Cavity Processing and Cleanroom Techniques

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SRF2015 Tutorial No. 8

Saturday, September 12th, 2015

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Preface

- Tutorial will introduce basics and methods for SRF cavity processing and cleanroom techniques
- Additional information for sections can be found in appendix identified on slides by:

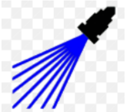
See APPENDIX for more details

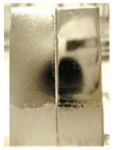
- Practical applications presented will focus on low beta quarter-wave and half-wave cavities
- However there are many publications on elliptical cavity processing and cleanroom assembly available online and in past tutorials

Sections

1. SRF coldmass and cavity workflow
2. SRF cavity receiving and inspection
3. Degreasing
4. Ultra pure water and high pressure rinse
5. Mechanical surface preparation
6. Removal by chemical etching
7. Preparation by electro polishing
8. Safety considerations
9. Heat treatments
10. Cleanroom protocols
11. Cleanroom assembly techniques





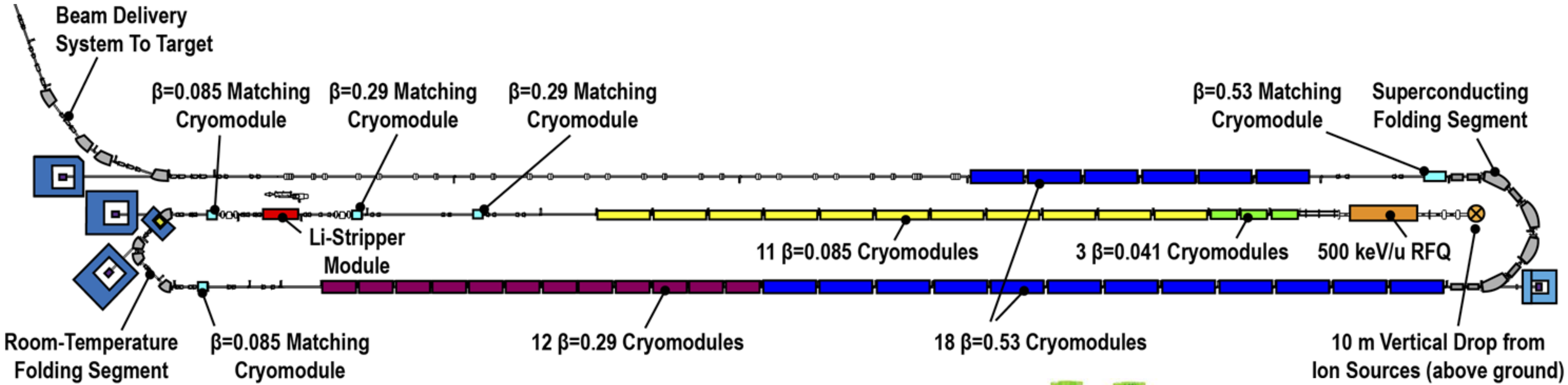





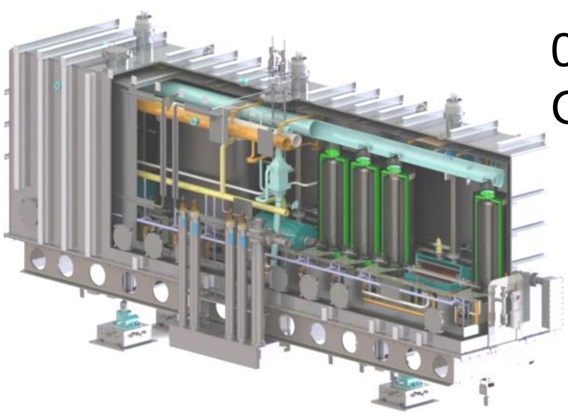
1. SRF Coldmass and Cavity Workflow

SRF Cavity Processing and Coldmass Assembly for LINACS

- SRF cavities are used for accelerators across the world
- Fabricated from SC material niobium metal which has T_c of 9.7 K
- Many cavities required to construct accelerator systems ranging from tens to hundreds of cavities



FRIB has 332 SRF cavities



0.085QWR Cryomodule

SRF Cavities All Shapes and Sizes

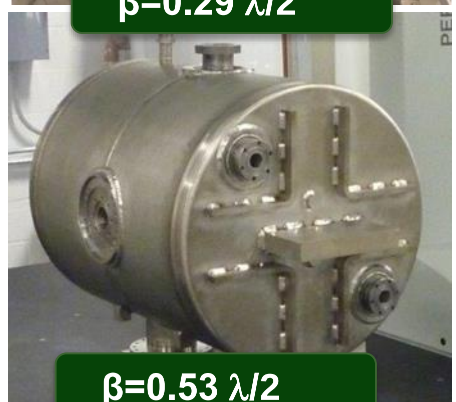
There are elliptical type cavities used for high-beta :

And then there are low-beta; λ/4 (QWR), λ/2 (HWR), single-spoke and multi-spoke.

β=0.041 λ/4

β=0.085 λ/4

β=0.29 λ/2



β=0.53 λ/2

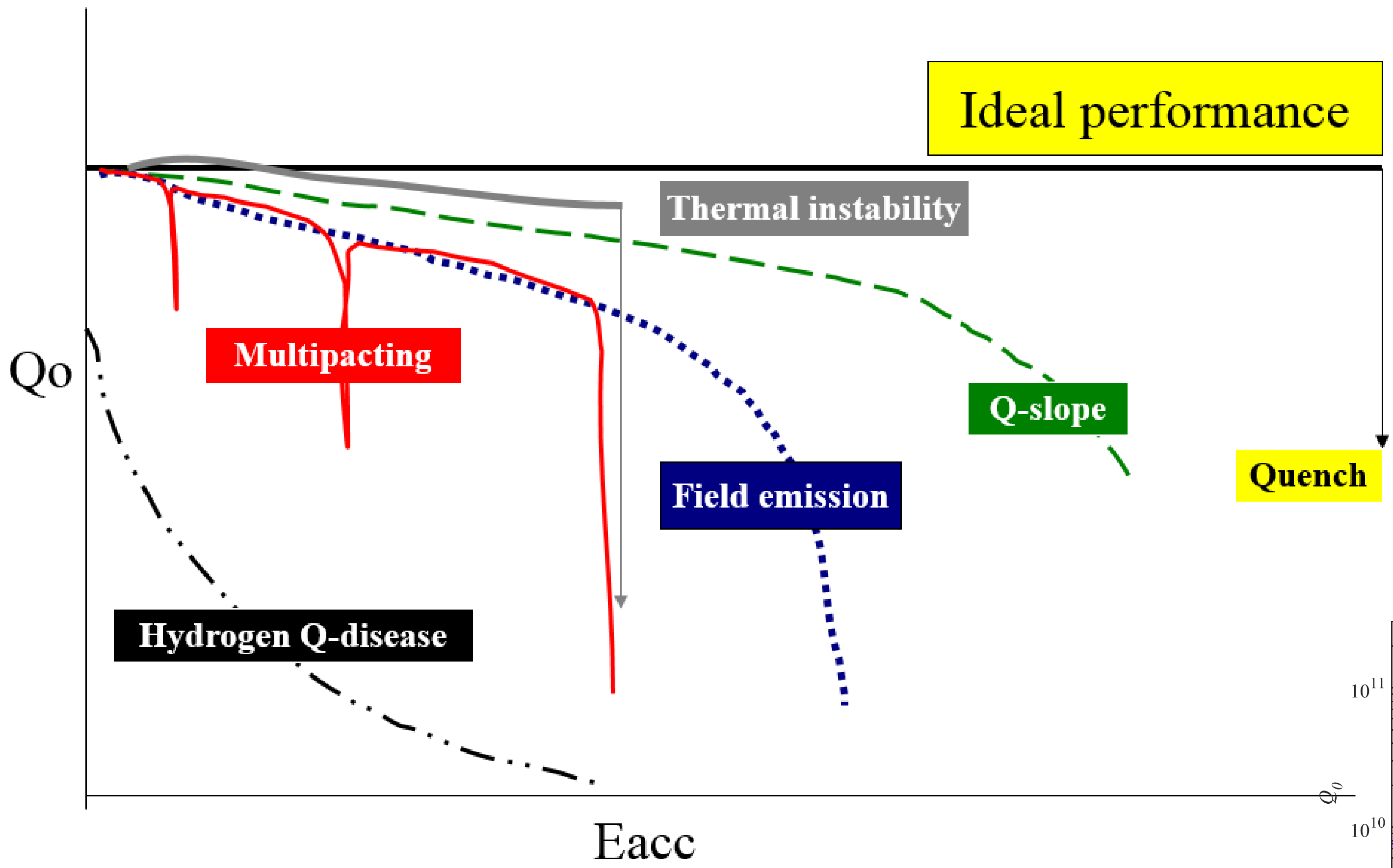
β=0.12 λ/4

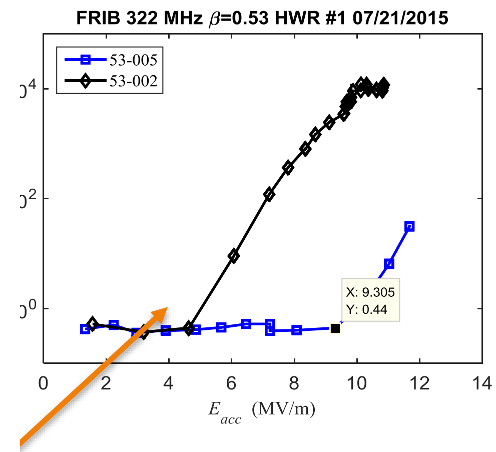
16 Quarter-Wave Resonator, 88.05 MHz, beta 0.12
S. Bousson, IPN Orsay, MSU – 11th August 2011

SRF Cavity Requirements to Achieve High Performance

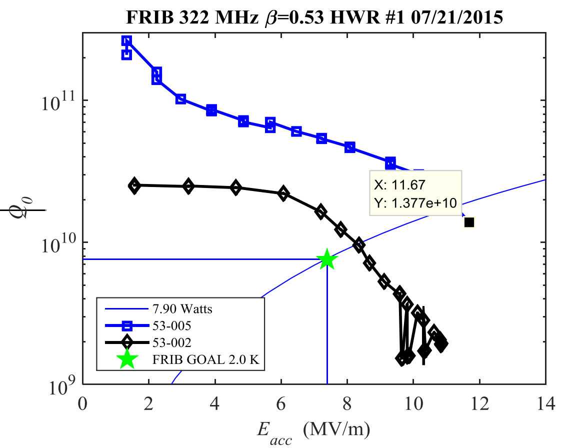
- High RRR niobium material (>250 RRR residual resistance)
- Defect and inclusion free surfaces
- Accurate geometry to meet high tolerance RF surface shapes
- Leak free welds and seal surfaces to provide ultra high vacuum space
- Smooth RF surface – surface roughness < 2 R_a
- Contamination, grease and particle free surfaces on RF beamline
- Meticulous procedure and quality assurance program required to deliver production quantity cavities
- **ALL OF THESE REQUIREMENTS MUST BE MET TO HAVE HIGH PERFORMANCE CAVITY!**

Common Cavity Performance Limitations and Issues





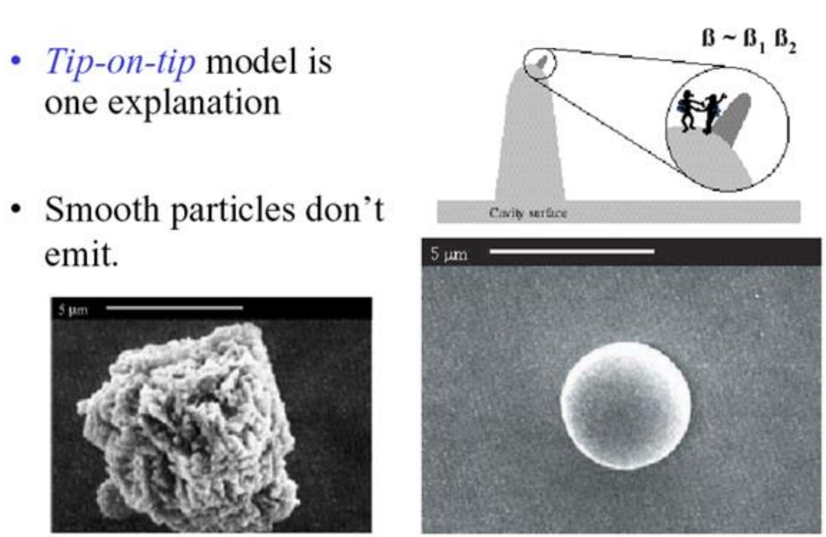
FIELD EMISSION

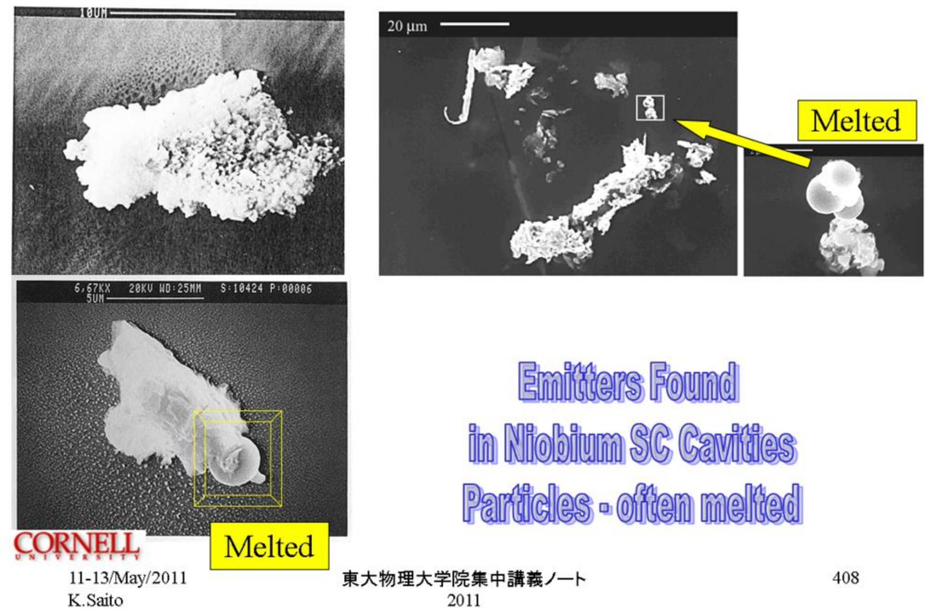


Kenji Saito, Lecture note in Tokyo University on May 2011

Fabrication and Processing Errors Cause Performance Problems!

- Most common performance limitation related to field emission
- Caused by submicron particulate and surface imperfections
- **MAIN GOAL: CLEAN, particle free, smooth RF surface**



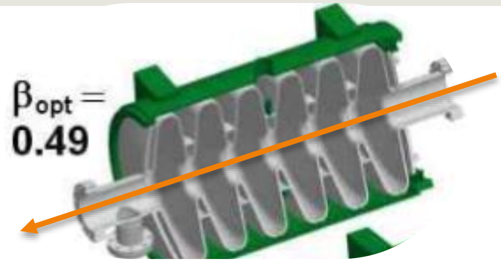


Better processing, cleaning and assembly techniques push out field emission onset level !

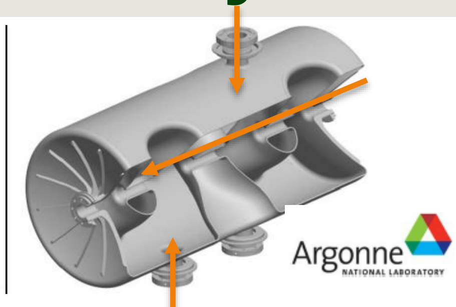
Kenji Saito, Lecture note in Tokyo University on May 2011

Processing Techniques are Developed Based on Cavity Geometry

- Elliptical access through beam port *equator difficult to reach.
- QWR, HWR and spoke complicated structure fabrication, cleaning, processing and assembly more involved
- Cavity mechanical design consider access for cleaning and processing all surfaces.
- Cavities have critical surfaces for:
  - RF performance,
  - UHV vacuum seals
  - tuning mechanism
  - cryogenic connections



Cross-section of the β=0.49 RIA elliptical cavity.



Section view of the ANL β ≅ 0.63 triple-spoke cavity

QWR have removable bottom flange for access to clean and process

HWR have custom cleaning ports on each end for access to clean and process

PROTOTYPE SUPERCONDUCTING TRIPLE-SPOKE CAVITY FOR BETA = 0.63
K.W. Shepard, M.P. Kelly, J.D. Fuerst, M. Kedzie, and Z.A. Conway, ANL, Argonne, IL 60439, U.S.A.

Low Beta Coldmass Workflow

Major Component

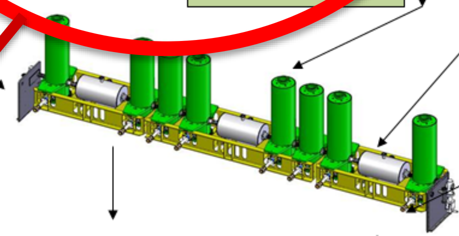
Nb material acceptance testing (Cavity Fabrication Group)

COLDMASS RAILS
- Q/A
- Inspect & check field
- Degrease rails & components
- Assemble cold mass cart
- Assemble end plates

CAVITY
- Q/A – leak check - inspection
- Degrease – bulk etch & custom etch (150 microns) & H degas (600C)
- Degrease USC cavity & vacuum components
- Light etch (30 microns) & HPR
- Assemble to test stand, pump down & leak check
- Low temp bake
- RF vertical test prep
- RF vertical test & certify
- Prep insert for cleanroom
- Purge & remove from insert → 20% reprocess
- Install cavity to rails

SOLENOID
- Q/A & final alignment machining
- Inspect & degrease
- USC & dry solenoid
- Install solenoid

FUNDAMENTAL POWER COUPLER
- Q/A
- Process FPC components
- Assemble FPC to test stand
- Pump down & leak check
- FPC bake and HP conditioning
- Purge & remove from test stand
- Install FPC to cavity

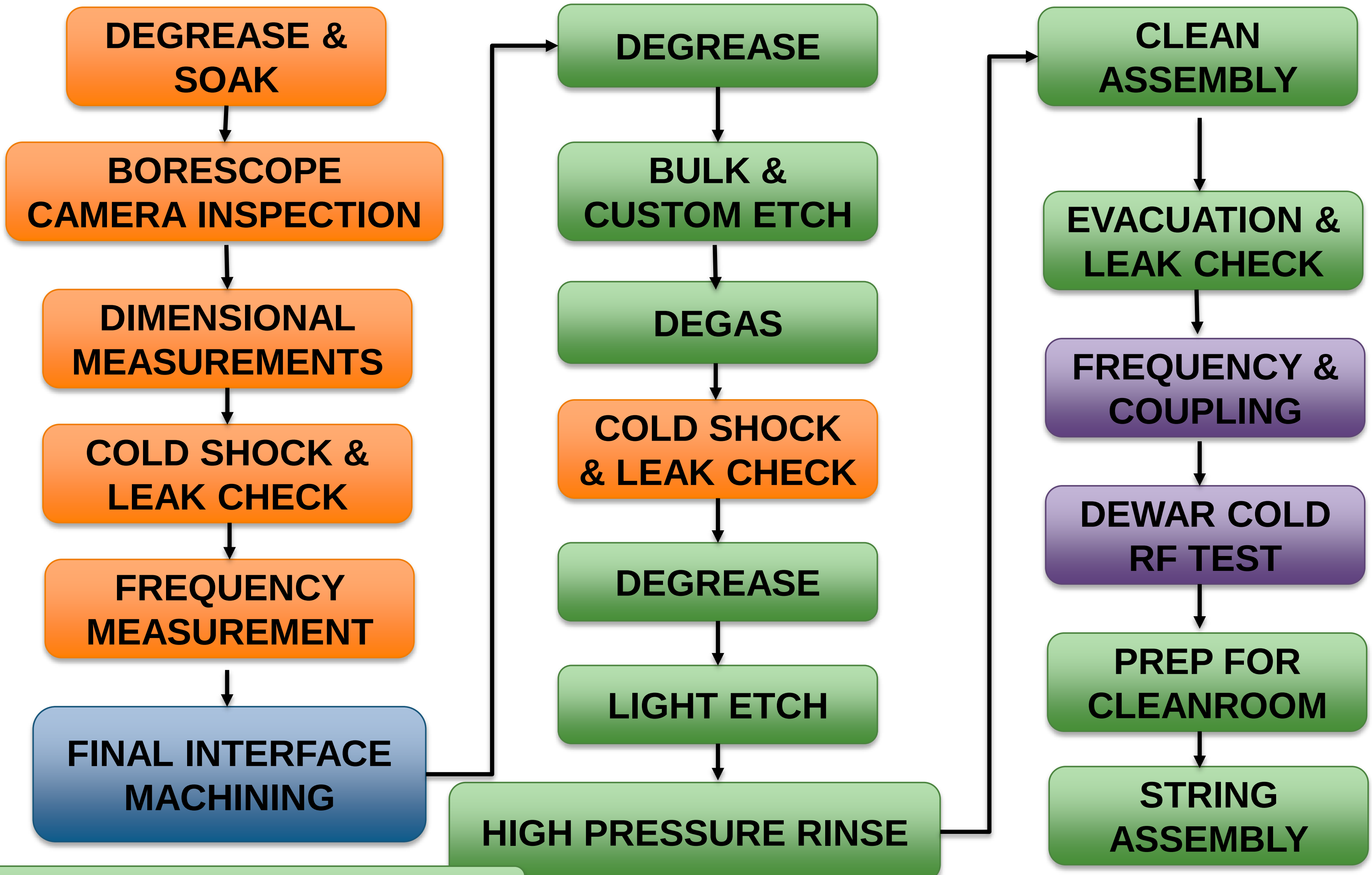


- Coldmass pump down, leak check & diagnostic checks
- Remove from cleanroom
- Cryomodule Assembly

Color Key:
- Vendors
- Inspection Group
- Processing & Coldmass Assembly Group
- RF Test & Measurement Group
- Use if necessary to recover lower Q

Lecture to focus on cavity processing and assembly

Low Beta Cavity Floor Router

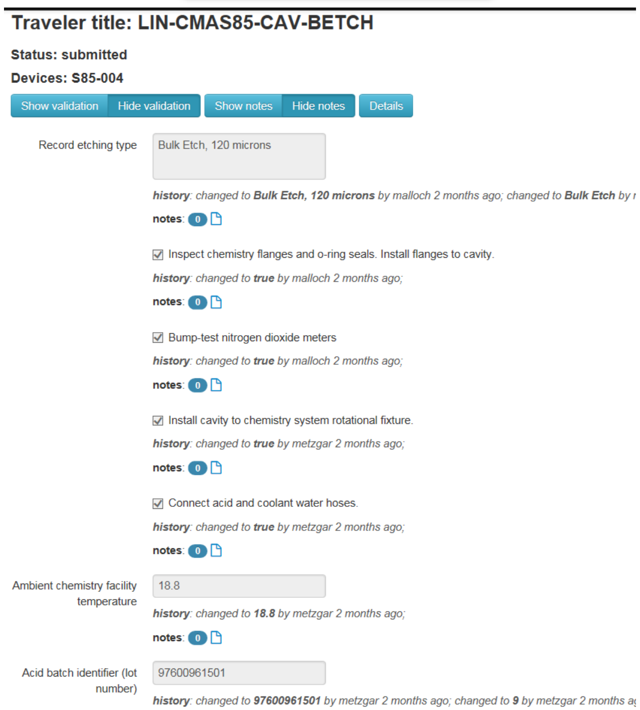


See APPENDIX for more details

Importance of Work Control for High Quality and Repeatability

- Each process step has **standard operating procedure** and **router checklist** to ensure repeatability and accuracy in work flow
- **E-travelers** to collect variables and data for each router step
- Non-conformance tracking & resolution important to catch fabrication errors early in production and **take corrective actions**

E-traveler



Traveler title: LIN-CMAS85-CAV-BETCH

Status: submitted

Devices: S85-004

Show validation | Hide validation | Show notes | Hide notes | Details

Record etching type: Bulk Etch, 120 microns

history: changed to **Bulk Etch, 120 microns** by malloch 2 months ago; changed to **Bulk Etch** by r

notes: 0

☑ Inspect chemistry flanges and o-ring seals. Install flanges to cavity.

history: changed to **true** by malloch 2 months ago;

notes: 0

☑ Bump-test nitrogen dioxide meters

history: changed to **true** by malloch 2 months ago;

notes: 0

☑ Install cavity to chemistry system rotational fixture.

history: changed to **true** by metzgar 2 months ago;

notes: 0

☑ Connect acid and coolant water hoses.

history: changed to **true** by metzgar 2 months ago;

notes: 0

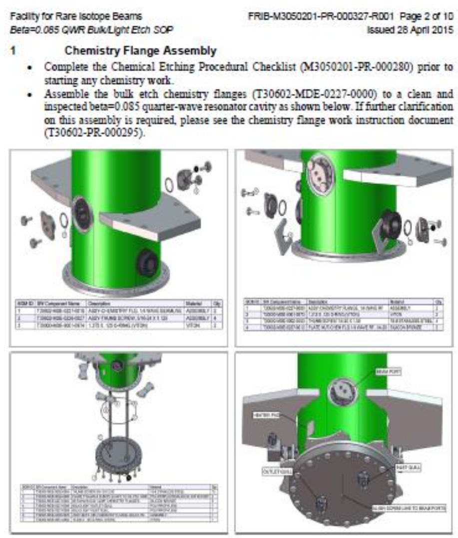
Ambient chemistry facility temperature: 18.8

history: changed to **18.8** by metzgar 2 months ago;

notes: 0

Acid batch identifier (lot number): 97600961501

history: changed to **97600961501** by metzgar 2 months ago; changed to **9** by metzgar 2 months a

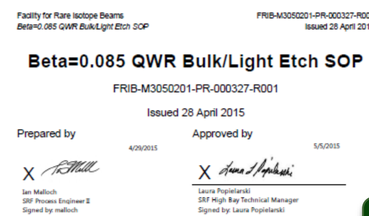


Facility for Rare Isotope Beams
Beta=0.085 QWR Bulk/Light Etch SOP
FRIB-M3050201-PR-000327-R001 Page 2 of 10
Issued 28 April 2015

1 **Chemistry Flange Assembly**

- Complete the Chemical Etching Procedural Checklist (M3050201-PR-000280) prior to starting any chemistry work.
- Assemble the bulk etch chemistry flanges (T30602-MDE-0227-0000) to a clean and inspected beta=0.085 quarter-wave resonator cavity as shown below. If further clarification on this assembly is required, please see the chemistry flange work instruction document (T30602-PR-000295).

Standard Operating Procedure (SOP)



Facility for Rare Isotope Beams
Beta=0.085 QWR Bulk/Light Etch SOP
FRIB-M3050201-PR-000327-R001
Issued 28 April 2015

Beta=0.085 QWR Bulk/Light Etch SOP

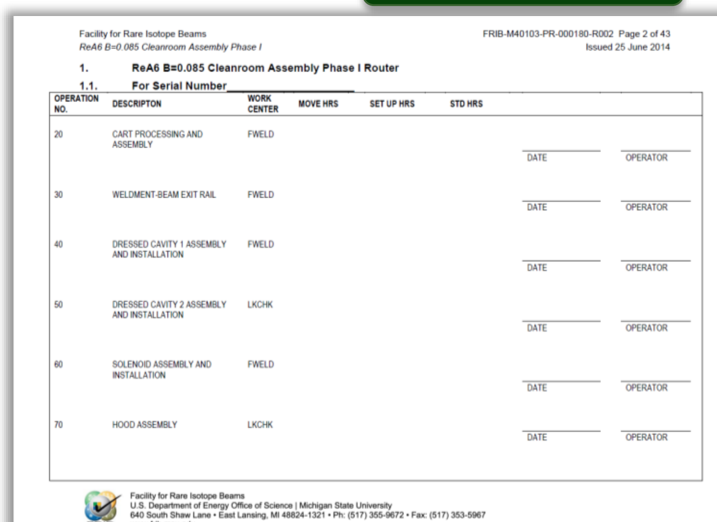
FRIB-M3050201-PR-000327-R001

Issued 28 April 2015

Prepared by: 4/29/2015 — Ian Malloch, SRF Process Engineer II, Signed by: malloch

Approved by: 5/5/2015 — Laura Popielarski, SRF High Bay Technical Manager, Signed by: Laura Popielarski

Floor Router



Facility for Rare Isotope Beams
ReA6 B=0.085 Cleanroom Assembly Phase I
FRIB-M40103-PR-000180-R002 Page 2 of 43
Issued 25 June 2014

1. **ReA6 B=0.085 Cleanroom Assembly Phase I Router**

1.1. **For Serial Number** ______________

OPERATION NO.	DESCRIPTON	WORK CENTER	MOVE HRS	SET UP HRS	STD HRS		
20	CART PROCESSING AND ASSEMBLY	FWELD				DATE	OPERATOR
30	WELDMENT-BEAM EXIT RAIL	FWELD				DATE	OPERATOR
40	DRESSED CAVITY 1 ASSEMBLY AND INSTALLATION	FWELD				DATE	OPERATOR
50	DRESSED CAVITY 2 ASSEMBLY AND INSTALLATION	LKCHK				DATE	OPERATOR
60	SOLENOID ASSEMBLY AND INSTALLATION	FWELD				DATE	OPERATOR
70	HOOD ASSEMBLY	LKCHK				DATE	OPERATOR

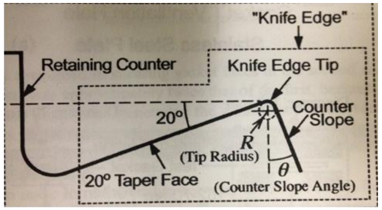
FRIB
Facility for Rare Isotope Beams
U.S. Department of Energy Office of Science | Michigan State University
640 South Shaw Lane • East Lansing, MI 48824-1321 • Ph: (517) 355-9672 • Fax: (517) 353-5967
www.frib.msu.edu

2. SRF Cavity Receiving and Inspection

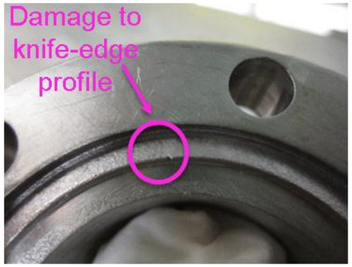
SRF Cavity
Quality Acceptance Inspection

- All cavities are inspected
- Inspection may include:
  - Vendor report reviewed & material certs.
  - critical dimensions measured
  - Visual inspection by scopes& cameras
  - Frequency measurement
  - Coupling measurements
  - Cold shock & vacuum leak check
  - Water soak to expose any iron inclusions

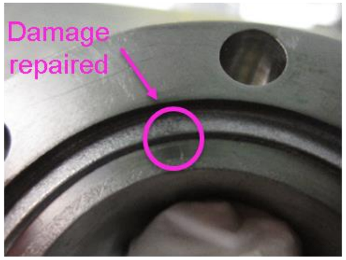
Post Soak



Conflat Standard		Tip radius (mm)	Counterslope angle (degrees)
American	A	0.050	5
European	E	0.105	40



Nonconformance – identified and defined (vacuum leak)



Nonconformance Action - vendor rework/protective covers

- All critical surfaces are inspected→ RF surfaces and electron beam welds inspected with digital borescope
- All sealing surfaces are checked with magnifying tools

Borescope

Coordinate Measurement Machine

This is what your hand looks like to an SRF cavity, washed or not!

1. Always wear powder free latex or nitrile gloves while touching cavities or coldmass components.
2. Always cover seal surfaces and cavity ports with caps

3. Degreasing

What is clean?

I'm usually DIRTY upon arrival

- Clean surfaces **free of films and particulate**
  - Films = Grease, skin oil, soap residue, polymers
  - Particulate = dust, dirt, dry skin
- Contamination causes field emission and performance limitations
- **Purpose to make all surfaces free from films and particulate !**

Grease in tapped holes

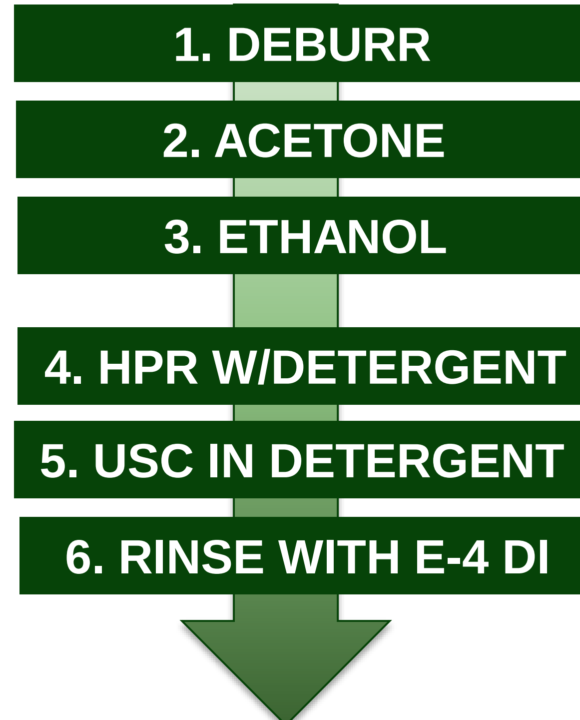
Residue on cavity surface

Grease from vacuum components

1. DEGREASE
2. REMOVE PARTICULATE
3. INSPECT

Degreasing

- Remove gross contamination: burrs, grease or oil from fabrication steps
- Wipes should minimize lint
- Solvent wiping or degreasing with acetone and/or ethanol useful to superficial residue
- High pressure spraying at ~ 1000 psi with detergents
- Ultra sonic cleaning with detergents effective



Detergent (solution)	pH of solution	Uses	May Corrode
Micro 90 ® (0.5-2%)	9.7	Nb, NbTi, SS	Zinc, Al, Cu, Ni
Surface Cleanse 903 ®	6.5	Cu, Al, other	May craze polycarbonate
Tikopur R33 (1%)	9.9	Nb, NbTi, SS	
Citranox ® (1-2%)	2.5	Remove metal oxides	Soft metals

Ultra Pure Water Requirements

- E-1 and E-4 used for FRIB production SRF facility
  - Some labs use E-1.1 -1.2
- E-4 (filtered DI) water only for USC outside of cleanroom
- E-1 (Ultra pure water) all other points of use (POU) including:
  - Chemistry tools
  - Final cleanroom USC
  - Cleanroom POU
  - High pressure rinse
- The key to pure water systems are: reduction of particles, total organic carbon (TOC), and silica

D5127 – 13

TABLE 1 Requirements for Water at the Point of Distribution in the Electronics and Semiconductor Industries[A]

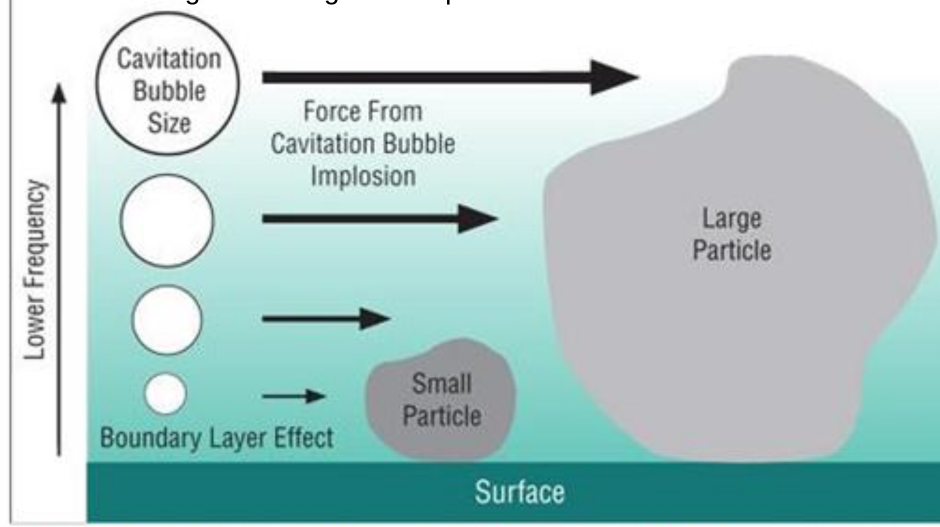
Parameter	Type E-1	Type E-1.1	Type E-1.2[B]	Type E-1.3[B]	Type E-2	Type E-3	Type E-4
Linewidth (microns)	1.0–0.5	0.35–0.25	0.18–0.09	0.065–0.032	5.0–1.0	>5.0	—
Resistivity, 25°C (On-line)	18.1	18.2	18.2	18.2	16.5	12	0.5
TOC (µg/L) (on-line for <10 ppb)	5	2	1	1	50	300	1000
On-line dissolved oxygen (µg/L)	25	10	3	10	—	—	—
On-Line Residue after evaporation (µg/L)	1	0.5	0.1		—	—	—
On-line particles/L (micron range)							
>0.05 µm				500			
0.05–0.1		1000	200	N/A	—	—	—
0.1–0.2	1000	350	<100	N/A	—	—	—
0.2–0.5	500	<100	<10	N/A	—	—	—
0.5-1.0	200	<50	<5	N/A	—	—	—
1.0	<100	<20	<1	N/A	—	—	—
SEM particles/L (micron range)							
0.1–0.2	1000	700	<250	N/A	—	—	—
0.2–0.5	500	400	<100	N/A	3000	—	—
0.5–1	100	50	<30	N/A	—	10 000	—
10	<50	<30	<10	N/A	—	—	100 000
Bacteria in CFU/Volume							
100 mL Sample	5	3	1	N/A	10	50	100
1 L Sample			10	1			
10 L Sample				1			
Silica – total (µg/L)	5	3	1	0.5	10	50	1000
Silica – dissolved (µg/L)	3	1	0.5	0.5	—	—	—
Anions and Ammonium by IC (µg/L)							
Ammonium	0.1	0.10	0.05	0.050	—	—	—
Bromide	0.1	0.05	0.02	0.050	—	—	—
Chloride	0.1	0.05	0.02	0.050	1	10	1000
Fluoride	0.1	0.05	0.03	0.050	—	—	—
Nitrate	0.1	0.05	0.02	0.050	1	5	500
Nitrite	0.1	0.05	0.02	0.050	—	—	—
Phosphate	0.1	0.05	0.02	0.050	1	5	500
Sulfate	0.1	0.05	0.02	0.050	1	5	500
Metals by ICP/MS (µg/L)							
Aluminum	0.05	0.02	0.005	0.001	—	—	—
Antimony				0.001			
Arsenic				0.001			
Barium	0.05	0.02	0.001	0.001	—	—	—
Boron[C]	0.3	0.1	0.05	0.050	—	—	—
Cadmium				0.010			
Calcium	0.05	0.02	0.002	0.001	—	—	—
Chromium	0.05	0.02	0.002	0.001	—	—	—
Copper	0.05	0.02	0.002	0.001	1	2	500
Iron	0.05	0.02	0.002	0.001	—	—	—
Lead	0.05	0.02	0.005	0.001	—	—	—
Lithium	0.05	0.02	0.003	0.001	—	—	—
Magnesium	0.05	0.02	0.002	0.001	—	—	—
Manganese	0.05	0.02	0.002	0.010	—	—	—
Nickel	0.05	0.02	0.002	0.001	1	2	500
Potassium	0.05	0.02	0.005	0.001	2	5	500
Sodium	0.05	0.02	0.005	0.001	1	5	1000
Strontium	0.05	0.02	0.001		—	—	—
Tin				0.010			
Titanium				0.010			
Vanadium				0.010			
Zinc	0.05	0.02	0.002	0.001	1	5	500
Temperature Stability (K)				±1			
Temperature Gradient (K/10 min)				<0.1			
Dissolved Nitrogen On-line (mg/L)				8-18			
Dissolved Nitrogen Stability (mg/L)				±2			

Ultra Sonic Bath Parameters

- 40 kHz operation
- Temperature control to140^{0}F
- Timer 30-60 min
- Hot water more effective
- 0.5– 3% detergent in DI water
- Recirculation pump with filter
- New solution for final steps
- Thoroughly rinse with E-4

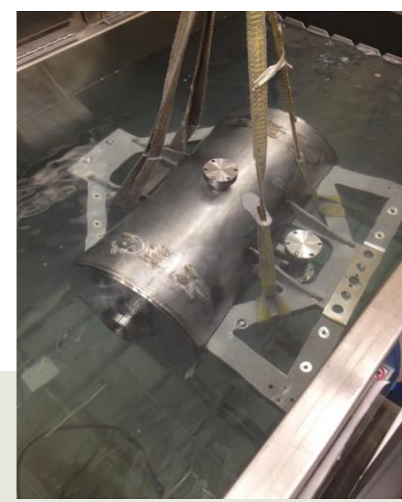
See APPENDIX for more details

Article From: 10/19/2011 Production Machining, John Fuchs from Cleaning Technologies Group



In ultrasonic cleaning, as the frequency decreases, the cavitation bubbles get larger (and the number of bubbles decreases). Larger (more energetic) bubbles are more effective on larger particles.

