FRIB

Cavity Processing and Cleanroom Assembly Review of Literature

Laura L. Popielarski

SRF2019 Tutorial No. 6
Friday, June 28th, 2019

MICHIGAN STATE UNIVERSITY

U.S. DEPARTMENT OF ENERGY | Office of Science

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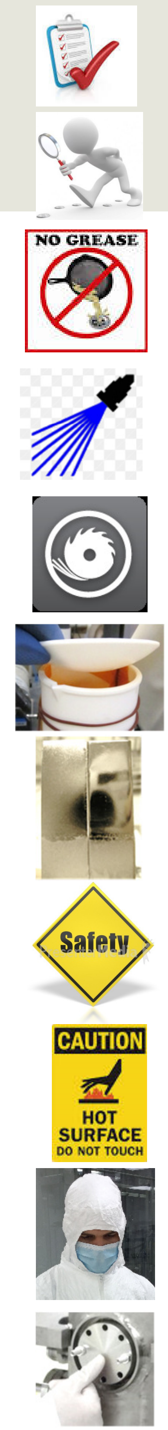
Preface

- Tutorial will introduce basics for SRF cavity processing and cleanroom techniques based on published processes
- 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
- References for each slide's content are shown on the slide for you to read up on further information
- A complete list of all references are located at the end

Sections

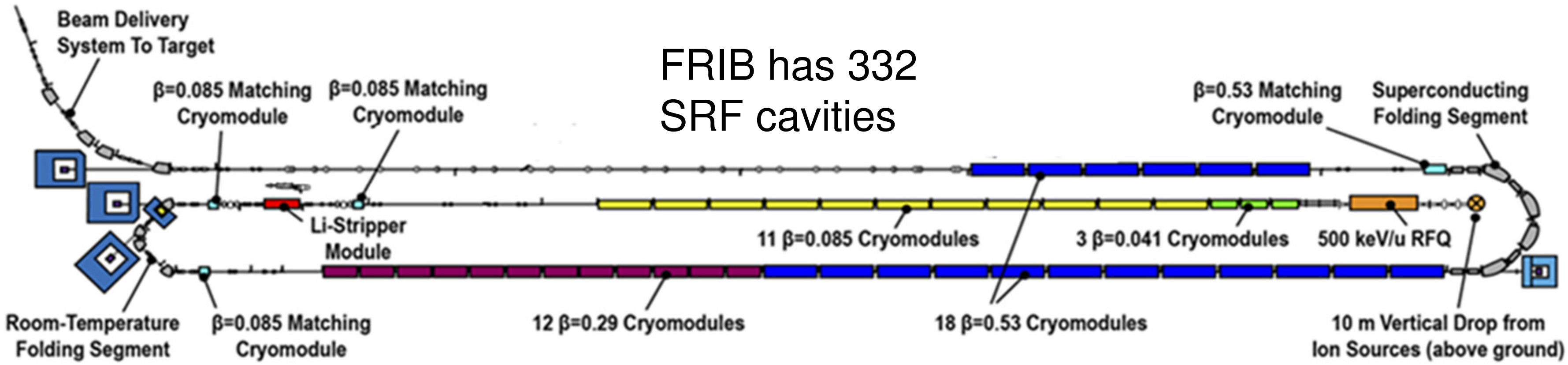
1. SRF coldmass and cavity workflow
2. SRF cavity receiving and inspection
3. Degreasing
4. Ultra pure water and high pressure rinse
5. Mechanical surface preparation
6. Removal by chemical etching
7. Preparation by electro polishing
8. Safety considerations
9. Heat treatments
10. Cleanroom protocols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



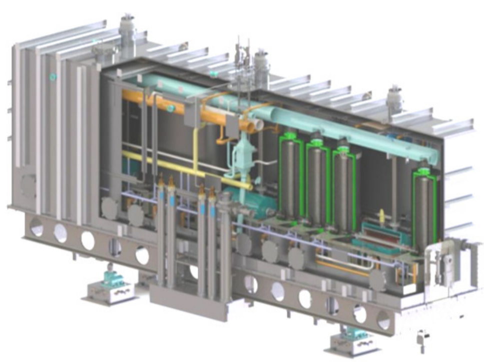
1. SRF Coldmass and Cavity Workflow

SRF Cavity Processing and Coldmass Assembly for LINACS

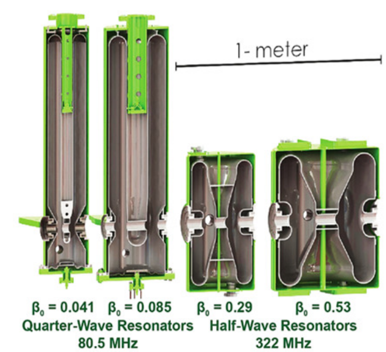
- SRF cavities are used for accelerators across the world
- Fabricated from SC material niobium metal which has T_c of 9.25 K
- Many cavities are required to construct accelerator systems ranging from tens to hundreds of cavities



FRIB has 332 SRF cavities



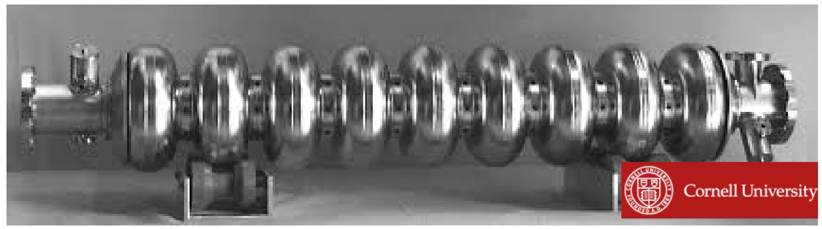
0.085 QWR Cryomodule



FRIB Cryomodule Design and Production, T. Xu, et.al, Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, USA M. Wiseman, Thomas Jefferson National Accelerator Facility, Newport News, VA, USA M. Kelly, Argonne National Laboratory, Lemont, IL, USA R. Laxdal, TRIUMF, Vancouver, Canada K. Hosoyama, High Energy Accelerator Research Organisation, Tsukuba, Japan 1also at INFN - Laboratori Nazionali di Legnaro, Legnaro (Padova), Italy. Proceedings of LINAC2016, East Lansing, MI, USA

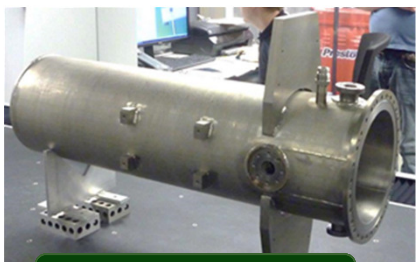
SRF Cavities All Shapes and Sizes

There are elliptical type cavities used for high-beta



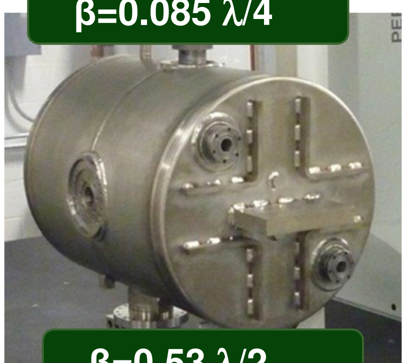
Design Topics for Superconducting RF Cavities and Ancillaries H. Padamsee, Cornell University, CLASSE, Ithaca, New York. www.lns.cornell.edu

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



β=0.085 λ/4

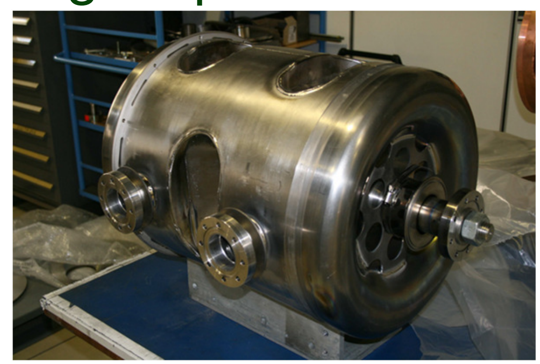
β=0.041 λ/4



β=0.53 λ/2

Superconducting Resonators Development For The FRIB And ReA LINACS at MSU: Recent Achievements and Future Goals, Facco, Proceedings of IPAC2012, New Orleans, Louisiana, U.S.A.

β=0.29 λ/2



β=0.12 λ/4

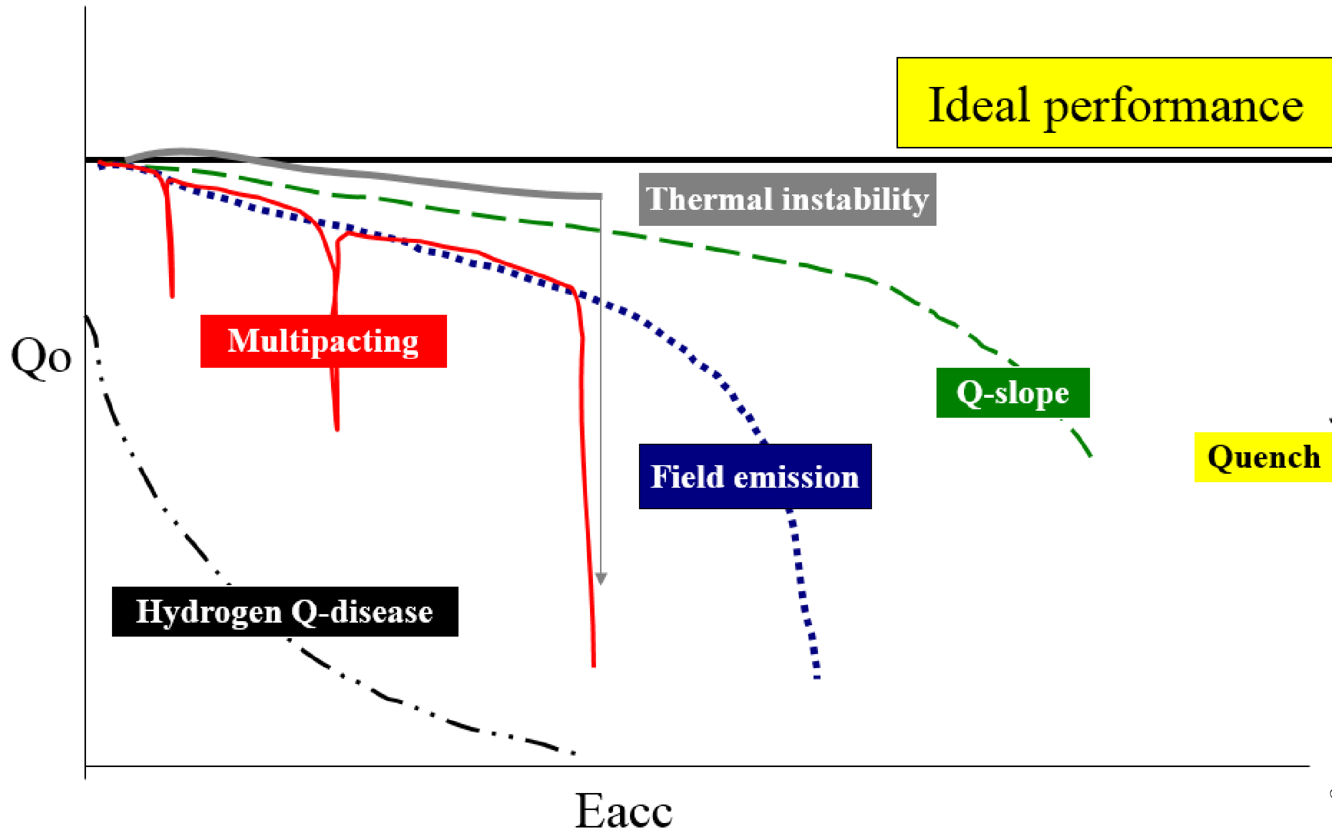
Development of a Beta 0.12, 88 MHZ, Quarter Wave Resonator and its Cryomodule for the Spiral 2 Project, G. Olry, J-L. Biarrotte, S. Blivet, S. Bousson, C. Commeaux, C. Joly, T. Junquera, J. Lesrel, E. Roy, H. Saugnac, P.Szott, CNRS/IN2P3/IPNO, Orsay, France Proceedings of the 12th International Workshop on RF Superconductivity

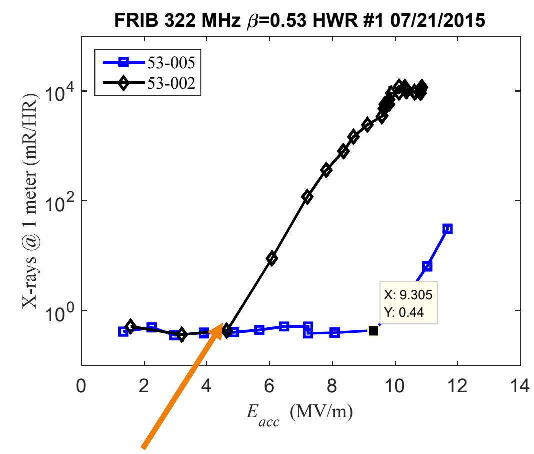
SRF Cavity Requirements to Achieve High Performance

- High RRR niobium material
- 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!**

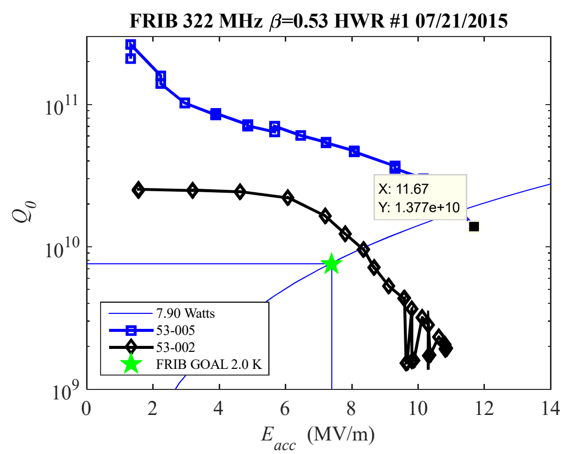
Published by CERN in the Proceedings of the CAS-CERN Accelerator School: Superconductivity for Accelerators, Erice, Italy, 24 April – 4 May 2013, edited by R. Bailey, CERN–2014–005 (CERN, Geneva, 2014). Design Topics for Superconducting RF Cavities and Ancillaries H. Padamsee, Cornell University, CLASSE, Ithaca, New York

Common Cavity Performance Limitations and Issues





FIELD EMISSION



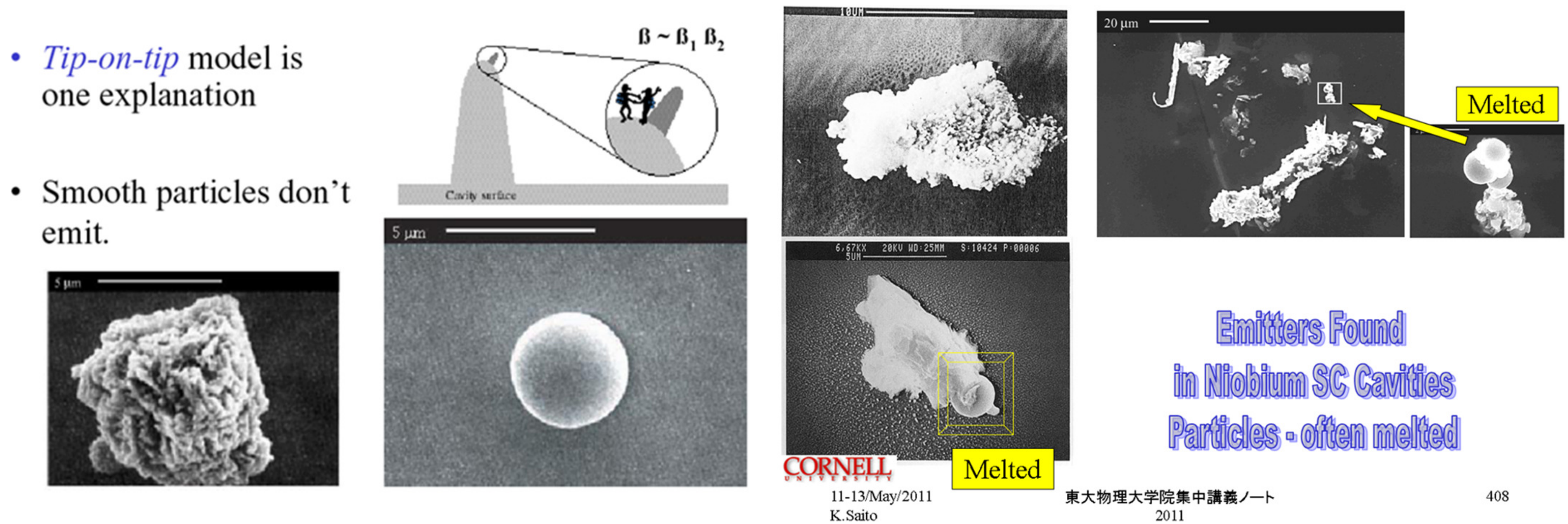
Kenji Saito, Lecture note in Tokyo University on May 2011

Limits in cavity performance Detlef Reschke DESY Tutorial, SRF 2007
https://accelconf.web.cern.ch/accelconf/srf2007/TUTORIAL/PDF/Tutorial_3c.pdf

H. Padamsee, "50 years of success for SRF accelerators — a review," Superconductor science and technology, vol. 30, p. 05003 (23 pp), 2017.

Fabrication and Processing Errors Cause Performance Problems!

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

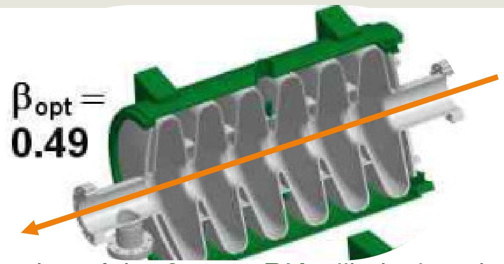


Better processing, cleaning and assembly techniques push out field emission onset level !

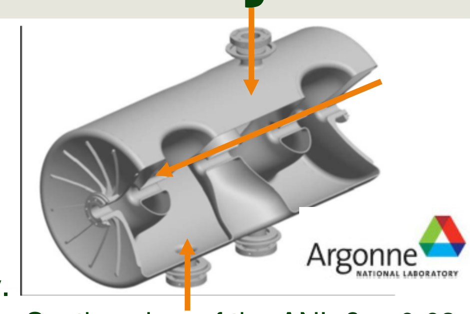
Kenji Saito, Lecture note in Tokyo University on May 2011
The Nature of Field Emission From Microparticles and the Ensuing Voltage Breakdown Hasan Padamsee and Jens Knobloch. Floyd R. Newman Laboratory of Nuclear Studies, Cornell University, Ithaca, NY 14853. Presented as an invited talk at the RF '98 conference.SRF 981021-14. https://www.bnl.gov/edm/papers/HP_srf981021-14.pdf
Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, U.S.A. 2005.

Processing Techniques are Developed Based on Cavity Geometry

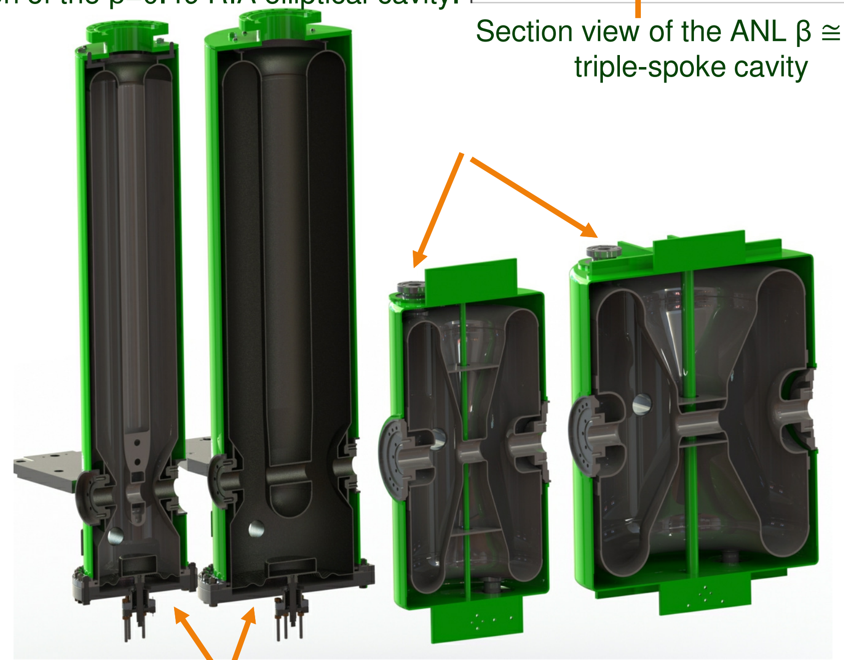
- Elliptical access through beam port *equator difficult to reach.
- QWR, HWR and spoke complicated structure. Fabrication, cleaning, processing and assembly more involved
- Cavity mechanical design consider access for cleaning and processing all surfaces.
- Cavities have critical surfaces for:
  - RF 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

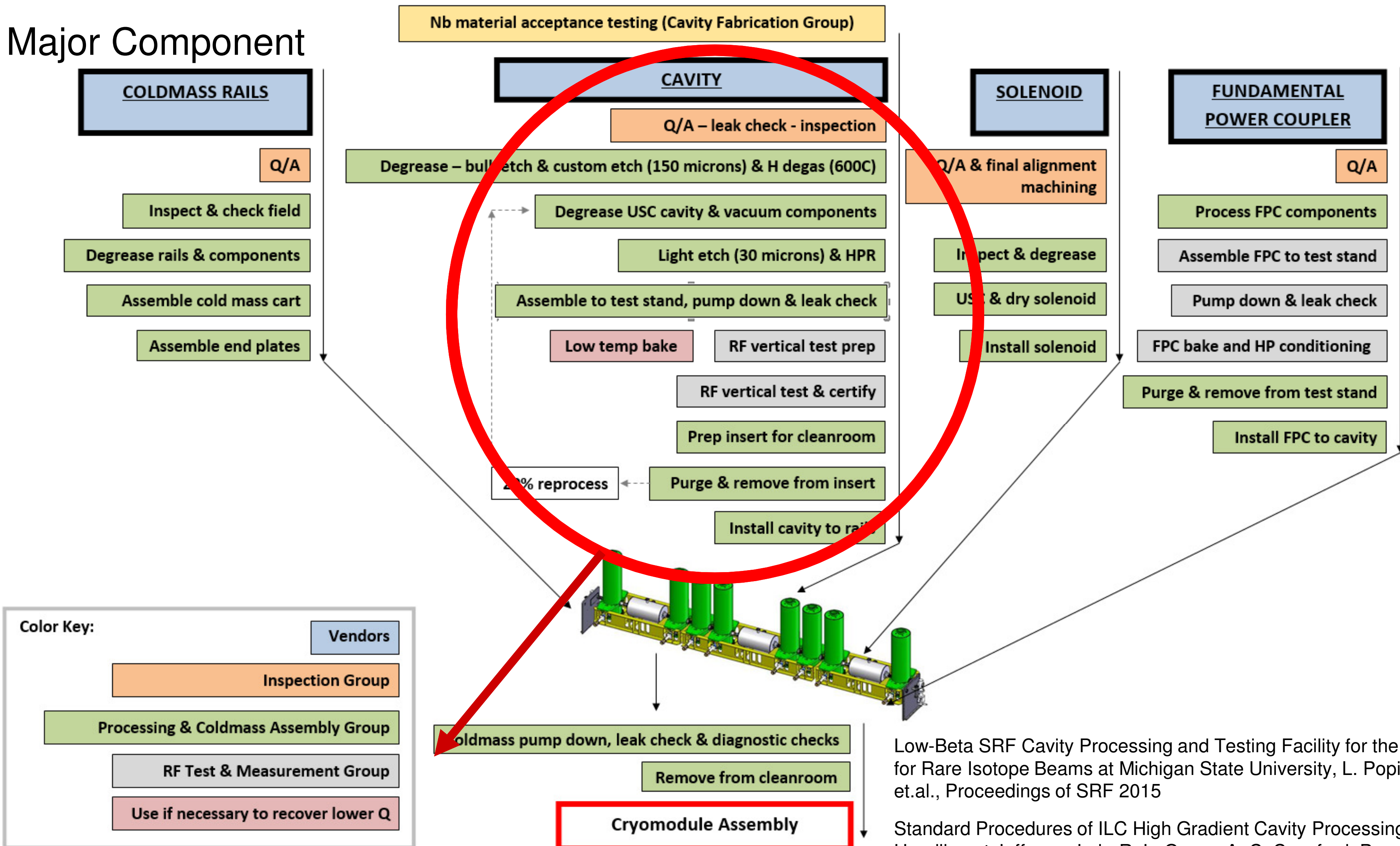
HWR have custom cleaning ports on each end for access to clean and process

FRIB Cryomodule Design and Production* T. Xu, et.al. Proceedings of LINAC2016, East Lansing, MI, U.S.A.

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

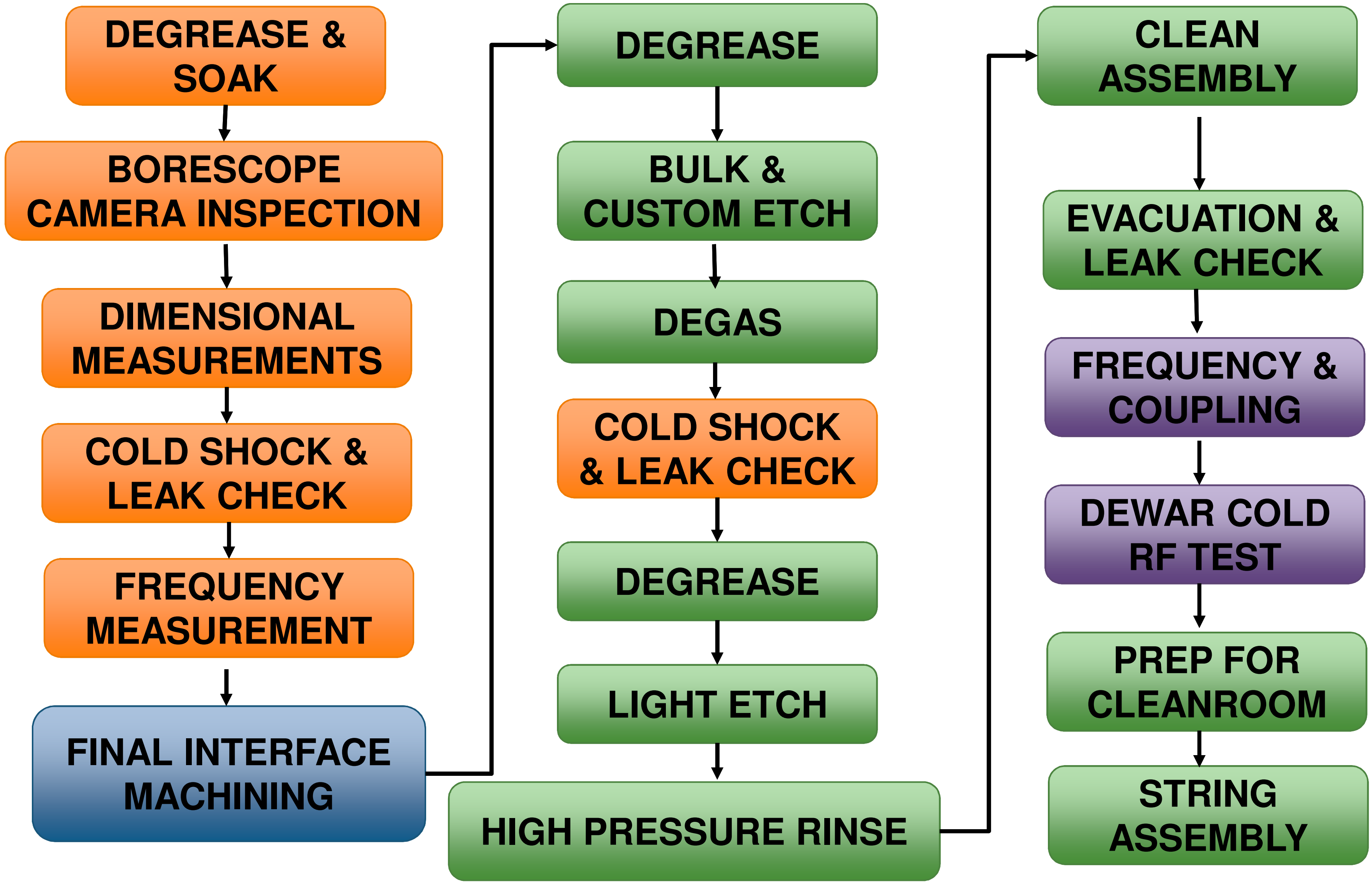
Major Component



Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

Standard Procedures of ILC High Gradient Cavity Processing and Handling at Jefferson Lab, R. L. Geng, A. C. Crawford, Proceedings of SRF 2011

Low Beta Cavity Floor Router



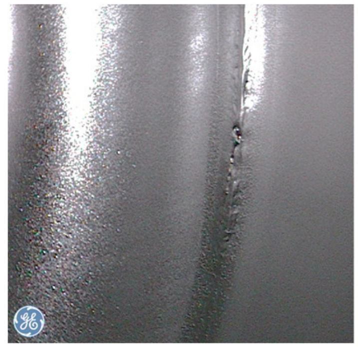
Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

2. SRF Cavity Receiving and Inspection

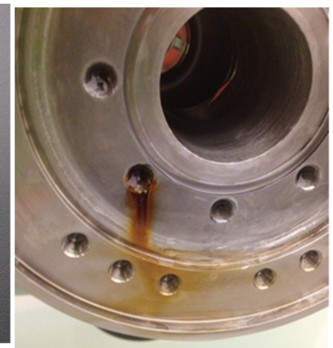
SRF Cavity
Quality Acceptance Inspection

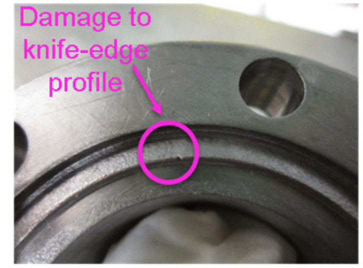
- All cavities are inspected
- Inspection may include:
  - Vendor report reviewed & material certs.
  - Critical dimensions measured
  - Visual inspection by scopes & cameras
  - Frequency measurement
  - Coupling measurements
  - Cold shock & vacuum leak check
  - Water soak to expose any iron inclusions

Borescope

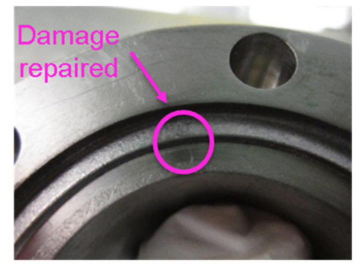


Post Soak



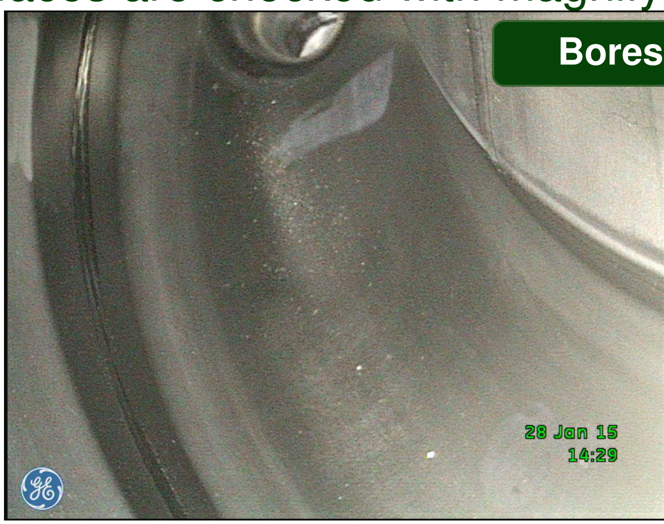


Nonconformance – identified and defined (vacuum leak)

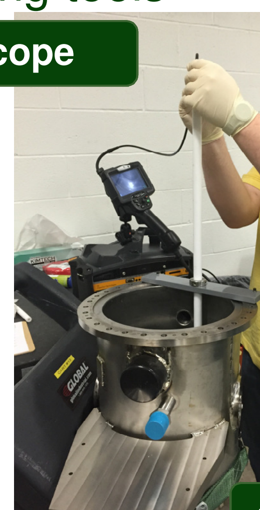


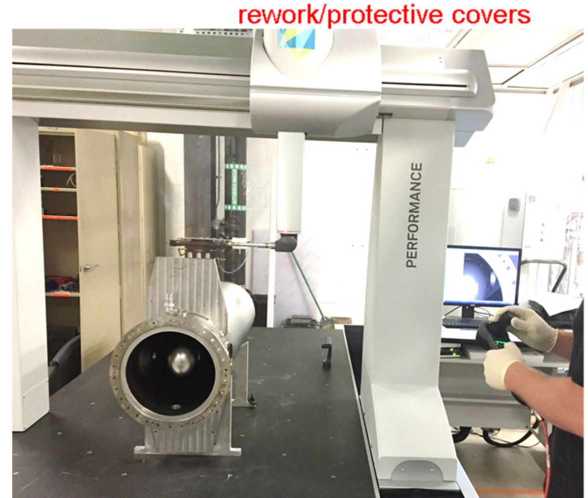
Nonconformance Action - vendor rework/protective covers

- 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

Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF2015

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

http://www.scitechinc.ca/products/oil-field-industrial-4/

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

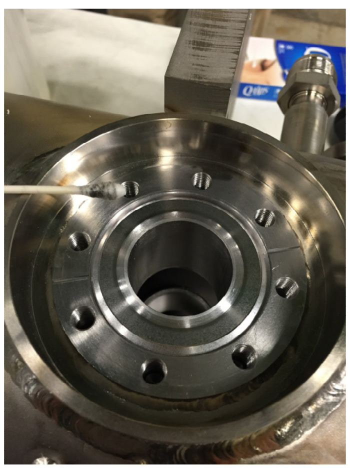
3. Degreasing

What is clean?

