SURFACE PREPARATION
CLEAN ROOM TECHNIQUES

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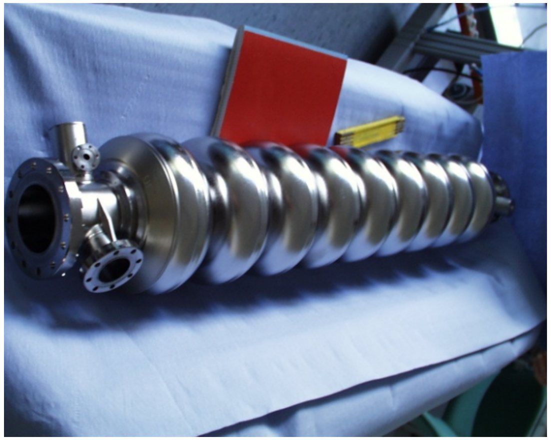
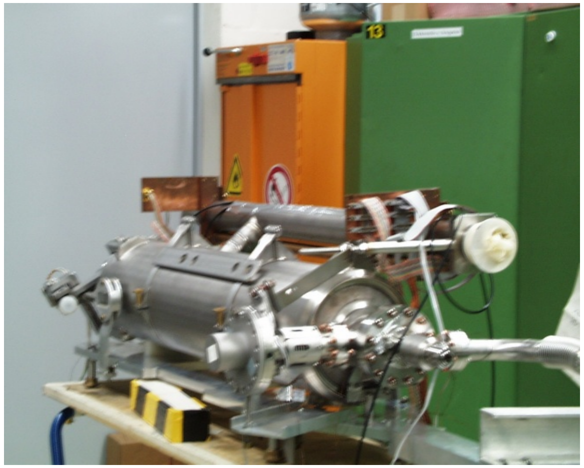
Required Procedures for Qualifying SRF Cavities

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of internal bulk damage layer of niobium from fabrication (150um)
- Removal of hydrogen gas (absorbed during chemistry) from bulk Nb
- Chemical removal of internal surface for clean assembly (10-20um)
- High Pressure Rinsing to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation

Basic Steps





Preparation step A
Removal of damage layer / post purification / tuning

↓

Preparation step B
Final cleaning and assembly for vertical test

↓

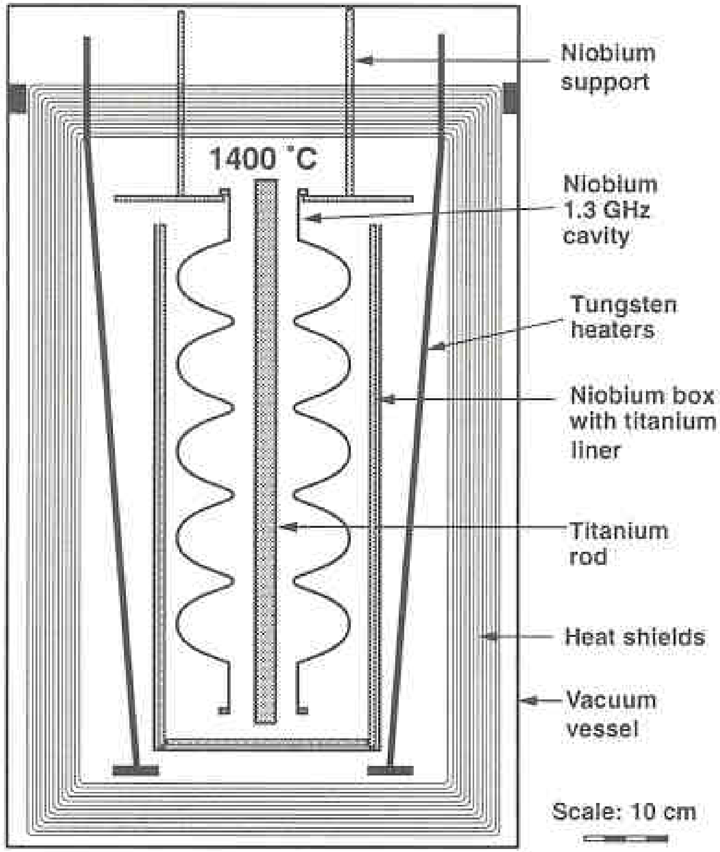
Preparation step C
Welding of connection to H vessel / He vessel welding

↓

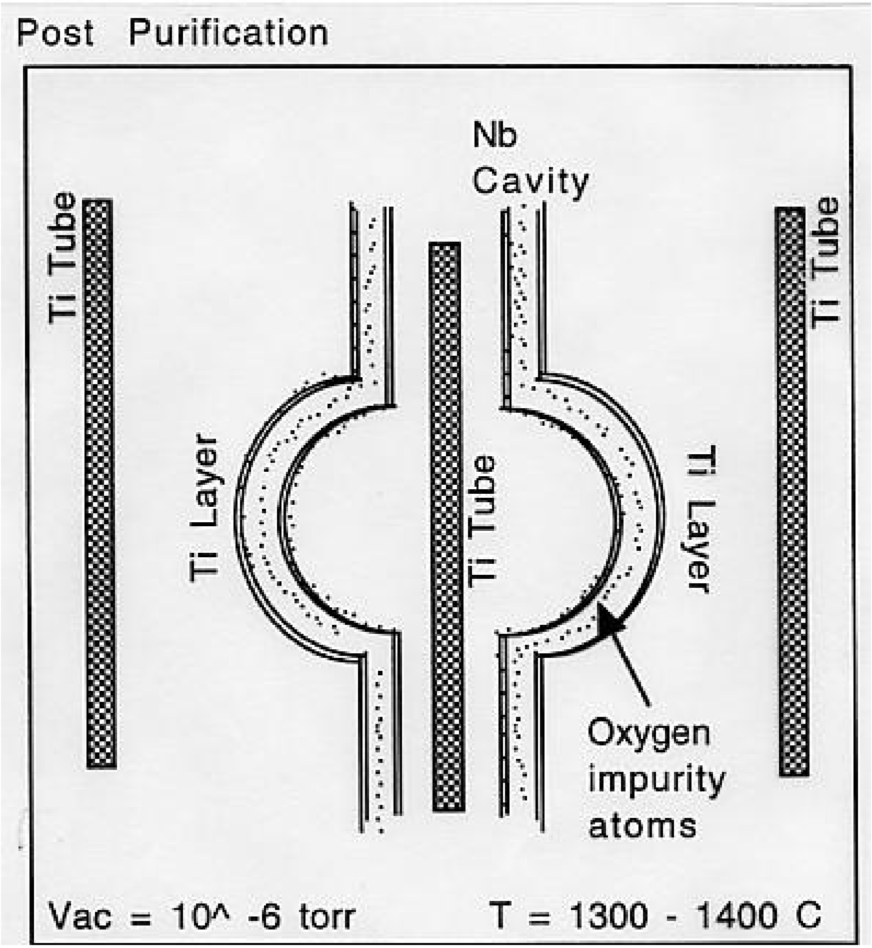
Preparation step D
Final cleaning and assembly for module / horizontal test

Post Purification

- Cavity is fired at 1350°C with Ti for several hours
  - Ti will cover the Nb surface and cleans the Nb bulk by solid state diffusion
  - RRR is increased from 300 to 500-600
- Cavity is soft after firing, yield strength is reduced from 50 to 10 N/mmTi must be removed by etching around 80 μm from the surface

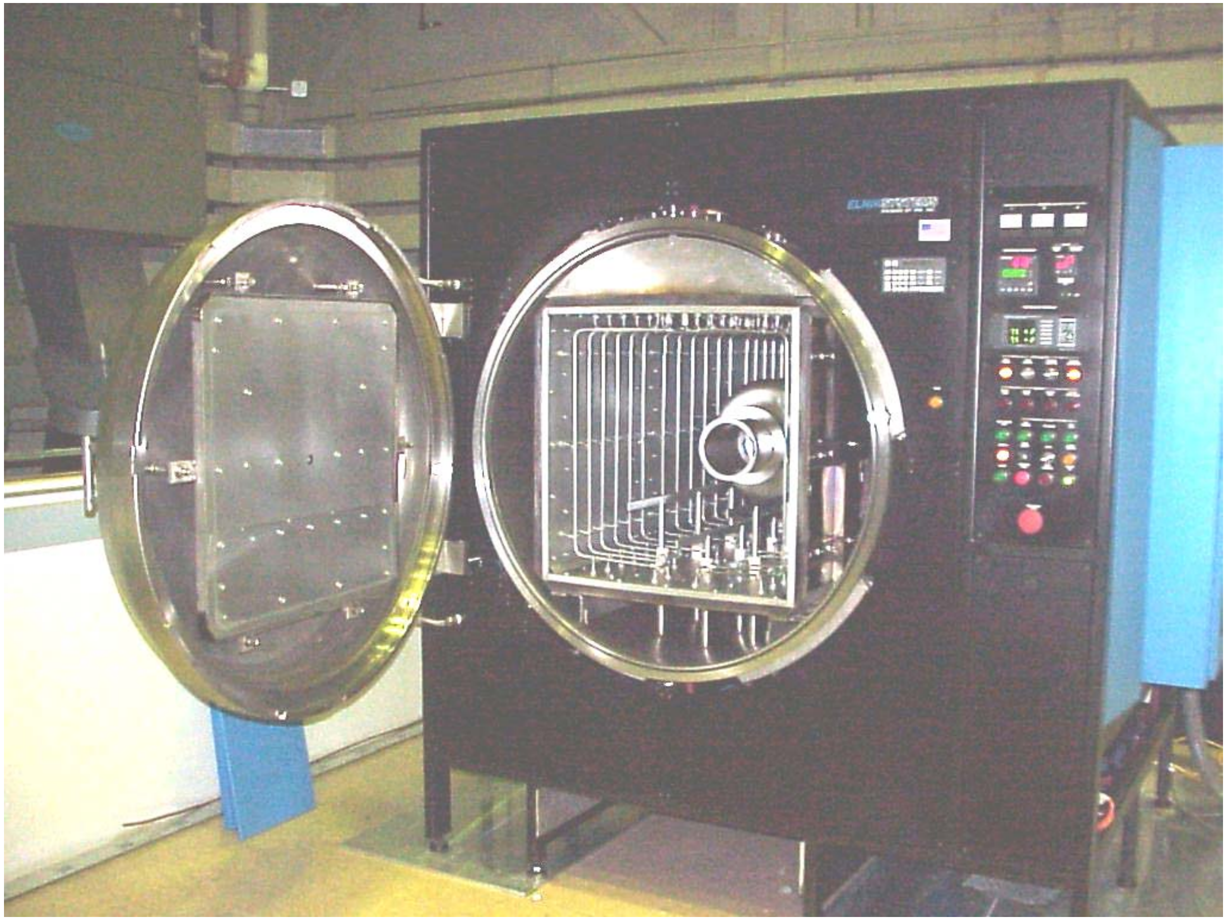


Post Purification



High Temperature Heat Treatment

Heat Treatment Furnace at Jlab up to 1250C



Ultrasonic Degreasing:

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:

- Ultrasonic degreasing with detergent and ultra pure water
- Usually performed in Hepa filtered air
- Water quality is good, 18M-Ohm-cm, Filtration >0.2um
- 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.

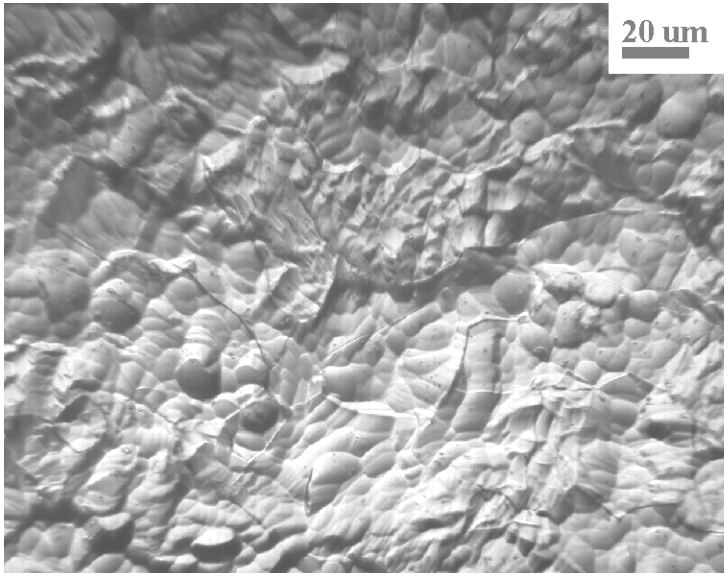
Acid Etching of Sub-components & Cavities:

