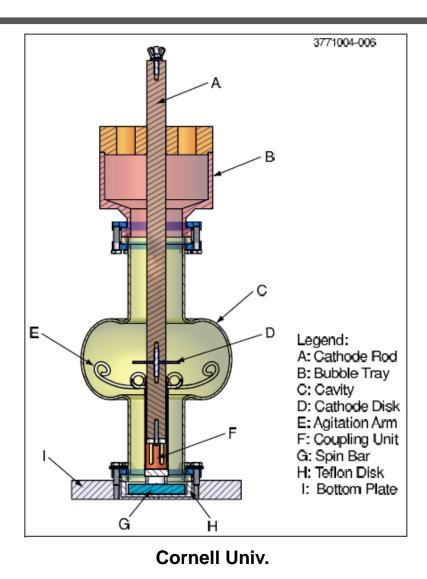
SRF cavities for ATLAS and future FRIB

Argonne Nat'l Lab

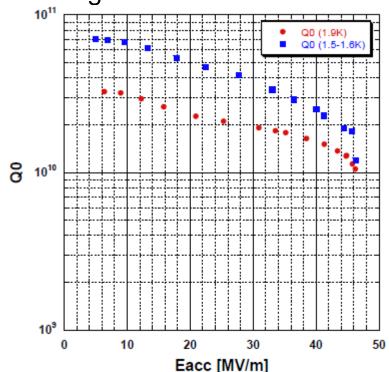




Vertical EP



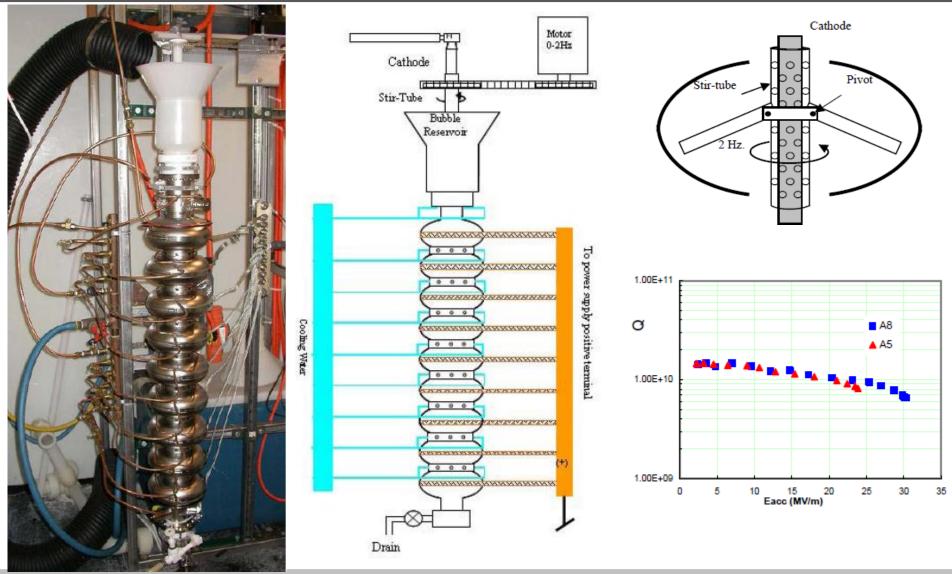
- No rotary acid seals
- Twice removal rate than horizontally rotating EP
- No sliding electrical contacts
- No large acid reservoir and heat exchanger