200 gallon (FRIB cavity size) volume with weir



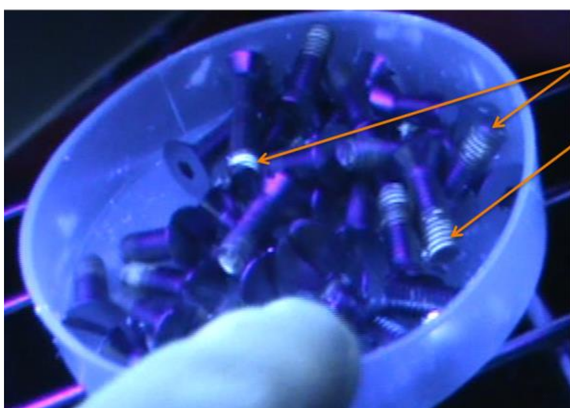
Manual High Pressure Rinse

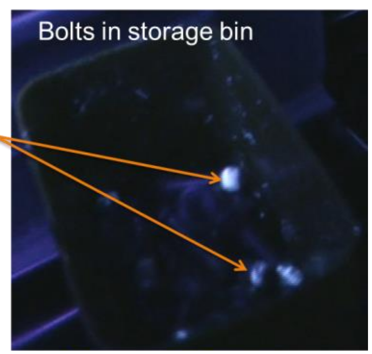
- Very large components not fit in USC rinsed manually
- Detergent & DI water deliver to nozzles
- String rails, carts, cryomodule components, cryogenic lines
- Force from high pressure water dislodges particles
- Also used in cleanrooms final rinsing flanges and fasteners

← wash down booth

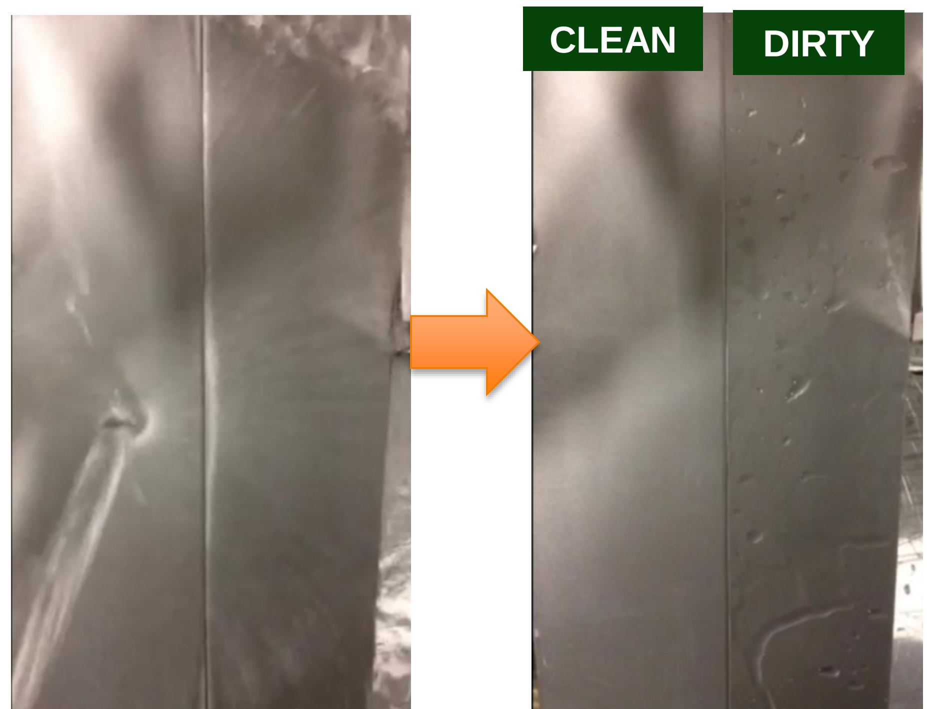
Tests to Check Cleanliness

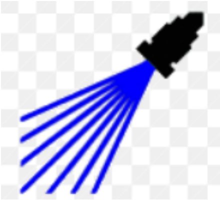
- **Total organic carbon content measurements**
- Visual inspection and white poly wipe
- **Water break free test**
- Clean gas spray & count
- Surface particle counts
- **UV light inspection**
- Residual gas analysis





Grease easily detected on bolt threads
Identify bolts that have not been cleaned properly





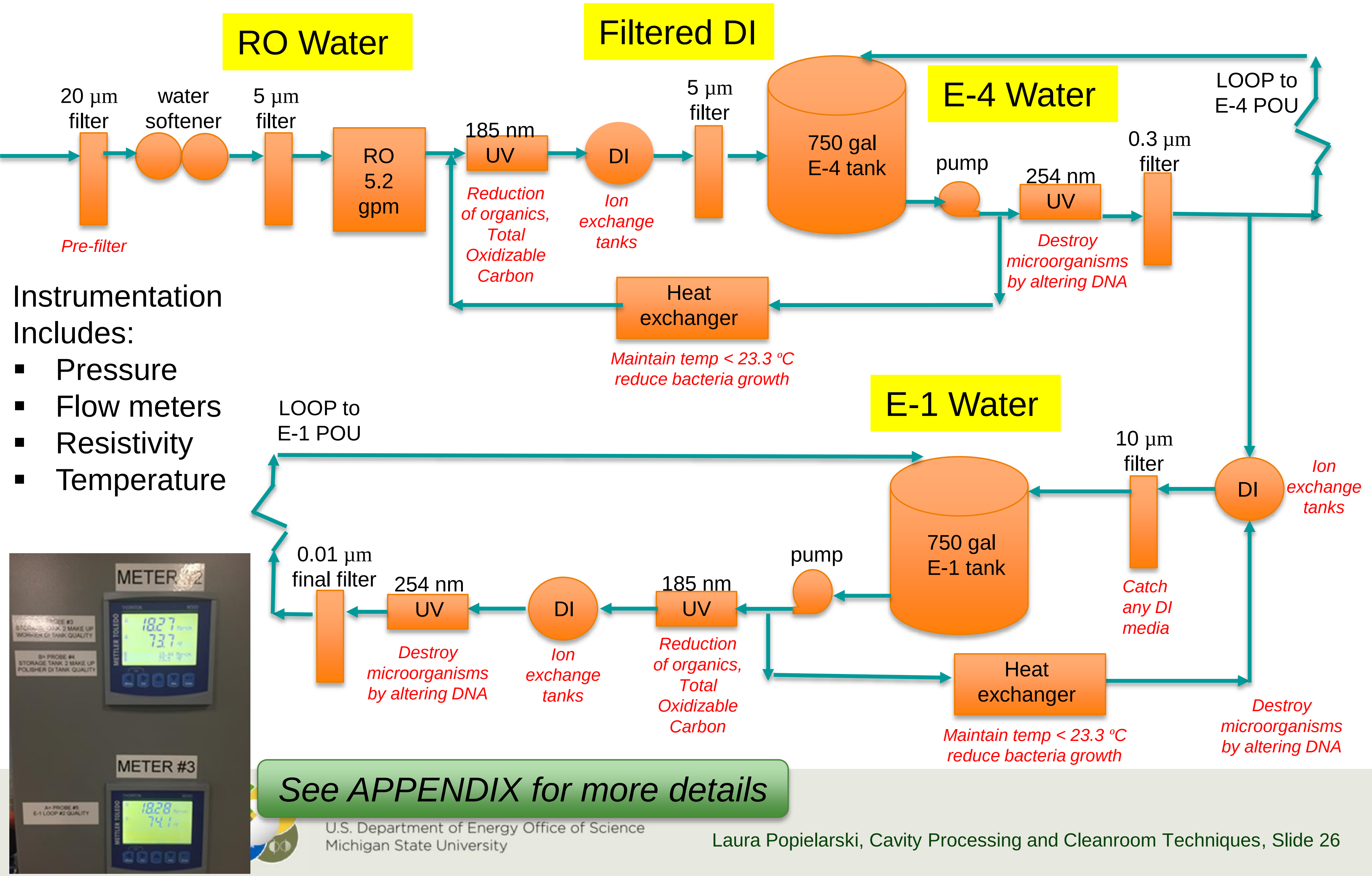
4. Ultra Pure Water and High Pressure Rinse

UPW System Design

Water System	RO make UP	Storage Size
R & D Pre Clean DI	0.15 (216 gpd)	150 gal
R & D UPW	0.5 gpm (720 gpd)	450 gal
Production UPW (E-1 & E-4)	5 gpm (7200 gpd)	750 + 750

- Key design criteria:
  - Water velocity 3-5 ft/s flow in continuous loop
  - Reduce dead legs and fittings, water should not stop flowing
  - Reduce total length of pipe travel by optimizing POU layout
  - Butt welded pipe with no seams best, zero-dead leg valves
  - Optimize RO water make-up rate and tank storage based on process needs

UPW System Process Diagram



Instrumentation Includes:

- Pressure
- Flow meters
- Resistivity
- Temperature

See APPENDIX for more details

UPW System Equipment

Reverse osmosis system

Water softener and brine tank

Virgin DI beds

185 nm UV sterilizer bulb units

254 nm UV sterilizer bulb unit →

Destroy microorganisms by altering DNA

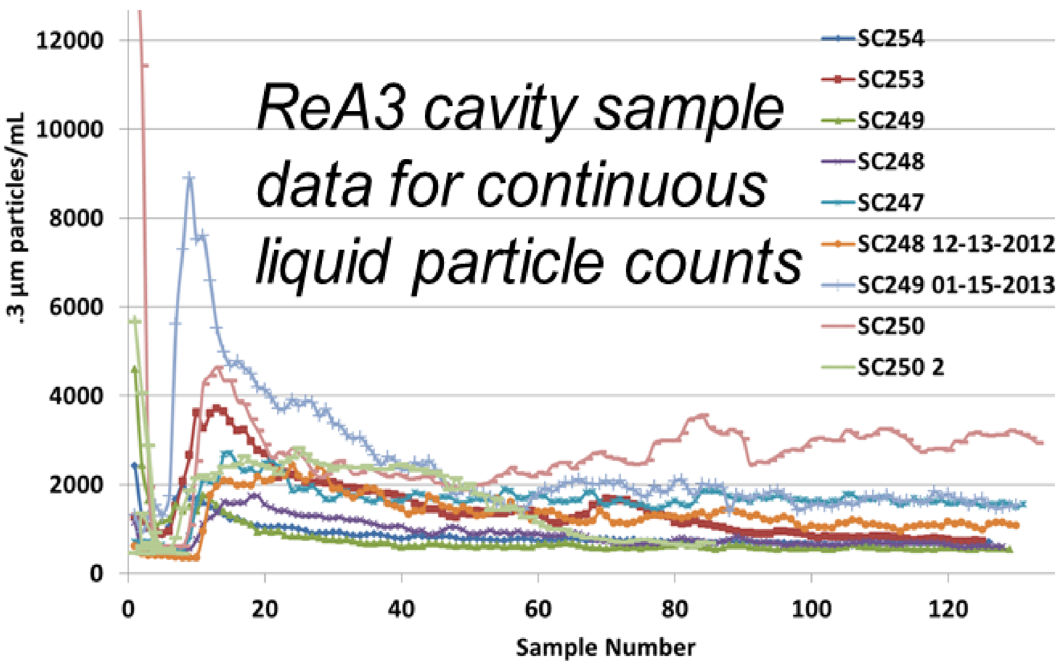
See APPENDIX for more details

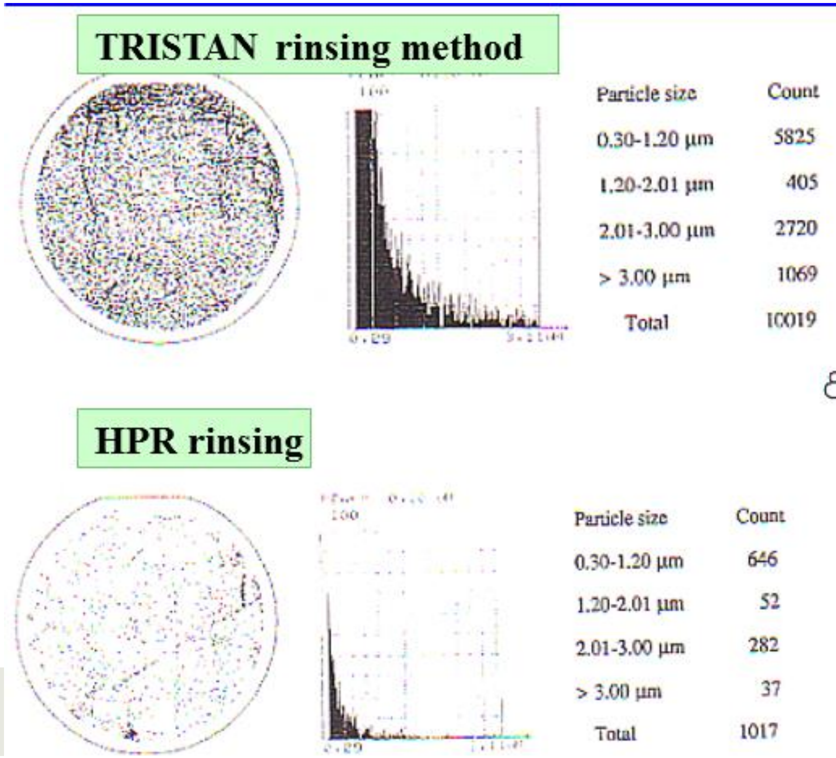
Reduction of organics, Total Oxidizable Carbon

← Sub-micron post filter 0.02 μm

Ultra Pure Water at High Pressure Used for Final Cleaning

- After bulk processing residual particulate must be removed
- High pressure rinse (HPR) application to SRF cavity developed by Peter Kneisel and Kenji Saito in 1993
- High pressure ultra pure water sprayed on all internal surfaces to knock off particulate

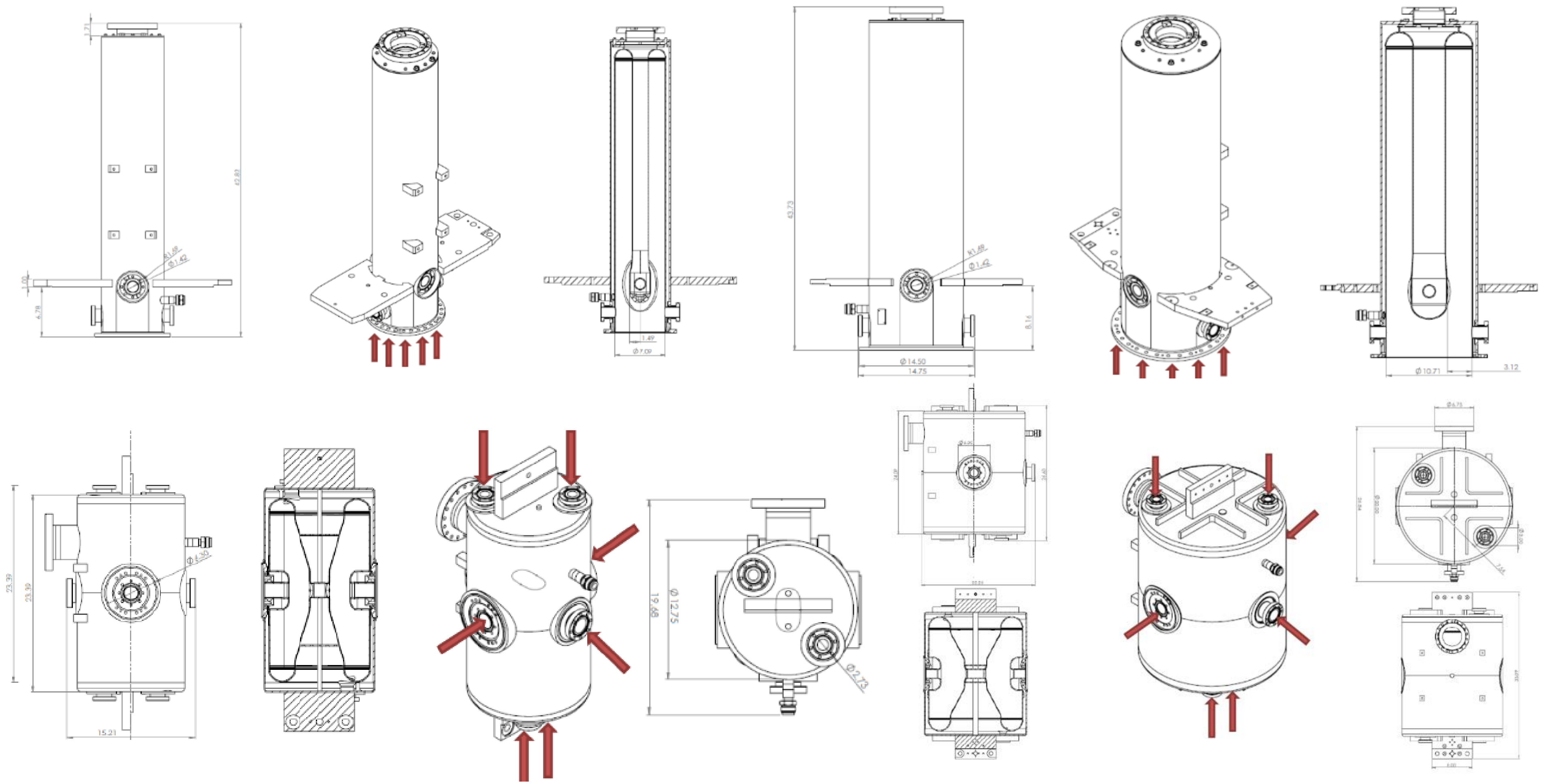




Kenji Saito, Lecture note in Tokyo University on May 2011

Access to Internal Surfaces for Cleaning

- Multiple cavity geometries require versatile high pressure rinse system
- Ports on HWR specifically designed for cleaning access

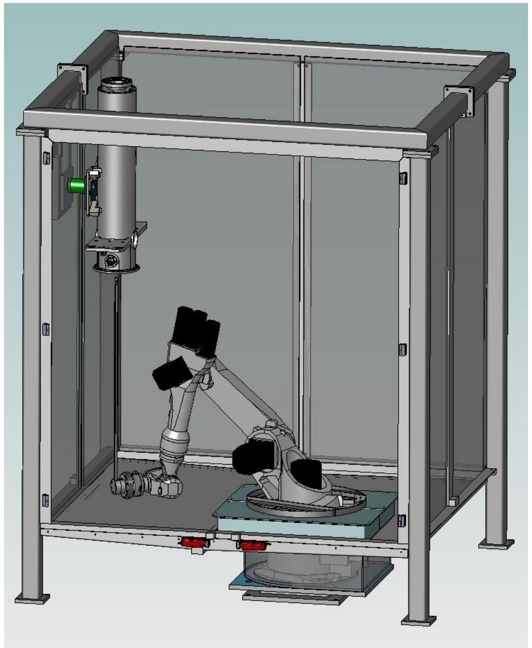


Rotation and Translation Methods

<u>SRF R & D 2000</u>
Nozzle stationary
Cavity moves up & down on table while spinning on rotating table

<u>NSCL ReA Project 2008</u>
Wand moves up and down on linear actuator
Cavity spinning on rotating table

- Reduce touch labor to cavity
- Reduce tooling
- Reduce cavity motion and moving



<u>FRIB Project 2015</u>
Wand moves up and down and oscillates on robotic arm
Cavity stationary

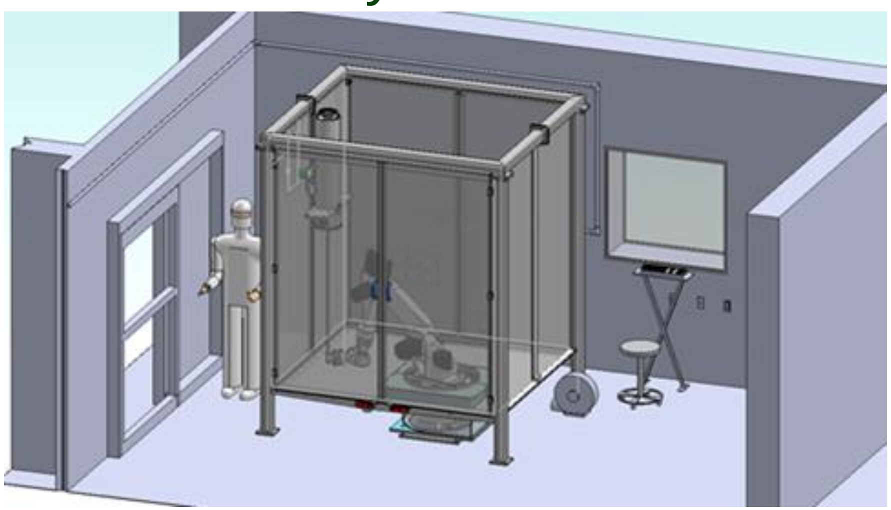
HPR Design Considerations and Cleaning Variables

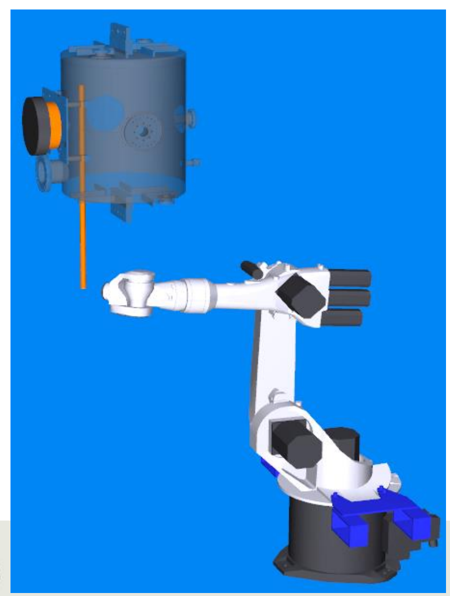
- 1000-1500 psi at point of use
- Pure gas over pressure
- Alignment
- Motion → rotation and translation, avoid spiral affect
- Materials → Cleanroom and E-1 water compatible, low friction
- Nozzle and jet design
- Duration→ Depends on cavity type, and surface area
- Post-Rinse → dry in ISO 5 cleanroom, away from all movement or people

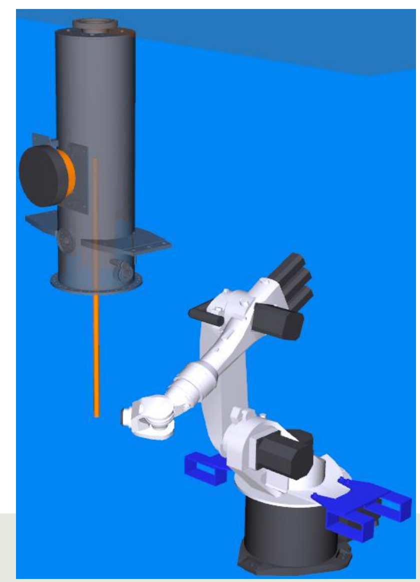
See APPENDIX for more details

Automated High Pressure Rinse Future for Multi-Port Cavities

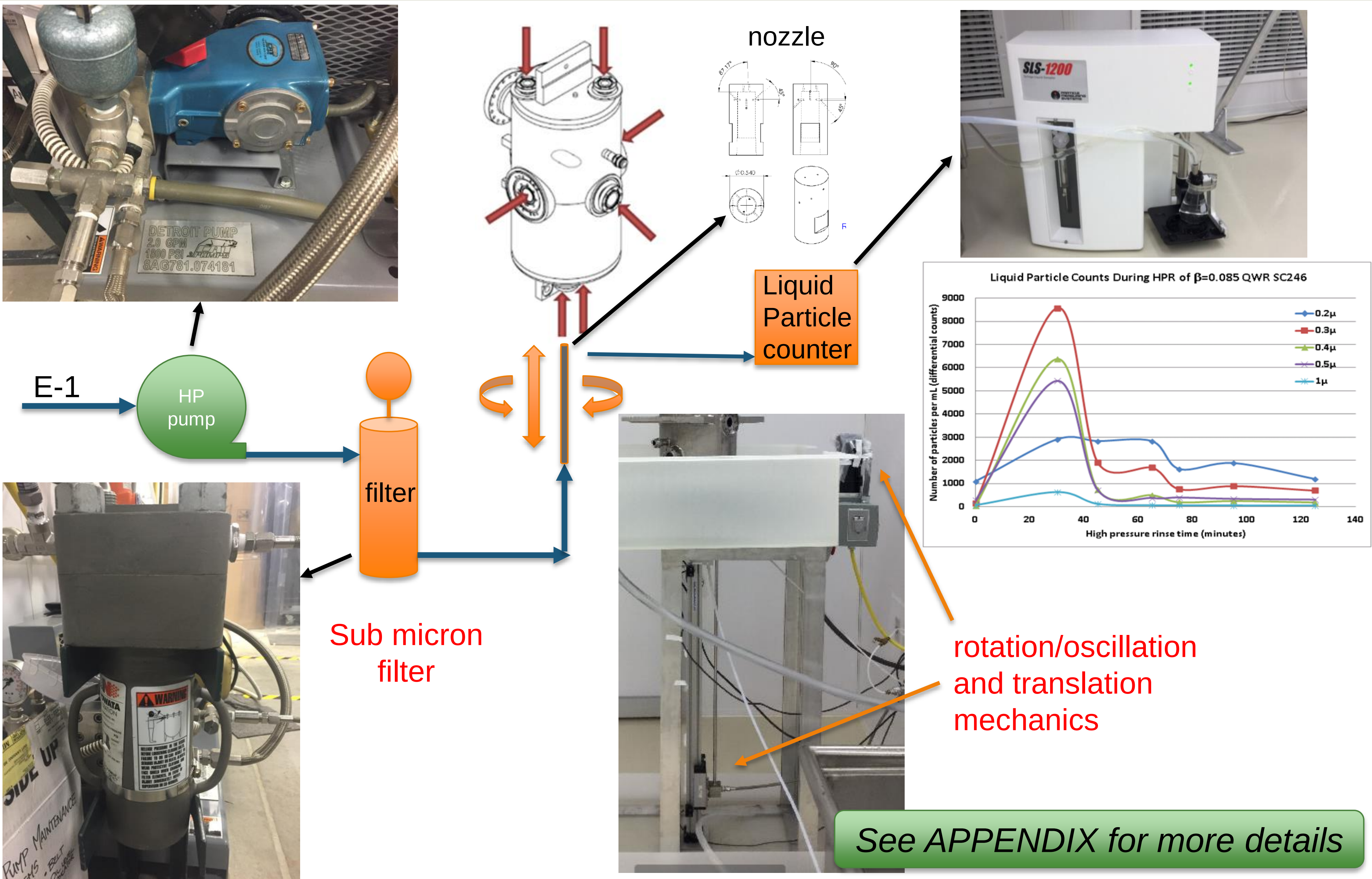
- Robotic high pressure rinsing tool is improvement over present techniques, reduce processing times and labor requirements
- Real inspection data coordinates uploaded to HPR HMI for individual serialized cavity for custom automated process
- Reduces chance of interference, drops operator touch labor and lowers risk to cavity contamination
- Automated system can be versatile for use with various geometries







Major Components for Effective High Pressure Rinse System

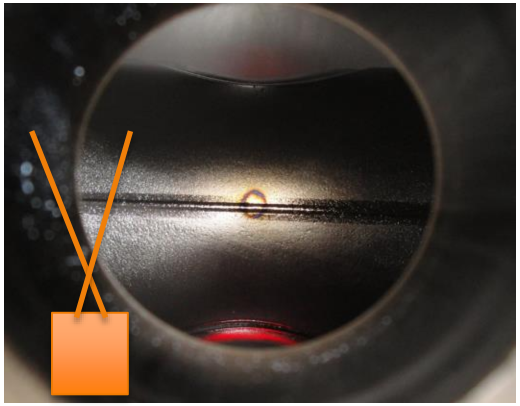


High Pressure Rinse Systems Around the World

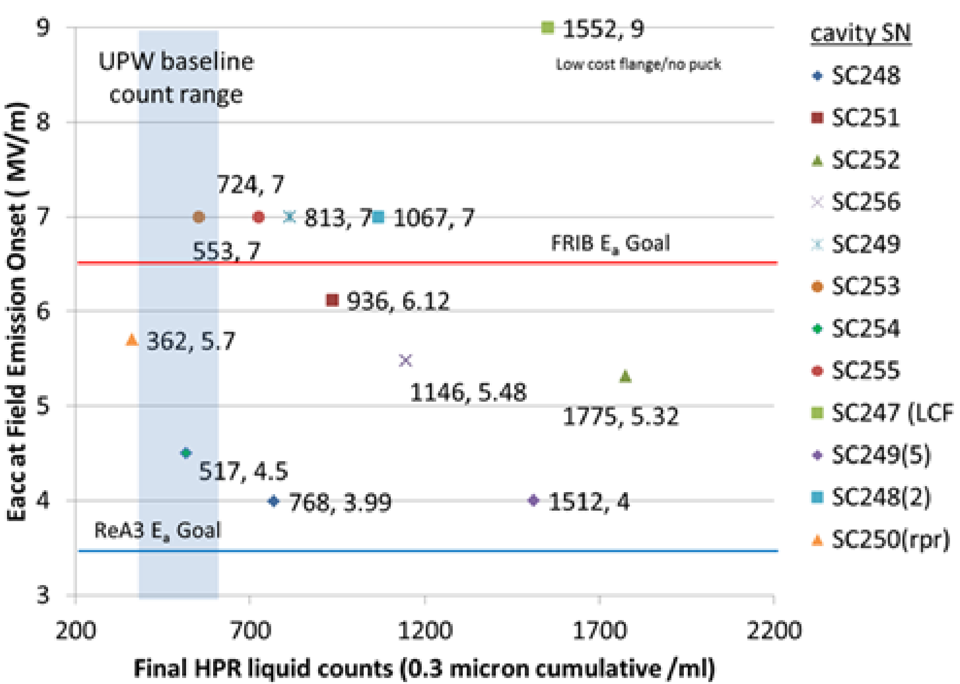
ANL's HPR - Courtesy of Michael Kelly

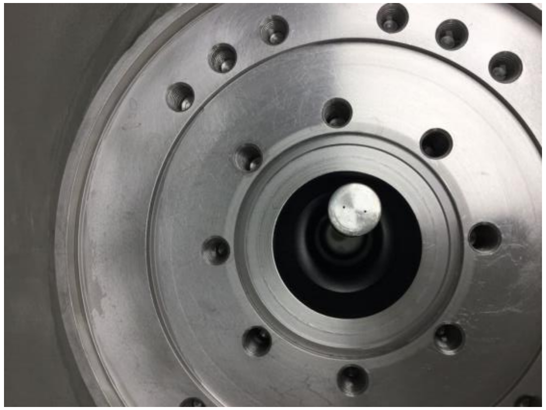
High Pressure Rinse Concerns

- Oxidation caused by force at surface, must make sure continuous motion
- Wand and nozzle alignment with ports
- Liquid particle counts not perfect indicator field emission onset, useful for relative cleanliness & rinsing complete
- Use dummy cavities for commissioning



Part rotating & wand translating but the two jets spraying from the top too close to IC & created small circle. Wand penetration was reduced.