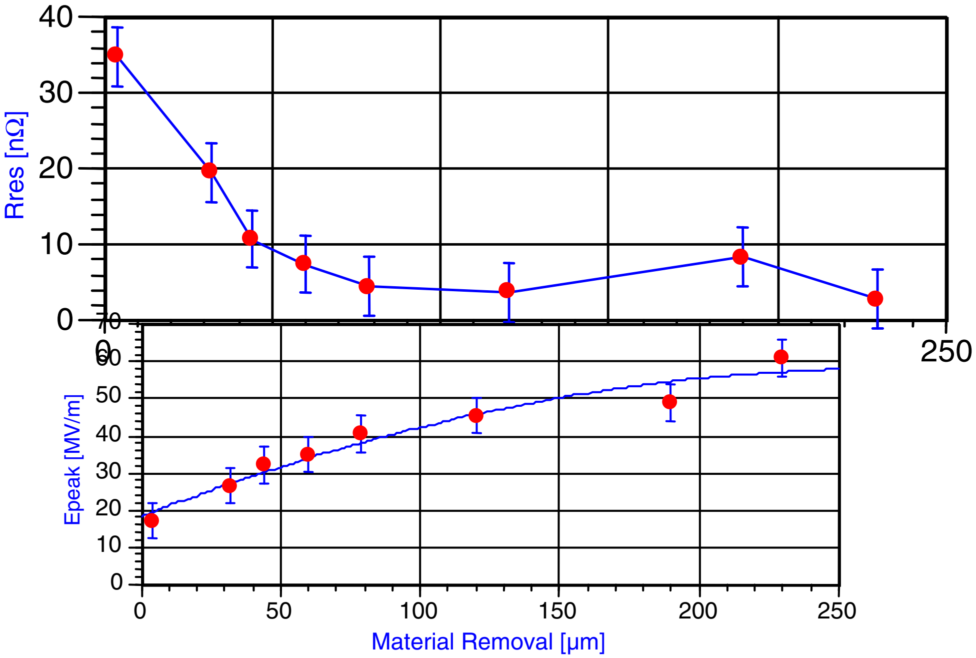
- Sub-components require
  - Removal of oxides which come from fabrication steps → lower losses and improve sealing
- Cavities require:
  - Interior chemistry to remove damaged surface layer incurred in welding and deep drawing (100-200um)
  - Exterior chemistry to remove surface oxides that occurred in welding (10-30um)

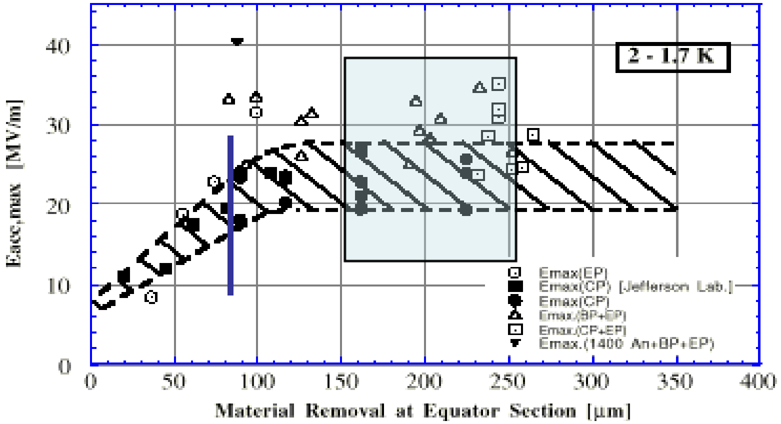
Implementation: (BCP or EP)

- 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





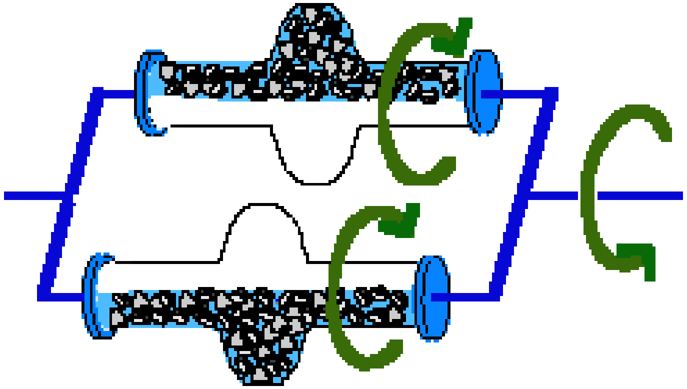


P. Kneisel

K. Saito

Alternative Method of Material Removal → Barrel Polishing

Centrifugal Barrel Polishing (CBP)



[T.Higuchi, K. Saito, SRF 2003]

Implementation:

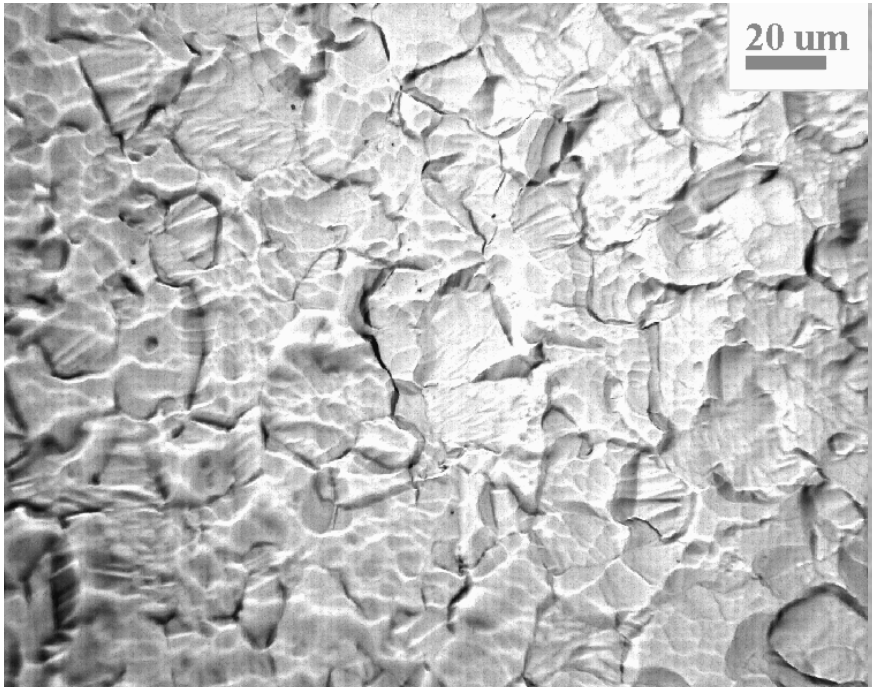
- Plastic stones and liquid abrasive added inside cavity and rotated

- Stones rubbing on surface removes material thus smoothing the surfaces (including weld areas)

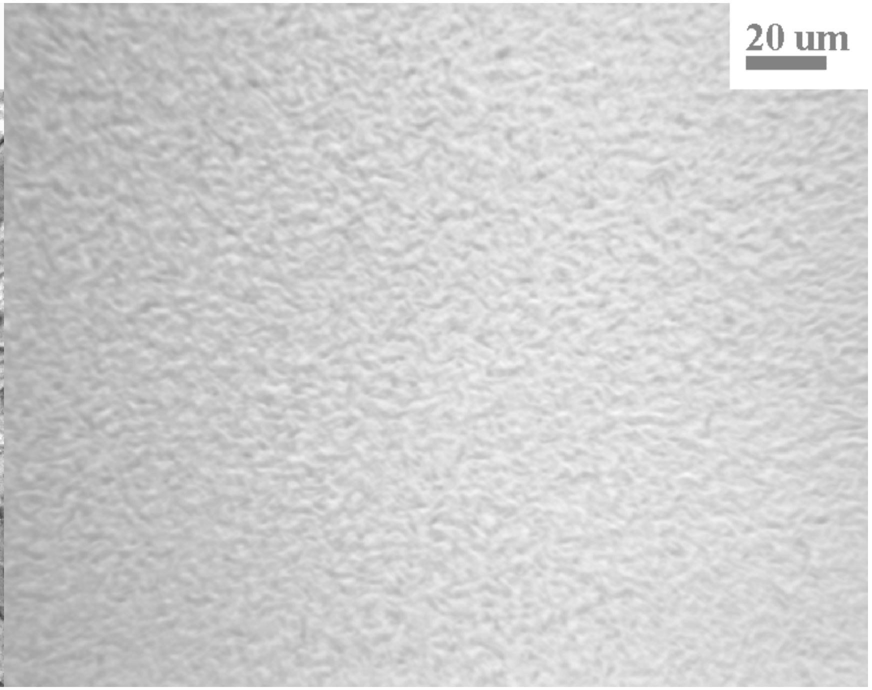
- Benefit is less overall chemistry needed (80um), KEK

- Removal of material 2x on equators then irises

Niobium Material Removal by Chemistry



Niobium surface after BCP

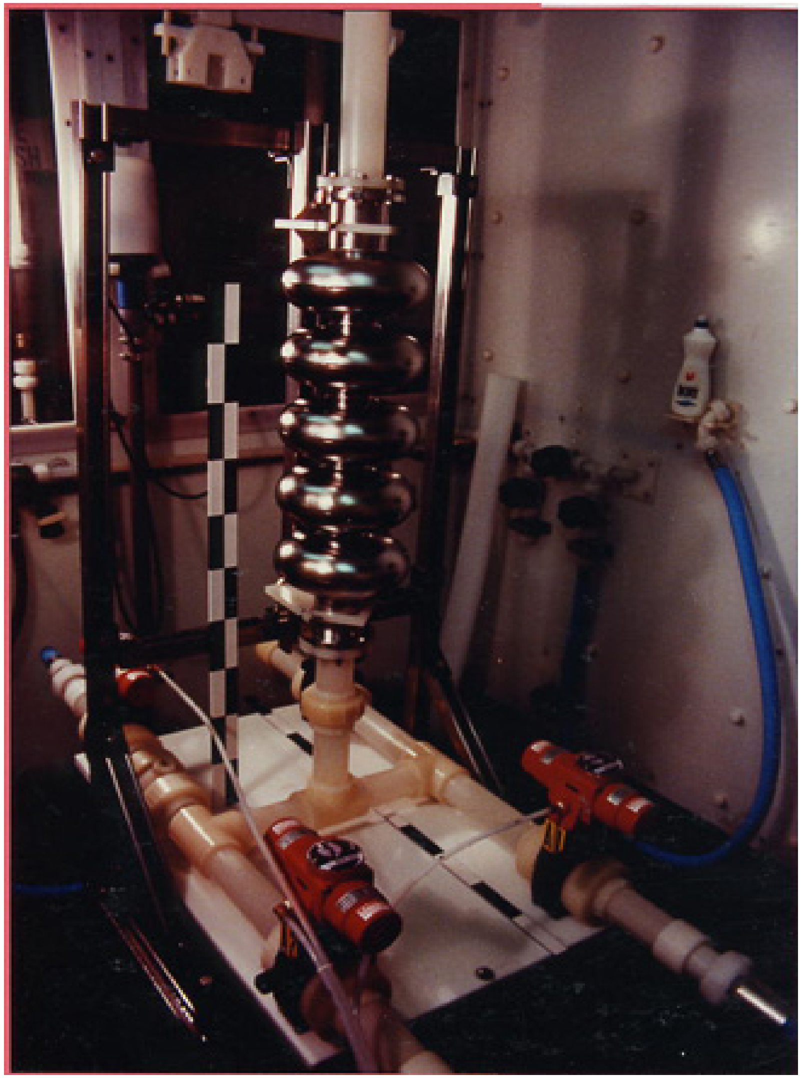


Niobium surface after EP

Chemical Etching, Final Treatment

- Buffered Chemical Polish BCP by mixture of HF, HNO_3, H_3PO_4 at 1:1:2
  - Cooled at 15°C to avoid H pick up
  - Closed system for cooling and cleanliness
- Fast rinsing with pure water
- High pressure rinsing with ultra-pure water
- Drying and clean assembly

Chemical Etching



Buffered Chemical Polish (BCP)

HF (49%), HNO_3 (65%), H_3PO_4 (85%)

Mixture 1:1:1 , or 1:1:2 by volume typical

Reaction:

Brown gas

Oxidation

$2Nb + 5HNO_3 \rightarrow Nb_20_5 + 5NO_2$

Reduction

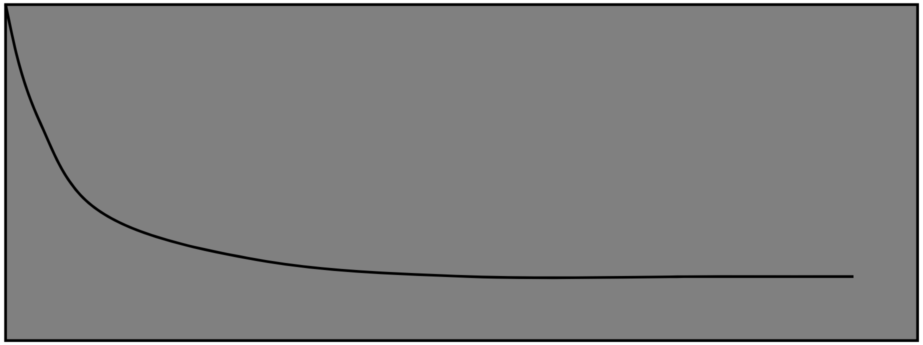
$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F\ 0.5H_2O + 1.5H_2O$