- 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 !**

I'm usually DIRTY upon arrival

1. DEGREASE
2. REMOVE PARTICULATE
3. INSPECT



Grease in tapped holes

Residue on cavity surface

Grease from vacuum components

Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, Popielarski, L., R. Oweiss, J. Popielarski, I. M. Malloch, L. J. Dubbs, and K. Elliott. In Conf. Proc., vol. 1205201, no. IPAC-2012-WEPPC065, pp. 2357-2359. 2012.

Degreasing

- Remove gross contamination: burrs, grease or oil from fabrication steps
- Solvent wiping or degreasing useful to superficial residue, lint free wipes
- High pressure spraying with detergents
- Ultra sonic cleaning with detergents effective

1. DEBURR

2. ACETONE

3. ETHANOL

4. HPR W/DETERGENT

5. USC IN DETERGENT

6. RINSE WITH E-4 DI

*All detergents are commercially available

*Detergent (solution)	pH of solution	Uses	May Corrode
[1]Micro 90 ® (0.5-2%)	9.7	Nb, NbTi, SS	Zinc, Al, Cu, Ni
[2]Surface Cleanse 930 ®	6.5	Cu, Al, other	May craze polycarbonate
[3]Tickopur R33 (1%)	9.9	Nb, NbTi, SS	
[4]Citranox ® (1-2%)	2.5	Remove metal oxides	Soft metals

1. https://www.ipcol.com/cleaners/micro-90
2. https://www.ipcol.com/cleaners/surface-cleanse-930
3. https://www.sigmaaldrich.com/catalog/product/sigma/z660035?lang=en&region=US
4. https://alconox.com/citranox/

Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, Popielarski, L., In Conf. Proc., vol. 1205201, no. IPAC-2012

Saito, K. "Gradient yield improvement efforts for single and multi-cells and progress for very high gradient cavities." Proc. SRF2007. Beijing (2007).

Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.

Burrill, A. Buffer Chemical Polishing and RF Testing of the 56 MHz SRF Cavity. No. BNL-82024-2009-IR. Brookhaven National Lab.(BNL), Upton, NY (United States), 2009.

Mammosser, J. "SRF cavity preparation and limitations." Lecture of SRF2009, Berlin, Germany.

Ultra Pure Water Requirements

- 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

Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.

SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, et.al., Proceedings of LINAC2014, Geneva, Switzerland

Cooper, C., et al. "Cavity Processing Research Laboratory at Fermilab: SRF cavity processing R&D." Proceedings of SRF 2011.

D5127 – 13

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

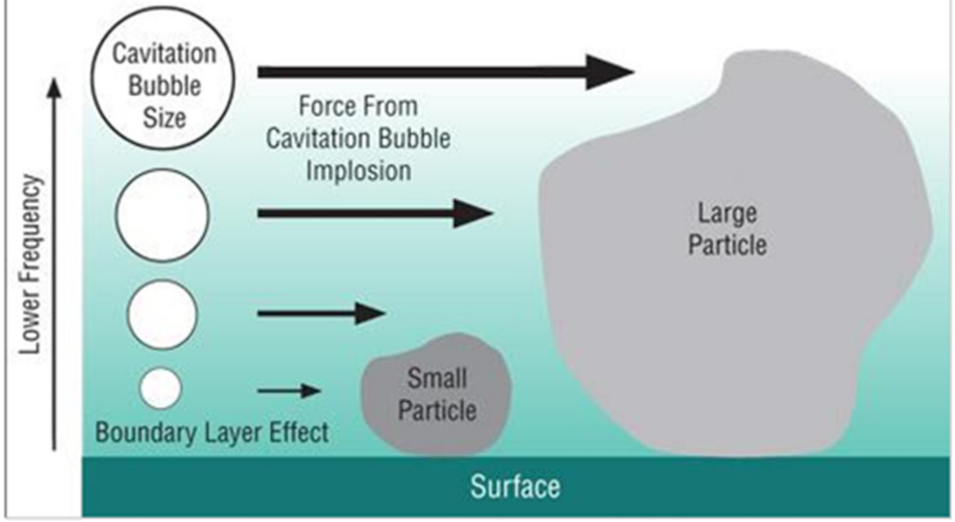
Parameter	Type E-1	Type E-1.1	Type E-1.2[B]	Type E-1.3[B]	Type E-2	Type E-3	Type E-4
Linewidth (microns)	1.0–0.5	0.35–0.25	0.18–0.09	0.065–0.032	5.0–1.0	>5.0	—
Resistivity, 25°C (On-line)	18.1	18.2	18.2	18.2	16.5	12	0.5
TOC (µg/L) (on-line for <10 ppb)	5	2	1	1	50	300	1000
On-line dissolved oxygen (µg/L)	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							
Vanadium							
Zinc	0.05						500
Temperature Stability (K)							
Temperature Gradient (K/10 min)							
Dissolved Nitrogen On-line (mg/L)							
Dissolved Nitrogen Stability (mg/L)							

ASTM D5127-13, Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries, ASTM International, West Conshohocken, PA, 2013, www.astm.org.

General Use of Ultra Sonic Cleaners

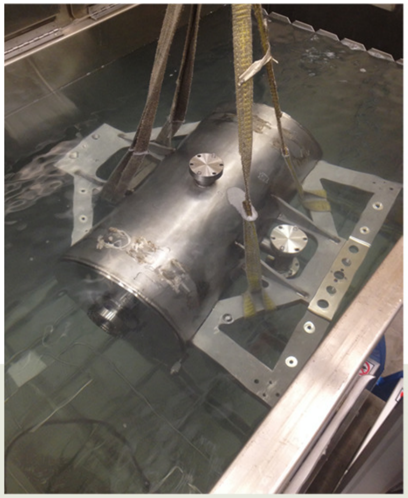
- Frequency of operation
- Temperature control
- Cleaning time
- Hot water more effective
- Detergent use
- Recirculation pump with filter
- New solution for final steps
- Thoroughly rinse

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 volume with weir



Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, Popielarski, L., In Conf. Proc., vol. 1205201, no. IPAC-2012

Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.

Article From: 10/19/2011 Production Machining, John Fuchs from Cleaning Technologies Group
https://www.productionmachining.com/articles/meeting-medical-manufacturings-tough-demands

Manual High Pressure Rinse

- Very large components that do 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

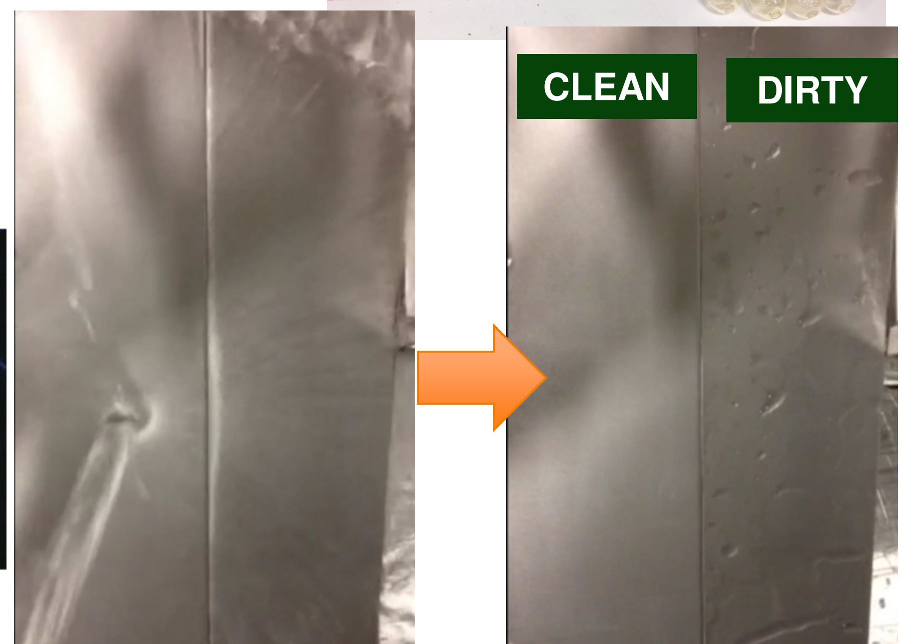
SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, et.al., Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, 48824, U.S.A. Proceedings of LINAC2014, Geneva, Switzerland

← wash down booth

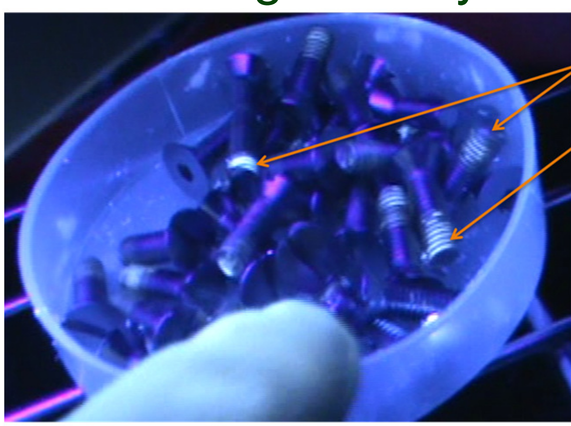
Superconducting Accelerator Activities at TRIUMF/ISAC, R. E. Laxdal, G. Clark, K. Fong, A. Mitra, M. Pasini, R. Poirier, I. Sekachev, G. Stanford, TRIUMF, 4004 Wesbrook Mall, Vancouver, Canada. LINAC 2002

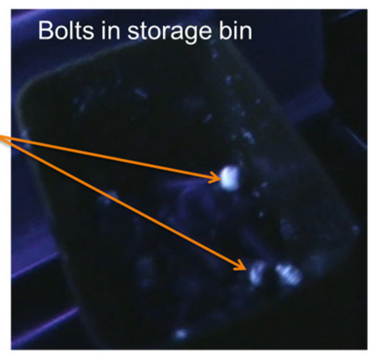
Verification

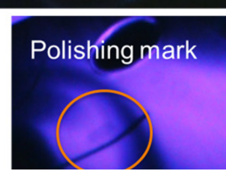
- Total organic carbon (TOC) content measurements
- Visual inspection and white poly wipe
- Water break free test
- Clean gas spray & count
- Surface particle counts
- UV light inspection
- Residual gas analysis



Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.



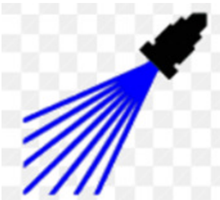




Grease easily detected on bolt threads
Identify bolts that have not been cleaned properly

Advanced Technologies for Determination of Surface Cleanliness, Technological Engineering, volume XI, number 1/2014ISSN 1336 -5967. Jan Kudlacek, Petr Chabera. CVUT Prague, Faculty of Mechanical Engineering, Prague, Czech Republic.

Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, DESY, Hamburg, Germany. CARE Conf-05-033-SRF.



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

SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, et.al., Proceedings of LINAC2014, Geneva, Switzerland

Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005

Ultrapure Water for SRF Applications, T. Reilly, USPAS, January 2015. https://www.jlab.org/indico/event/98/contribution/16/material/slides/0.pdf

UPW System Equipment

Reverse osmosis system

Water softener and brine tank

Virgin DI beds

185 nm UV sterilizer bulb units

Reduction of organics, Total Oxidizable Carbon

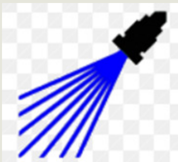
254 nm UV sterilizer bulb unit →

Destroy microorganisms by altering DNA

← Sub-micron post filter 0.02 μm

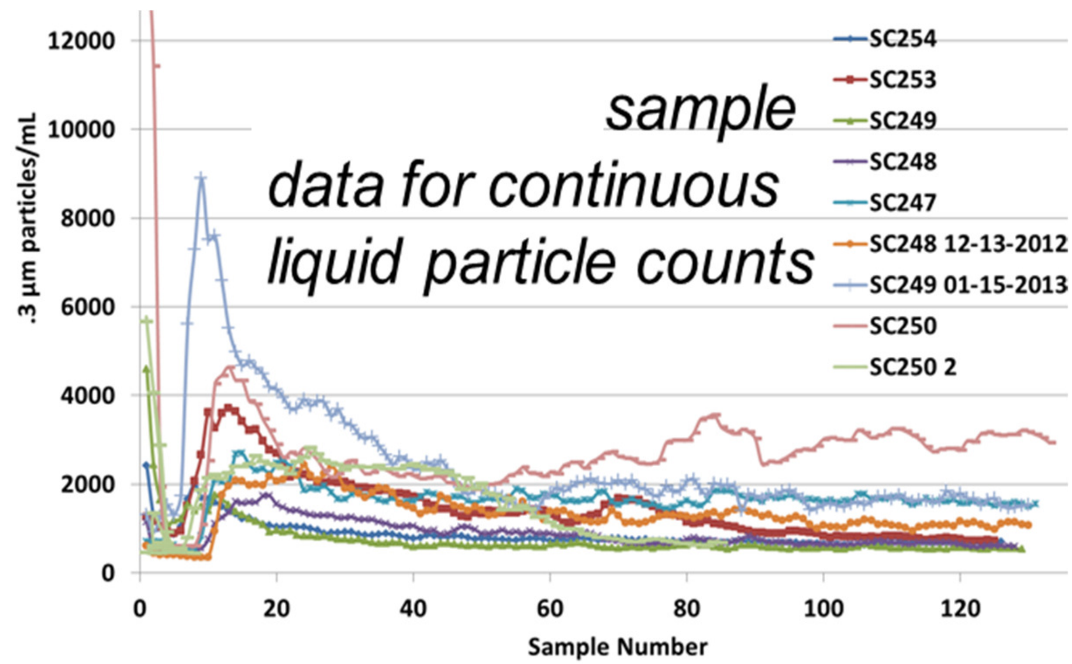
SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, et.al., Proceedings of LINAC2014, Geneva, Switzerland

Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, DESY, Hamburg, Germany. CARE Conf-05-033-SRF.

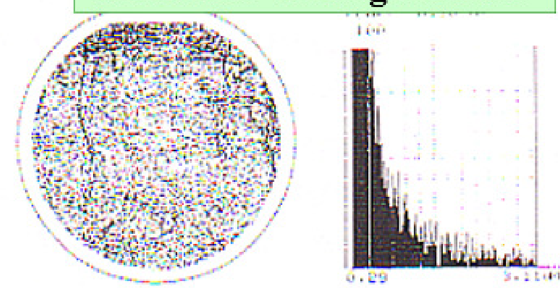


Ultra Pure Water at High Pressure Used for Final Cleaning

- 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

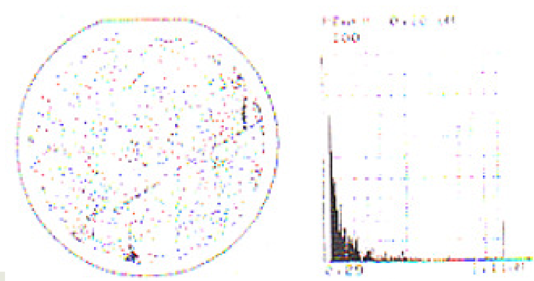


TRISTAN rinsing method



Particle size	Count
0.30-1.20 µm	5825
1.20-2.01 µm	405
2.01-3.00 µm	2720
> 3.00 µm	1069
Total	10019

HPR rinsing

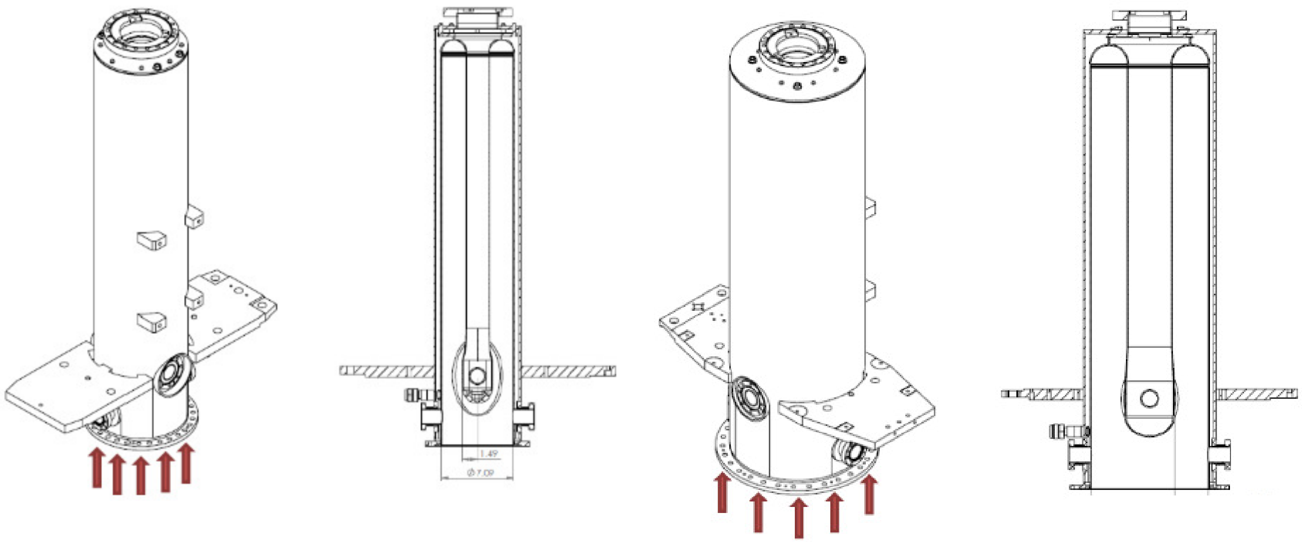


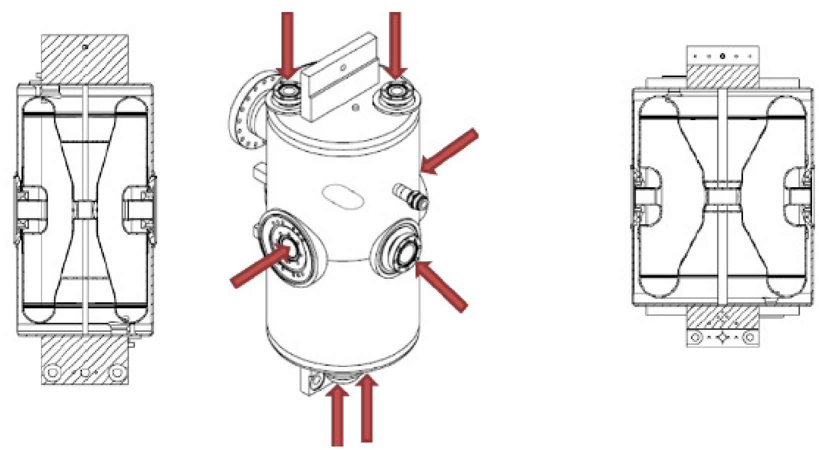
Particle size	Count
0.30-1.20 µm	646
1.20-2.01 µm	52
2.01-3.00 µm	282
> 3.00 µm	37
Total	1017

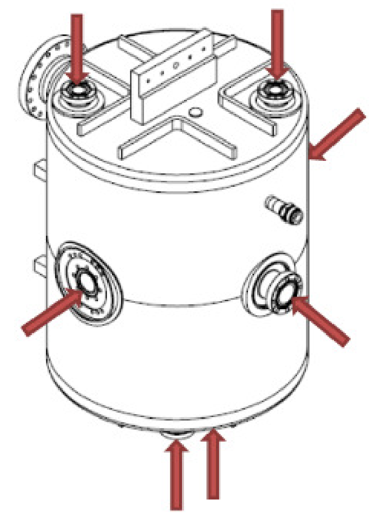
KEK Preprint 94-5 April 1994 A Study of Ultra-clean Surface for Niobium SC Cavities, K. SAITO, H. MIWA, K. KUROSAWA, P. KNEISEL, S. NOGUCHI,E. KAKO, M. ONO, T. SHISHIDO and T. SUZUKI.

Access to Internal Surfaces for Cleaning

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







Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

High Pressure Rinse Systems Around the World

A Flexible System for the High Pressure Rinsing of SRF Cavities, R. Murphy, et.al., Proceedings of SRF2011.

Status of SRF Facilities at SNS, J. Saunders, et.al., IPAS 2015.

A New High Pressure Rinsing System Established at DESY, A. Matheisen, et.al., Proceedings of SRF 2009.

Design and Implementation of an Automated High-Pressure Water Rinse System for FRIB SRF Cavity Processing, I. Malloch, et.al., Linac 2016.

http://ipnwww.in2p3.fr/IMG/pdf/dasupratechclean_room.pdf

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

A Flexible System for the High Pressure Rinsing of SRF Cavities, R. Murphy, et.al., Proceedings of SRF2011.

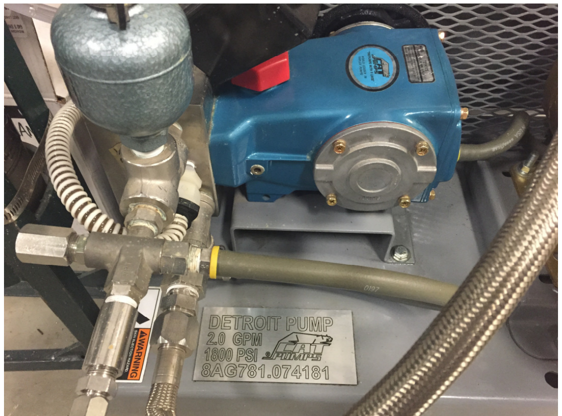
Status of SRF Facilities at SNS, J. Saunders, et.al., IPAS 2015.

A New High Pressure Rinsing System Established at DESY, A. Matheisen, et.al., Proceedings of SRF 2009.

Design and Implementation of an Automated High-Pressure Water Rinse System for FRIB SRF Cavity Processing, I. Malloch, et.al., Linac 2016.

http://ipnwww.in2p3.fr/IMG/pdf/dasupratechclean_room.pdf

Major Components for High Pressure Rinse System

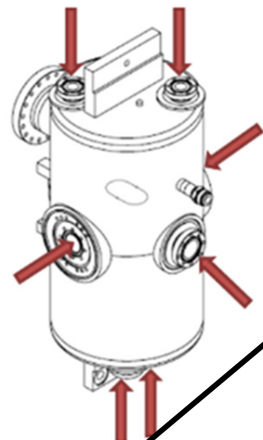


E-1

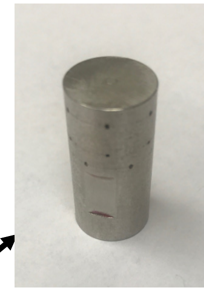
HP pump

filter

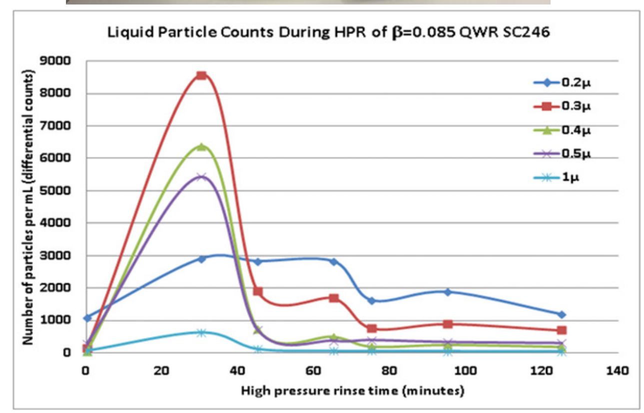
Sub micron filter

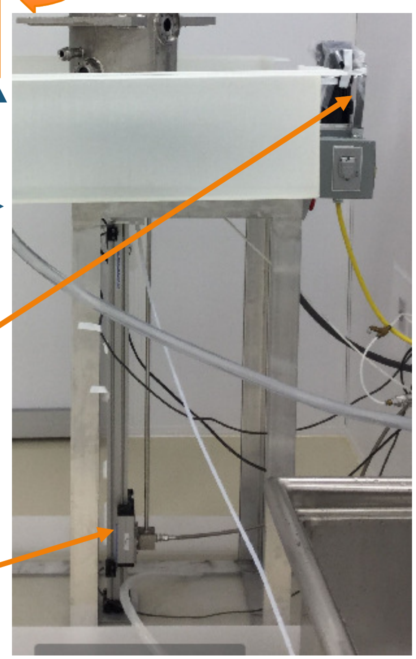


nozzle



Liquid Particle counter





rotation/oscillation and translation mechanics

A Flexible System for the High Pressure Rinsing of SRF Cavities, R. Murphy, et.al., Proceedings of SRF2011.

Status of SRF Facilities at SNS, J. Saunders, et.al., IPAS 2015.

A New High Pressure Rinsing System Established at DESY, A. Matheisen, et.al., Proceedings of SRF 2009.

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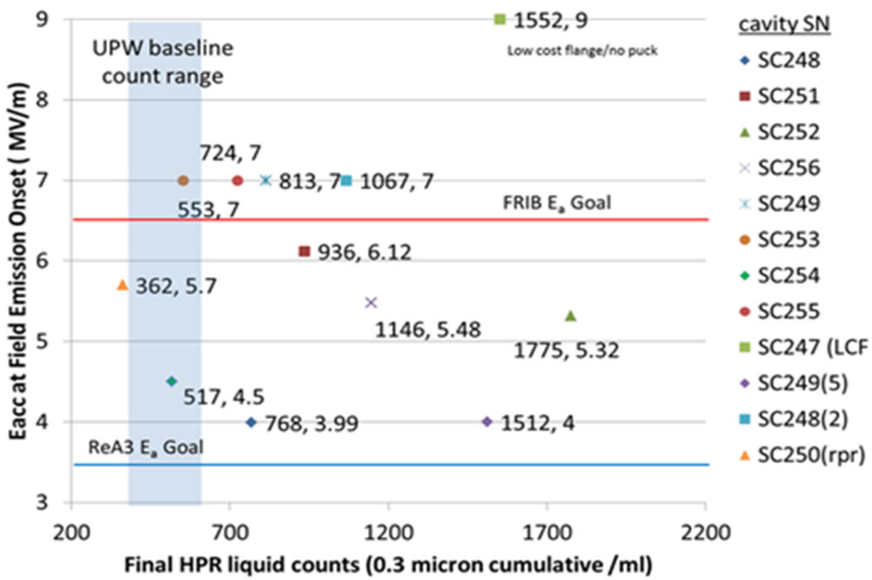
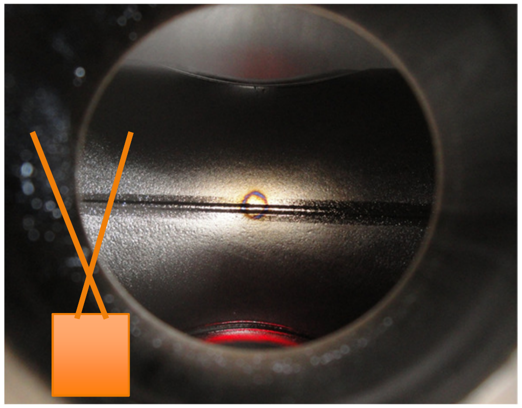
http://ipnwww.in2p3.fr/IMG/pdf/dasupratechclean_room.pdf

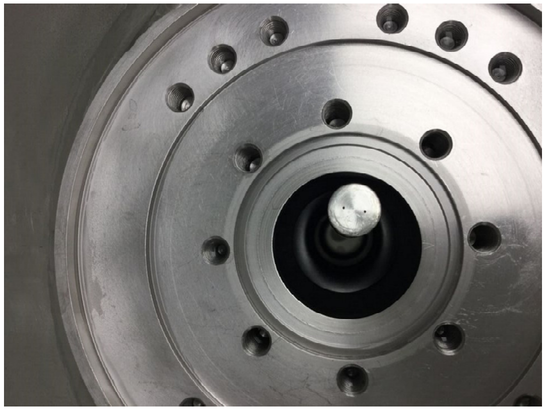
High Pressure Rinse Concerns

- 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

Investigation into the Effectiveness of the JLAB High Pressure Rinse System, J. Mammosser, T. Rothgeb, T. Wang, A.T. Wu, Jefferson Lab, Newport News, VA 23606, USA. PAC 2003.
High Pressure Rinsing System Studies, D. Sertore, M. Fusetti, P. Michelato, C. Pagani, INFN Milano-LASA, I-20090 Segrate (MI), Italy T. Higo, J. Hong, K. Saito, KEK, Tsukuba, Japan, G. Ciovati, T. Rothgeb, JLAB, Newport News, VA 23606, USA A. Matheisen, N. Krupka, DESY, Hamburg, Germany. Proceedings of SRF2007, Peking Univ., Beijing, China

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
- Mechanical Abrasives include:
  - Manual polishing (power tools, sandpaper, Scotch Brite ™)
  - Tumbling (Centrifugal Barrel Polishing)
- Chemical Reaction
  - Buffered Chemical Polishing (BCP)
  - Electropolishing (EP)

Study on Particle 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, U.S.A. PAC 2013.