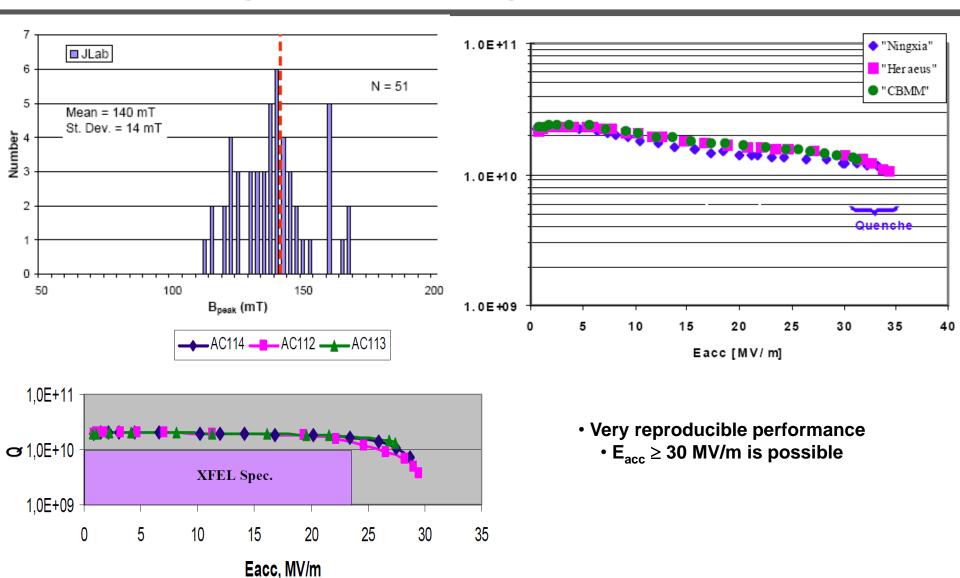
Vertical EP





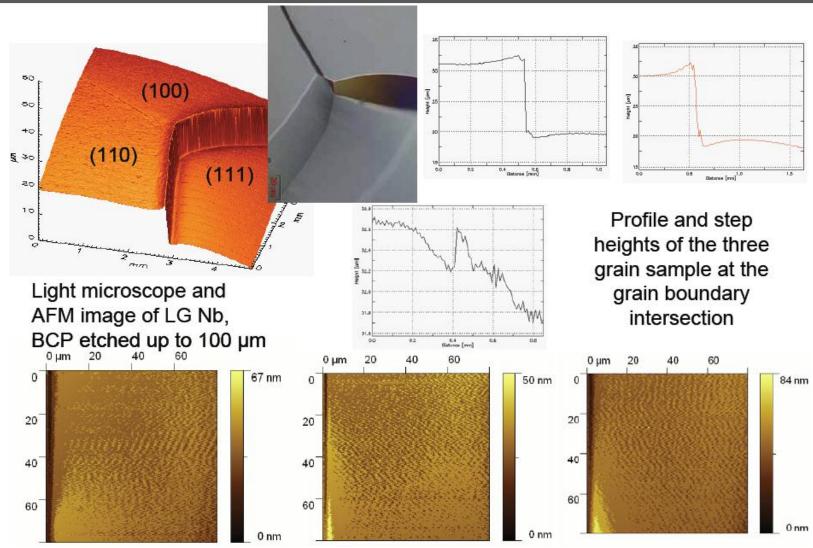


Challenge to EP: Large Grain Nb & BCP





Large-Grain Nb Surface After BCP

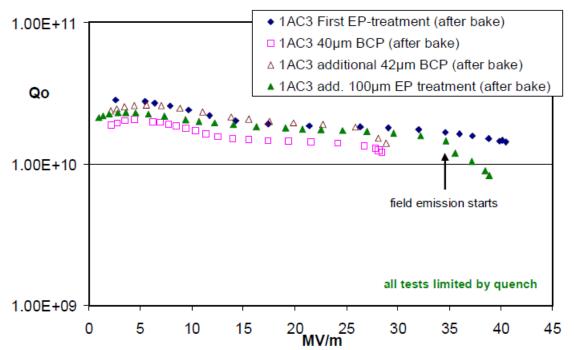


Roughness of fine-grain Nb sample treated by EP is ~ 250 nm





Challenge to EP: Large Grain Nb & BCP



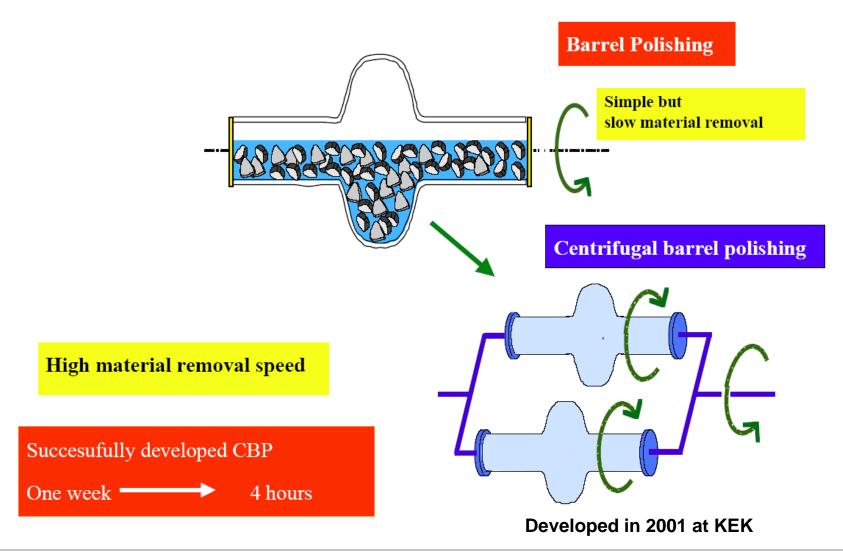
| | Quench gradient after final EP | Quench gradient after final BCP |
|------------|-----------------------------------|------------------------------------|
| Large | | |
| grain Nb | (33 – 43) MV/m | (25 – 30) MV/m |
| Fine grain | $(36 \pm 4) \text{MV/m}$ | Data not sufficient |
| Nb | (30 ± 4) W V/III | Data not sunicient |

- Studies at DESY show higher Eacc after EP even for large-grain Nb
- The typical performance of large-grain Nb cavities treated by BCP would satisfy the requirements for most accelerator projects





Centrifugal Barrel Polishing

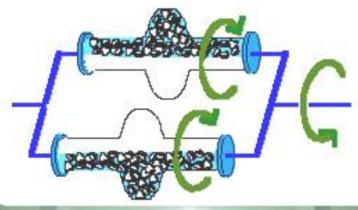






CBP Implementation

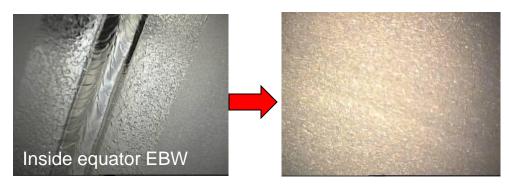
Centrifugal Barrel Polishing (CBP)





Implementation:

- Plastic stones and liquid abrasive added inside cavity and rotated
- Stones rubbing on surface removes material thus smoothing the surfaces (including weld areas)
- Benefit is less overall chemistry needed (80 μm) and smooth weld areas



• Removal of material 2x on equators then irises. Average removal rate $\sim 5~\mu\text{m/h}$





Barrel Polishing Machine at JLab







• Removal rate $\sim 3 - 4 \mu m/h$







Required Procedures for Qualifying SRF Cavities

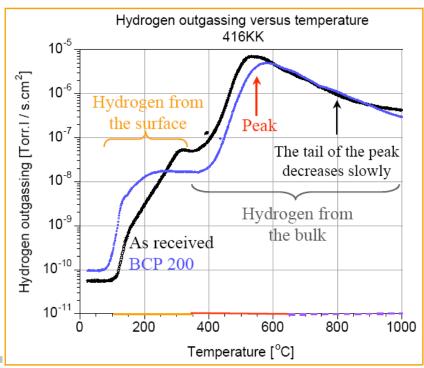
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Heat Treatment for H-degassing

