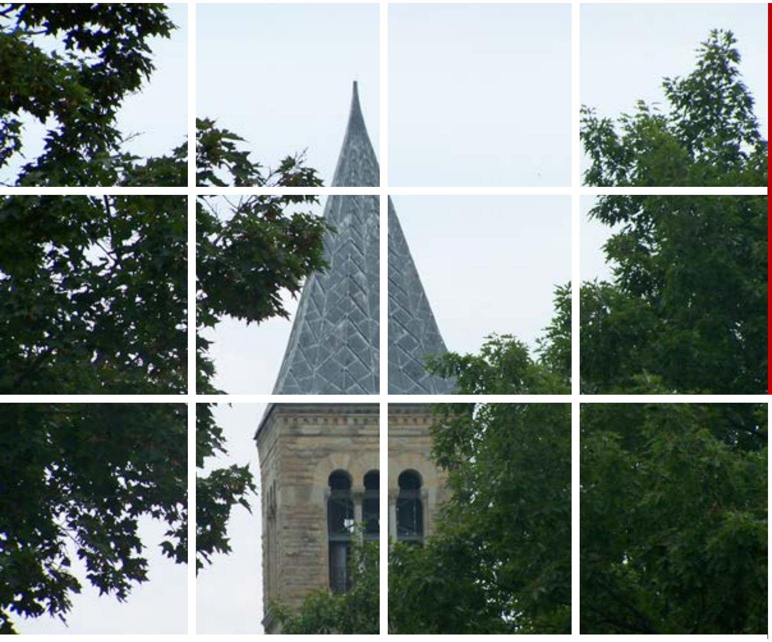

Vacuum Science and Technology for Accelerator Vacuum Systems

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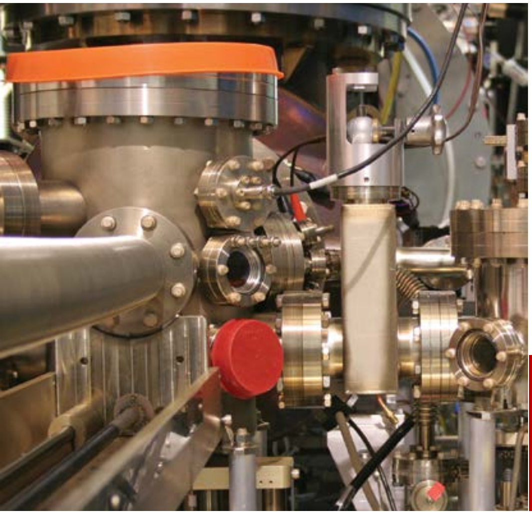
UCDAVIS

Cornell Laboratory
for Accelerator-based Sciences
and Education (CLASSE)



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SESSION 5.4: FABRICATION AND CLEANING

- *Cleanness is the foundation in achieving UHV and required vacuum system performance.*
- *Cleanness starts with vacuum component material selection, and fabrication.*
- *Proper fabrication processes leads to easier and less aggressive post-machining cleaning.*
- *Cleanness requires a development of UHV practice and culture, besides the adequate facilities.*
- *UHV clean and Particle-free are very different requirements, though most modern accelerator vacuum systems demanding both.*

Material Preparation Techniques

- **Vacuum materials may be prepared for finish machining by the following techniques:**
  1. *Rough Machining*
  2. *Metal Stamping*
  3. *Water-jet cutting*
  4. *Laser cutting*
  5. *Plasma arc cutting*
  6. *Bead/sand blasting*

- **When plasma arc cutting, make sure that sufficient material allowance is made for complete removal of the heat affected zone (HAZ) during final machining.**

- **Bead/sand blasting should only be permitted on material with large amounts of mill scale or heavy inclusions from contact with metallic or organic material.**

Material Finishing Techniques

- The preferred technique for finishing vacuum materials is machining (turning and milling).

- The following techniques should be avoided or at least approved on a case-by-case basis:
    1. *Grinding*
    2. *Honing*
    3. *Electric Discharge Machining (EDM)*
    4. *Chemical milling*
    5. *Glass bead blasting*

- EDM (specially wire-EDM) is acceptable when DI-water is used as dielectric fluid. Otherwise more aggressive etching may be necessary.

- Glass bead blasting may be permitted with new clean beads when an optically dispersive surface is required. (Alumina beads are better choice.)

Material Finishing Techniques (cont.)

- *When machining will not produce the required surface finish, polishing may be permitted. When polishing, care should be taken to avoid excessive rubbing or contact pressure.*
- *The following abrasives are acceptable for UHV components.*
  - *3M Scotch - Type S, Silicon Carbide (color: gray), 500 grit*
  - *Brite - Type A, Aluminum Oxide (color: maroon), 240 grit*
  - *3M Wet or Dry Fabricut Cloth - Aluminum oxide or silicon carbide, 600 grit*
  - *Mineral oil suspended diamond pastes*
- ***Modern extrusions with proper dies can achieve almost "mirror-like" inner surface finishes.***
- *Very smooth inner surface finish via a process called "Abrasive Flow machining, or AFM". This process was employed for the LCLS's long undulator beam pipes.*

Cutting Fluids for Finish Machining

If feasible, only alkali-detergent like cutting fluids should be used for the finish machining. This type of oil-free fluids are fully water soluble, and are readily removed by solvents. Some example fluids are:

Relton A-9	*Rapid Tap*
Tap Magic	*Trim Tap*
Tapmatic #1 or #2	*RD2-195*
"Pearl" Kerosene by Chevron Chem CO	*Dip Kool 868*
"Tool Saver" by Do All Corp.	*DIP Kool 862*
Cutzol EDM 220-30	*Dip Kut 819H*
Sunnen Man-852 Honing Oil	*No Sul #6871*
Vytron Concentrate	*Kool Mist #88*
Rust-Lick G-25-J	*Cimcool 5 Star 40*
Wheelmate #203	*Cimperial # 1011*
Aqua Syn 55 by G-C Lubricants CO	*Haloform CW-40*
Cold Stream Coolant by Johnson Wax CO	*Trim Sol*
"Acculube" by Lubricating Systems Inc.	*Trim9106CS*
Micro Drop "Advanced System Lubricant" by Trico	*CINDOL 3102*
Micro Drop "New Vegetable Based" by Trico	*PenWalt #DP 1131*

Outgassing (with RGA) tests of machined specimen (after cleaning) may be necessary to establish acceptability of a machining fluid.

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Dry-Machining or Solvent as Cutting Fluid

- *In reality, machining on a finished chamber is needed. In those cases, dry-machining is required.*
- *In some facilities, such as during TPS's (Taiwan Light Source) aluminum chamber fabrication, machining in a Clean Room using ethanol as cutting fluid has been implemented.*

Oil-free CNC Machining in Clean Room

Rough Machining → (Wait 3 days) → Intermediate Machining → (Wait 1 day) → Precision Machining

Rough Machining	Intermediate Machining	Precision Machining
Tool enter increment: 0.3 ~ 0.5mm	Tool enter increment: 0.1 ~ 0.2mm	Tool enter increment: < 0.05mm

CNC Machining Processes (with 99.5% Ethanol as lubricant)

Suggested UHV Handling & Assembly Guidelines

- No food, drink, or smoking allowed in CLEAN AREA.
- Limit entry and exit into CLEAN AREA.
- Hydrocarbons (oils, grease) and dust-collecting materials (cardboard) must be minimized.
- Equipment brought into CLEAN AREA must be clean. Carts, chambers, stands, and tools must be free of oils and dust.
- Wood must be minimized. If wood is required, it should be wrapped in "ULO" plastic sheet and/or cleanroom tape.
- A special set of tools expressly for use on vacuum components should be kept in the CLEAN AREA.

UHV Handling & Assembly Guidelines (Cont.)

- *Metal tools must be degreased. After degreasing, tools should be kept in clean trays and handled with clean gloves.*
- *No cadmium plated, lead, or painted tools should be permitted. Chrome and nickel plated tools are permitted.*
- *Aluminum foil shall be in accordance with ASTM B479, type designated as DRY ANNEAL A, (oil free). Each piece of foil should be used only once and then discarded.*
- *Aluminum foil and lint-free tissue should be stored in clean boxes with lids.*
- *Only use pens for writing in CLEAN AREA, do not use pencils. Minimize the use of paper. If paper is used, it should be "cleanroom type."*

UHV Handling & Assembly Guidelines (Cont.)

- *Clean vacuum parts and open chambers should be covered with foil at all times when work is not being performed.*
- *Do not wear wooly sweaters in CLEAN AREA.*
- *No sandpaper or abrasives allowed.*
- *Hands should be kept out of pockets (this introduces lint).*
- *Clean parts should be handled with new polyethylene gloves used inside 100% stretch nylon gloves.*
- *Gloved hands which touch cleaned parts and tools should touch nothing else (this includes your face, hair, etc.). Gloves which touch unclean surfaces should be replaced immediately.*

UHV Handling & Assembly Guidelines (Cont.)