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

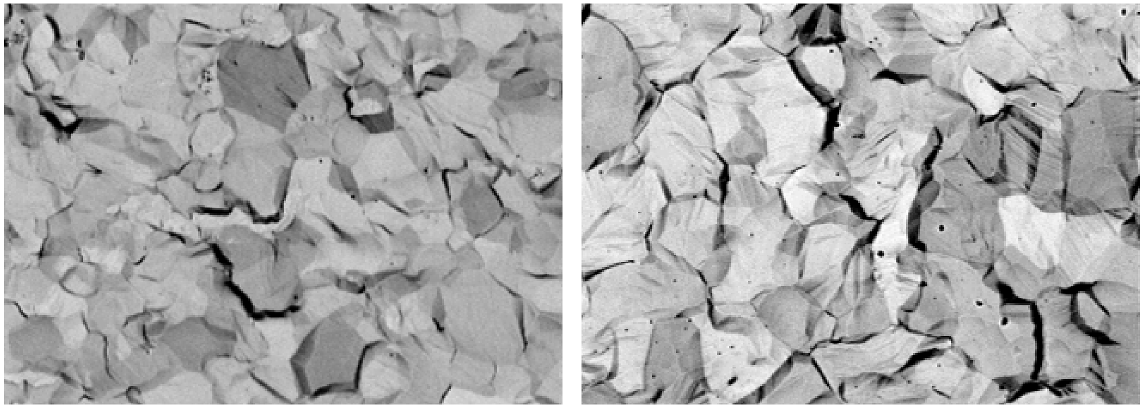


Figure 5. *SEM scans of an as-received sample (left) and an abraded sample (right) after more than 100 microns of etching. Note the unusually high concentration of black particulate spots on the abraded sample.*

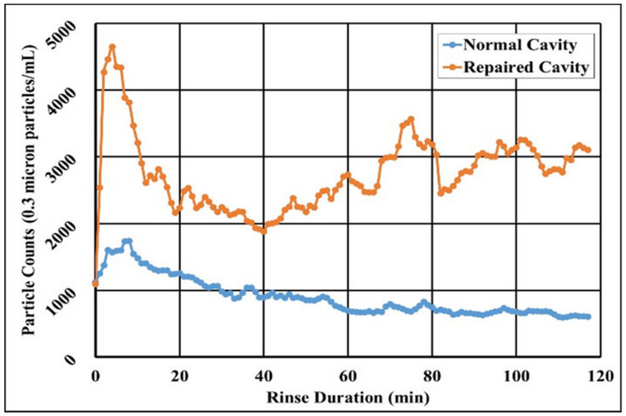


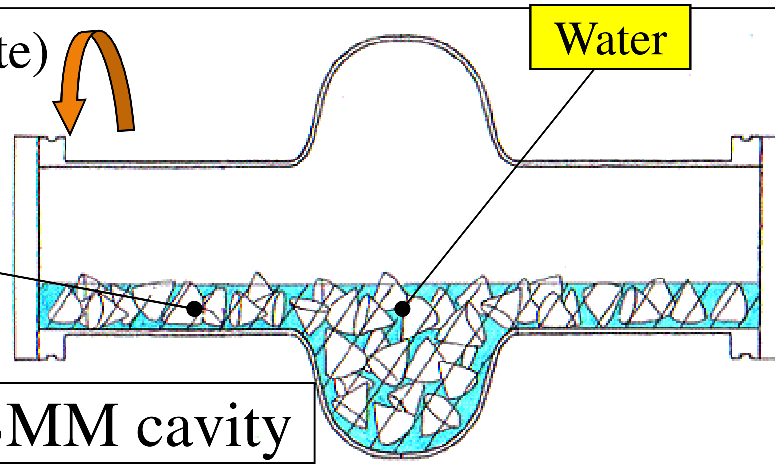
Figure 6. *Liquid particle counts as a function of high-pressure rinse time comparing a normal and repaired cavity.*

Study on Particle 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, U.S.A. PAC 2013

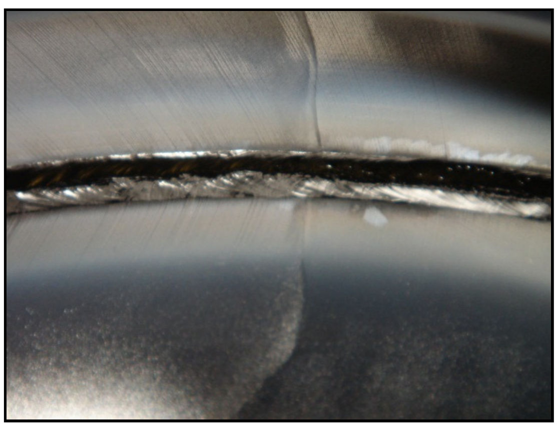
Centrifugal Barrel Polishing (CBP)

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

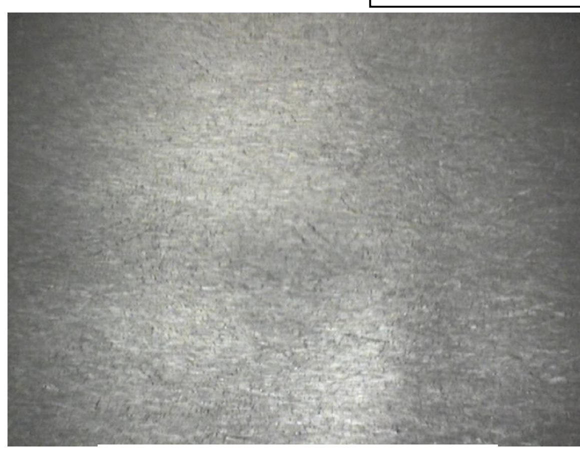
Rotated ~100 rpm on an oppositely rotating table



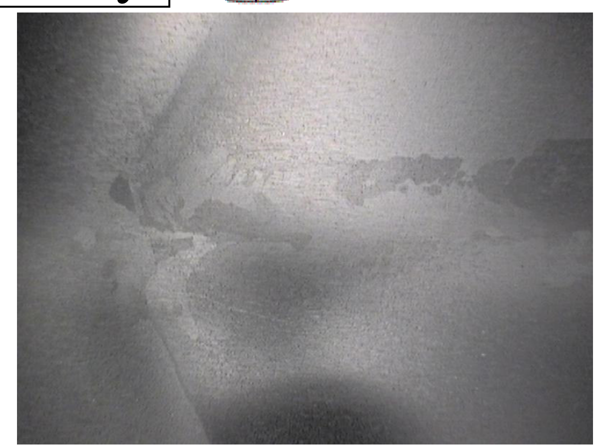
CBMM cavity



Before CBP (equator EBW seam)



After CBP



After light CP(10μm)

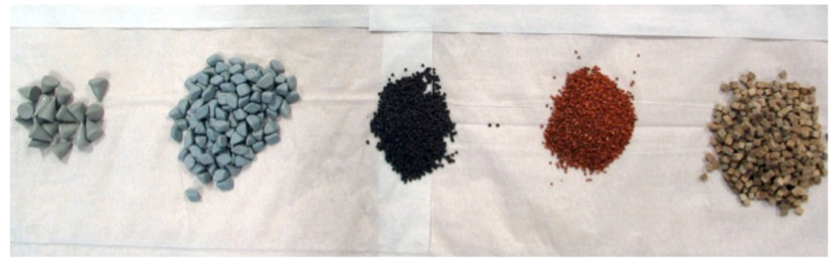
Centrifugal Barrel Polishing of Cavities Worldwide, C. Cooper, Fermi National Accelerator Laboratory, Batavia, IL, U.S.A. Kenji Saito,KEK, High Energy Accelerator Research Organization, Tsukuba, Japan B. Bullock, Cornell University, LEPP – SRF Group, Ithaca, NY,U.S.A. S. Joshi, Raja Raman Center for Advanced Technology, Indore, India A. Palczewski, Thomas Jefferson Accelerator Lab, Newport News, VA, U.S.A. Proceedings of SRF2011, Chicago, IL U.S.A.

Kenji Saito, Lecture note in Tokyo University on May 2011

CBP at Other Labs

- Fermilab developed one way 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.



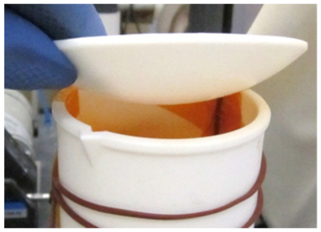
Medias are tumbled inside. The grey cones (far left) are a plastic with aluminum silicate, used for bulk removal. The powder blue media (second from left) are ceramic abrasives, useful as a first-pass media. A hardwood cut into small cubes (far right) is also a useful abrasive.

Mirror-like finish can be achieved

Fermilab

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

Centrifugal Barrel Polishing of Cavities Worldwide, C. Cooper, Fermi National Accelerator Laboratory, Batavia, IL, U.S.A. Kenji Saito,KEK, High Energy Accelerator Research Organization, Tsukuba, Japan B. Bullock, Cornell University, LEPP – SRF Group, Ithaca, NY,U.S.A. S. Joshi, Raja Raman Center for Advanced Technology, Indore, India A. Palczewski, Thomas Jefferson Accelerator Lab, Newport News, VA, U.S.A. Proceedings of SRF2011, Chicago, IL U.S.A.



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

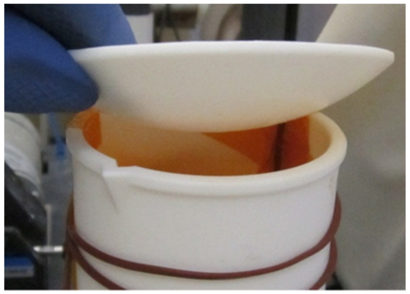
RECIPE- 1:1:2 acid mixture
1 part Hydrofluoric (HF) acid (49% w/w)
1 part Nitric (HNO_3) acid (70% w/w)
2 parts Phosphoric acid (85% w/w) [Buffer; not involved in reaction]

- 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, U.S.A.

Chemical Reaction Mechanism



Brown NO_2 gas

Nitric acid oxidizes the Nb

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

HF reacts with Nb oxide

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

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

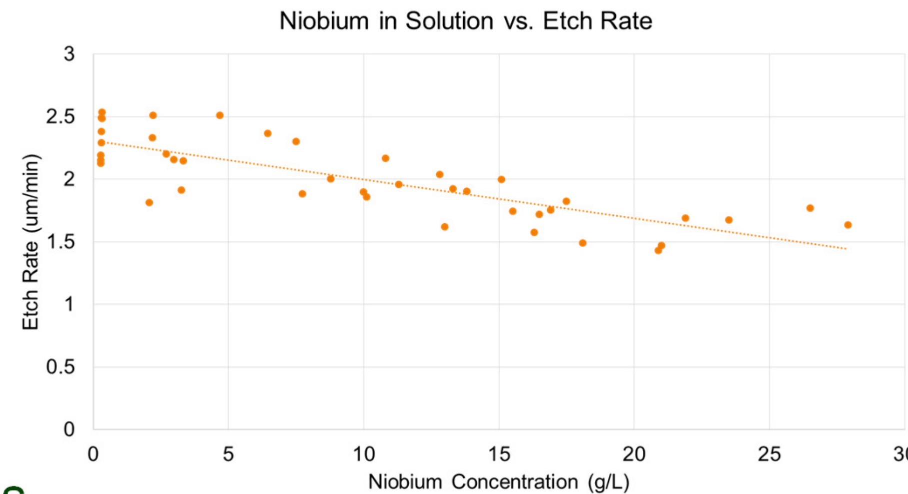
The reaction is exothermic !

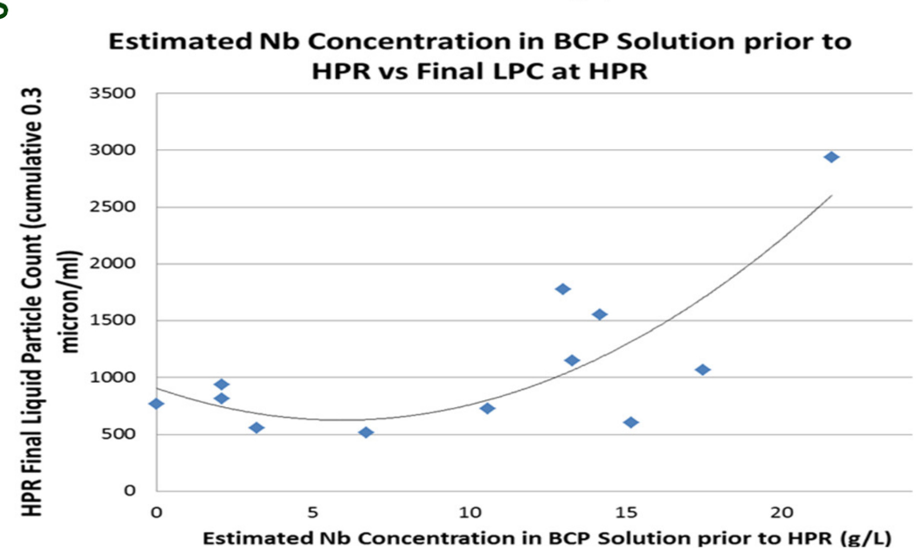
HEAT OF REACTION RESULTS SUMMARY		
Average Heat of Reaction =	-607	kJ/mol
Standard Deviation =	17.6	kJ/mol
Theoretical Heat of Reaction =	-678.9	kJ/mol
Percent Error =	***10.5%***	

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, U.S.A. IPAC 2012.

BCP Process Variables And Considerations

- Acid temperature to reduce hydrogen
- Acid flow rate
- System pressure
- Ultra sonic thickness measurement (USTM) on bare cavities: understand etch rates and removal uniformity
- BCP etching very repeatable if variables kept constant
- Acid niobium concentration to optimize etch rate and decrease contamination



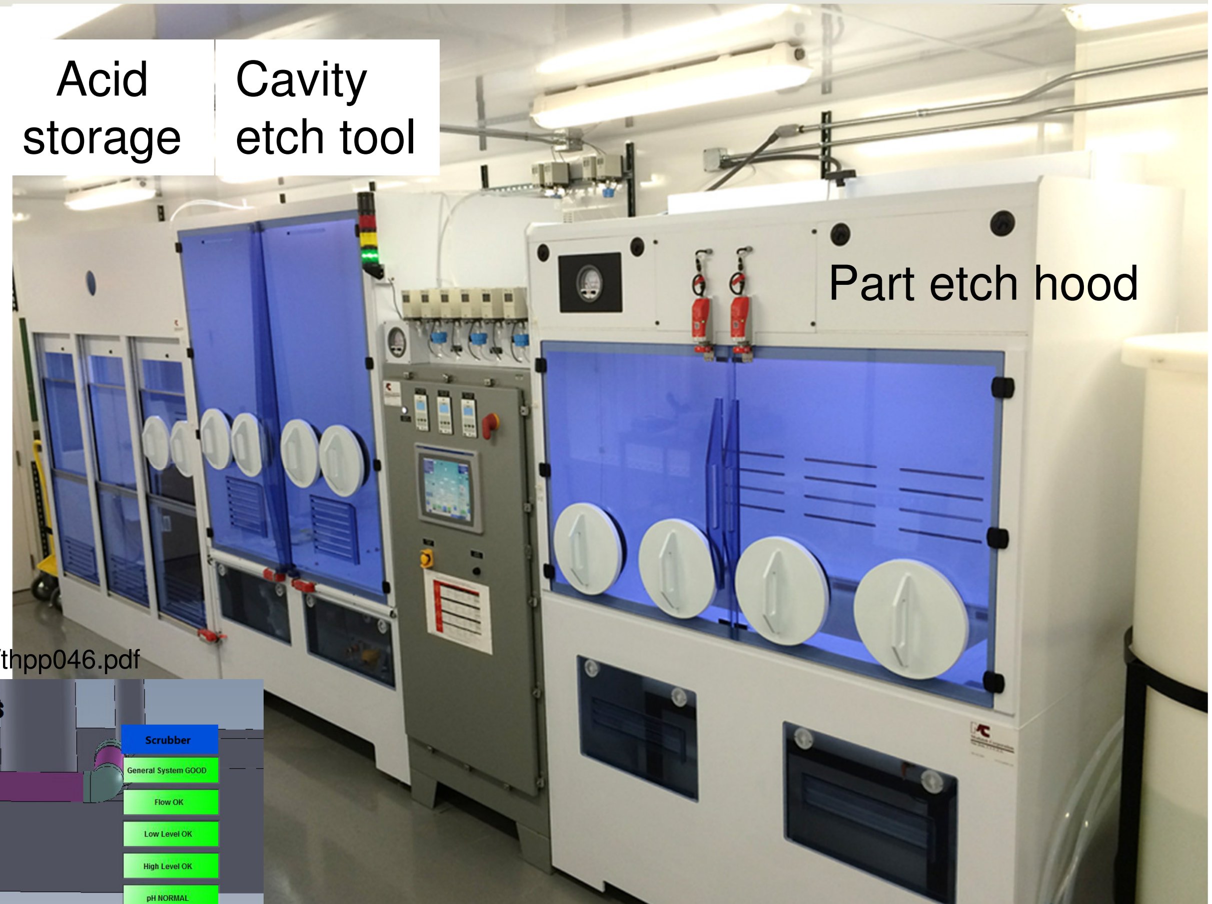


SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

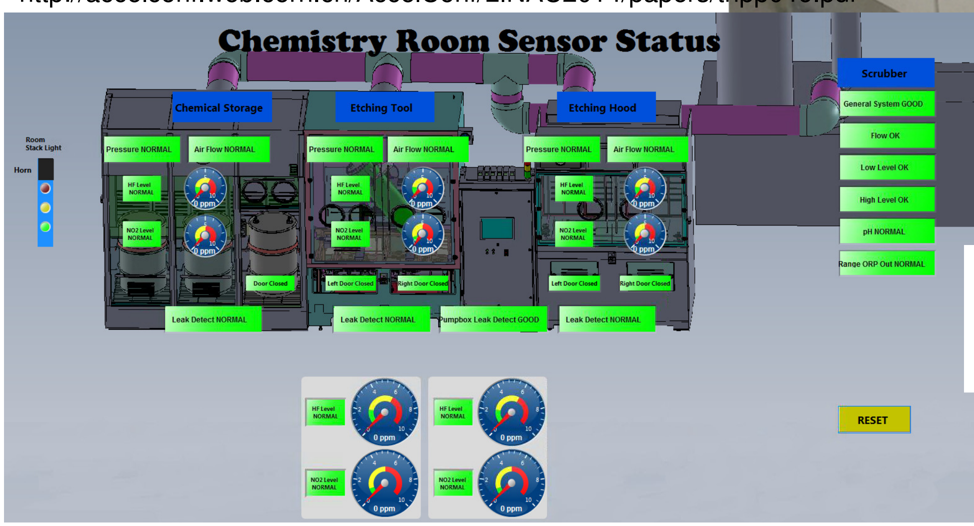
Investigation of BCP Parameters for Mastery of SRF Cavity Treatment, F. Éozénou, E. Cenni, G. Devanz, T. Percerou, T. Proslier, C. Servouin, M.L.L. Nghiem* Irfu, CEA, Université Paris-Saclay, F- 91191 Gif-sur-Yvette, France *Université Pierre et Marie Curie, 4 place Jussieu, F- 75252 Paris, France. SRF 2017, Lanzhou, China. 18th International Conference on RF Superconductivity

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



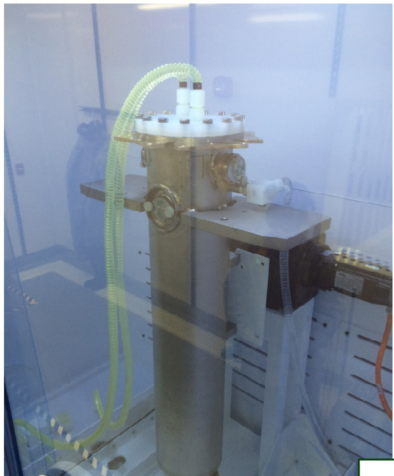
http://accelconf.web.cern.ch/AccelConf/LINAC2014/papers/thpp046.pdf



← Exterior monitors display all tool and facility alarm status.

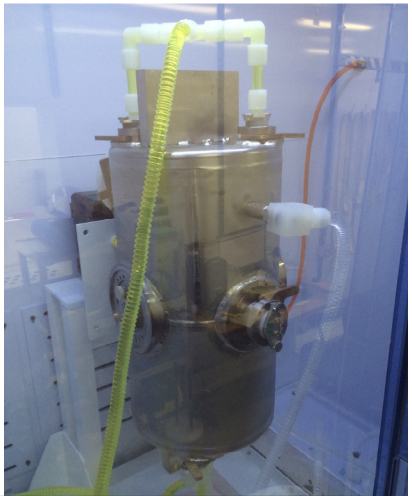
SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

Etching Configurations for Low Beta Cavities



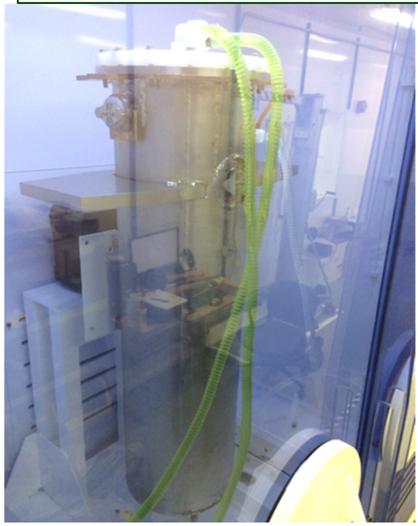
β=0.041 QWR

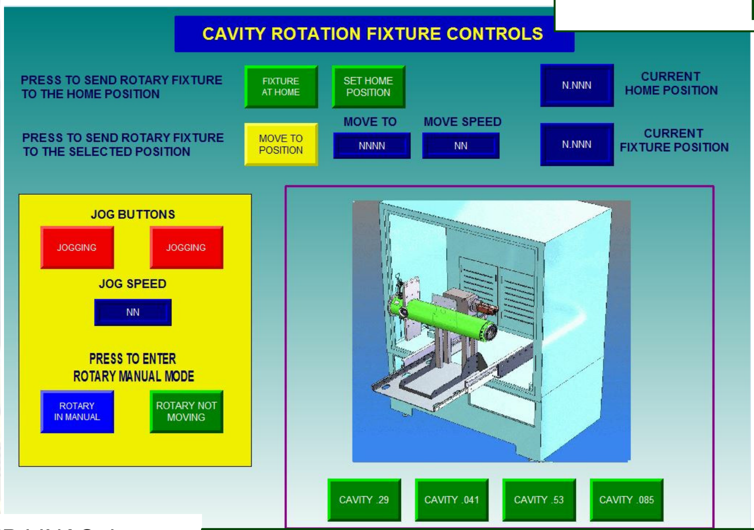
85001 tuned by custom etching in high electric field region

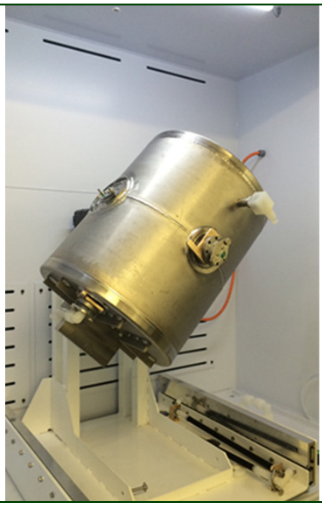


β=0.29 HWR

β=0.53 HWR







Tooling allows easy access for installation and rotation, used for QWR etching and differential etching for tuning.

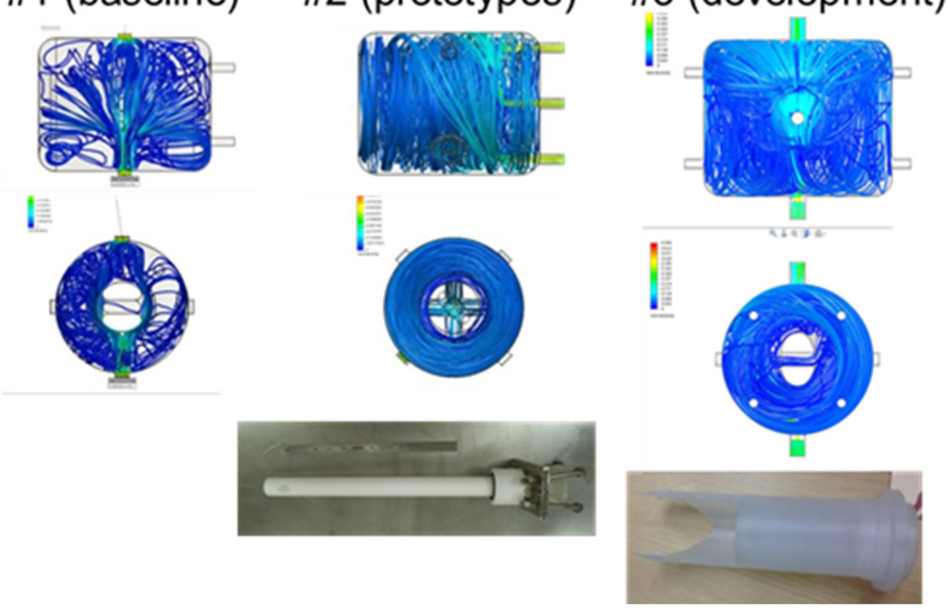
Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

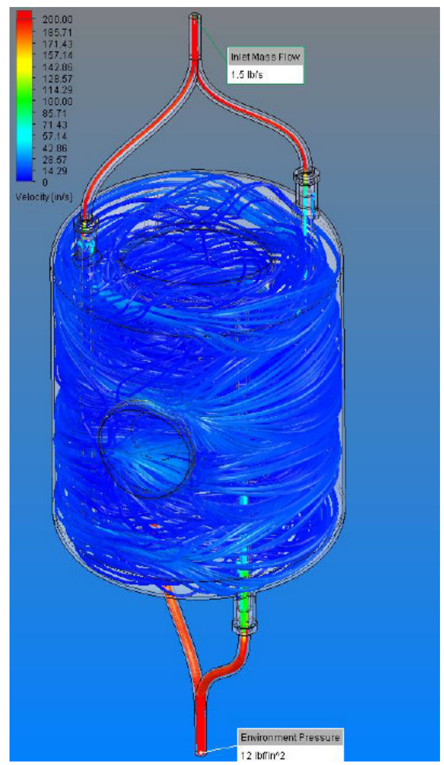
Interface Tooling is as Important as Equipment Design

- 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

#1 (baseline) #2 (prototypes) #3 (development)



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



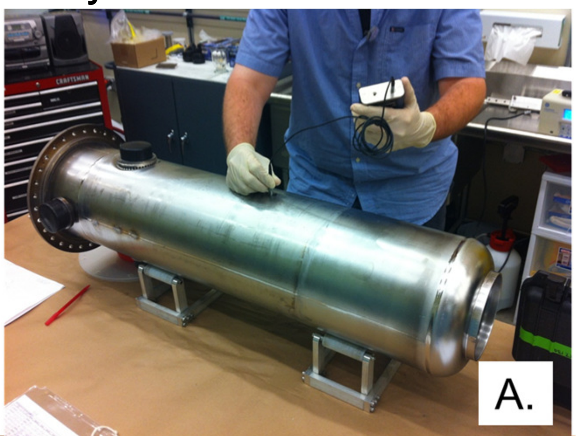
Buffered Chemical Polishing Development for the Beta=0.53 Half-Wave Resonator at Michigan State University, L. Popielarski, et.al., Proceedings of SRF2011

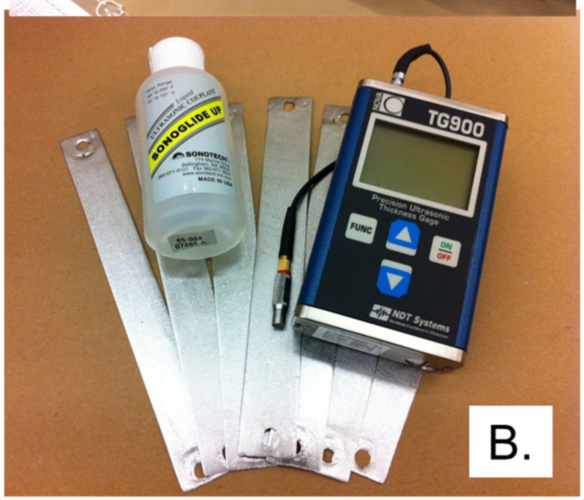
SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, et.al., Proceedings of SRF 2015

Optimization of the BCP Processing of Elliptical Nb SRF Cavities, C. Boffo, C. Cooper, A. Rowe, FNAL, Batavia, IL 60510, U.S.A. G. Galasso, University of Udine, Italy. Proceedings of EPAC 2006, Edinburgh, Scotland.

Etch Removal Visualization for β=0.53 HWR

Ultra sonic thickness measurements on undressed cavity.