- H absorption occurs during chemical and/or mechanical material removal
- Reduce bulk H concentration in Nb to avoid Q-disease
- The heat treatment also "stress-relieves" the Nb
- Different parameters at different labs:
 - 600 °C/10 h at JLab
 - 800 °C/2 h at DESY
 - 750 °C/3 h at KEK



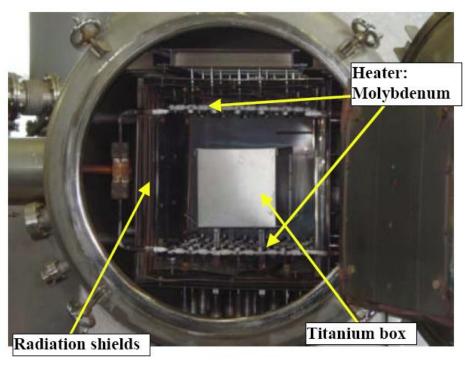




High Temperature Vacuum Furnace



Heat Treatment Furnace at JLab up to 1250 °C, P ≤ 10⁻⁶ Torr



Vacuum furnace in KEK : Temp.= 1300 °C max, Vac. = 1xE-6 torr

Use Residual Gas Analyzer to monitor the partial pressure of residual gases during heat treatment





Required Procedures for Qualifying SRF Cavities

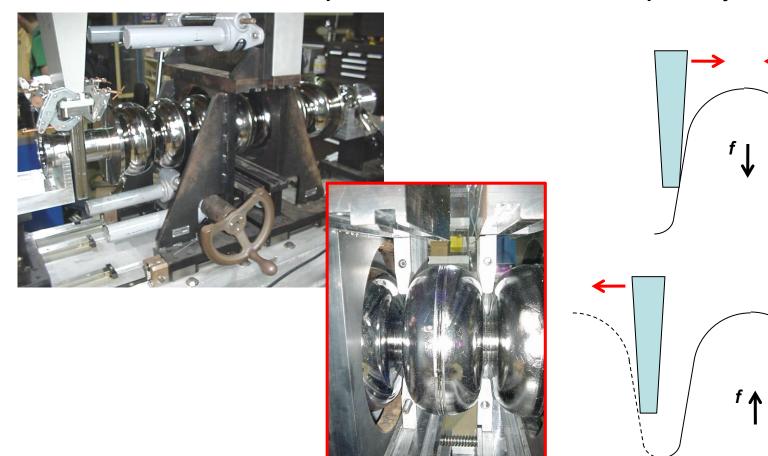
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Mechanical Tuning

 Small mechanical adjustments to the cavity's cells to obtain flat field profile and desired frequency







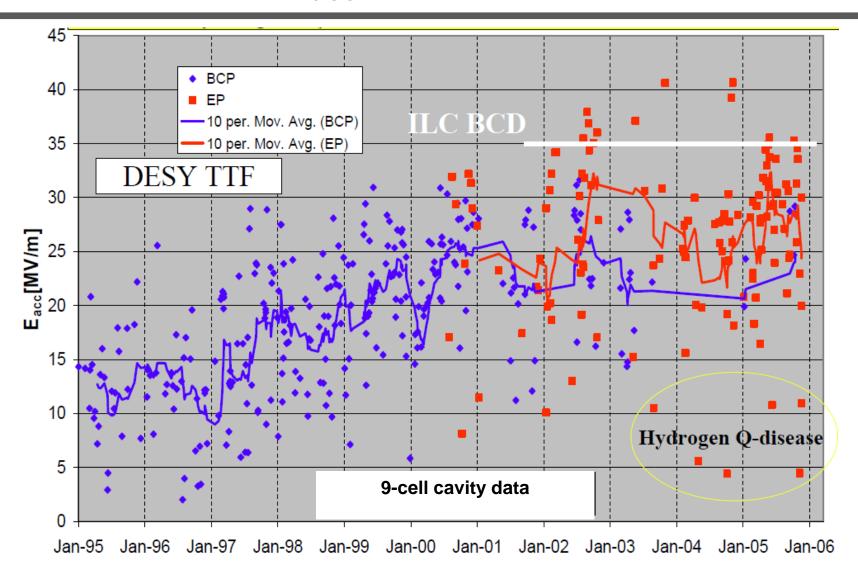
Post-EP Cleaning

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EP: high E_{acc} but large scattering

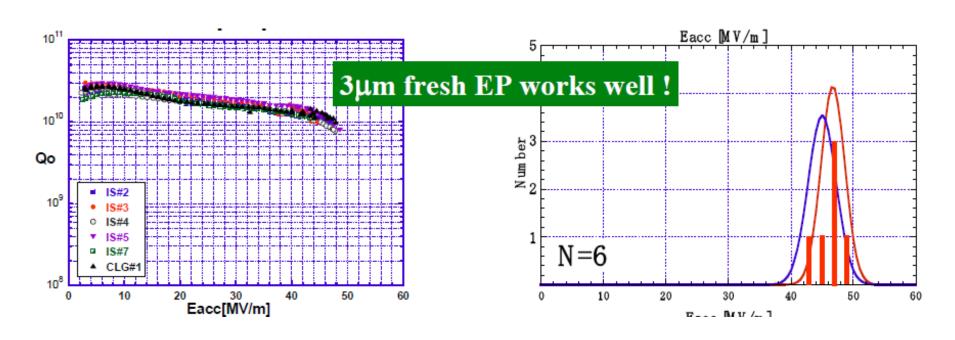






Post-EP Cleaning Processes

- Ethanol Rinse (DESY)
- "Flash" BCP (10 μm) (DESY)
- "Flash" EP (3 μm, fresh acid, no re-circulation) (KEK)
- Ultrasonic Degreasing with Micro-90 and hot water (JLab)