Wand misalignment

5. Mechanical Surface Preparation

Surface Preparation
Bulk Damage Removal

- Visual blemishes and damaged layers of ~ 100-200 μm from Nb sheet fabrication skin passes

WELD SPATTER

SCRATCHES

INCLUSION

GOUGES

PITTING

- Damaged removed by mechanical abrasives and/or chemical reaction methods
- Mechanical Abrasives include:
  - Manual polishing (power tools, sandpaper, Scotch Brite ™)
  - Tumbling (Centrifugal Barrel Polishing)

- Chemical Reaction
  - Buffered Chemical Polishing (BCP)
  - Electropolishing (EP)

STUDY ON PARTICULATE RETENTION ON POLISHED NIOBIUM SURFACES AFTER BCP ETCHING*
I. Malloch#, C. Compton, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA

Mechanical Abrasion Concerns

- Abrasives cause extended degradation of the repaired region, even after etching
- Particulate contamination after using abrasives
- Consider methods/materials used
- Apply to smallest area
- Extended etching and high-pressure rinse cycles after repair
- Pursue less aggressive repair solutions

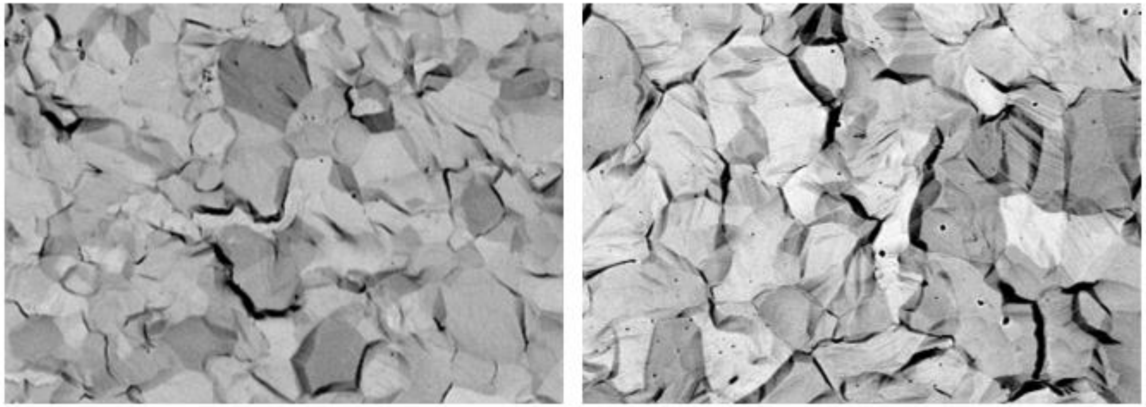


Figure 5. *SEM scans of an as-received sample (left) and an abraded sample (right) after more than 100 microns of etching. Note the unusually high concentration of black particulate spots on the abraded sample.*

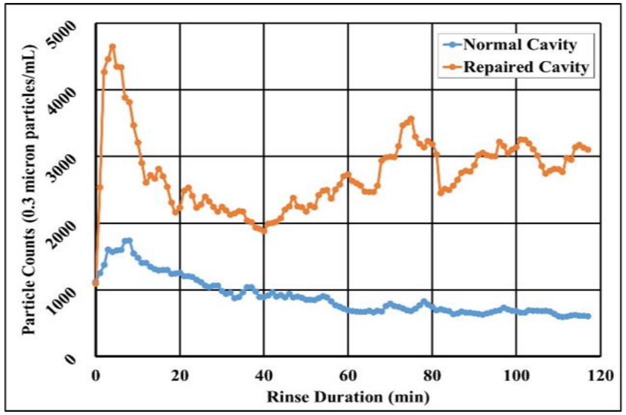


Figure 6. *Liquid particle counts as a function of high-pressure rinse time comparing a normal and repaired cavity.*

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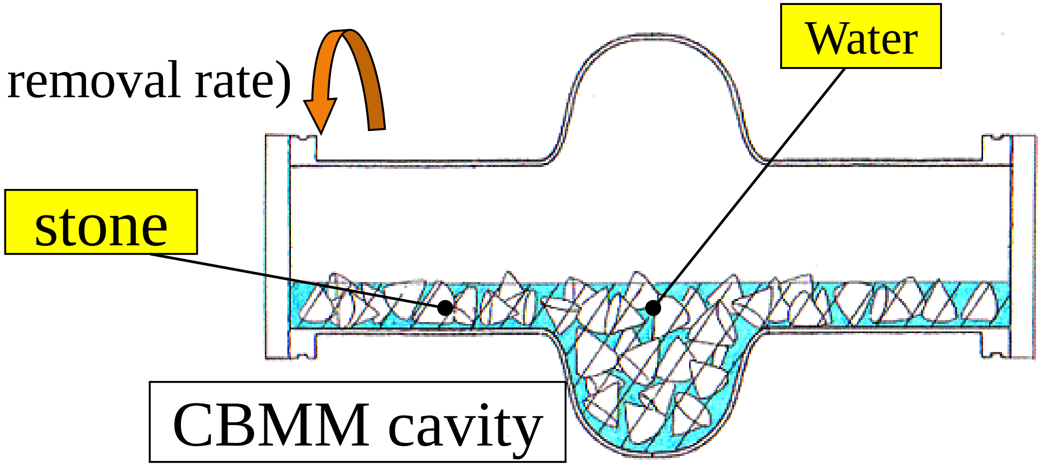
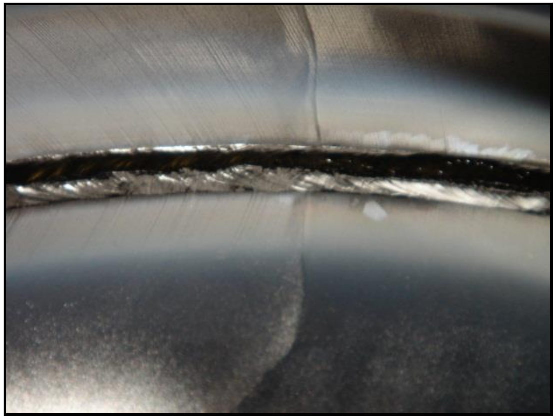
Centrifugal Barrel Polishing (CBP)

*Only has been used for elliptical cavities

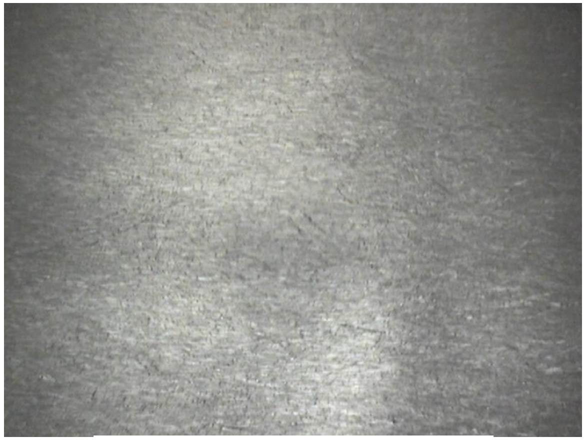
Rough stone : 5 times (15 microns/day removal rate)
Green stone : Once
Brown stone : Once
White stone : Once
Totally ~ 200 **μm removed @ equator**

Rotated ~100rpm on a oppositely rotating table

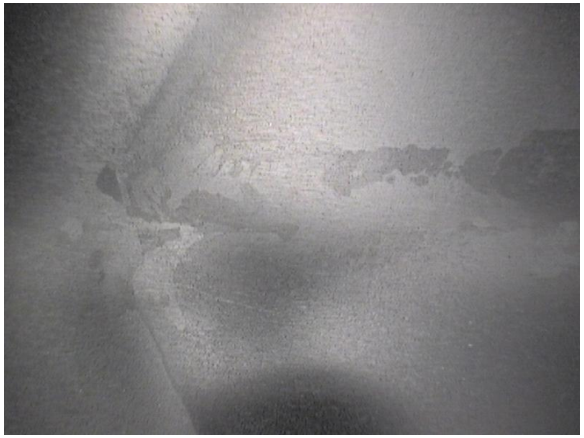




Before CBP (equator EBW seam)



After CBP



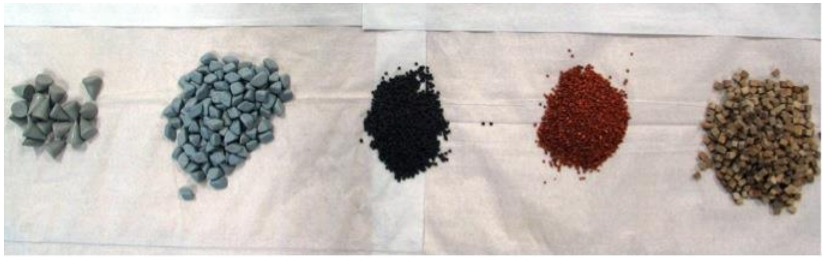
After light CP(10μm)

Kenji Saito, Lecture note in Tokyo University on May 2011

CBP at Other Labs

- Fermilab developed method of ultra-fine polishing for ILC cavities

The tumbling machine can hold two nine-cell accelerating cavities, rotating them up to 115 turns per minute. The rinsing device (right) washes the media out. Cavities must be absolutely free of any extraneous material after tumbling.



Medias are tumbled inside. The grey cones (far left) are a plastic with aluminum silicate, used for bulk removal. The powder blue media (second from left) are ceramic abrasives, useful as a first-pass media. A hardwood cut into small cubes (far right) is also a useful abrasive.

Mirror-like finish can be achieved

Fermilab

http://newsline.linearcollider.org/2011/03/03/tumbling-opens-possibilities/

6. Removal by Chemical Etching

Niobium Removal by Buffered Chemical Polish

- Standard acid etching mixture for niobium cavities is referred to as **buffered chemical polish or BCP**
- Chemical mixture reacts with metal surface to "ETCH" away layers of niobium
- Removal of 150 microns is optimum for RF performance

RECIPE- 1:1:2 acid mixture
1 part Hydrofluoric (HF) acid (49% w/w)
1 part Nitric (HNO_3) acid (70% w/w)
2 parts Phosphoric acid (85% w/w) [Buffer; not involved in reaction]

- The reactant HF is very **TOXIC ! → HF**
- The product gas is also **TOXIC !→ NO_2**
- **Some labs have used ratio of 1:1:1**

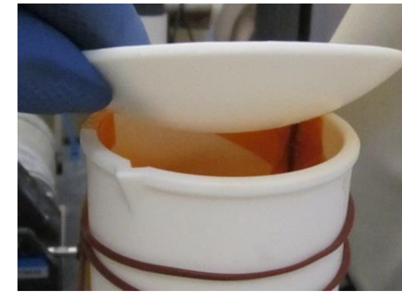
If there is BCP then there is HF! Safety is important!

NIOBIUM REACTION KINETICS: AN INVESTIGATION INTO THE REACTIONS BETWEEN BUFFERED CHEMICAL POLISH AND NIOBIUM AND THE IMPACT ON SRF CAVITY ETCHING*
I. Malloch, L. Dubbs, K. Elliott, R. Oweiss, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA

Chemical Reaction Mechanism

Nitric acid oxidizes the Nb

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



Brown NO_2 gas

HF reacts with Nb oxide

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

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

The reaction is exothermic !

HEAT OF REACTION RESULTS SUMMARY		
Average Heat of Reaction =	-607	kJ/mol
Standard Deviation =	17.6	kJ/mol
Theoretical Heat of Reaction =	-678.9	kJ/mol
Percent Error =		*10.5%*

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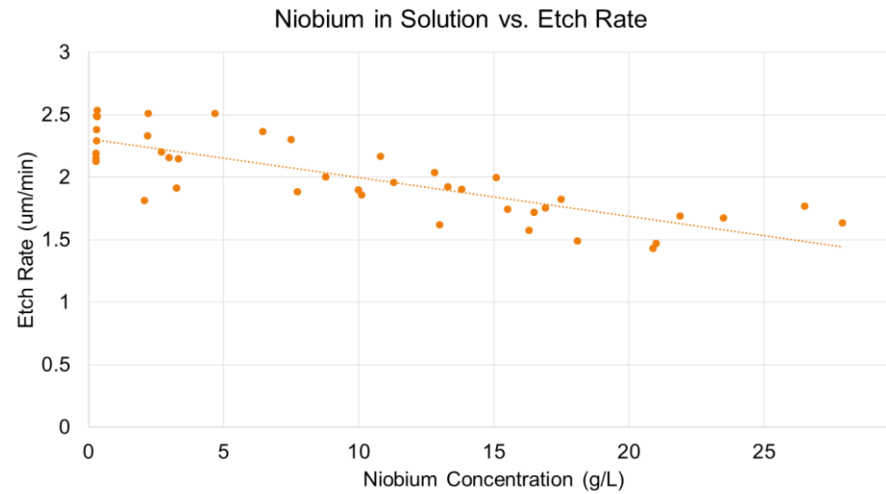
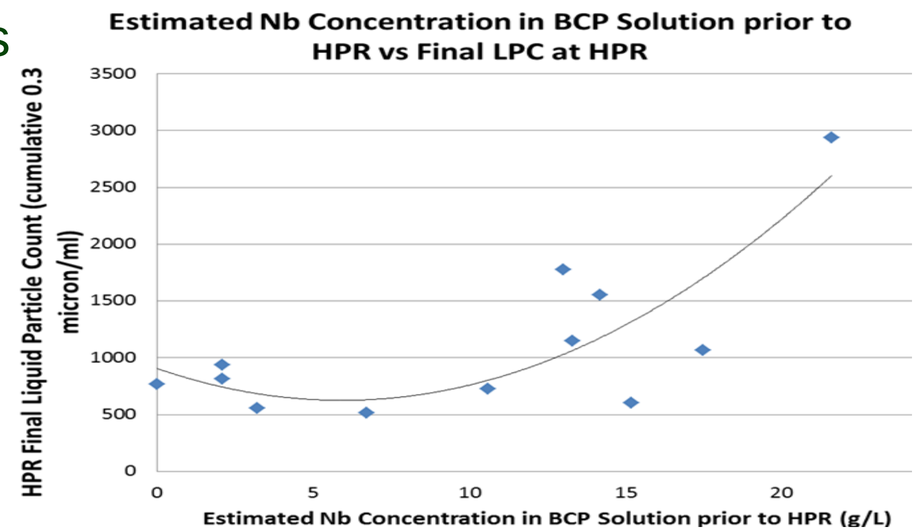
BCP Process Variables And Considerations

- Acid temperature 13-15 ºC to reduce hydrogen
- Acid flow rate ~ 5-10 gpm
- System pressure < 20 psi
- Ultra sonic thickness measurement (USTM) on bare cavities: understand etch rates and removal uniformity
- BCP etching very repeatable if variables kept constant
- Swap acid when concentration reaches 10-15 g Nb/L to optimize etch rate and decrease contamination

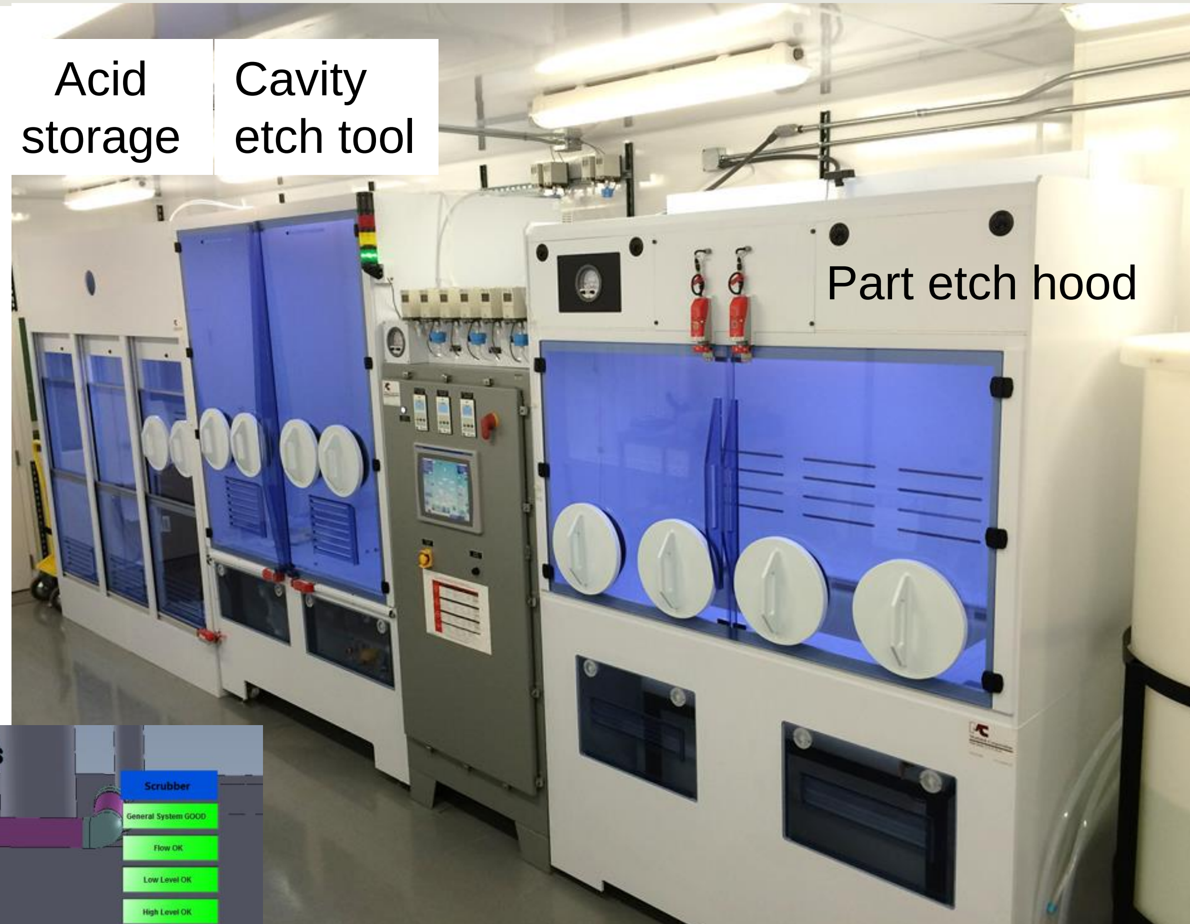
See APPENDIX for Design Considerations

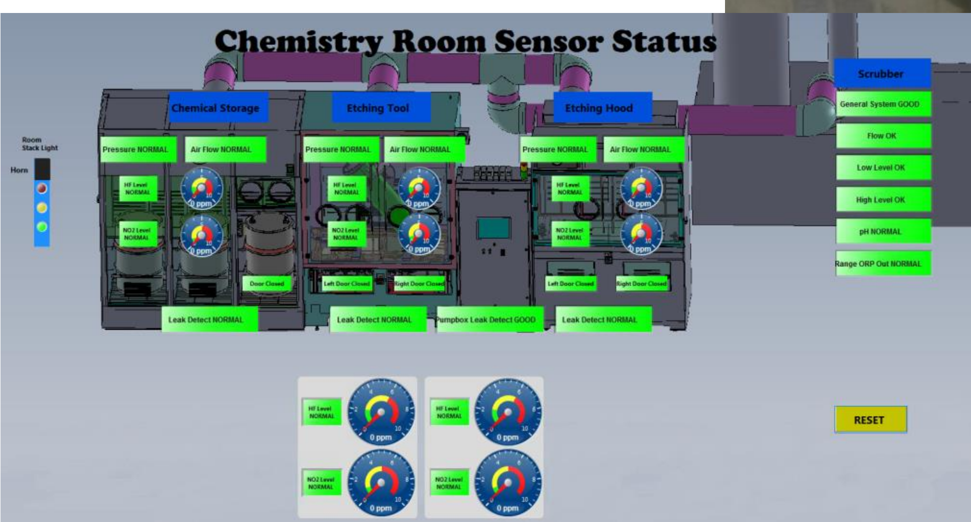




Automated Chemical Etching Facility for Production Cavities

- State-of-the-art chemical process equipment
- Safe and reliable, user friendly HMI
- Sophisticated controls, safety interlocks and alarms to eliminate exposure to BCP and toxic chemical vapors

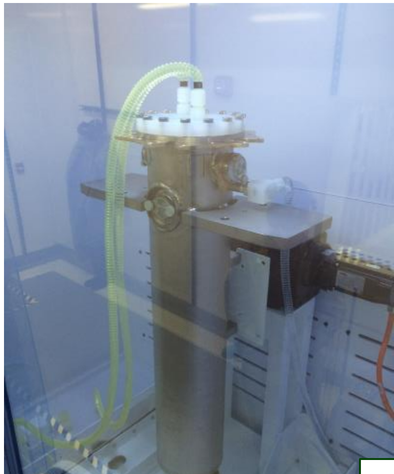




← Exterior monitors display all tool and facility alarm status.

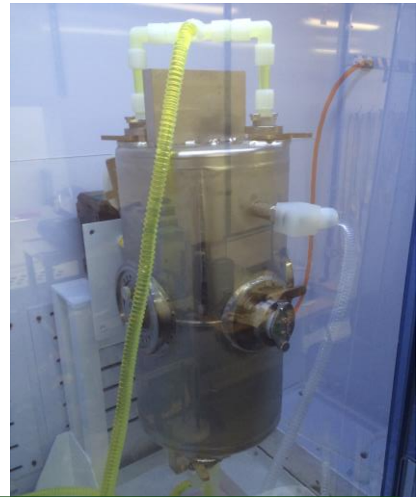
See APPENDIX for P&ID

Etching Configurations for Low Beta Cavities



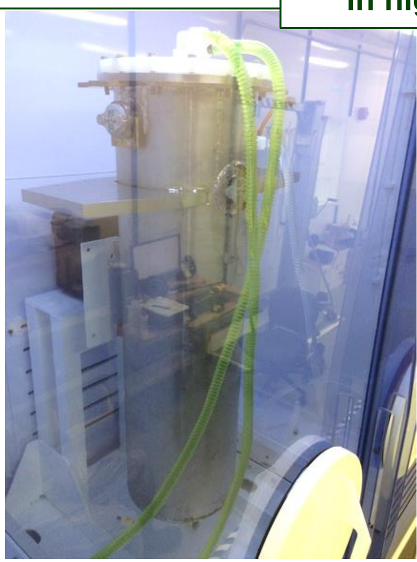
β=0.041 QWR

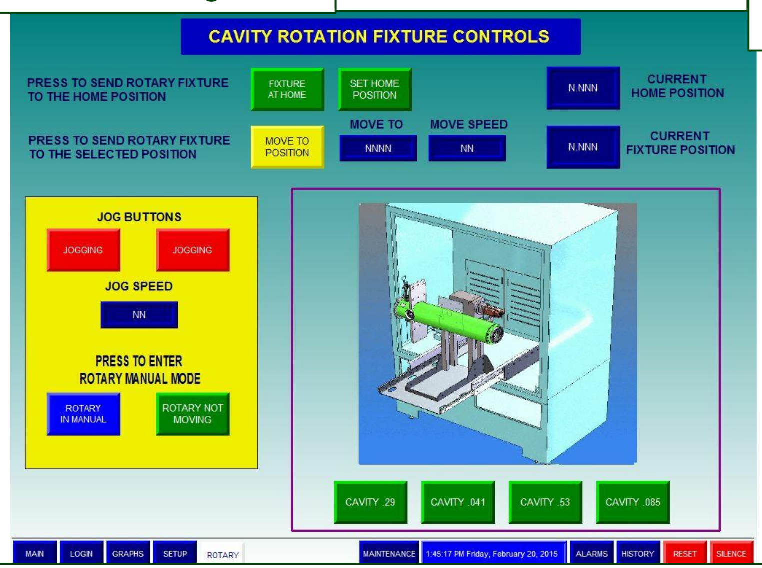
85001 tuned by custom etching in high electric field region



β=0.29 HWR

β=0.53 HWR





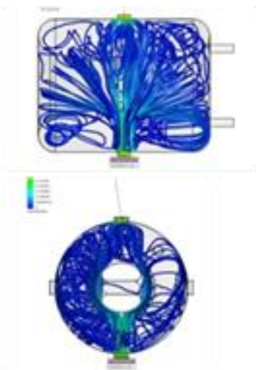
Tooling allows easy access for installation and rotation of all FRIB cavity types, rotation required for QWR etching and differential etching for tuning.

Interface Tooling is as Important as Equipment Design

- Tooling is for acid dispersion, sealing and masking areas
- Chemical input and return quills designed for optimum velocity profile to achieve near uniform removal→ Shape of cavity makes it difficult to achieve perfect uniformity
- Reduce high removal areas

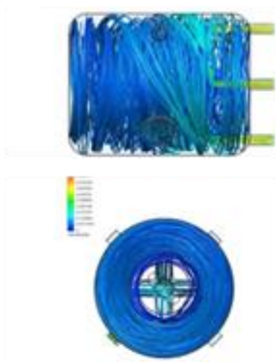
#4 PRODUCTION: Chemical input quills designed to optimize velocity profile and etch uniformity

#1 (baseline)



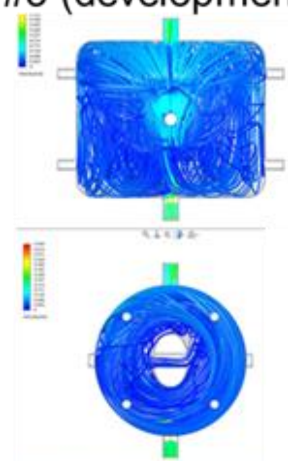
One acid inlet, poor mixing and uniformity

#2 (prototypes)

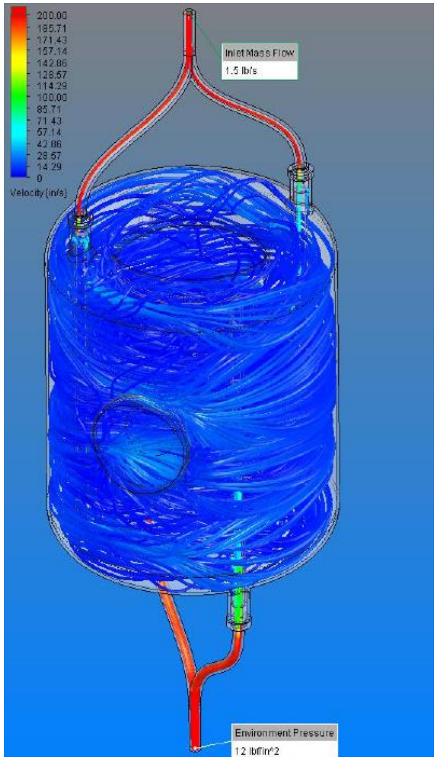


4 chemical wands disperse acid through cleaning ports to create mixing effect, improved uniformity

#3 (development)

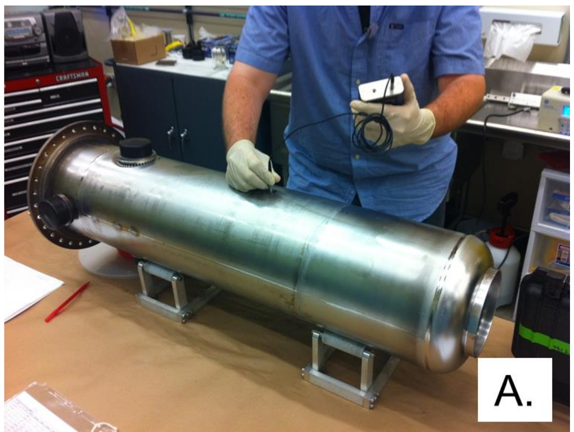


One chemical insertion wand simplifies assembly, while maintaining uniformity

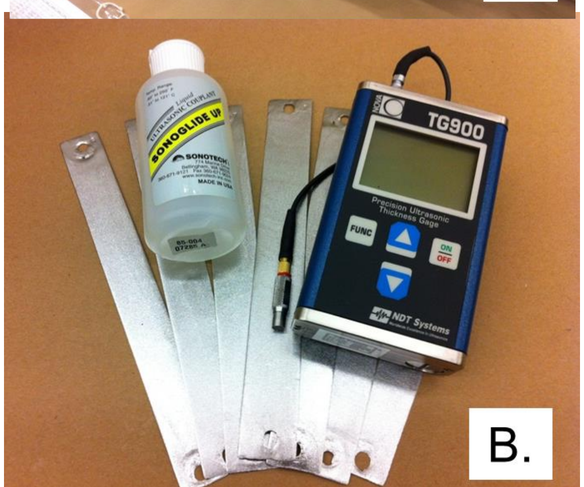


Etch Removal Visualization for β=0.53 HWR

USTM on undressed cavity to better understand the removal uniformity or variance.



A.

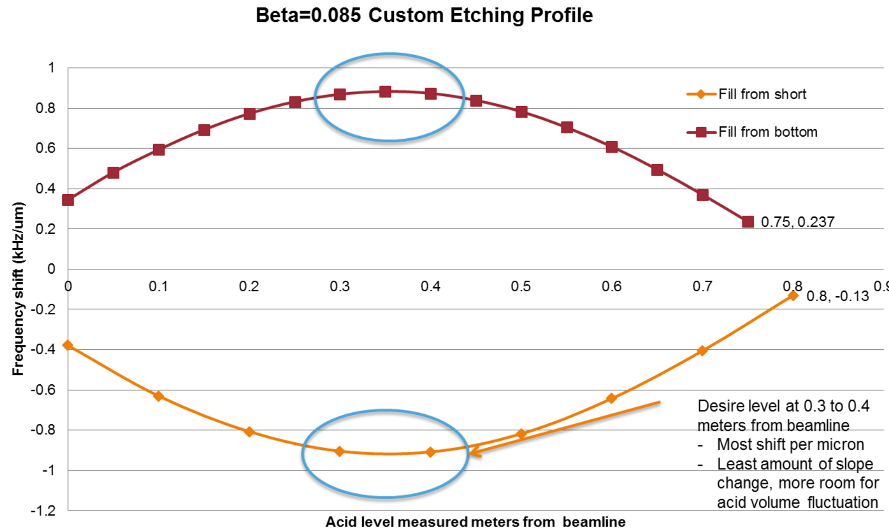


B.

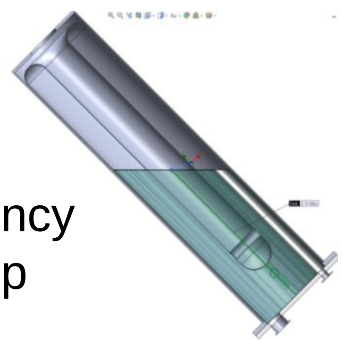
147	119	137	142	142	160	145	124	155	152	140	163	INNER CONDUCTOR
150	104	137	155	178	137	122	107	147	160	146	165	
145	112	135	145	147	152	132	114	160	160	173	157	
145	117	145	165	183	163	130	127	173	173	185	157	
152	131	119	168	183	147	160	127	180	193	201	166	RINSE PORT S.P.
157	145	147	188	X	140	155	135	170	180	152	150	
145	150	188	185	157	155	157	150	152	203	165	145	
140	122	175	188	107	157	152	127	152	173	86	150	OUTER CONDUCTOR
145	124	157	160	79	152	165	122	152	147	86	152	
160	170	180	157	64	170	193	185	191	152	122	157	
168		221	168	122	170	196		218	163	145	163	
196	BP1	185	168	RF2	188	236	BP2	180	163	RF1	152	
198		183	170		178	224		175	165		180	
163		211	168	74	168	188		213	168	130	165	
155	160	183	160	61	165	191	178	188	155	94	155	
147	124	163	152	81	150	160	127	160	152	74	152	
147	130	175	183	157	157	160	124	145	173	107	152	
155	150	188	183	168	142	152	152	160	175	163	152	FIDUCIAL SHORT PLATE
151	151	150	157	150	142	140	142	147	152	157	145	
168	114	130	173	173	163	150	145	135	180	183	170	
150	119	145	163	168	147	140	119	165	160	183	168	INNER CONDUCTOR
145	109	127	145	142	157	135	114	145	155	196	160	
142	107	124	137	152	150	127	107	140	152	163	147	
155	122	135	142	150	157	137	117	140	152	150	147	

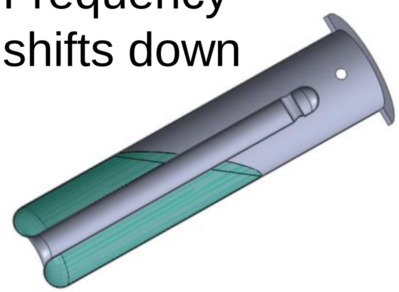
Differential Etching for Frequency Compensation

- Spot etch removal based on the electromagnetic design of the cavity
- Removal in the high E-field region removal yields upward frequency shift
- Removal in high magnetic field regions yields downward frequency shift

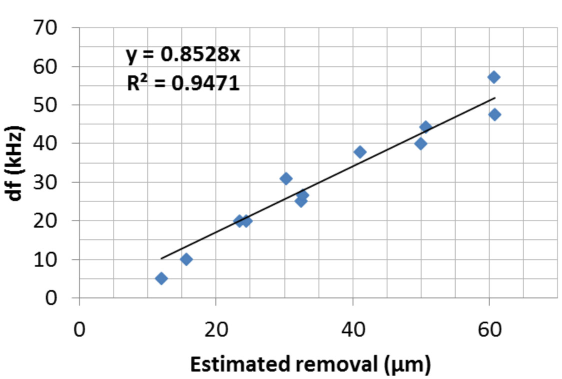


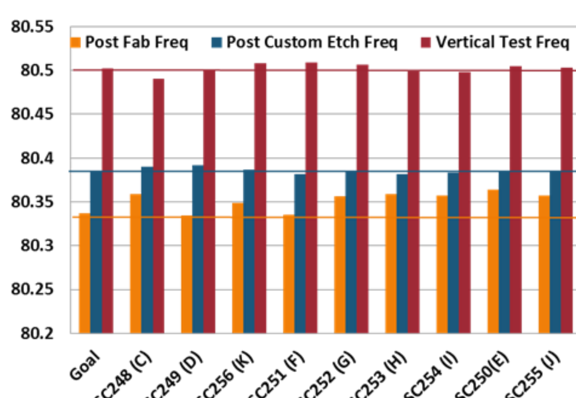
Frequency shifts down

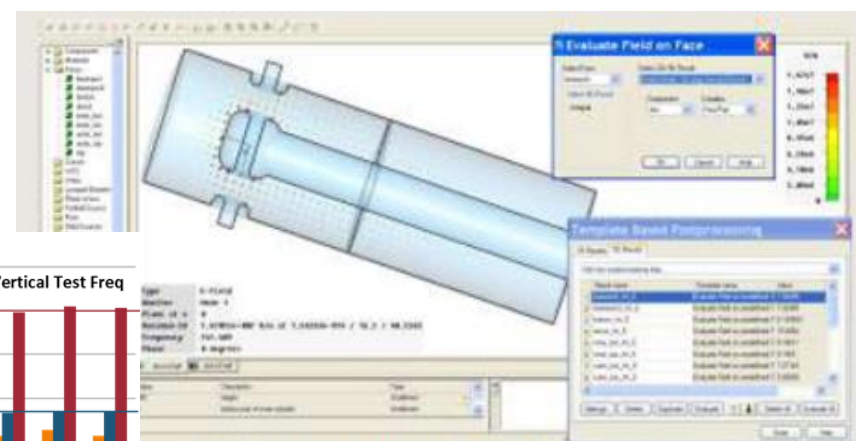




Frequency shifts up





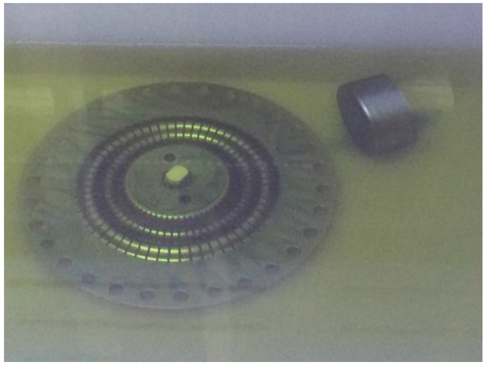


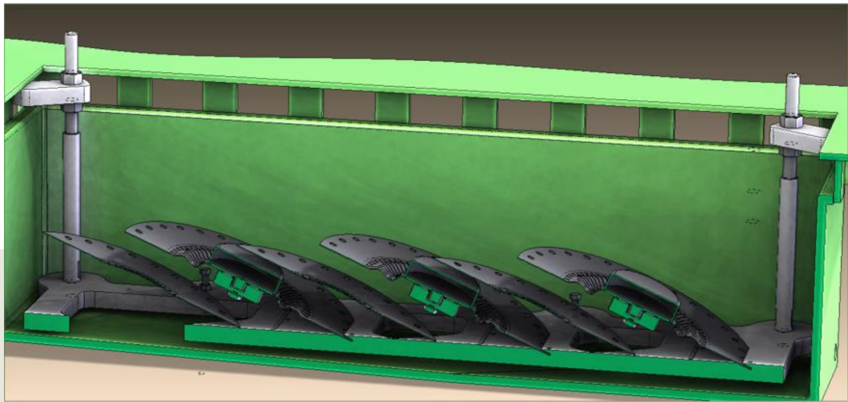
Differential etching for frequency compensation
Differential sensitivity for ½
2 kHz/um for

TRIUMF

Niobium Part Etching

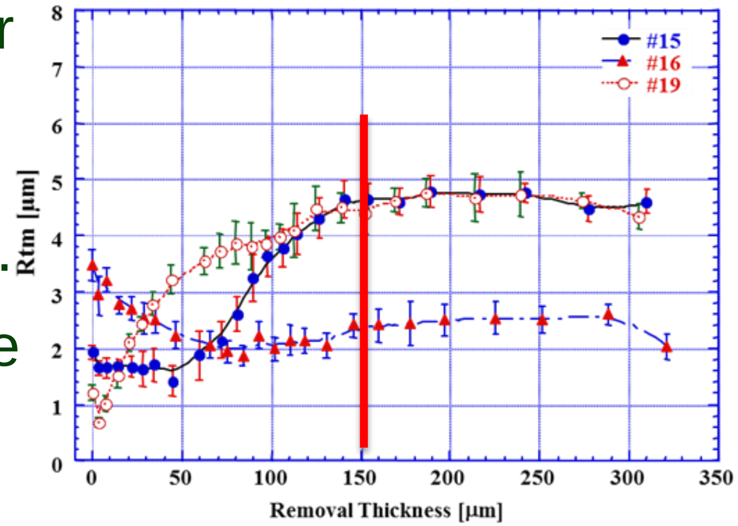
- Parts are etched with BCP for:
  - Preparing subcomponents for electron beam welding
  - QWR tuning plate assemblies
  - Material R & D samples
- Cold acid ~ 13-15 ° C
- Automatic BCP fill and drain and UPW fill and drain rinse cycles in closed ventilated hood.
  - Batch etching in tank
  - Wiping with polyester cloths or swabs
- Mask non-niobium components
- Fixture parts:
  - to allow gas bubbles to escape
  - And keep from touching each other

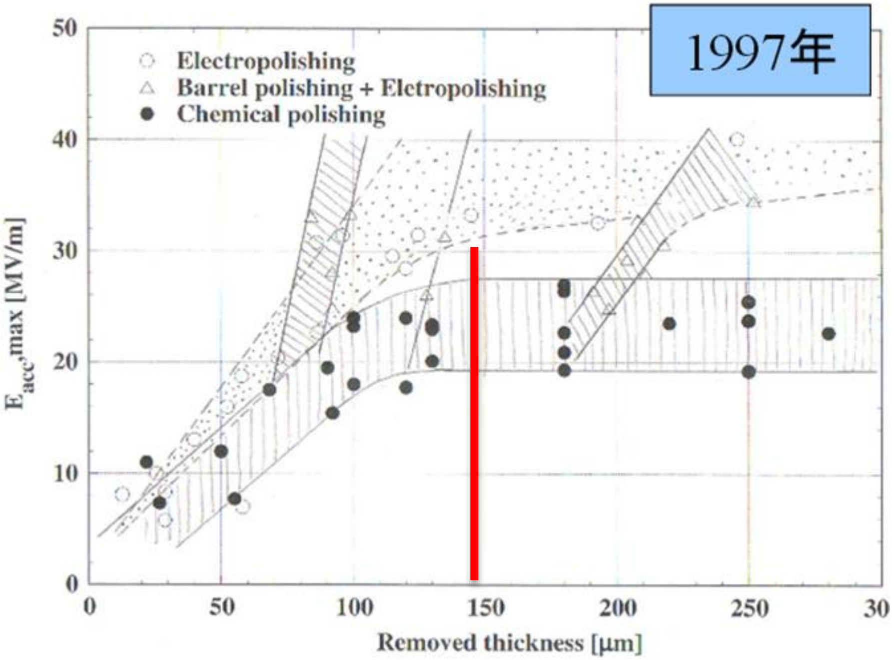




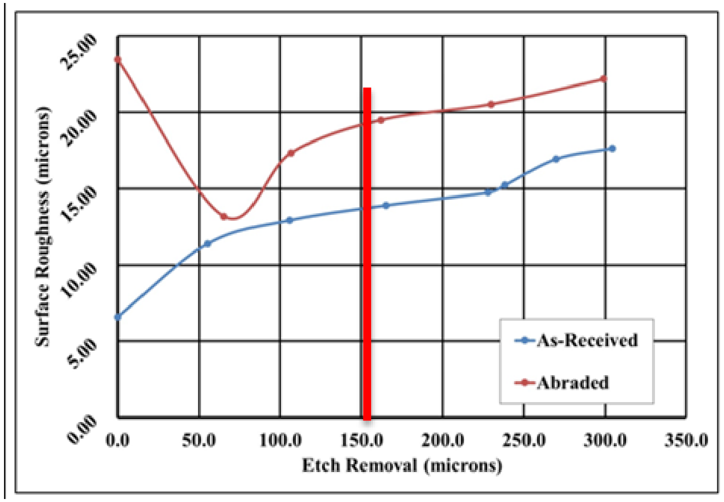
Why 150-200 microns?