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	

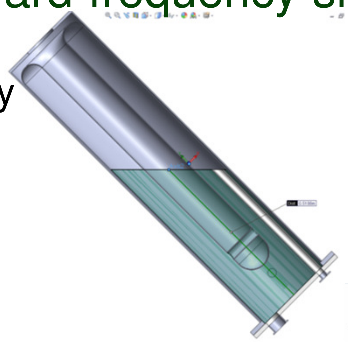
http://accelconf.web.cern.ch/accelconf/srf2015/papers/mopb095.pdf

SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

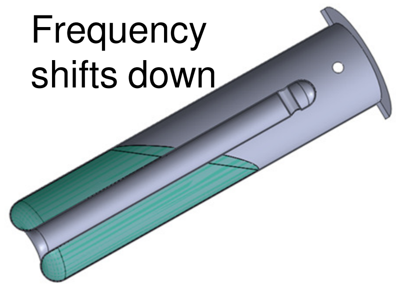
Differential Etching for Frequency Compensation

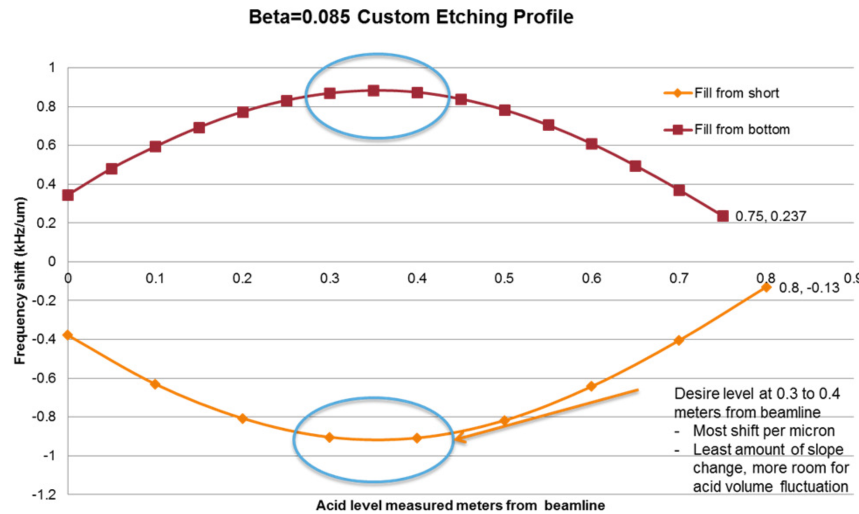
- Spot etch removal based on the electromagnetic design of the cavity
- Removal in the high E-field region removal yields upward frequency shift
- Removal in high magnetic field regions yields downward frequency shift

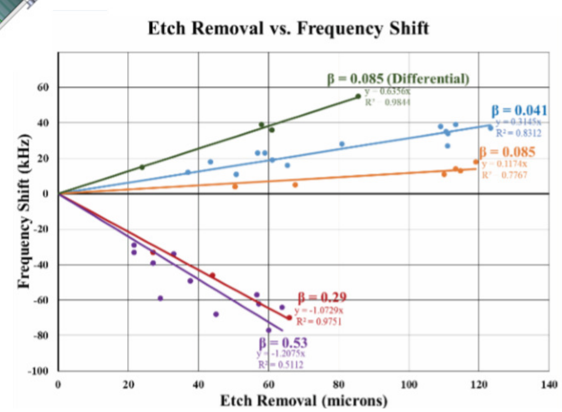
Frequency shifts up



Frequency shifts down

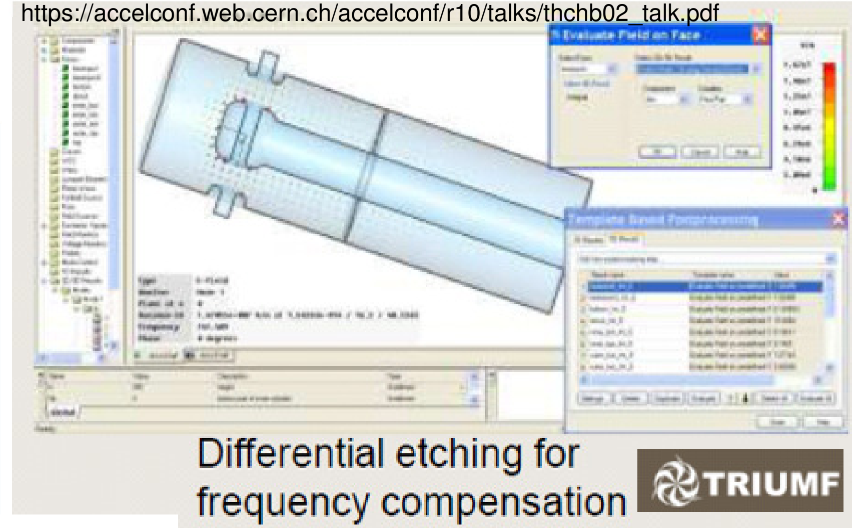






Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

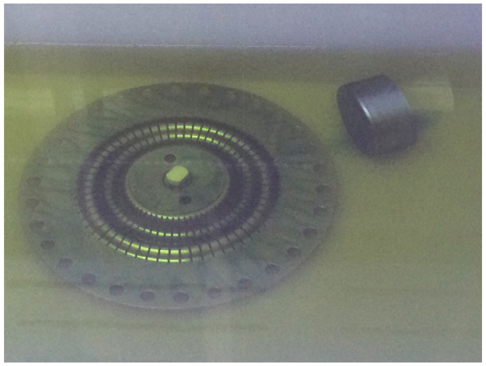
https://accelconf.web.cern.ch/accelconf/r10/talks/thchb02_talk.pdf

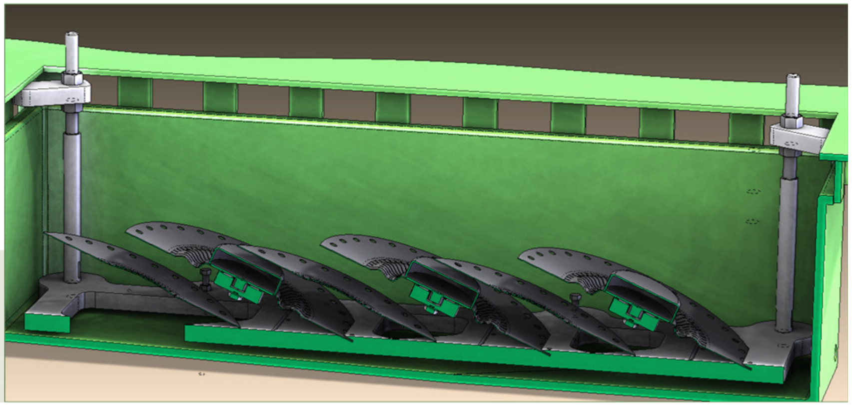


SCRF Development at TRIUMF, V. Zvyagintsev, R.E. Laxdal, B. Amini, C. Beard, R. Dawson, T. Emmens, K. Fong, A. Grasselino, P. Harmer, D. Kishi, P. Kolb, A. Koveshnikov, D. Lang, M. Laverty, R. Leewe, C. Laforge, D. Longuevergne, M. Marchetto, A.K. Mitra, T. Ries, I. Sekachev, R. Shanks, B. Waraich, F. Yan, Q. Zheng, TRIUMF, Vancouver, Canada. R.S. Orr, W. Trischuk, University of Toronto, Ontario, Canada, R. Edinger, PAVAC Industries, Richmond, Canada RUPAC 2010 Protvino

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





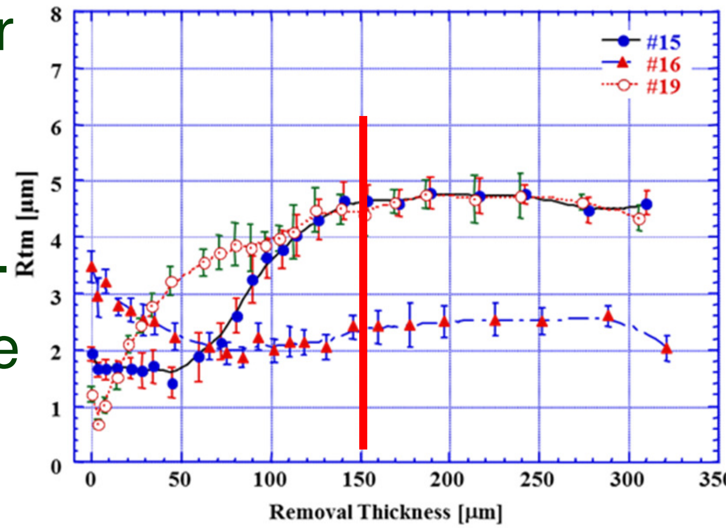
Handbook of Accelerator Physics and Engineering 2nd Printing, A. Chao, M. Tigner, Section 5.3 Fabrication of Niobium RF Structures, T. Hays, H. Padamsee, Cornell U. D. Proch, DESY. Pg. 304. 1999

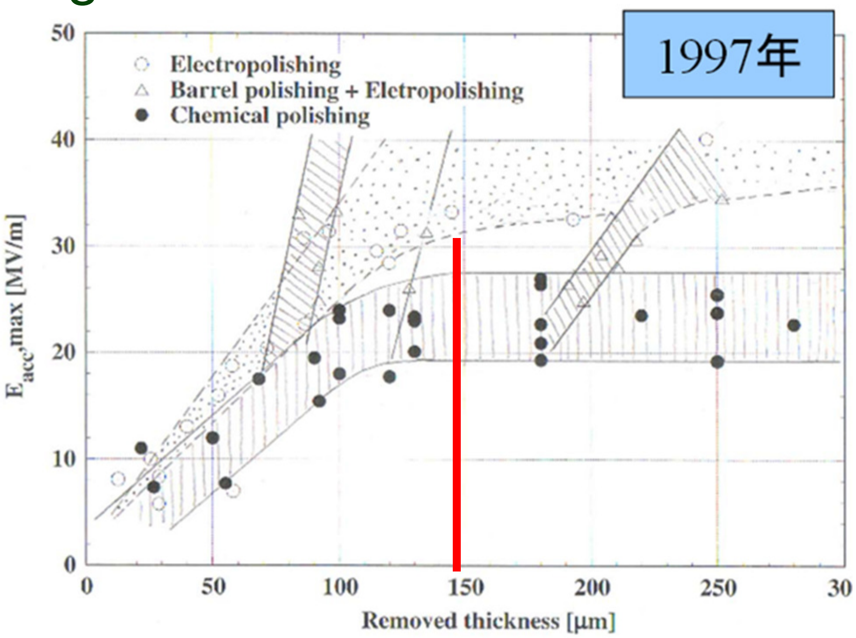
SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

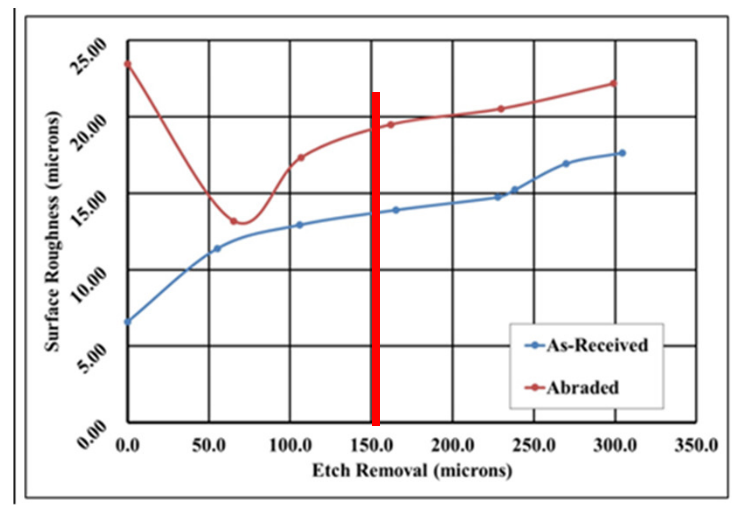
Why 150-200 microns?

- 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





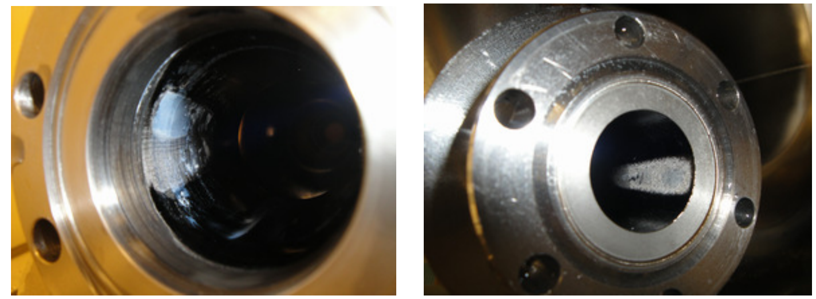


Superiority of Electropolishing over Chemical Polishing on High Gradients, Kenji SAITO, et.al., Proceedings of the 1997 Workshop on RF Superconductivity, Abano Terme (Padova), Italy.

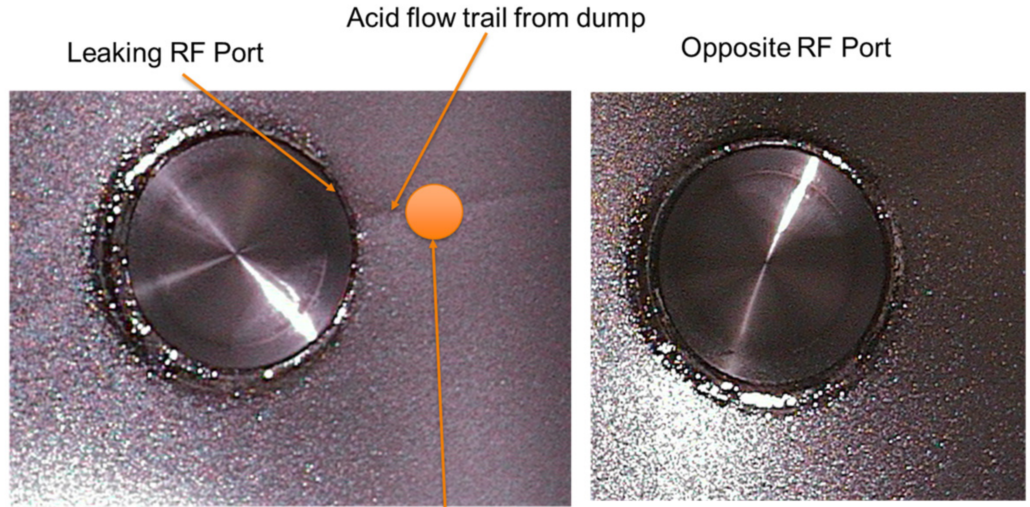
Study on Particle Retention on Polished Niobium Surfaces after BCP Etching, I. Malloch, C. Compton, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. PAC 2013.

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

Optimization of the BCP Processing of Elliptical Nb SRF Cavities, C. Boffo, C. Cooper, A. Rowe, FNAL, Batavia, IL 60510, U.S.A. G. Galasso, University of Udine, Italy. Proceedings of EPAC 2006, Edinburgh, Scotland.

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, Italy. SRF 2001.

SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

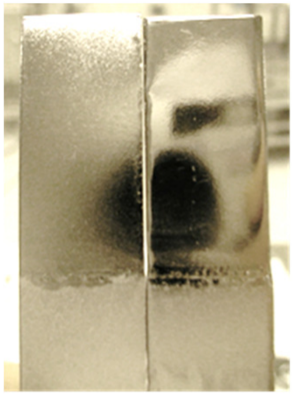
BCP Tools at Other Labs

http://uspas.fnal.gov/materials/08UMD/Surface_Preparation.pdf

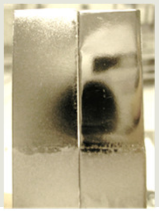
https://www.bnl.gov/cad/ardd/Chemical_Processing.asp

Commissioning of JLAB Vertical Cavity Processing System for SRF Nb Single Cell and Multi-cell Cavity with HF-Free Pulse-Reversed Electropolishing, H. Tian, L. Phillips, J. Musson, C. Seaton, M. Lester, and C. E. Reece Jefferson Lab, Newport News, VA 23606, USA. IPAC2018, Vancouver, BC. Canada.

SRF Activities at IPN Orsay, Sebastien Bousson, CNRS/IN2P3/ IPN Orsay – Paris-Sud University, MSU – 11th August 2011



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

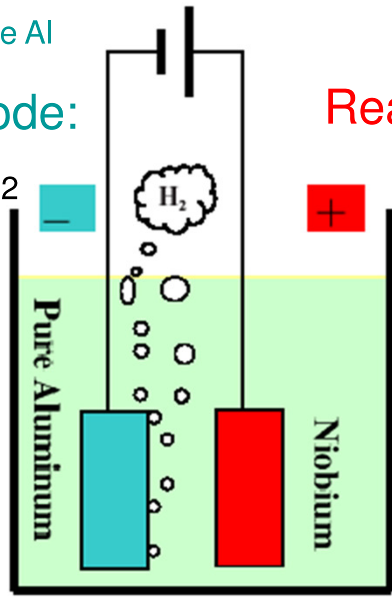
Overall: $2Nb + 10HF + 2H_2O \rightarrow 2H_2NbOF_5 + 5H_2 \uparrow$

Al-1350-O EC, 99.5% pure Al

Reaction at cathode:

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

Hydrogen gas produced



Reactions at anode:

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

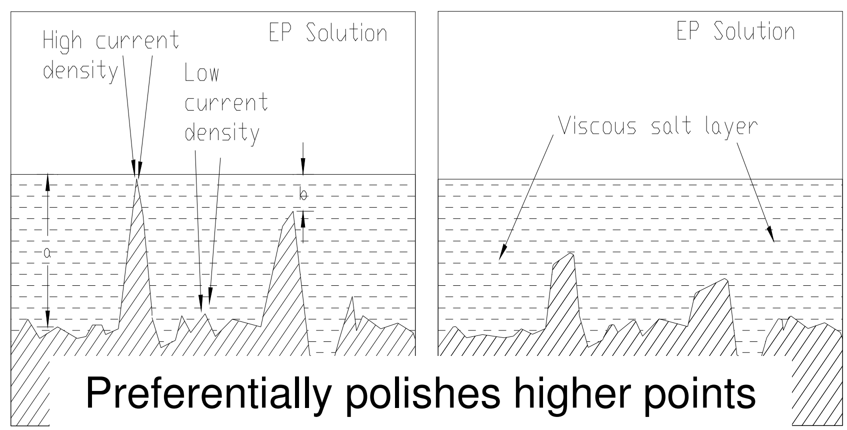
$2H_20 \rightarrow O2 + 4H+ + 4e-$

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

Acid Mixture:

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

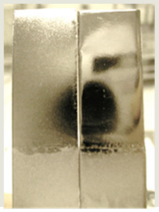
Sulfuric acid is source of free ions



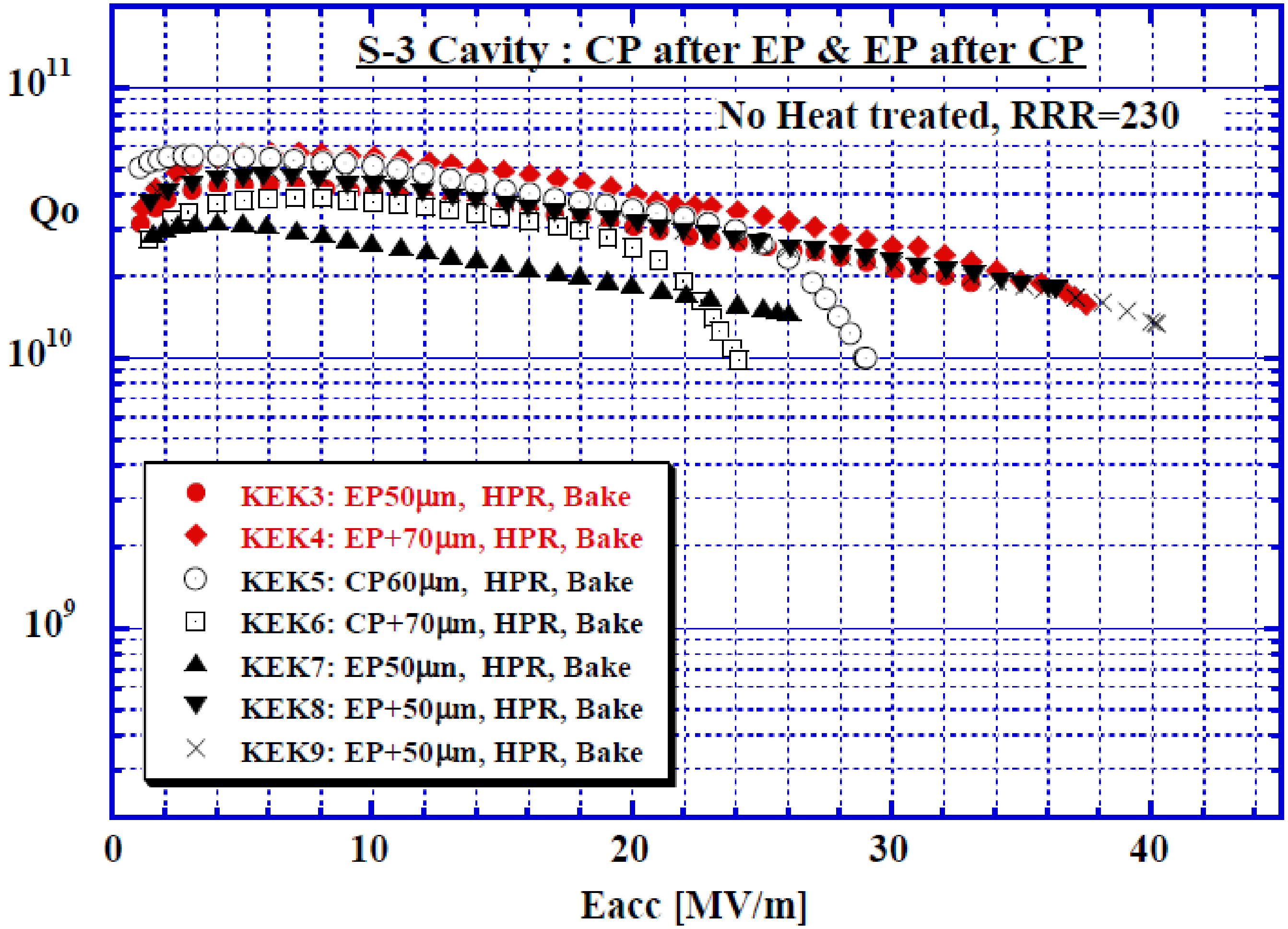
Preferentially polishes higher points

Kenji Saito, Lecture note in Tokyo University on May 2011

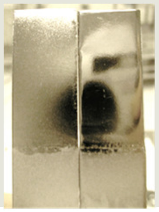
Development of Electropolishing Technology for Superconducting Cavities, K. Saito, KEK, 1-1 Oho, Tsukuba-shi, Ibaraki-ken, Japan. Proceedings of the 2003 Particle Accelerator Conference.



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

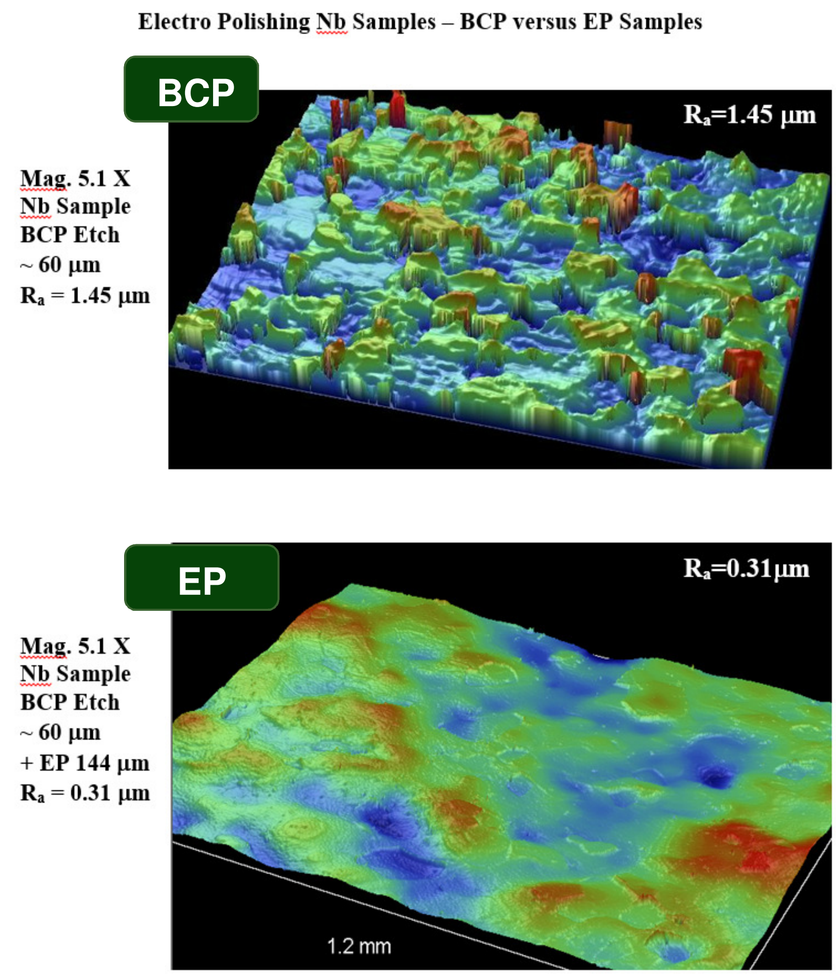


Development of Electropolishing Technology for Superconducting Cavities, K. Saito, KEK, 1-1 Oho, Tsukuba-shi, Ibaraki-ken, Japan. Proceedings of the 2003 Particle Accelerator Conference.



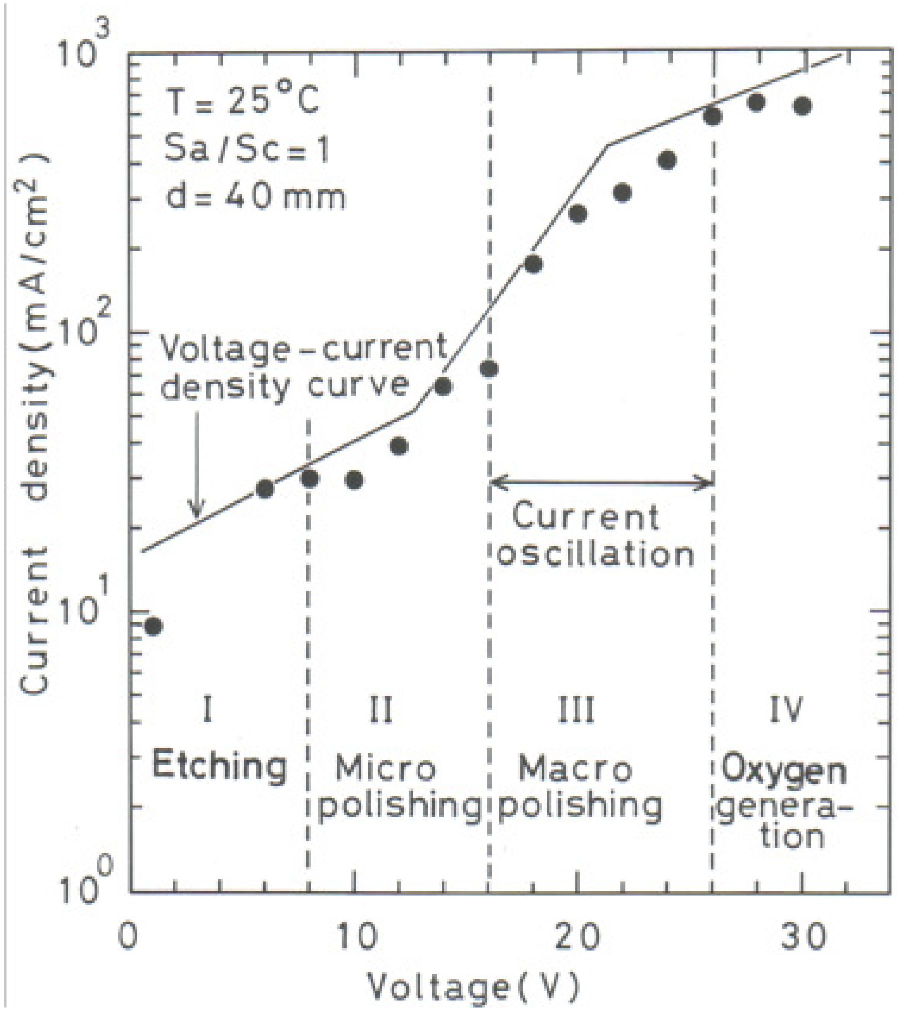
Some Comments on EP

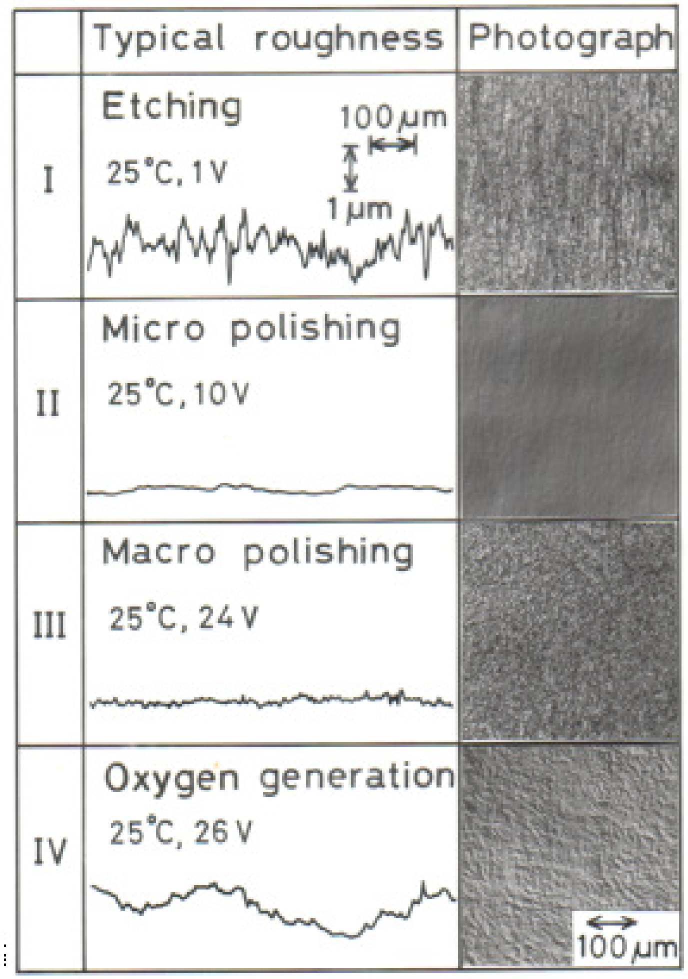
- **On EP samples roughness drops below 1 um after 150 um of material removed** (L. Lilje, Improved Surface Treatment of SC TESLA Cavities)
- **The main difference between BCP and EP is the smoothening of the grain boundaries**
- Increases gradients, up to 40 MV/m, fundamental limit
- Decreases Q-slope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



Development of Electropolishing Technology for Superconducting Cavities, K. Saito, KEK, 1-1 Oho, Tsukuba-shi, Ibaraki-ken, Japan. Proceedings of the 2003 Particle Accelerator Conference.