- **Replace gloves with a new, clean pair at the beginning of each shift and following breaks.**

- **Hands should be washed before wearing clean gloves.**

- **Clean-room quality protective clothing (lab coats, hats, hair nets, face masks) should be worn when working on vacuum components in CLEAN AREA.**

UHV Handling & Assembly Guidelines (Cont.)

- **Replace gloves with a new, clean pair at the beginning of each shift and following breaks.**
- **Hands should be washed before wearing clean gloves.**
- **Clean-room quality protective clothing (lab coats, hats, hair nets, face masks) should be worn when working on vacuum components in CLEAN AREA.**

A Generic Cleaning Procedure

Mechanical Cleaning

↓

Degreasing or Solvent Cleaning*

↓

Detergent Cleaning*

↓

Chemical Etch*

↓

Electrolytic Polishing*

↓

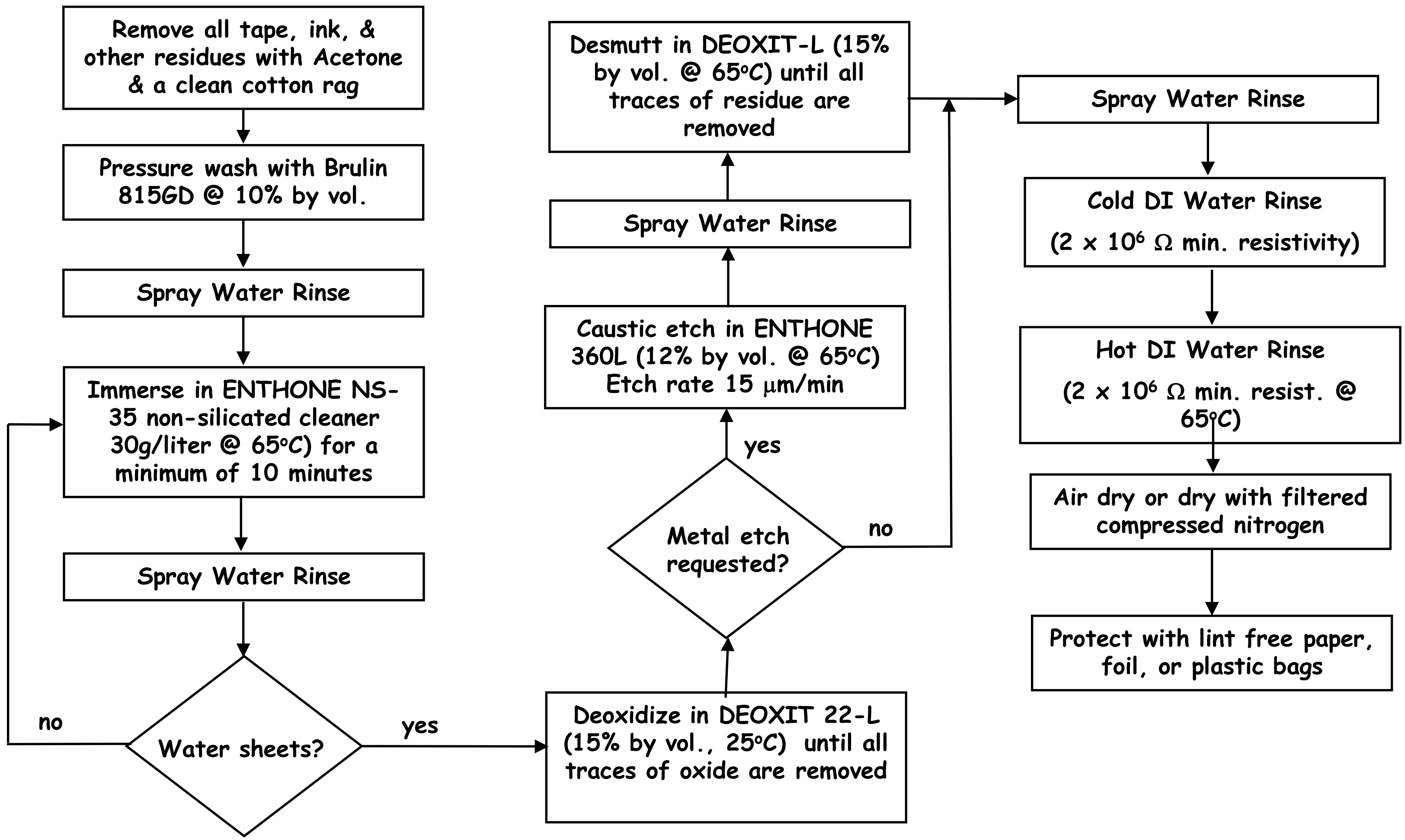
High Pressure Rinse

↓

Degassing

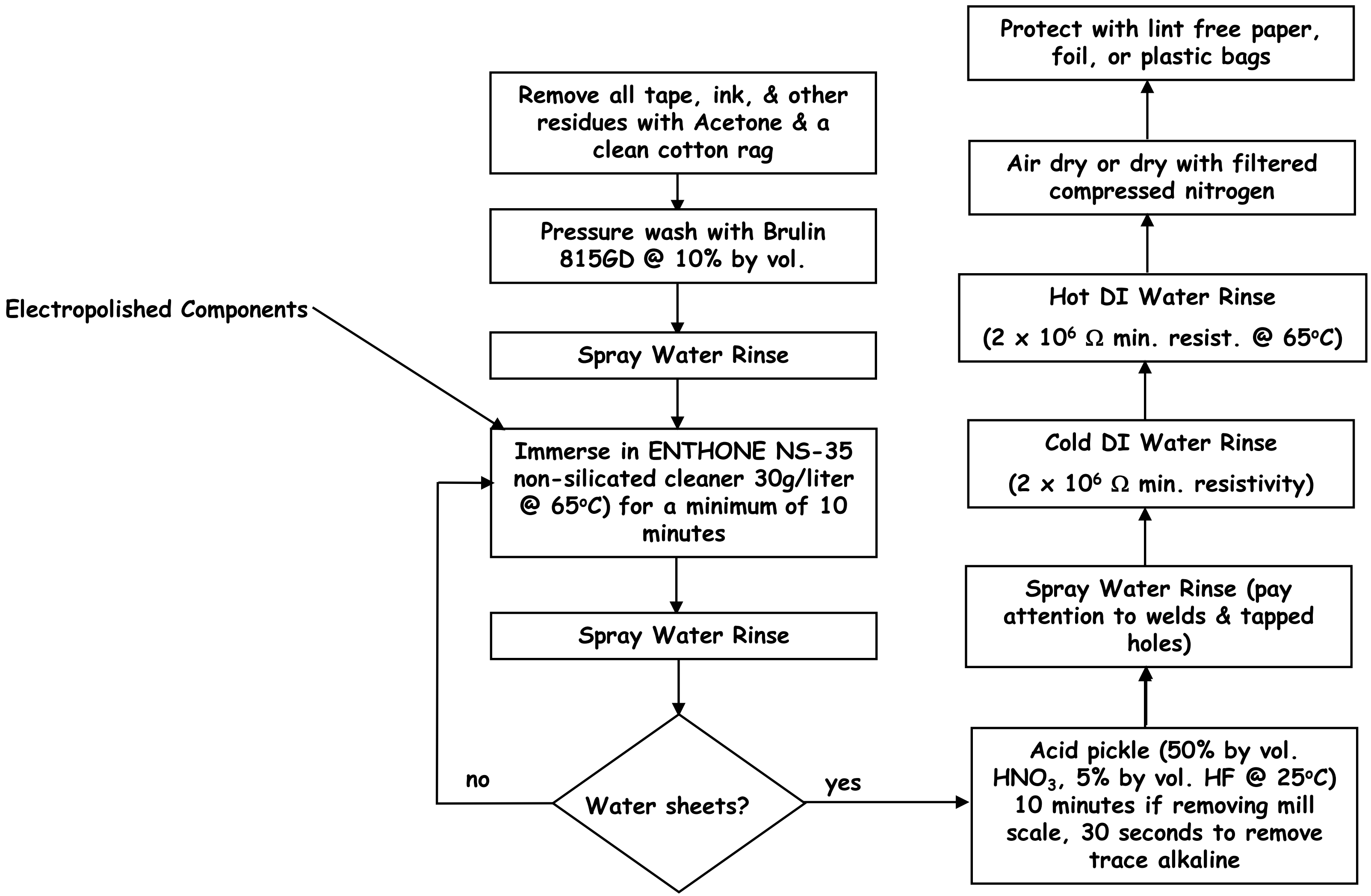
*Typically these steps are proceeded by a water rinse to avoid contamination of subsequent baths.