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

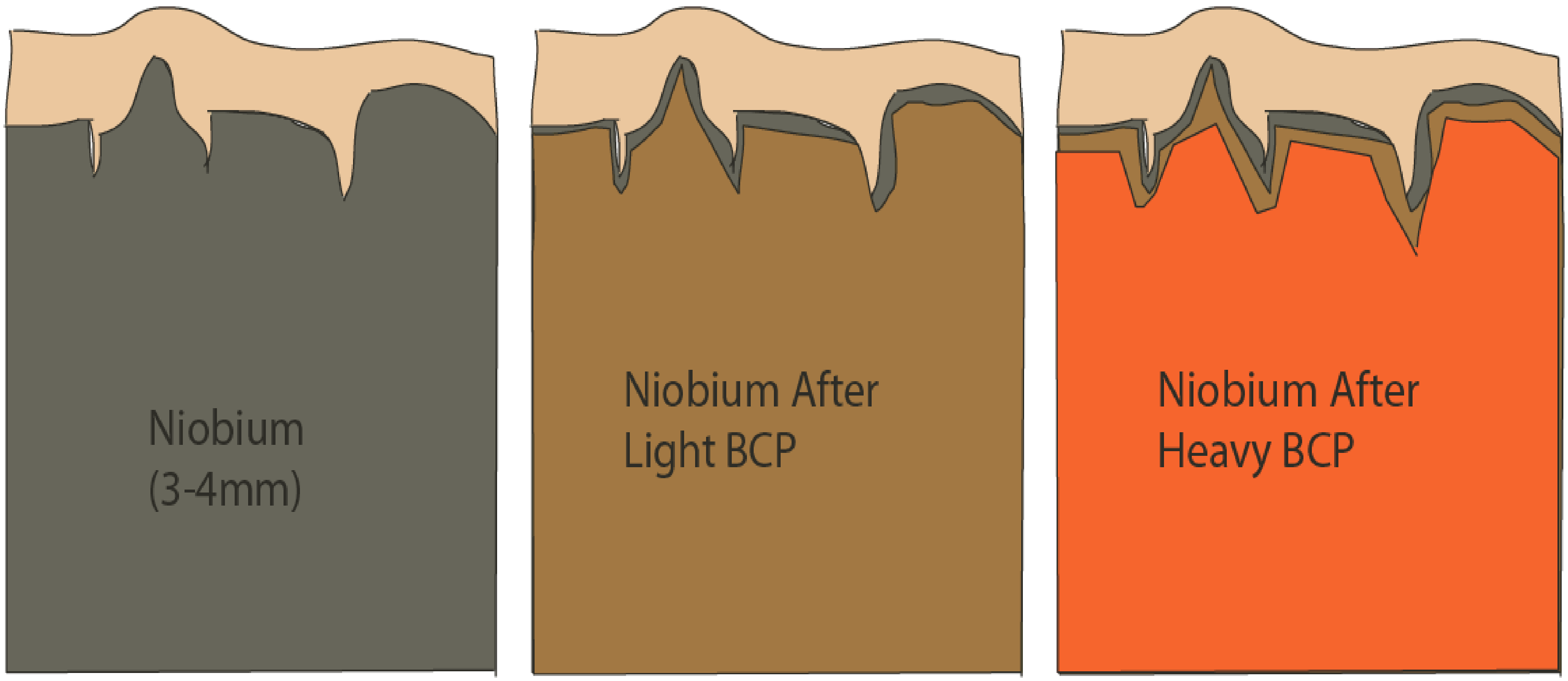
Use of BCP:

- 1:1:1 still used for etching of subcomponents (etch rates of 8um/min)
- 1:1:2 used for most cavity treatments
  - Mixing necessary → reaction products at surface
  - Acid is usually cooled to 10-15C (1-3um/min) to control the reaction rate and Nb surface temperatures (reduce hydrogen absorption)

Acid Wasted After 15g/L Nb



Effects of BCP on The Niobium Surface



(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

BCP Cabinet JLab

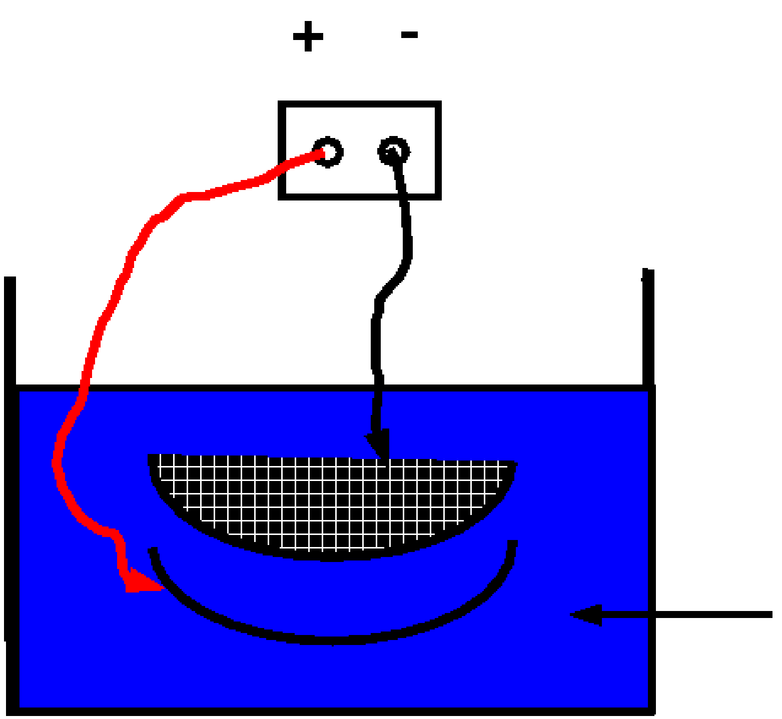
Implementation:

- 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

Electropolishing of Niobium

Electropolishing of half cells

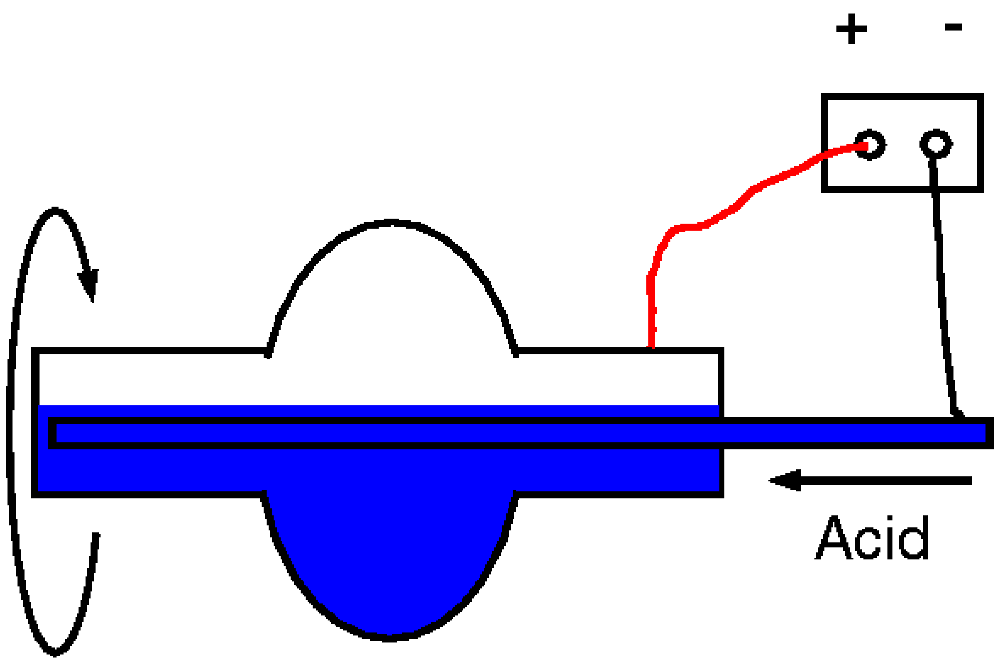
(Scheme)



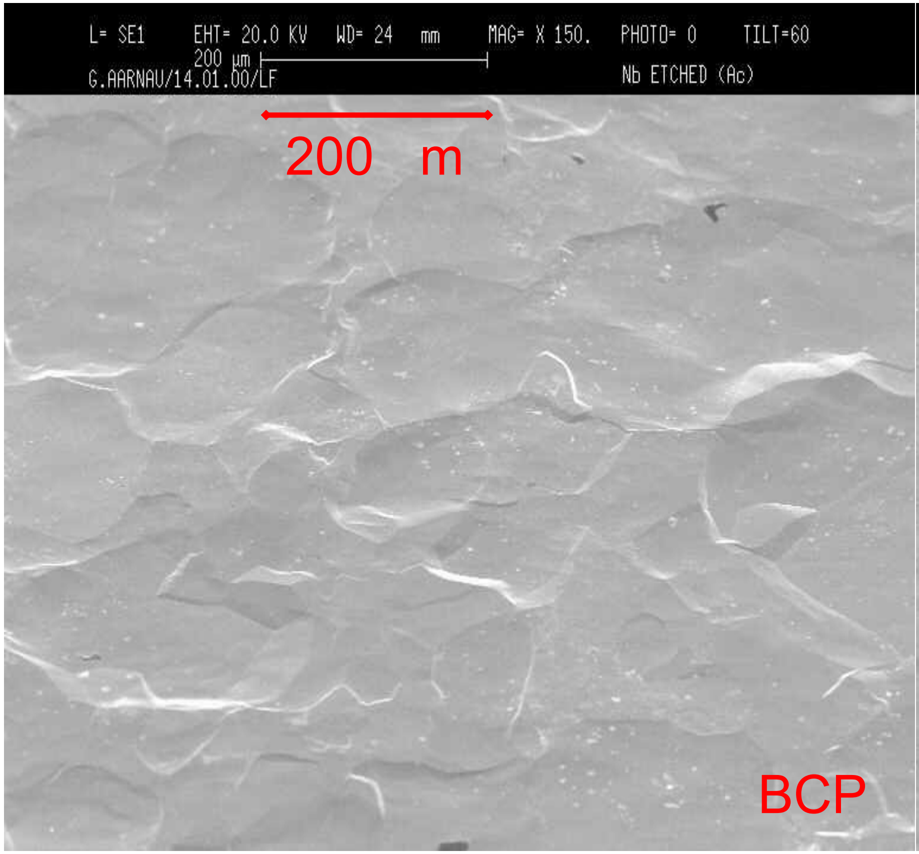
- EP Electrolyte (KEK / Siemens)
- 90 % H_2SO_4
- 10 % HF
- 30 °C
- 0,5 µm/min

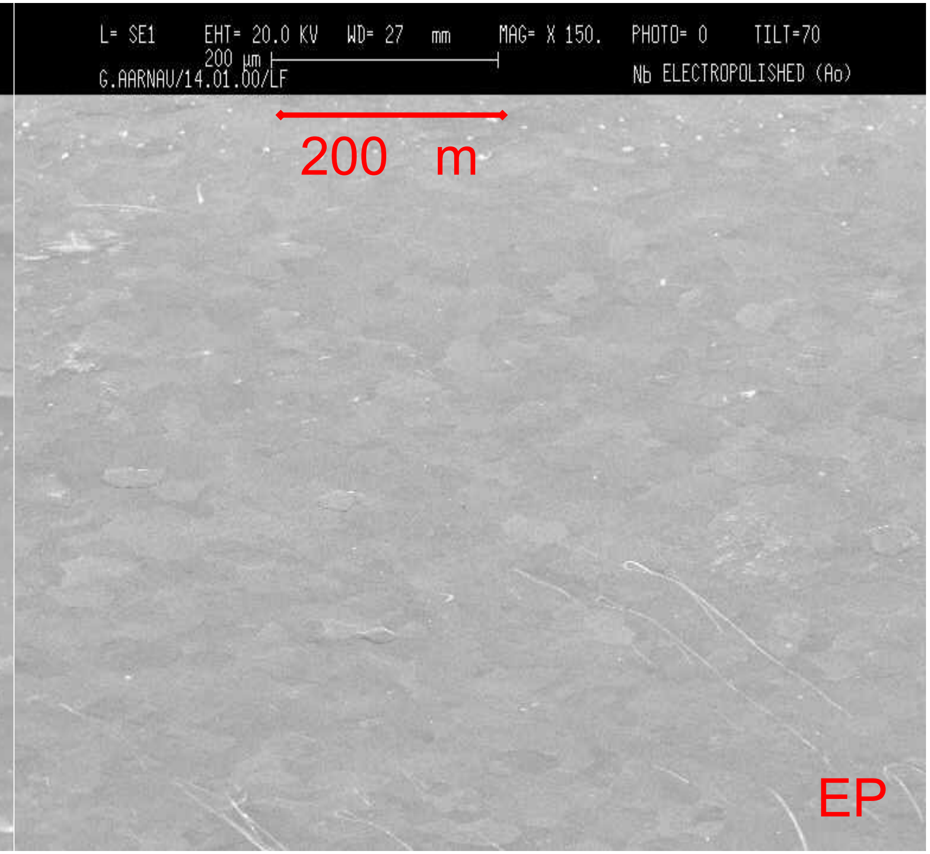
Electropolishing

Electropolishing of 1-cell cavities
(Scheme)