Required Procedures for Qualifying SRF Cavities

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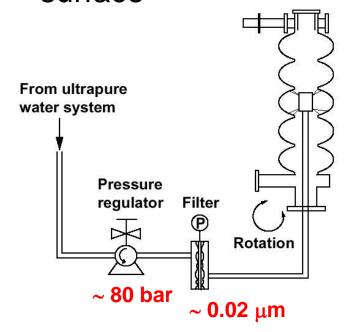
Low-temperature baking

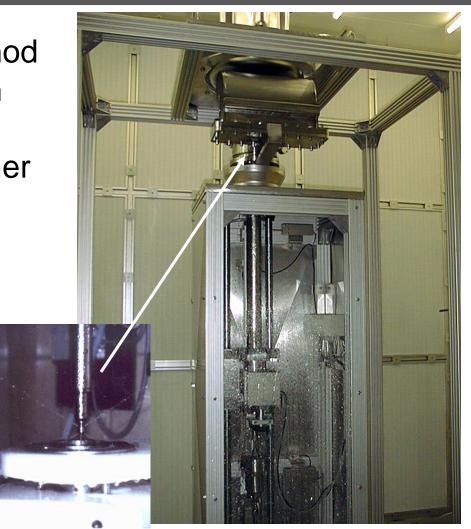




High Pressure Rinsing (HPR)

 SRF cavities cleaning method to remove particulates from handling and contaminants after chemistry from the inner surface





ACCEL Instruments





High Pressure Rinsing (HPR)

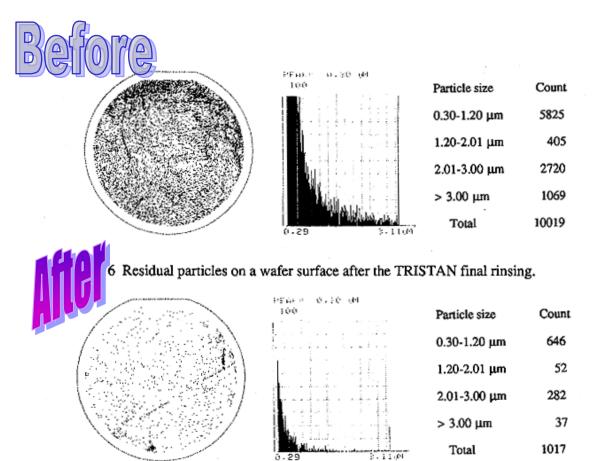
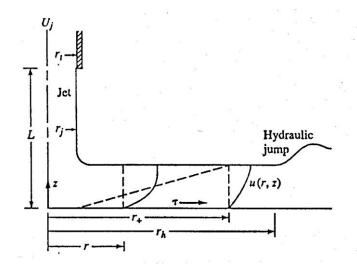


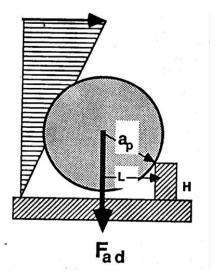
Fig. 7 Residual particle on a wafer surface after HPR.





Particle Removal Mechanism





- Hydrodynamic model allows estimating the shear stress τ of the water jet, which depends on flow rate and pressure
- Particle removal by rolling if the water shear stress is greater than a critical shear stress τ₀, related to the particle size, adhesion force and surface roughness _____

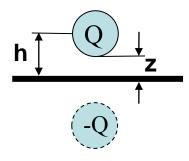
$$\tau_0 = \frac{F_{ad}}{44a_p^2} \sqrt{2\frac{H}{a_p} + \left(\frac{H}{a_p}\right)^2}$$





Adhesion Forces

Particle of diameter d



Adhesion forces:

Coulomb

• Capillary
$$F = \alpha \frac{Q^2}{4\pi\varepsilon_0 d^2}$$

$$F = 2\pi\gamma a$$

• Van der Waals $F=2\pi\gamma d$ γ : surface tension

Electrical double layer

$$F = \frac{7.2 \text{eV}}{16\pi} \frac{d}{z^2}$$

$$F \propto \frac{\Delta \Phi^2 d}{z}$$

Example: 1 μm glass particle on

water 1.4×10⁻⁷ N

4.5×10⁻⁷ N

3×10⁻⁸ N

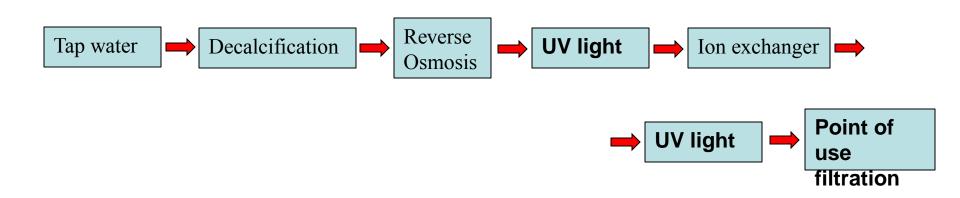
1×10⁻⁸ N



Ultrapure Water Quality

- Water quality of ultrapure water for SRF cavities preparation:
 - Resistivity: 18.2 MΩcm
 - Total oxydable carbon (TOC): < 5 ppb
 - Particulate counts (> 0.3 μm/l): < 10
 - Bacteria counts: < 0.1 CFU/100ml

Typical water purification stages:







HPR QA

- Online monitoring of TOC, resistivity and particulate counts
- Collection of water from rinsed cavity for particulate analysis