Cleaning of Aluminum Components



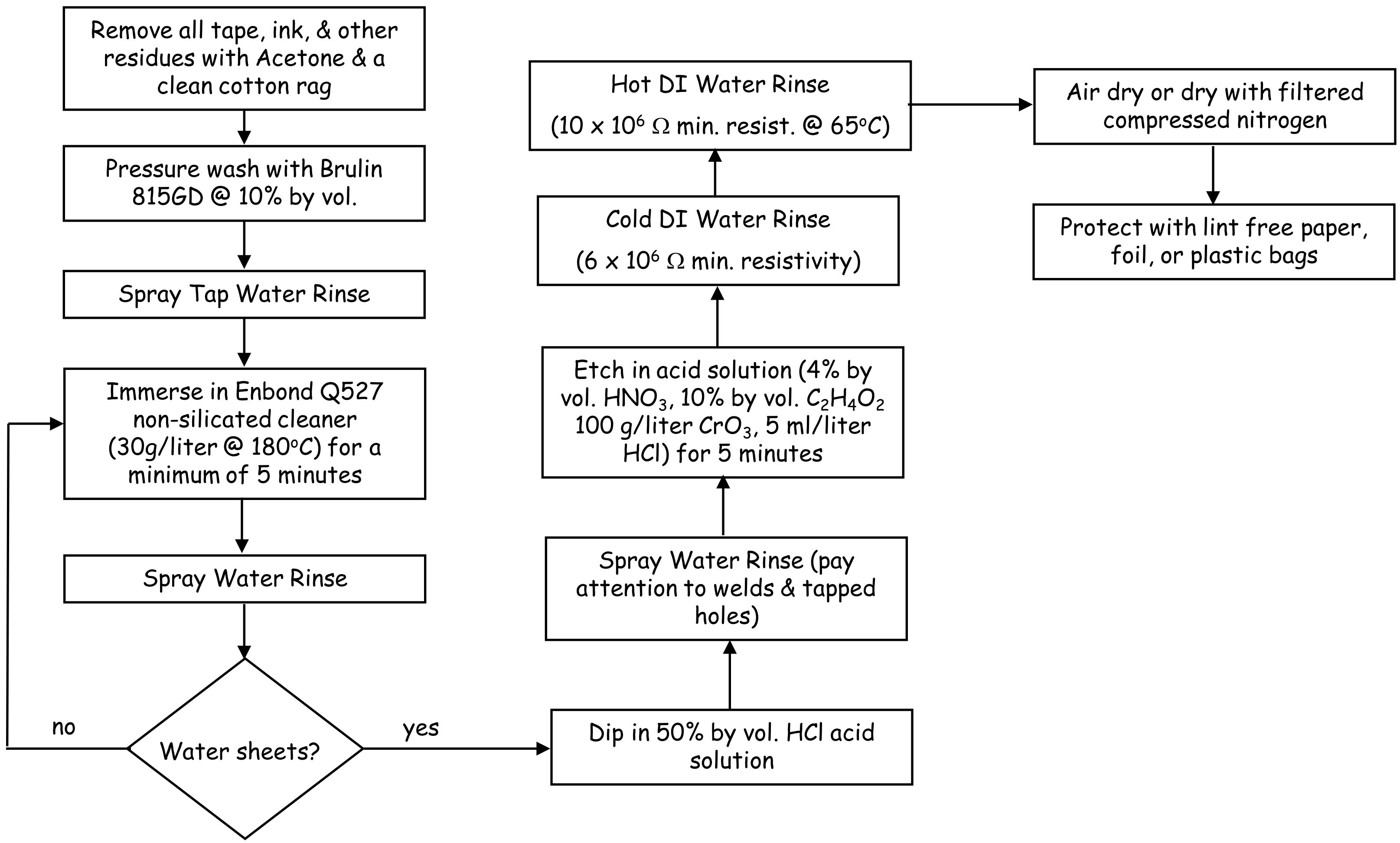
Ref. LLNL MMED Plating Shop Procedure

Cleaning of Stainless Steel Components



Ref. LLNL MMED Plating Shop Procedure

Cleaning of Copper and Glidcop Components



Ref. LLNL MMED Plating Shop Procedure

Cleaning Procedure Varies with Labs

CLASSE, Cornell Vacuum Part Cleaning Request Card

CHEM. ROOM JOB TICKET

Part name:
Submitted by:
Date required:

Accnt #:
Date submitted:
Material:
Quantity:

These basic cleaning usually sufficient

- ❑ Hand-scrub, soap & water
- ❑ Detergent & ultrasonic
- ❑ Solvent only in ultrasonic

Chemical etch:

- ❑ S.S. cold polish
- ❑ Copper cleaner
- ❑ BCP 1:1:2
- ❑ Aluminum etch
- ❑ Citric acid
- ❑ Citranox & water

Special instructions (times, temp., formulas, etc.):

These aggressive cleanings are used only necessary, such as parts for vacuum braze

Some of the More Aggressive Recipes

<u>NEW Copper Cleaner:</u>

1450 M.L. D.I. H_2O
1750 M.L. HNO_3
800 M.L. H_3PO_4

<u>OLD Copper Cleaner:</u>

1200 M.L. D.I. H_2O
1600 M.L. HNO_3
800 M.L. H_3PO_4
400 M.L. HF

<u>Aluminum Etch:</u>

sodium Hydroxide
Dissolve 6 oz. NaOH in 4 liters Hot H_2O

<u>Desmut:</u>

500 M.L. D.I. H_2O
190 M.L. H_2SO_4=Sulfuric
5 oz. Deox #7 Chromate
Then add 3300 M.L. D.I. H_2O

<u>S.S. Cold Polish</u>

500 M.L. H_2O
716 M.L. HF
1000 M.L. HNO_3
Then add 1684 M.L. Hot H_2O

<u>S.S. Pickle:</u>

400 Pickle
1000 M.L. HCl
120 M.L. HNO_3
2480 M.L. Hot H_2O

<u>Ti Pickle:</u>

80 M.L. HF
1200 M.L. HNO_3
2720 M.L. D.I. H_2O

By using water-soluble cutting fluid, these more aggressive etching were used much less at CLASSE, Cornell.

Cleaning with Ozonate Water

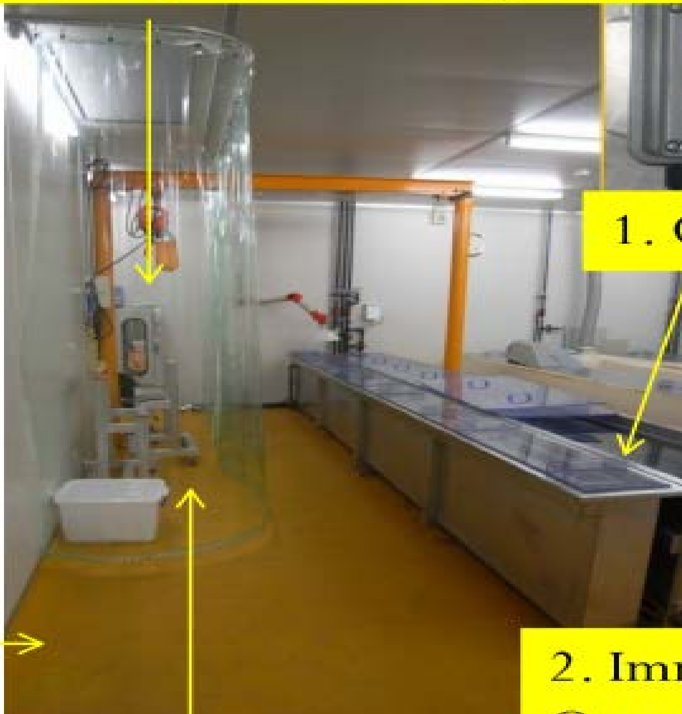
- *An effective cleaning method with ozone gas for the reduction of carbon contamination from the surface has been investigated by T. Momose [1].*
- *Another method of ozonate water cleaning for the superconducting RF cavities studied by K. Asano [2] shows good results of removing the surface contaminations.*
- *As a much more environmental friendly cleaner, ozonate water is also find wide-spread use in semiconductor industries for wafer cleaning.*
- *In TPS and NSLS II vacuum system fabrication, ozonate water cleaning has been employed as the primary cleaning for the aluminum chambers. (Data for Cu showed less benefit for ozonate water, as compared to Citranox® cleaning.)*

[1] T. Momose, Y. Maeda, K. Asano and H. Ishimaru, J. Vac. Sci. Technol. A13(3), 515 (1995).
[2] K. Asano, T. Furuya, S. Mitsunobu, T. Tajima and T. Takahashi, "Stable Performance of 508-MHz Superconducting RF Cavities for KEK B-Factory", KEK Preprint 95-191 (1996).