Electropolishing Characteristics With Nb

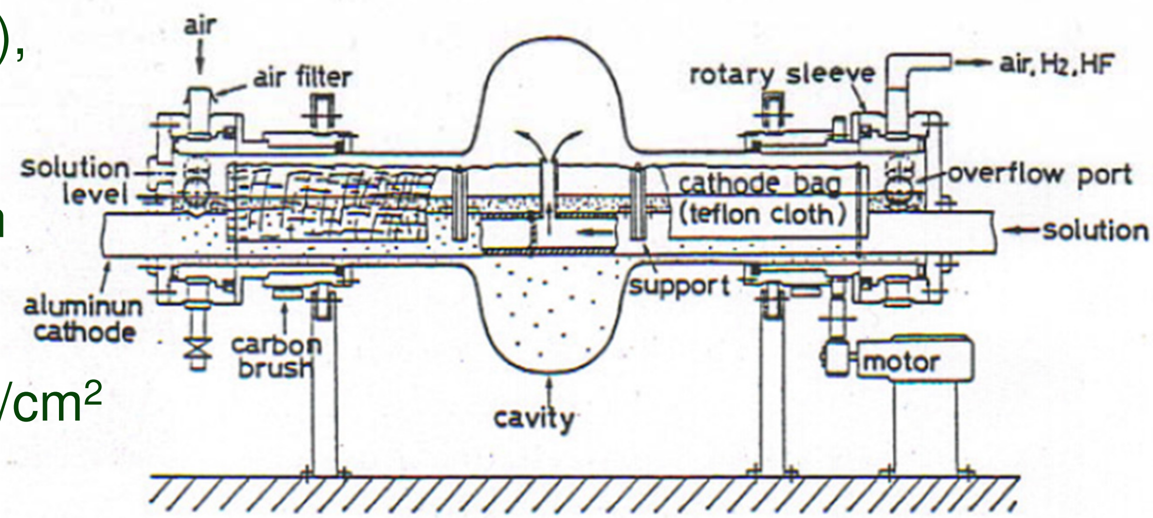


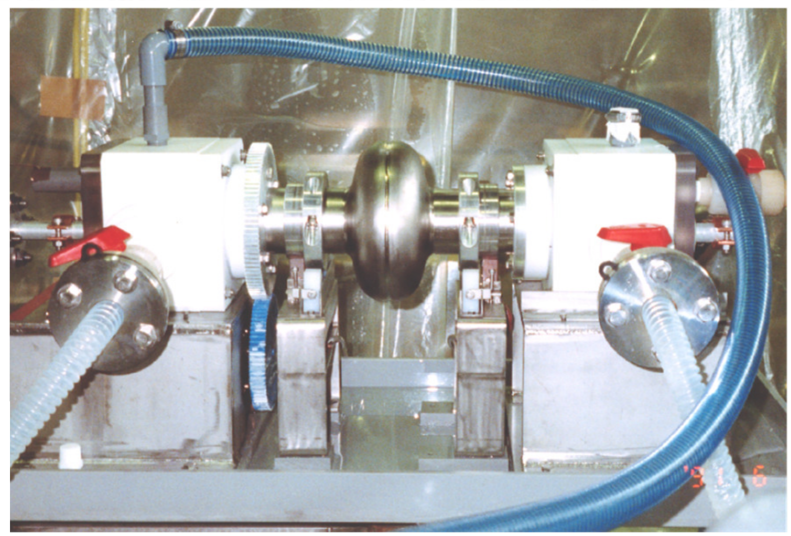


R & D of Superconducting Cavities at K E K, Kenji SAITO, Yuzo KOJIMA, Takaaki FURUYA, Shinji MITSUNOBU, Shuichi NOGUCHI, Kenji HOSOYAMA, Toshiharu NAKAZATO1, Tsuyoshi TAJIMA, Kiyomitsu ASANO KEK, National Laboratory for High Energy Physics 1-1 Oho, Tsukuba-shi, Ibaraki-ken, 305 Japan Keigo INOUE, Yohsuke IINO Mitsubishi Heavy Industries,Ltd., Nagoya Aircraft works 10, Oye-cho, Minato-ku, Nagoya, 455 Japan Hirotoshi NOMURA Plating, Co., Ltd. 5-12-20 Himejima, Nishiyodogawa-ku, Osaka, 555 Japan and Koichi TAKEUCHI Tokyo Denkai, Ltd. 1-3-20 Higashisuna, Kohtoh-ku, Tokyo, 136 Japan. Proceedings of the Fourth Workshop on RF Superconductivity, KEK, Tsukuba, Japan.

EP General Process Constraints

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

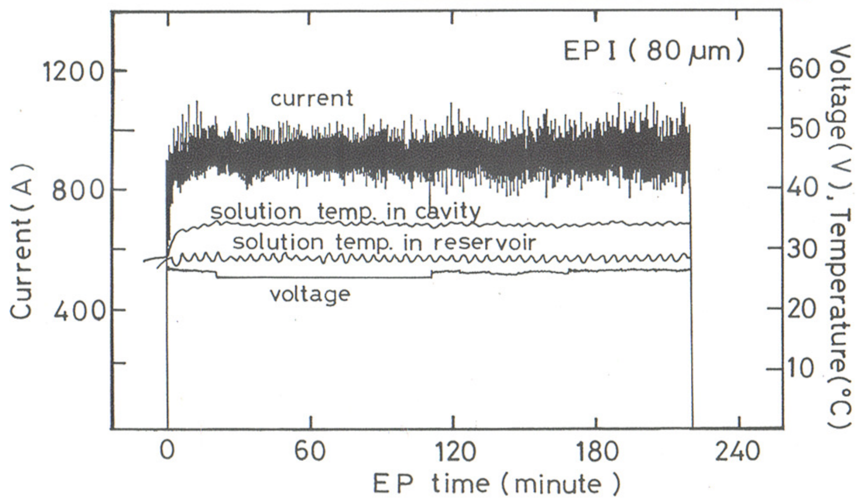


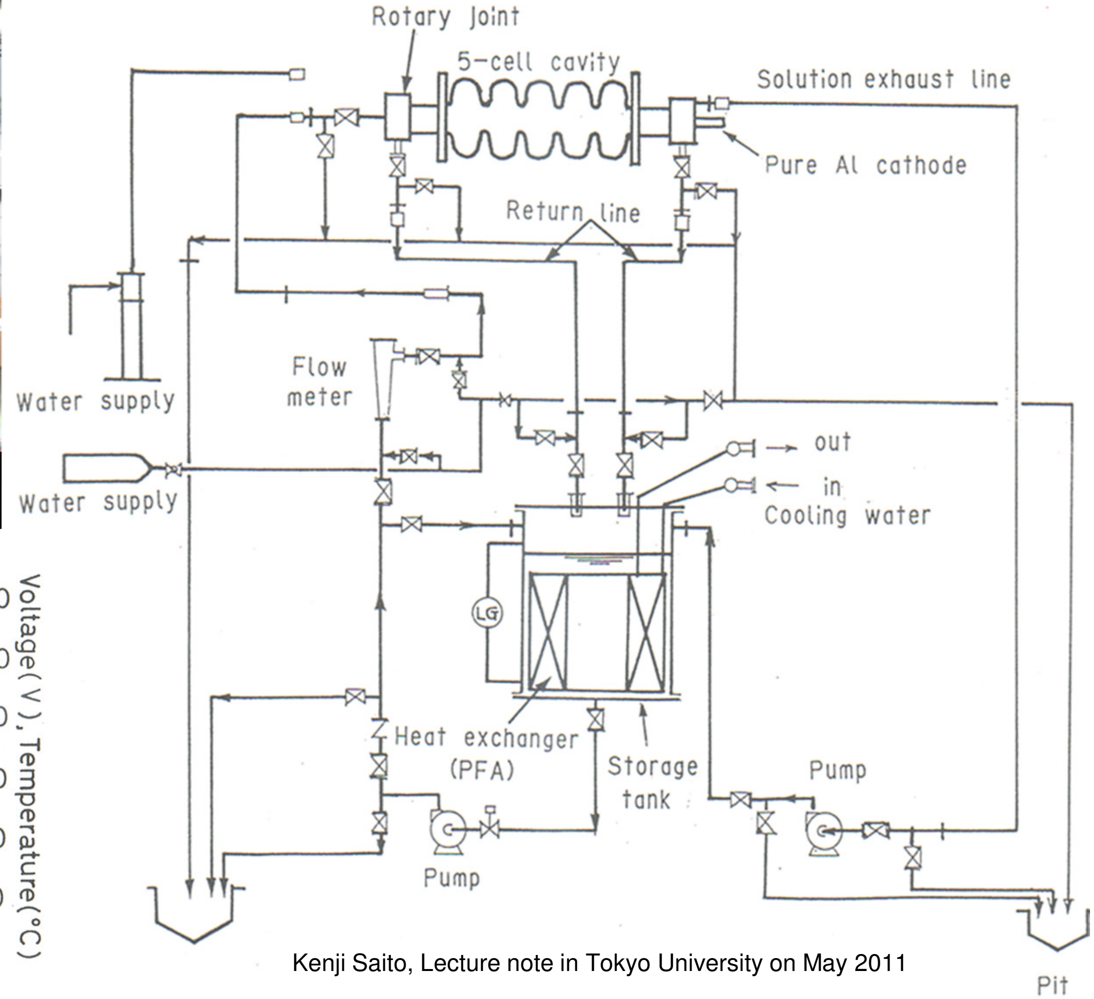


Kenji Saito, Lecture note in Tokyo University on May 2011
Bhashyam, S. "Comparison of Electropolishing and Buffered Chemical Polishing – A Literature Review", TD-03-046
Schultz, E., "Engineering Solutions for the Electro-Polishing of Multi-Cell Superconducting Accelerator Structures"
Geng, R.L. "Continuous Current Oscillation Electropolishing and Applications to Half-Cells"
Padamsee, H., "RF Superconductivity for Accelerators"
Development of Electropolishing Technology for Superconducting Cavities, K. Saito, KEK, 1-1 Oho, Tsukuba-shi, Ibaraki-ken, Japan. Proceedings of the 2003 Particle Accelerator Conference.

EP Process Diagram

TRISTAN 508MHz 5-cell cavity

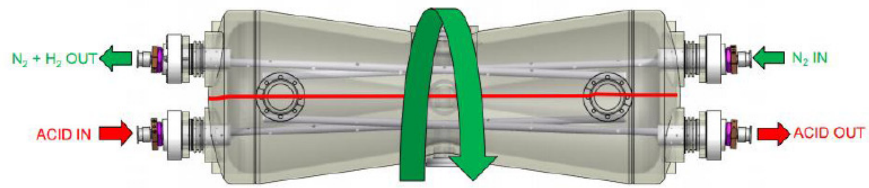


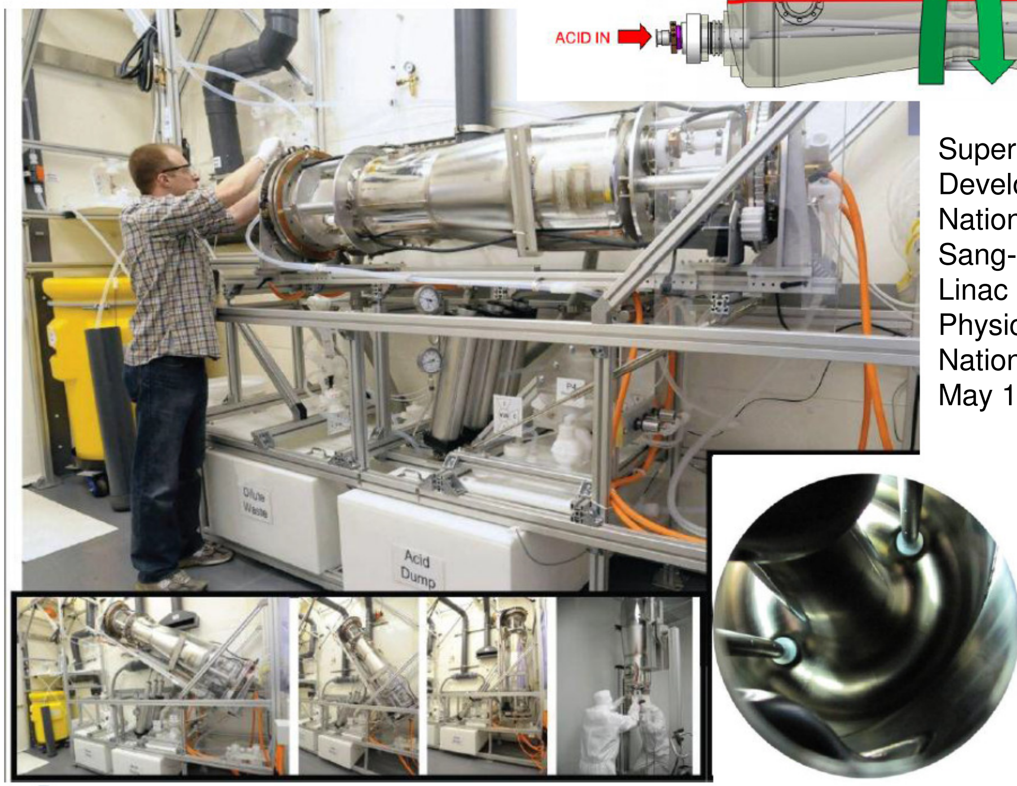


Kenji Saito, Lecture note in Tokyo University on May 2011

R & D of Superconducting Cavities at K E K, Kenji SAITO, Yuzo KOJIMA, Takaaki FURUYA, Shinji MITSUNOBU, Shuichi NOGUCHI, Kenji HOSOYAMA, Toshiharu NAKAZATO1, Tsuyoshi TAJIMA, Kiyomitsu ASANO KEK, National Laboratory for High Energy Physics 1-1 Oho, Tsukuba-shi, Ibaraki-ken, 305 Japan Keigo INOUE, Yohsuke IINO Mitsubishi Heavy Industries,Ltd., Nagoya Aircraft works 10, Oye-cho, Minato-ku, Nagoya, 455 Japan Hirotoshi NOMURA Plating, Co., Ltd. 5-12-20 Himejima, Nishiyodogawa-ku, Osaka, 555 Japan and Koichi TAKEUCHI Tokyo Denkai, Ltd. 1-3-20 Higashisuna, Kohtoh-ku, Tokyo, 136 Japan. Proceedings of the Fourth Workshop on RF Superconductivity, KEK, Tsukuba, Japan.

Half Wave Resonator at ANL



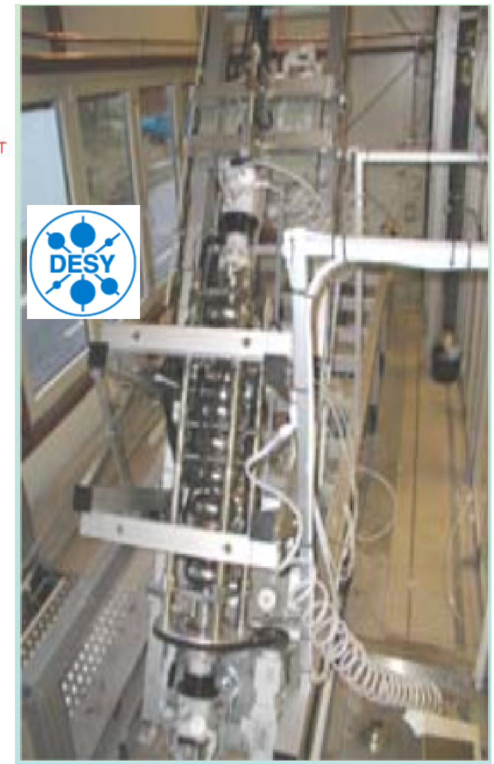


Superconducting RF Cavities Development at Argonne National Laboratory
Sang-hoon Kim on behalf of Linac Development Group in Physics Division at Argonne National Laboratory
May 10, 2014

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

Preparation Sequences for Electro-polished High Gradient Multi-cell Cavities at DESY, A.Matheisen, B.v.d.Horst , B.Petersen; S.Sägebarth; P.Schilling, Deutsches Elektronen Synchrotron DESY, Hamburg, Notkestraße 85, 22602 Hamburg, Germany

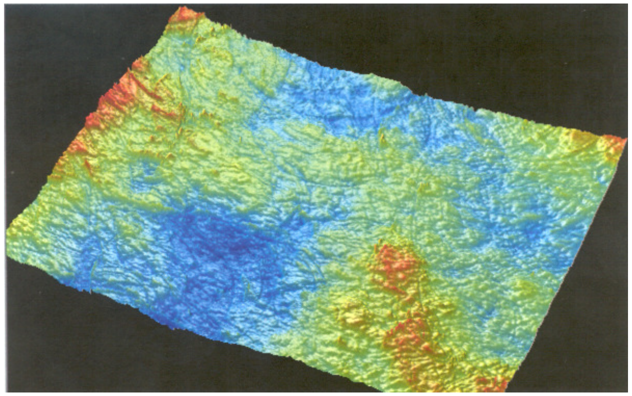
Current State of Electropolishing at ANL, T. Reid, R. Murphy, M. P. Kelly, S. M. Gerbick Argonne National Laboratory, Argonne, IL 60439, U.S.A. Proceedings of SRF2011, Chicago, IL, U.S.A.

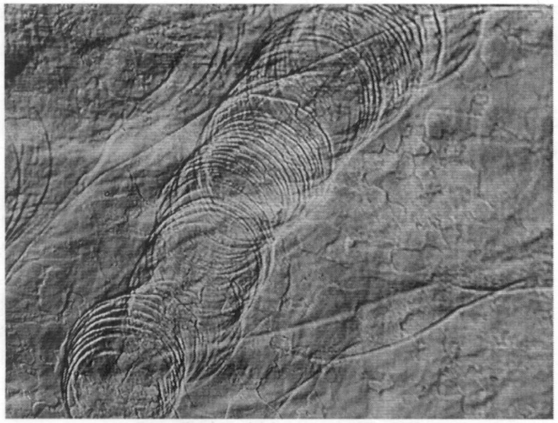
Superconducting RF Cavities Development at Argonne National Laboratory
AKPA - IBS Symposium on Special Topics in Physics, May 9-10, 2014, The University of Chicago. Sang-hoon Kim on behalf of Linac Development Group in Physics Division at Argonne National Laboratory.

Hydrogen Produced During EP

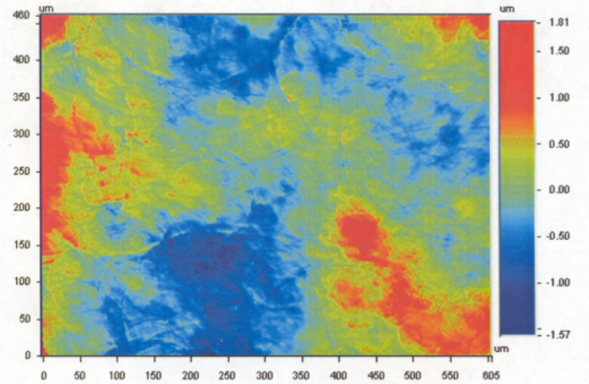
- PTFE mesh or bag used to cover the cathode to break and reduce hydrogen bubbles from reaching anode surface

10.2 X mag. 3-D Optical – 100+ μm EP





10.2 X mag. 2-D Optical – 100+ μm EP



Basic Studies for the Electropolishing Facility at DESY, N. Steinhau-Kühl; R. Bandelmann, K. Escherich, D. Keese, M. Leenen, L. Lilje, A. Matheisen, H. Morales, P. Schmüser, E. Schulz, M. Seidel, J. Tiessen Deutsches Elektronen Synchrotron DESY, Hamburg, Notkestraße 85, 22602 Hamburg, Germany. SRF Workshop 2011.

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

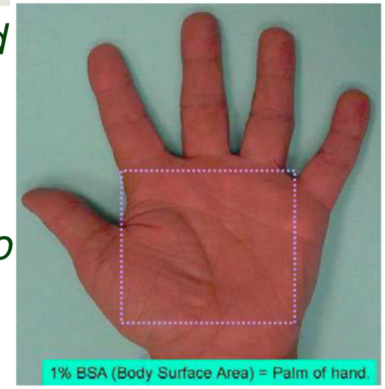
XFEL X-Ray Free-Electron Laser Update on the Experiences of Electro polishing of Multi-cell Resonators at DESY, N. Steinhau-Kühl, A. Matheisen, L. Lilje, B. Petersen, M. Schmökel, H. Weitkämper, Deutsches Elektronen Synchrotron DESY, Hamburg, Notkestraße 85,22602 Hamburg, Germany

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

Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

SRF Cavity Processing and Chemical Etching Development for the FRIB LINAC, I. Malloch, M. LaVere, E. Metzgar, L. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, U.S.A. Proceedings of SRF 2015, Whistler, BC, Canada.

Chemical Safety Data Sheets.
BCP: https://www.puritanproducts.com/wp-content/uploads/products/BUFF_CHEM_50_25_25_SDS_6940.pdf
NO2: https://www.airgas.com/msds/001041.pdf

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

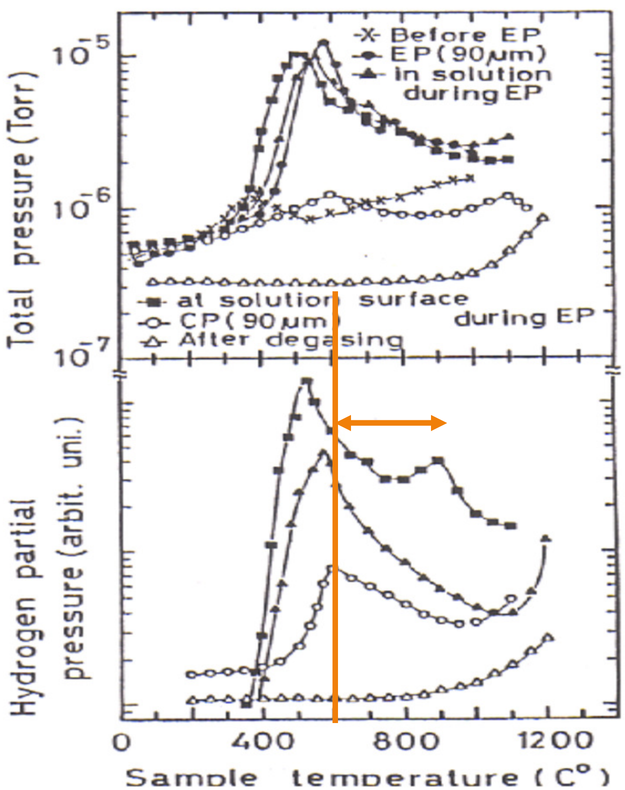
First aide triage response to HF: rinse 3-5 min. and start applying calcium gluconate

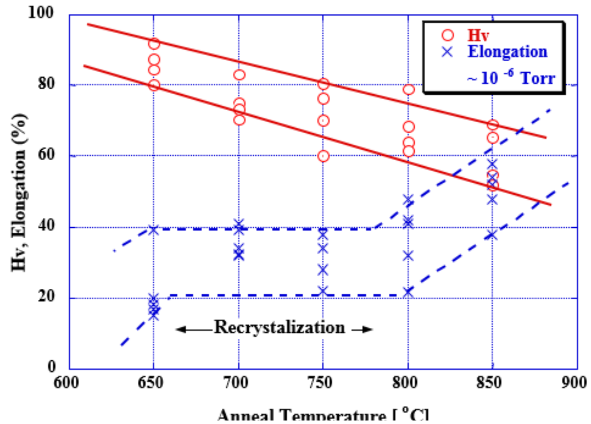
Chemical Safety Data Sheets.
BCP: https://www.puritanproducts.com/wp-content/uploads/products/BUFF_CHEM_50_25_25_SDS_6940.pdf
NO2: https://www.airgas.com/msds/001041.pdf

9. Heat Treatments

Why Heat Treatment?

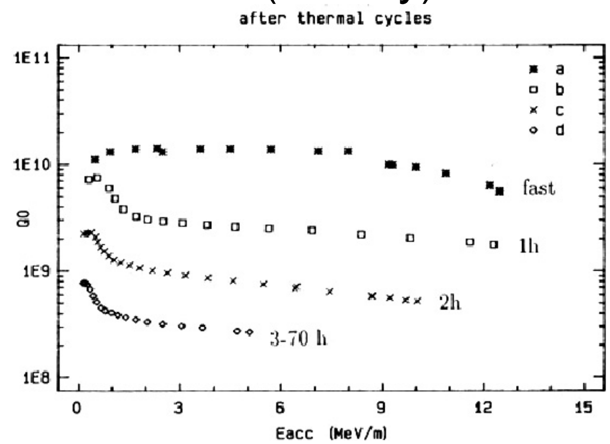
- Observe Q_0 drop after niobium soak around 100 K for 1+ hours
- Fast cool down of niobium < 1 hr is required to avoid the Q_0 drop (disease)
- Hydrogen degasification proven to eliminate the Q_0 drop even during a slow cool down
- Degassing effective starting ~600 °C





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

(Saclay)



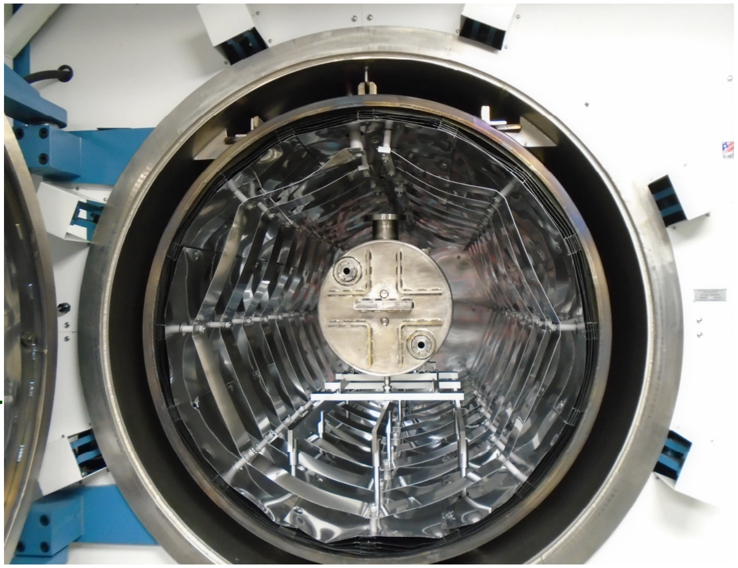
Q_0 drop after various soak times ~100 K

K. Saito, et al.,in Proceedings of the 4th Workshop on RF Superconductivity (SRF 1989), Tsukuba, Japan, 1989, edited by Y. Kojima (National Laboratory for High-Energy Physics (KEK), Tsukuba, Japan, 1990), pp. 635–694.

Q Degradation of Niboium Cavities due to Hydrogen Contamination, B. Bonin, Groupe d'Etude des Cavites Supraconductrices, DSM/DphN/STAS, CEN Saclay, 91191 Gifsur Yvette, France, R.W. ROTH Fb 08 / Experimentalphysik, Bergische Universitiit Wuppertal Gaussstrasse 20, Postfach 100127, 5600 Wuppertal 1, Germany. Particle Accelerators, 1992, Vol. 40, pp.59-83

Heat Treatment to Remove Hydrogen Uptake from Processing

- Risk of Q-disease eliminated by hydrogen degassing step after the bulk chemistry
- 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



Various recipes used world-wide

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

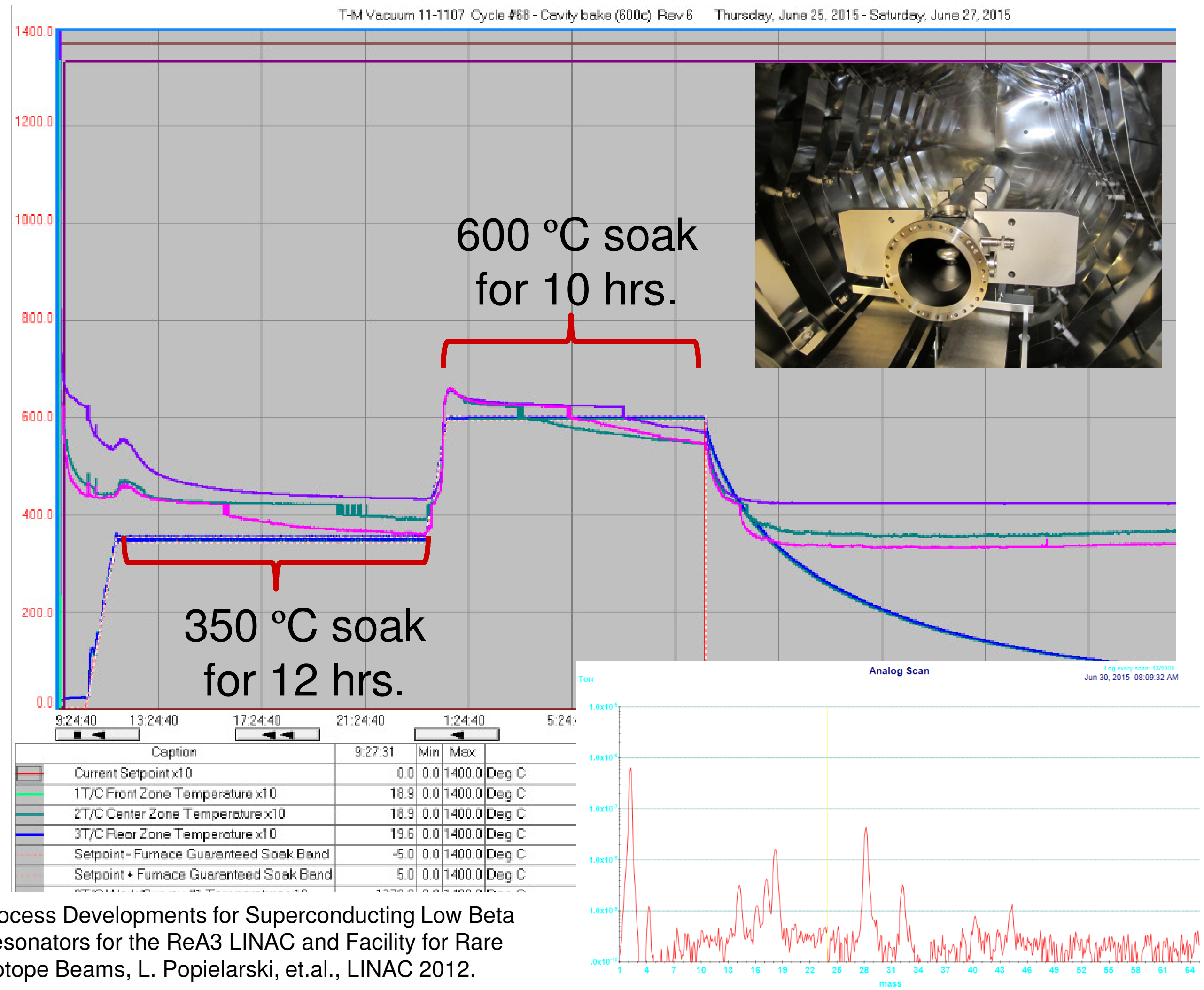
Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

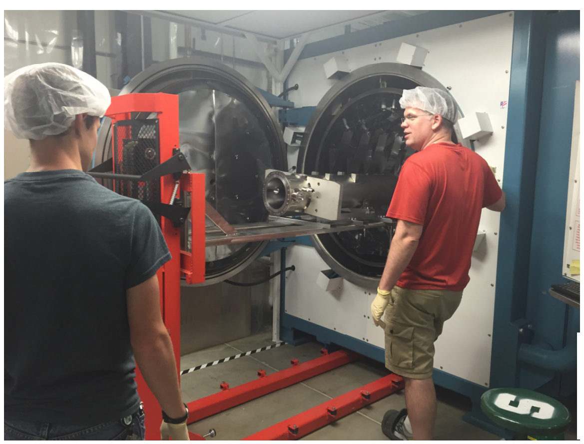
Proceedings of the 4th Workshop on RF Superconductivity (SRF 1989), Tsukuba, Japan, 1989, edited by Y. Kojima (National Laboratory for High-Energy Physics (KEK), Tsukuba, Japan, 1990), pp. 635–694.