Surface Roughness of Niobium



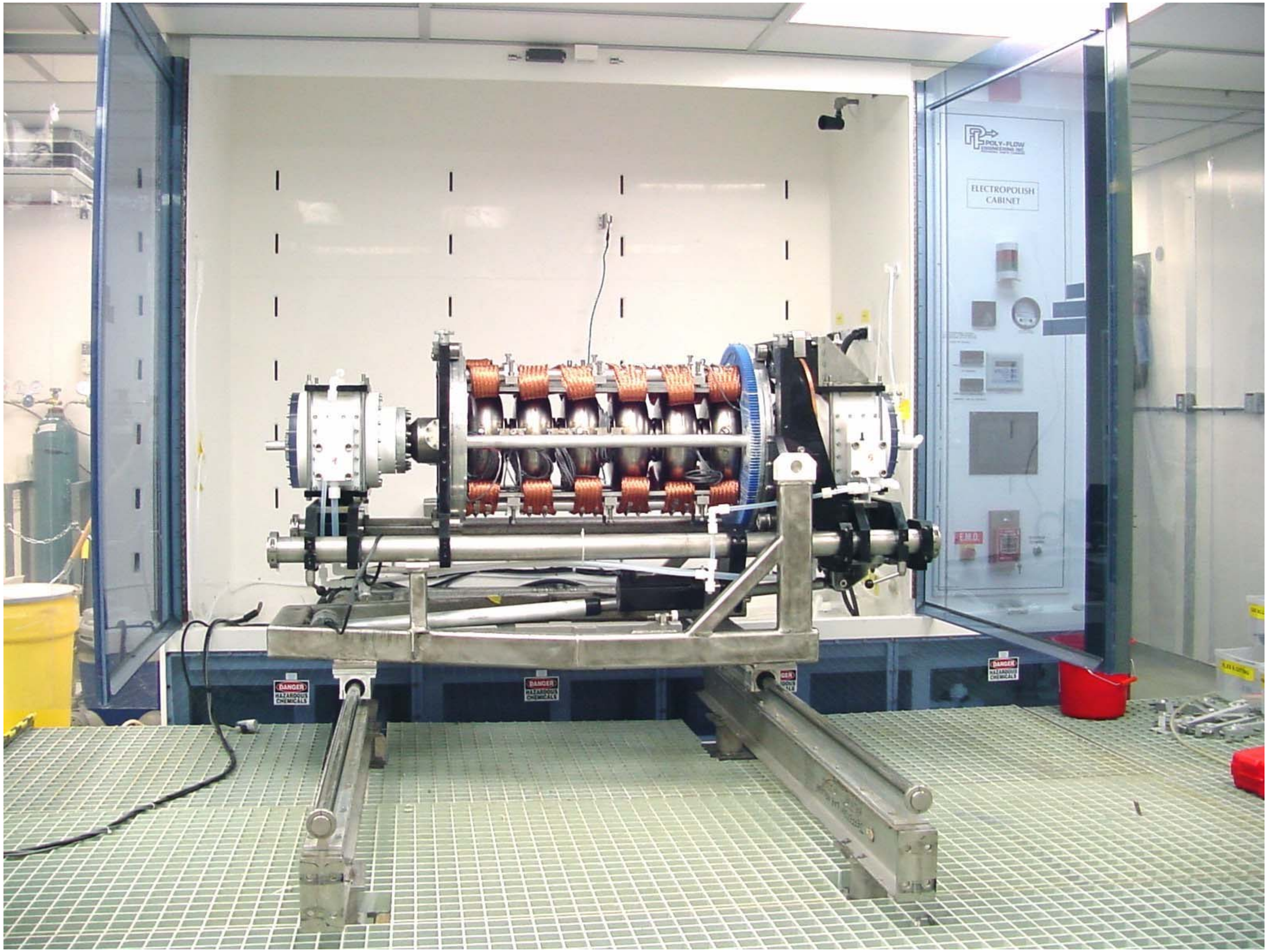


- Standard: chemical etching
  - HF, HNO_3, H_3PO_4
- Electro polishing

Electropolishing Systems

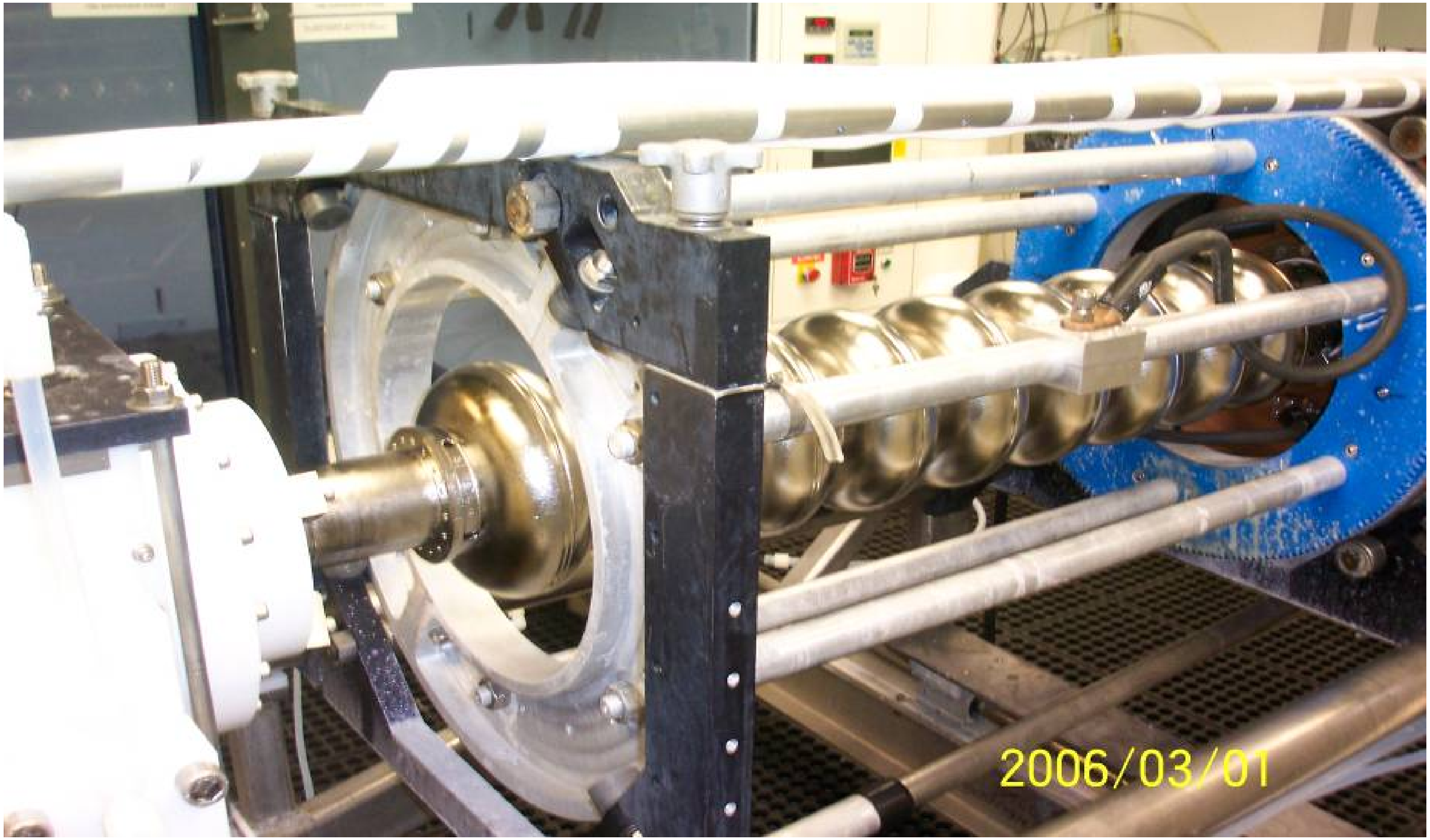
DESY

Electropolishing Systems



JLAB

Electropolishing Systems



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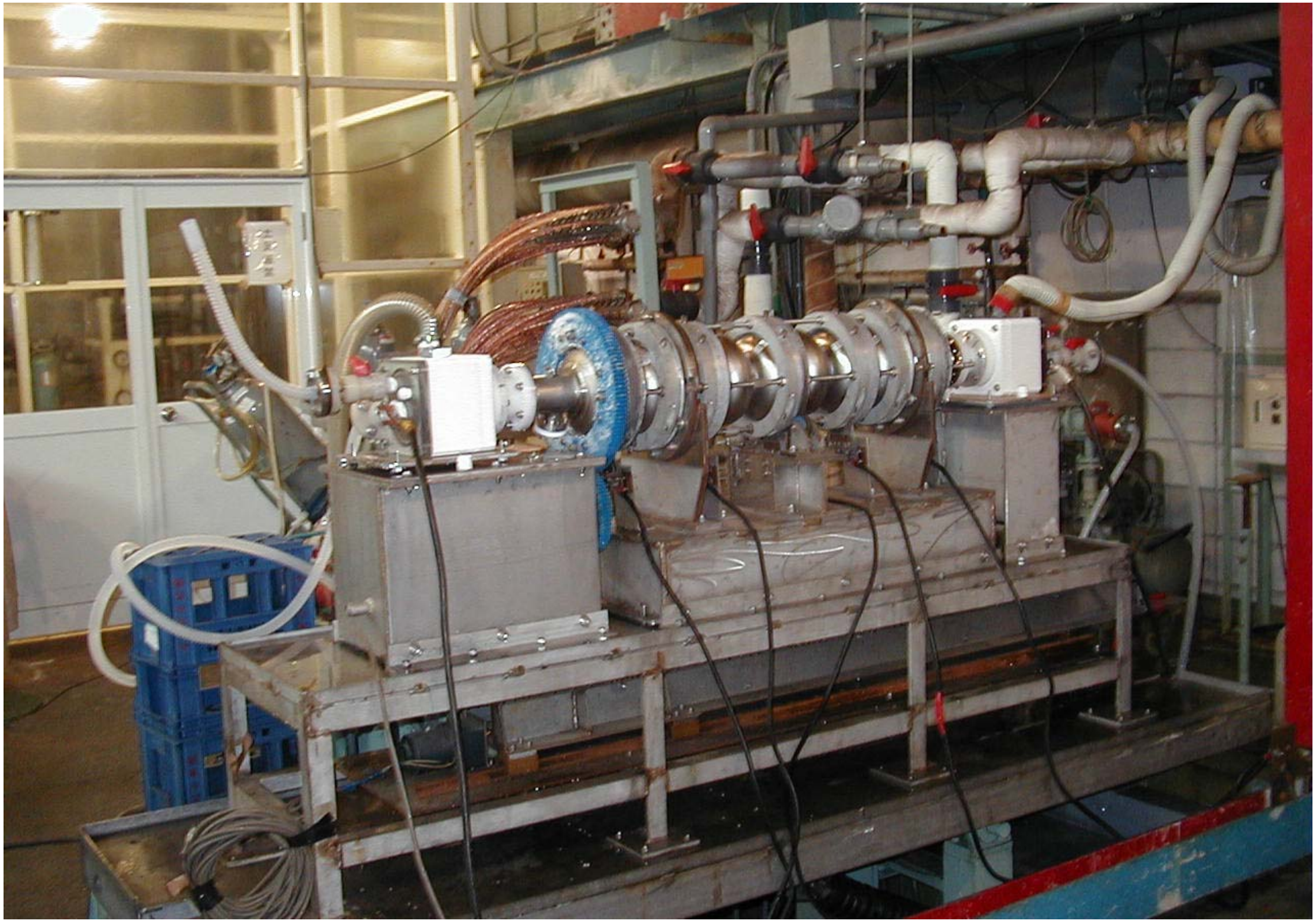
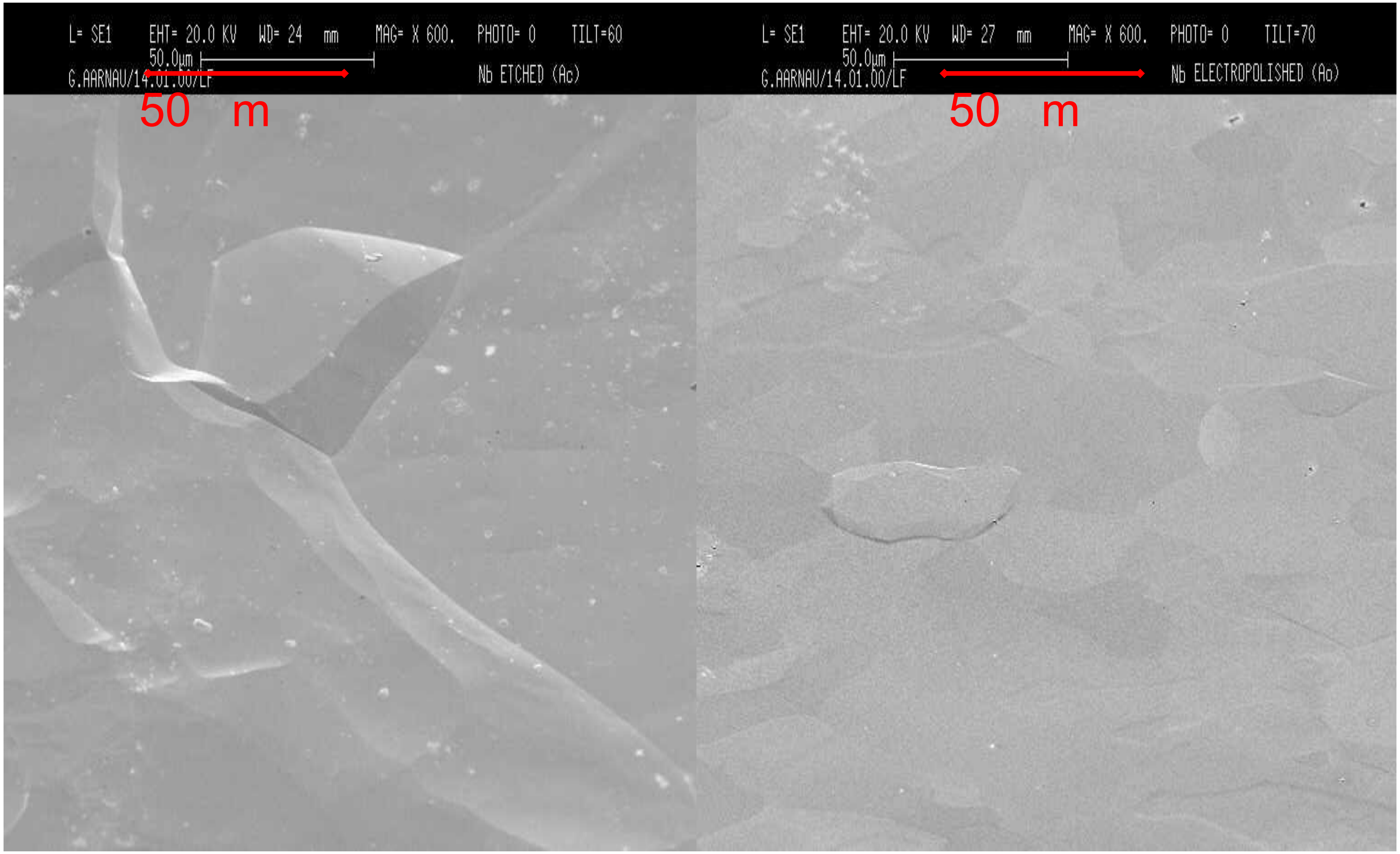
Electropolishing of 9-cell Resonators (Nomura Plating & KEK)