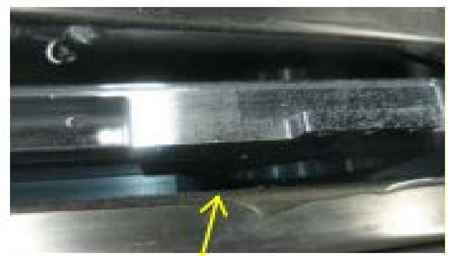
TPS's Ozonate Water Cleaning

Ozonated Water Cleaning (> 20 ppm)

4. Transport to Welding room



1. Ozonate water vessel



2. Immerse chamber in Ozonate water (30 min)

3. Drying in Clean booth

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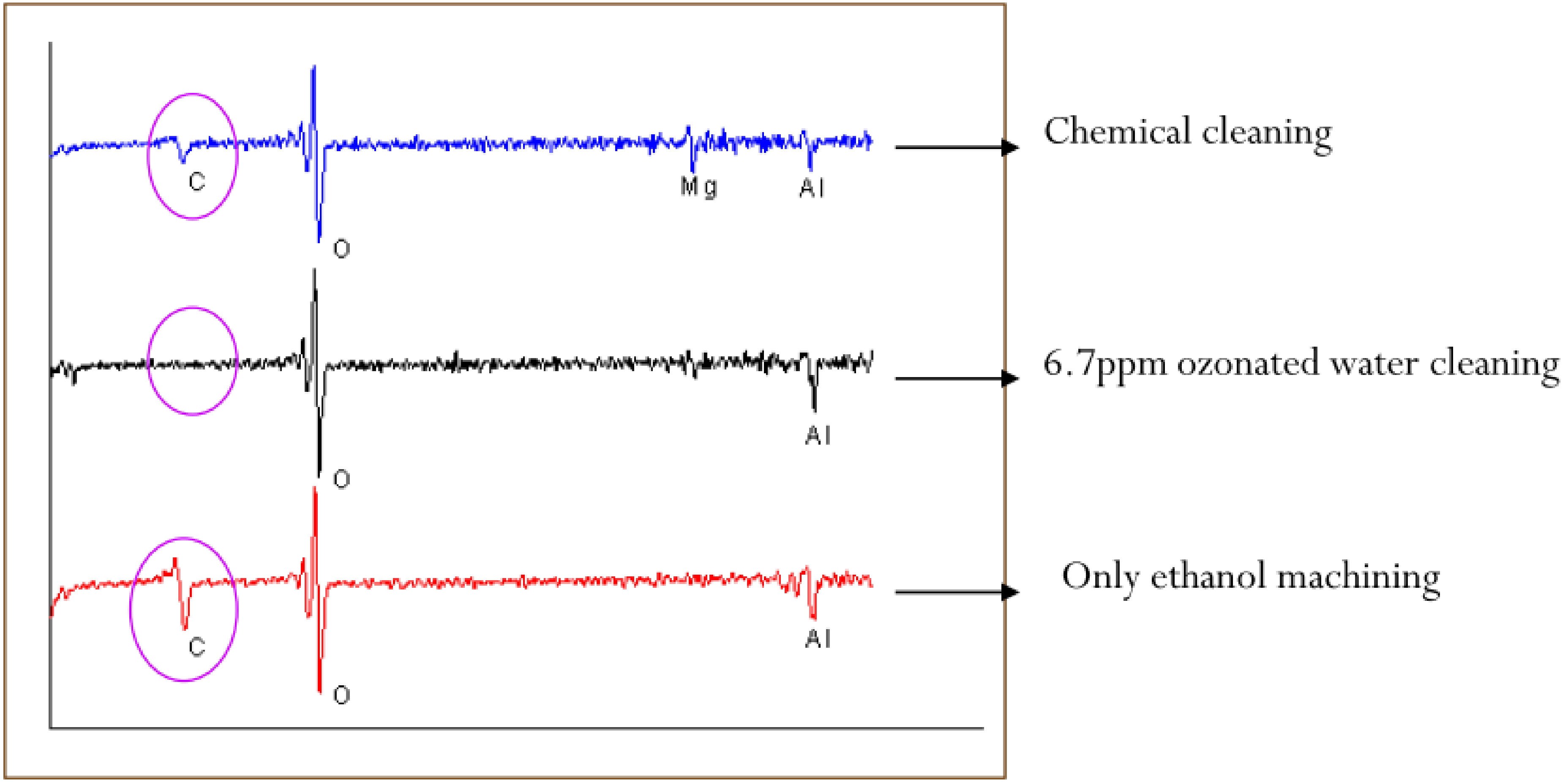
IUVSTA WS-63, Avila, Spain, Sep. 14-19, 2010

Surface analysis (AES) for Al samples



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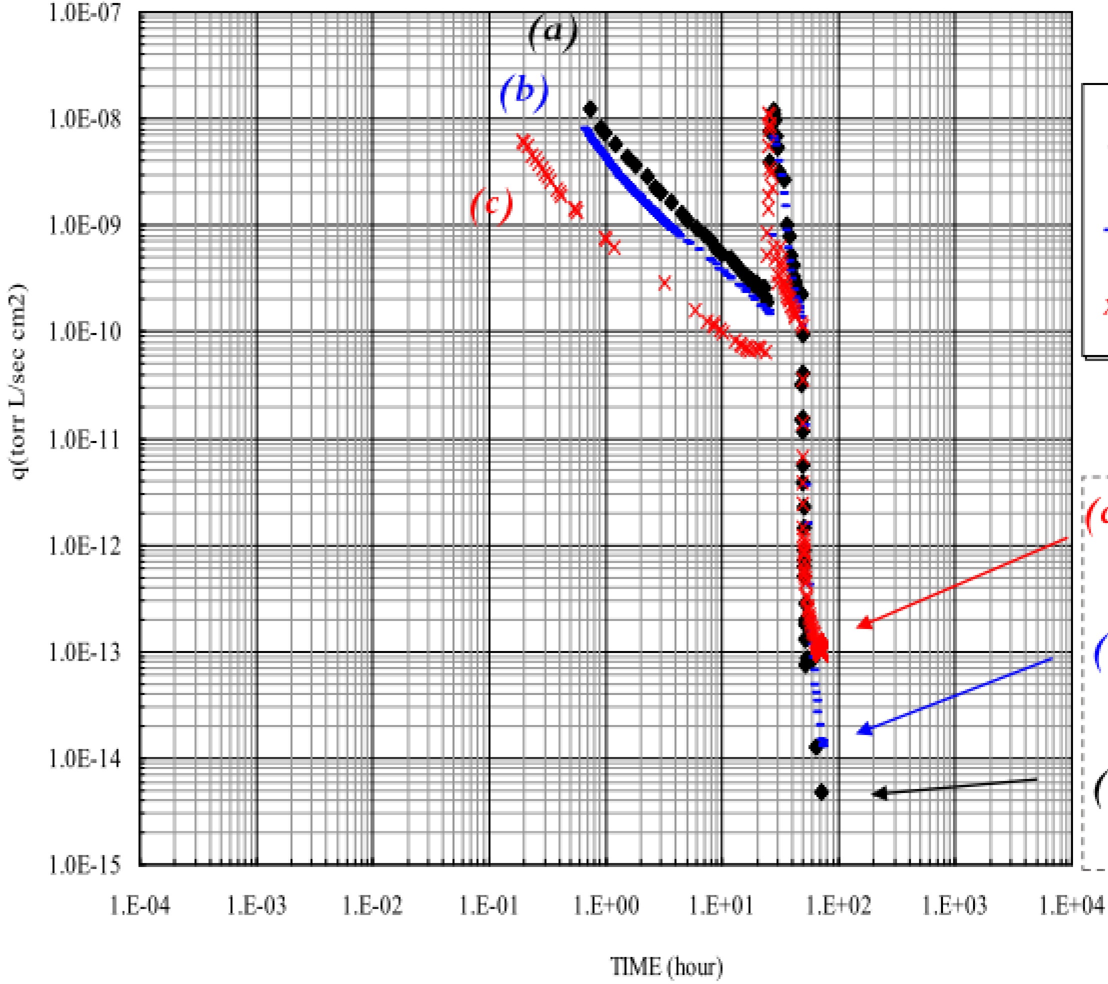
Thermal Outgassing Rate (Aluminum)



◆◆◆ *(a) O_3 Water Cleaning*

---- *(b) Ethanol CNC Machining*

xxx *(c) Chemical Cleaning*

After Baking :