Heat Treatment Furnace Operation

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

http://inspirehep.net/record/1482071/files/tupb022.pdf

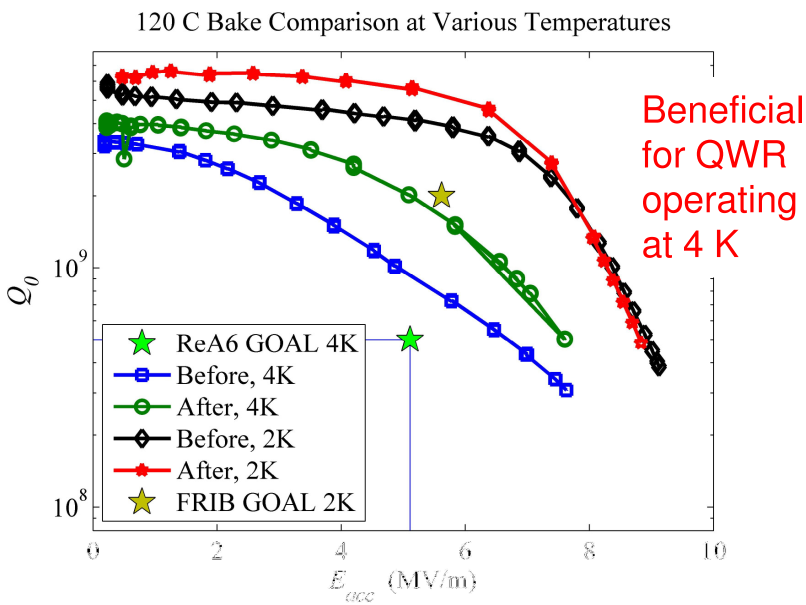


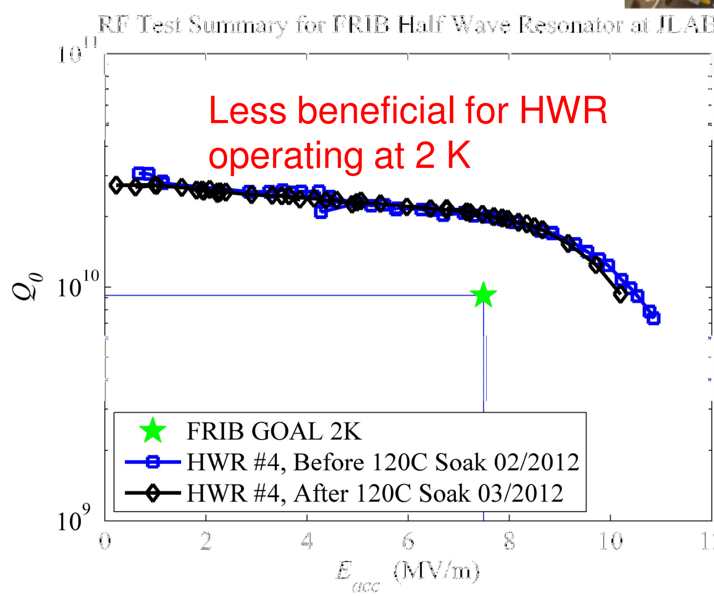


Process Developments for Superconducting Low Beta Resonators for the ReA3 LINAC and Facility for Rare Isotope Beams, L. Popielarski, et.al., LINAC 2012.

120 ºC Low Temperature Bake

- Bake at 100 ºC-150 ºC 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
- Related to oxygen diffusion into the niobium, causing changes of the structure niobium/oxide interface on a nanometer scale



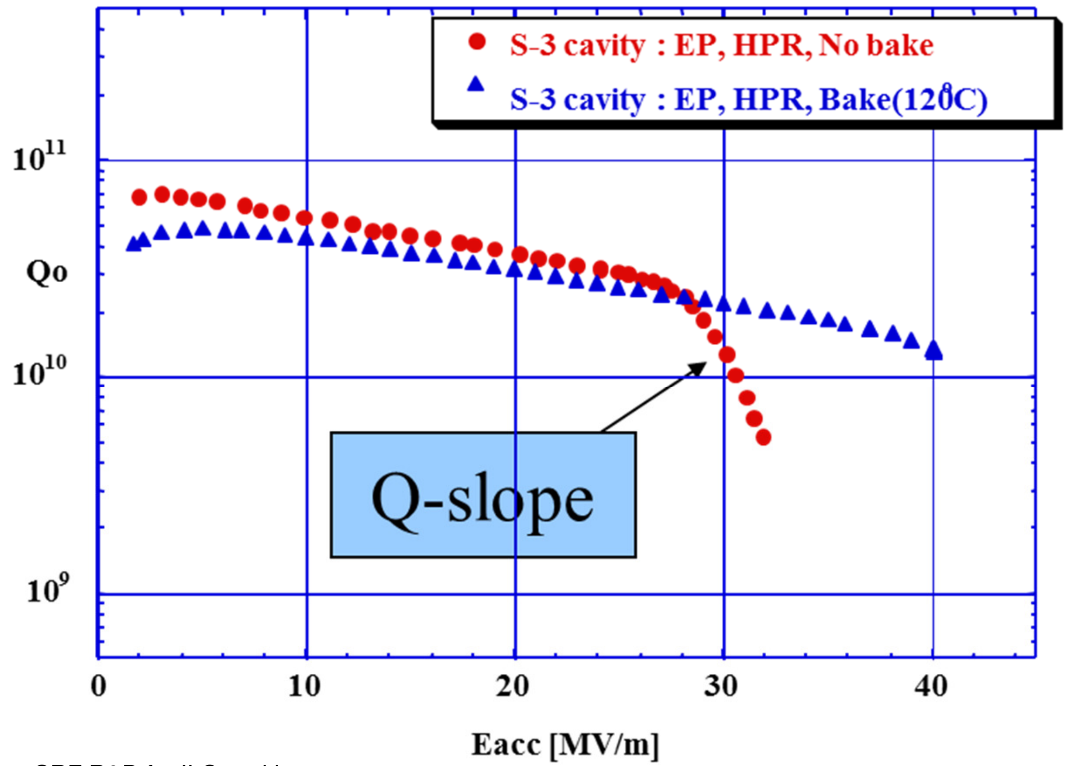


Effect of Low Temperature Baking on Niobium Cavities, G. Ciovati, P. Kneisel, G. Myneni, Jefferson Lab, Newport News, VA 23606, W. A. Lanford, Department of Physics, SUNY Albany, Albany, NY 12222, SRF 2003

Process Developments for Superconducting Low Beta Resonators for the ReA3 LINAC and Facility for Rare Isotope Beams, L. Popielarski, et.al., LINAC 2012.

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



Geng, Rongli. Sun . "Overview of high gradient SRF R&D for ILC cavities at Jefferson Lab". United States. https://www.osti.gov/servlets/purl/1115723.

Kenji Saito, Lecture note in Tokyo University on May 2011

10. Cleanroom Protocols

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

Why do we use cleanrooms?

- Cleanrooms are 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!

Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, CARE Conf-05-033-SRF.

First FRIB Beta=0.53 Prototype Coldmass Build, D. Victory, K. Elliott, B. Oja, J. Popielarski, M. Wilbur, LINAC 2016.

What is Contamination?

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

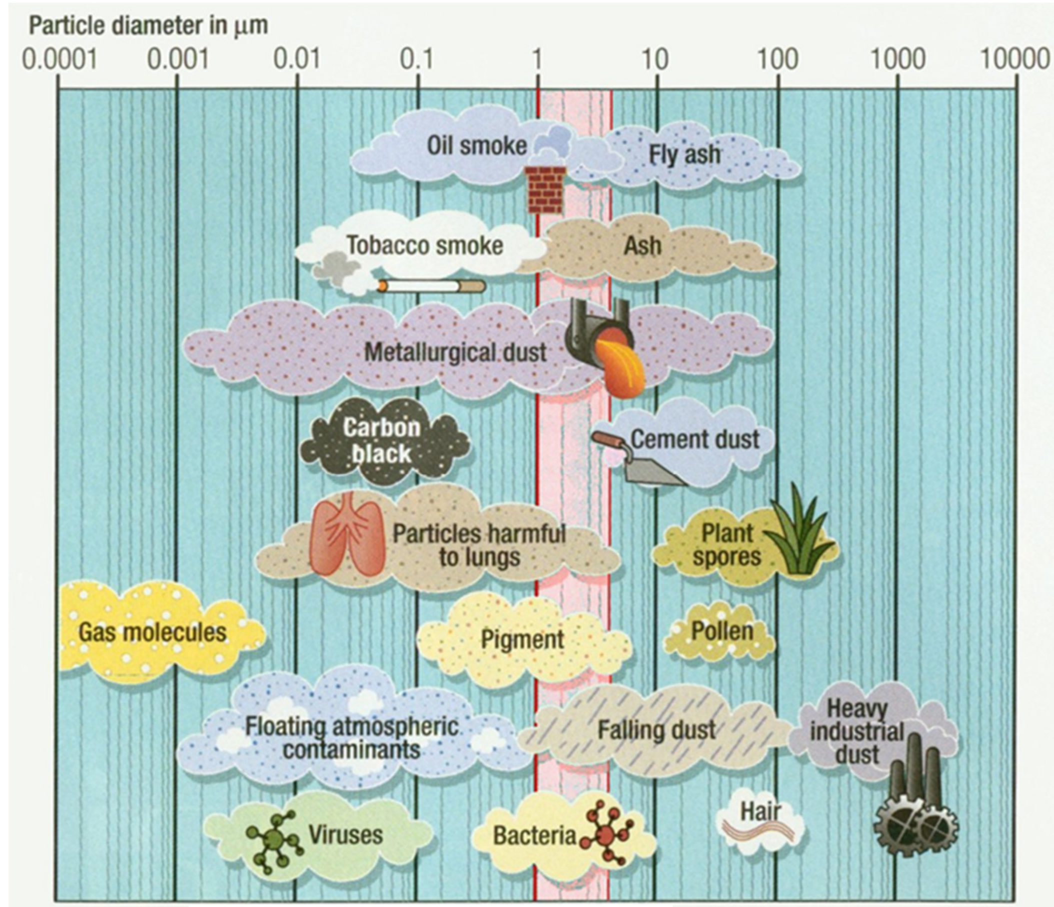
Facilities	People	Tool Generated	Fluids	Product generated
Air conditioning debris	Clothing debris (lint, fibers etc.)	Brooms, mops and dusters	Cleaning chemicals	Aluminum particles
Construction material (sheet rock, saw dust etc.)	Spittle	Vibrations	Floor finishes or coatings	Cleanroom debris
Paint and 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	

Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.

Quality Control of Cleanroom Processing Procedure of SRF Cavities for Mass Production, R. Oweiss, K. Elliott, A. Facco, M. Hodek, I. Malloch, J. Popielarski, L. Popielarski, K. Saito Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA +INFN - Laboratori Nazionali di Legnaro, Padova, Italy. LINAC 2012.

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

Quality Control of Cleanroom Processing Procedure of SRF Cavities for Mass Production, R. Oweiss, K. Elliott, A. Facco, M. Hodek, I. Malloch, J. Popielarski, L. Popielarski, K. Saito Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA +INFN - Laboratori Nazionali di Legnaro, Padova, Italy. LINAC 2012.
Reschke, Detlef. "Cleanliness techniques." 12th International Workshop on RF Superconductivity, Cornell, USA. 2005.

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

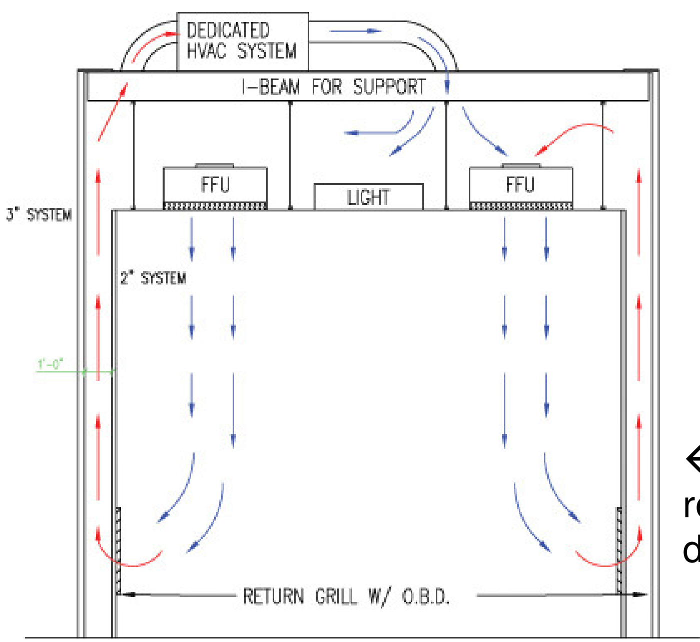
SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, LINAC 2014.

CERN SRF Assembling and Testing Facilities, J. Chambrillon, M. Therasse, O. Brunner, P. Maesen, O. Pirotte, B. Vullierme, W. Weingarten CERN, Geneva, Switzerland. SRF2011.

W. Whyte (17 October 2001). Cleanroom Technology: Fundamentals of Design, Testing and Operation. John Wiley & Sons. ISBN 978-0-471-86842-2.

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

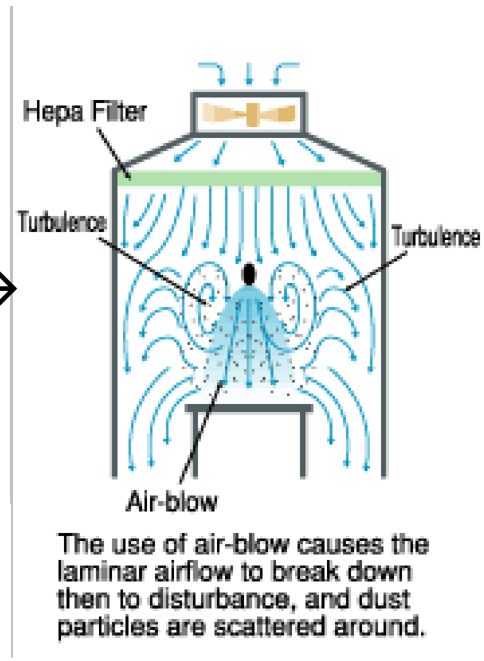


Figure 1: Turbulent Flow

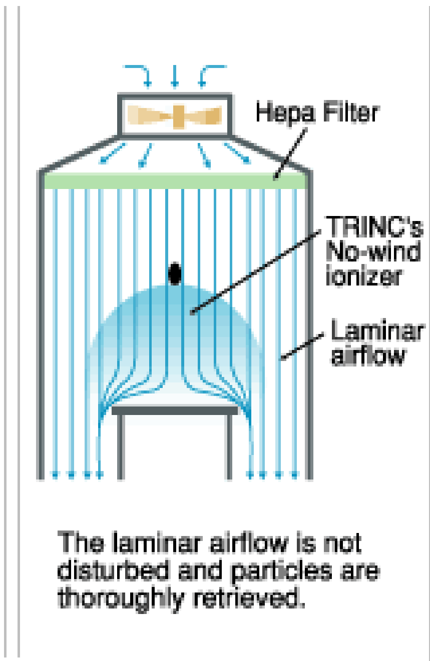


Figure 2: Laminar Flow

W. Whyte (17 October 2001). Cleanroom Technology: Fundamentals of Design, Testing and Operation. John Wiley & Sons. ISBN 978-0-471-86842-2.

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

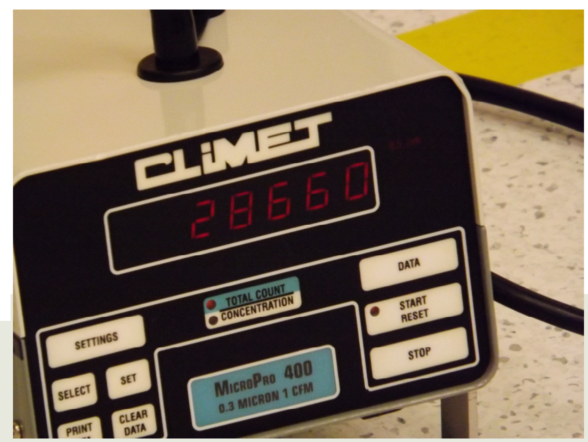
Table 1 Federal Standard 209 class limits

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

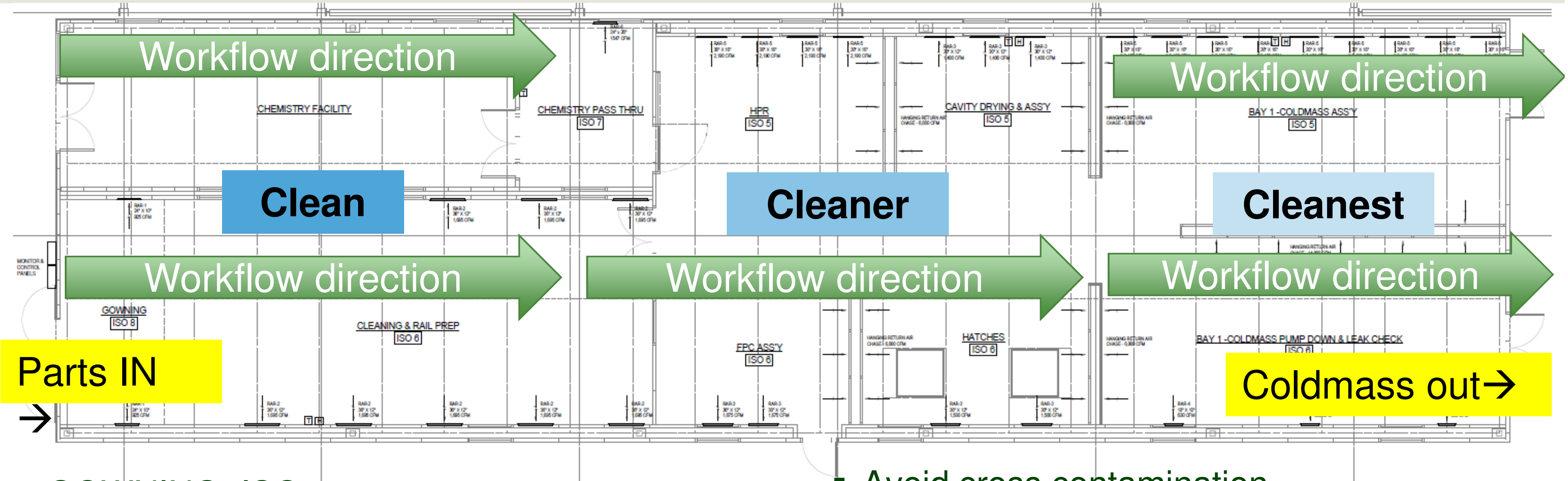
ISO 14644-1 Classes	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8
FS 209 Classes	Class 1	Class 10	Class 100	Class 1000	Class 10 000	Class 6 100 000

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



W. Whyte (17 October 2001). Cleanroom Technology: Fundamentals of Design, Testing and Operation. John Wiley & Sons. ISBN 978-0-471-86842-2.

SRF Production String Assembly Cleanroom Classification

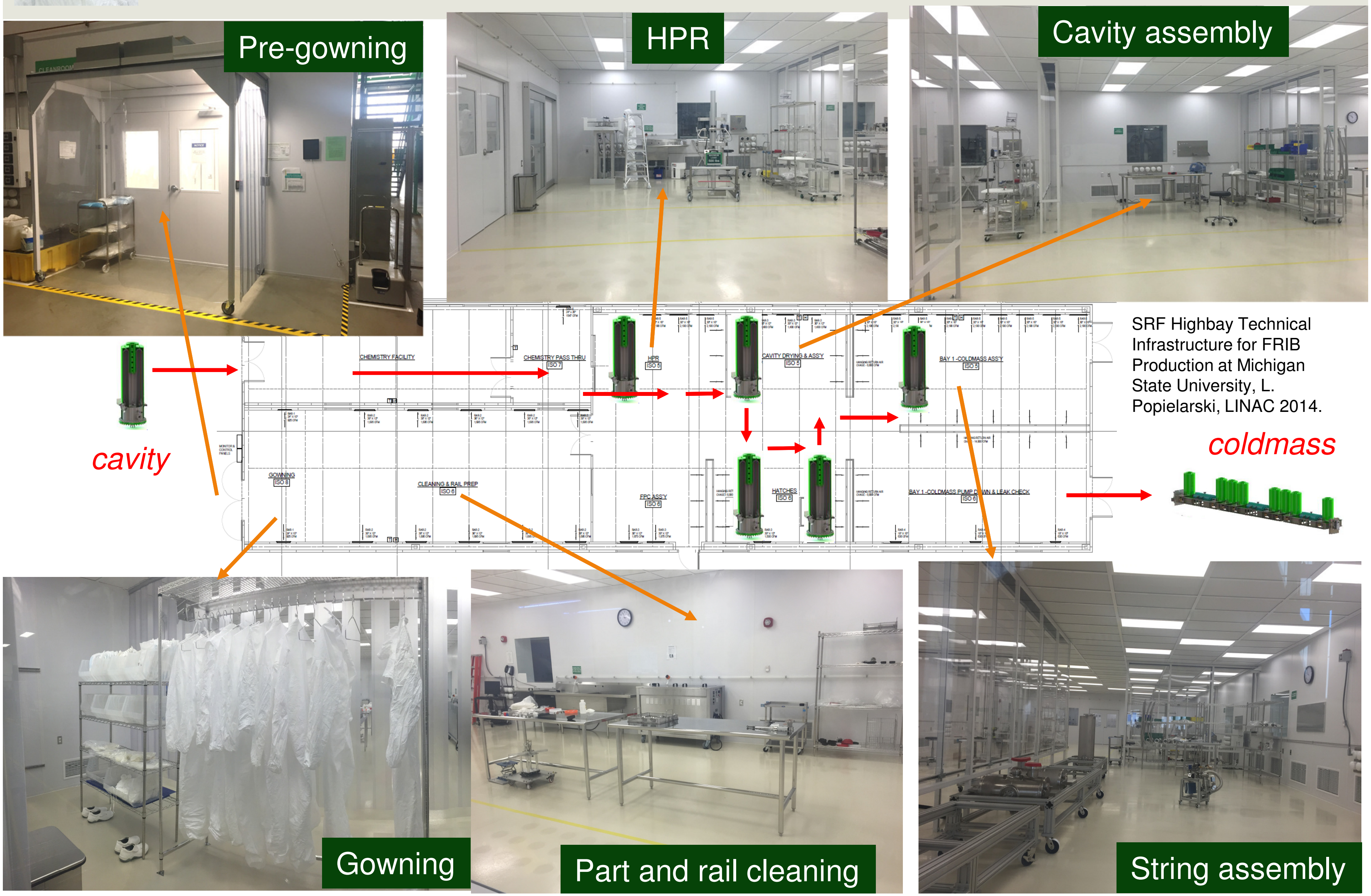


- GOWNING: ISO 8
- CLEANING & RAIL PREP: ISO 6
- HPR: ISO 5 & FPC ASS'Y: ISO 6
- CAVITY DRYING & ASS'Y: ISO 5
- HATCHES: ISO 6
- COLDMAS ASS'Y BAY 1: ISO 5
- COLDMASS PUMP DOWN & LEAK CHECK: ISO 6

- 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

SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, LINAC 2014.

Tour of a Production SRF Cleanroom



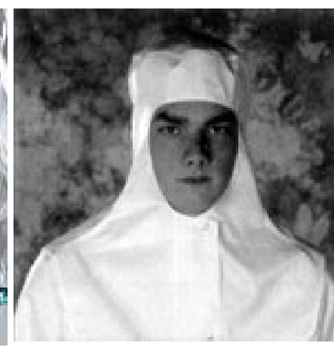
SRF Highbay Technical Infrastructure for FRIB Production at Michigan State University, L. Popielarski, LINAC 2014.

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
- Prohibiting risky items and behaviors
- Regular maintenance focusing on high traffic areas

ALL COVERALLS MANUFACTURED IN CLEANROOMS! CHECK ISO CERT.



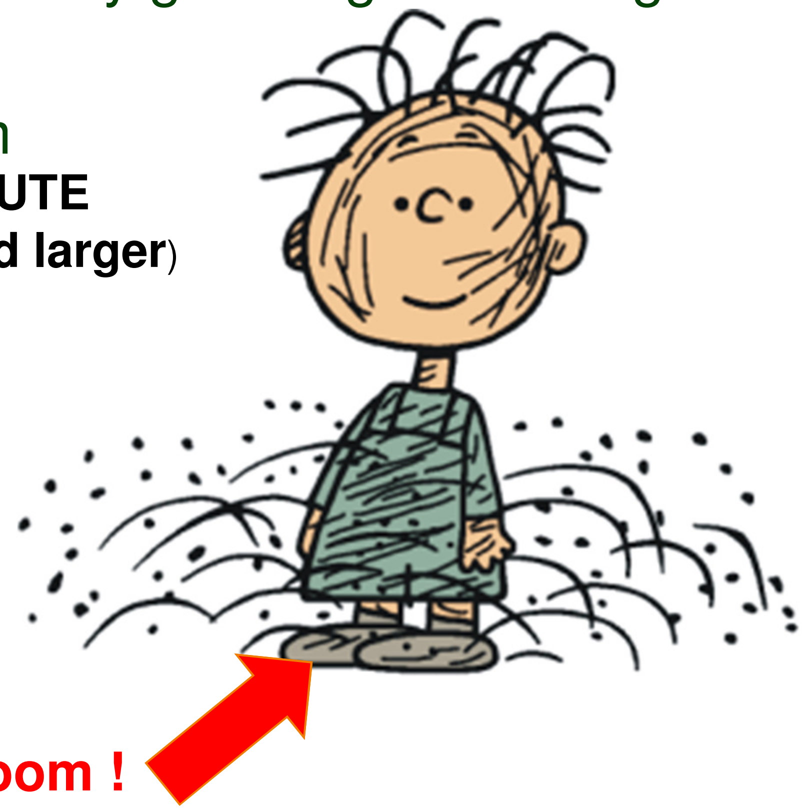




Microchemical Journal,45, 336-342 (1992) Human Contamination Control in a Clean Room’ S. VIDALI LIS Linen Supply, Italiana SpA Piazza De Angeli, 9-20146 Milan, Italy Received August 13, 1991; accepted February 1992

Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, CARE Conf-05-033-SRF.

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



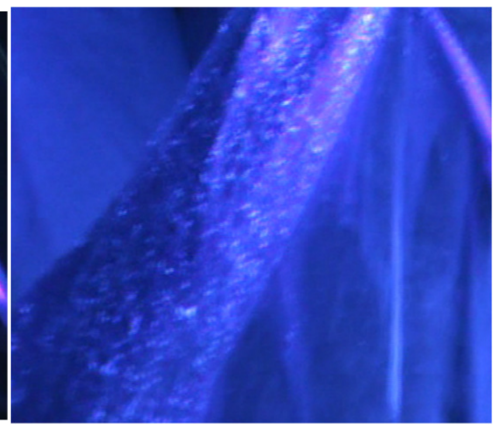
PEOPLE ACTIVITY	PARTICLES/MINUTE (0.3 microns and larger)
Motionless (Standing or Seated)	100,000
Walking about 2 mph	5,000,000
Walking about 3.5 mph	7,000,000
Walking about 5 mph	10,000,000
Horseplay	100,000,000

This is what people look like to a cleanroom !

Microchemical Journal,45, 336-342 (1992) Human Contamination Control in a Clean Room' S. VIDALI LIS Linen Supply, Italiana SpA Piazza De Angeli, 9-20146 Milan, Italy Received August 13, 1991; accepted February 1992
Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, CARE Conf-05-033-SRF.

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

Microchemical Journal,45, 336-342 (1992) Human Contamination Control in a Clean Room' S. VIDALI LIS Linen Supply, Italiana SpA Piazza De Angeli, 9-20146 Milan, Italy Received August 13, 1991; accepted February 1992

Optical contamination control in the Advanced LIGO ultra-high vacuum system, Margot H. Phelpsa, Kaitlin E. Gushwaa, and Calum I. Torriea,baLIGO Laboratory, California Institute of Technology, 1200 E. California Blvd., Pasadena, CA USA 91125; bSUPA, School of Physics and Astronomy, Kelvin Building, University of Glasgow, Glasgow G12 8QQ UK

General Cleanroom Concerns

- Similar metals gall in CR→ use approved grease or different materials
- 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.