- Shown that 150-200 microns is optimum for cavity performance
- If remove too much the material could become thin and affect mechanical stability.
- Additional etching does not improve surface roughness





Kenji Saito, Lecture note in Tokyo University on May 2011

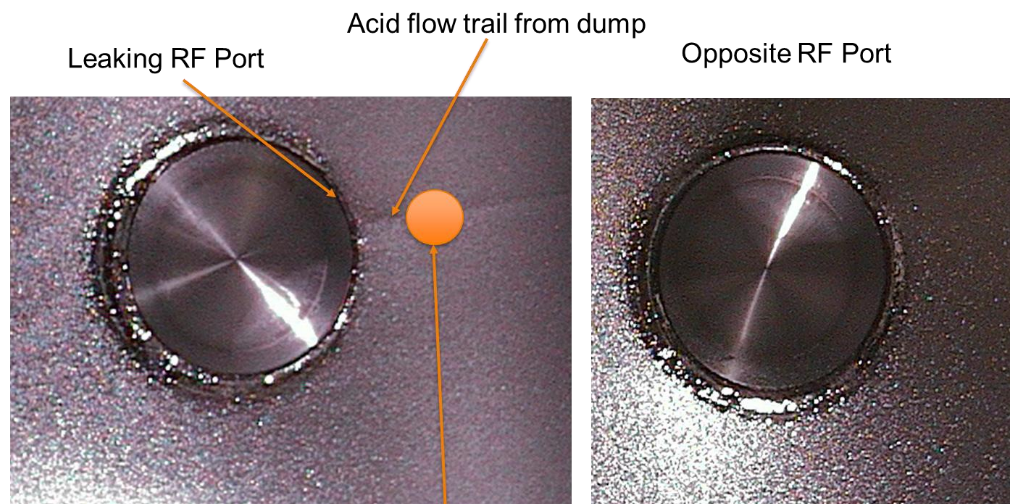


STUDY ON PARTICULATE RETENTION ON POLISHED NIOBIUM SURFACES AFTER BCP ETCHING*
I. Malloch#, C. Compton, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA

BCP Concerns and Remedies

- Vapor marks →
  - allow gas to escape by agitation or rotation
- High removal areas → tool design
  - Material thinning & mechanical stability.
  - Etch through poor quality welds
  - Inspect after etching!
- Streaking →
  - From slow drain or dump
  - Goal for fast even drain
- Other mixtures of acid →
  - Tried around the world, to slow etch removal, slow down the reaction to avoid hydrogen uptake, less hazardous (V. Palmieri)

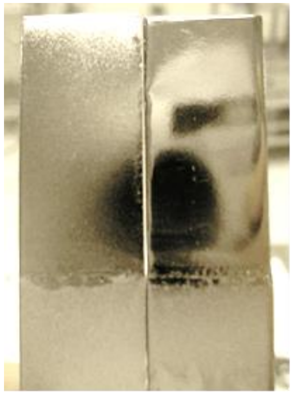
Vapor and gas build up in ports



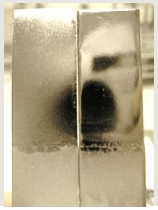
1.656 mm thickness measured with USTM after last differential etch (~425 microns estimated removal). Initial thicknes was 2.075 mm

BESIDES THE STANDARD NIOBIUM BATH CHEMICAL POLISHING
V. Palmieri, F. Stivanello, S.Yu. Stark, INFN – LNL, Legnaro (Padua), ITALY C. Roncolato, INFM – Research Unit of Padua, Padua, ITALY, M. Valentino, INFM – Research Unit of Naples, Naples, ITAL

BCP Tools at Other Labs



7. Preparation by Electropolishing



Electropolishing (EP) Niobium

- EP applied to reduce surface roughness and create smoother surface
- Cleaner because easier to remove particulate and less field emitters

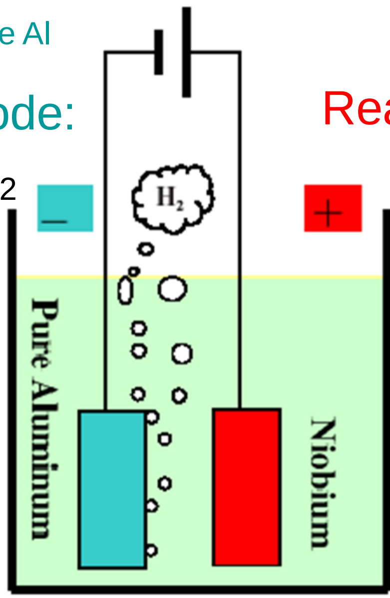
Overall: $2Nb + 10HF + 2H_2O \rightarrow 2H_2NbOF_5 + 5H_2 \uparrow$

Al-1350-O EC, 99.5% pure Al

Reaction at cathode:

$2H^+ + 2e^- \rightarrow H_2$

Hydrogen gas produced



Reactions at anode:

$2Nb + 5H_2O \rightarrow Nb_2O_5 + 10H+ + 10e-$

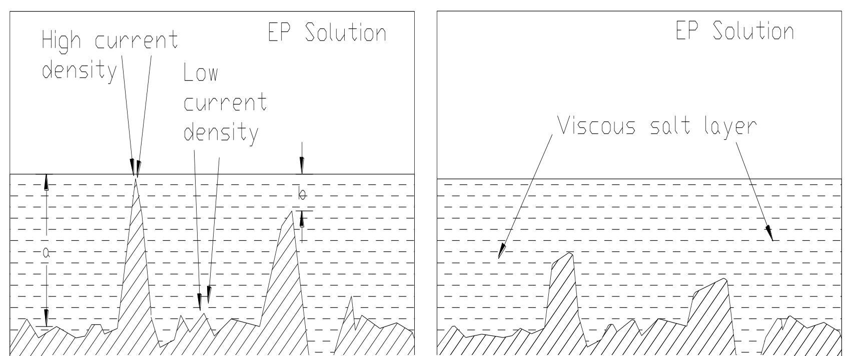
$2H_20 \rightarrow O2 + 4H+ + 4e-$

- Applied current creates niobium oxide
- HF reacts with Nb oxide

Acid Mixture:

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

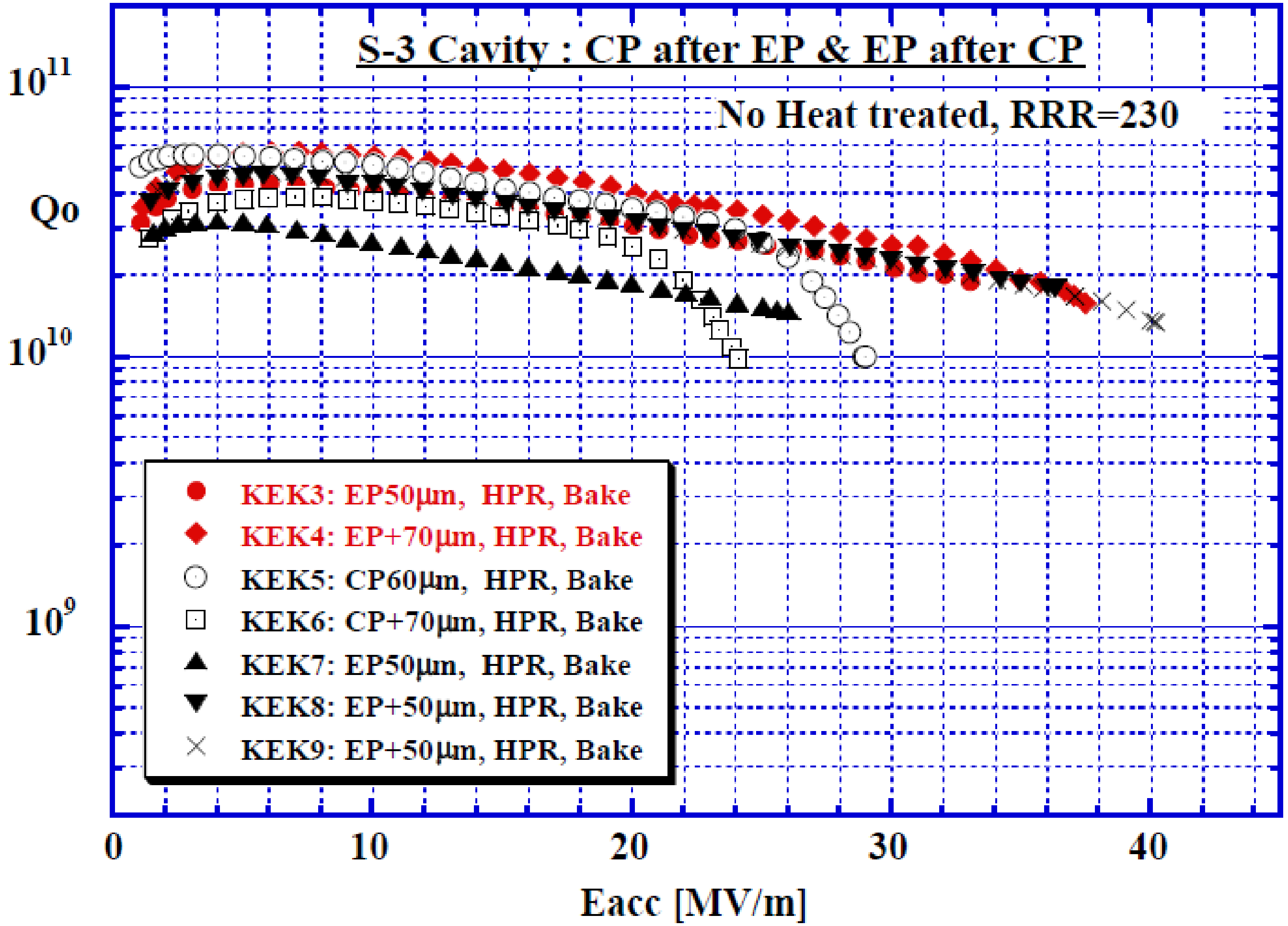
Sulfuric acid is source of free ions



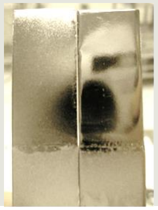
Preferentially polishes higher points

Kenji Saito, Lecture note in Tokyo University on May 2011

Evidence of the Superiority of EP over BCP with High Gradient Performance

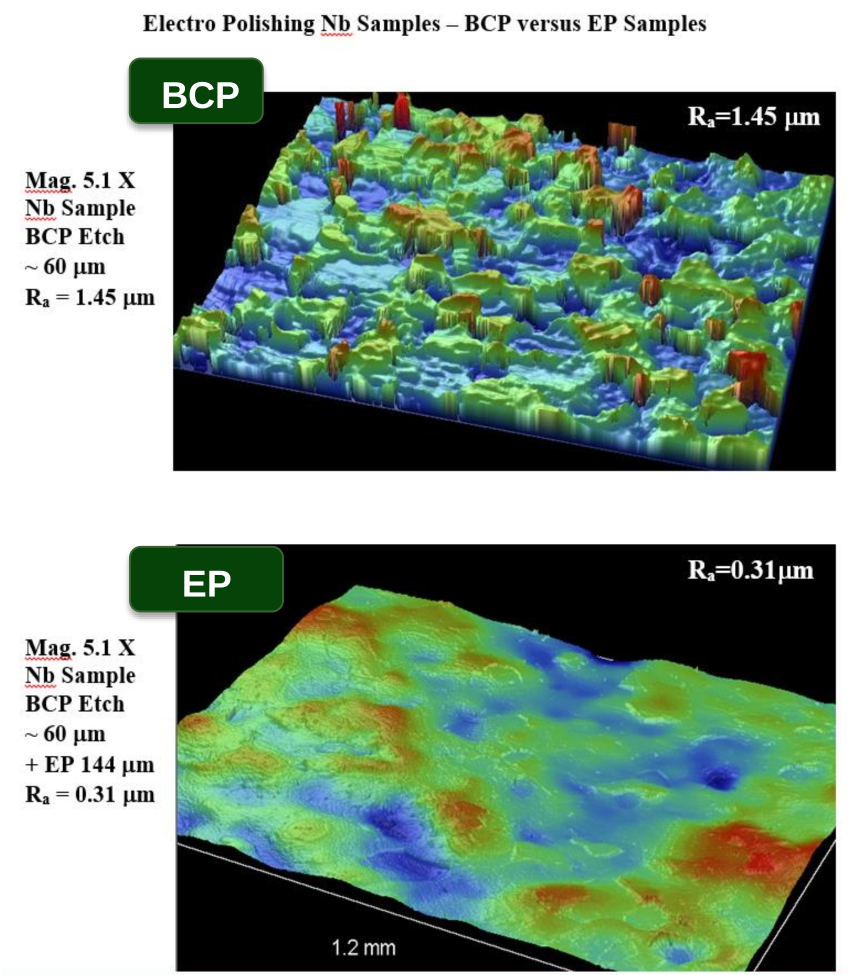


Kako, E and Saito, K. "Development of Electropolishing Technology for Superconducting Cavities", PAC 2003

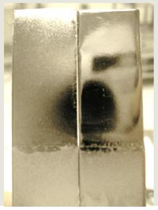


Some Comments on EP

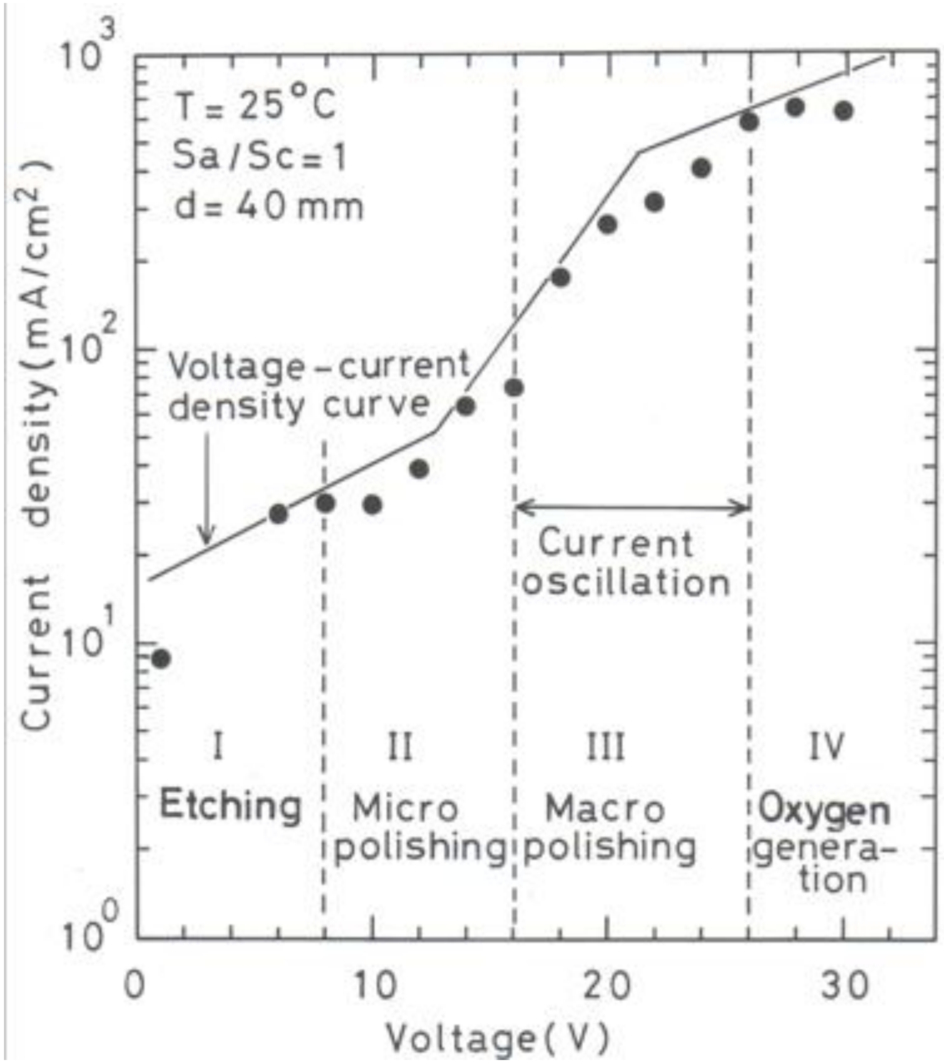
- **On EP samples roughness drops below 1 um after 150 um of material removed** (L. Lilje, Improved Surface Treatment of SC TESLA Cavities)
- **The main difference between BCP and EP is the smoothening of the grain boundaries**
- Increases gradients, up to 40 MV/m, fundamental limit [6]
- Decreases Q-slope appearance

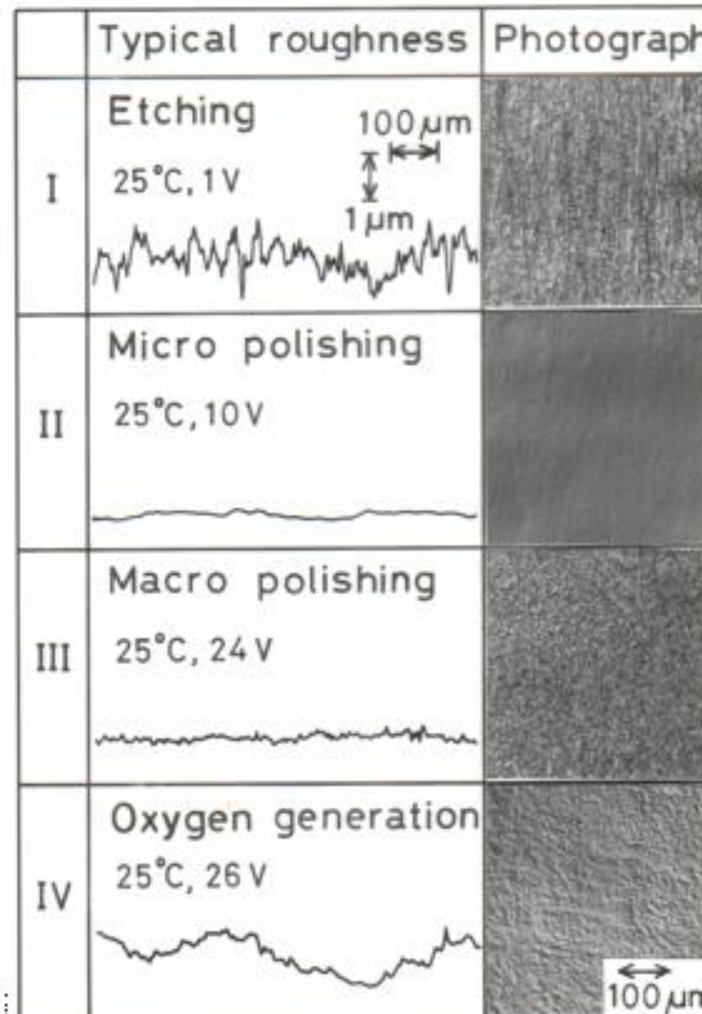


See APPENDIX for more details



Electropolishing Characteristics With Nb





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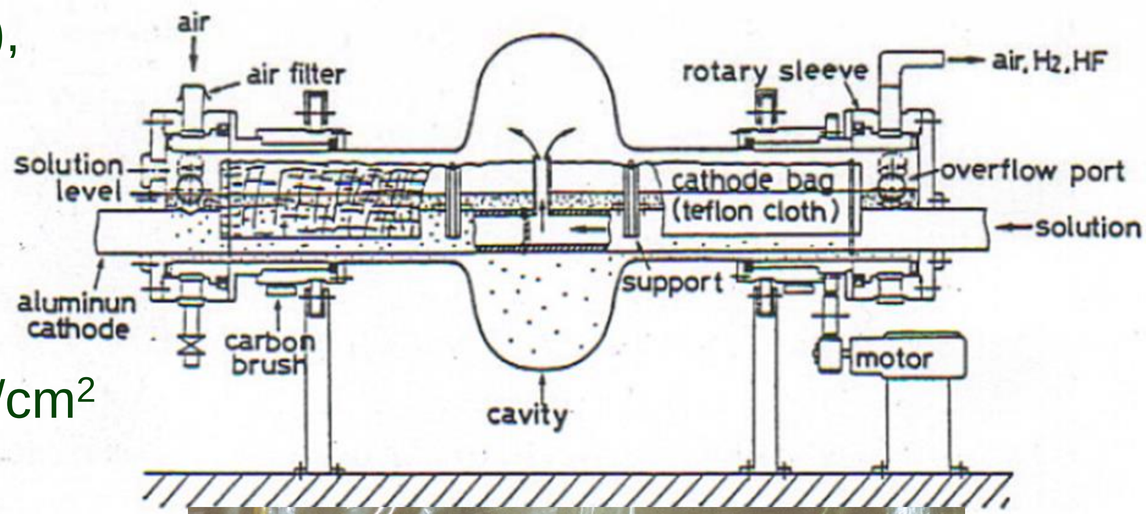
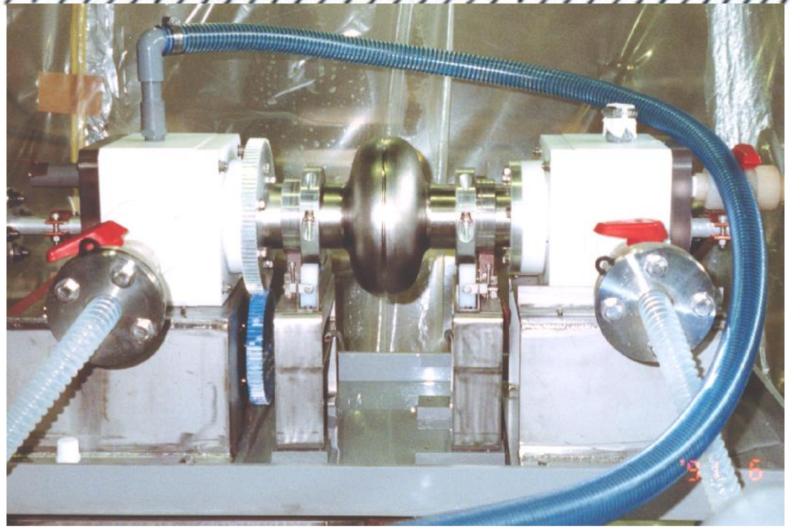
EP General Process Constraints

- **EP Solution:** 1 part HF (48%), 9 parts H_2SO_4 (98%) [1]
- **Temperature:** 20-40 °C
- **Voltage:** 10-25 V, depends on bath temperature [7]
- **Oscillation :** 0.1-0.3 Hz [2]
- **Current density**: 10 – 50 mA/cm^2
- **Acid flowrate:** 60 l/min [5]
  - depends on cavity surface area [6]
- **RPM:** 0.4 – 1 rpm [5], 1-9 rpm [7]
- **Etch rate:** ~0.5 μm/min
- Cathode and cavity only metallic
  - parts in contact with acid [7]
- Ability to dilute hydrogen gas

See APPENDIX for more details





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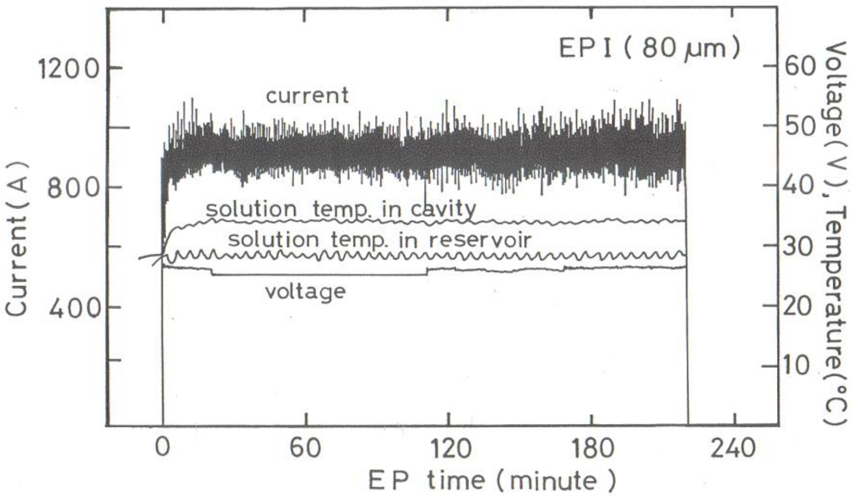
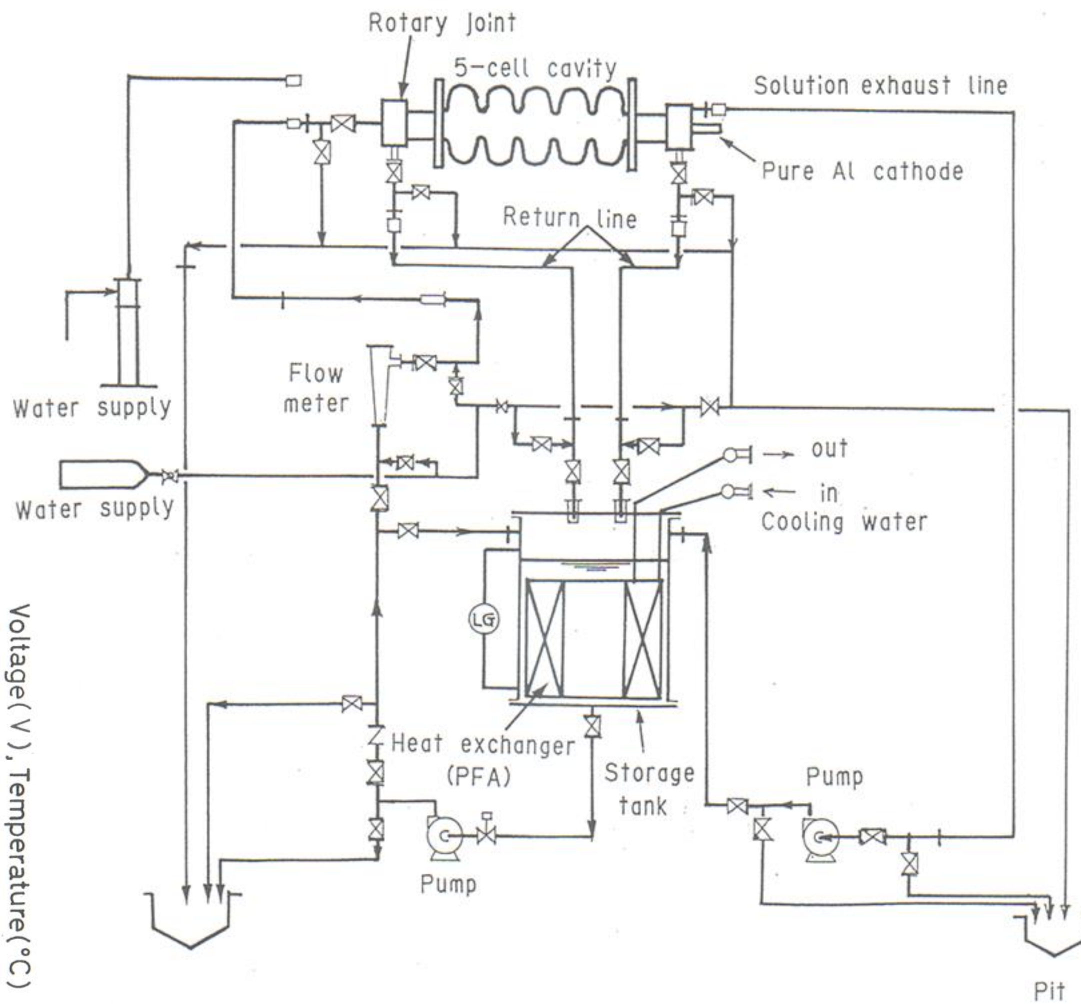
EP Process Diagram





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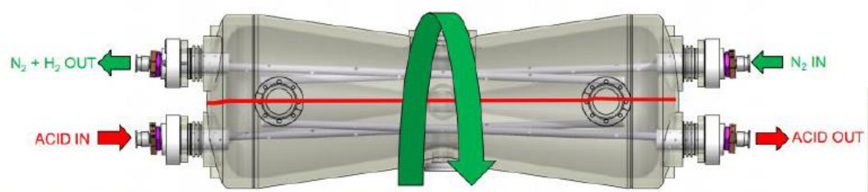
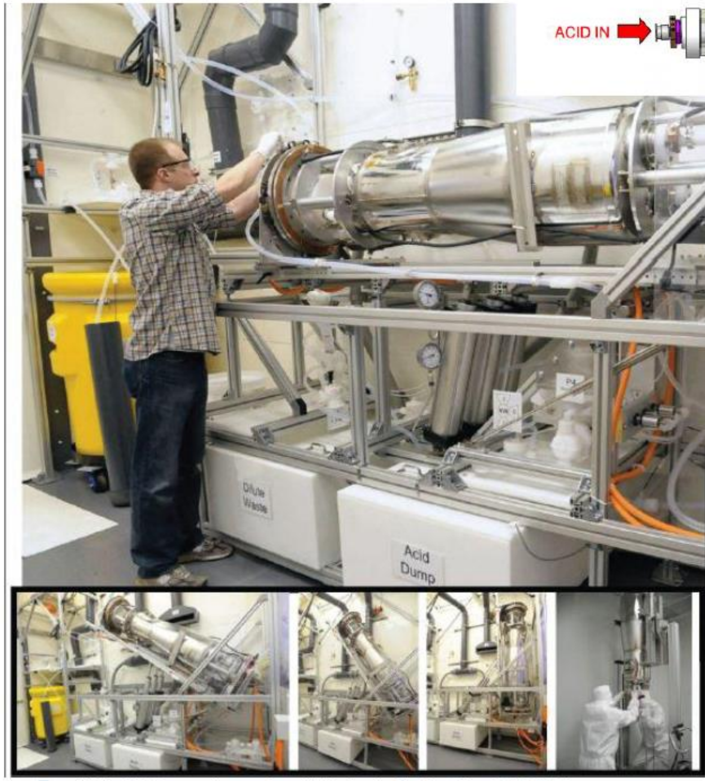
TRISTAN 508MHz 5-cell cavity

Half Wave Resonator at ANL

Electro-Polishing

Argonne NATIONAL LABORATORY



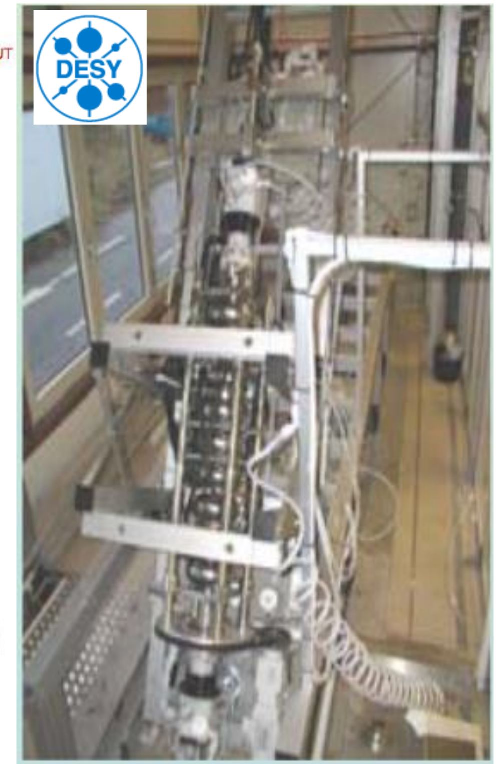


Superconducting RF Cavities Development at Argonne National Laboratory
Sang-hoon Kim on behalf of Linac Development Group in Physics Division at Argonne National Laboratory
May 10, 2014

BEFORE EP

AFTER 12HRS OF EP
150µm Nb REMOVED

AKPA - IBS Symposium on Special Topics in Physics, May 9-10, 2014

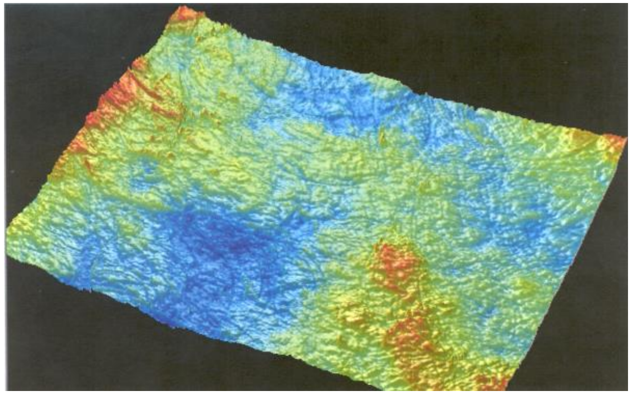


Cavity assembled to EP stand

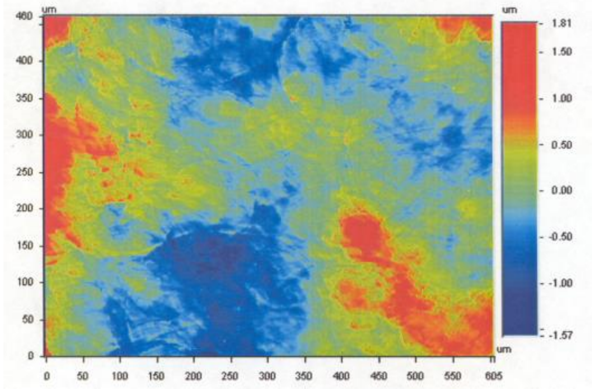
Hydrogen Produced During EP

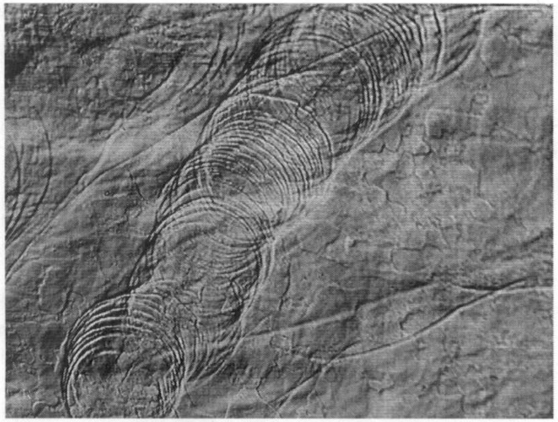
- PTFE mesh or bag used to cover the cathode to break and reduce hydrogen bubbles from reaching anode surface

10.2 X mag. 3-D Optical – 100+ μm EP



10.2 X mag. 2-D Optical – 100+ μm EP





Bubble traces – “Basic Studies for the Electro Polishing Facility at DESY”, N. Steinhau-Kuhl

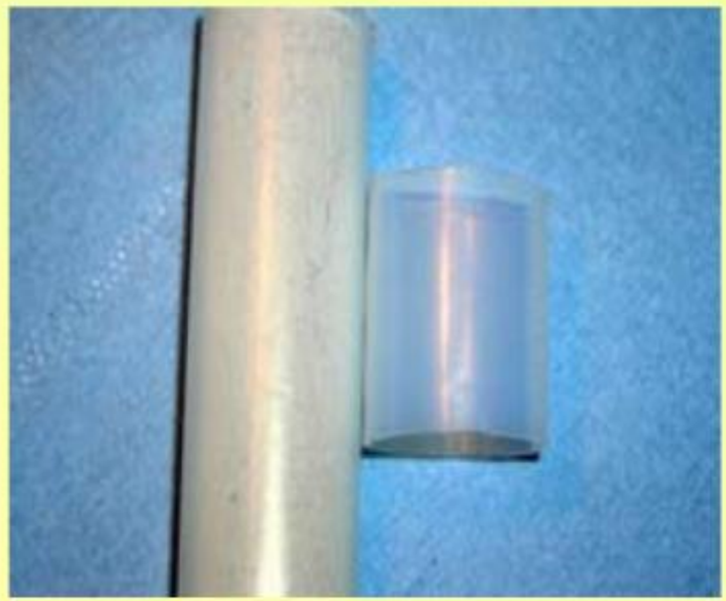
Sulfur Contamination Issues with EP

- Post EP cleaning is required to remove sulfur contamination either with an ethanol rinse or other detergent

Sulphur

During the EP process crystalline sulphur segregates out of the acid. After a few hours a thin film of sulphur was found on tubing surface. Sulfur is water insoluble, and it's not to be excluded that the sulfur is also on the cavity surface after the HPR. To remove this sulfur we are planning to rinse the cavity with ethanol. The solubility of sulfur in ethanol at 20°C amounts to 1,14g S / 100g C_2H_5OH. A small test shows that it's possible to remove the sulphur layer with ethanol (see the pictures).

Tube with a thin sulphur layer



Tube before and after ethanol rinsing

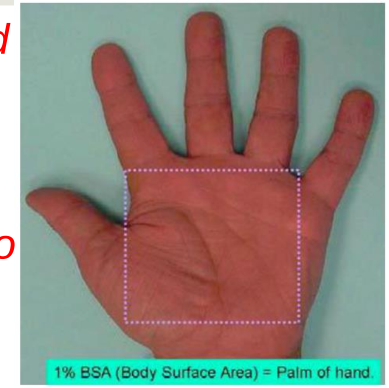
XFEL X-Ray Free-Electron LaserUpdate on the experiences of electro polishing of multi-cell resonators at DESY
N. Steinhau-Kühl, A. Matheisen, L. Lilje, B. Petersen, M. Schmökel, H. Weitkämper, Deutsches Elektronen Synchrotron DESY, Hamburg, Notkestraße 85,22602 Hamburg, Germany

8. Safety Considerations

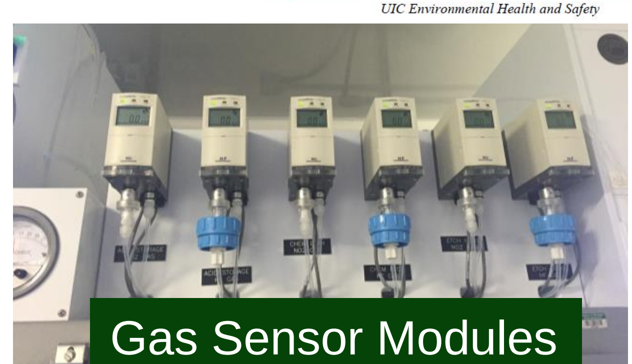
BCP Hazards and Controls

- **Chemical Hazards:**
  - Exposure to BCP (HF acid & vapor)
  - Exposure to NO_2 by products
- **Controls:**
  - Air pressure and flow alarms in each tool and room for ventilation system
  - HF & NO_2 sensors and alarms in each tool and room
  - Leak detection in each containment
  - Level sensors on tanks
  - pH alarms on heat exchanger
  - Sashes and doors to all tools interlocked
- Operators protected from hazards

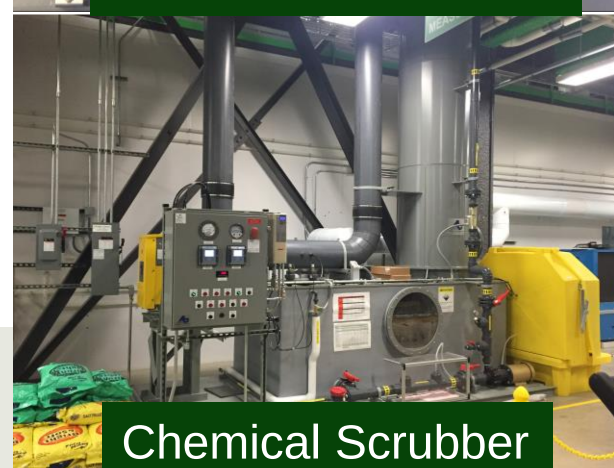
Concentrated HF acid at 50% or stronger to 1% or more to BSA can be fatal



UIC Environmental Health and Safety



Gas Sensor Modules



Chemical Scrubber

BCP Administrative Controls and Procedures

- Most important is training staff on hazards and **RESPONSE**
  - Maintain restricted access to only trained and authorized staff
  - Write and review procedures prior to performing work
  - Have an emergency response plan documented and training in place
- Wear all required Personal Protective Equipment (PPE)
- Safety shower and eyewash installed near facility

Have Emergency Kit Ready With Calcium Gluconate

Ensure local responders know how to handle HF emergency

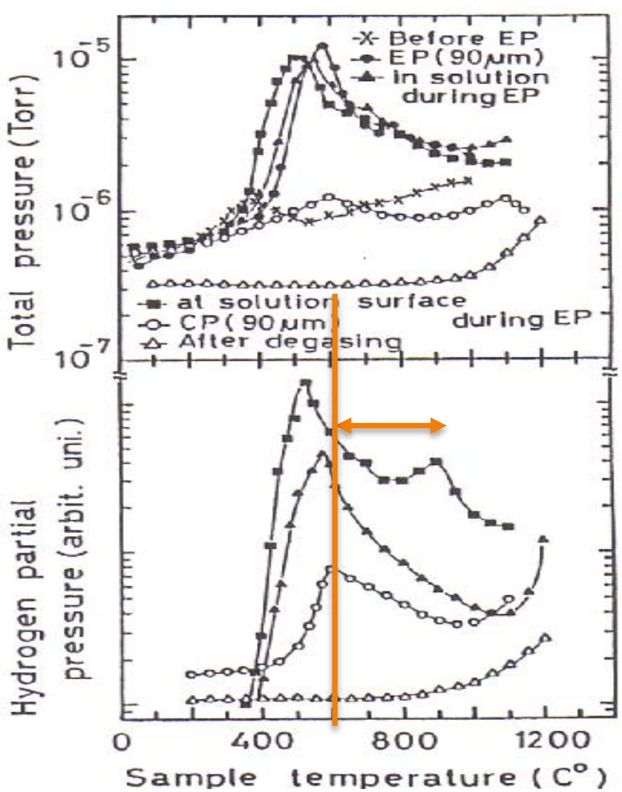
First aide triage response to HF: rinse 3-5 min. and start applying calcium gluconate

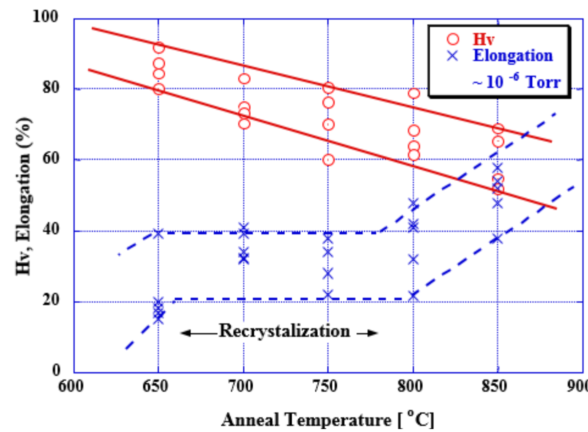
See APPENDIX for more details

9. Heat Treatments

Why Heat Treatment?

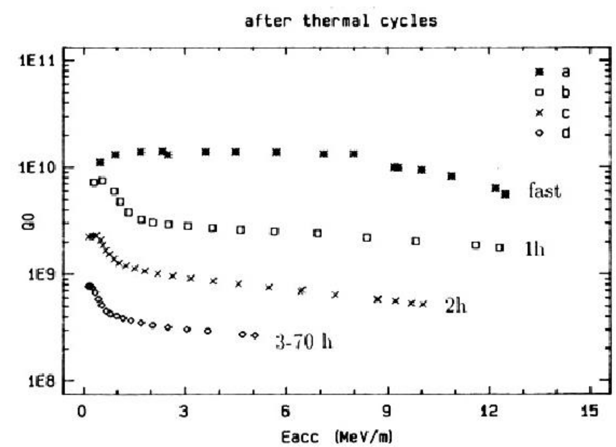
- Clearly observed Q_0 drop after niobium soak around 100 K for 1+ hours
- Fast cool down of niobium < 1 hr is required to avoid the Q_0 drop (disease)
- Hydrogen degasification proven to eliminate the Q_0 drop even during a slow cool down
- Degassing effective starting ~600 °C





Partial recrystallization begins ~ 600ºC and full recrystallization begins ~800 ºC

(Saclay)

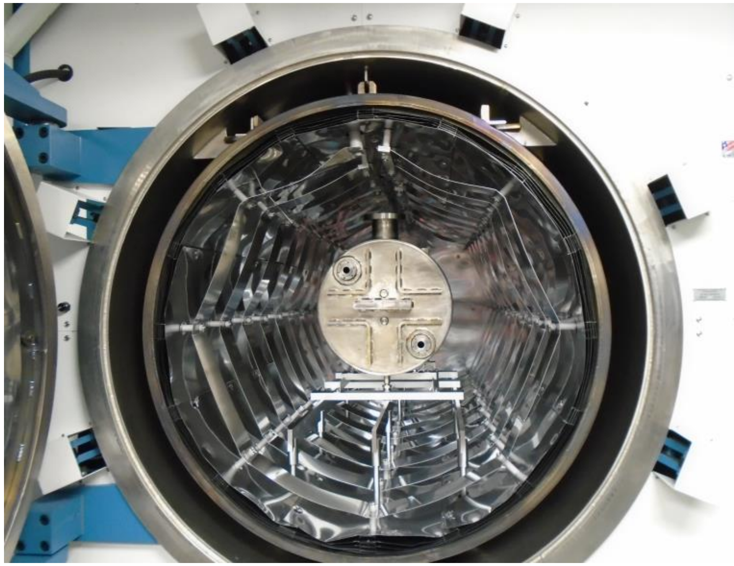


Q_0 drop after various soak times ~100 K

Kenji Saito, Lecture note in Tokyo University on May 2011

Heat Treatment to Remove Hydrogen Uptake from Processing

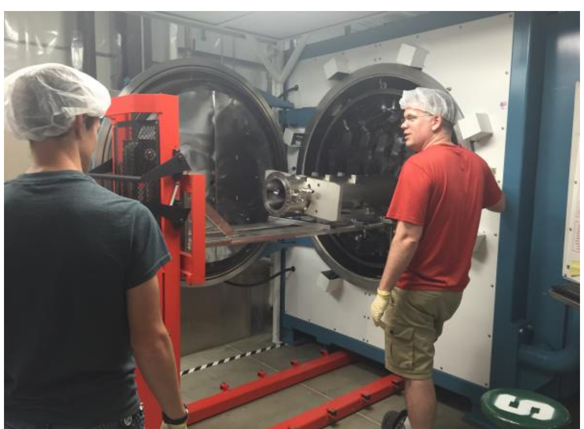
- Risk of Q-disease eliminated by hydrogen degassing step after the bulk chemistry
- Cavities fired in high temperature vacuum furnace 10 hours at 600 °C pressure 1E-5 torr Start with soak for 12 hours at 350 °C
- Heat treatment drives much hydrogen from the bulk niobium material
- Cavity must be degreased and dry
- Furnace must be kept clean and dry, and located in a clean zone

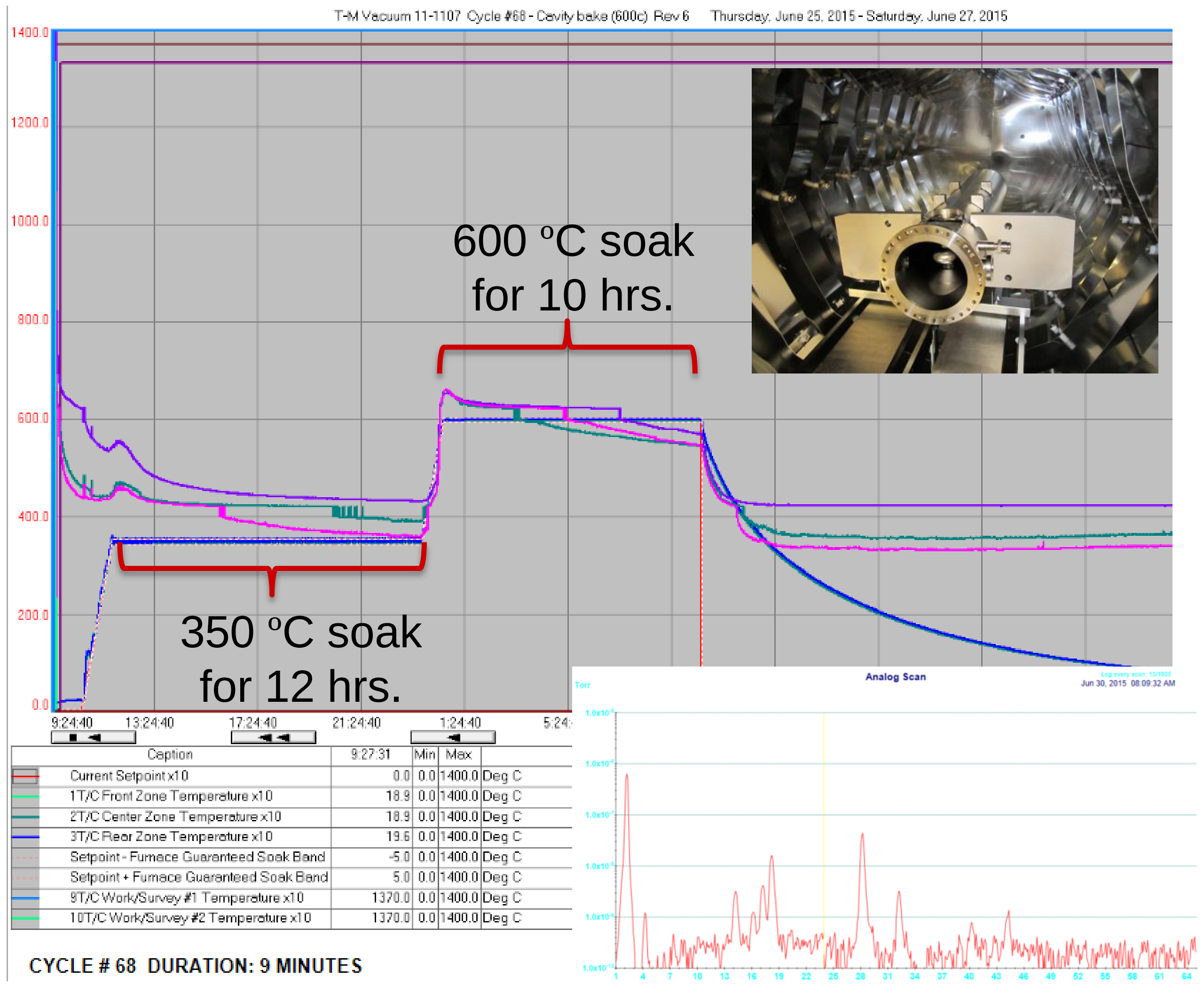


Temperature	Time	Purpose	Notes
600 °C	10 hrs.	Hydrogen degassing, non-annealing	Use for geometry like QWR that cannot have IC droop
800 °C	2 hrs.	Annealing (recrystallization) remove hydrogen	Nb becomes soft, allows easier tuning of elliptical
> 1000 °C	2 hrs.	Post-purification, full recrystallization	Vacuum annealing, usually Nb surrounded by titanium getter material/foil

Heat Treatment Furnace Operation

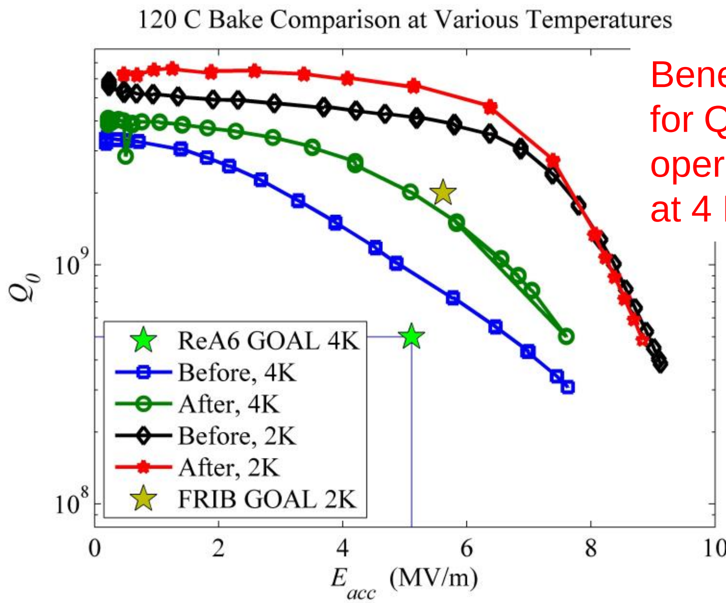
- Partial pressures and temperature recorded versus time using residual gas analyzer (RGA)



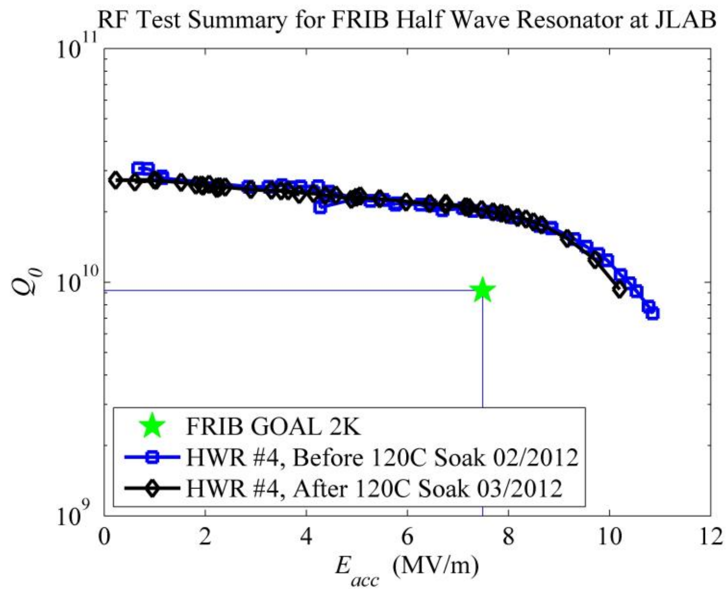


120 °C Low Temperature Bake

- Bake at 100 °C-150 °C under UHV for > 24h has beneficial effects on the BCS surface resistance and the high field Q-drop
- Improved low beta cavity performance at 4 K but not required to meet FRIB cavity performance specifications at 2 K
- It has been related to oxygen diffusion into the niobium, causing changes of the structure niobium/oxide interface on a nanometer scale



Beneficial for QWR operating at 4 K

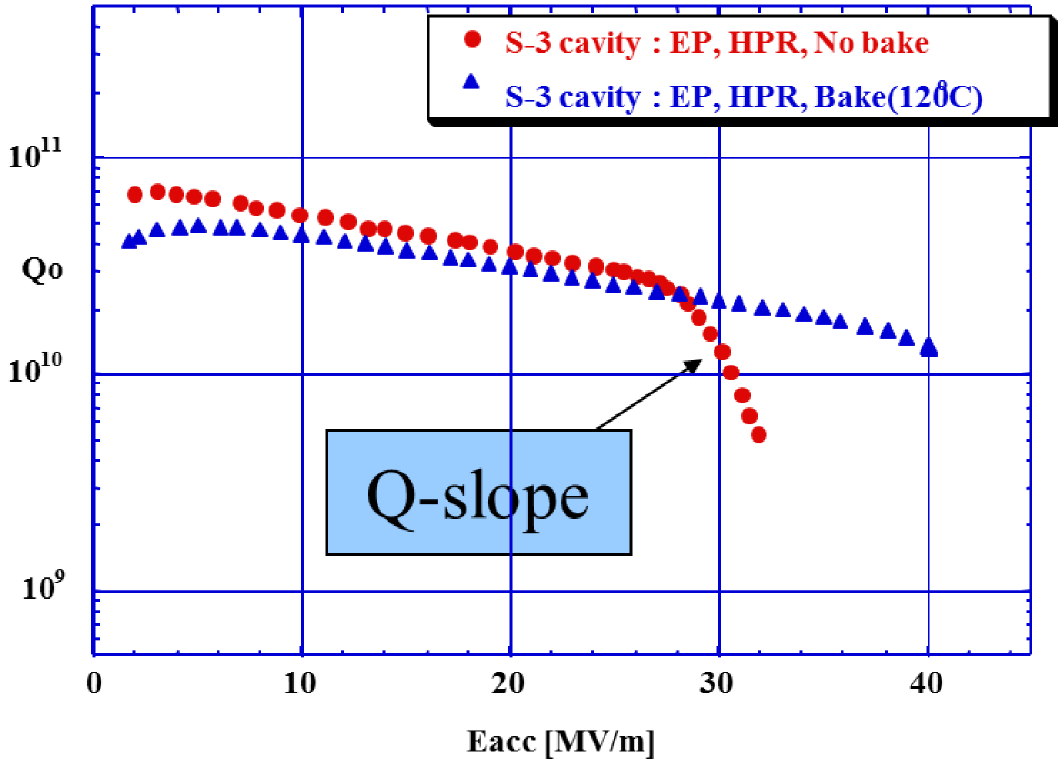


Less beneficial for HWR operating at 2 K

EFFECT OF LOW TEMPERATURE BAKING ON NIOBIUM CAVITIES *
G. Ciovati†, P. Kneisel, G. Myneni, Jefferson Lab, Newport News, VA 23606
W. A. Lanford, Department of Physics, SUNY Albany, Albany, NY 12222, SRF 2003

Bake Electropolished Elliptical Cavity

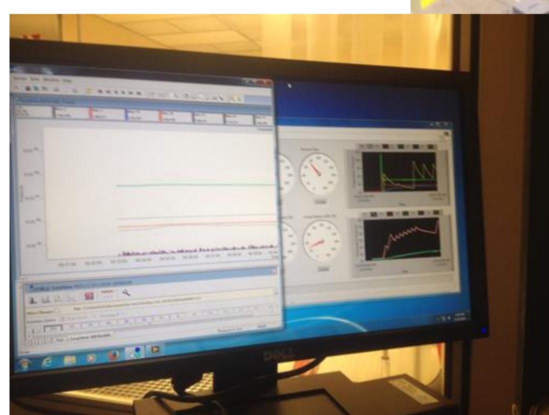
- Bake at 100ºC-150ºC under UHV for > 24 hr. has beneficial effects on the BCS surface resistance and the high field Q-drop
- Effective for electropolished elliptical cavities



Kenji Saito, Lecture note in Tokyo University on May 2011

Low Temperature Bake Method

- Warm, dry gas 120 °C through helium vessel
- Custom shaped thermal insulation jacket wrapped around exterior
- Ramp to 120 °C (over ~ 3 hours) and hold with controllers for 36-48 hours
- Interlock to shut off if temperature rises
  - Indium seal melt at 156 °C
- Pressured kept less than 1E-5 torr
- Cavity actively pumped and under vacuum
- Pressure and temperature versus time is recorded and RGA partial pressure logged

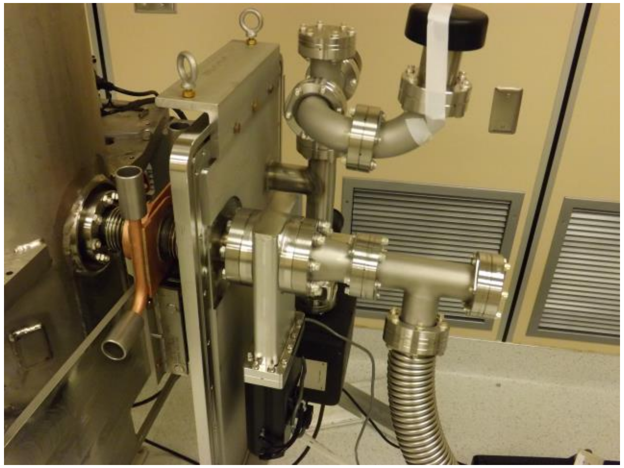


10. Cleanroom Protocols

"But mom, all you said was 'get all your stuff up off the floor!' "

Why do we use cleanrooms?

- Cleanrooms used to prevent contamination in SRF components by producing a **low particle environment**
- Small amounts of particulate can cause field emission in coldmasses
- Contamination will stay in a coldmass forever!
- A clean cavity is a high performing cavity
- **Everything** that goes into the cleanroom must go through a cleaning process!



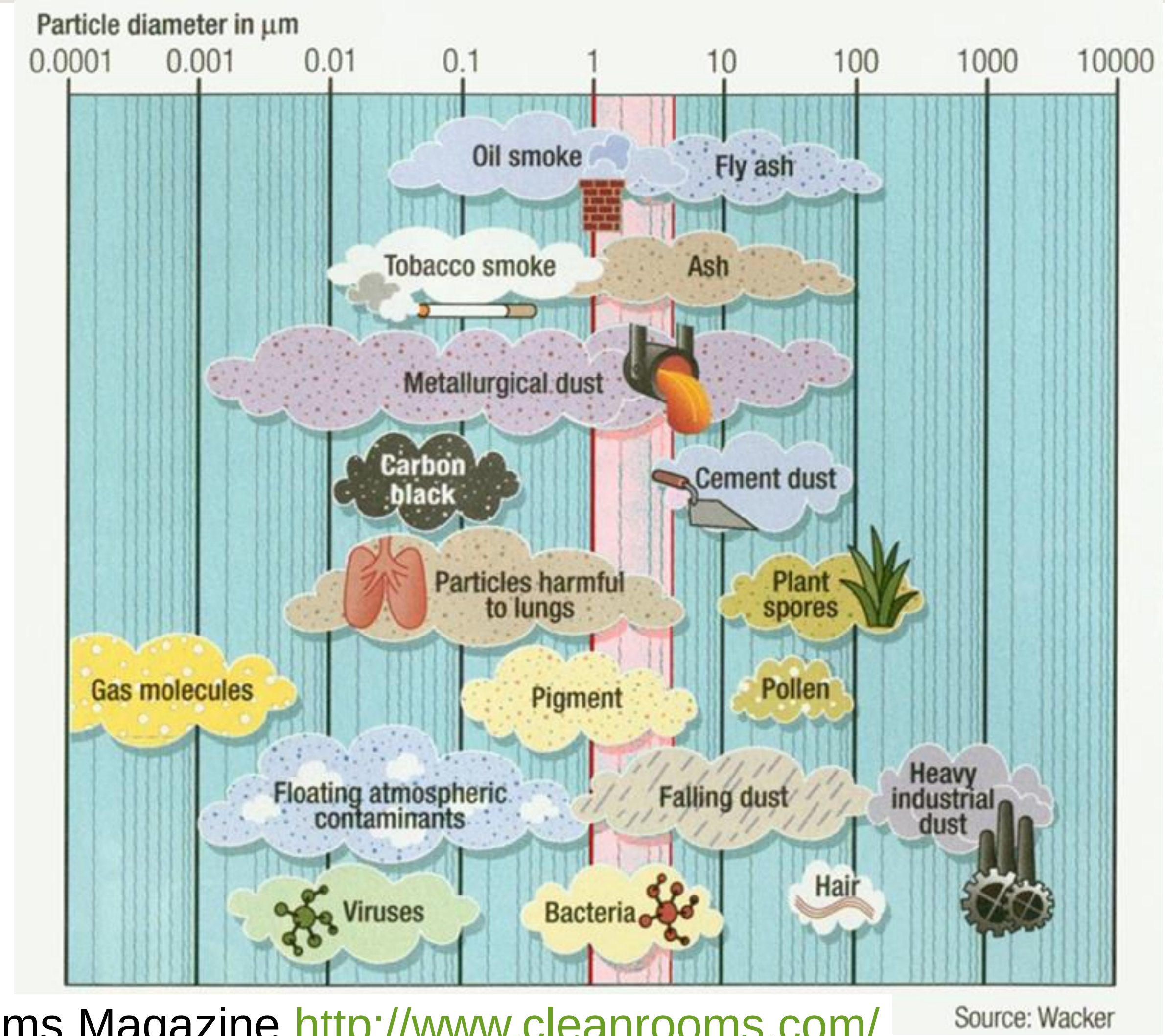
What is Contamination?

- A process or act that causes materials or surfaces to be soiled with contaminating substances
- 2 types
  - Film type
  - Particulate

Facilities	People	Tool Generated	Fluids	Product generated
Air conditioning debris	Clothing debris (lint, fibers etc.)	Brooms, mops and dusters	Cleaning chemicals	Aluminum particles
Construction material (sheet rock, saw dust etc.)	Spittle	Vibrations	Floor finishes or coatings	Cleanroom debris
Paint and coatings	Cosmetics and perfume	Lubricants and emissions	Bacteria, organics and moisture	Quartz flakes
Room air and vapors	Hair	Friction and wear particles	Plasticizers (outgasses)	Silicon chips
Spills and leaks	Skin flakes and oil		Deionized water	
Walls, floors and ceilings			Particulates floating in air	

What is particulate?

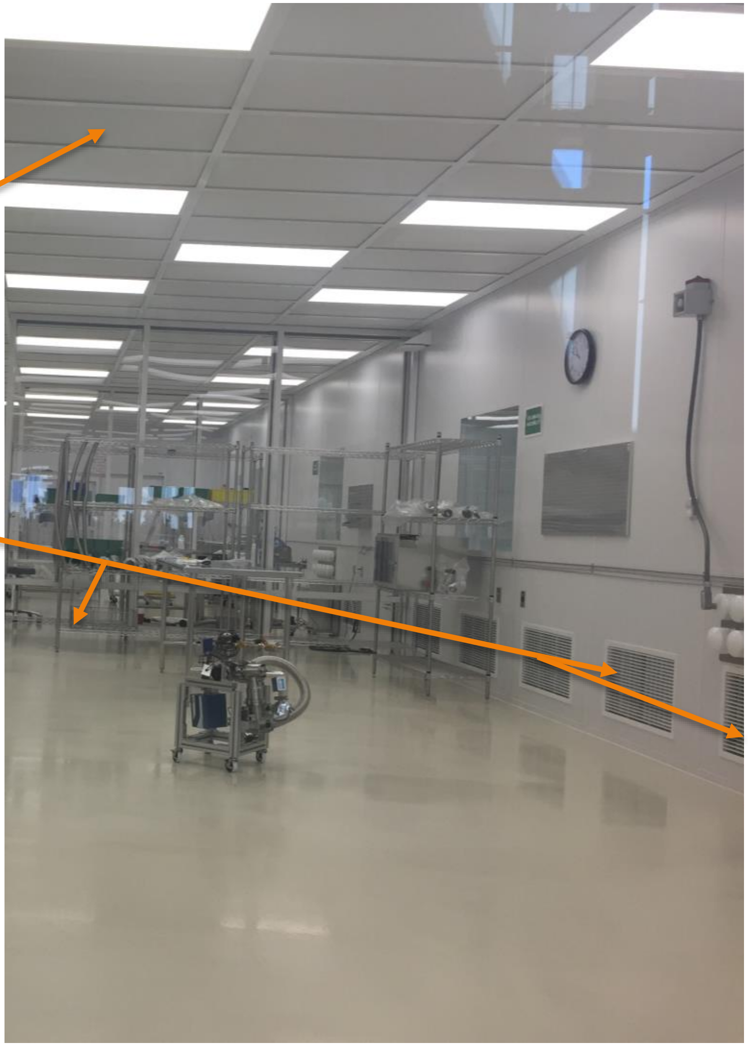
- Particulate is submicron solid matter suspended in the air
- **You cannot see particulate!**
- Certify at 0.5 micron
- Human hair is 100 microns, 200x larger!
- Class 1,000 means there are less than 1,000 particles of 0.5 micron size per cubic foot
- A average high bay space has about a half million!



Cleanrooms Magazine http://www.cleanrooms.com/

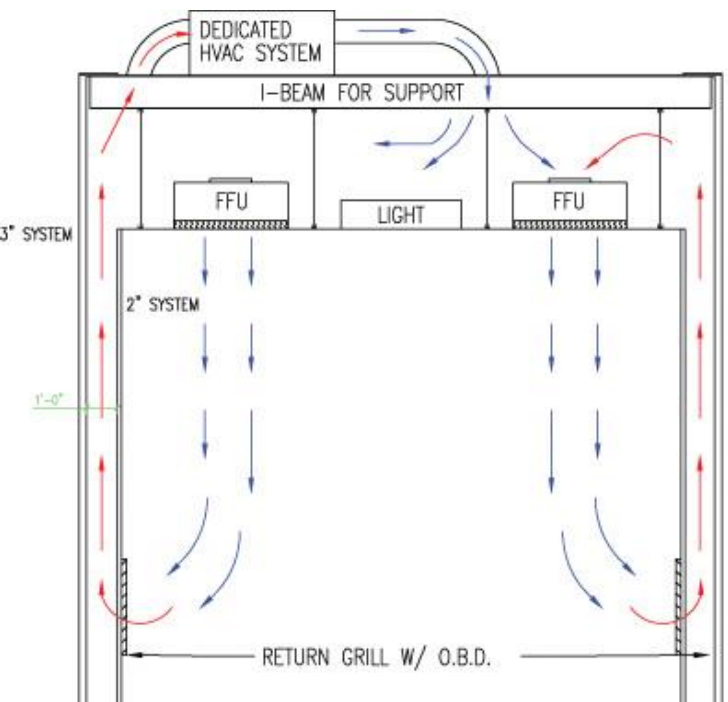
What is a Cleanroom?

- “Controlled Environment” that limits airborne contamination
- Controlled parameters:
  - Air Filtration
    - » Pre-filters on air intake
    - » HEPA Filters in the ceiling tiles
  - Air flow velocity and direction
    - » Laminar down then to return wall vents
  - Pressurization
    - » Higher pressure air from clean to dirty zones
  - Temperature
    - » Set at comfortable level
  - Humidity
    - » Set at comfortable level
- Cleanroom is isolated from other lab or production floor spaces with barriers



How does it work?

- HEPA filters remove particulate and create a **laminar air flow** environment
- Particulates flow to floor and are exhausted through vents
- Room pressure is higher than surrounding areas pushing contamination out
- CR air is quantified and certified following ISO 14644-1 guidelines
- A cleanroom MAINTAINS pre-cleaned items in a decontaminated state
- A cleanroom will NOT clean items



Single pass air flow design →

← Air return recirculation design

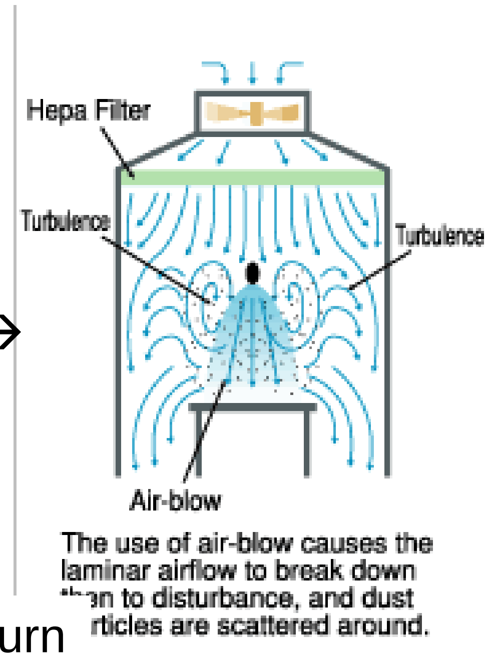


Figure 1: Turbulent Flow

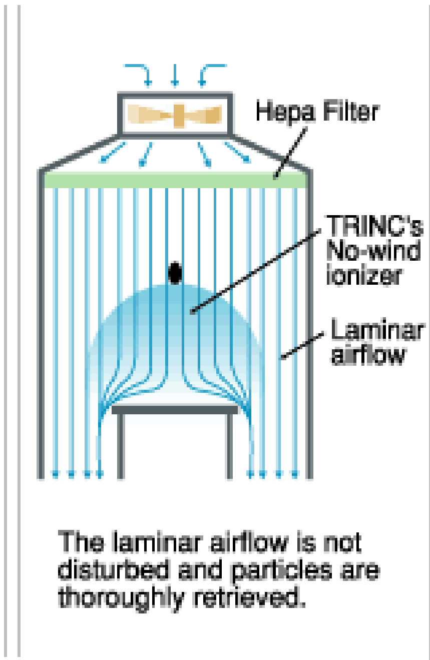


Figure 2: Laminar Flow

How do I know it works?
ISO Standard and Federal Standard 209 (obsolete)

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	35	8	
ISO Class 4	10 000	2 370	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	8 320	293
ISO Class 7				352 000	83 200	2 930
ISO Class 8				3 520 000	832 000	29 300
ISO Class 9				35 200 000	8 320 000	293 000

Table 1 Federal Standard 209 class limits

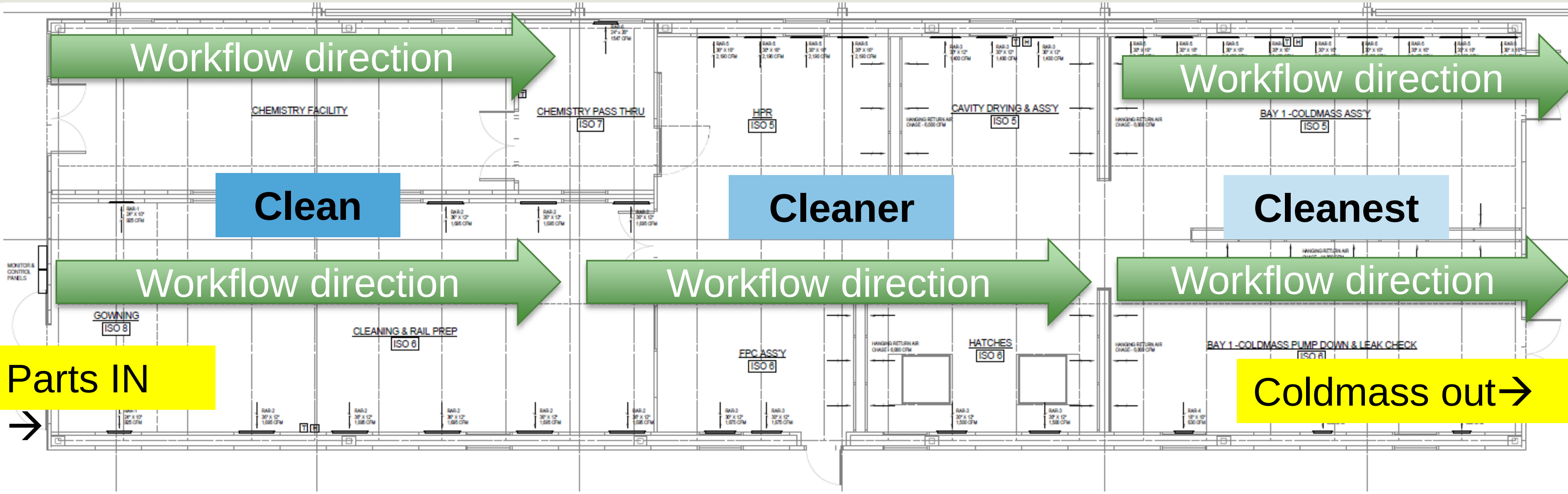
Class	Particles / ft³				
	≥ 0.1 μm	≥ 0.2 μm	≥ 0.3 μm	≥ 0.5 μm	≥ 5.0 μm
1	35	7.5	3	1	NA
10	350	75	30	10	NA
100	NA	750	300	100	NA
1,000	NA	NA	NA	1,000	7
10,000	NA	NA	NA	10,000	70
100,000	NA	NA	NA	100,000	700

Table 3 Comparison between selected equivalent classes of FS 209 and ISO 14644-1

ISO 14644-1 Classes	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8
FS 209 Classes	Class 1	Class 10	Class 100	Class 1000	Class 10 000	Class 6 100 000

- Various particle counting systems used to certify clean spaces: handheld, portable, real time on-line.

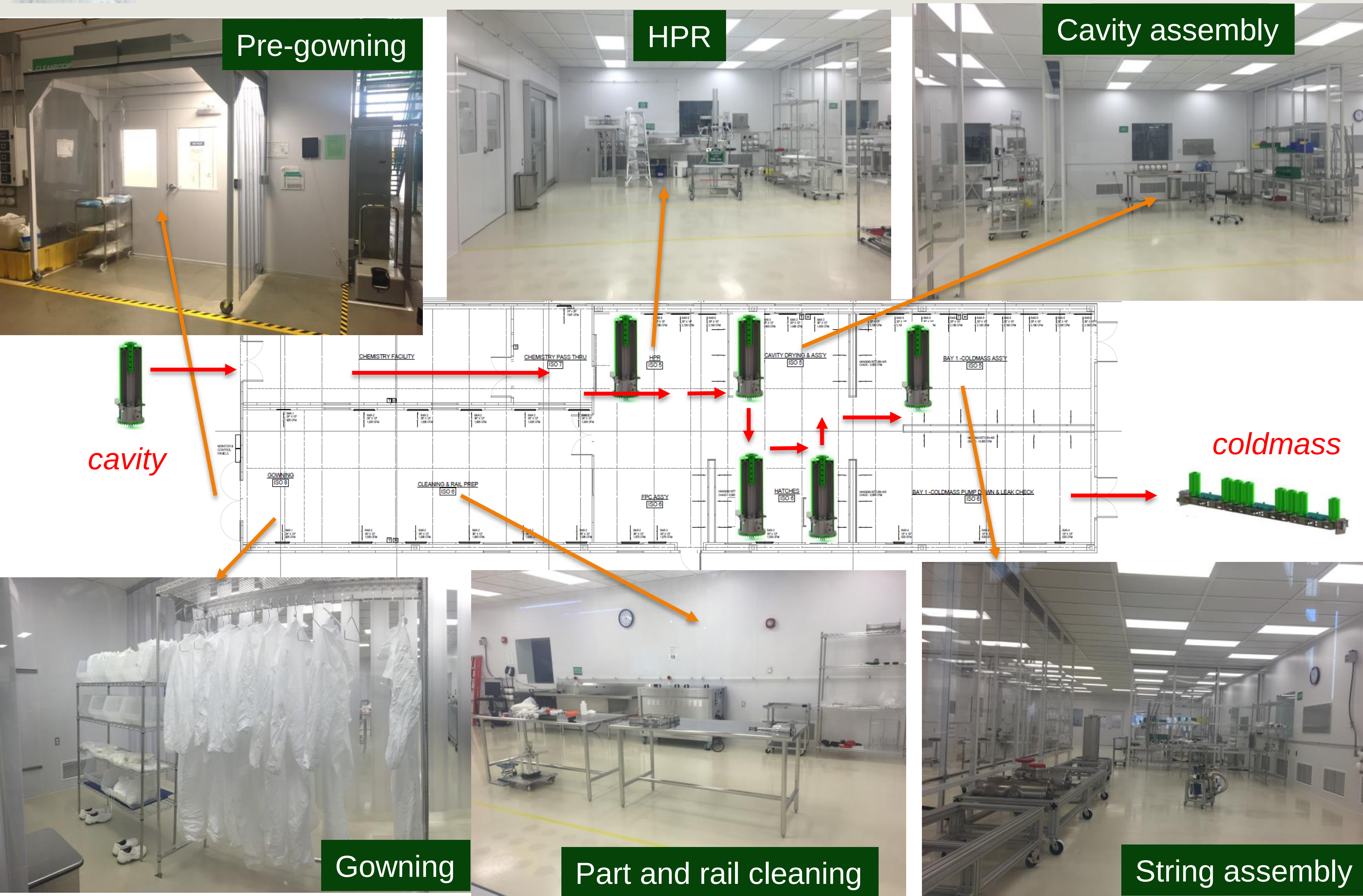
SRF Production String Assembly Cleanroom Classification



- GOWNING: ISO 8
- CLEANING & RAIL PREP: ISO6
- HPR: ISO 5 & FPC ASS'Y: ISO5
- CAVITY DRYING & ASS'Y: ISO 5
- HATCHES: ISO 5
- COLDMAS ASS'Y BAY 1: ISO 5
- COLDMASS PUMP DOWN & LEAK CHECK: ISO 6

- Avoid cross contamination
- Workflow should go in one direction
- Design layout and workflow so you do not go backwards through the process
- Transitional areas for entrance and exit

Tour of a Production SRF Cleanroom



Controlling Human Contamination

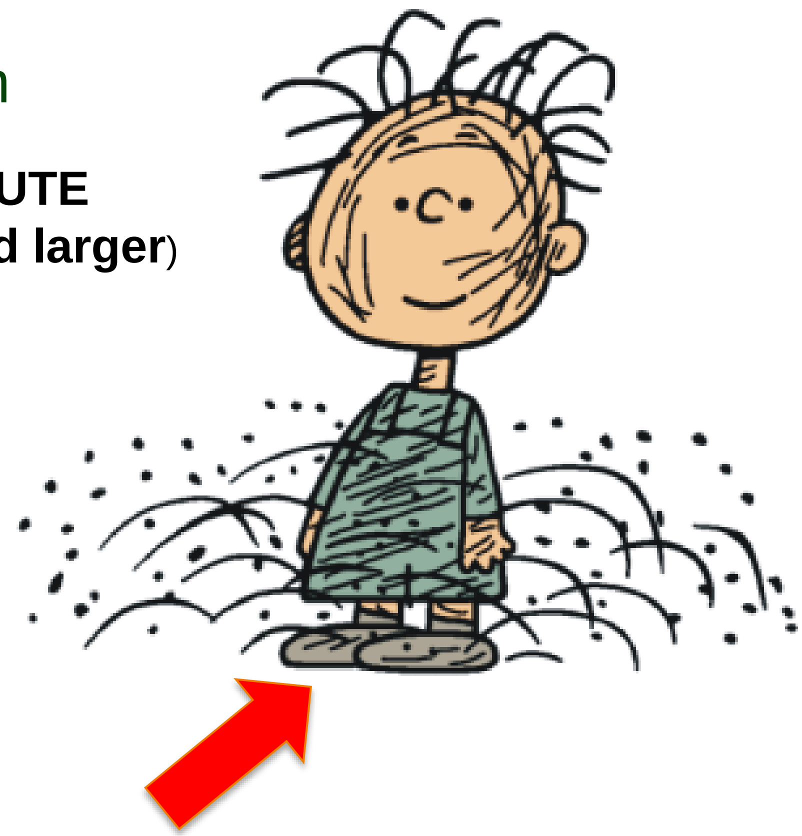
- 5-10 million particles from skin, hair, dirt, clothing
- Must use approved garments (class 100 or less)
- "**bunny suit"** controls human contamination
- Protects cleanroom by creating a barrier
- Includes Coveralls, Boot covers, and Hood
  - Gloves, hairnet, and mask are also required
- Coveralls on in transitional "gowning" area
- Cleaning EVERYTHING that goes in
- Prohibiting risky items and behaviors
- Regular maintenance focusing on high traffic areas

ALL COVERALLS MANUFACTURED IN CLEANROOMS! CHECK ISO CERT.

Do I have to wear it?

- Absolutely, every time you enter, even if only grabbing something or flipping one switch!
- YOU are the dirtiest thing in a cleanroom

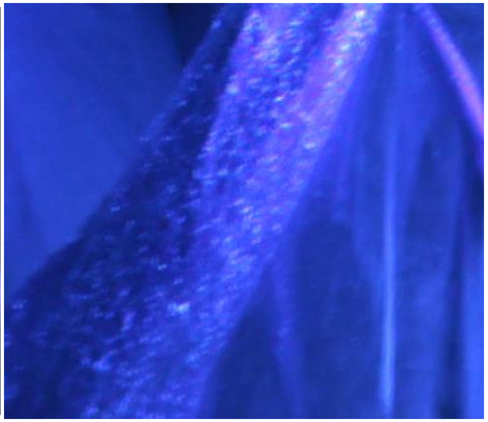
PEOPLE ACTIVITY	PARTICLES/MINUTE (0.3 microns and larger)
Motionless (Standing or Seated)	100,000
Walking about 2 mph	5,000,000
Walking about 3.5 mph	7,000,000
Walking about 5 mph	10,000,000
Horseplay	100,000,000



This is what people look like to a cleanroom !

Cleanroom Garment Tips

- Change top layer of gloves often during clean assembly
- Gloves, face mask and hair net are disposable, new ones every time
- New set of garments each time for ultra-critical activities like SRF cavity assembly
- Store garments so internal and external surface do not touch



Clear indication of concentrated particles and fibers on the inside of coverall
Concentrated particles around coverall neck area inside and outside of cover
*Re-establish importance of how to store coverall so not to cross-contaminate *Importance of changing the coveralls

See APPENDIX for more details

General Cleanroom Concerns

- Similar metals gall in CR→ use approved grease
- Allergies to latex glove → cleanroom nitrile
- Plated tools and fasteners could flake → stainless steel
- Eyeglass fog up with face mask → contacts or anti-fog wipes
- Can get warm in CR coverall → wear layers

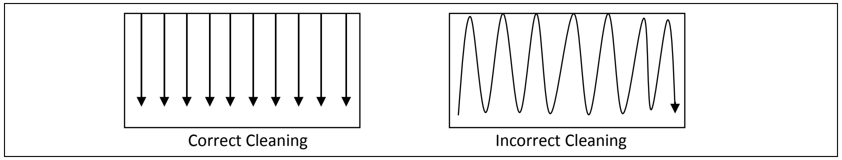
Stainless Steel will gall! Similar metals will gall together when there is no film barrier between them.

Good Cleanroom Practices

- Use tacky mat at entrance of cleanroom
- Put on gloves as soon as you enter the cleanroom
- Sensitive parts packaged in approved material, backfilled with filtered nitrogen gas and sealed before leaving CR

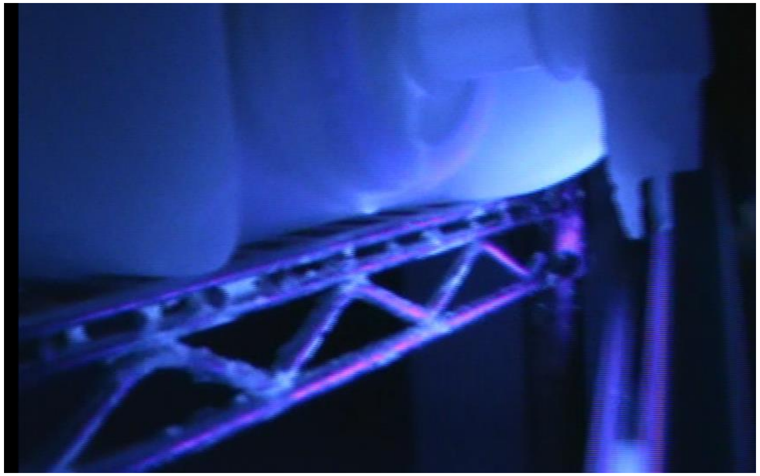
- Move sensitive parts away from doorway
- Maintain positive air pressure
- Wipe down all items brought into cleanroom with lint free wipers and ethanol

- Move slowly to reduce air turbulence
- Reduce mechanical vibrations in tools or equipment
- Tape wheels to carts
- Regular maintenance

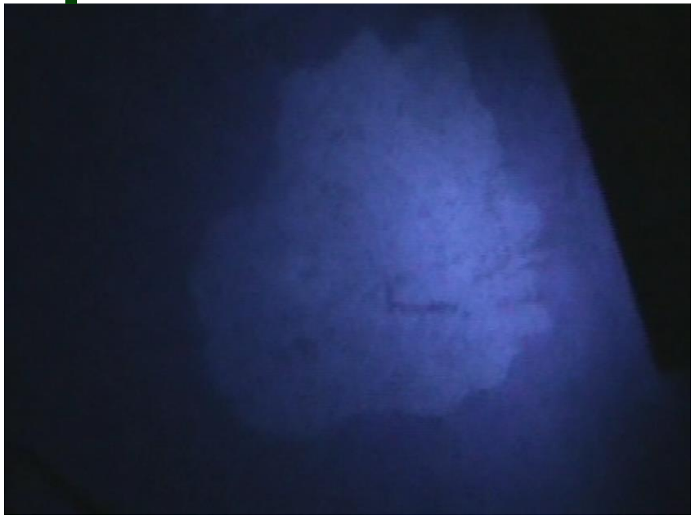


See APPENDIX for more details

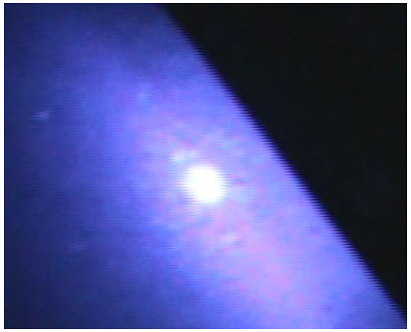
Contamination Detection Using Ultra Violent Lamp Fluorescence



Micro90 Degrease Dispenser

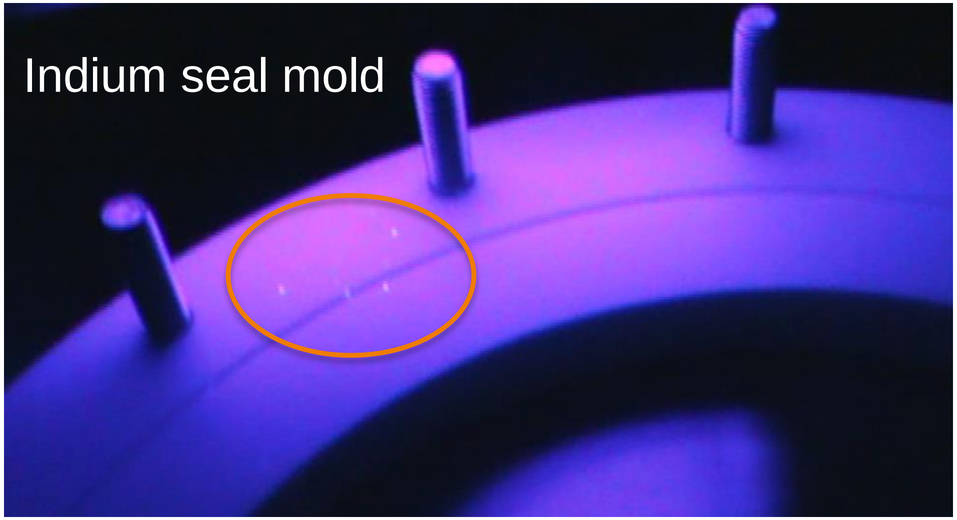


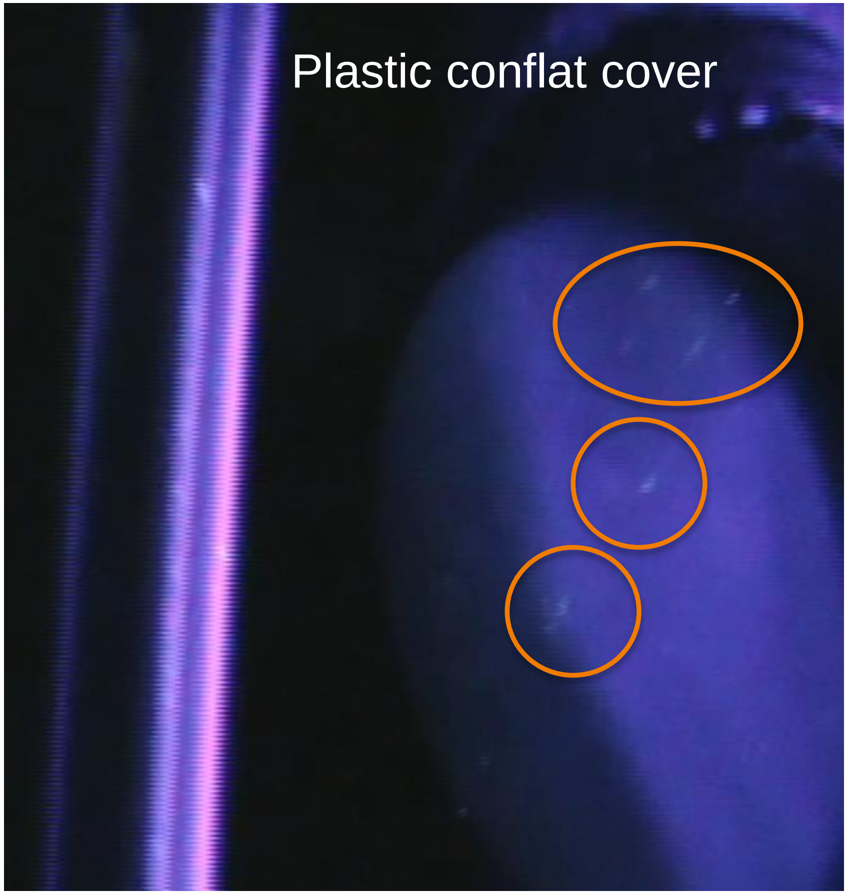
Easily detect spills, detergents and water marks with UV light

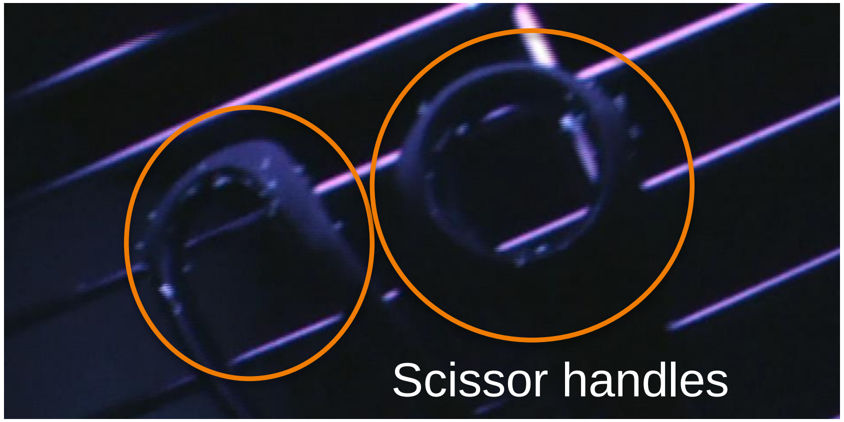


Mop areas of high traffic often!

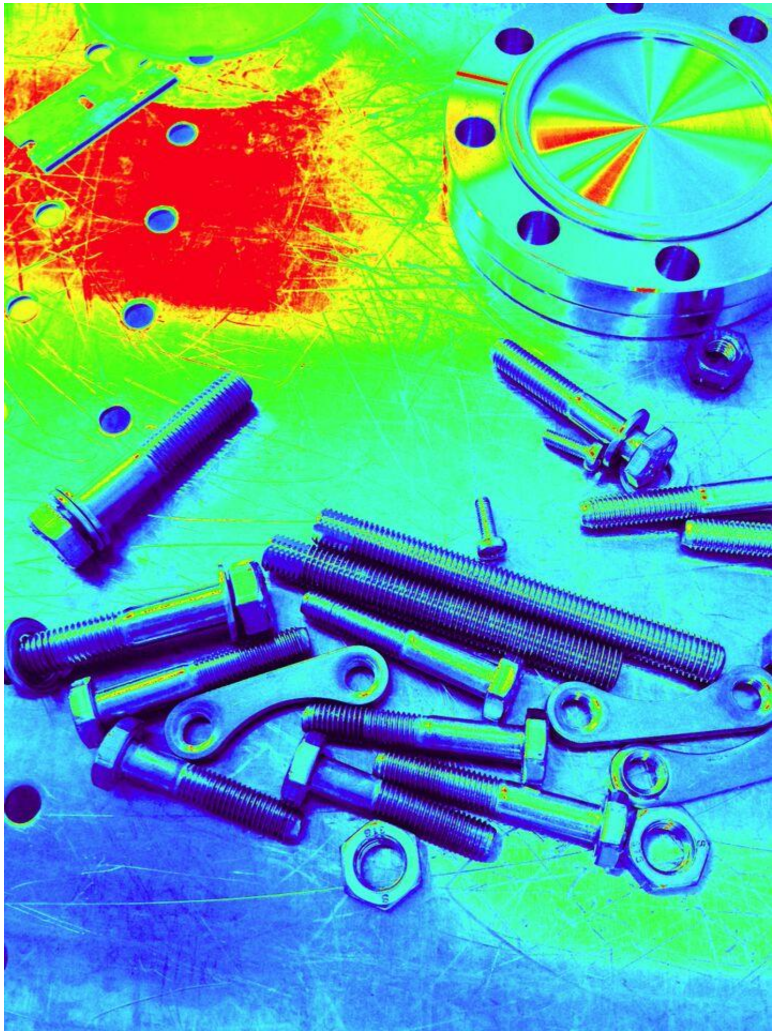
Tool Inspection for Macro Particle Detection







11. Cleanroom Assembly Techniques



Surface Particle Specifications

- Surface particle counts performed on accessible cavity surfaces and all vacuum flanges
- Counts < 1 particles/in^2 at 0.3 μm
- Better than 1246D Level 1

INSTITUTE OF ENVIRONMENTAL SCIENCES AND TECHNOLOGY

Contamination Control Division
Standard 1246D

IEST-STD-CC1246D

Product Cleanliness Levels and Contamination Control Progra

Table 1. Particulate cleanliness levels

Cleanliness Level	Particle size (µm)	Maximum allowable concentration limits for particles of stated size and larger Particles per 0.1 m^2 of surface area or 0.1 liter of gas or liquid (N)
1	1	1
5	1	2.8
	2	2.3
	5	1
10	1	8.4
	2	6.9
	5	2.9
	10	1
25	2	53.1
	5	22.7
	15	3.3
	25	1
50	5	166
	15	24.6
	25	7.2
	50	1
100	5	1780
	15	264
	25	78.4
	50	10.7
	100	1
200	15	4180
	25	1230
	50	169
	100	15.8
	200	1
300	25	7450
	50	1020
	100	95
	250	2.2
	300	1
500	50	11800
	100	1090
	250	26.3
	500	1
750	50	95800
	100	8910
	250	213
	500	8.1
	750	1
1 000	100	42600
	250	1020
	500	38.7
	750	4.7
	1 000	1

Note 1. Concentration limits shown are maximum cumulative particle count for particles equal to and larger than the stated particle sizes, for surface or liquid, to meet a specified cleanliness level. Sampling areas other than 0.1 m^2 shall be calculated to the basis of 0.1 m^2. Areas may be estimated if total area is considered by both parties to be too difficult to measure within two significant figures. This condition shall be noted and low/high ranges shall be used. Parts with a total significant surface area less than 0.1 m^2 and which have had the entire critical surface area sampled shall be accepted on the basis of actual count.

Note 2. Values in the table are from equation 5.1, rounded down to three significant digits, and expressed to no more than one decimal place.

Table 4. Sampling and measurement techniques for surfaces, liquids, and gases

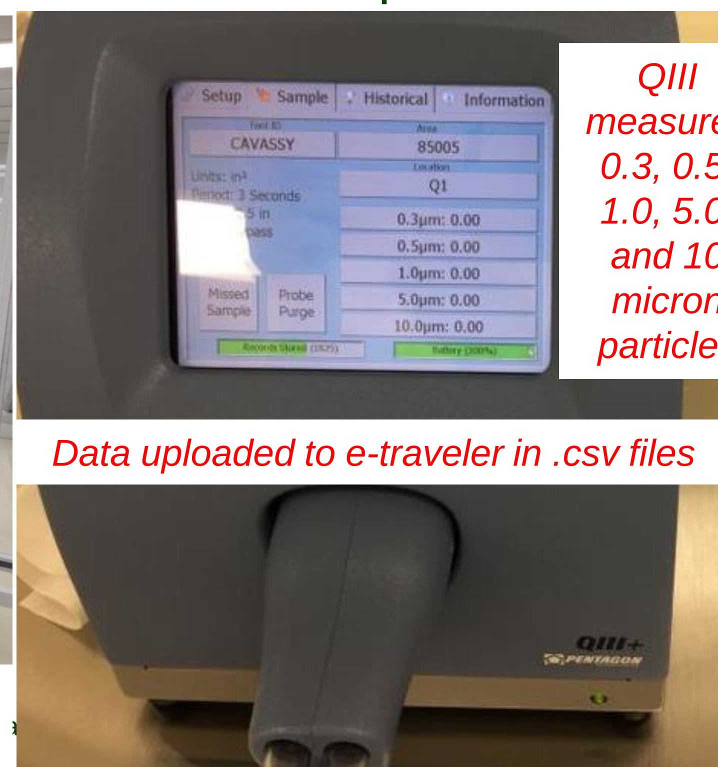
	Sampling Techniques	Measurement Techniques
Surfaces	ASTM F51 ASTM F303 ASTM F306 ASTM E1216 ASTM E1234	ASTM F311 ASTM F312 ASTM F331 ASTM E1235
Liquids	ASTM F302 ASTM F303 ASTM F1094	ASTM F311 ASTM F312 ASTM F331 ARP 598
Gases	ASTM F25 ASTM F50 ASTM F307 ASTM F318 ASTM F327	ASTM F25 ASTM F50 ASTM F312 ASTM F331 ARP 743

Particle Free Quality Control

- Surface particle counts performed using diagnostic probe
- Displaces particles on surface using pressurized air and then vacuums into laser particle counter → automated method
- The probe does not touch RF surface but hovers closely to surface
- Similar method can be achieved with pressurized filtered nitrogen gas to displace particles on the surface and collect with handheld air particle counter

ISO 5 /Class -100 Surface particle counts of tuning plate

ISO 5 /Class -100 Surface particle counts of QWR

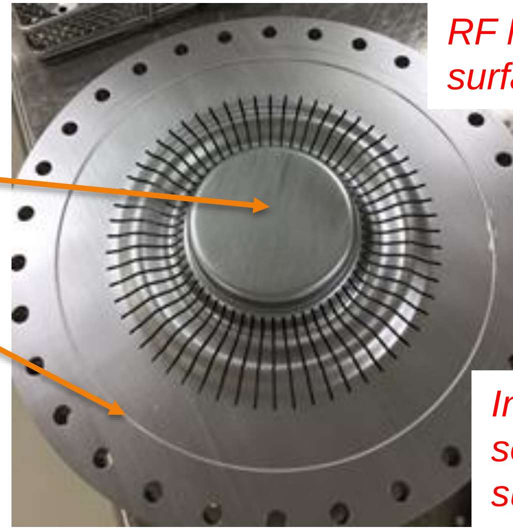


QIII measures 0.3, 0.5, 1.0, 5.0, and 10 micron particles

Data uploaded to e-traveler in .csv files

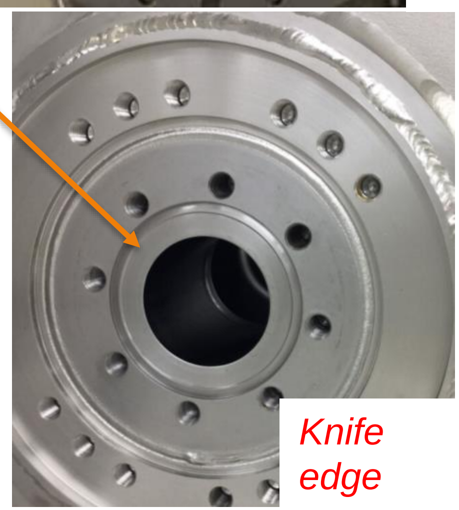
Part Inspection Essential Prior to Final Assembly

- Final look at:
  - Gaskets
  - RF Surfaces
  - Coupler antennas
  - All seal surfaces
  - Knife edges
- If there are any dings, scratches or imperfections the part must be replaced or repaired
- Last time to *"see"* surfaces before coldmass installation



RF Nb surface

Indium seal surface

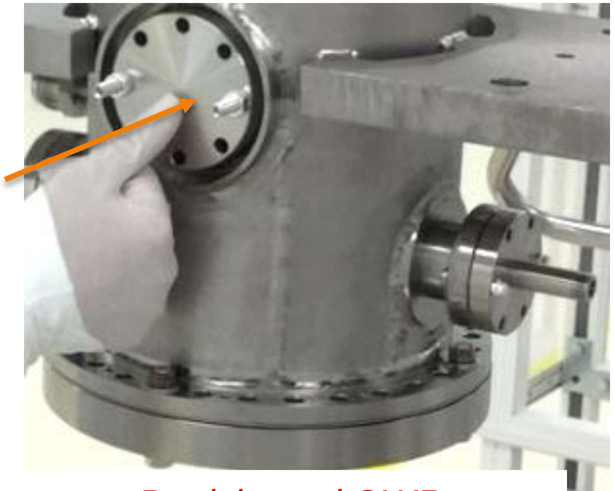


Knife edge

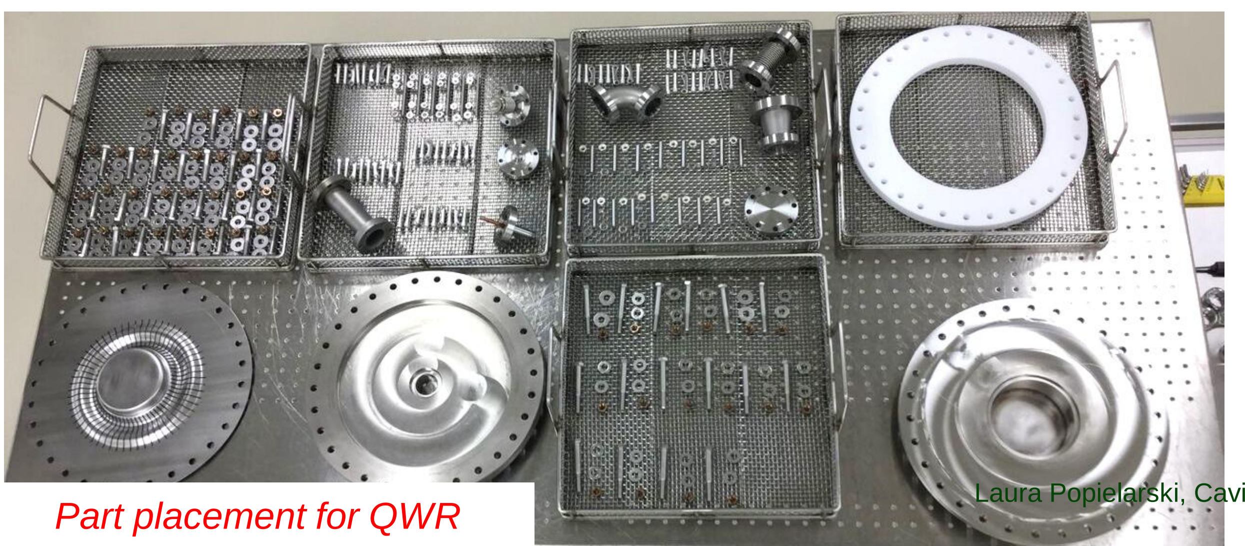
Copper gaskets

Clean Assembly Techniques

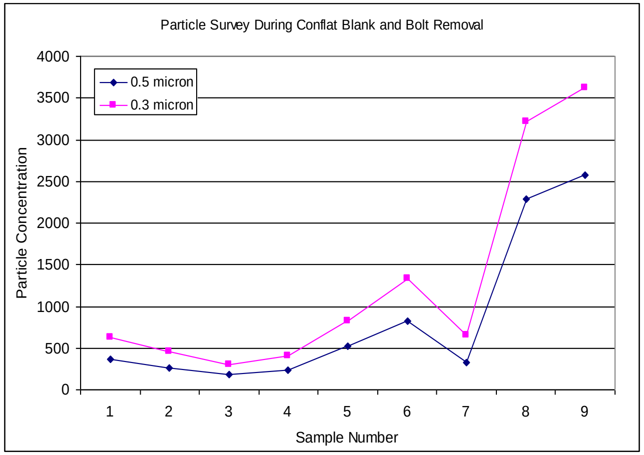
- Define part placement so not to reach over clean parts & for repeatable set-up
- Make particle tight seals with few bolts then move to higher ISO class for all bolt population and torque
- Do not touch vacuum or RF surface with gloves, always lift by edges of flanges
- Do not put arms or hands over ports, keep distance from cavity
- All tools cleaned the same method as components



Particle seal QWR

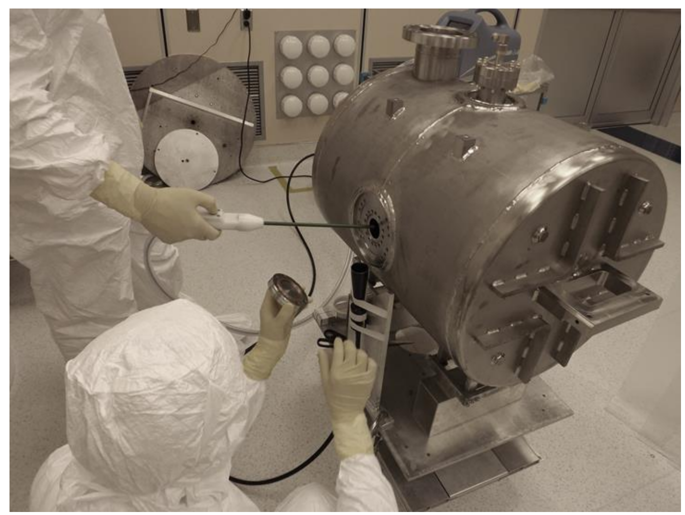


Part placement for QWR



Clean Assembly Concerns

- Errors in gasket installation
  - Slipping flange on copper gasket
    - » Always work from bottom of cavity
  - Dropping gaskets
    - » Work slowly
- Flange hole misalignment
  - Can cause galling and stuck bolts
    - » Tooling can be useful for alignment
    - » Alignment pins
- Background air particle counts too high (> 30 0.5 micron/CF)
  - Ensure no other tasks or major movements are occurring near clean assembly area
    - » Ladders
    - » Rolling carts
    - » Other assembly



Issue: The beam line assembly

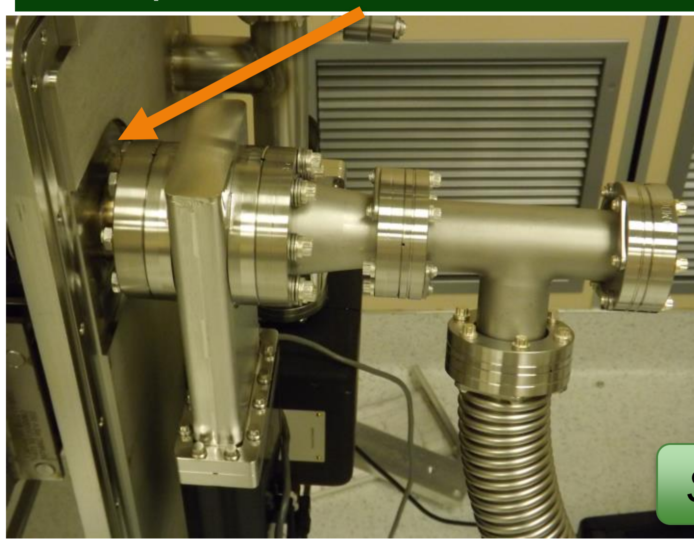
Close proximity vertical flange arrangement

- All subcomponents leak checked prior to assembly
- Particulate counts prior to assembly ensure no contamination source present
- Special tools may be required: bellow compression, gasket holding, low profile wrenches for small gaps, and coupler installation support.
- Slight positive pressure in cavities or string may be used to reduce migration of particles into space (ref. DESY)

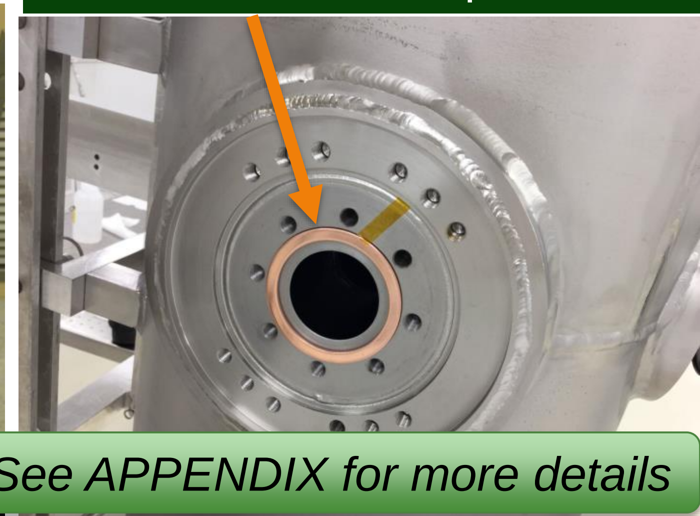
Cavity String Assembly Mechanical Steps at DESY (updated) (technical note), Tug Arkan / Brian Smith, May 12, 2006

Bellows compression tool

Low-profile wrench used here



Gasket held in place



See APPENDIX for more details

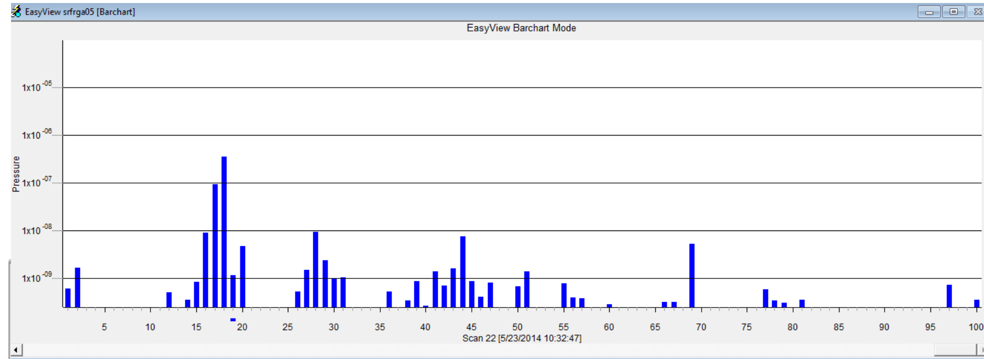
Evacuation for UHV Operation

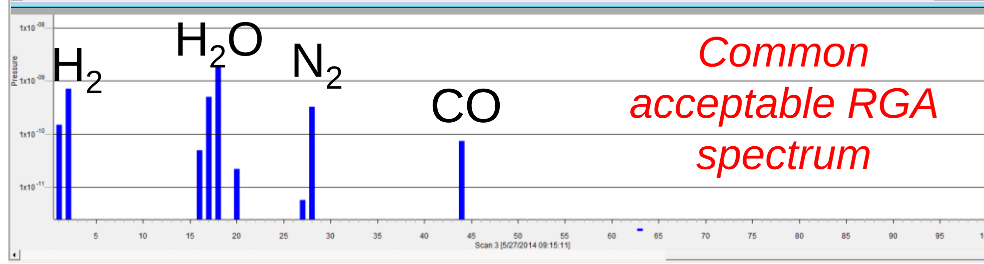
- Vacuum cart/manifold required to pump down and vent cavity for RF testing and coldmass string assembly
- Major components of a vacuum manifolds include:

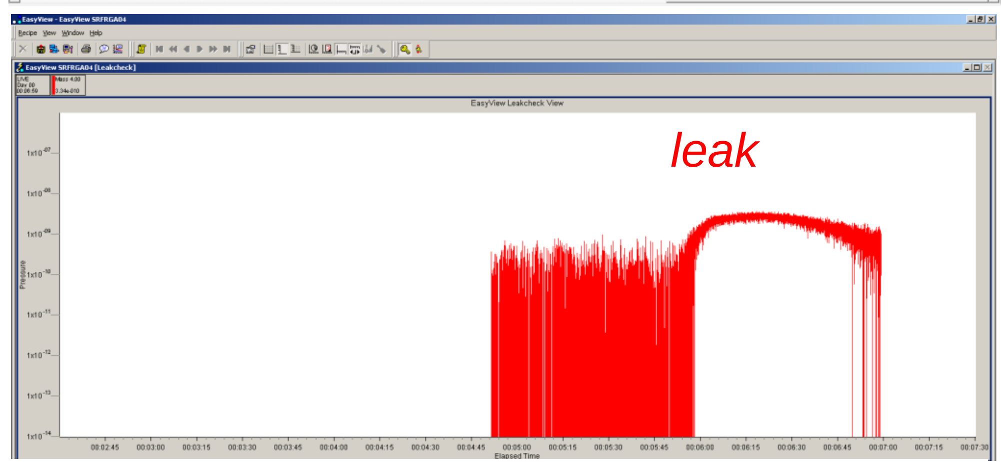
Component	Task
Dry scroll	Rough pump to 1 torr level
Turbo molecular pump	High vacuum pumping <1E-8 torr
High pressure gauge (eg. Pirani)	1-999 torr
Low pressure gauge (ion gauge, cold cathode gauge)	< 1 mtorr
Residual gas analyzer	Partial pressure of gas in vacuum system, can identify contamination and leaks
Burst disk or pop off	Release pressure in over pressure event
Purge lines	Slow purge up to atmosphere
Isolation and/or shut off valves	All metal valve to isolate purge lines or other gas processing lines

Vacuum Quality Characterization

- Residual gas analyzer (RGA) filament and software output partial pressure of detected molecules real time
- Used to detect leaks, contamination, and outgassing
- Libraries define ‘fingerprints’ for common contaminants
- Typical peaks:
  - Air leak, water, nitrogen, helium
  - Hydrocarbons:
    - » High mass peaks – back streaming of oil or vacuum grease
    - » Lower mass peaks - solvents



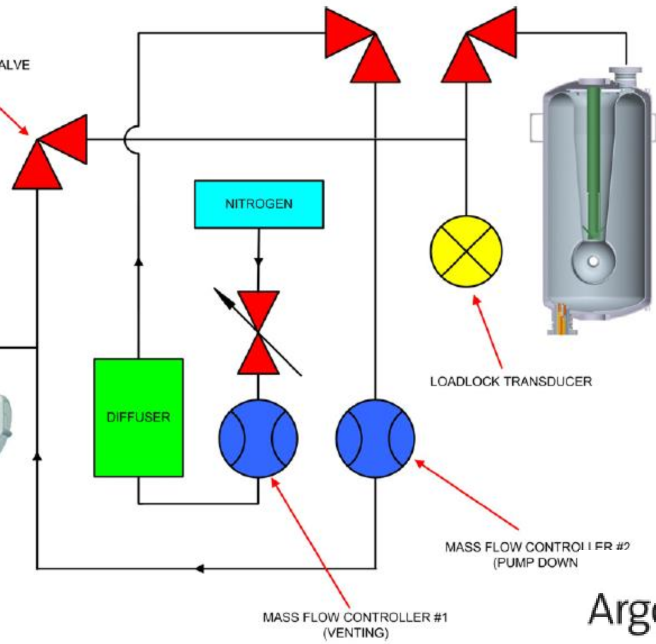




(http://www.mksinst.com/docs/R/SpectraBulletin208.pdf)

Automated Slow Pump and Purge System

- The purpose of all steps is to remove all particulate from cavity assembly
- Same philosophy for evacuation and venting cavity systems applies!
- Must avoid introducing any particles or migration of particles within the system
- Automated systems control rate of pumping and venting using mass flow controllers and diffusers, to keep flow out of turbulent range
- Systems also include submicron filters to keep purge gas very clean
- Typical time for a FRIB cavity venting is ~ 20-30 minutes and 4-6 hours for a full coldmass string
- Both single cavity test stands and full coldmasses have been pumped and vented multiple times and continue to maintain performance

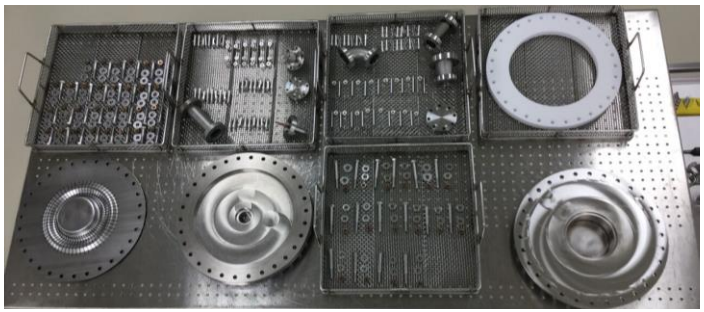


PARTICLE FREE PUMP DOWN AND VENTING OF UHV VACUUM SYSTEMS
K. Zapfe and J. Wojtkiewicz, Deutsches Elektronen Synchrotron DESY, D-22607 Hamburg

A CLEAN PUMPING AND VENTING SYSTEM FOR SRF CAVITIES AND CRYOMODULES
S.M. Gerbick, M.P. Kelly, Argonne National Laboratory, Argonne, IL 60439, U.S.A.

ACHIEVING THE FINAL GOAL…

CLEAN PARTS →

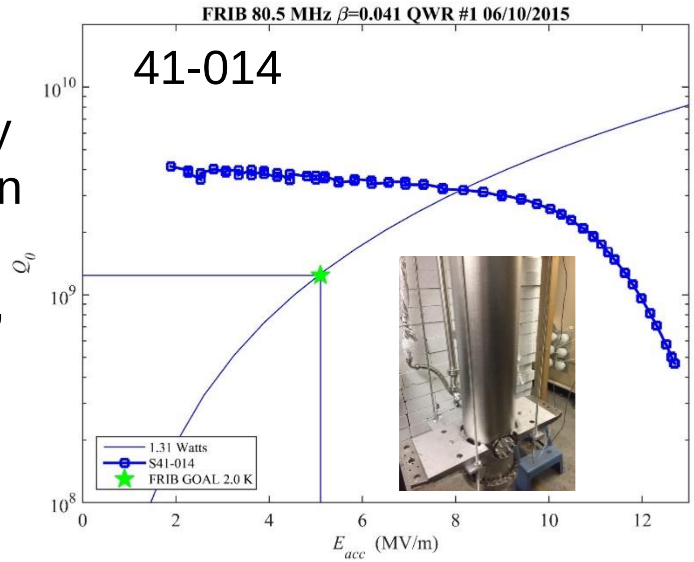


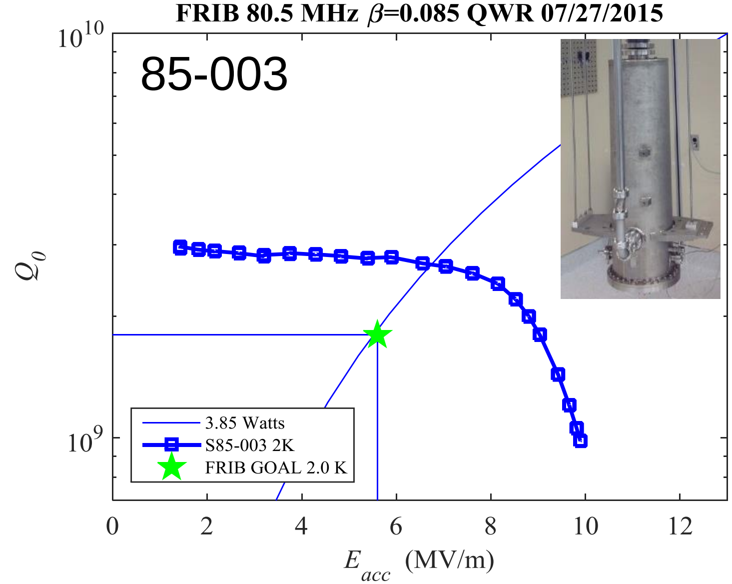
CLEAN CERTIFIED CAVITY

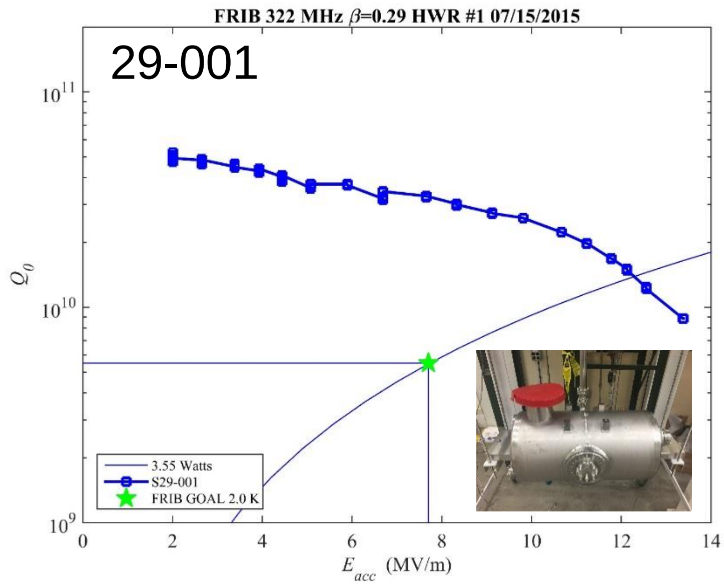
COLDMASS

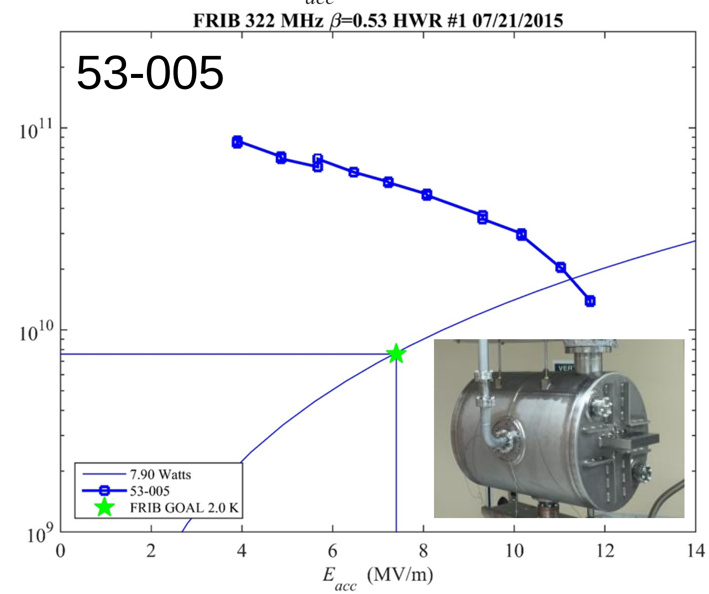
Cavities Exceeding Performance Specification…

Production articles of all four FRIB cavity types have been certified for use in cryomodules, following standard processing procedures in the new SRF Highbay. The results shown indicate specifications are met with margin.





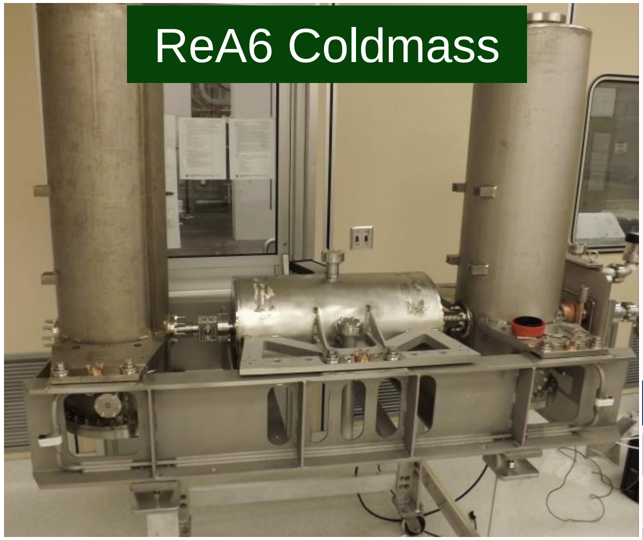




And High Performing Cryomodules!

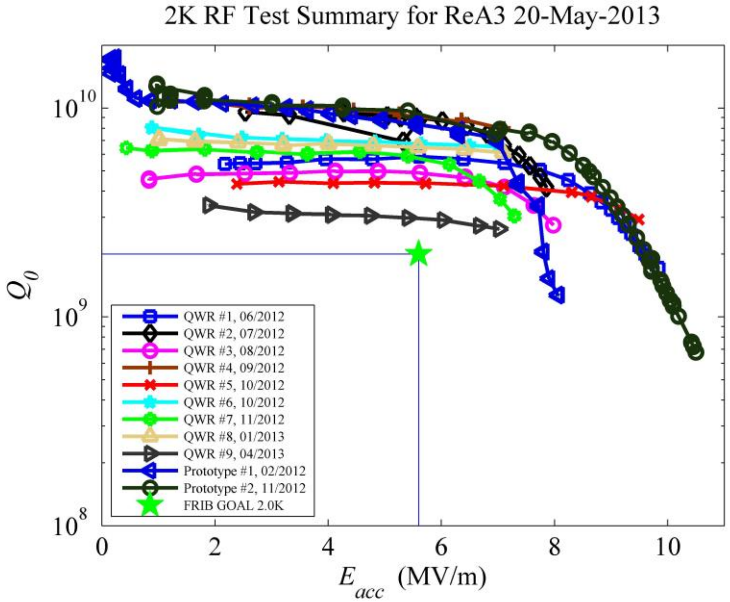
- Cavities capped in cleanroom several months until full string is complete and evacuated
- ReA3 and ReA6 cryomodules performance support method of storage

ReA6 Coldmass



ReA3 Coldmass

	Gradient
2 K test	E_a (MV/m)
Measured QWR 1	6.2
Measured QWR 2	6.2
FRIB goal 2 K	5.6



System performance excellent. Operation reliable within specifications

- QWR performance very good, no x rays, large margin in E_a
- Resonators and cryomodule mechanical stability excellent

Final Comments

- Cavity cleaning and surface preparation is critical to SRF accelerator performance!
- Always take care to protect the cavity surfaces at all steps, one tiny mistake can cause detrimental outcome to cavity performance.
- The baseline techniques are developed for FRIB processing and cleanroom assembly…
- **however much more can be learned during the FRIB production run and shared for future accelerators.**

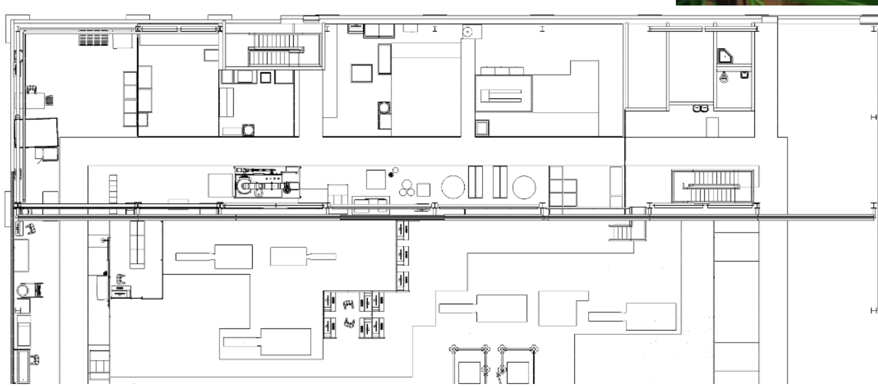
FRIB Solution to Process and Assemble Low Beta Cavities

- Low beta cavity shapes are complicated to process for mass production
- Consequently, specific processing and assembly equipment is required for these cavities
- Key elements for cavity production and assembly include that infrastructure and processes are
  - Cost effective → keep project costs on budget
  - Reliable → Be effective to deliver specifications
  - Repeatable → Keep reworks low & high quality

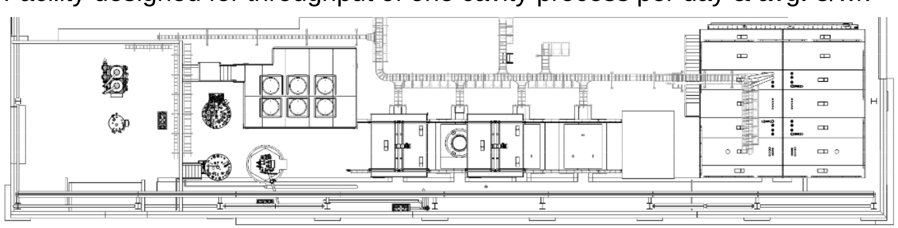
Production Inputs

Cold mass components →

Resources →



Facility designed for throughput of one cavity process per day & avg. 3/wk

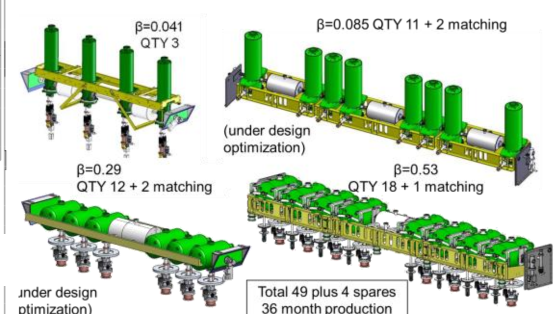


High bay floor ~ 23,400 ft^2

Cavity QA & Inspect= 1,400 ft^2
Furnace Area = 1,065 ft^2
Cleanroom Prep= 1,700 ft^2
Process Support= 1,050 ft^2
Chemistry facility = 535 ft^2
Processing CR= 2,500 ft^2
Cold mass CR = 1,440 ft^2
VTA/CMTF= 5,100 ft^2
Cryogenic systems= 1,800 ft^2

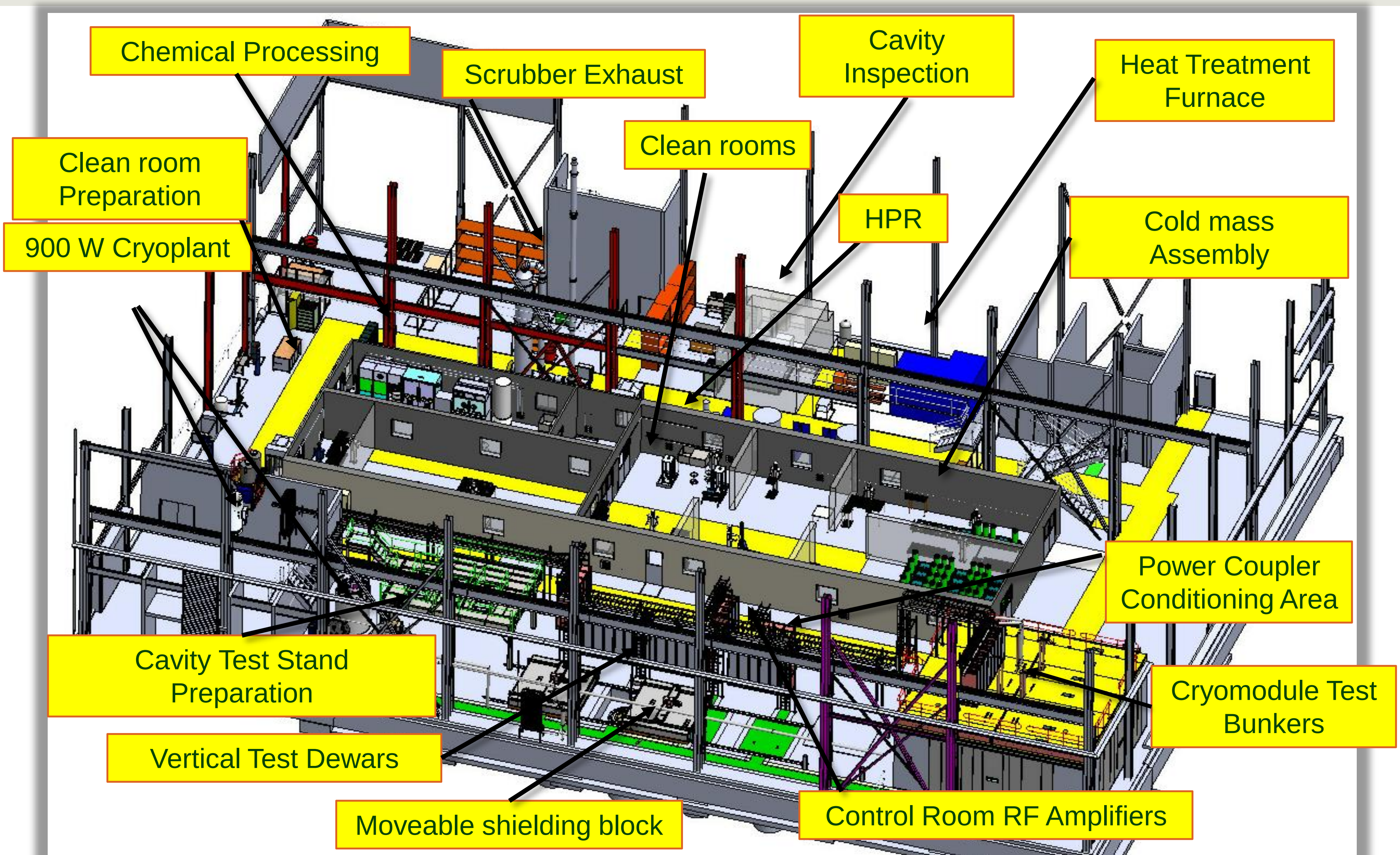
Production Outputs

→ Cold masses



→ Certified Cryomodules

SRF Low Beta Processing & Cleanroom Facility for Production



Acknowledgement

- Thanks to Kenji Saito for providing me with content for this lecture, especially for electropolishing sections
- Thanks to staff from the FRIB Cryomodule Department, Cavity Processing and Coldmass Assembly Group for providing pictures and information

Thank you for your attention!

THE END

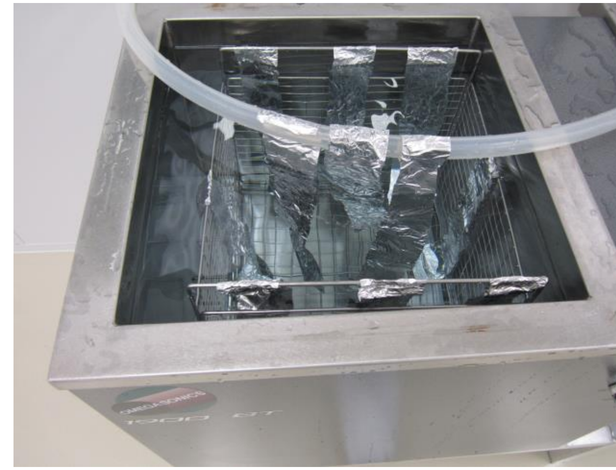
APPENDIX

Many Critical Steps to Deliver Quality RF Surface & Beamline

	Major Work Flow Step	Why?	Environment
INSPECTION	1. Receive and Tagging	track production floor cavity	highbay
	2. USC Degrease #1 - 1 hr, 140 C	Remove gross contaminations and grease films	highbay
	3. Salt Water Soak - over night	Expose any foreing inclusions like iron	highbay
	4. Cavity Drying	Remove water droplets and vapor	highbay
	5. Surface Checks and Boroscope	Inspect welds, critical surfaces	highbay
	6. Dimensional Measurements	Ensure cavity dims & surfaces meet tolerance	ISO 8 cleanzone
	7. Frequency Check #1	Ensure cavity withing tuning range tolerance	highbay
	8. Cold shock/Leak Check #1	Ensure welds & seals are good for UHV	highbay
	QA Inspect #1	Hold for document review	
BULK PROCESSING	9. USC Degrease #2 - 1 hr, 140 C	Remove contaminations and grease films	highbay
	10. Bulk Etch (120-150 MICRONS)	Remove internal damage layer and contamination	chemistry lab
	11. Frequency Check #2	Define differential etch removal quantity	highbay
	12. Differential Etching (~10-80 microns	Tune cavity to desired frequency	chemistry lab
	13. Cavity Drying	Remove water droplets and vapor	ISO 7 cleanzone/highbay
	14. Leak Check #2	Ensure weld integrity after chemical etching	highbay
	15. USC Degrease #3	Remove contaminations and grease films	highbay
	16. Heat Treatment Degas	Move hydrogen from surface into bulk Nb	ISO 7 cleanzone
	17. Cold Shock/Leak Check	Ensure weld integrity after heat treatment	highbay
	18. Frequency & coupling check	Ensure desired frequency	highbay
	QA Inspect #2	Hold for document review	
FINE PROCESSING	19. USC Degrease Cavity #4	Remove contaminations and grease films	highbay
	20. Light Etch (10-30 microns)	Remove oxide layers and any contamination	chemistry lab
	21. High Pressure Rinse	Remove particulate from internal surfaces, ports	ISO 5 cleanroom
	22. Clean Cavity Test Stand Assembly	Hermetically seal all ports to RF test stand, leak check	ISO 5 cleanroom
	23. Low Temperature Bake	Degrease high field Q-slope and reduce MP	highbay
	24. Dewar Test (vertical test)	Certify cavity perforamnce at real op conditions	highbay
	Final QA Inspect	Hold for document review	
	25. Install Cavity to string	Final clean assembly to coldmass	ISO 5 cleanroom

Ultra Sonic Cleaning Effective

- Cleaners commissioned using foils and part cleaning checks
- Smaller tanks (40 gal & 90 gal) for vacuum components, couplers, tuning plates
- E-1 water replaced for each batch
- Special baskets required
- Items must be submerged for cleaning & trapped air must be released.
- Placement of parts in cleaner is important
- Must be cautious of knife edges, sealing surfaces and RF surfaces
- Fixtures required for items that cannot be placed into baskets: cavities, solenoids

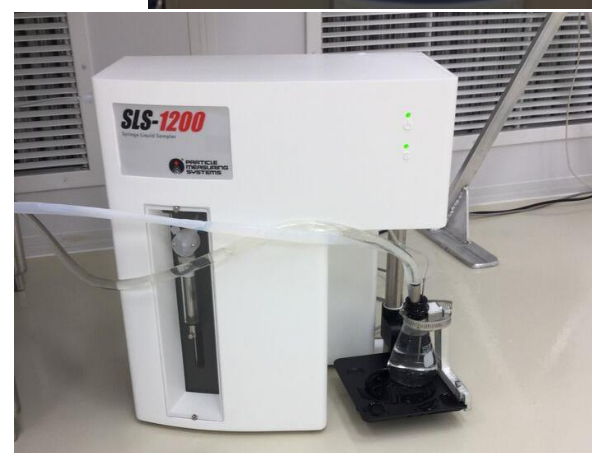


Commissioning USC with foil

Parts baskets

UPW System Testing

- Total silica: < 0.5 μg/L
- Total organic carbon (TOC): < 15 ppb
- **Resistivity: 18.1 Meg-Ohm - cm**
- **Liquid particle counts: < 20 particle/ml at 0.3 μm**
- Bacteria: < 3 CFU (colony forming unit)/100mL
- Flow rate: 12 gpm
- Temperature: ~74 ºF (23.3 ºC)
  - Keep water < 75 F by heat exchanger to reduce bacteria growth
- Maintenance important to keep quality
- Annual system sterilization



HPR Design Considerations and Cleaning Variables

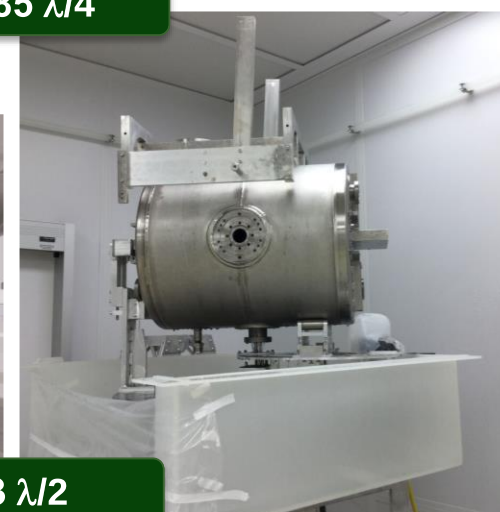
- Pressure → 1000-1500 psi at point of use
- Flow → 1-4 gpm E1 water with POU filtration down to 0.5 microns
- Pure gas over pressure used to reduce entry of particle into cavity from CR
- Alignment → Achieved by high tolerance fixtures, cameras, mirrors or automated alignment systems
- Interface → Important that the wand/nozzle assembly does not interfere with the cavity RF surface, scratch or dent
- Motion → Generally a combination of rotation and translation with cavity tooling to deliver water through a wand/nozzle assembly. Program to avoid spiral affect
- Materials → Cleanroom and E-1 water compatible, low friction
- Quality → Portion of rinsate is actively drained to liquid particle counter.
  Base E-1 counts taken before HPR process and system purged prior to set-up
- Duration→ Depends on cavity type, 2-3 hours or ~6 s/in^{2}, may require longer time if LPC are high or cavity is very large
- Post-Rinse → Cavity to dry in ISO 5 cleanroom, away from all movement or people

FRIB QWR and HWR HPR Set-ups


β=0.041 λ/4


β=0.085 λ/4


β=0.29 λ/2

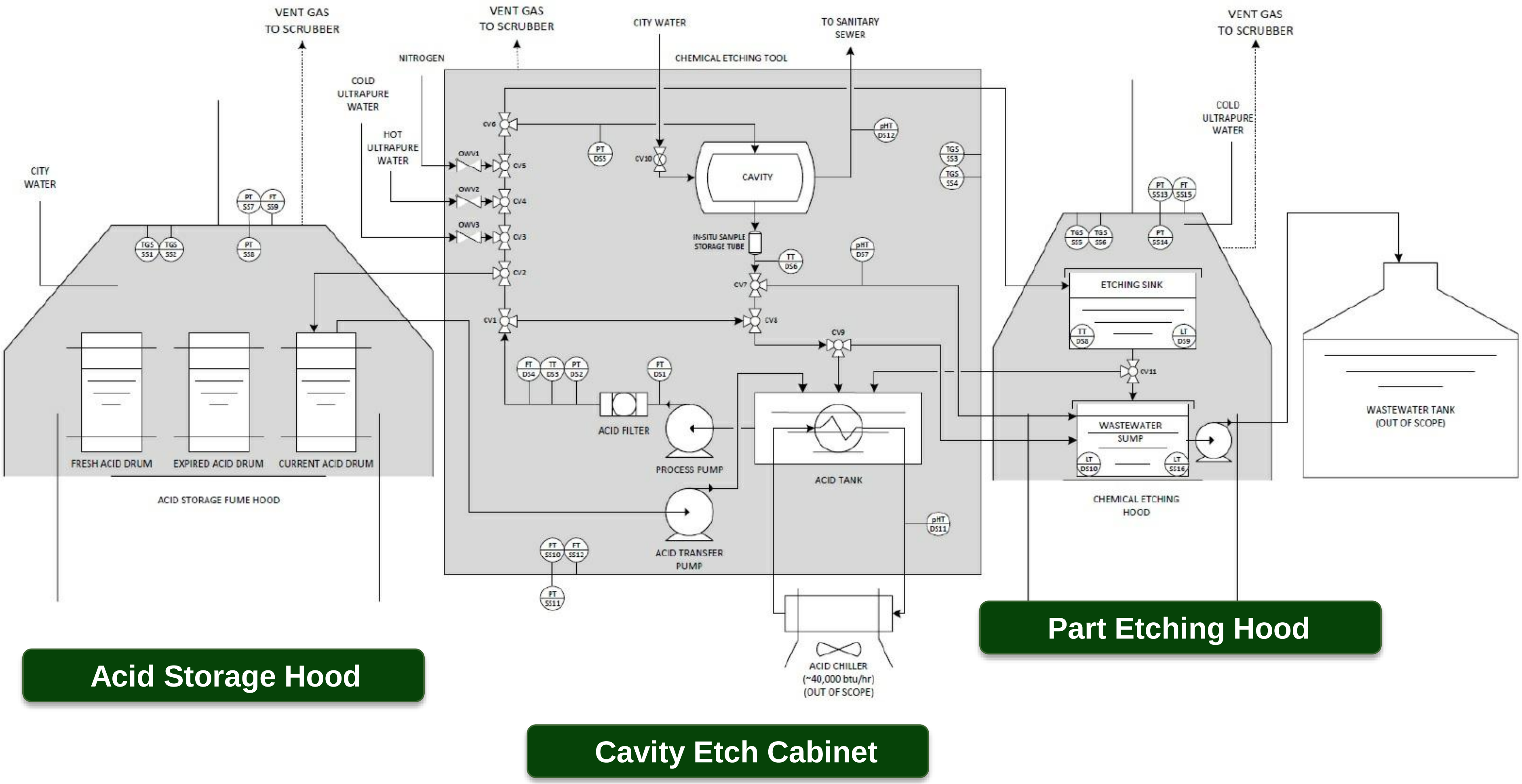



β=0.53 λ/2

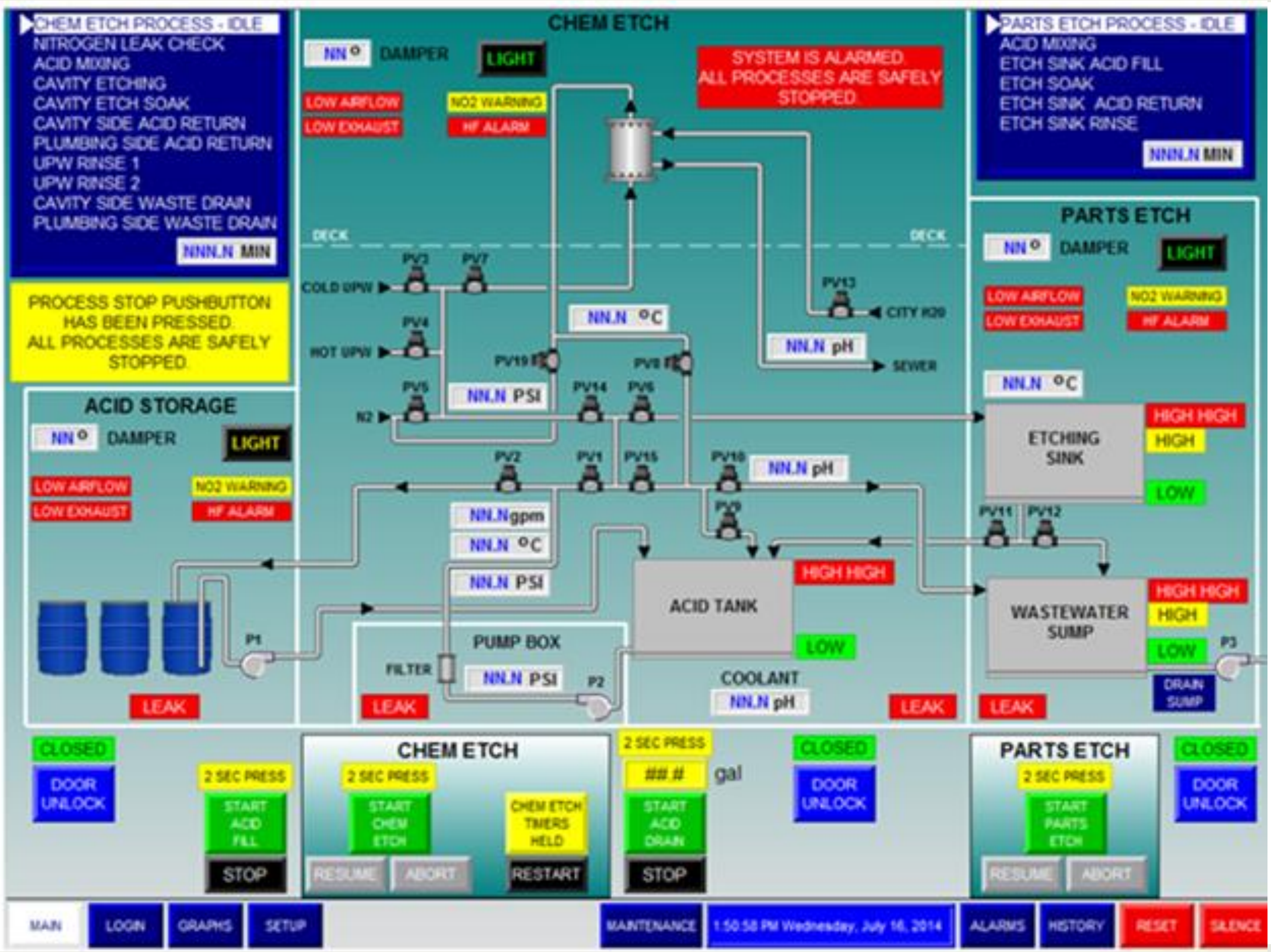
MSU BCP Facility Evolution 2000-2015

2000 R&D **2002** Small project **2014 Production**

Production Chemistry Diagram



Human Machine Interface for Etching Tools



BCP Equipment Mechanical Design Considerations

- **Plumbing:** usually closed loop and use acid pumps to move fluids, mixing process and an etching process
- **Long term material compatibility**: with BCP mixture is limited to PVDF and PTFE → all wetted parts must be compatible
- **Other materials:** are compatible for limited duration for ancillary use such as transfer hoses, tooling, containment
- **BCP:** mixture is procured from industry in pre-mixed 55 gal drums
- **Optimize pipes:** to reduce stagnant fluids and mixing water and acid, good drainage
- **Instrumentation:** Active filtration, flow rate, temperature and pressure sensors, heat exchanger/s (acid cooling), pH, gas sensors
- **Waste:** spent acid tanks, acidic rinse water tanks, or neutralization systems
- **Containment:** Secondary and tertiary containment for tanks and plumbing for leaks and/or spills. Pallets, double pipe, tanks
- **Ventilation:** Negative pressure required to remove toxic vapors and byproduct. Usually a chemical scrubber is designed for typical process

SRF Chemistry Facility Training Requirements

- SRF cavity preparation safety governed and approved by Environment Health and Safety (*)
  - MIOSHA Hazardous Work in Laboratories Standard
  - Michigan State University Chemical Hygiene Plan
  - SRF Chemistry Facility Training

Chemical Hygiene Plan

Michigan St

Office of Radiation, Che
C-124 Research C
East Lansing,
517-

Nove

FRIB-M30500MA-000043-R001

FRIB

SRF Processing & Coldmass Assembly Group Chemistry Facility Training

Laura Popielarski, Chemical Engineer

National Superconducting Cyclotron Laboratory (NSCL)

Office of Environmental Health and Safety (EHS)

Michigan State University, East Lansing, Michigan

Rev 3. 03-11-2013

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Training Requirements

	Requirement	Level I visitor	Level II observer	Level III research aide on BCP	Level IV assistant operator	Level V lead operator
1	Attend SRF Chemistry Room Training Presentation					
2	Complete *EHS Chemical Hygiene & Laboratory Safety/Hazardous Waste* (online)					
3	Read *MSU FRIB Chemistry Facility Handbook*					
4	Read MSDS sheets and Honeywell Document					
5	Read *BCP and HF Safe Use and Accidental Exposure Procedure*					
6	Read *BCP Exposure Emergency Response Plan* and Emergency Kit Contents					
7	Read *Sodium Hydroxide Use and Exposure Procedure*					
8	Read *SRF Chemistry Room Exposure Quick Reference Guide*					
9	Watch Chem Room First Aid Presentation by W. Smith Chandler, MMPH, MS, FACOEM, Jefferson Lab					
10	Complete *ChemMax3 Export Control Training*					
11	Complete *Personal Protective Equipment Training*					
12	Complete Chemistry Facility Introduction & Basic Tour					
13	Complete Chemistry Facility on-the-job Tour					
14	Complete Chemistry Facility on-the-job training					
15	Issued (1) 25 g tube of Calcium Gluconate 2.5% made by Pharma Science, MSDS and BCP Exposure Emergency Response Instructions					

DCC: S30105-MA-000053

DCC: S30105-PR-000082

DCC: S30105-PR-000082

DCC: S30105-PR-000083

DCC: Z00000-EX-000058

DCC: S30105-PR-000083

Chemistry facility is access level controlled, and multi-step training is required

HF Quick Reference Response Guide

Treatment of Hydrofluoric Acid (HF) Exposure

Quick Reference

NOTE: In addition to the usual medical history, the physician should obtain the following information: concentration of HF, date and time of exposure, duration of exposure, how exposure occurred, body parts exposed/affected, first aid measures instituted (what, when, how long). Injuries due to dilute HF solutions or low concentrations of vapors may result in delays in clinical presentation up to 24 hours following exposure.

	SKIN BURNS		EYE EXPOSURE	INHALATION		INGESTION
FIRST AID	**CONCENTRATED HF** Water Wash THEN Iced Benzalkonium Chloride* 0.13% Soaks OR Calcium Gluconate 2.5% Gel	**DILUTE HF** Water Wash THEN Iced Benzalkonium Chloride* 0.13% Soaks OR Calcium Gluconate 2.5% Gel	**ALL HF** Water Wash OR Saline Wash	**CONCENTRATED HF** Oxygen AND 2.5% Calcium Gluconate[4] by Nebulizer	**(Mild Exposures) DILUTE HF** Oxygen THEN Consider 2.5% Calcium Gluconate[4] by Nebulizer	**ALL HF** DO NOT INDUCE VOMITING Milk or Water THEN Milk of Magnesia OR Mylanta®+
MEDICAL TREATMENT	**CONCENTRATED HF** Debride (if necessary) THEN Continue Soaks OR Calcium Gluconate 2.5% - 5% Injection[2,4] AND Observe for/Treat Systemic Effects[3] (especially if > 25 sq. in.)	**DILUTE HF** Debride (if necessary) THEN Continue Soaks OR Calcium Gluconate 2.5% Gel OR Calcium Gluconate 2.5% - 5% Injection[2,4] Systemic Effects[3] Unlikely	**ALL HF** Topical Tetracaine Hydrochloride THEN 1% Calcium Gluconate Irrigation[4] AND Consult Opthamologist	**CONCENTRATED HF** Continue Calcium Gluconate by Nebulizer Observe and Treat for Respiratory Distress, Bronchoconstriction, Pulmonary Edema, Systemic Effects[3] (Inhaled Steroids and/or Bronchodialators as Needed)	**DILUTE HF** Continue Calcium by Gluconate Nebulizer Observe Serious Effects Unlikely Inhalation of HF Fumes from Diluted Acid is Uncommon	**ALL HF** Lavage with Calcium Chloride or Calcium Gluconate AND Treat Systemic Effects[3]

1. This is a brief summary of First Aid and Medical Treatment measures. The text of the brochure "RECOMMENDED MEDICAL TREATMENT FOR HYDROFLUORIC ACID EXPOSURE" must be consulted for more complete information.

2. 2.5% calcium gluconate injections must be used if the soaks or gel do not significantly relieve pain in 30-40 minutes. Injections may also be used as the primary treatment, especially for larger and/or deeper burns.

3. Systemic effects include hypocalcemia, hypomagnesemia, hyperkalemia, cardiac arrhythmias, and altered pulmonary hemodynamics. TREATMENT includes cardiac monitoring, monitoring serum calcium, fluoride, magnesium, and electrolytes; administration of IV calcium gluconate, correcting magnesium and electrolyte imbalance, and, in extreme cases, hemodialysis.

4. Calcium gluconate is normally supplied in ampules containing 10% calcium gluconate. Concentrations less than 10% are obtained by diluting with normal saline.

* Benzalkonium chloride is a high molecular weight quaternary ammonium compound available as Zephiran® a Registered Trademark of Sanofi Pharmaceuticals, New York, NY 10016
+ Registered trademark. Johnson & Johnson - Merck. Fort Washington, PA 19034

For additional reference charts or information on properties, storage and handling, or medical treatment for hydrofluoric acid, contact:

Honeywell Specialty Materials
101 Columbia Road
Morristown, NJ 07962

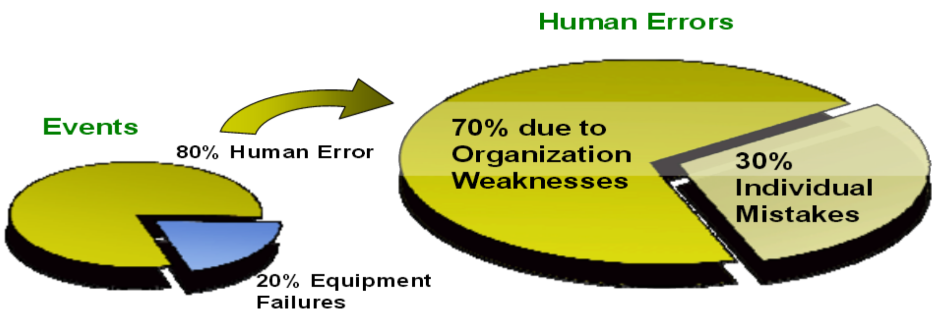
In the event of a medical emergency with this product, call the 24-hour Honeywell emergency telephone number:
800-498-5701

This foldout chart is also available as a laminated 15" x 23" wall poster.

Honeywell

Review Human Factors Early in the Design and Procedure Development

- End-users involved with equipment design and commissioning
- Review the procedure with your assistant before performing tasks, even if it has been done often, safety briefing
- Second independent verification on critical tasks, settings
- Reduce interruptions when performing critical tasks
- Post ‘do not distract’ or ‘do not disturb’ signs
- Carefully review Change Orders/Process Changes
- Employees stop work and ask questions if conditions change
- Clearly communicate and check understanding
- Maintain, inspect and test equipment



Challenges with Electropolishing

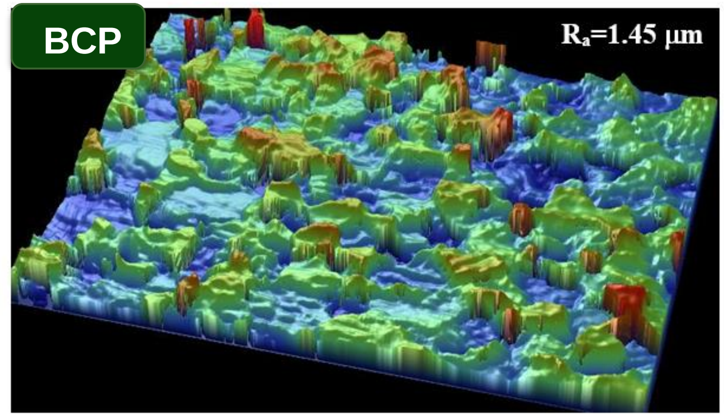
- Etch rate slower than BCP (0.5 μm/min versus 2 μm/min)[3] [5]
- Sulfur surface contamination, ultrasonic rinsing in H_2O_2 [5], ethanol rinse or other detergent cleaning required
- Hydrogen production
  - Can cause Q-disease inside cavity, surround cathode with Teflon cloth [5]
  - Hydrogen gas must be diluted outside of cavity with nitrogen to 4% (Lower Flammability Limit) [7]
- Bake cavity at high temperatures 100 ° C [1] to 800 ° C [5] to remove hydrogen
- Possible problems with multipacting [3]
- Ways to prolong life of acid solution [6]
- Acid replacement within cavity structure [7]
- Sometimes poor electropolishing around equator of elliptical cavities, need special cathode shapes
- Other areas of oscillation, etching pits on surface, and bubble traces on surface [8]

Benefits of Electropolishing (EP) over Buffered Chemical Polishing (BCP)

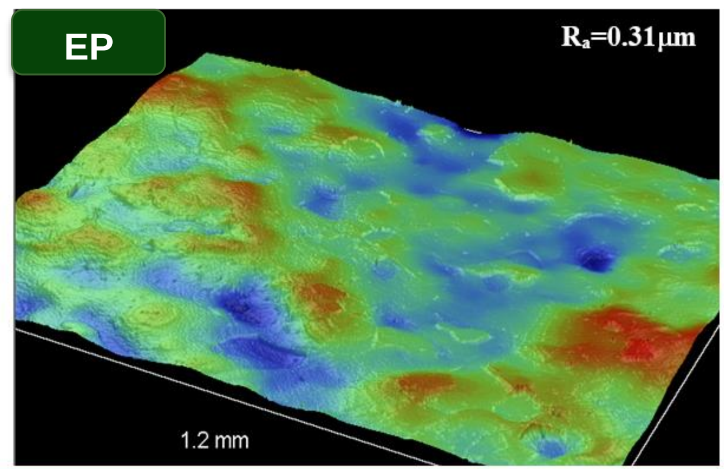
- Increases gradients, up to 40 MV/m, fundamental limit [6]
- Reduces grain boundary steps (1 µm versus 5 µm for BCP) [1]
- Decreases Q-slope appearance (BCP Q-slope @ ~25 MV/m, EP Q-slope @ ~35 MV/m, and 40 MV/m with ~100 °C bake-out) [1]
- Can use niobium material with RRR=200 [1]
- Bright and smooth surface [3]
- Can decreases field emissions [3]
- Goal should be < 2 µm surface roughness for best results [6]
- BCP may not improve surface after a depth of ~100 µm [10]

Electro Polishing Nb Samples – BCP versus EP Samples

Mag. 5.1 X
Nb Sample
BCP Etch
~ 60 µm
R_a = 1.45 µm



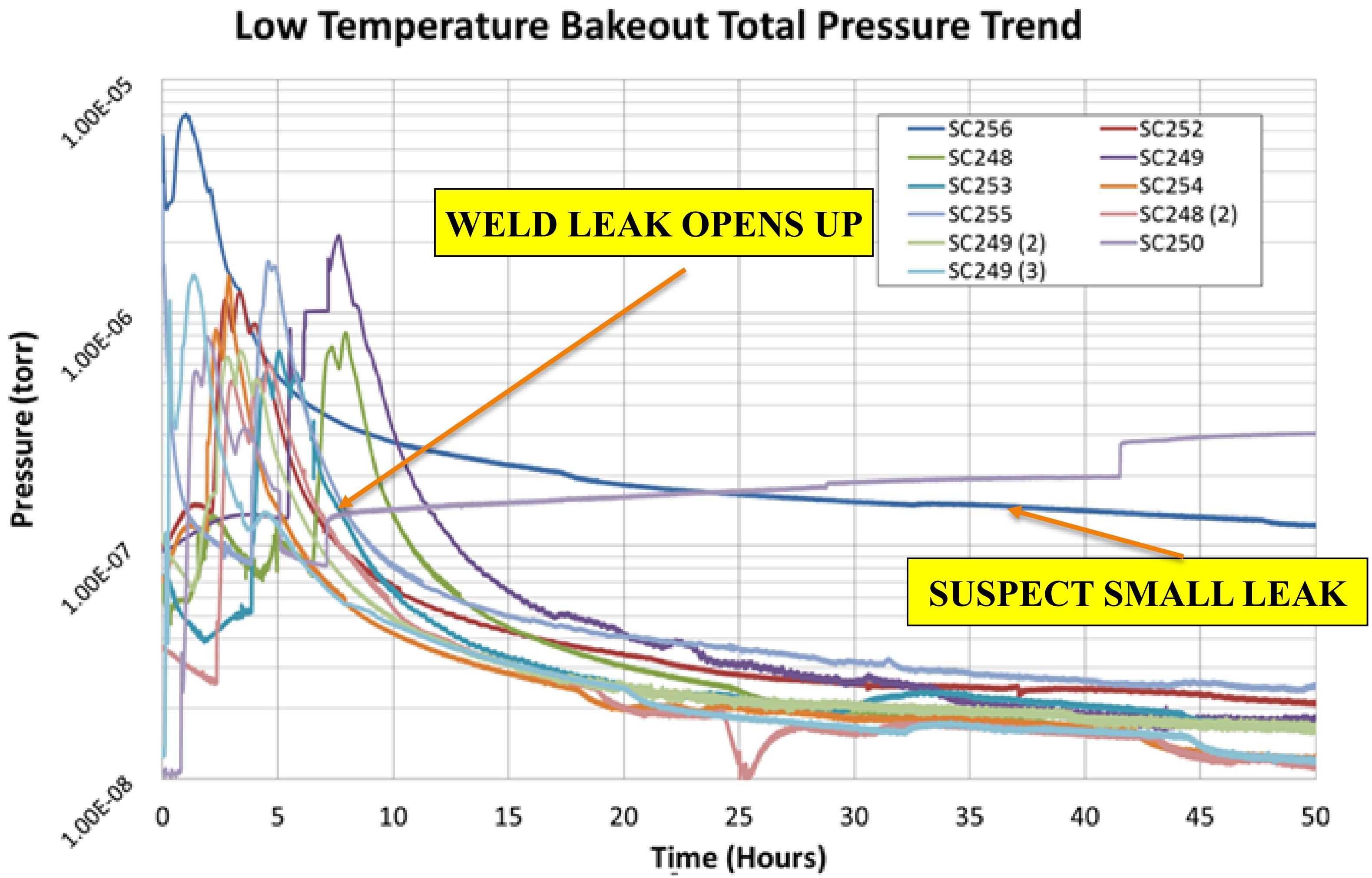
Mag. 5.1 X
Nb Sample
BCP Etch
~ 60 µm
+EP 144 µm
R_a = 0.31 µm



References for EP Section

- [1] Bhashyam, S. “Comparison of Electropolishing and Buffered Chemical Polishing – A Literature Review”, TD-03-046
- [2] Geng, R.L. “Continuous Current Oscillation Electropolishing and Applications to Half-Cells”
- [3] Lilje, L. “Electropolishing of Niobium Cavities”, Rissen 2002
- [4] Lilje, L. “Electropolishing of Niobium Mono-cell Cavities at Henkel Electropolishing Technology LTD. (Germany)”
- [5] Padamsee, H., “RF Superconductivity for Accelerators”
- [6] Saito, K. “Development of Electropolishing Technology for Superconducting Cavities”, PAC 2003
- [7] Schultz, E., “Engineering Solutions for the Electro-Polishing of Multi-Cell Superconducting Accelerator Structures”
- [8] Steinhau-Kuehl, N., “Basic Studies for the Electro Polishing Facility at DESY”
- [9] Steinhau-Kuehl, N., “Electro Polishing at DESY”
- [10] Xue, Q, “Modeling and Optimization of the Chemical Etching Process in Niobium Cavities”

Low Temperature Bake Pressure Trend for QWRs



What must stay clean?

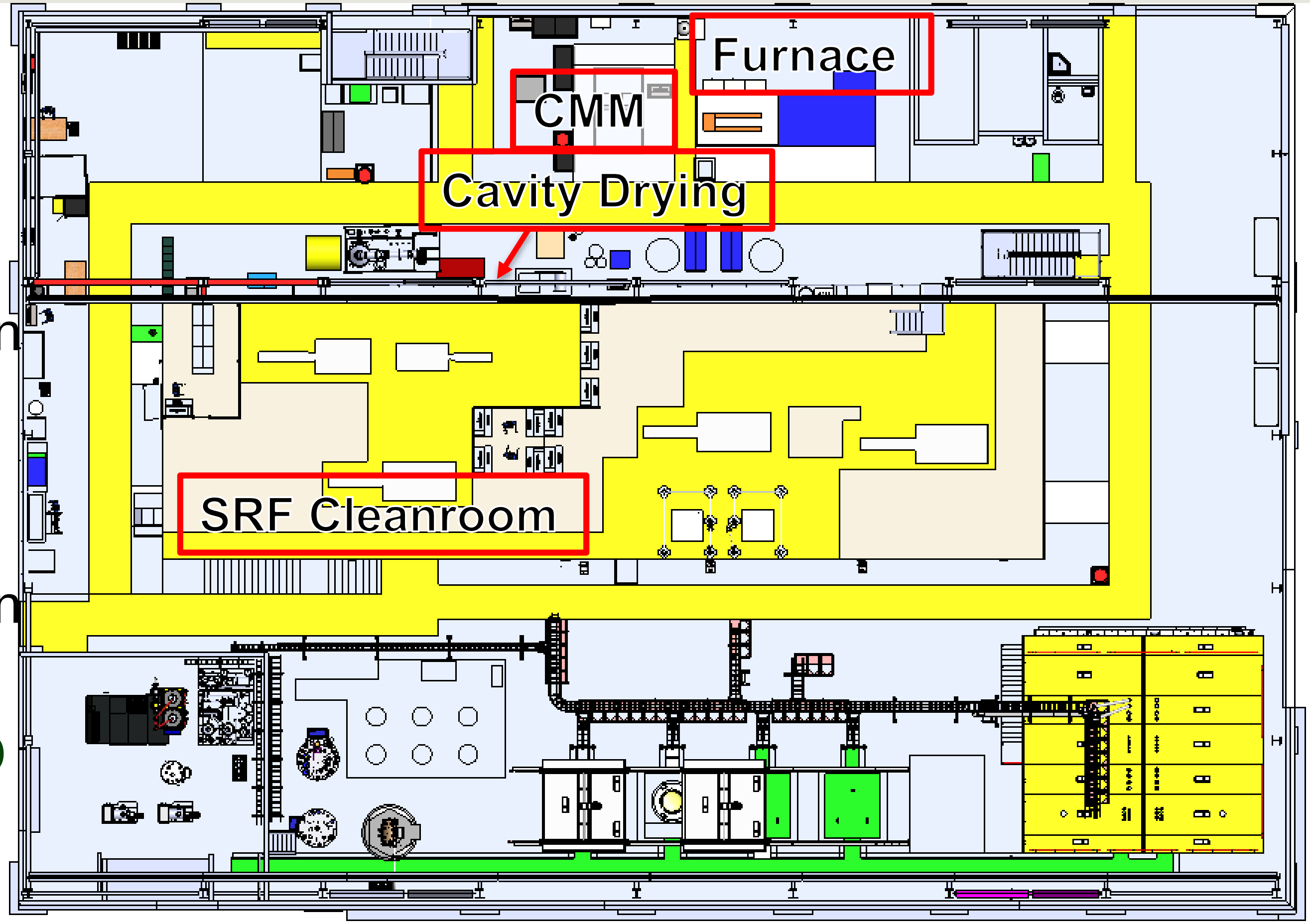
- All SRF surfaces
  - Niobium and all surfaces in the cavity vacuum space
- All cavity vacuum space items
  - Flanges, bellows, antennas, pressure gauges, and pumps
- All items that go into the cleanroom
  - People, tools, fasteners, cavities, fixtures, instruments, etc.
- Any item/fixture that contacts SRF surfaces or vacuum components
- Furniture, racks, carts, lifting fixtures…everything

What are the steps to prep cleaning?

1. Remove all tape
2. Disassemble assemblies to singular components
3. Wipe metal items with acetone until lint-free wipe remains white
   -Take care to get in every crevice and hole, especially dead tapped holes
4. Wipe or ultrasonic clean all items with 1% Micro90® Solution and rinse with E-4 water
5. Wipe all items with alcohol until lint-free wipe remains clean
6. Take cleaned item straight to cleanroom part entrance

Low Beta Facility Cleanroom Classifications

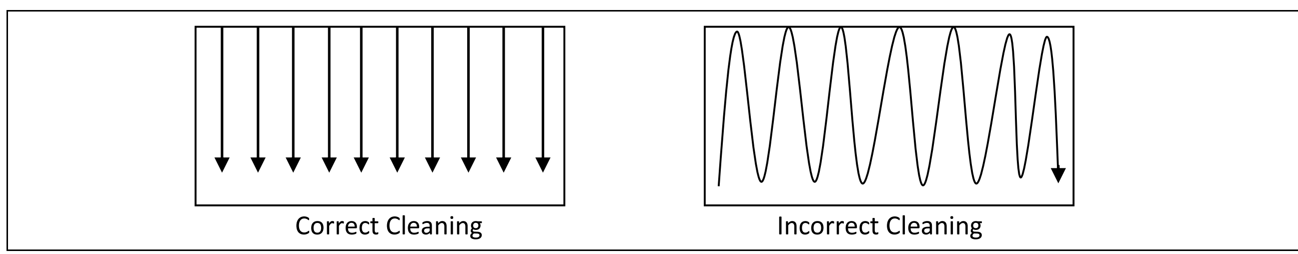
- CMM: ISO 8
  - Reduce dirt on CMM
  - Isolate area
- Furnace: ISO 7
  - Reduce contamination in furnace and on cavities
- Cavity Drying: ISO 7
  - Reduce contamination before furnace
- SRF Cleanroom: ISO 5-7
  - Eliminate contamination in cold mass



With good training, maintenance and cleanroom protocol better ISO <u>can be achieved</u>!

Cleanroom Maintenance for Certification

- Mop cleanroom with ultrapure water (UPW) on regular basis
- Clean gowning room and around assembly areas often
- Do not use powdered detergents
- Floors and horizontal surfaces are a priority when cleaning
- Keep log book of each cleaning



Controlling Human Contamination
Operational Rules

- Avoid natural fiber clothing
- Jewelry and watches should not be worn
  - Can tear gloves.
- Cosmetics should be limited
  - Causes: Outgassing, cross contamination
  - Avoid perfumes and colognes
- Use appropriate gloves for application
  - Cleanroom gloves not paper box gloves
- Wipe glasses clean before entering CR
- Food, drink, and gum not allowed
- Smoking not allowed in or near CR
- Wipe down down smart phones and other approved devices

Cleanroom Equipment

- Polypropylene, plastic or stainless steel (SS) furniture and equipment
- Furniture with non-shedding surfaces & free of scratches
- Shelves with open grate to minimize air turbulence
- CR dedicated tools, non plated tools SS preferred
- Filter exhausts from vacuum systems or cooling systems with HEPA filter, or exhaust outside of CR

SRF Component Cleaning Steps

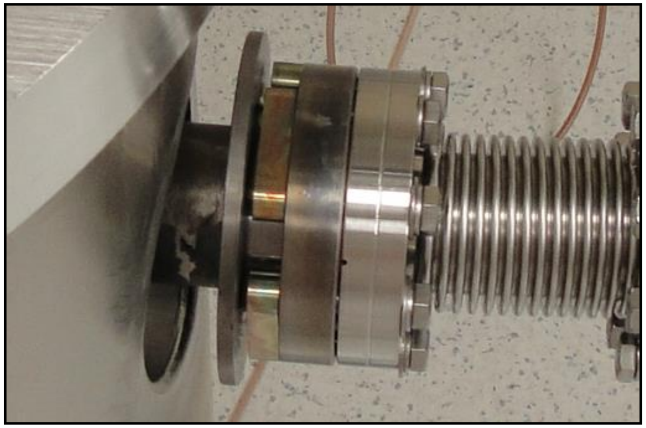
	Task	Location	PPE	Environment Classification	Materials	Outcome/Note
1. Degreasing procedure (in pink) may vary but still meet cleanliness and vacuum requirements	inspect, Degrease with Acetone, degrease with ethanol as needed	CR Prep Area	nitrile gloves, safety glasses	no cleanroom class	reagent grade acetone, low line kimwipes	remove gross contamination, oil, dirt, machine chips, marker, tape residue
	1 minute rinse with E-4 (RO/DI) water, inspect	CR Prep Area	nitrile gloves, safety glasses	no cleanroom class	DI water	
	60 min USC in 1% solution Micro 90 (OR Surface Cleanse) & E-4 DI water at 140 F, rinse with E-4 RO/DI water (RF surface first, then Beam line), inspect	CR Prep Area	nitrile gloves, safety glasses	no cleanroom class	Micro90, DI water, 200+ gal E-4	TOC < 200 ppb in rinse water
	60 min USC in 1% solution Micro 90 (OR Surface Cleanse) & E-4 RO/DI water at 140F, rinse with E-4 RO/DI water (fasteners)	CR Prep Area	nitrile gloves, safety glasses	no cleanroom class	E-4 water, surface cleanse, 10 gal USC	
2. Particle Elimination Procedure (in yellow) may vary from but still meets cleanliness and vacuum requirements	40 min USC in E-1 UPW at 140F for RF or beamline surfaces, inspect	Rail Prep CR	full cleanroom garmets	class 10,000	E-1 UPW, 20 gal, 200 gal	TOC < 200 ppb in rinse water, LPC < about 300 cumulative 0.3 micron sized/ml
	high pressure rinse at 1200 psi, 2gpm, with E-1 (UPW) for predetermined time ~ 5 sec per in^2 of surface area for SRF cavity RF surface	HPR WC	full cleanroom garmets, safety glasses	class 100 or better	E-1 UPW, 2 gpm	TOC < 200 ppb in rinse water, LPC < about 300 cumulative 0.3 micron sized/ml
	40 min USC in E-1 UPW at 140F for fasteners	Rail Prep CR	full cleanroom garmets	class 10,000	E-1 UPW, 20 gal USC	TOC < 200 ppb in rinse water, LPC < about 300 cumulative 0.3 micron sized/ml
	Rinse parts (non-RF cavity) with UPW for 1 minute each, inspect	Rail Prep CR	full cleanroom garmets	class 100 or better	E-1 UPW	TOC < 200 ppb in rinse water, LPC < about 300 cumulative 0.3 micron sized/ml
3. Drying Procedures (in orange) may vary from but still meets cleanliness and vacuum requirements	Place part in nitroge[illegible]cator and set to 4% RH	assembly CR	full cleanroom garmets, class 100	class 100	filtered dry nitrogen, desiccator	sub micron filtration <0.05 micron
	OR Dry in a [illegible] oven	[illegible]	[illegible]	[illegible]	class 100 table	
	spray part with 40-90 [illegible]ltered dry nitrogen in class 100 area, perform surface particle counts just prior [illegible] assembly/installation	assembly CR	full cleanroom garmets, class 100 certified	class 100	filtered dry nitrogen	Mil-Spec 1246C Table 1. Level 1
4. Packaging Procedure (in blue) may vary from but still meets cleanliness and vacuum requirements	Remove when RH 4% is reached and set on clean class 100 table	assembly CR	full cleanroom garmets, class 100 certified or equiv	class 100		
	inspect, Place component in class 100 certified poly bag, backfill with dry inert gas like nitrogen, fold edge over		full cleanroom garmets, class 100 certified	class 100	class 100 cert. poly bag	
	Place bagged componet with folded edge in another class 100 certified poly bag, back fill with dry nitrogen and seal		full cleanroom garmets, class 100 certified	class 100	class 100 cert. poly bag	
	label outside bag with class 100 certified label, with predefined information (date, PN, SN, initial of technician) (PN, SN on exterior packaging too)		full cleanroom garmets, class 100 certified	class 100	class 100 label system, tape	
5. MSU Vendor/Part Qualification Procedures	Inspect packaging	CR Prep Area	nitrile gloves	no cleanroom class		
	Demagnetize in packaging (needs review)	CR Prep Area	nitrile gloves	no cleanroom class	magnetometer, demagnetizer	all FRIB coldmass components < 15 milligauss
	Prepare package for cleanroom	CR Prep Area	nitrile gloves, safety glasses			
	Perform QIII surface particle counts of outside package	SRF Cleanroom	full cleanroom garmets, class 100 certified	class 10,000	QIII	required for validation testing only
	Perform QIII surface particle counts of interior package	SRF Cleanroom	full cleanroom garmets, class 100 certified	class 10,000	QIII	required for validation testing only
	Open exterior bag in class 100	SRF Cleanroom	full cleanroom garmets	class 100		
	Perform subcomponent particle counts	SRF Cleanroom	full cleanroom garmets	class 100	QIII	
	perform QIII surface particle counts on parts	SRF Cleanroom	full cleanroom garmets	class 100	QIII	Mil-Spec 1246C Table 1. Level 1
	Perform liquid particle counts on rinsate sample for part	SRF Cleanroom	full cleanroom garmets	class 100	LPC	total cumulative particle 0.3 micron and greater < about 300 particle/ml
	perform QIII surface particle counts on part post rinse	SRF Cleanroom	full cleanroom garmets	class 100	QIII	Mil-Spec 1246C Table 1. Level 1
	Assemble part to bake manifold, pump, leak check	SRF Cleanroom	full cleanroom garmets	class 10,000	manifold bake system	requirement varies by part
	Bake at 120-140 C for 24-48 hours, analyze RGA	SRF Cleanroom	full cleanroom garmets	class 10,000	manifold bake system	requirement varies by part

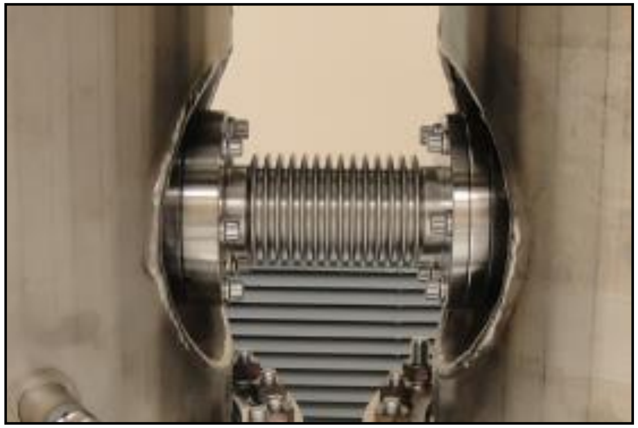
Final surface particle count

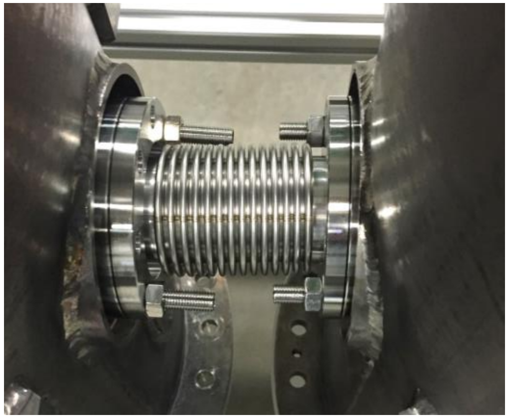
1. USC Degrease
2. USC particle removal
3. Drying and particle inspection
4. Clean packaging
5. MSU Vendor and part qualification

Issue: The beam line assembly

Close proximity vertical flange arrangement





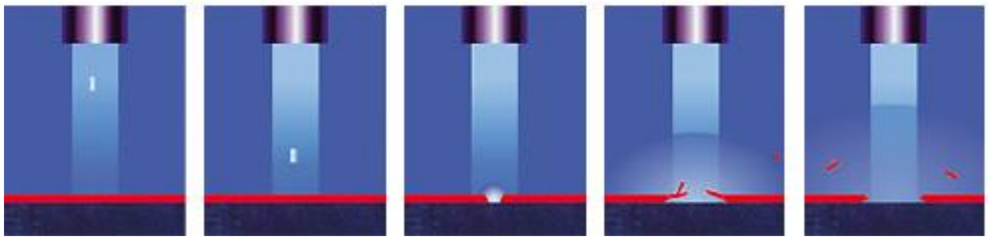


Particle Contamination Risk	Resolution
Human contamination	Follow protocol, minimize the number of workers*
Difficult to assemble vertical beam line seal	Use installation fixtures: clips, tape, wedge, etc.
Challenge to fit flexible coupling	Use compression and extension tools
Bolt & nut particulate	Perform work slowly
Bolt & nut particulate	Filtered nitrogen shower
Bolt & nut particulate	Make particle seal w/two bolts, turn nut*
Final module beam line connection in LINAC	Portable CR, fully gowned, particle counts before assembly < 100 – 0.5 μm/CF*

MEASUREMENTS ON PARTICLE CONTAMINATION DURING CAVITY ASSEMBLY#, F. Zhu, R. Bandelmann, T. Ebeling, N. Krupka, A. Matheisen, B.Petersen, D. Reschke, DESY, Notkestraße 85, 22603 Hamburg, Germany, Institute of Heavy Ion Physics, Peking University, 100871, Beijing, China

What is Dry Ice Blasting (Cleaning)?

Dry ice blasting is known by several names: dry ice blasting, dry ice cleaning, CO2 blasting, dry ice dusting, and even environmentally sustainable cleaning. Cold Jet dry ice blasting is an efficient and cost-effective way for industries to maximize production capability and quality. Dry ice blasting is similar to sand blasting, plastic bead blasting or soda blasting where media is accelerated in a pressurized air stream to impact a surface to be cleaned or prepared. But that's where the similarity ends.



Instead of using hard abrasive media to grind on a surface (and damage it), dry ice blasting uses soft dry ice, accelerated at supersonic speeds, and creates mini-explosions on the surface to lift the undesirable item off the underlying substrate. If you want to read all the technical details, see the How CO2 Blasting Works page.

Dry ice blasting:

•is a non-abrasive, nonflammable and nonconductive cleaning method
•is environmentally responsible and contains no secondary contaminants such as solvents or grit media
•is clean and approved for use in the food industry
•allows most items to be cleaned in place without time-consuming disassembly
•can be used without damaging active electrical or mechanical parts or creating fire hazards
•can be used to remove production residue, release agents, contaminants, paints, oils and biofilms
•can be as gentle as dusting smoke damage from books or as aggressive as removing weld slag from tooling
•can be used for many general cleaning applications

Cold Jet dry ice blasting uses compressed air to accelerate frozen carbon dioxide (CO2) "dry ice" pellets to a high velocity. A compressed air supply of 80 PSI/50 scfm can be used in this process. Dry ice pellets can be made on-site or supplied. Pellets are made from food grade carbon dioxide that has been specifically approved by the FDA, EPA and USDA.

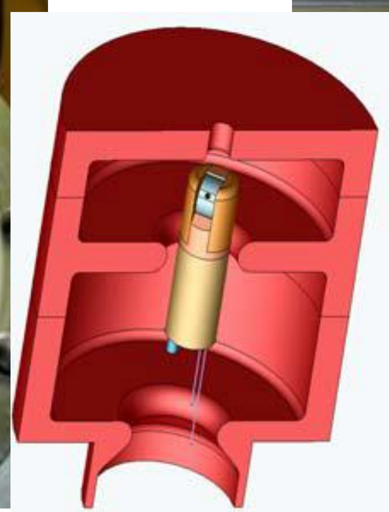
Carbon dioxide is a non-poisonous, liquefied gas, which is both inexpensive and easily stored at work sites.

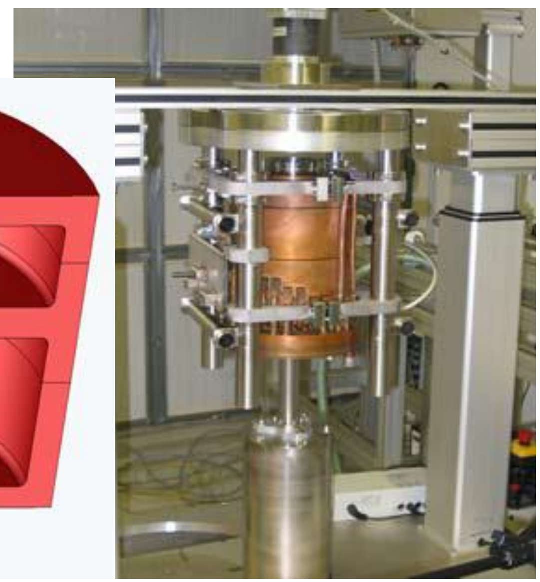
http://www.coldjet.com/en/information/what-is-dry-ice-blasting.php

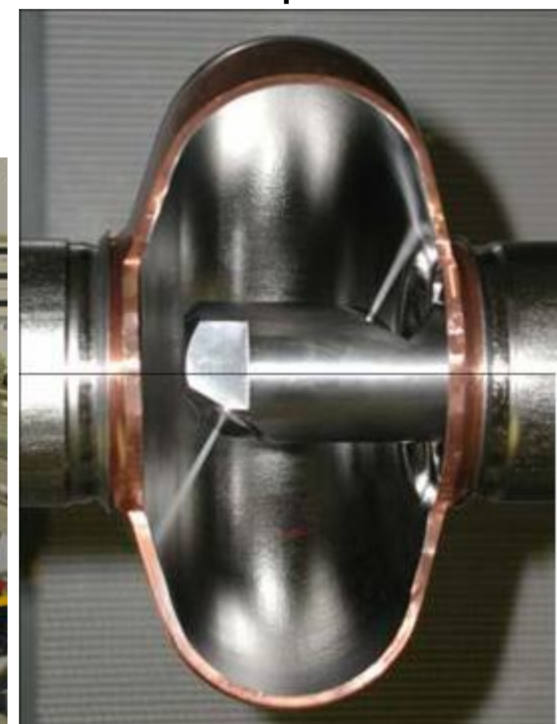
Dry-Ice Cleaning for SRF

- DESY: horizontal + vertical cleaning stands (presented at SRF 2007)
- Tasks: mostly cleaning of the copper RF gun cavity of the photo injector of FLASH + XFEL
  - Goal: effective removal of particle => with no oxidation of Cu
  - installation for horizontal cleaning of (1-3) cell cavities in reliable operation

  successful horizontal cleaning of Nb single-cells







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SRF Coldmass Component Workflow