(c) Chemical cleaning

$q_{72} = 9.5\times10^{-14}$ *Torr·L/s·cm²*

(b) Ethanol CNC Machining

$q_{72} = 1.4\times10^{-14}$ *Torr·L/s·cm²*

(a) O_3 Water Cleaning

$q_{72} = 4.8\times10^{-15}$ *Torr·L/s·cm²*

** 1 Torr·L/(s·cm²) ~ 1330 Pa·m/s*

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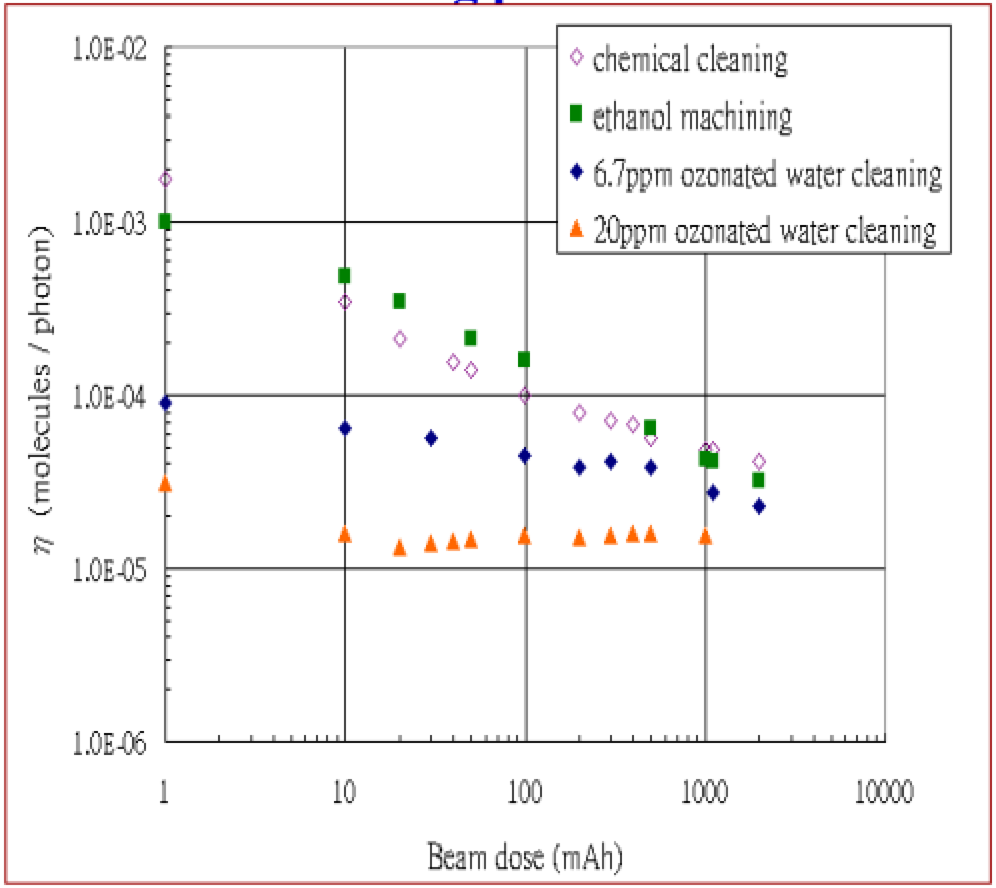
PSD yield for Aluminum samples

Yield of photon stimulated desorption (η) for various cleaning processes :



- η of ozonate water cleaning is more than one order of magnitude lower than those of only ethanol machining or chemical cleaning at 1mAh.
- η decreases with beam dose increases.
- Al samples with ozonate water cleaning maintain the lowest η at the beam dose through 1000 mAh.

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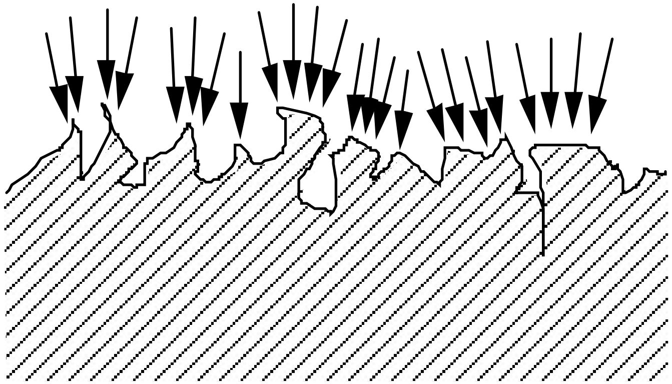
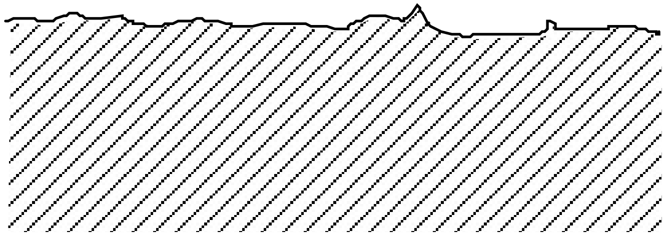
Electropolish

- Consider as a "reverse" electroplating technique.
- Metal is removed from the "high spots" due to higher current density.
- Surface metal is rich in H_2 and fluid until degassed.
- Electropolish produces a bright metallic finish.
- With proper rinsing and a post bake step, very low outgassing rates can be achieved.

Electric Field Lines are concentrated at peaks producing higher chemical milling rates



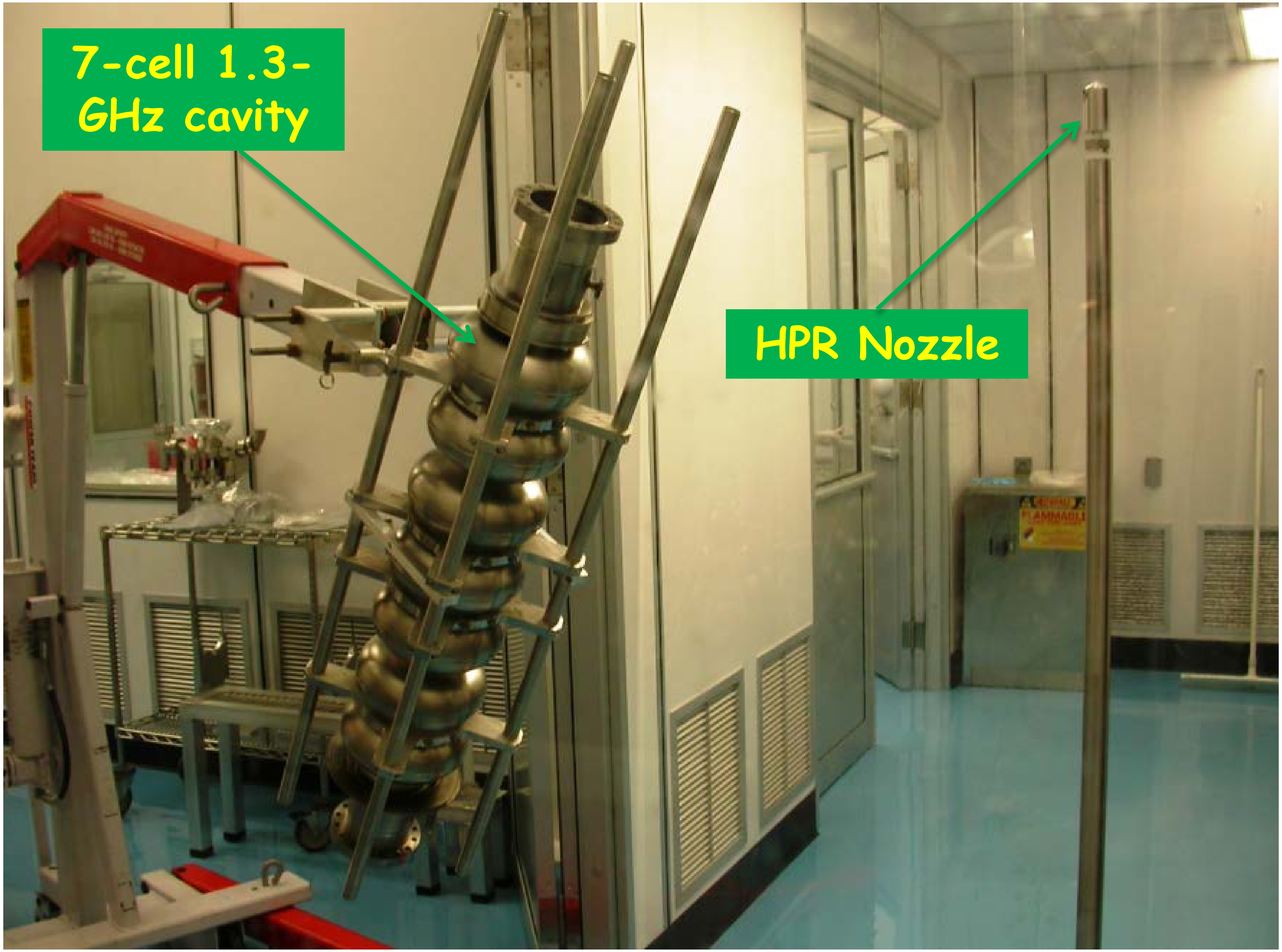
Resulting surface has reduced peaks, reduced surface area, and reduced outgassing



High Pressure Rinse (HPR)

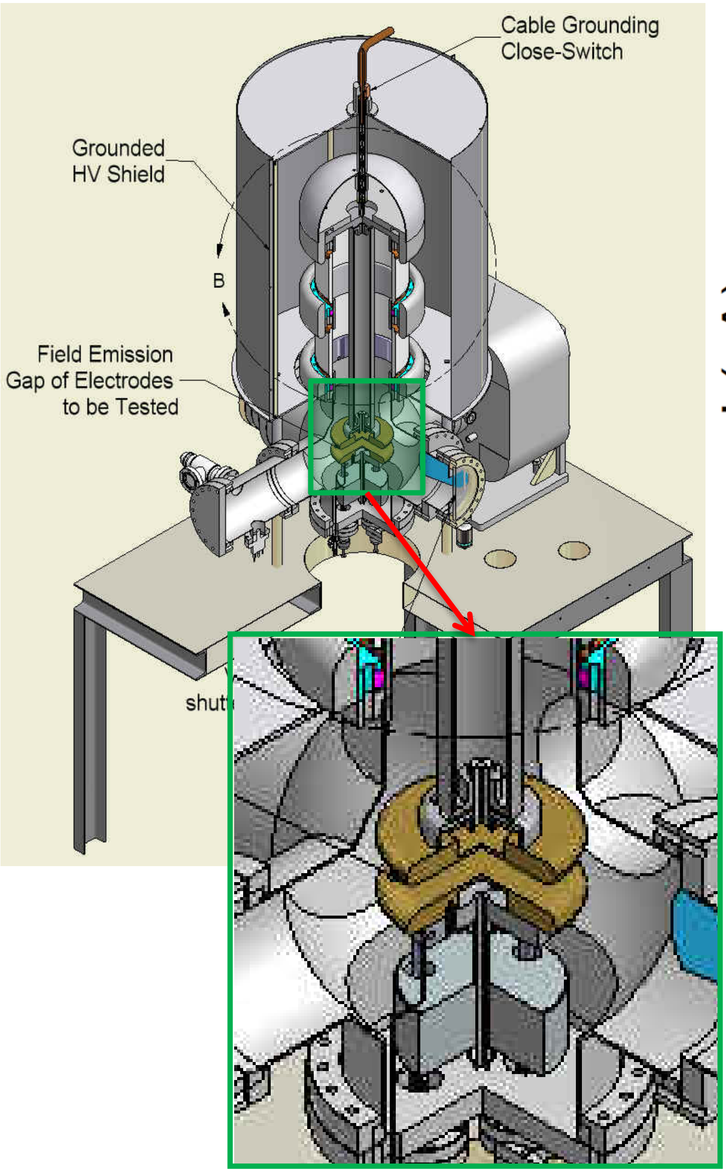
- *Fluid used can be tap water, deionized water, or with a detergent to assist in cleaning.*
- *With a detergent, this process is used early in the cleaning process. With deionized water, it is one of the final steps.*
- *Use of high fluid velocity to dislodge particles from the surface.*
- *Most effective cleaning method for particles in the 1 μm range.*
- *High pressure rinse can be effective on large parts, as well as small parts.*

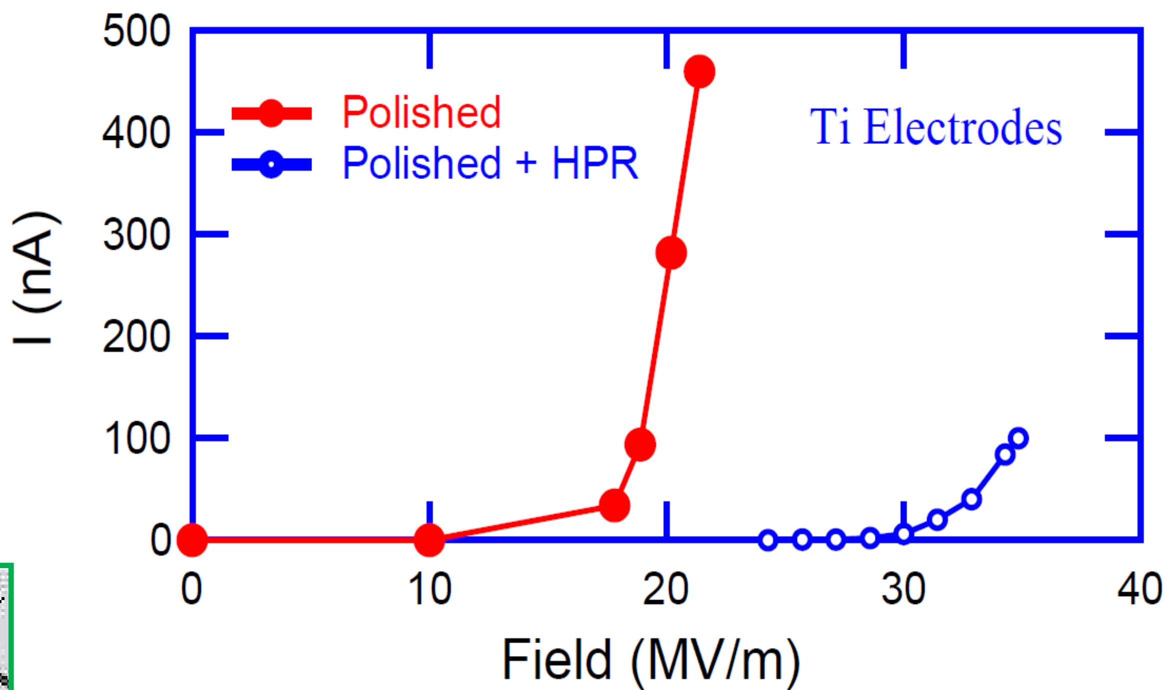
HPR SRF Cavity – Final Cleaning 1



HPR SRF Cavity – Final Cleaning 2

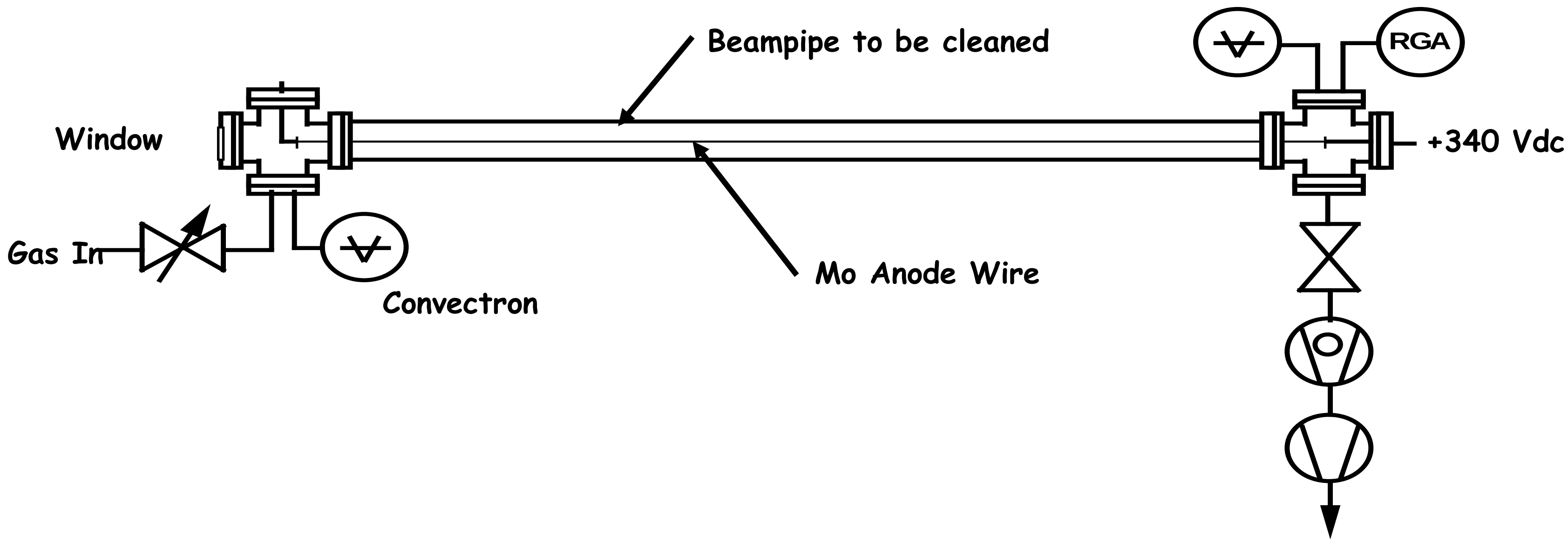
HPR HV Electrode for ERL





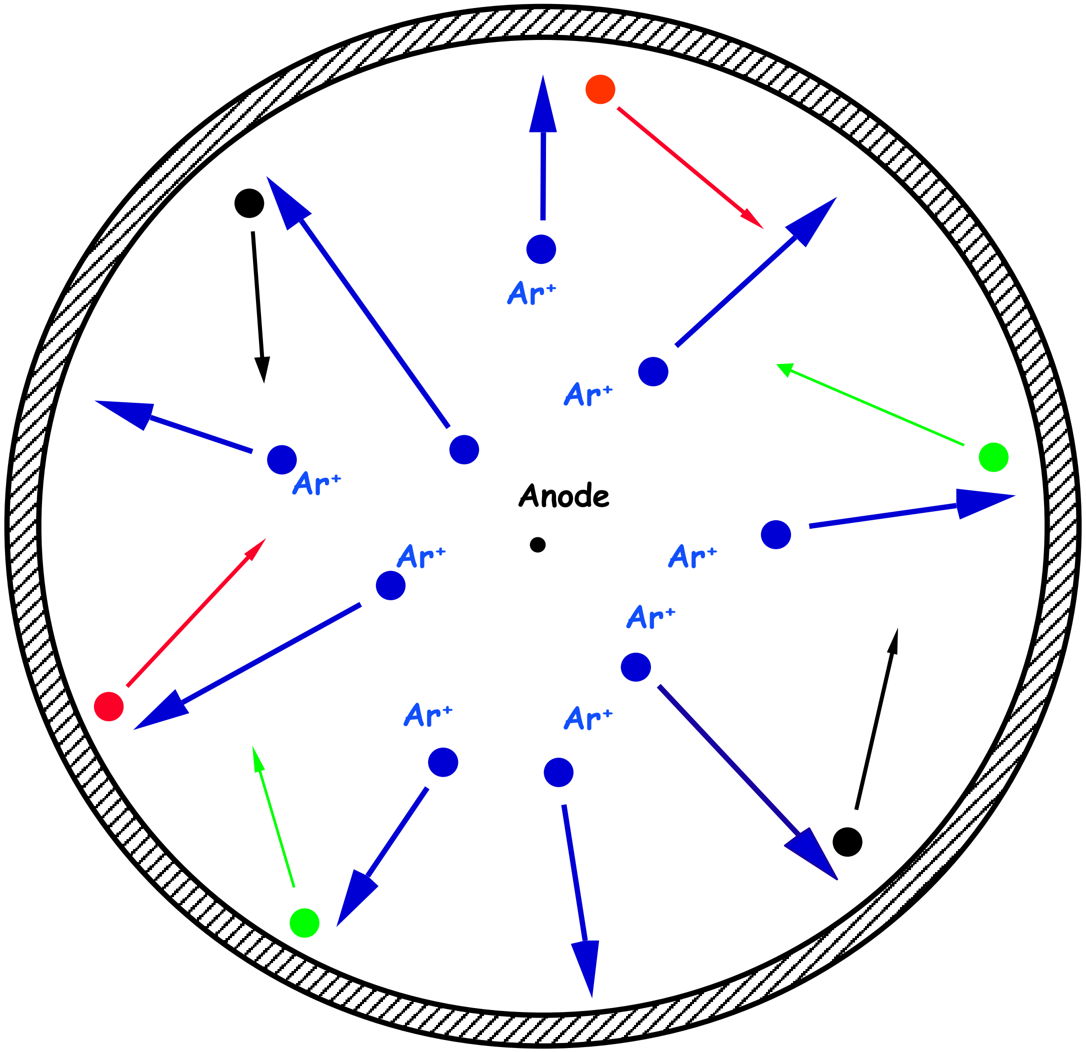
HPR approved to be very effective to remove field emitters for Cornell ERL DC gun structure

Glow Discharge Cleaning Schematic



- Glow discharge cleaning is useful in removing surface contamination such as C, S, H_2O, and organics.
- Must be a flowing system to prevent readsorption.
- Typical gases used are Ar, Ar-O_2, H_2.
- Glow discharge cleaning can leave higher levels of Ar and O_2 in the metal surface.
- A 200°C bakeout is still required after glow discharge cleaning.

Glow Discharge Cleaning



Various surface treatments evaluated by XPS

Surface analysis by x-ray photoelectron spectroscopy (XPS)

Surface Treatment	XPS Surface Atom %					
	Cu	O	N	C	Cl	Ar
Chem. Cleaning (old SLAC recipe)	22.4	22.5	11.9	41.6	1.6	-
Chem. Cleaning (new SLAC recipe)	43.4	36.8	-	17.9	1.9	-
GDC - 95% Ar, 5% O_2 (2×10^{19} ions/cm^2)	50.6	40.0	-	8.0	-	1.4
GDC - 95% Ar, 5% O_2 (2×10^{18} ions/cm^2)	48.6	42.0	-	8.0	-	1.4
GDC - 100% H_2 (2×10^{18} ions/cm^2)	64.2	23.6	-	12.2	-	-

Ref. "Processing of OFE Copper Beam Chambers for PEP-II High Energy Ring", Hoyt et al, 1995 Particle Accelerator Conference

Bakeout and Degassing

- *Vacuum firing of components will result in low outgassing rates*

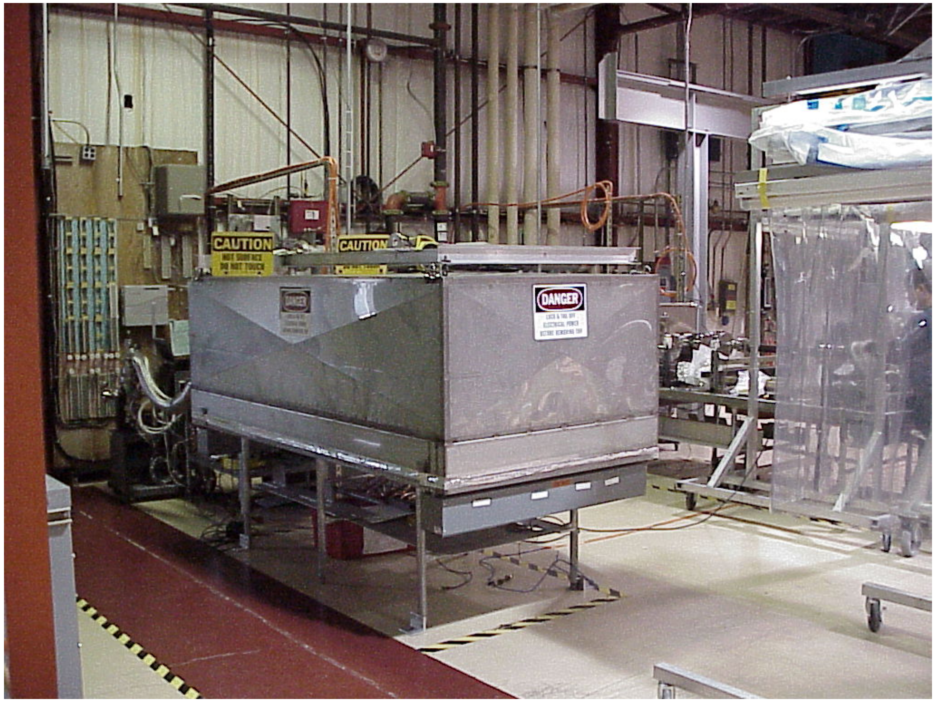
  (T = 800°C - 1000°C, P~10^{-4} Torr for several hours).
  - *Bulk H_2 is depleted from metal*
  - *Works well for stainless steels*
  - *copper and aluminum are annealed*

- *Heating systems for bakeout*
  - *Ovens are the easiest to use*
  - *Heater tapes with insulation*
  - *Nichrome wire covered with ceramic beads*
  - *Calrods or heater bands with insulation*
  - *Heater blankets (built-in insulation)*

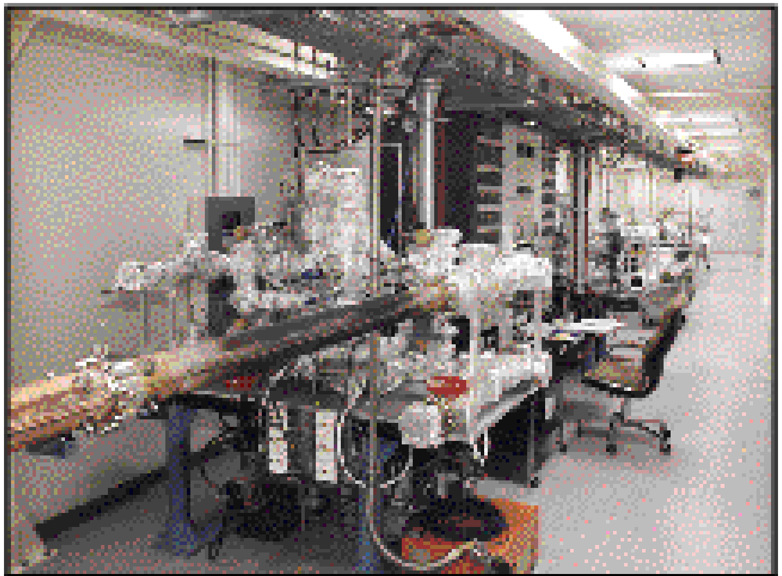
SLAC Glow Discharge and Bakeout Station

Glow Discharge Station

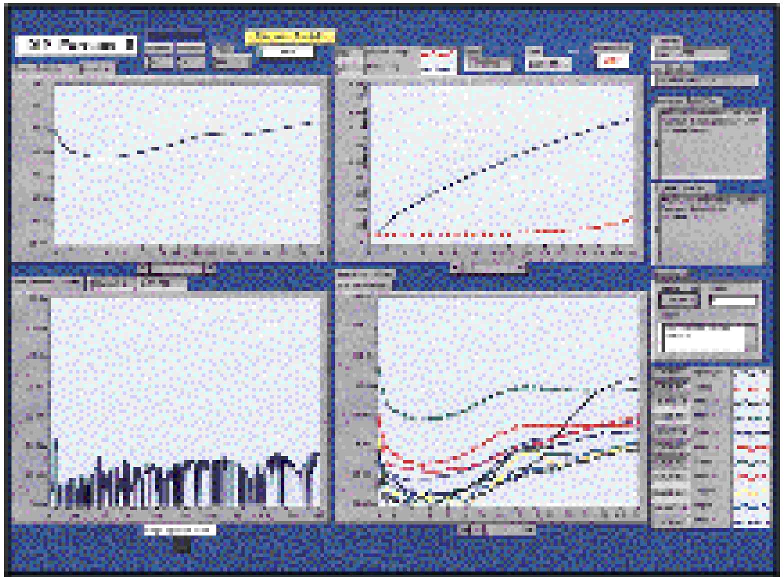
Bakeout Oven Enclosure (200°C)



LLNL Glow Discharge and Bakeout Station

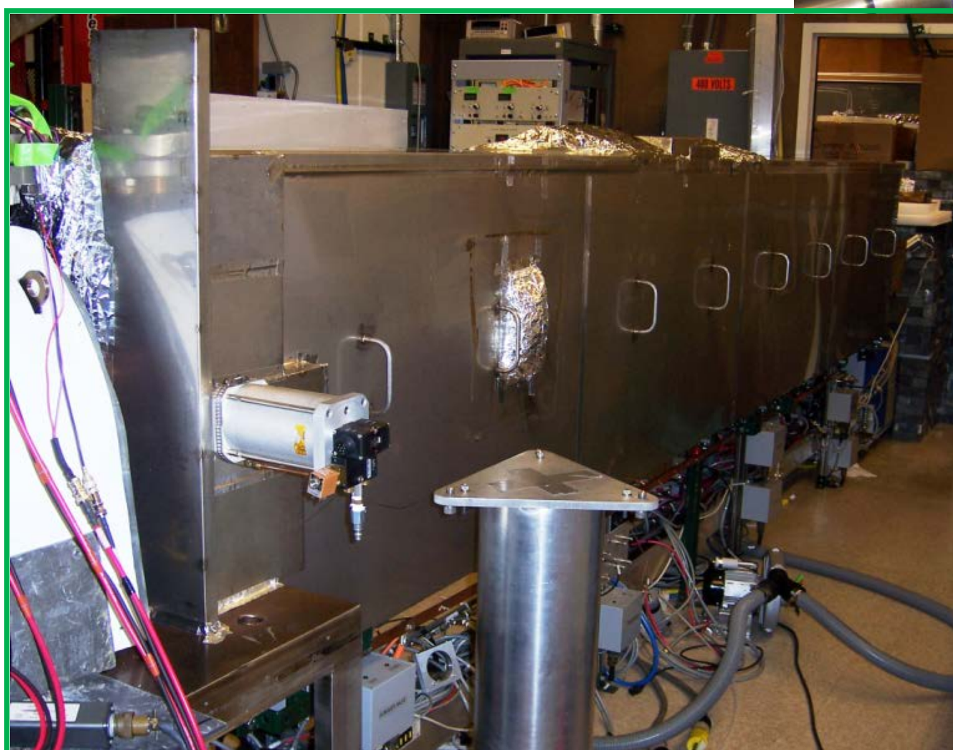


ATEG glow discharge and bake station



RGA and LabView software records the results of processing

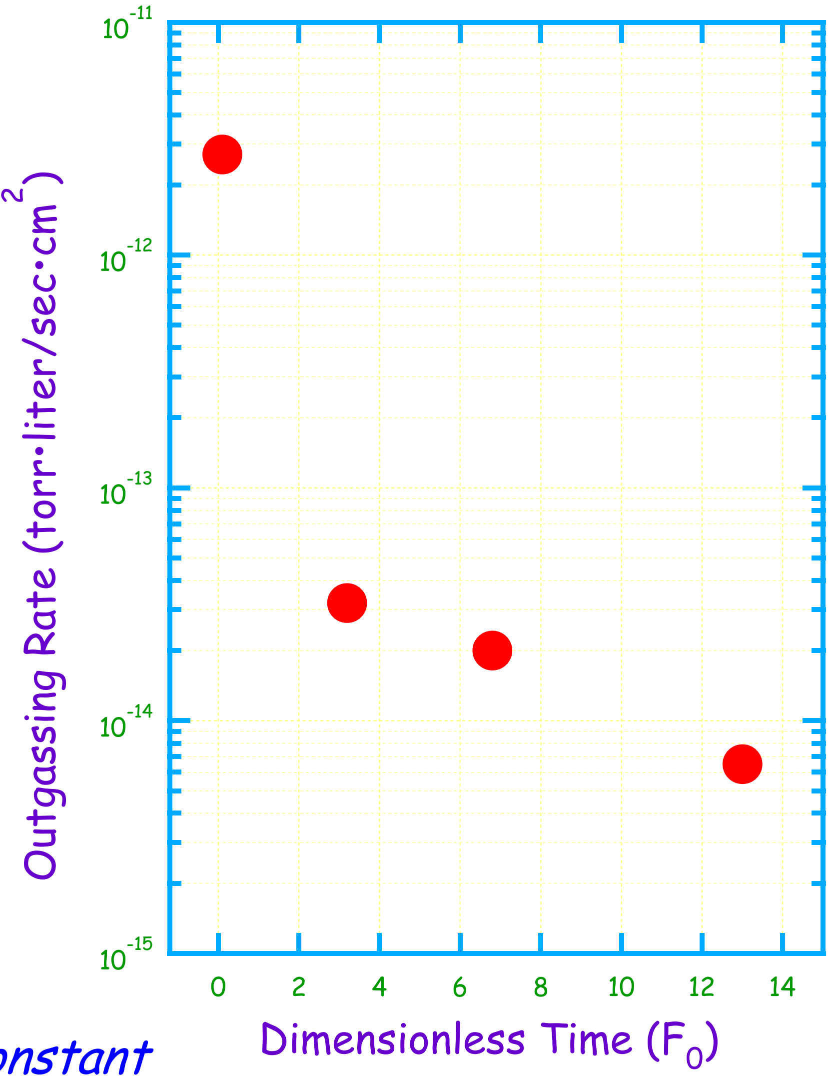
In-situ bakeout of Cornell ERL Beamline



Cornell's Recipe for Degassing SSTs

- *The heat-treated (400°C bake in air for a duration of up to 100 hr) all stainless steel materials for the gun chamber.*
- *SST outgassing rates (after a 150°C vacuum bakeout) below 10^{-13} torr·l/s·cm² achieved with F_0>3, a dimensionless time scale, F_0.*

$$F_0 = 4Dtd^{-2}$$



- *t – heating time; d –thickness*
- *$D=D_0 \exp(-E_d/kT)$ – Hydrogen diffusion constant*