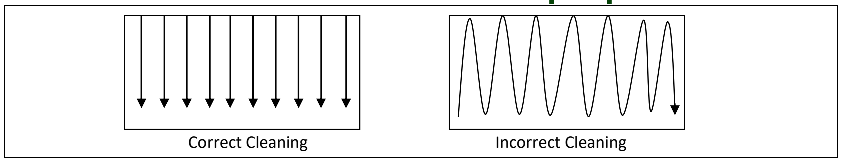
Production Status of SRF Cavities for the Facility for Rare Isotope Beams Project, C. Compton, A. Facco, S. Miller, J. Popielarski, L. Popielarski, A. Rauch, K. Saito, G. Velianoff, E. Wellman, K. Witgen, T. Xu, Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, 48824, U.S.A. SRF 2015

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

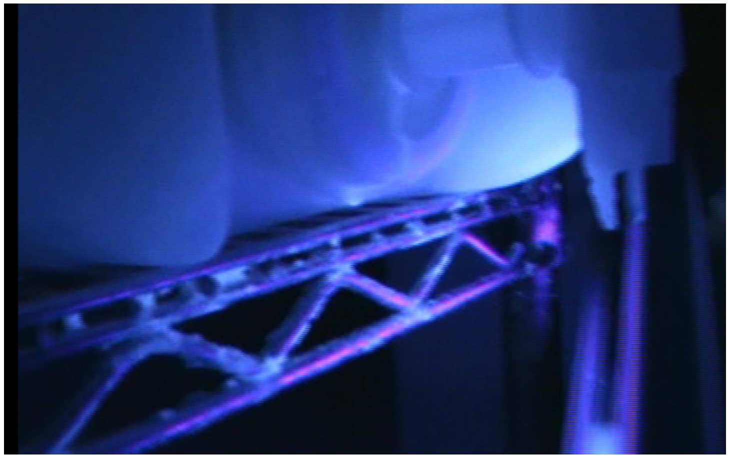
- Move sensitive parts away from doorway
- Maintain positive air pressure
- Wipe down all items brought into cleanroom with lint free wipers and ethanol

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

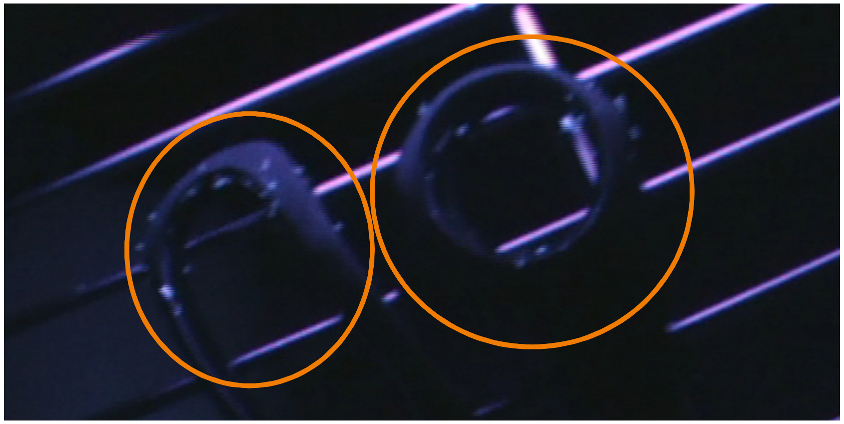


Quality Control of Cleanroom Processing Procedure of SRF Cavities for Mass Production, R. Oweiss, et.al., LINAC 2012.
Clean-room Facilities for High Gradient Resonator Preparation, K.Escherich, A.Matheisen, N.Krupka, B.Petersen, M.Schmökel, CARE Conf-05-033-SRF.
https://indico.cern.ch/event/555785/contributions/2283555/attachments/1352708/2043324/Cleanroom_practises_v6.pdf

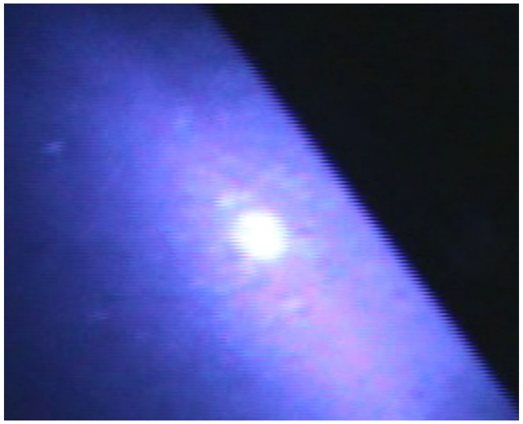
Contamination Detection Using Ultra Violet Lamp Fluorescence



Degreaser Dispenser



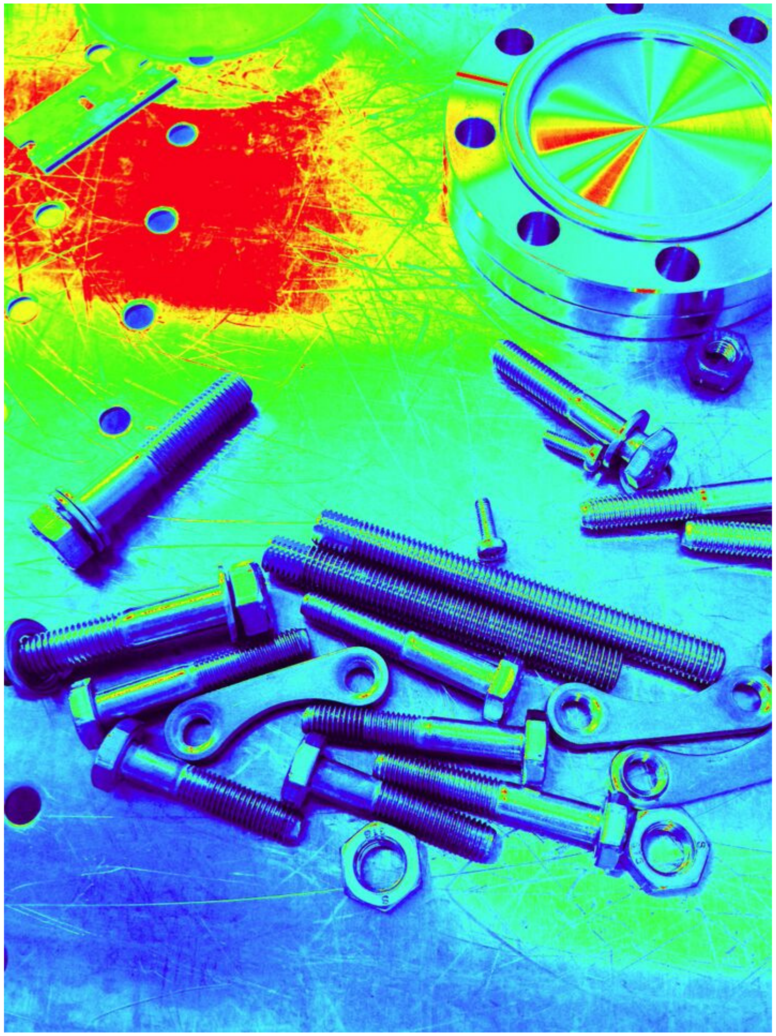
Detect detergents and water marks with UV light



Mop areas of high traffic often!

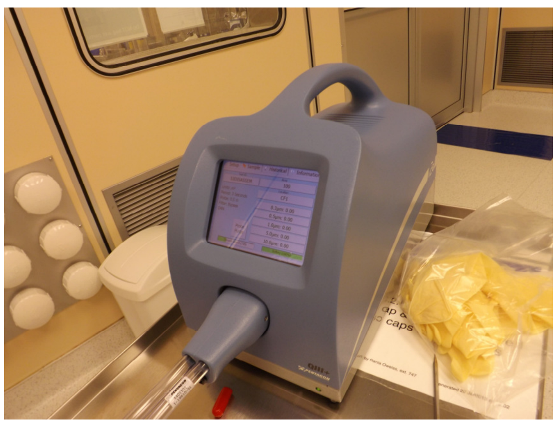
Optical contamination control in the Advanced LIGO ultra-high vacuum systemMargot H. Phelpsa, Kaitlin E. Gushwaa, and Calum I. Torriea,baLIGO Laboratory, California Institute of Technology, 1200 E. California Blvd., Pasadena, CA USA 91125; bSUPA, School of Physics and Astronomy, Kelvin Building, University of Glasgow, Glasgow G12 8QQ UK
https://www.degagecorp.com/education-detail.php?cid=22

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

INSTITUTE OF ENVIRONMENTAL SCIENCES AND TECHNOLOGY

MIL-STD-1246 C PRODUCT CLEANLINESS LEVELS CONTAMINATION

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

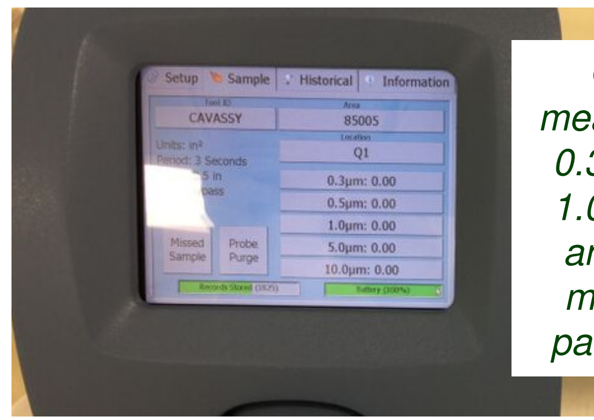
Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, L. Popielarski, L. Dubbs, K. Elliott, I. Malloch, R. Oweiss, J. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA. Proceedings of IPAC2012, New Orleans, Louisiana, USA

Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L.Popielarski, et.al., Proceedings of SRF2015, Whistler, BC, Canada.

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
- The probe does not touch RF surface but hovers closely to surface
- 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



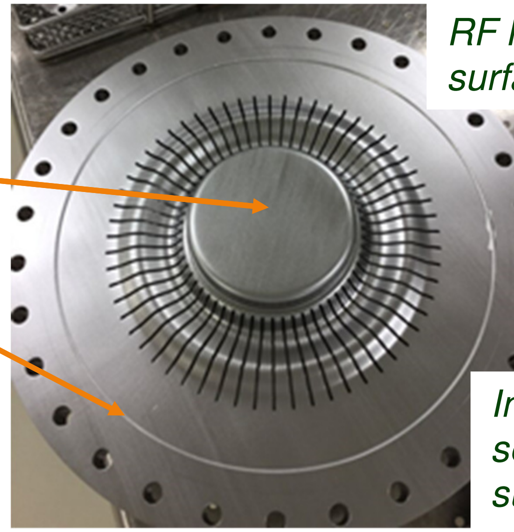
QIII measures 0.3, 0.5, 1.0, 5.0, and 10 micron particles

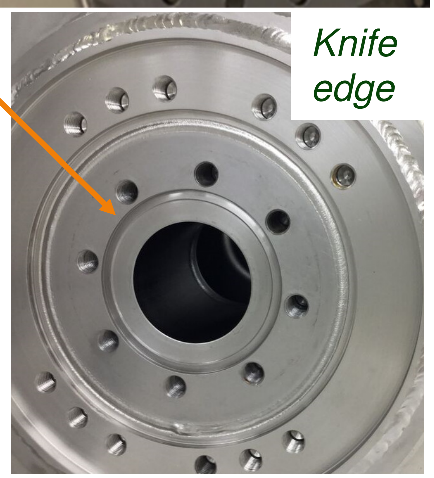
Data uploaded to e-traveler

Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, L. Popielarski, L. Dubbs, K. Elliott, I. Malloch, R. Oweiss, J. Popielarski, Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824, USA. Proceedings of IPAC2012, New Orleans, Louisiana, USA

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 is replaced or repaired
- Last time to *"see"* surfaces before coldmass installation



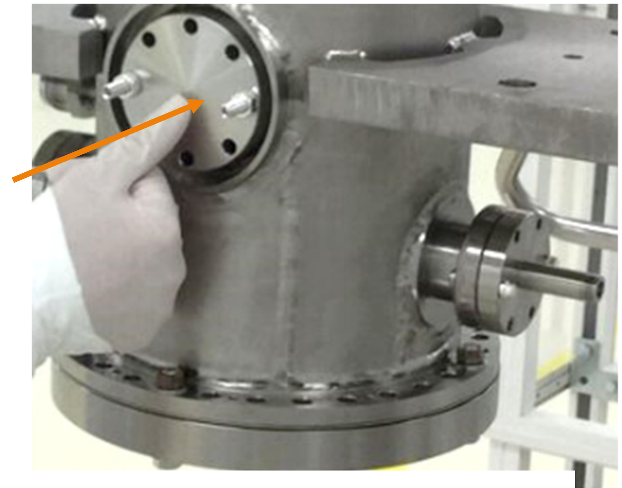


Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, L. Popielarski, et.al., Proceedings of IPAC2012, New Orleans, Louisiana, USA

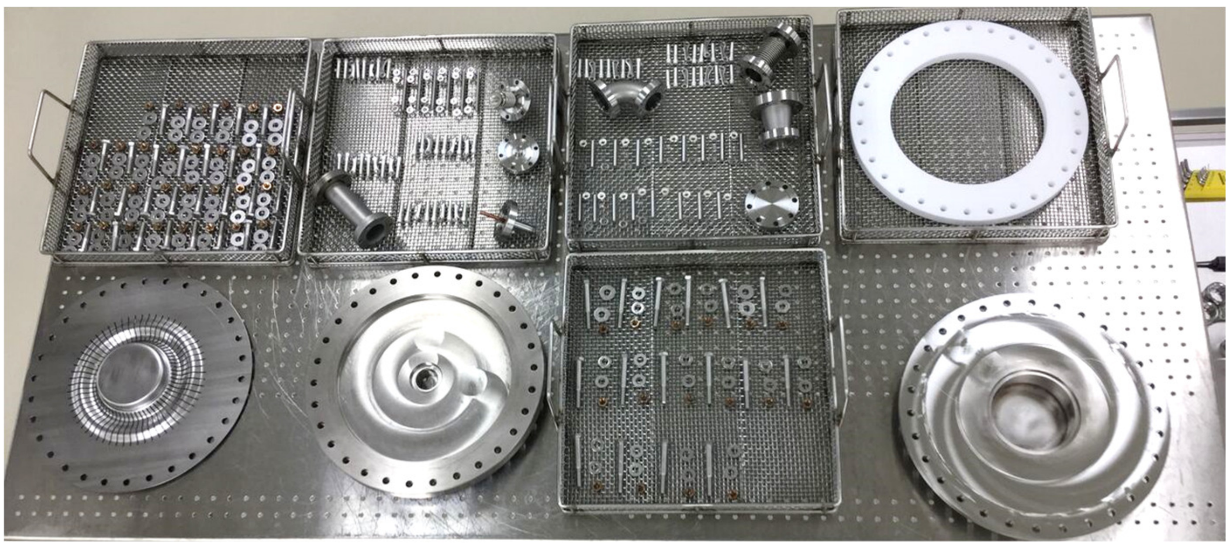
Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF2015, Whistler, BC, Canada.

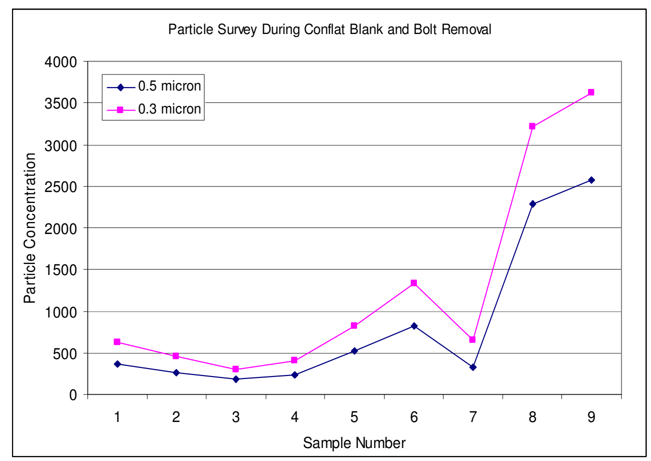
Clean Assembly

- 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, always lift by edges of flanges
- All tools cleaned the same as components



Particle seal QWR



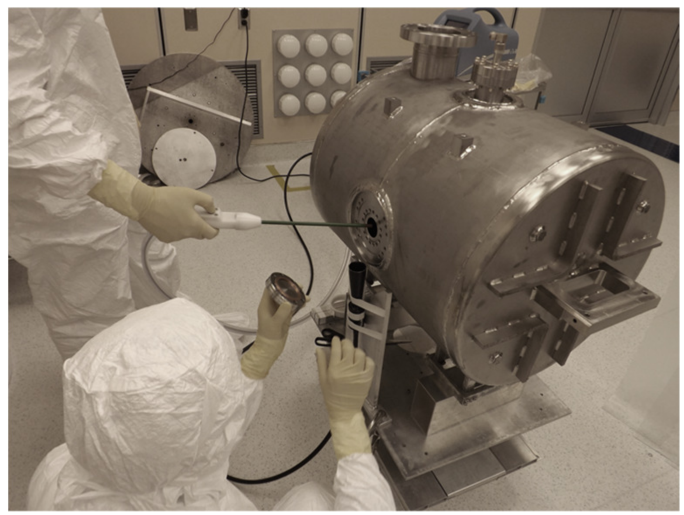


Cleanroom Techniques to Improve Surface Cleanliness and Repeatability for SRF Coldmass Production, L. Popielarski, et.al., Proceedings of IPAC2012, New Orleans, Louisiana, USA

First FRIB Beta=0.041 Production Coldmass Build, K. Elliott, S. Miller, B. Oja, J. Popielarski, L. Popielarski, D. Victory, M. Wilbur, T. Xu, Facility for Rare Isotope Beams, East Lansing, USA, M. Wiseman, Thomas Jefferson National Accelerator Facility, Newport News, USA. LINAC 2016.

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
- No other tasks or major movements are occurring near clean assembly area
    - » Ladders
    - » Rolling carts
    - » Other assembly



First FRIB Beta=0.041 Production Coldmass Build, K. Elliott, S. Miller, B. Oja, J. Popielarski, L. Popielarski, D. Victory, M. Wilbur, T. Xu, Facility for Rare Isotope Beams, East Lansing, USA M. Wiseman, Thomas Jefferson National Accelerator Facility, Newport News, USA. LINAC 2016.

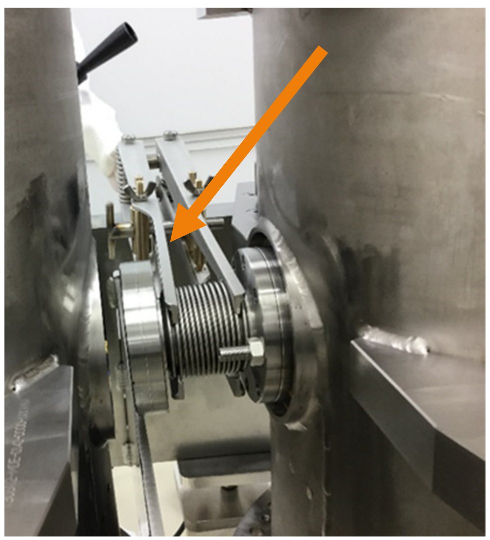
First FRIB Beta=0.53 Prototype Coldmass Build, D. Victory, K. Elliott, B. Oja, J. Popielarski, M. Wilbur, LINAC 2016.

Issue: The beam line assembly
Close proximity vertical flange arrangement

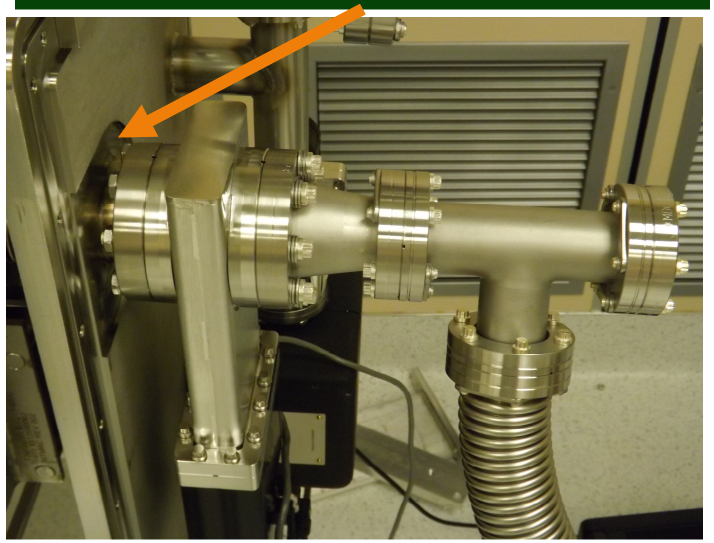
- All subcomponents leak checked prior to assembly
- Particulate counts prior to assembly ensure no contamination source present
- Special tools may be required: bellow compression, gasket holding, low profile wrenches for small gaps, and coupler installation support.
- Slight positive pressure in cavities 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

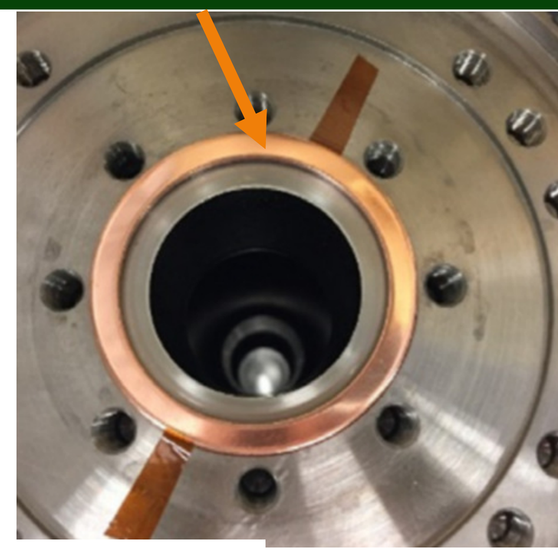
Bellows compression



Low-profile wrench used here



Gasket held in place



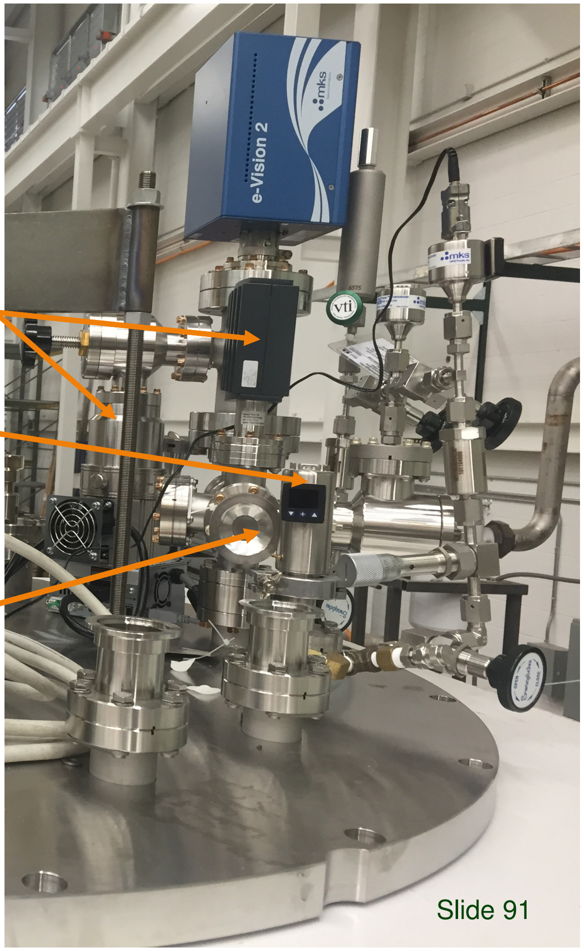
First FRIB Beta=0.041 Production Coldmass Build, K. Elliott, S. Miller, B. Oja, J. Popielarski, L. Popielarski, D. Victory, M. Wilbur, T. Xu, Facility for Rare Isotope Beams, East Lansing, USA M. Wiseman, Thomas Jefferson National Accelerator Facility, Newport News, USA. LINAC 2016.

First FRIB Beta=0.53 Prototype Coldmass Build, D. Victory, K. Elliott, B. Oja, J. Popielarski, M. Wilbur, LINAC 2016.

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, 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

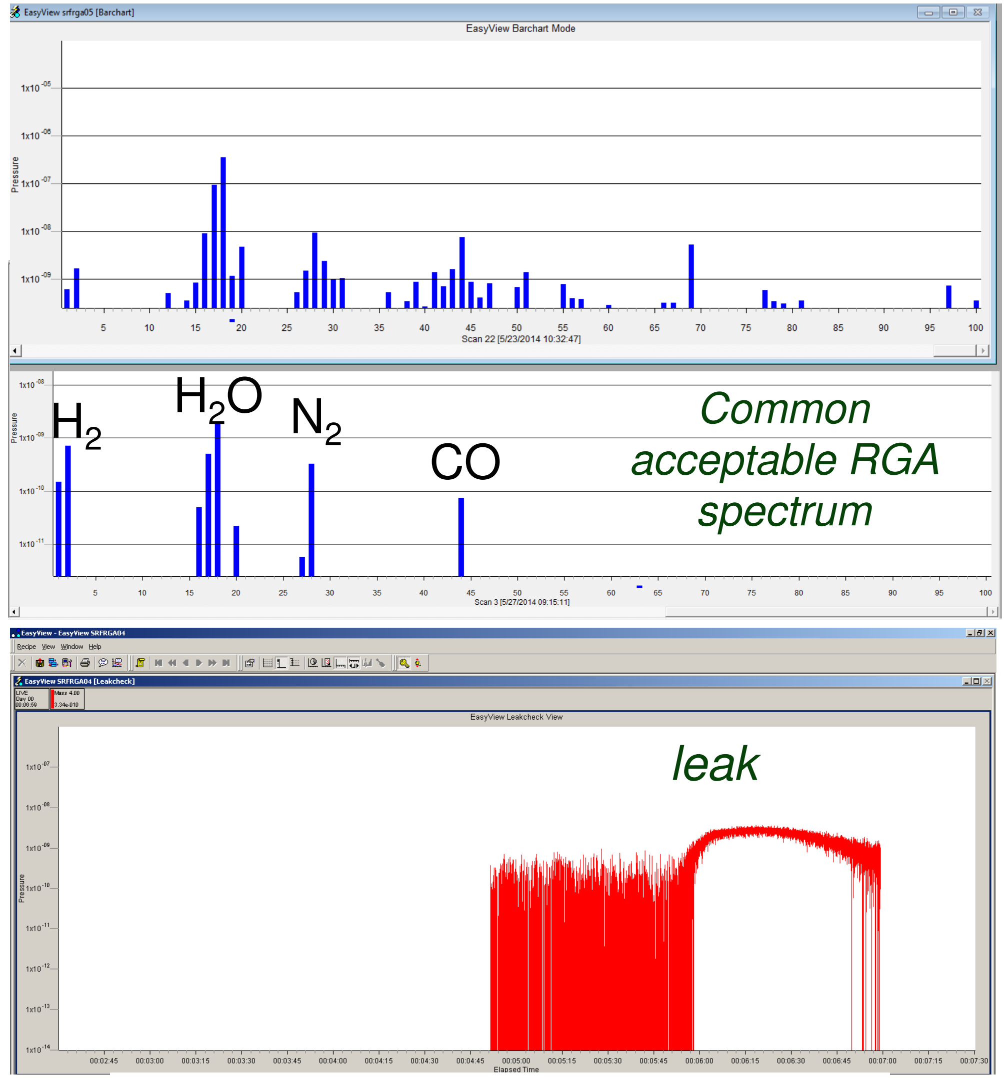


Particle Free Pump Down and Venting of UHV Vacuum Systems, K. Zapfe and J. Wojtkiewicz, Deutsches Elektronen Synchrotron DESY, D-22607 Hamburg. SRF 2009.

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. SRF 2009.

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

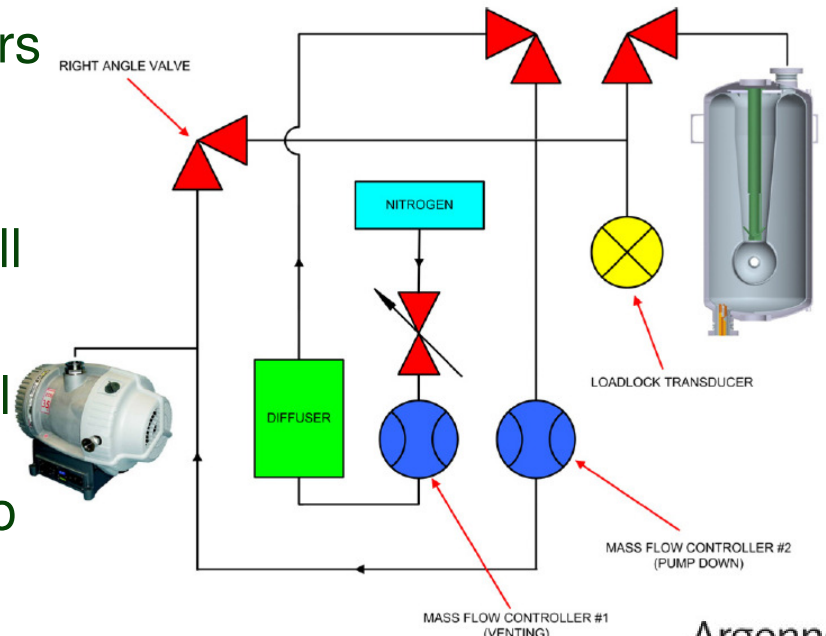


(http://www.mksinst.com/docs/R/SpectraBulletin208.pdf)

Cleanliness and Vacuum Acceptance Tests for the UHV Cavity String of the XFEL Linac, S. Berry, C. Boulch, C. Cloué, C. Madec, O. Napoly, T. Trublet, B.Visentin, CEA/DSM/irfu/SACM, Saclay, France, D. Henning, L. Lilje, M. Schmoekel, A. Matheisen, DESY, Hamburg, Germany. Proceedings of SRF 2015, Whistler, BC, Canada.

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 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. SRF 2009.

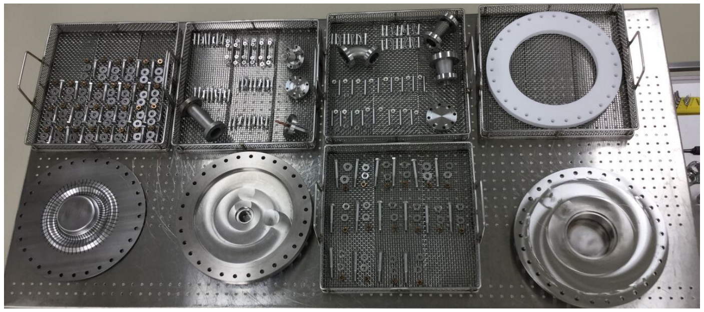
A Clean Pumping and Venting System for SRF Cavities and Cryomodules, S.M. Gerbick, M.P. Kelly, Argonne National Laboratory, Argonne, IL 60439, U.S.A. SRF 2009.