Surface Roughness of Niobium



BCP EP

Electropolish (EP)

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

Hydrogen Gas

Reaction:

Oxidation

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

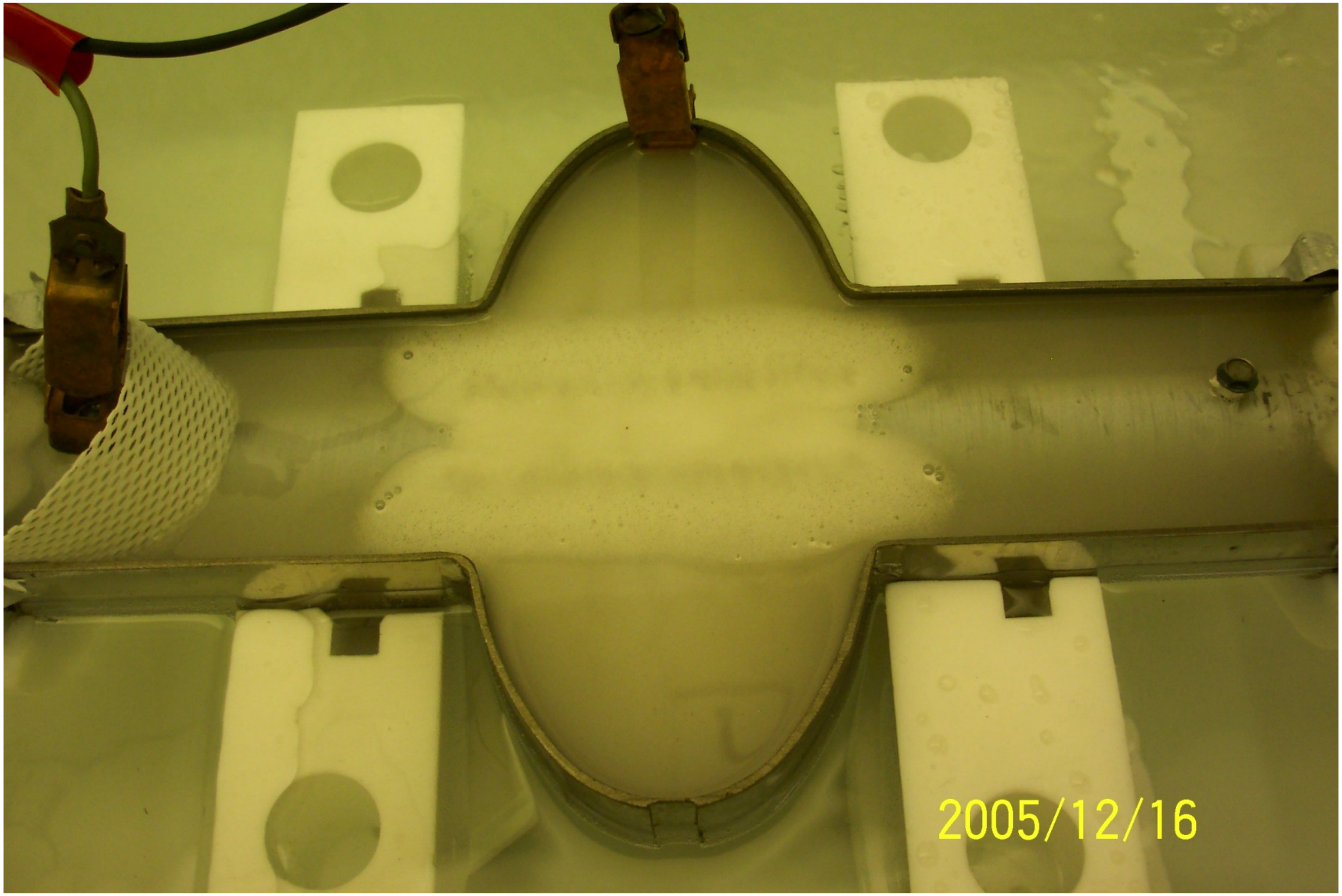
Reduction

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

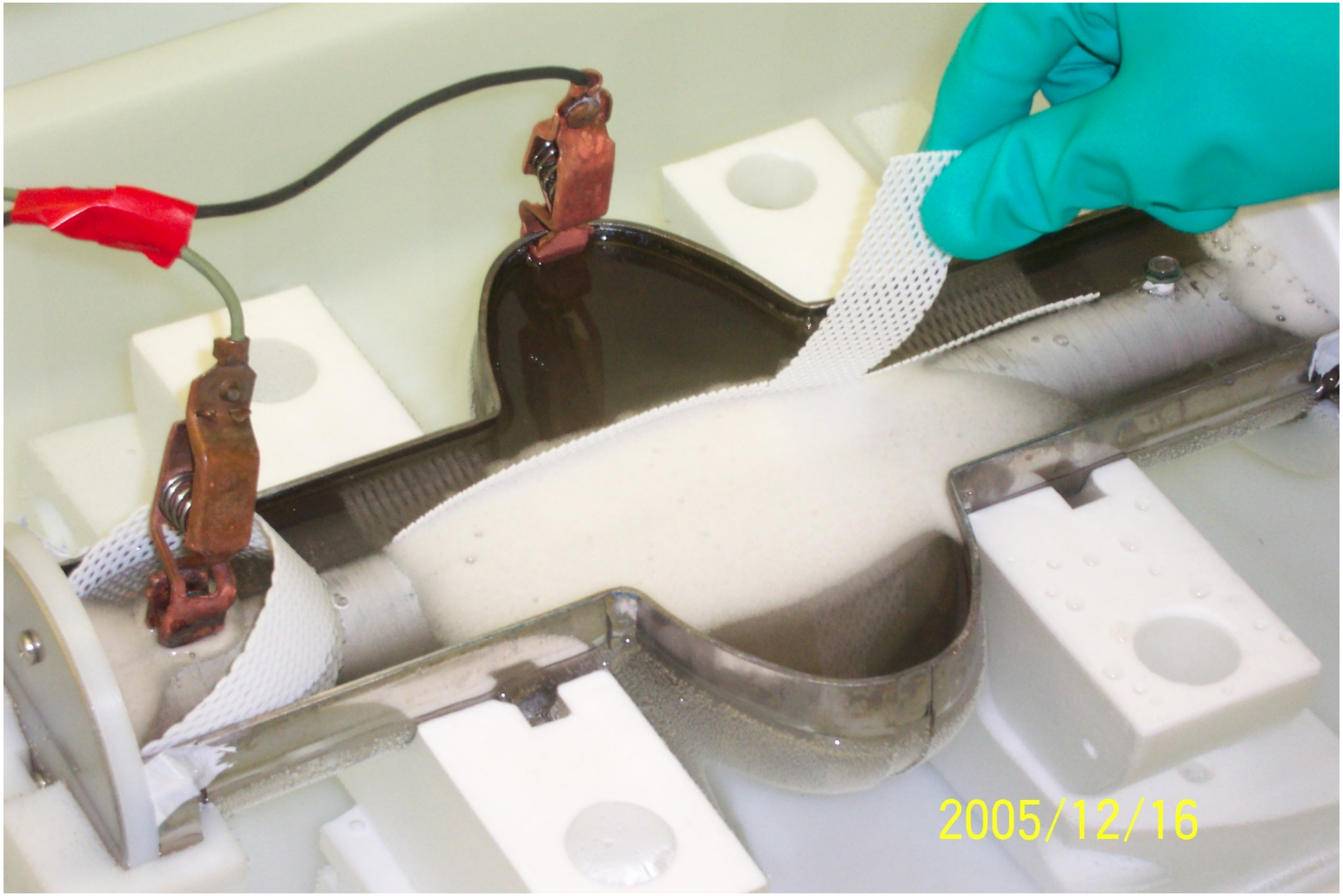
$$NbO_2F\ 0.5H_2O + 4HF \rightarrow H_2NbOF_5 + 1.5H_2O$$

These are not the only reactions that take place!

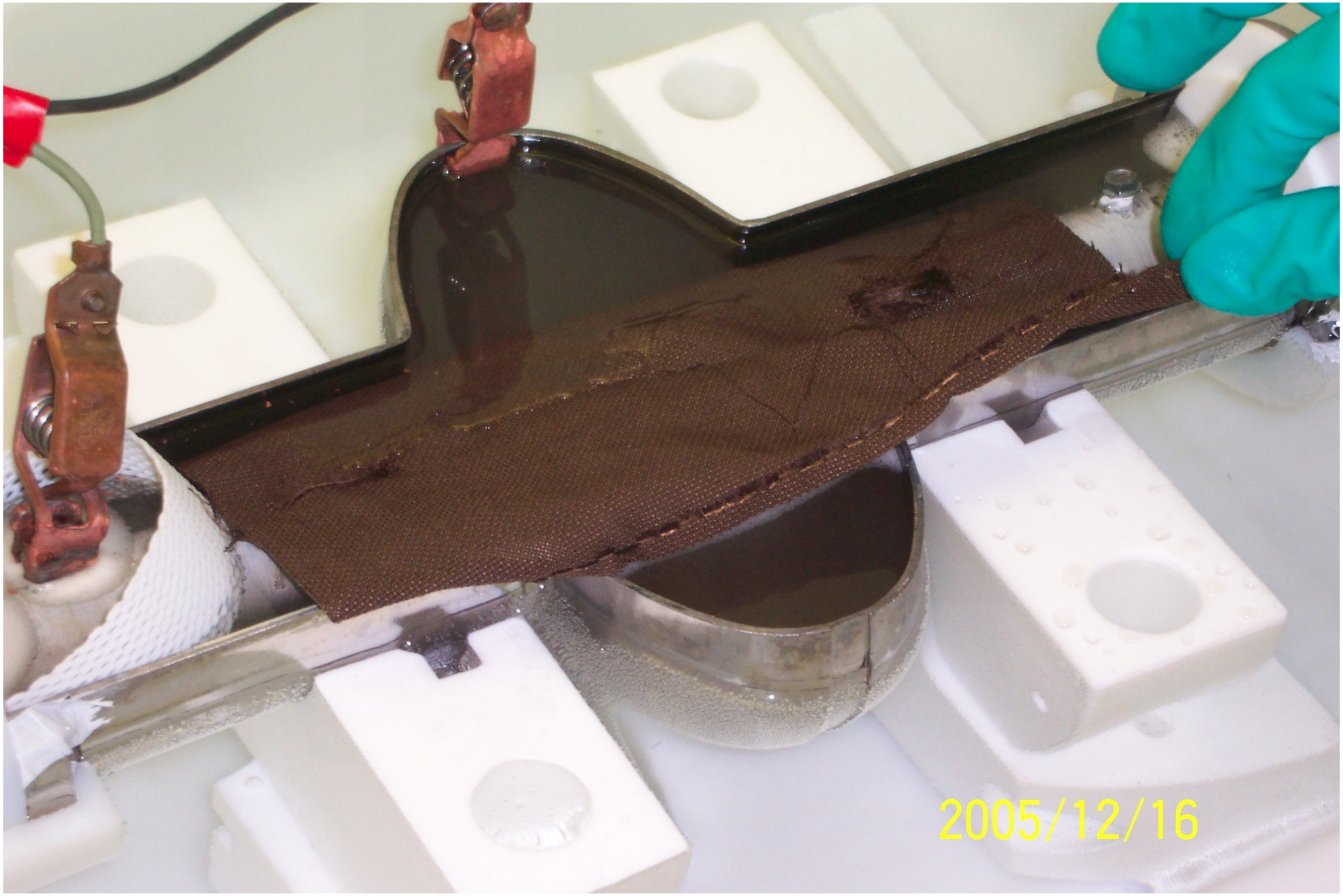
Hydrogen Gas Shielding Experiment



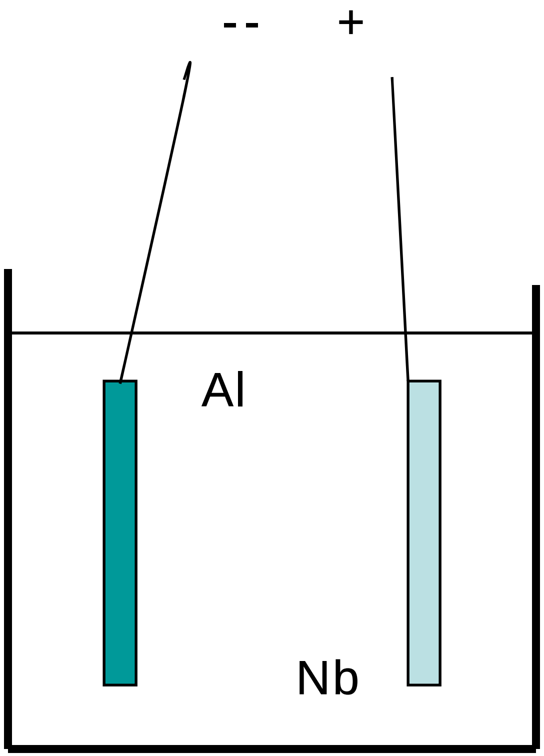
Perforated Teflon Sheet

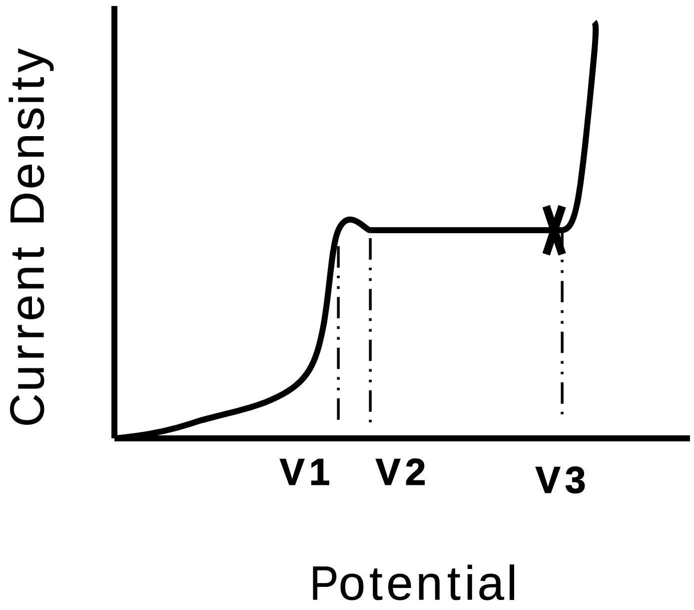


Fine Teflon Cloth (Numora Plating Co)



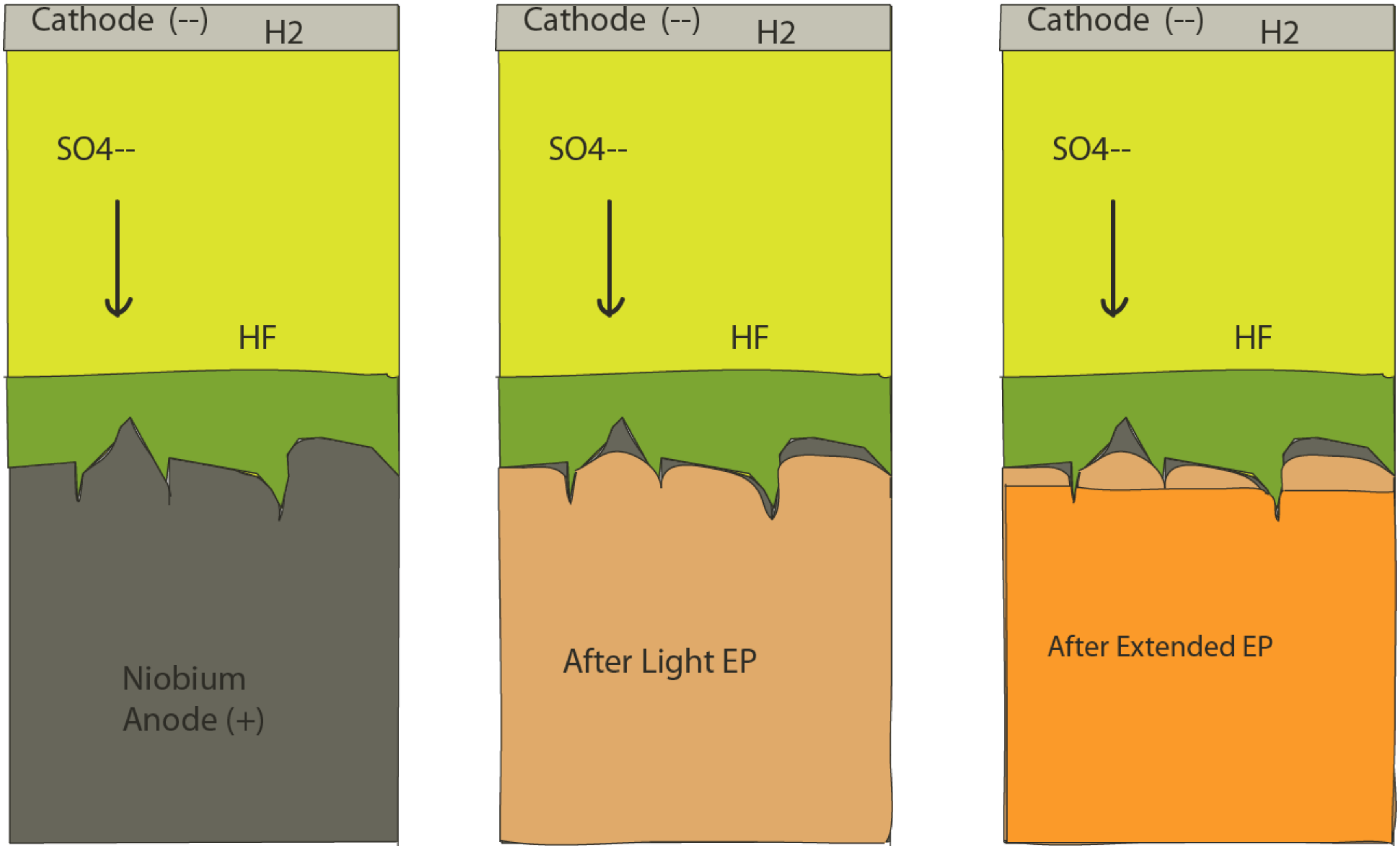
Basic Concepts of EP



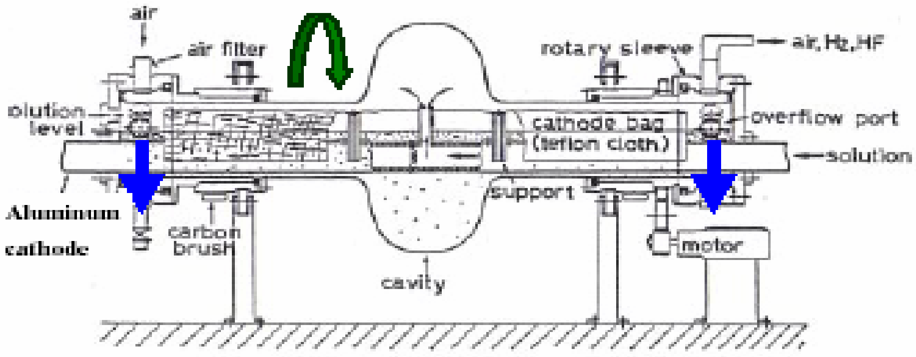


- **0-V2- Concentration Polarization occurs, active dilution of niobium**
- **V2-V3 – Limiting Current Density, viscous layer on niobium surface**
- **>V3 Additional Cathodic Processes Occur, oxygen gas generated**

Nb Surface Effects After EP



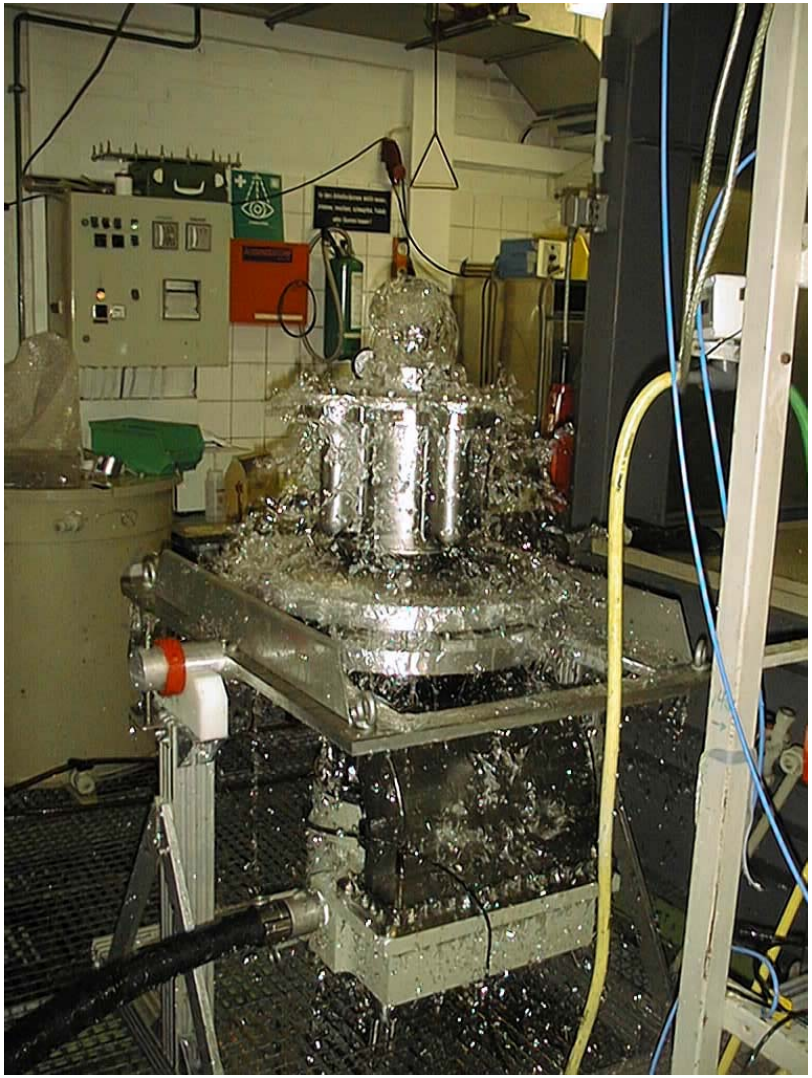
Horizontally Rotated Continuous EP (HRC-EP)



- 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 losses 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 of alcohol rinses
  - Full understanding of chemistry is still missing
  - Typically cavity processed horizontally, slowly rotated
  - Etch rate 2X on iris then equator (0.4um/min) –same as for BCP

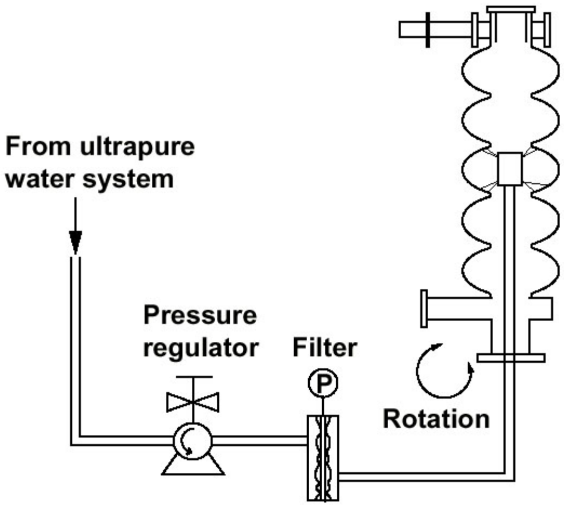
Continuous Flow Rinse

Ultrapure water (18 MΩ-cm)



High Pressure Rinsing (HPR)

- Rinsing of cavities with up to 1000 psi water jets removes many particles.



ACCEL Instruments

High Pressure Rinsing:

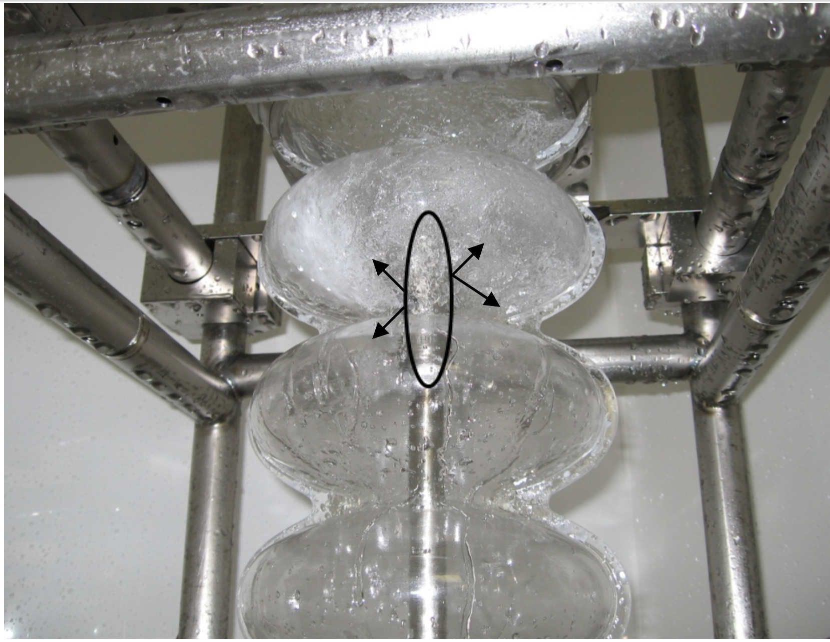
- The need for HPR surface cleaning:
  - Entire surface contaminated after chemistry, early field emission will result if not performed
  - Effective at removing particulates on the surface after assembly steps

ISSUES:

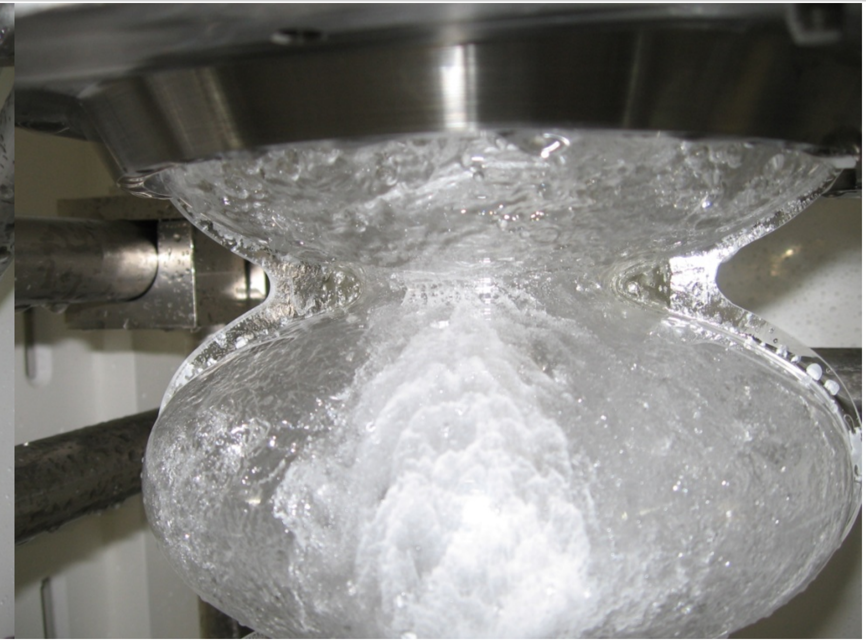
- HPR systems are still not optimized for the best surface cleaning performance
- Surface left in a vulnerable state, wet

JLab HPR Cabinet in Clean Room

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

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, auxiliary parts are attached ,high pressure rinsed again, dried and mounted onto the assembly bench.

- The most critical part of the assembly is the interconnection between two cavities, monitored by particle counting

String Assembly

Assembly: Vacuum Hardware

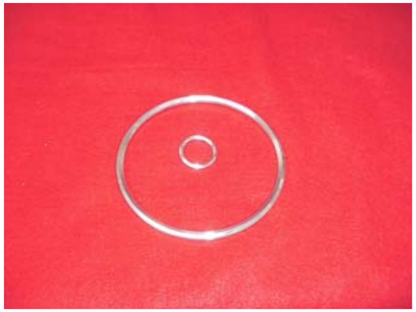
- The cavity strings have to be vacuum tight to a leak rate of < 1 $x10^{-10}$ torr l/sec
- The sealing gaskets and hardware have to be reliable and particulate-free
- The clamping hardware should minimize the space needed for connecting the beamlines

Assembly: Vacuum Hardware

- Present choice for TESLA cavities:

  diamond-shaped $AlMg_3$ –gaskets + NbTi flanges + bolts

- AlMg-Gasket



- Alternative:

  radial wedge clamp, successfully used for CEBAF upgrade cavities

- Radial Wedge Clamp

Cavity String Assembly in Clean Room

DESY

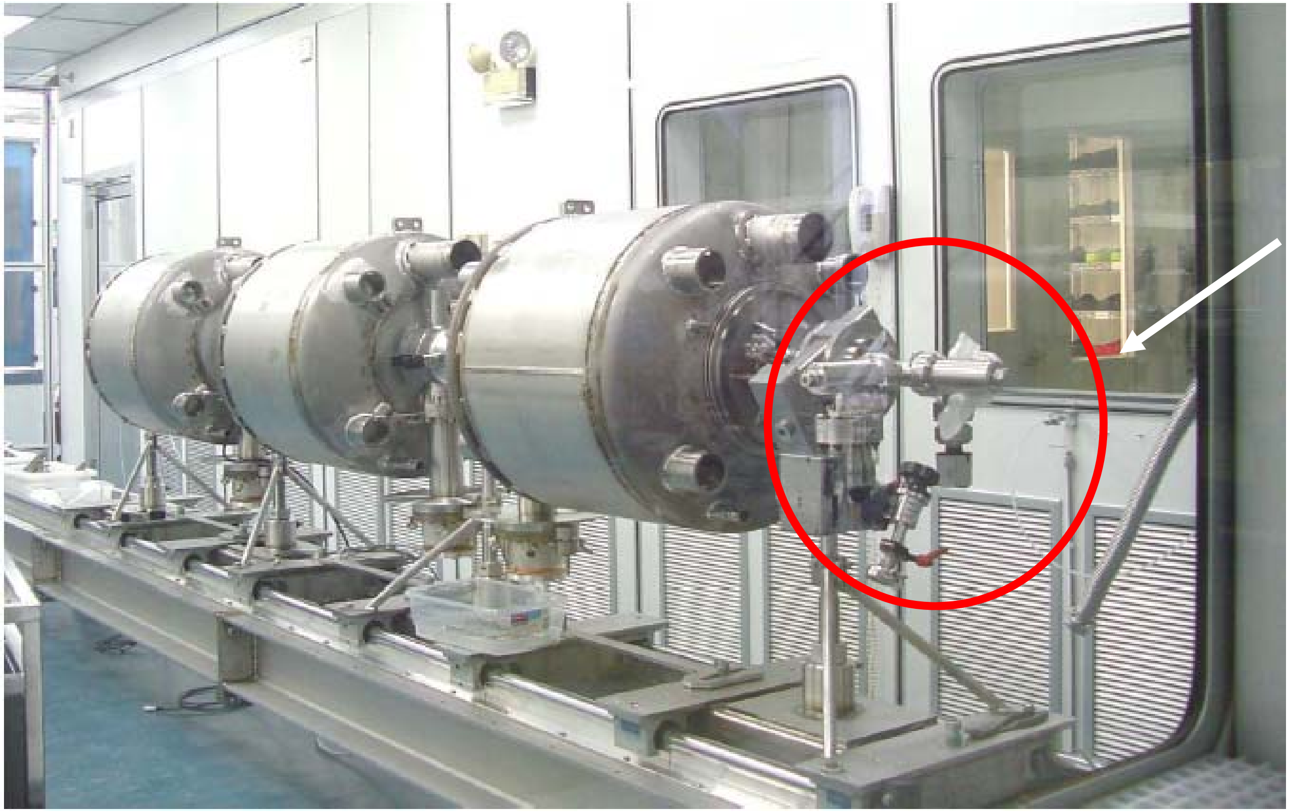
Cavity String Assembly in Clean Room

LEP

JLab Clean Room

Cavity String Assembly

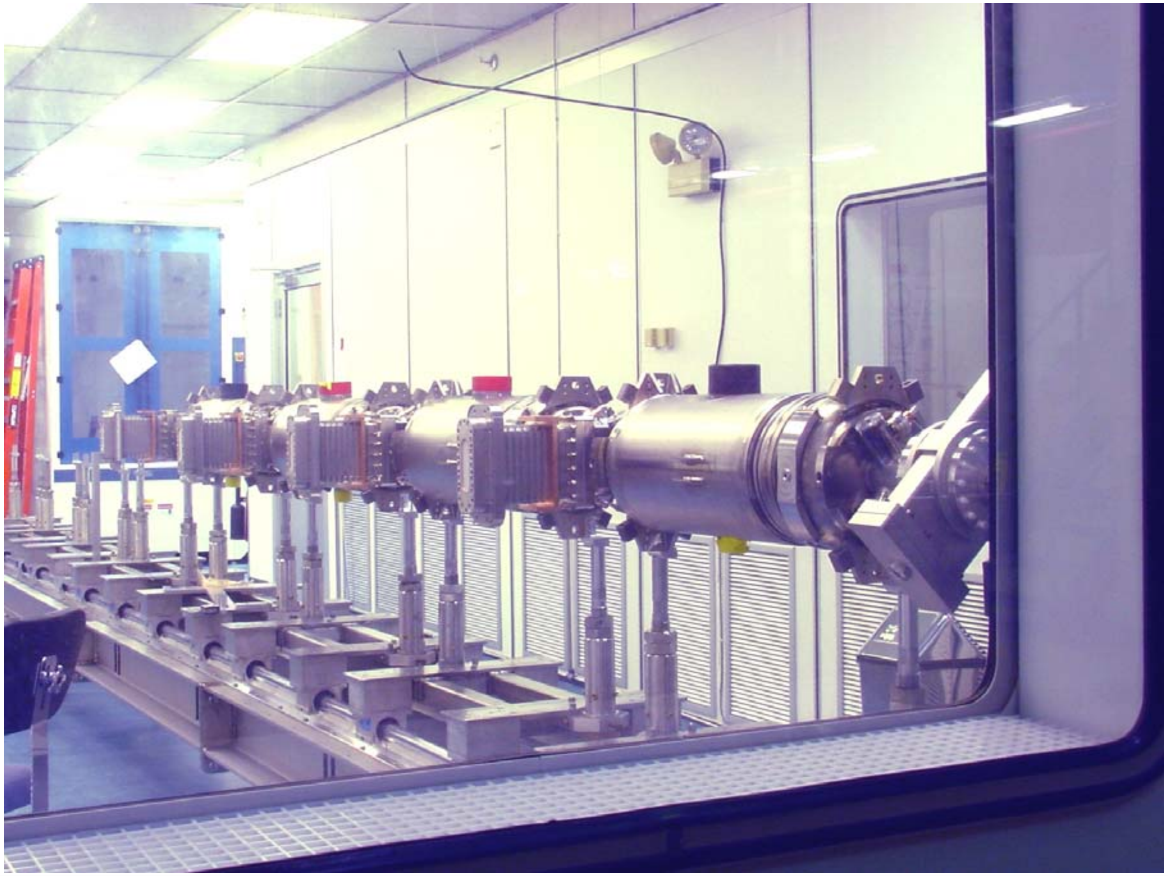
SNS Medium Beta Cavity String: three 6-cell 805 MHz cavities



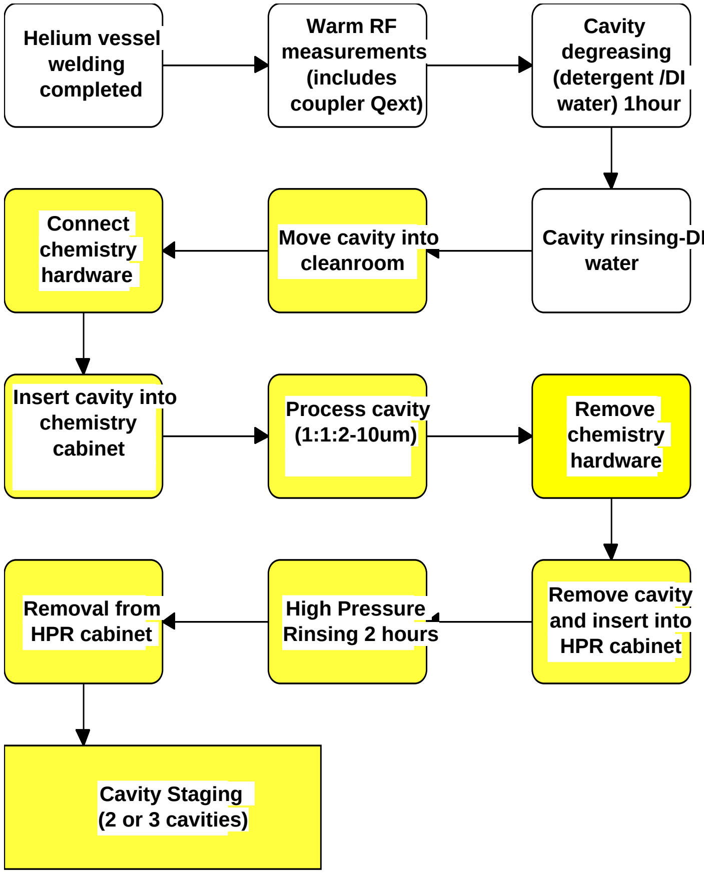
Pump-out assembly

String Assembly

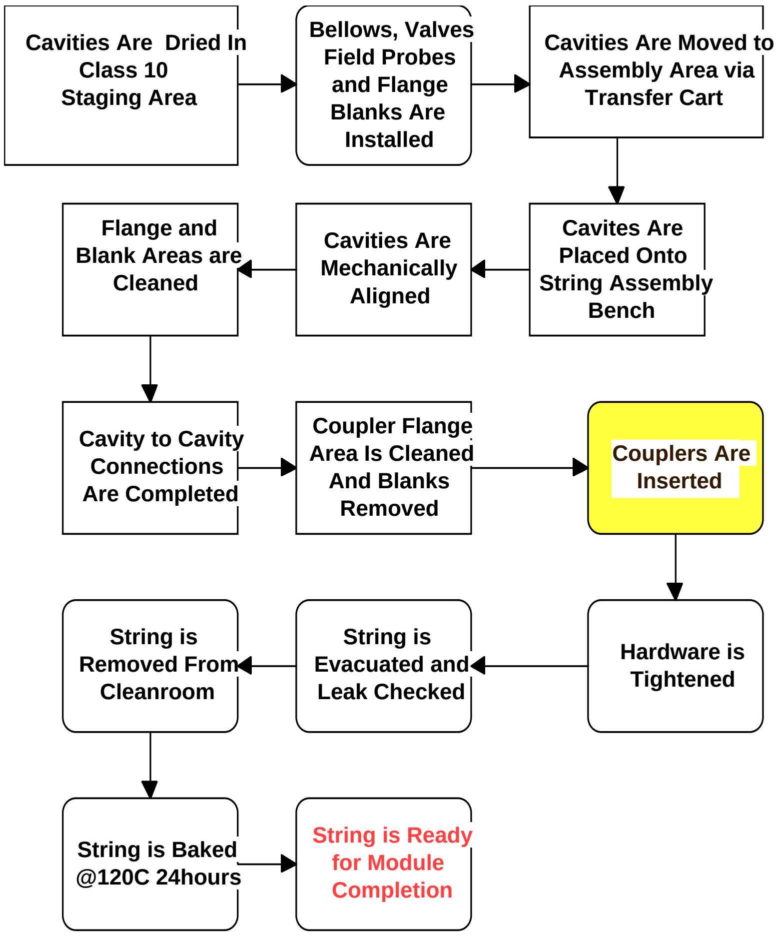
Jlab Upgrade String of eight 7-cell cavities in class 100
Clean room on assembly bench



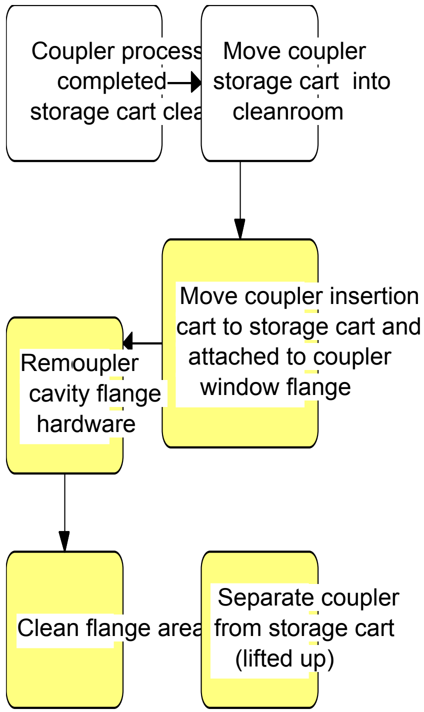
String Processing Sequence for SNS

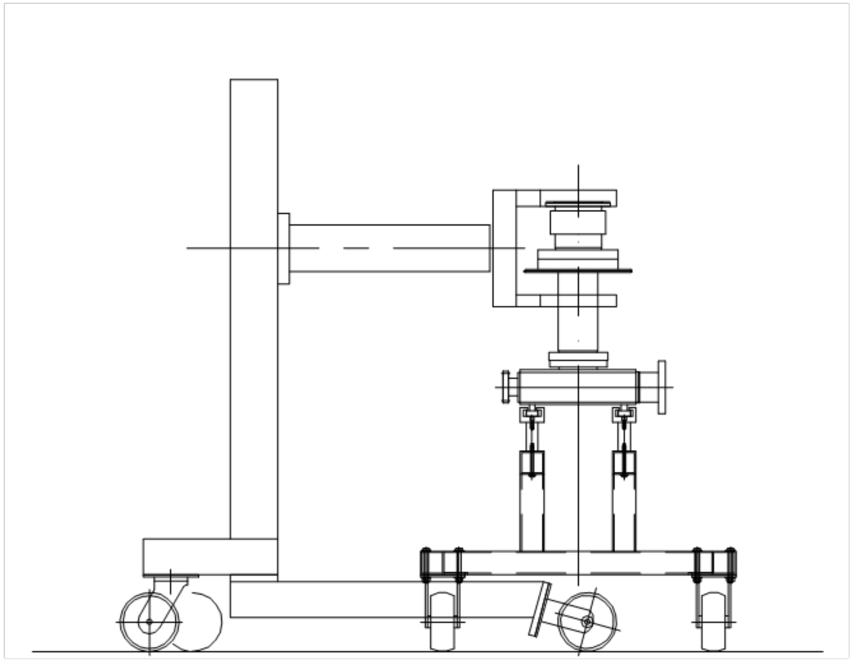


Cavity Assembly Sequence

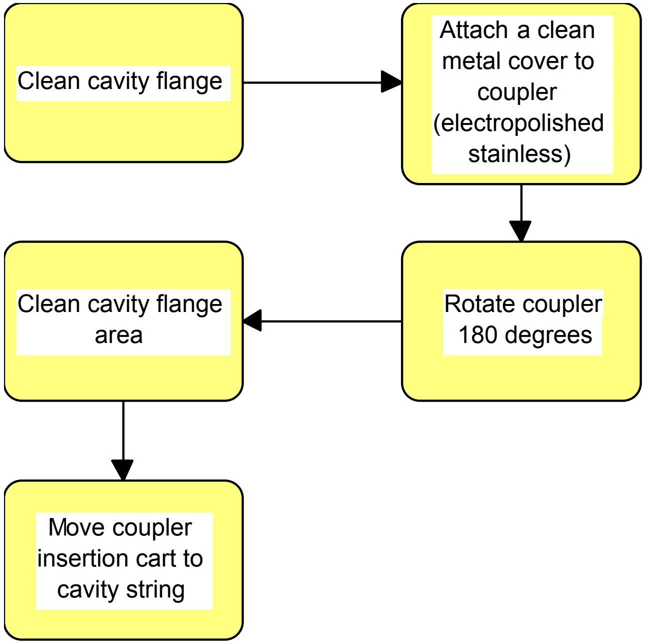


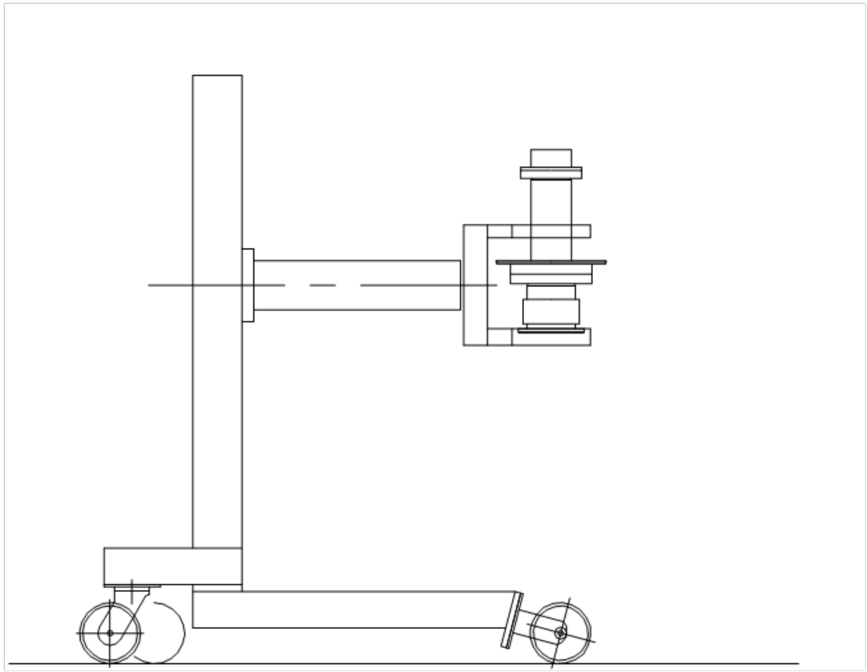
Coupler Insertion Procedure- Removal From Coupler Cleanroom Storage Cart



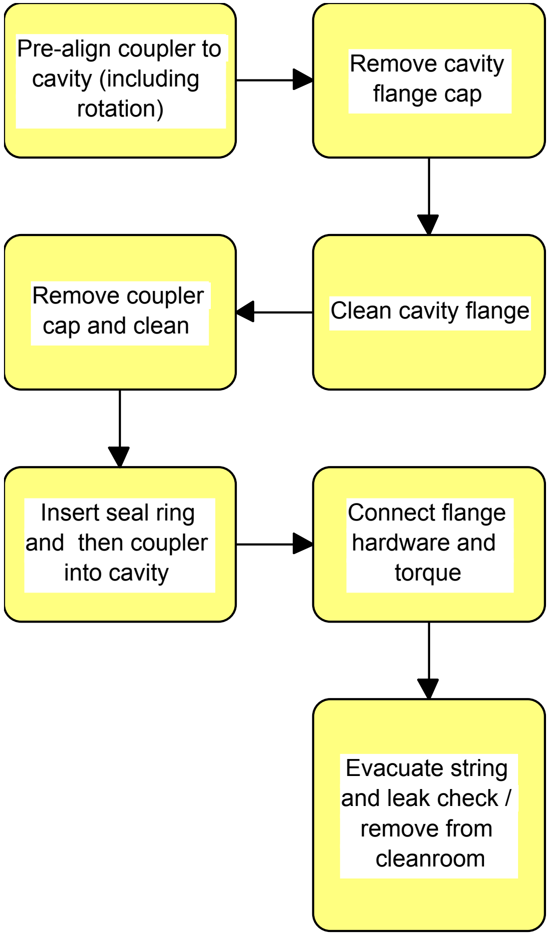


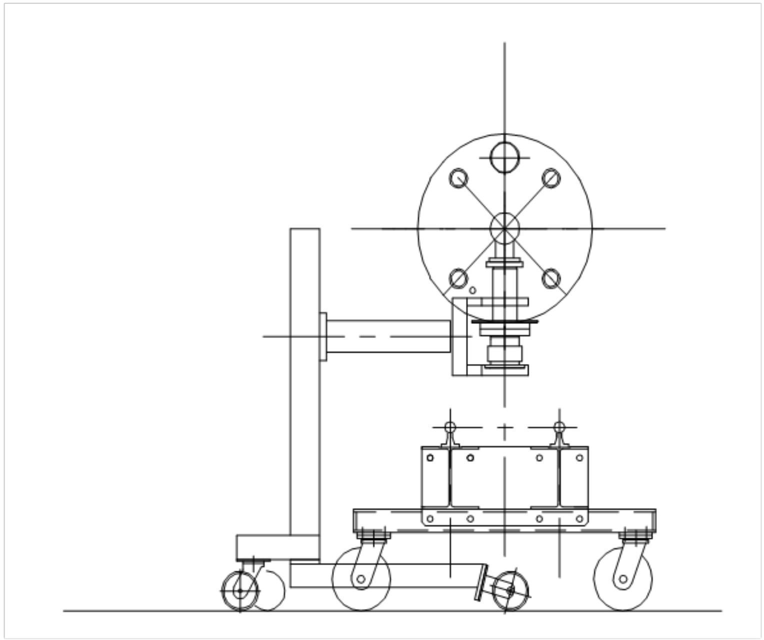
Coupler Insertion Procedure- Transporting to string tooling



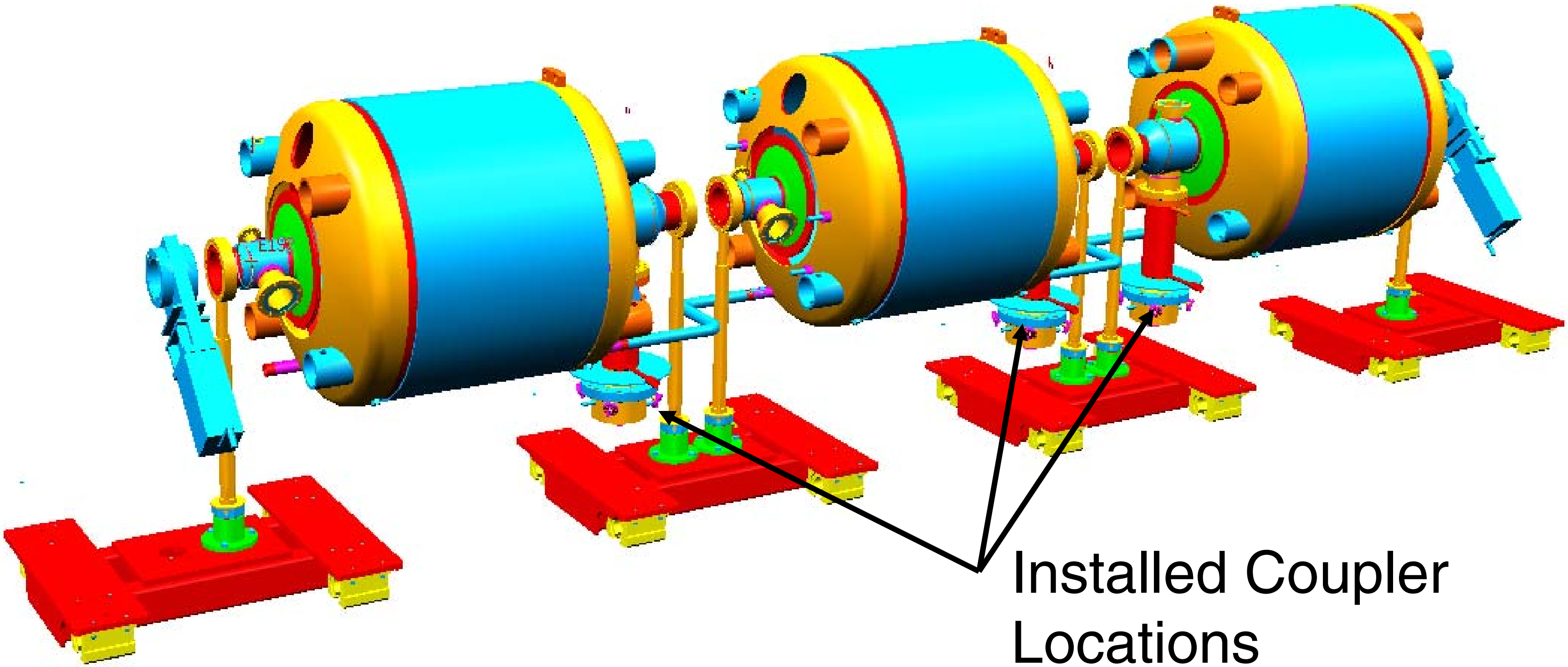


Coupler Insertion Procedure- Insertion into cavity





String Assembly



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, more process variables and not fully understood

Comments on Process Monitoring

- Currently
  - Process variables being monitored are poor indicators of cavity performance success or failure!
  - Cavity performance studies are narrowly focused and not conclusive
  - Knowledge of the performance impact generated with each process step is unknown

These must be addressed to reduce performance spread

Conclusions

- To be successful with current process steps:
- Must Set A Culture For High Quality
  - Fully document best practices and procedures
  - Provide routine training for the procedures
  - Expect quality during procedure implementation and monitor implementation progress
  - Record meaningful process data and continuously review

Conclusions

- We Should Push For Process Improvement:
  - Optimize HPR effectiveness for a given RF structure
  - Implement witness sample monitoring to develop an understanding of areas that need improvement
  - EP process – we need to develop a better understanding of the chemistry and improve process control and monitoring
  - Assembly process – we need to develop better online monitoring to reinforce personnel actions (particle counters) and improve hardware cleaning steps to reduce handling (automate)
  - We should not accept any new process or procedure without scientific evaluation
  - Reevaluate cavity flange designs → make cavity more assembly friendly