HPR Systems



HPR stand inside the clean room at JLab







HPR Systems





HPR stand inside the clean room at DESY





HPR spray heads optimization



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





HPR Jet Characterization





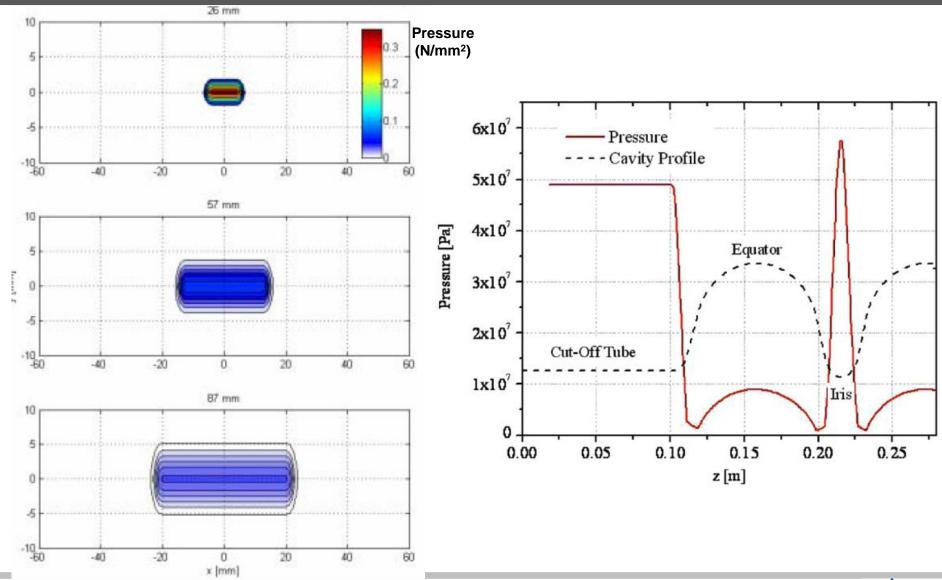
• Use a load cell to measure the force vs. distance of the water jet

$$F = \rho \cdot Q \cdot u \qquad u = \sqrt{\frac{2 \cdot p}{\rho}} \qquad \begin{array}{l} \text{u = velocity} \\ \text{Q = flow} \\ \text{p = pressure} \\ \text{p = density} \end{array}$$





Water Pressure vs. Distance







Different HPR Configurations

| Lab. | # nozzles | Tested nozzles | Flow [l/min] (1 nozzle) | Pump Press [bar] |
|----------------|--------------|-----------------------------------|----------------------------|------------------------|
| JLAB Prod | 2 | SSC-FAN: 1502 4002 40015 | 5@85 bar | 85 |
| JLAB R&D | 2 | SSC-FAN 1502 | 5@85 bar | 0.5 |
| | 9 | Φ=0.4 mm Sapphire | | 85 |
| KEK Tsukuba | 8 | Ф=0.6 mm SS | 1.5@70 bar | 70-50 |
| KEK | 8 | Φ=0.6 mm SS | 1.1@50 bar | 50-40 |
| Nomura | | Φ=0.6 mm SS | 0.9@40 bar | |
| DESY | 8 | Φ=0.6 mm Sapphire | 1.6@100 bar | 90-110 |

 "Fan" jet allows greater surface coverage compared to a standard round jet

 HPR Duration: 3 - 12 h on 9-cell cavity

• Cavity rotation: 2 – 20 rpm

Wand movement: 8 – 50 mm/min

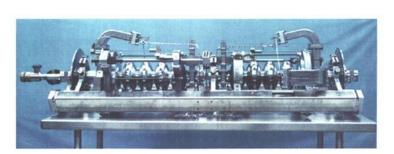




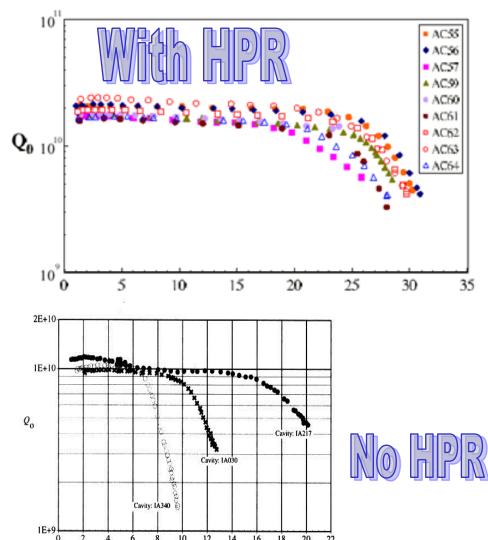
Performance Improvement After HPR



TESLA Cavities



CEBAF Cavities



E acc (MV/m)





HPR Issues



ISSUES:

- HPR systems are still not optimized for the best surface cleaning performance
- Surface left in a vulnerable state, wet



Dry Ice Cleaning

- Complementary method to HPR, developed at DESY
- Liquid CO₂ jet flowing through a nozzle and resulting in a snow/gas mixture at a temperature of 194 K

 Removal of hydrocarbons and sub-micron particles while keeping the surface dry by

- Thermal
- Mechanical
- Chemical
- Could be applied to a fully assembled cavity mounted horizontally as a part of a "cavity-string"





Required Procedures for Qualifying SRF Cavities

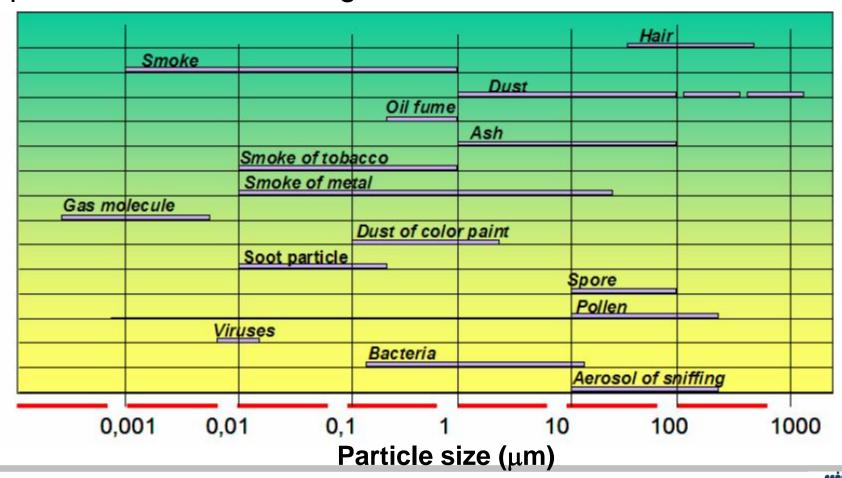
- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (~150 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
 - Additional "cleaning" steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking





Particulates in Air

 Cleanroom technology is required to prevent airborne particulates from settling on the surface of SRF cavities

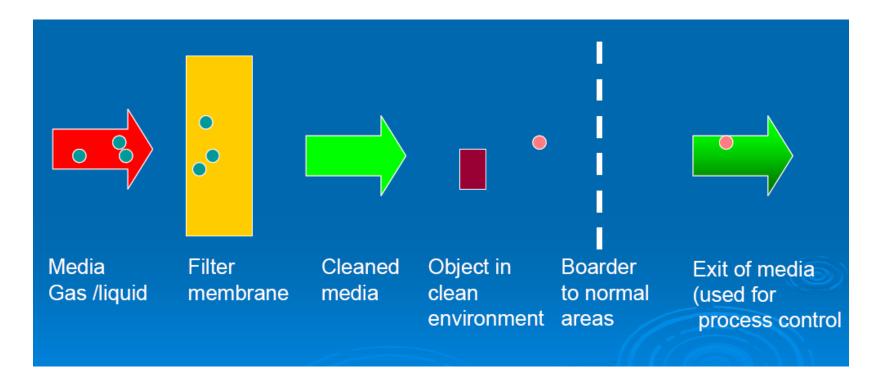




O_{LD} DOMINION

Cleanroom Technology

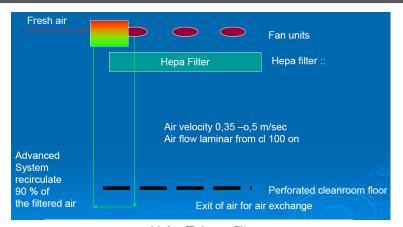
Cleanroom: a controlled environment in which all incoming air, water and chemicals are filtered to meet high standards of purity. Temperature, humidity and pressure are controlled, but the key element is air filtrations.

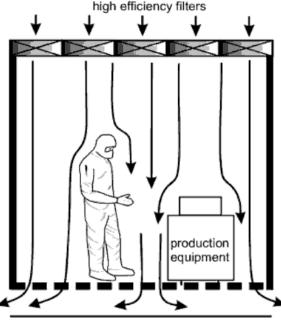




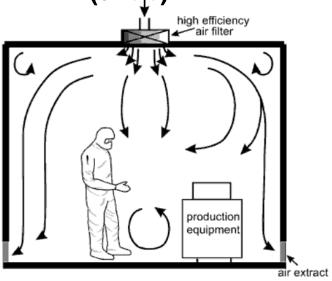


Type of Cleanrooms





Non-Unidirectional airflow type (JLab)



Unidirectional airflow type (DESY)





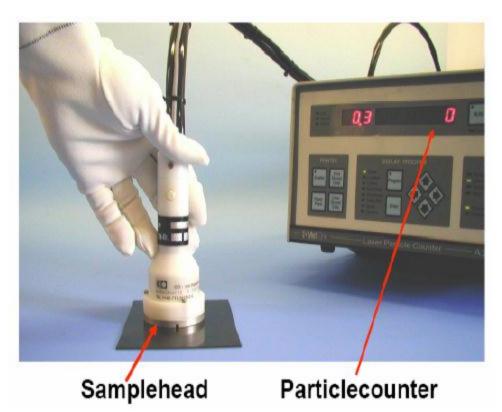
Cleanroom Classification

| ISO Classification number | Maximum concentration limits (particles/m³ of air) for particles equal to and larger than the considered sizes shown below | | | | | | | | | | |
|---------------------------------|--|----------------|---------|----------------|---------|-----------|-----------|----------|---------|----------------|--|
| | >=0.1μm>=0.2μm | | >=0.3µm | | >=0.5μm | | >=1μm | | >=5.0µm | | |
| ISO Class 1 | 10 | 2 | | | | | | | | | |
| ISO Class 2 | 100 | 24 10 | | | 4 | | | | | | |
| ISO Class 3 | 1 000 | 237 102 | | 2 35 | | 35 | | 8 | | | |
| ISO Class 4 | 10 000 | 2 370 | 1 020 | 1 020 | | 352 | | 83 | | | |
| ISO Class 5 | 100 000 | 23 700 | 10 200 | | 3 520 | | 832 | | 29 | | |
| ISO Class 6 | 1 000 000 | 237 000 | 102 000 | | 35 200 | 35 200 | | 8 320 | | 293 | |
| ISO Class 7 | | | | | 352 00 | 00 | 83 20 | 00 | 2 930 |) | |
| ISO Class 8 | | | | | 3 520 | 000 | 832 (| 000 | 29 30 | 00 | |
| ISO Class 9 | | | | | 35 200 | 000 0 | 8 320 000 | | 293 000 | | |
| ISO 14644-1 Classes | Class 3 | Class 4 | | Class 5 | | Class 6 | | Class 7 | | Class 8 | |
| FS 209 Classes | Class1 | Class 10 |) | Class 10 | 00 | Class 100 | 00 | Class 10 | , 000 | Class 100, 000 | |
| | | † | | | | | | | | | |
| | | Cavity assembl | y | Clean for S | | l | | | | | |





Particle Counters









People in Cleanrooms

- People are a major source of particulate contamination inside a clean room through:
 - Body Regenerative Processes Skin flakes, oils, perspiration and hair.
 - Behavior Rate of movement, sneezing and coughing.
 - Attitude Work habits and communication between workers.







Assembly: Vacuum Hardware

 The cavity strings have to be vacuum tight to a leak rate of < 1 x10⁻¹⁰ torr l/sec

 The sealing gaskets and hardware have to be reliable and particulate-free

 The clamping hardware should minimize the space needed for connecting the beamlines





Assembly: Vacuum Hardware

Present choice for ILC cavities:

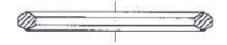
diamond-shaped AIMg₃ –gaskets + NbTi flanges + bolts

Also used for SNS cavities

Alternative:

radial wedge clamp, successfully used for CEBAF upgrade cavities

AlMg-Gasket





Radial Wedge Clamp







Cavity Assembly









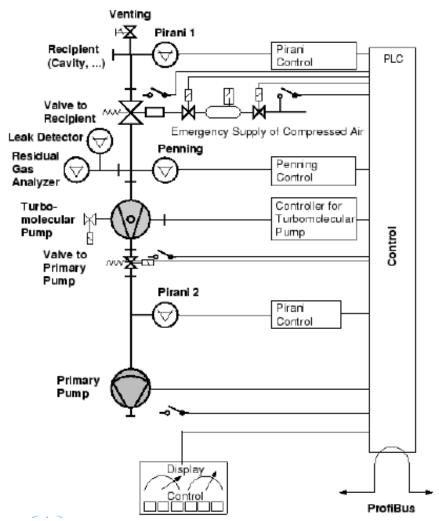
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- Low-temperature baking

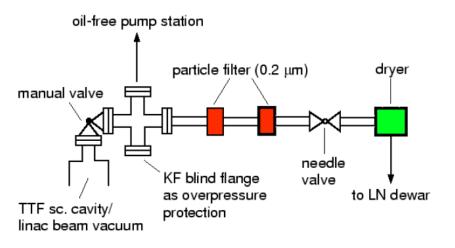




Clean Evacuation



- Oil-free pump stations with leak check and residual gas analyzer
- Laminar venting with pure, particle filtered N₂ or Ar



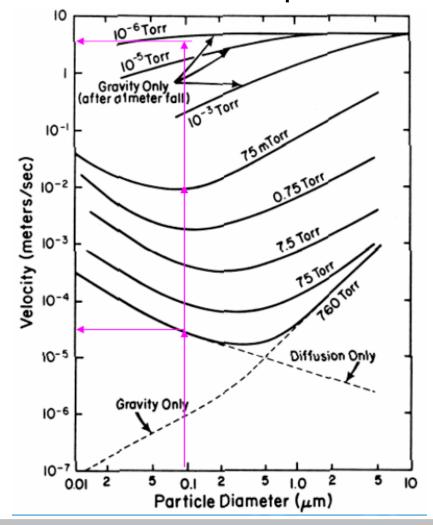




Clean Vacuum Systems

"Dirty" or "contaminated"
 (hydrocarbons, air leaks)
 vacuum system can re-contaminate the surface of a clean cavity!

Settling velocity for particles in air at room temperature







Required Procedures for Qualifying SRF Cavities

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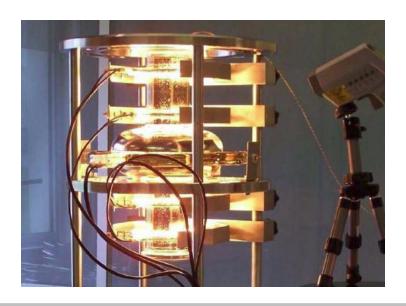


Low-Temperature Baking



Hot N₂ gas uniformly heats up the cavity (JLab)

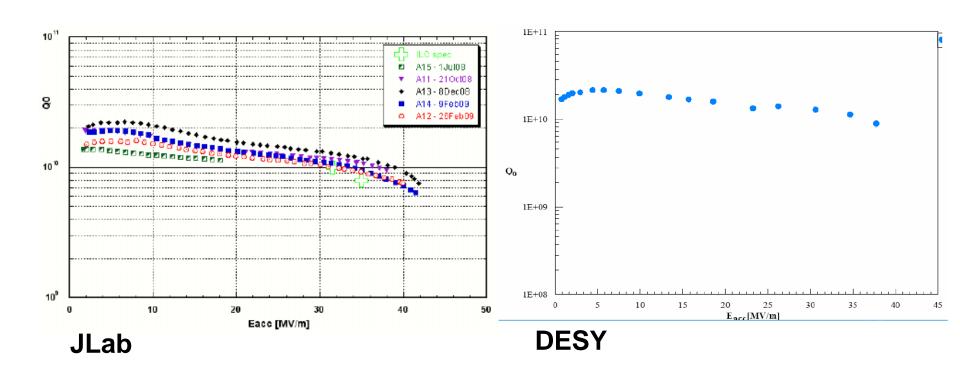
Infrared heaters heating the open cavity inside the cleanroom (Saclay)







If Everything Works Well...



 $E_p\cong 80$ MV/m, $B_p\cong 170$ mT can be achieved in the vertical test of 9-cell ILC cavities (~ 1 m² of Nb surface)





Additional Steps for Cavity String

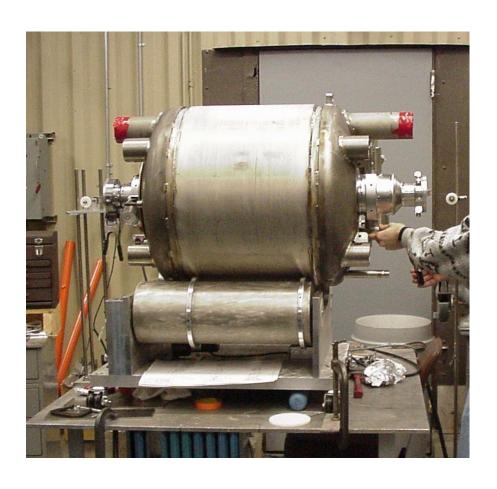
- Final mechanical tuning
- He-vessel welding
- Degreasing
- Final material removal (10-20 μm)
- Final HPR
- Horizontal assembly into cavity-string
- Evacuation of cavity string





Helium Vessel Welding









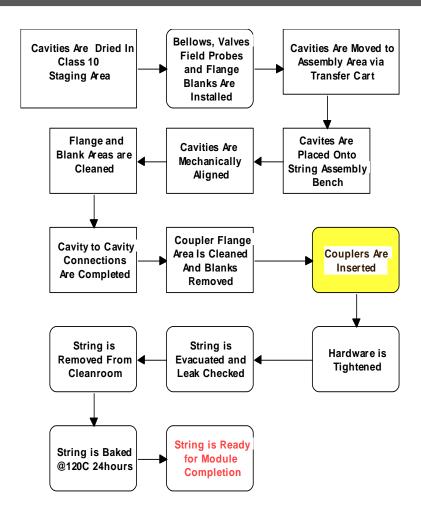
String Assembly

- A cavity string is assembled in a class 10 or class 100 clean room on an assembly bench over a period of several days after they have been qualified in a vertical or horizontal test.
- Prior to assembly, the cavities are high pressure rinsed for several hours, dried in a class 10 clean room, mounted onto the assembly bench and auxiliary parts are attached.
- The most critical part of the assembly is the interconnection between two cavities, monitored by particle counting





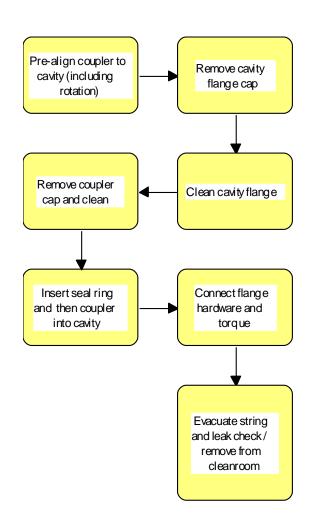
Example of Cavity Assembly Sequence

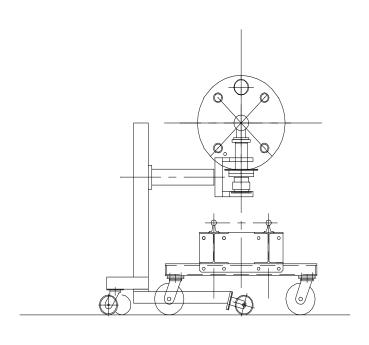






Coupler Insertion Procedure



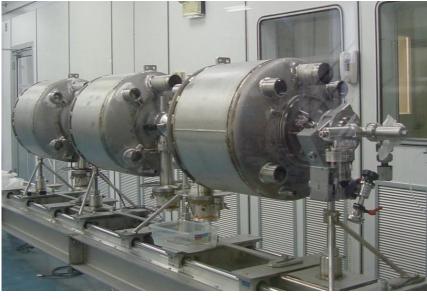






String Assembly





SNS β_g =0.61 string at JLab: 3 cavities per string

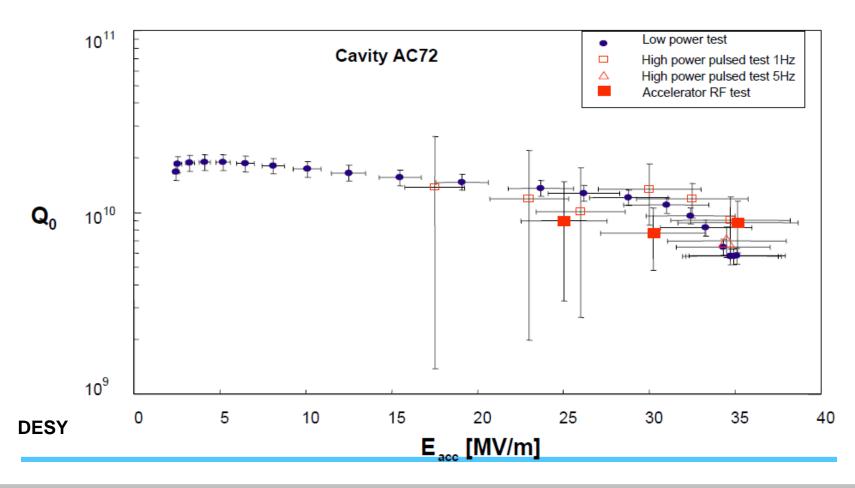
XFEL at DESY: 8 cavities per string

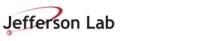




If Everything Works Well...

35 MV/m without field emission in operation with electron beam is possible!







Comments on Facilities and Process Steps

RF Cavities

 RF structures have excellent quality in materials and fabrication but flange designs require significant hardware for assembly and extensive manual labor → lots of room for errors

Facilities

- Cleanroom environments are typically excellent, easy to monitor
- DI water quality excellent in most cases, easy to monitor
- Sub-component cleaning not at same level with cleaning quality for cavities
- Many system failures reported, leading to large recovery times
- No two process system designs the same

Process Steps

- Assembly steps present the most interaction and largest source of particulate contamination, very difficult to monitor
- Subcomponent cleaning insufficient but easy to monitor
- BCP Chemistry in good control easy to monitor
- EP currently has less process control and more process variables.



History Plot of High Gradient