ACHIEVING THE FINAL GOAL…

CLEAN PARTS →

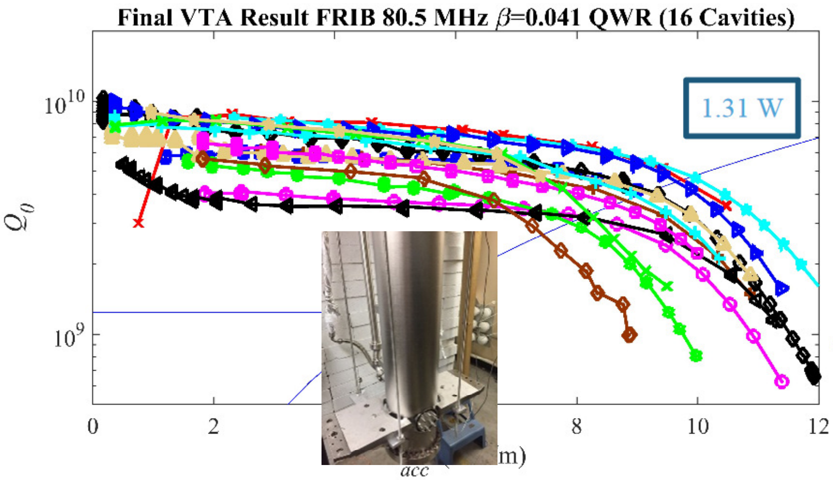
CLEAN CERTIFIED CAVITY →

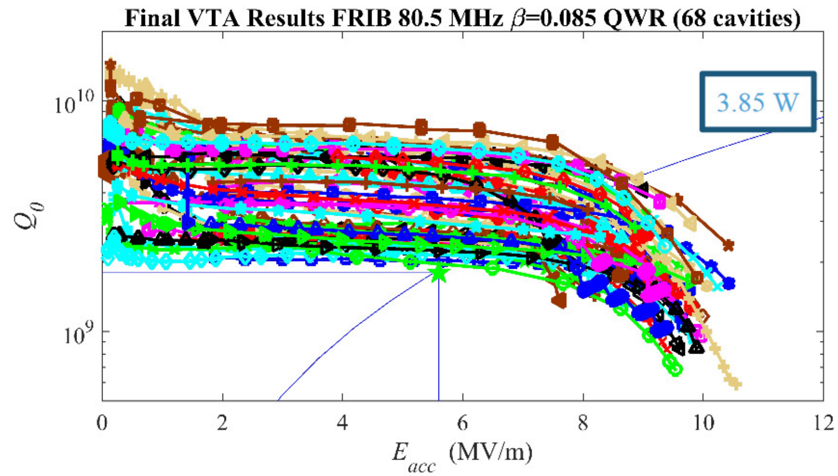
COLDMASS

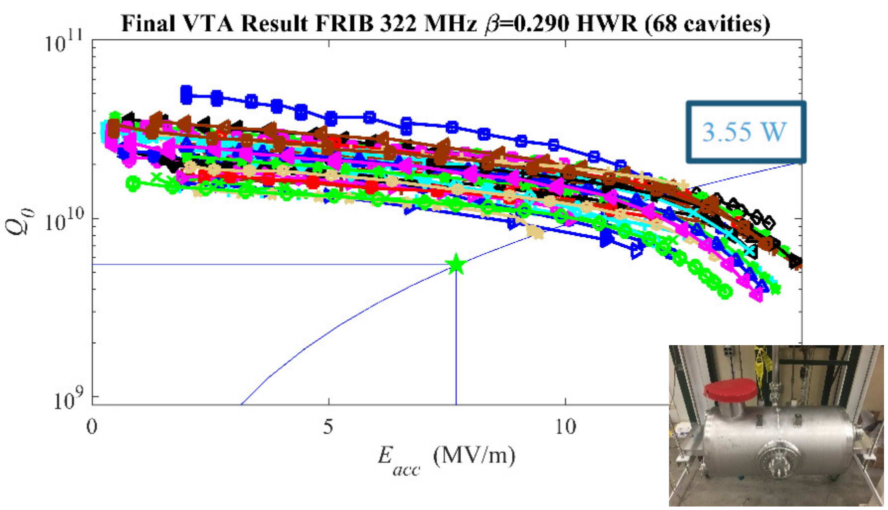


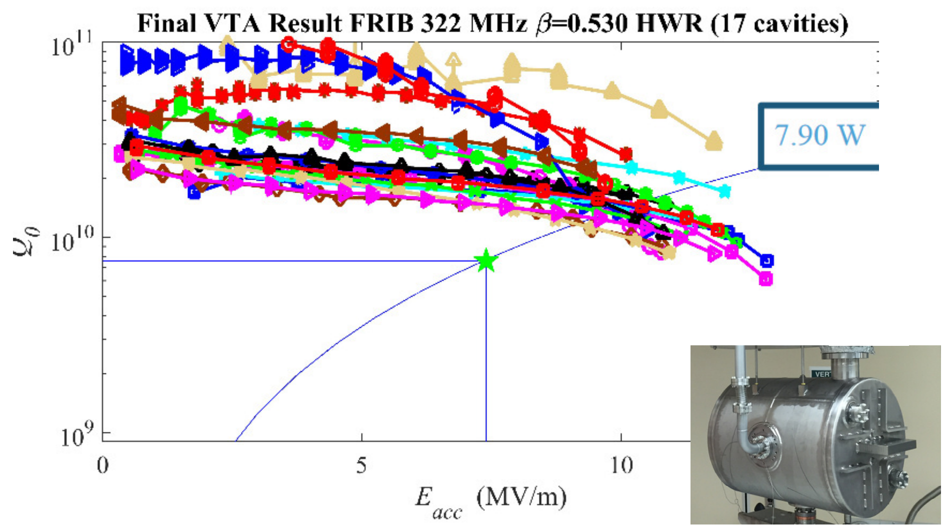
Low-Beta SRF Cavity Processing and Testing Facility for the Facility for Rare Isotope Beams at Michigan State University, L. Popielarski, et.al., Proceedings of SRF 2015

Cavities Exceeding Performance Specification…



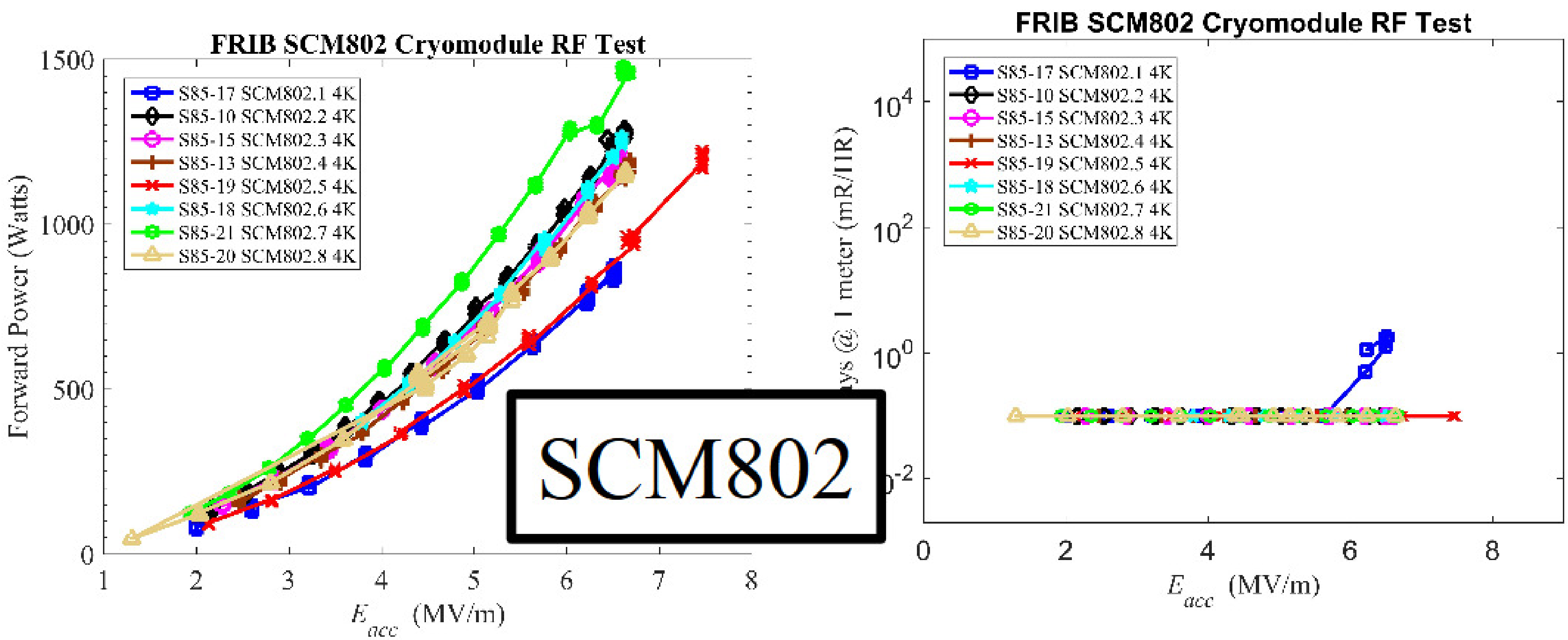






Performance Testing of FRIB Early Series Cryomodules, J.T. Popielarski, C. Compton, A. Ganshyn, W. Hartung, D. Luo, S.J. Miller, D.G. Morris, P.N. Ostroumov, L. Popielarski, K. Saito, S. Shanab, S. Stark, T. Xu, S. Zhao, Z. Zheng Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, USA. SRF 2017

And High Performing Cryomodules!



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

Performance Testing of FRIB Early Series Cryomodules, J.T. Popielarski, C. Compton, A. Ganshyn, W. Hartung, D. Luo, S.J. Miller, D.G. Morris, P.N. Ostroumov, L. Popielarski, K. Saito, S. Shanab, S. Stark, T. Xu, S. Zhao, Z. Zheng Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, USA. SRF 2017

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.
- Techniques were developed for FRIB processing and cleanroom assembly…
- **however much more can be learned during the production of future accelerators.**

Acknowledgement

- Thanks to Kenji Saito for providing me with content for this lecture, especially for electropolishing sections
- Thanks to the scientific community for the abundance of literature on processing and assembly of superconducting radio frequency cavities and associated hardware
- A complete list of citations for literature follow in the next slides

Thank you for your attention!

THE END

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[8] https://accelconf.web.cern.ch/accelconf/srf2007/TUTORIAL/PDF/Tutorial_3c.pdf https://www.classe.cornell.edu/public/SRF2005/talks/sunday/SuA01_talk_srf2005.pdf
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APPENDIX

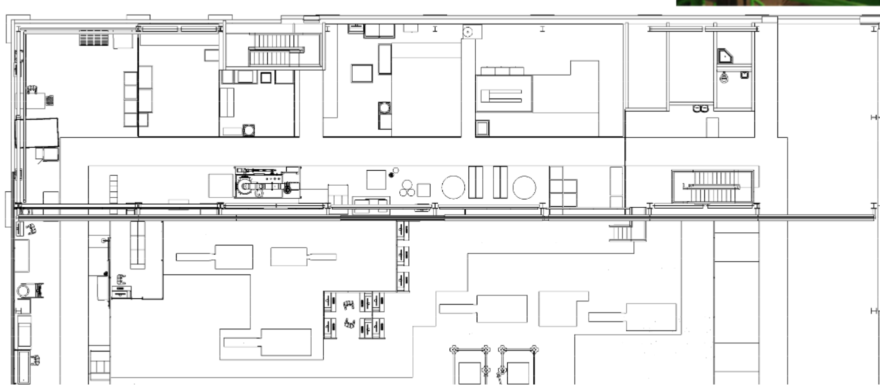
SRF Highbay at Michigan State University

- 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

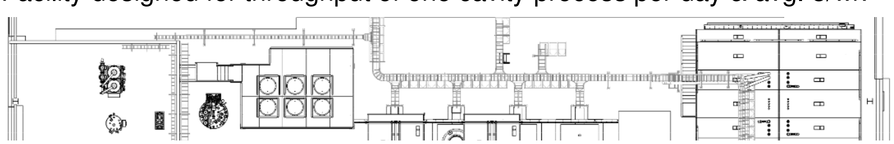
Production Inputs

Cold mass components →

Resources →

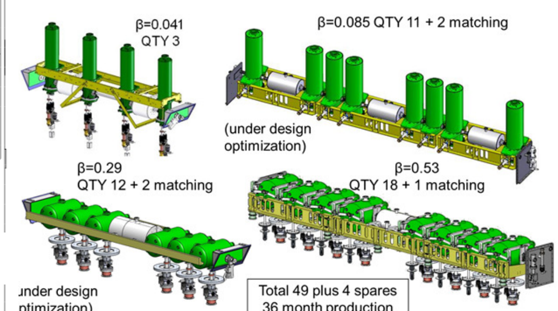


Facility designed for throughput of one cavity process per day & avg. 3/wk



Production Outputs

→ Cold masses



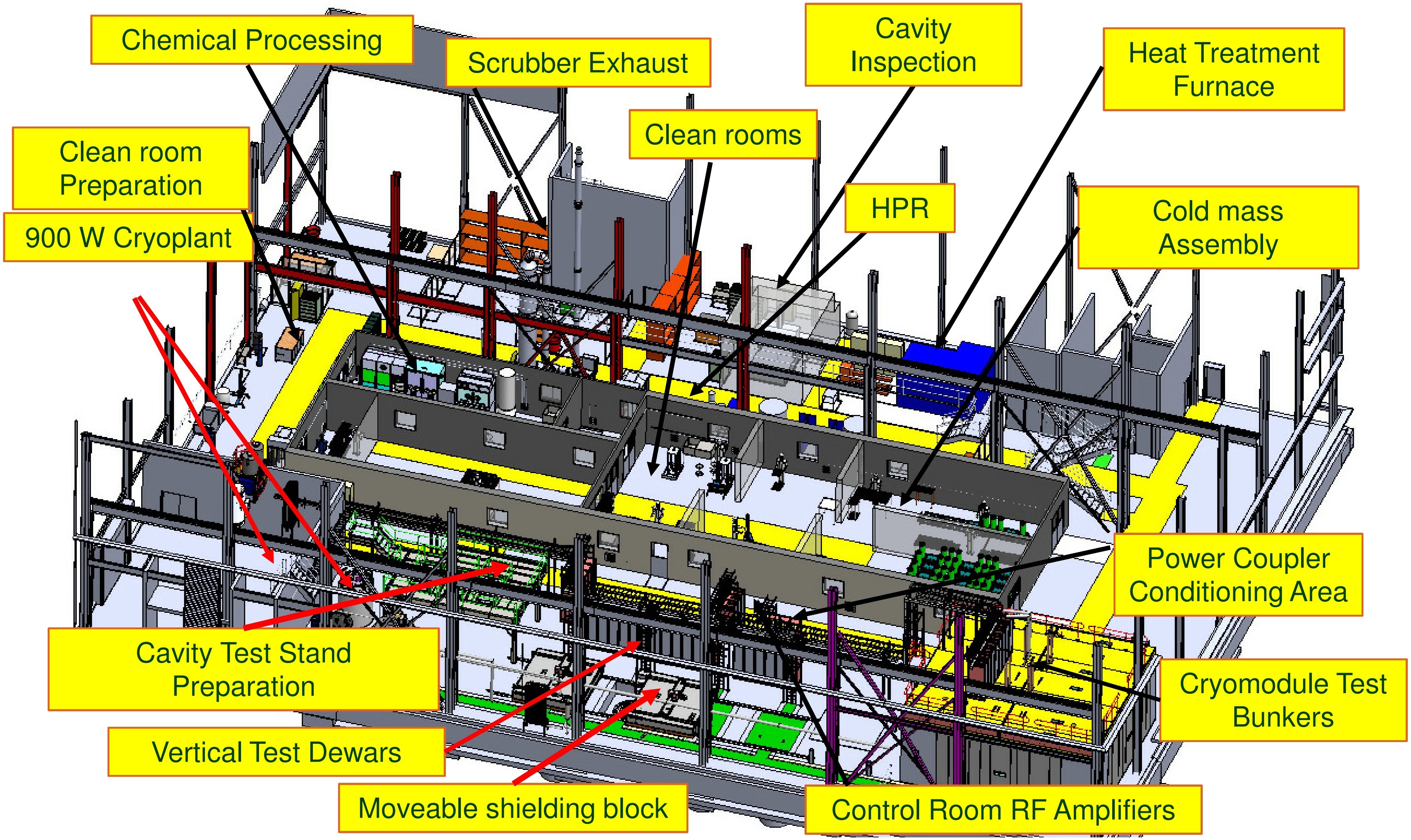
→ Certified Cryomodules

High bay floor ~ 23,400 ft²

Cavity QA & Inspect= 1,400 ft²
Furnace Area = 1,065 ft²
Cleanroom Prep= 1,700 ft²
Process Support= 1,050 ft²
Chemistry facility = 535 ft²
Processing CR= 2,500 ft²
Cold mass CR = 1,440 ft²
VTA/CMTF= 5,100 ft²
Cryogenic systems= 1,800 ft²

SRF HIGHBAY TECHNICAL INFRASTRUCTURE FOR FRIB PRODUCTION AT MICHIGAN STATE UNIVERSITY, L. Popielarski, Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, 48824, U.S.A. Proceedings of LINAC 2014,Geneva,Switzerland.

SRF Low Beta Processing & Cleanroom Facility for Production



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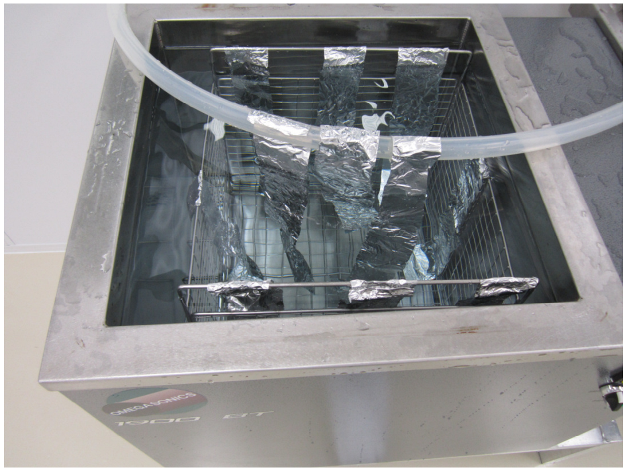
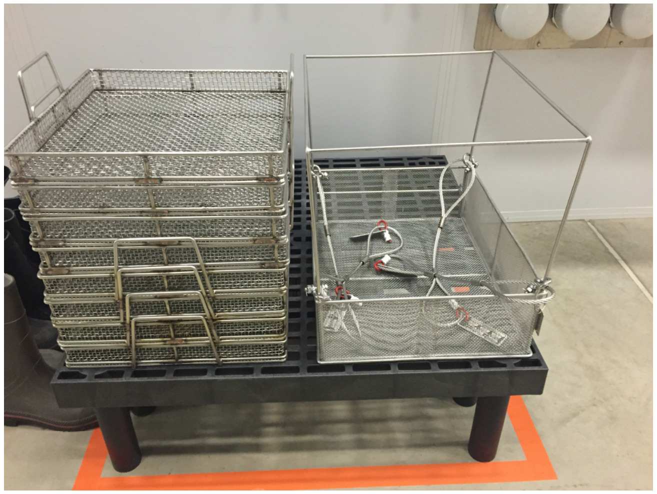
Ultra Sonic Cleaning Effective

- Cleaners commissioned using foils and part cleaning checks
- Smaller tanks 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

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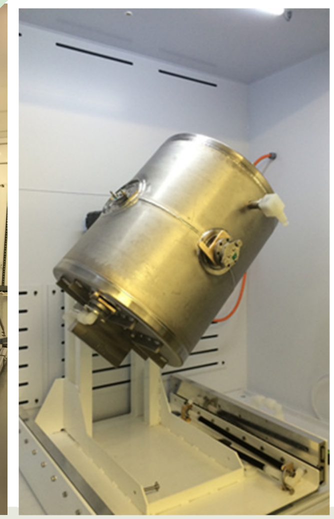
MSU BCP Facility Evolution 2000-2015



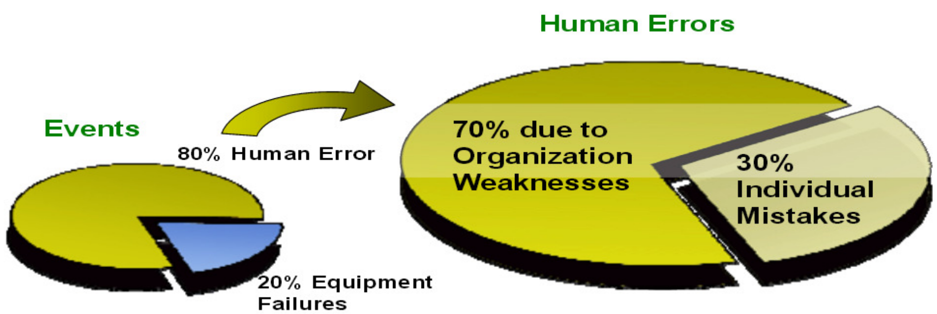
2000 R&D **2002** Small project **2014 Production**



Review Human Factors Early in the Design and Procedure Development

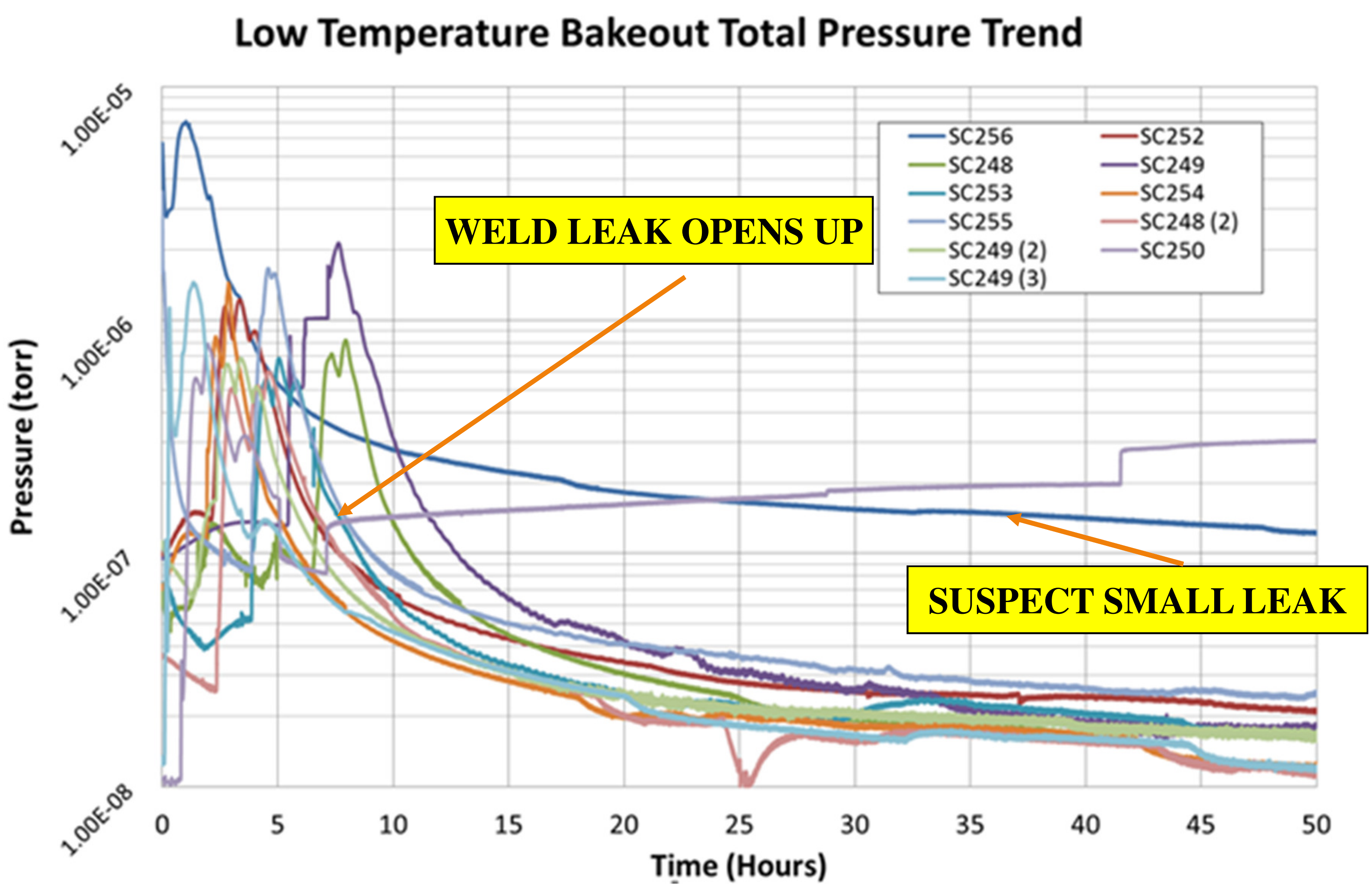
- End-users involved with equipment design and commissioning
- Review the procedure with your assistant before performing tasks, even if it has been done often, safety briefing
- Second independent verification on critical tasks, settings
- Reduce interruptions when performing critical tasks
- Post ‘do 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

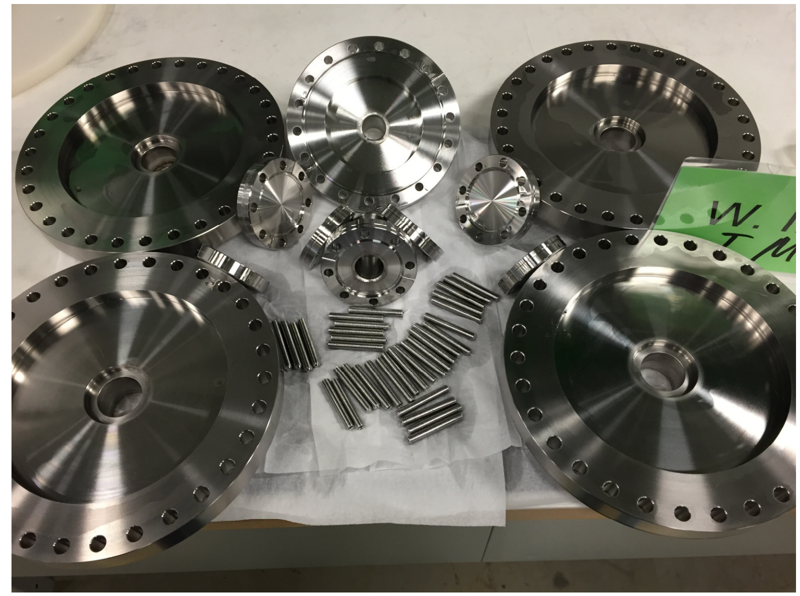
- Etch rate slower than BCP (0.5 μm/min versus 2 μm/min)
- Sulfur surface contamination, ultrasonic rinsing in H_2O_2, ethanol rinse or other detergent cleaning required
- Hydrogen production
  - Can cause Q-disease inside cavity, surround cathode with Teflon cloth
  - Hydrogen gas must be diluted outside of cavity with nitrogen to 4% (Lower Flammability Limit)
- Bake cavity at high temperatures 100 ° C to 800 ° C to remove hydrogen
- Possible problems with multipacting
- Ways to prolong life of acid solution
- Acid replacement within cavity structure
- 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

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



FIRST FRIB β=0.041 PRODUCTION COLDMASS BUILD, K. Elliott, S. Miller, B. Oja, J. Popielarski, L. Popielarski, D. Victory, M. Wilbur, T. Xu, Facility for Rare Isotope Beams, East Lansing, USA M. Wiseman, Thomas Jefferson National Accelerator Facility, Newport News, USA

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

https://indico.cern.ch/event/555785/contributions/2283555/attachments/1352708/2043324/Cleanroom_practises_v6.pdf

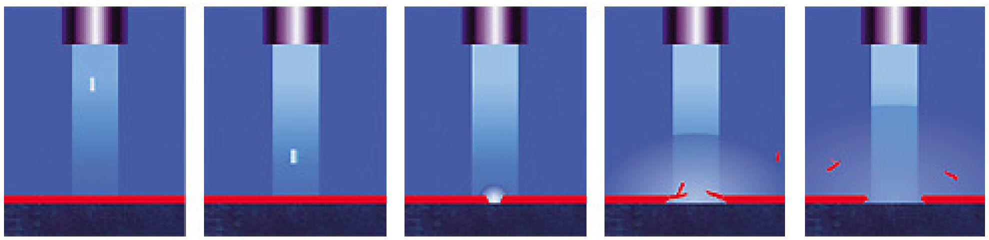
Cleanroom Equipment

- 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

W. Whyte (17 October 2001). Cleanroom Technology: Fundamentals of Design, Testing and Operation. John Wiley & Sons. ISBN 978-0-471-86842-2.

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.

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

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.

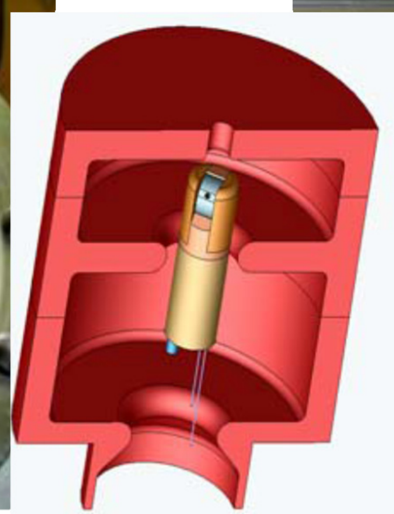
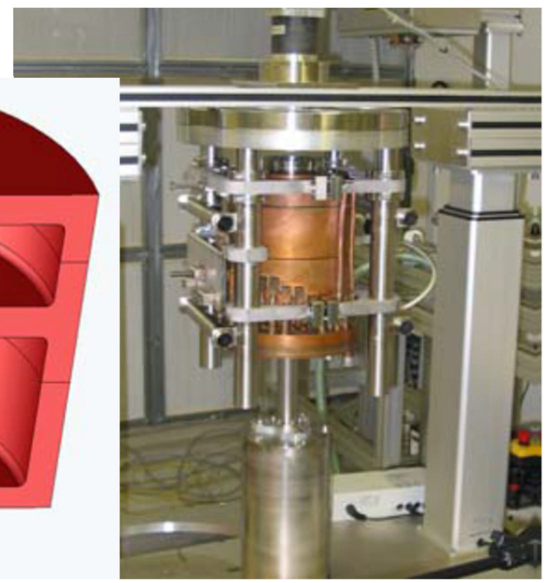
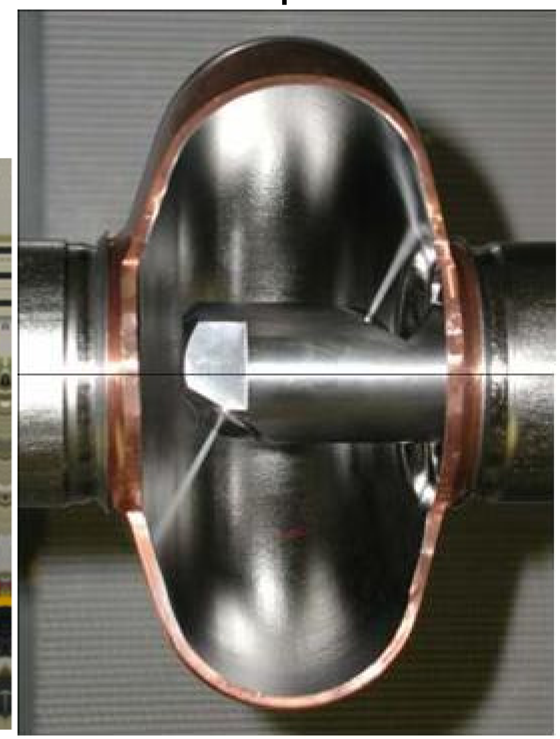
Carbon dioxide is a non-poisonous, liquefied gas, which is both inexpensive and easily stored at work sites.

FIRST EXPERIENCE WITH DRY-ICE CLEANING ON SRF CAVITIES D. Reschke, A. Brinkmann, DESY, D-22603 Hamburg, Germany D. Werner, Fraunhofer IPA, D-70569 Stuttgart, Germany G. Müller, FB C, University of Wuppertal, D-42097 Wuppertal, German
DRY-ICE CLEANING ON SRF-CAVITIES A.Brinkmann, J.Iversen, D.Reschke, J.Ziegler, DESY, D-22603 Hamburg, German

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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DRY-ICE CLEANING OF RF-STRUCTURES AT DESY, A. Brinkmann, J.Ziegler, Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany