

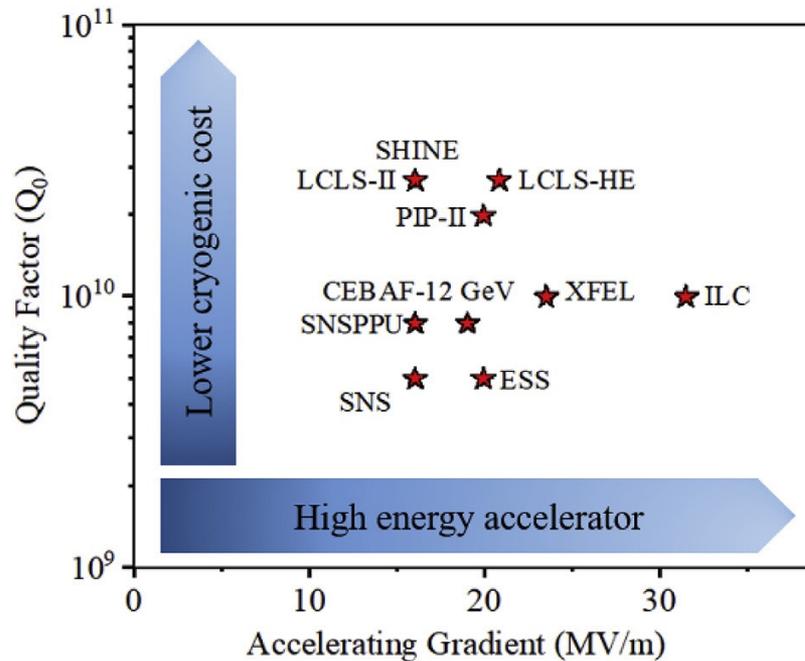
SRF'23 Lecture: SRF cavity fabrication and preparation



Rong-Li Geng
Jefferson Lab

Cavity fabrication and preparation

This lecture will present the fabrication and processing methods and techniques used for producing SRF cavities with high performance, including clean cavity assembly.



Niobium Material

- Niobium is the elemental superconductor with the highest critical temperature and the highest critical field
- Formability like OFHC copper
- Commercially available in different grades of purity (RRR > 250)
- Can be further purified by UHV heat treatment or solid state gettering

- High affinity to interstitial impurities such as C,H,O,N (in air at temperature < 150 °C)
- Require electron beam welding for joining
- Metallurgy not so easy
- Hydrogen can be easily absorbed, leading to Q-disease

Niobium as Compared with Other SC Materials

Material	Tc [K]	Hc/Hc1 [Oe]	Type	Fabrication
Pb	7.2	803/NA	I	Electroplating
Nb	9.25	1900/1700	II	Forming + EBW or film coating
Nb ₃ Sn	18.2	5350/300	II	Vapor diffusion or film coating or electroplating
MgB ₂	39	4290/300	II	Film coating

Niobium, bulk or coated, is the dominant material used in modern SRF systems.

High purity niobium sheets are supplied by industrial vendors in Americas, Europe and Asia



Niobium Production

- Niobium Ore in Araxa mine (open air pit) is Bariopyrochlor with 2.5% Nb_2O_5
- The ore is crushed and magnetite is magnetically separated from the pyrochlor.
- By chemical processes the ore is concentrated in Nb contents (50 –60 % of Nb_2O_5
- A mixture of Nb_2O_5 and aluminum powder is being reacted to reduce the oxide to Nb
- This Nb is the feedstock for the EBM processes

H.R.Salles Moura, "Melting and Purification of Nb", Proc.Intern.Sumposium Niobium 2001,Dec 2-5, 2001, Orlando Fl, p.147



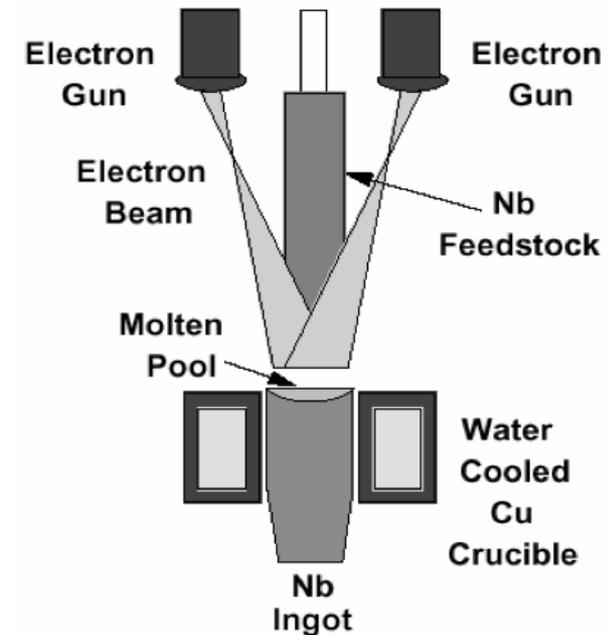
CBMM deposit in Araxa, Brazil



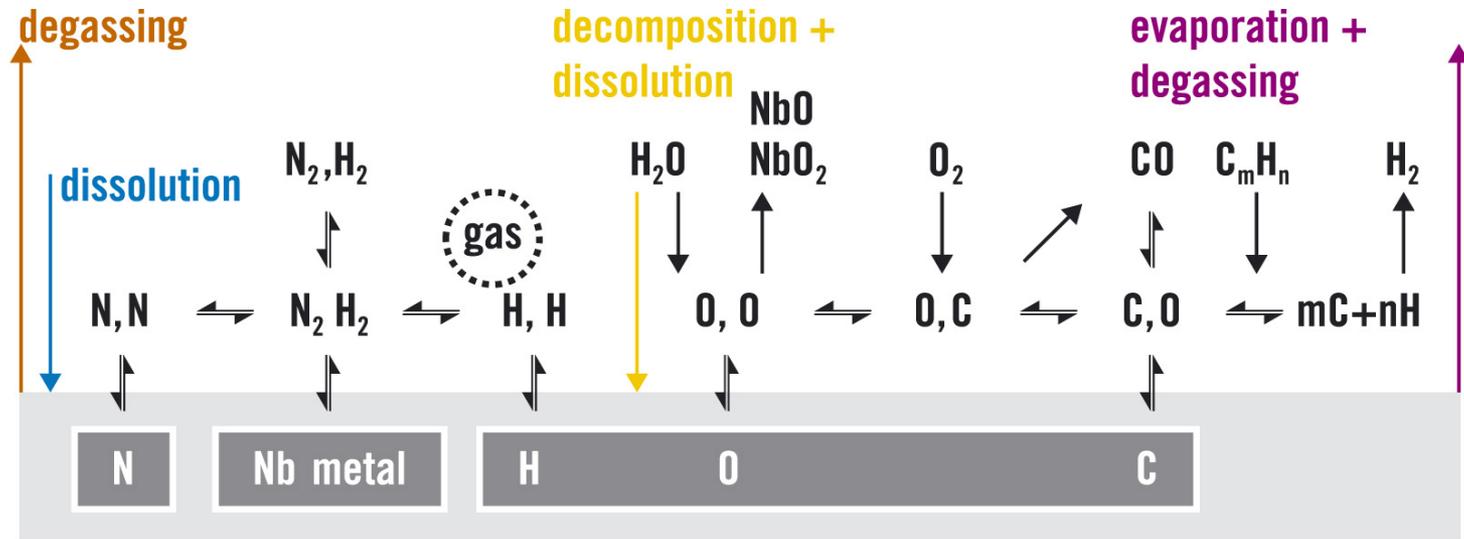
Electron beam melted ingots at CBMM

Electron Beam Melting

- High purity niobium is made by multiple electron beam melting steps under vacuum
 - This eliminates volatile impurities
- Several companies produce RRR niobium in large quantities
 - Cabot (USA), CBMM(Brazil), Tokyo Denkai (Japan), OTIC(China), Wah Chang (USA)
- RRR: Residual Resistivity Ratio
 - Resistivity at room temperature divided by the resistivity at 4.2K in the normal conducting state
 - Measure of purity
 - RRR scales roughly linearly with thermal conductivity at 4.2K, $\kappa(4.2K)[W/m-K] \sim RRR/4$



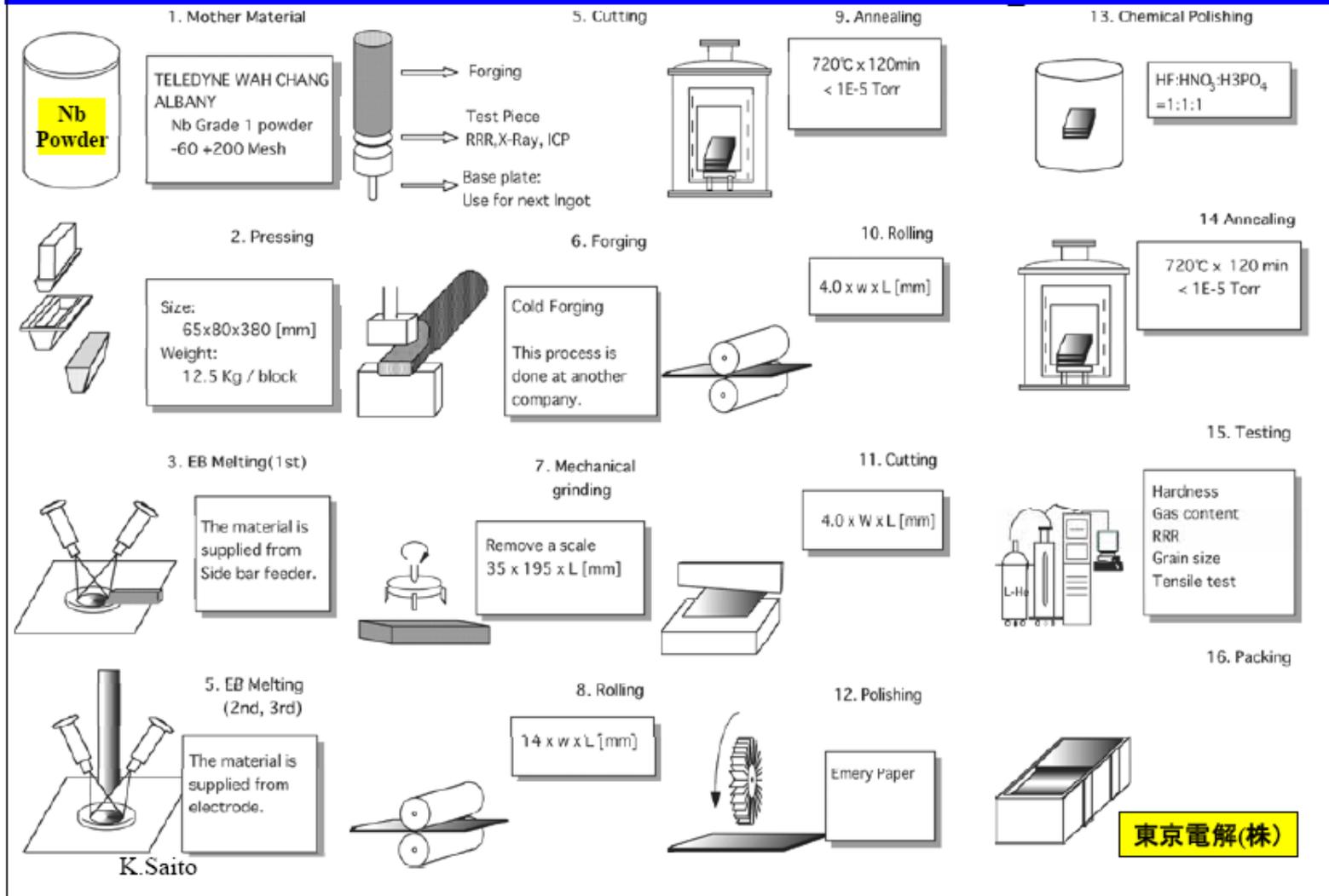
Nb EB Refining



Metal-gas and gas-gas reactions during Nb EB refining

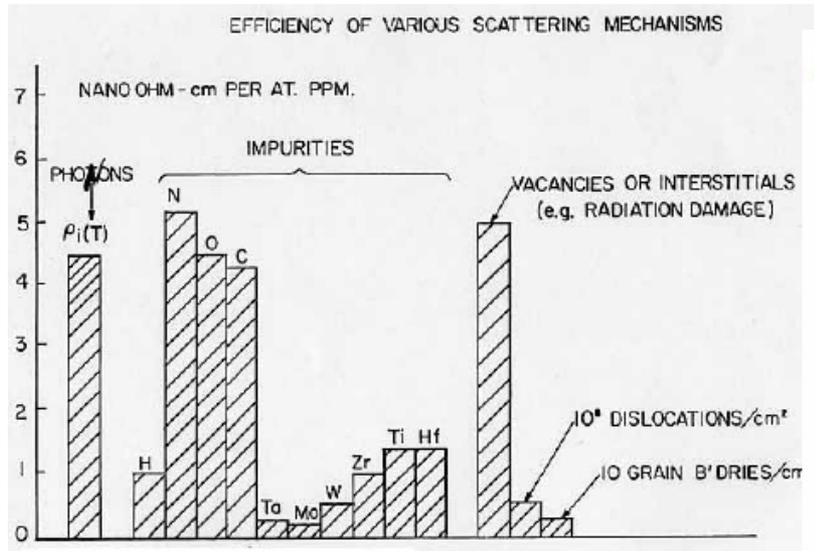
- The heating temperature is a compromise between the maximization of purification and minimization of the material losses by evaporation.
- RRR=300-500 are reachable currently.

Process flow of the industrial Nb production

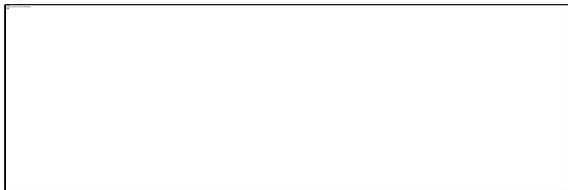


K. Saito, KEK

Influence of Impurities on RRR



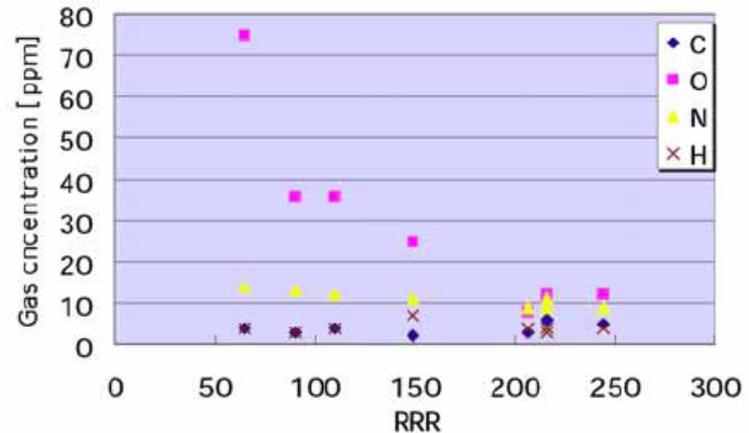
Contribution of different defects in the scattering mechanism:



$$r(300K) = 14.6 \text{ mW cm}$$

$$RRR_{ideal} \cong 35,000$$

Industrial Niobium Production – Intestinal impurities and RRR(Tokyo Denkai Co. Ltd.)

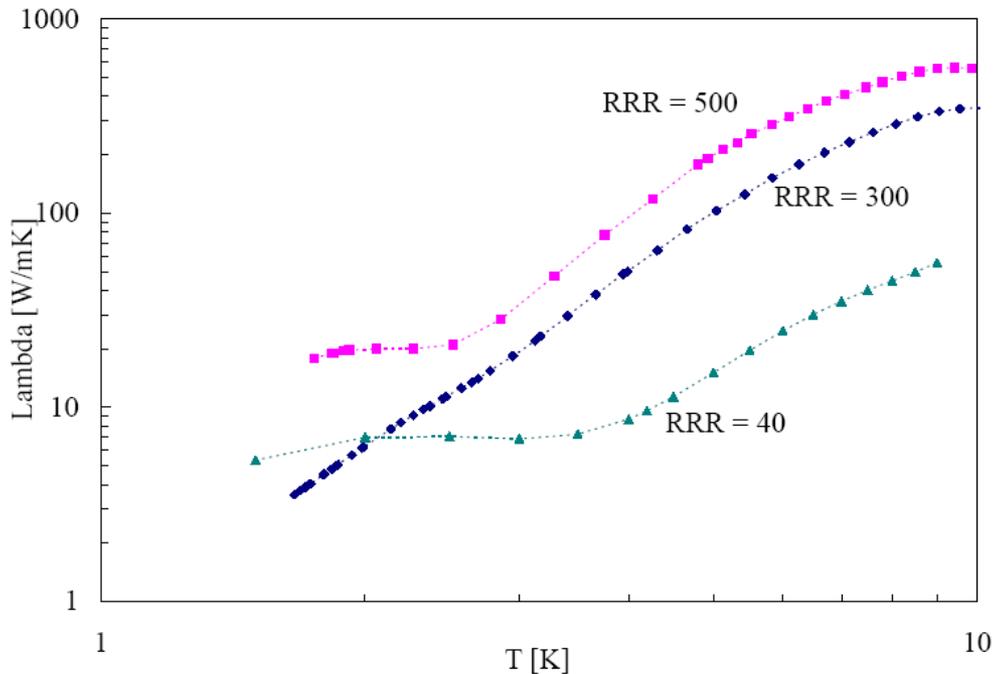


Relationship between RRR and nonmetallic impurities measured by Tokyo Denkai

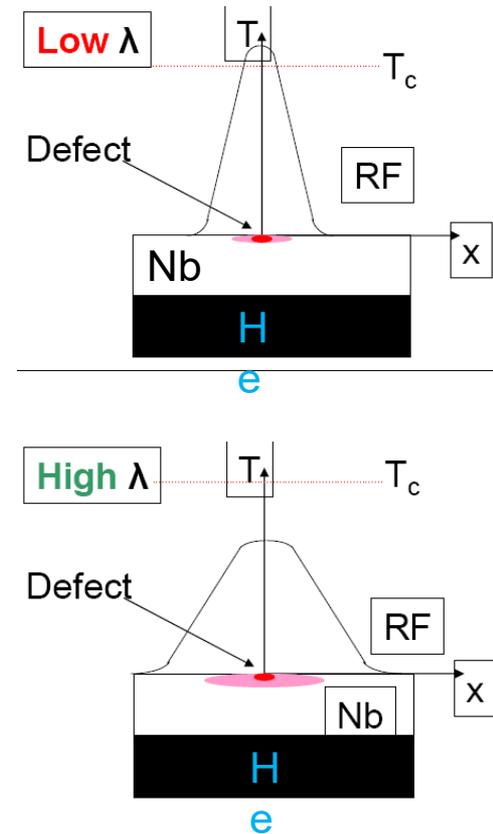
Expected RRR contribution for Nb for 1 wt ppm of impurities

Element	RRR	Element	RRR
H	2640	Zr	102 000–239 000
N	4230	Hf	200 000
C	4380	W	262 000–721 000
O	5580	Mo	717 000
Ti	53 700	Ta	1 140 000

Nb Thermal Conductivity

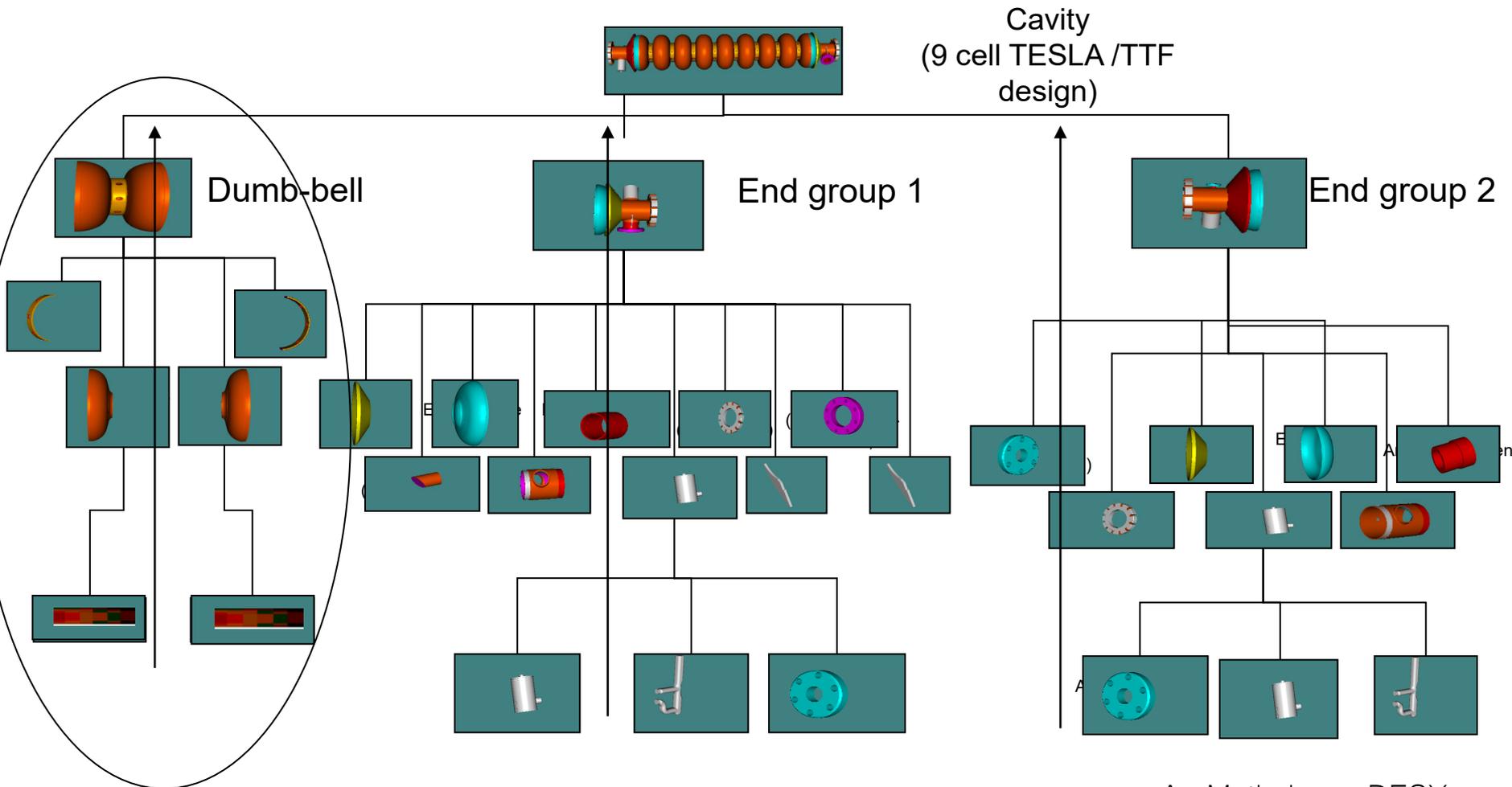


Rule of thumb:
 $\lambda(4.2\text{K}) = C \cdot \text{RRR}$
 $C \sim 0.25 \text{ W}/(\text{m K})$



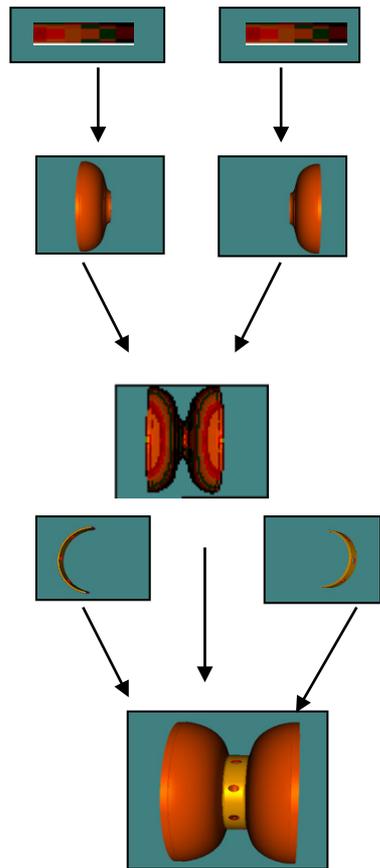
Main motivation to obtain high RRR (high purity) Nb is to improve the conduction of defect heat through the Nb wall containing defect temperature and avoiding destroy of SC state

Overview of Cavity Fabrication



A. Matheisen, DESY

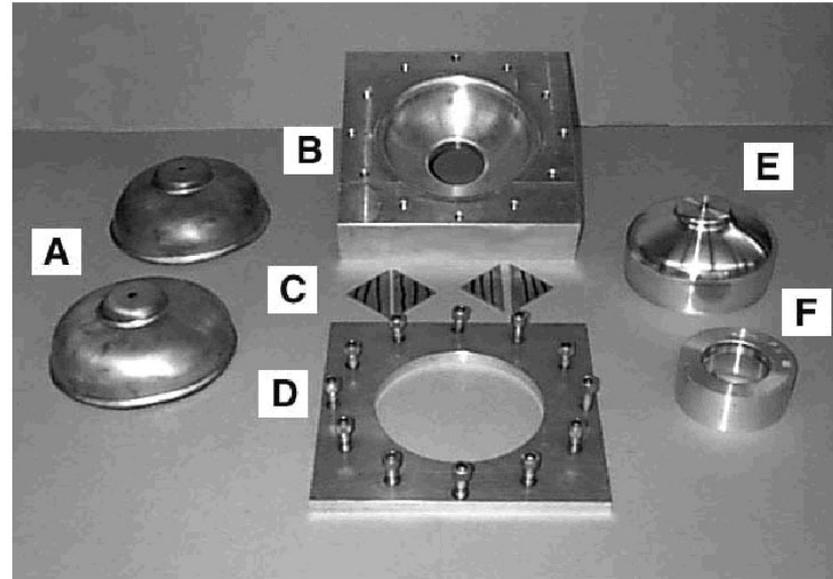
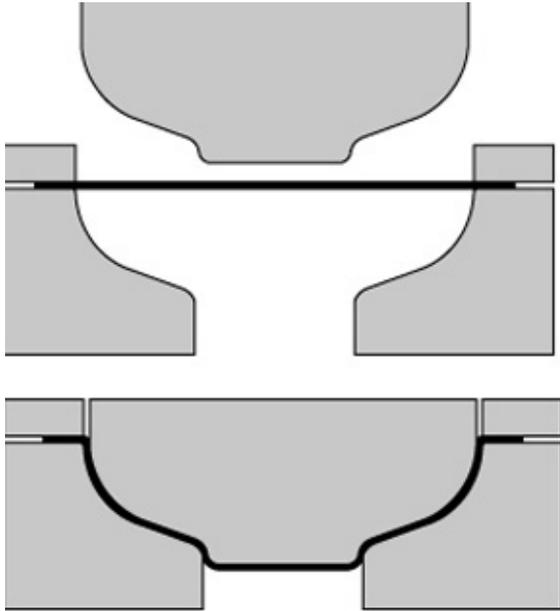
Example: Dumbbell Fabrication



Dumb- bell

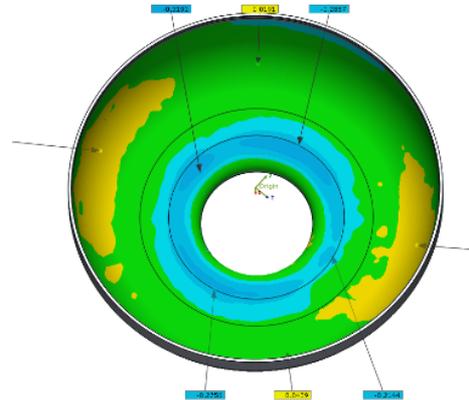
1. Mechanical measurement
2. Cleaning (by ultra sonic [us] cleaning +rinsing)
3. Trimming of iris region and reshaping of cups if needed
4. Cleaning
5. Rf measurement of cups
6. Buffered chemical polishing + Rinsing (for welding of Iris)
7. Welding of Iris
8. Welding of stiffening rings
9. Mechanical measurement of dumb-bells
10. Reshaping of dumb bell if needed
11. Cleaning
12. Rf measurement of dumb-bell
13. Trimming of dumb-bells (Equator regions)
14. Cleaning
15. Intermediate chemical etching (BCP /20- 40 μm)+ Rinsing
16. Visual Inspection of the inner surface of the dumb-bell
local grinding if needed + (second chemical treatment + inspection)

Deep Drawing



- Deep drawing with hydraulic press
 - 100 tons + 25 tons for iris coining for 10.25” discs (ILC)
 - 400 tons + 50 tons for iris coining for 15.5–16.5” discs (SNS)

Mechanical and RF QA/QC



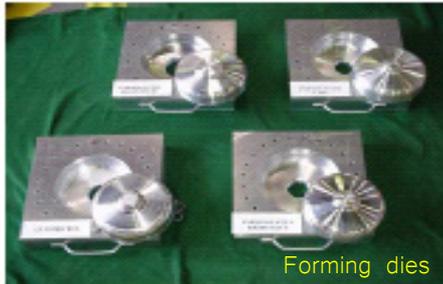
- Checking contour of half cell with a template of idea shape (photo on the left).
- Deviation from idea shape is revealed by gaps between half cell and template.
- This provides rapid feedback for initial forming trials.
- More sophisticated contour check is done using laser scanner (image on the right).
- The contour data can be used to compute the frequency deviation from the design value.
- Checking the RF frequency of the intermediate component.
- Two half cells are electron beam welded at iris, forming a dumbbell.
- Open ended half cells are covered with metal blanks, hold tightly against equator edge, forming a temporary cavity.
- The deviation of frequency from the design value is used to determine the cut length at equator weld prep.

Mechanical Grinding



- Mechanical grinding of visible local defects with aluminum oxide grinding discs
- Soaking parts in deionized water overnight reveals iron inclusion to guide grinding

Fabrication SNS/JLab

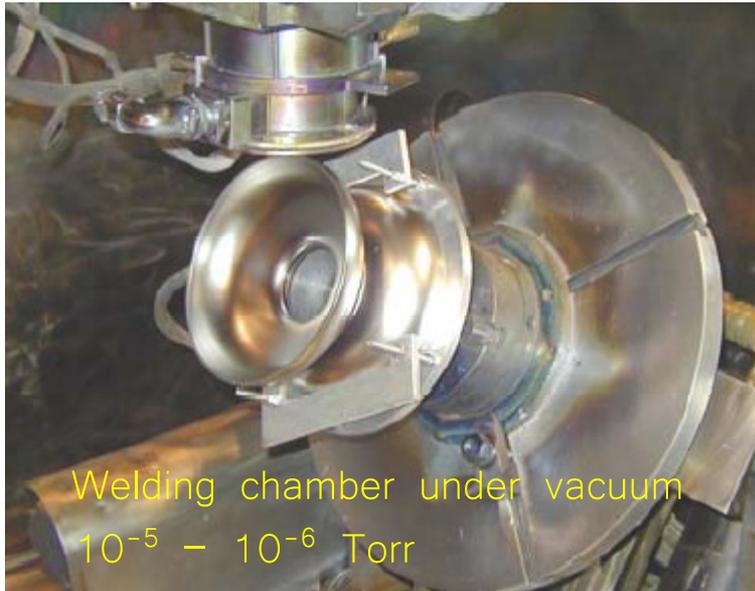


Video clip for half cell machining:
<https://www.youtube.com/watch?v=bMFYBWgGLPk>

courtesy of P. Kneisel

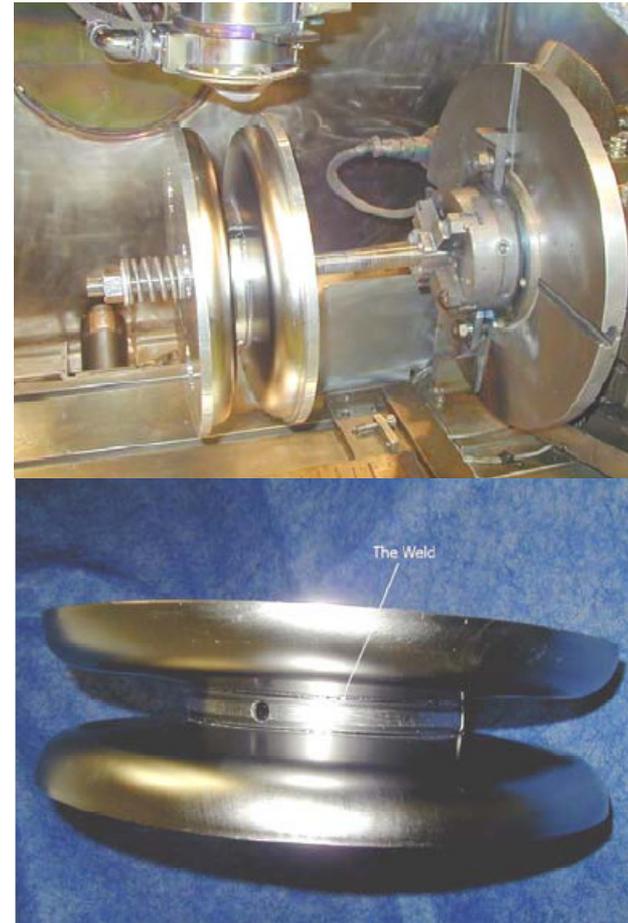
Electron Beam Welding (JLab)

Dumbbells



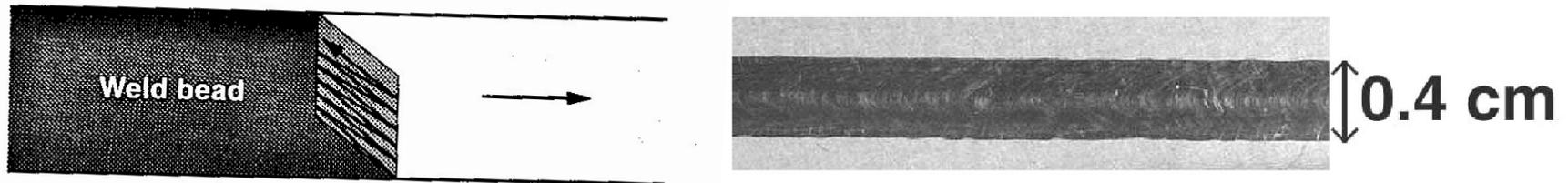
Tack- Welding: 4 tacks, focused beam
Voltage : 50 kV
Current: 15 mA
Rotational Speed : 20 inches/min
Distance of gun to work : 6 “
Final weld Current: 33 mA
Rotational speed: 18”/min
Focussing: elliptical pattern

Stiffening Rings

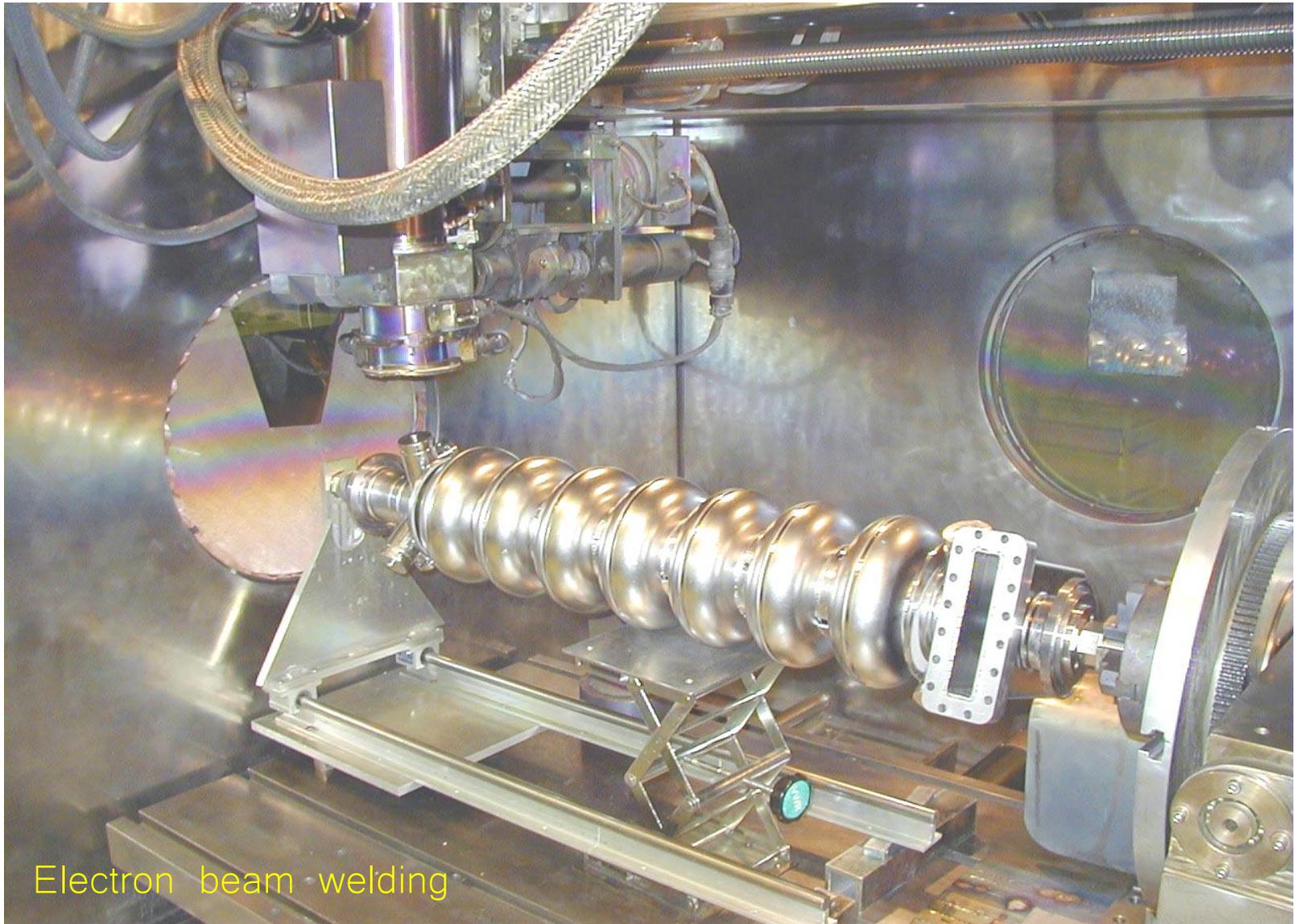


Rastered Beam in EBW

- High power density 10^7 W/cm² at the focus of electron beam
- Beam radius about 0.2 mm
- An electron transfers its kinetic energy to Nb material over a distance of about 0.01 mm largely in form of heat
- Beam is rastered during EBW.

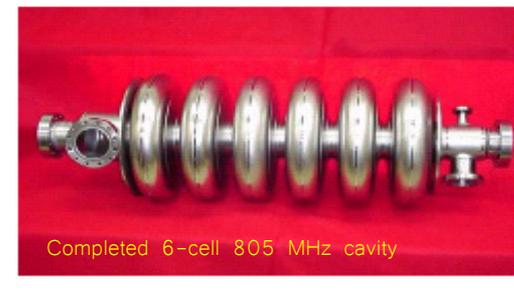
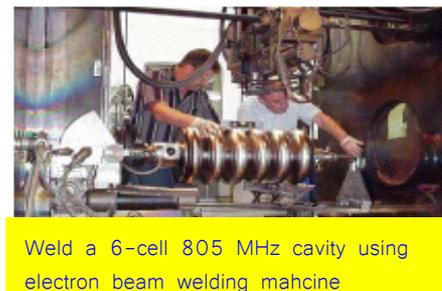
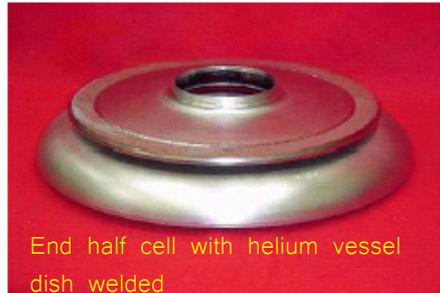


Rhombic raster pattern for the beam during EBW, produces a well-defined and reproducibly defocused beam



Electron beam welding

Fabrication SNS/JLab



Fabrication (JLab)

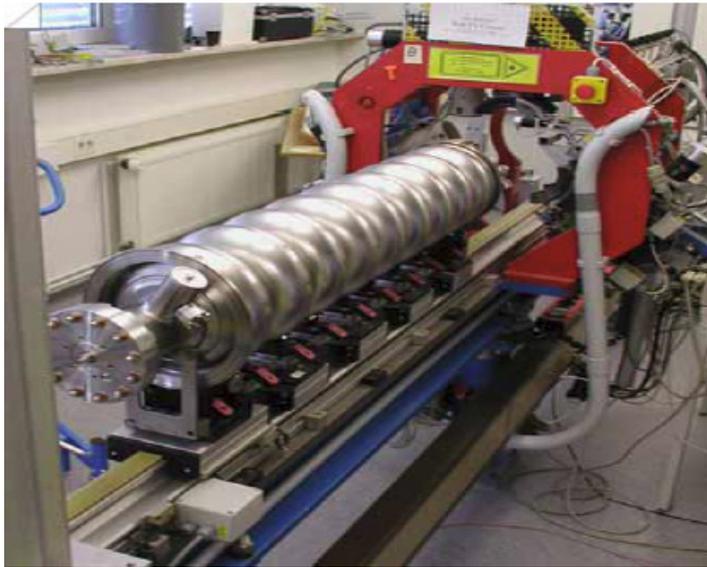
Endgroups



Cavity



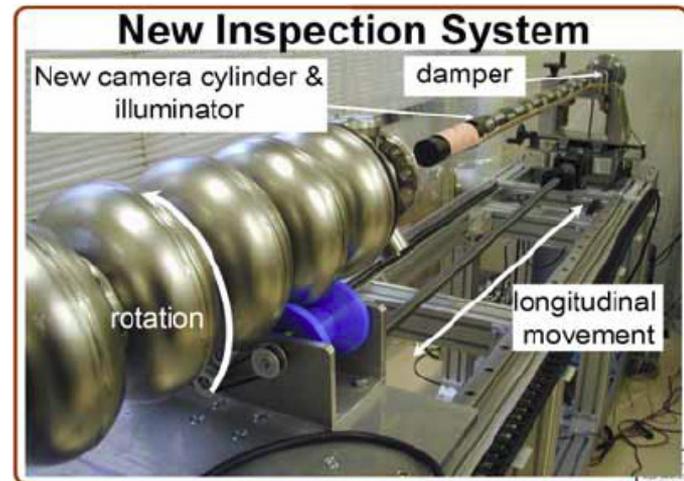
Cavity Inspection



Eccentricity measurement

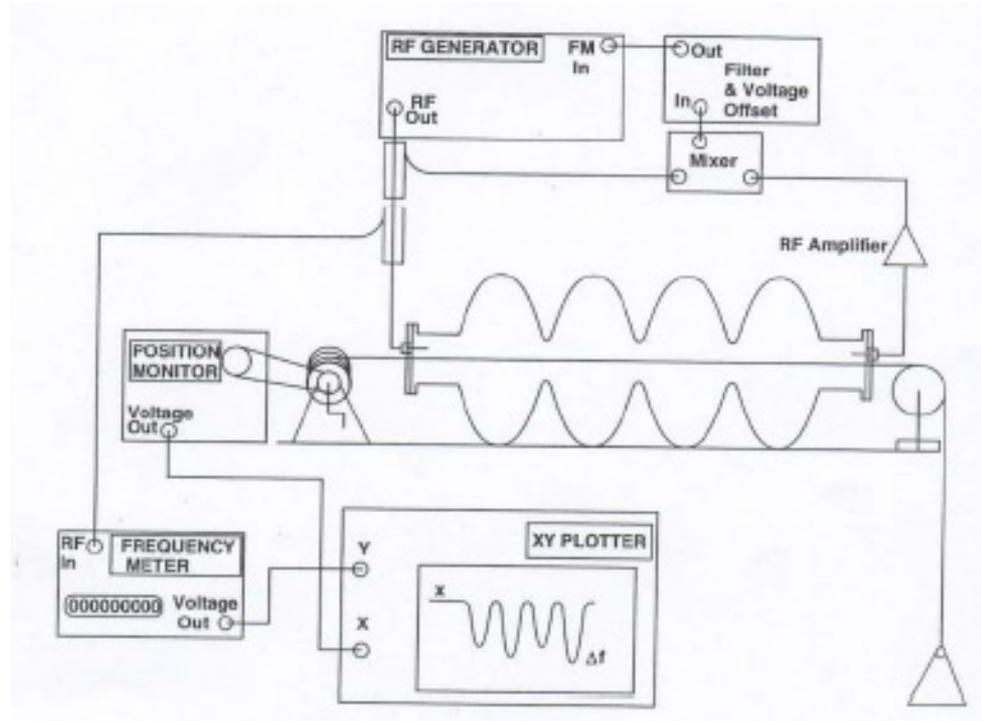
- Check of all mechanical tolerances
- Inspection of inner cavity surface
- Measure and adjust frequency and electrical field profile

Dimensional check



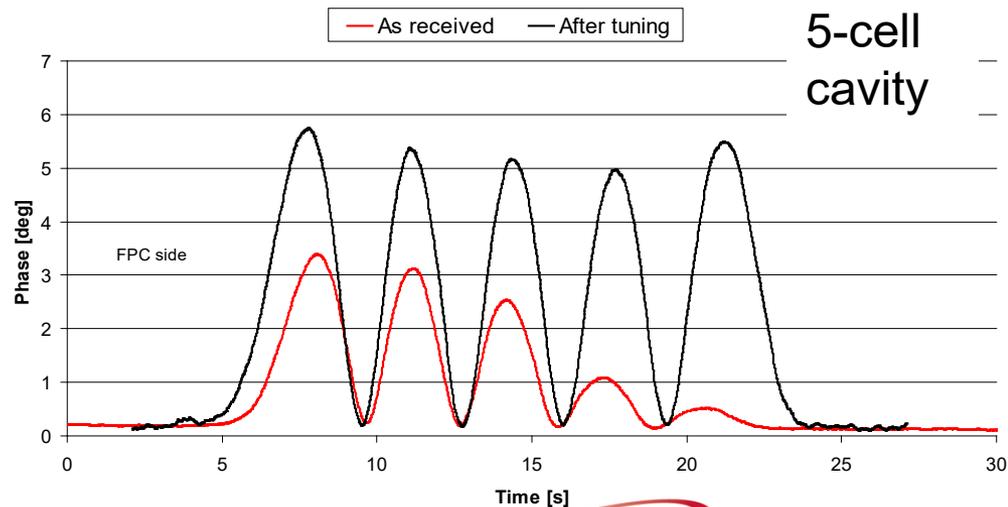
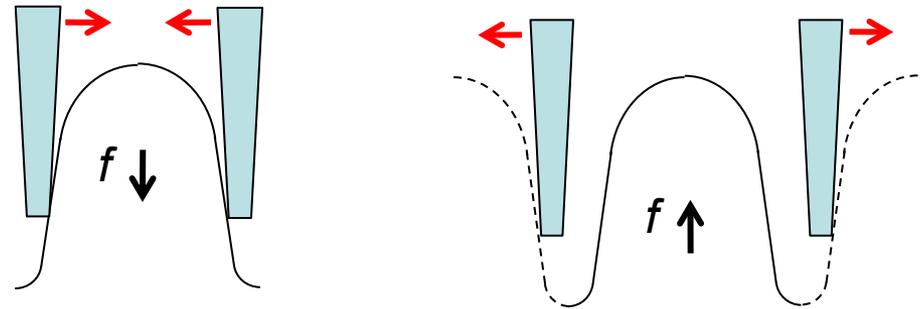
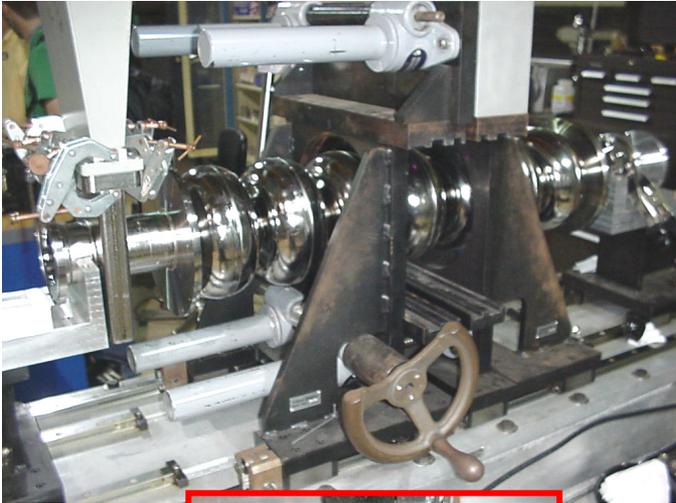
Cavity Tuning

Set-up for field profile measurements: a metallic needle is perturbing the RF fields while it is pulled through the cavity along its axis; the stored energy in each cell is recorded.



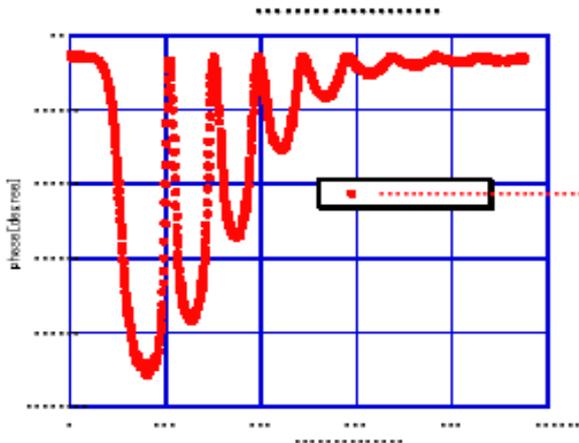
Cavity Tuning

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



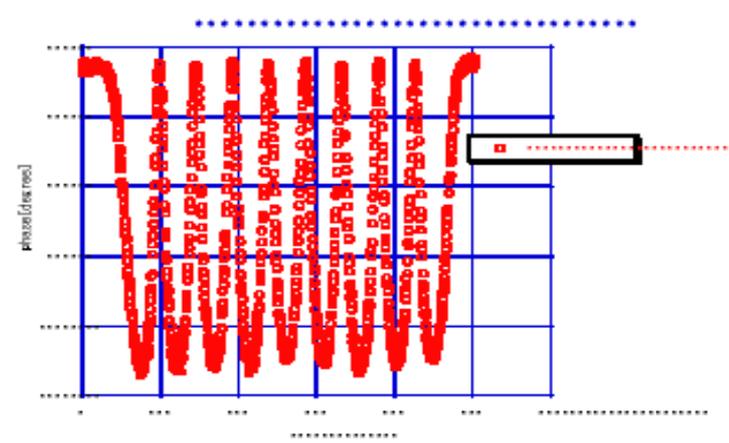
Field flatness after pre-tuning

• mode frequency 1298.774 MHz



Field flatness = 0.1 %
(as delivered to KEK)

• mode frequency 1298.547 MHz

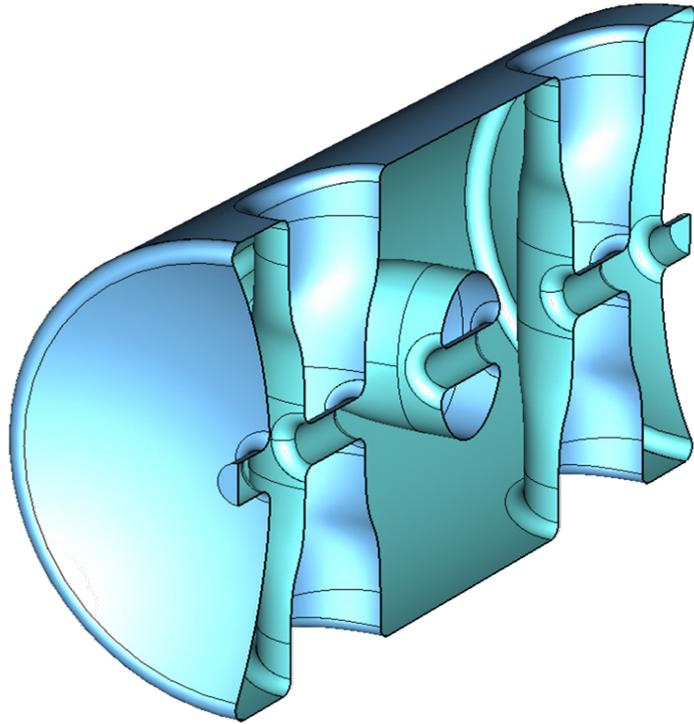


Field flatness = 98 %
(after pre-tuning)

Cavity	Field flatness (min/max)		Freq. target 1298.141 (MHz) @R.T.	
	as delivered	after pre-tuning	as delivered	after pre-tuning
1st	0.1%	98%	1298.774	1298.547
2nd	57.6%	Not yet	1301.447	Not yet
3rd	31.5%	Not yet	1301.577	Not yet
4th	51.5%	Not yet	1301.696	Not yet

Cell-to-cell coupling is as small as 1.6%, but no problem in pre-tuning.

Low- β Nb Cavity Fabrication



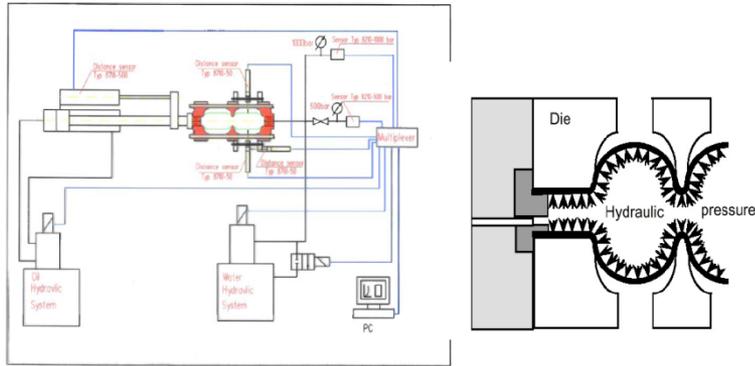
The ANL 345 MHz Triple-spoke cavities

Cavity components prior to EBW (AES)

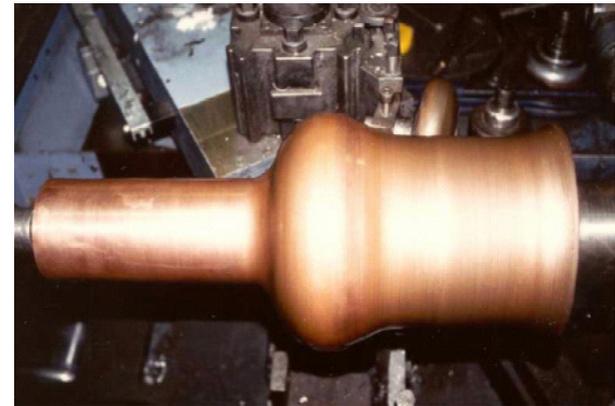
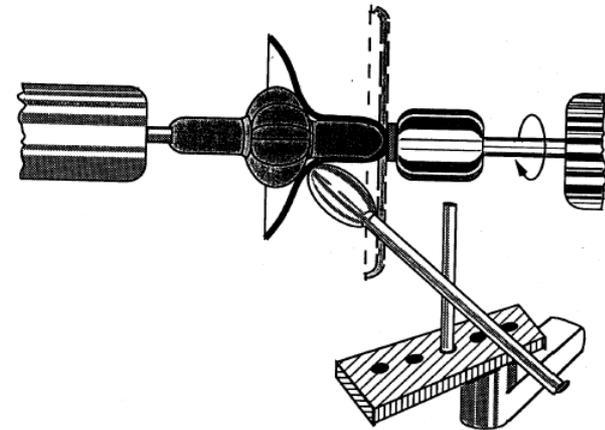


Alternative Fabrication – Seamless Cavity

Hydro forming (W.Singer,DESY)



Spinning (V.Palmieri,INFN Legnaro)



Alternative Fabrication

Nb Thin Film Coated Copper Cavity

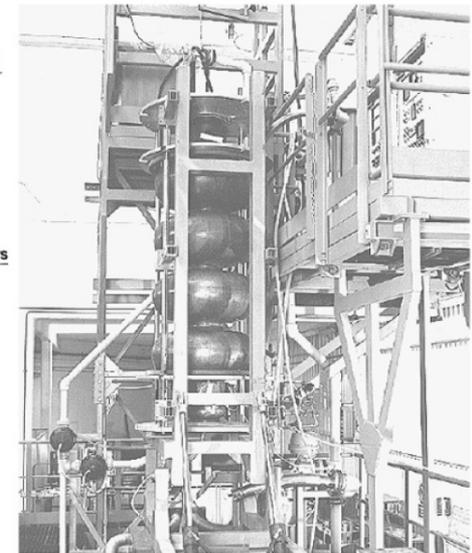
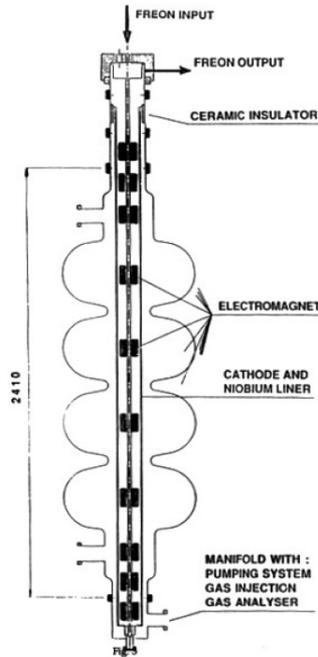
... .. CERN-JAPAN-RUSSIA Accelerator School, Nov. 6-14, 2002, Long Beach, California, USA
Niobium Film Coated Cavities

Developed in CERN for LEP II Superconducting RF cavities

$f=350$ MHz \longrightarrow big cavity (diameter :780mm) \longrightarrow Reduce Nb material for cost down



Copper half cell for LEP-II SC cavity



Fabrication of Low Beta Cavities

- Bulk Nb (by far the most used)

- highest performance, many manufacturers, any shape and f

- *performance* **** *cost* **



- Sputtered Nb on Cu (only on QWRs)

- high performance, lower cost than bulk Nb in large production, simple shapes

- *performance* *** *cost* ***



- Plated Pb on Cu (being abandoned)

- lower performance, lowest cost, affordable also in a small laboratory

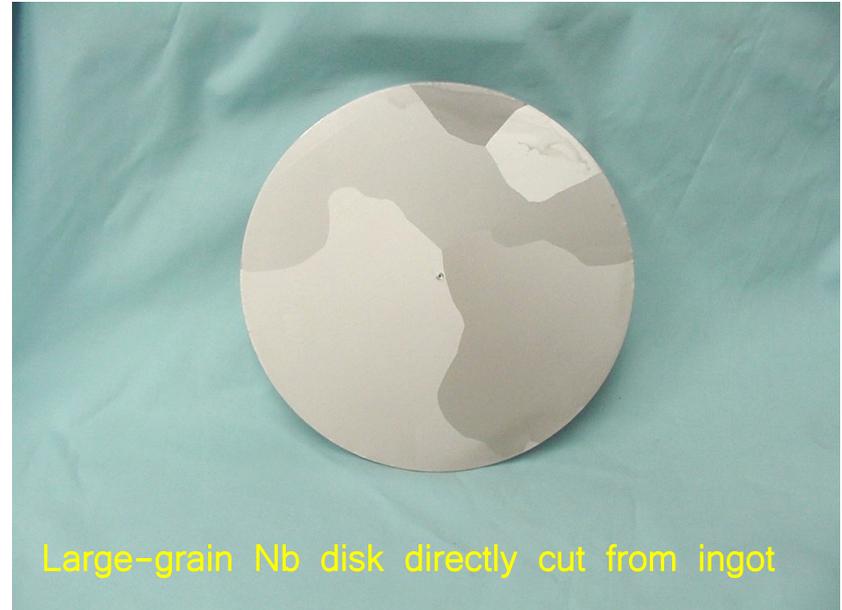
- *performance* ** *cost* ****



Courtesy A. Facco

Large-Grain Ingot Niobium

- Large-grain Nb disks from ingot directly
 - Wire EDM cut
 - Saw cut
 - Multi-wire slicing
- Eliminates many steps required for manufacturing fine-grain Nb sheets
- Some dimensional variation in formed components
- Standard electron beam welding
- Single crystal Nb?



Courtesy of K. Saito



Nb/Cu Clad Material

Advantages

- cost effective: allows saving a lot of Nb (ca. 4 mm cavity wall has only ca. 1 mm of Nb and 3 mm Cu). Especially significant for large projects like ILC
- bulk Nb microstructure and properties (the competing sputtering technique does not have such advantages)
- the treatment of the bulk Nb BCP, EP, annealing at 800°C, bake out at 150°C, HPR, HPP can be applied (excluding only post purification at 1400°C).
- high thermal conductivity of Cu helps for thermal stabilization
- stiffening against Lorentz - force detuning and microphonics can be easily done by increasing of the thickness of Cu layer.
- fabrication by seamless technique allows elimination of the critical for the performance welds especially on equator

W. Singer SRF 2005

Nb/Cu Clad Cavity Fabrication

- Nb/Cu laminated material is formed into a tube by:
 - Explosion bonding
 - Back extrusion
 - Hot bonding
- Nb/Cu clad cavity if formed from the tube by hydroforming
- Beam tubes/End groups are welded to the thin Nb layer by EBW. The Cu backing must be removed and cleaned at the weld joints



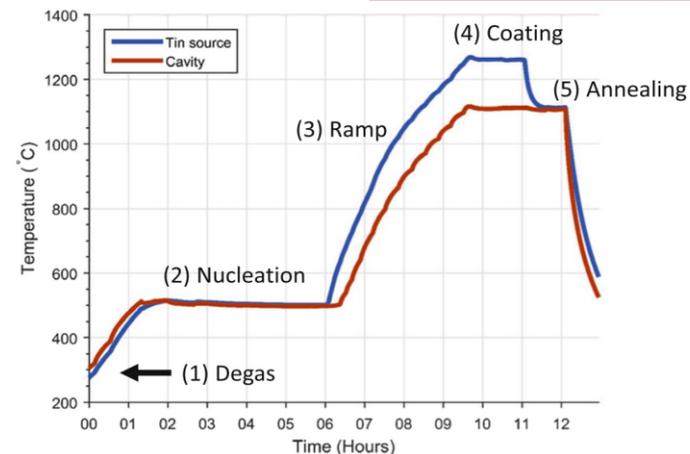
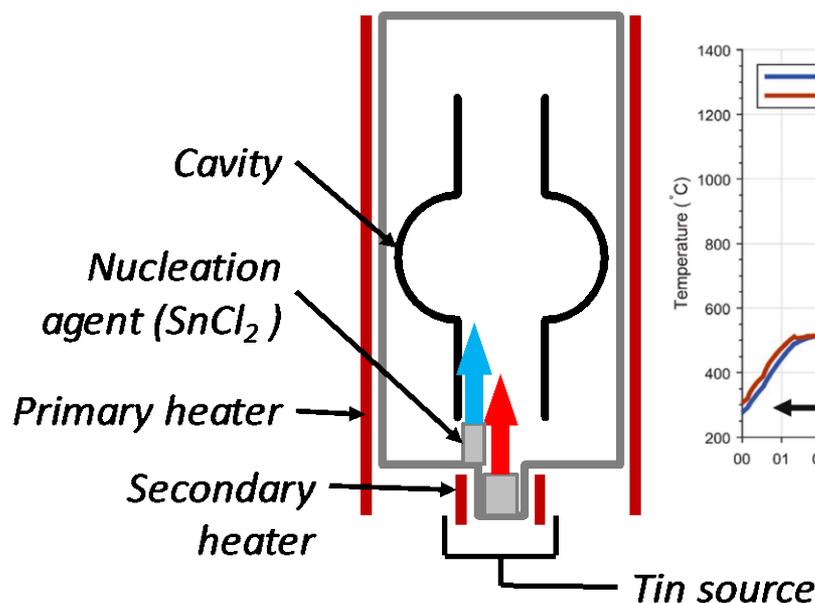
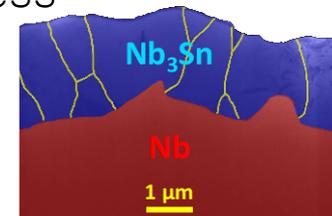
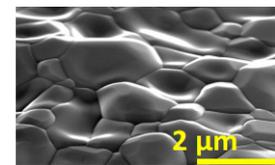
Four double cell NbCu clad cavities produced at DESY from KEK tubes (no cracks on the inside surface)



NbCu cavities hydroformed from explosively bonded tubes at DESY.

Nb₃Sn Cavity Fabrication

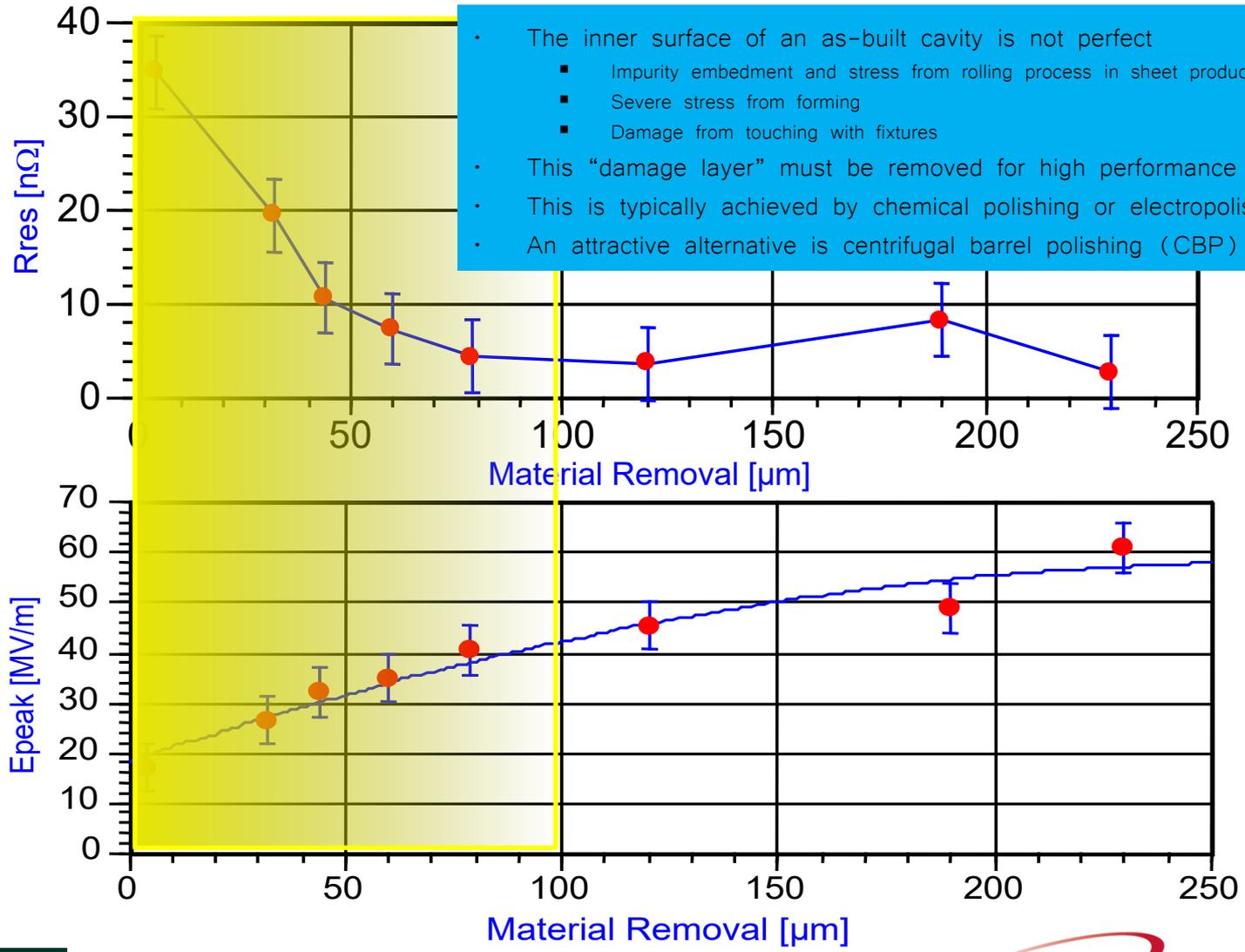
- Standard Nb cavity is fabricated
- Inner surface coated with Nb₃Sn via vapor diffusion process



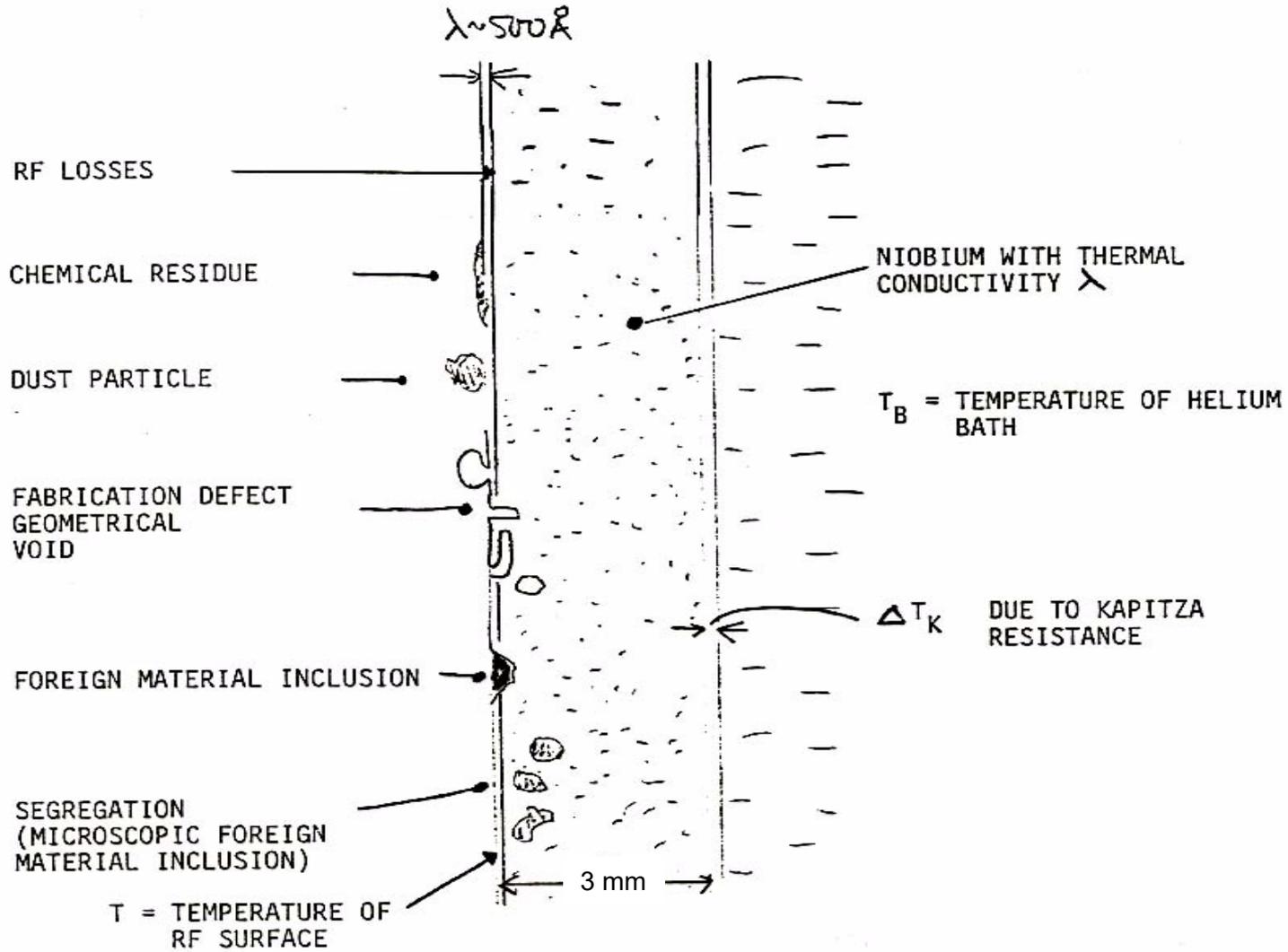
"Wuppertal" configuration, i.e., with secondary heater for the tin source
Optimized nucleation and temperature profile

S. Posen and M. Liepe, Phys. Rev. ST Accel. Beams 15, 112001 (2014).

Surface Processing: Remove “Damaged Layer”



Cavity RF Surface



Required Procedures for Qualifying SRF Cavities

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



Degreasing with Ultrasonic Agitation

Why is degreasing needed

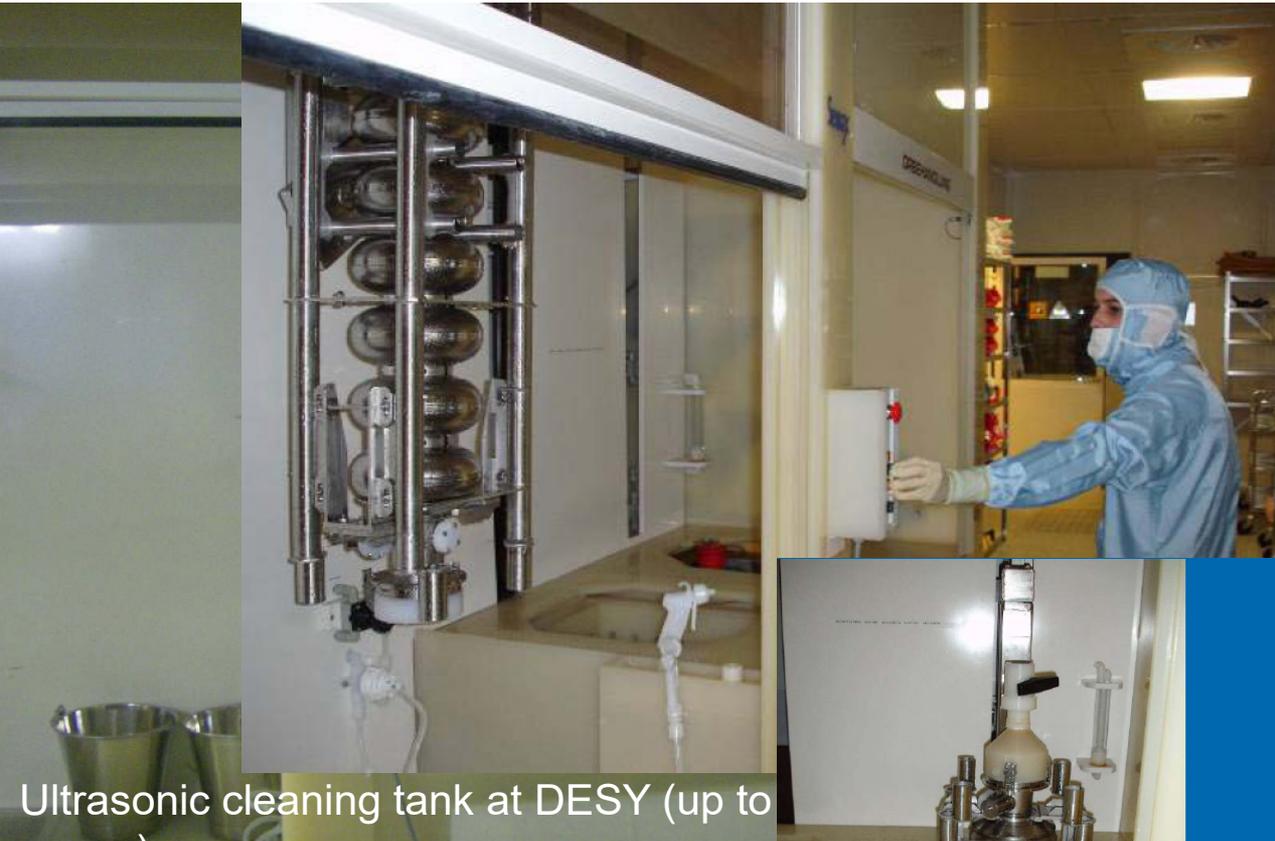
- To remove grease, oil and finger prints from cavity surfaces
- To remove surface contamination due to handling, RF measurements and QA inspection

Implementation:

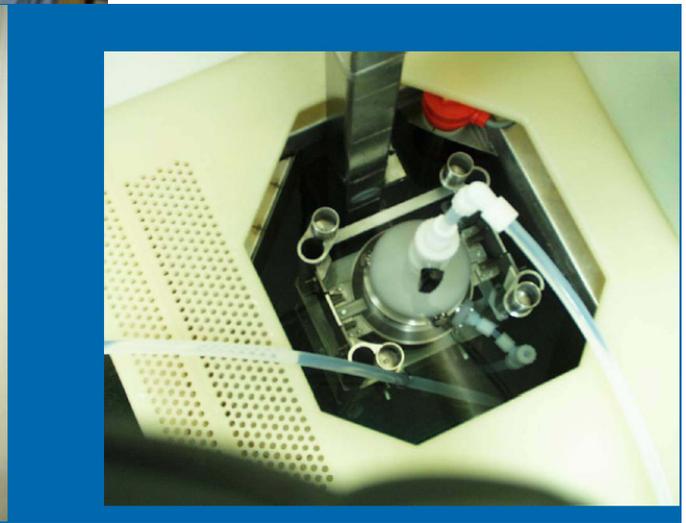
- Ultrasonic degreasing with detergent (Micro-90[®], Liqui-Nox[®]), 1%-2% concentration, and ultra pure water
- Usually performed in Hepa filtered air
- Water quality is good, 18 MW cm, Filtration > 0.2mm
- Manually or semi-automated processes available
- Problem: Parts are wet and vulnerable to particulate contamination



Ultrasonic Tanks for Cavity Cleaning



Ultrasonic cleaning tank at DESY (up to 1000L)



Acid Etching of Components & Cavities



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

Implementation: (BCP or EP)

- Subcomponents usually processed by hand in wet bench
- Acid quality usually electronic grade or better, low in contaminants
- Acid temperature control required to prevent additional absorption of hydrogen (Q-disease)
- Acid mixture difficult to QA

Chemistry of Nb Surface Etching

Buffered Chemical Polish (BCP)

HF (49%), HNO₃ (65%), H₃PO₄ (85%)

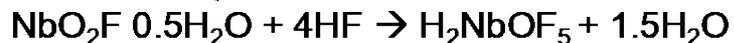
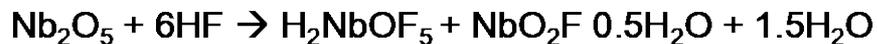
Mixture 1:1:1, or 1:1:2 by volume typical

Oxidation

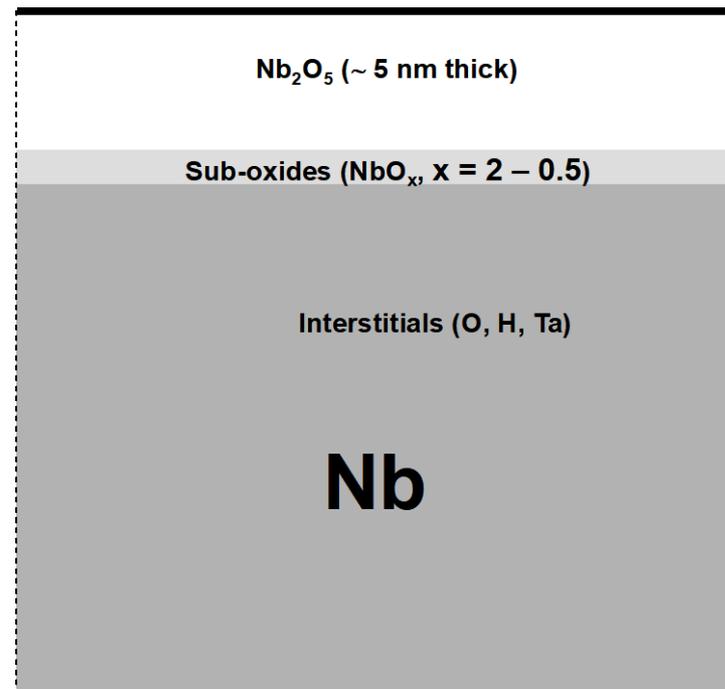


Brown gas

Reduction



Reaction exothermic! Use H₃PO₄ as “buffer” to slow reaction rate



BCP Systems for Cavity Etching

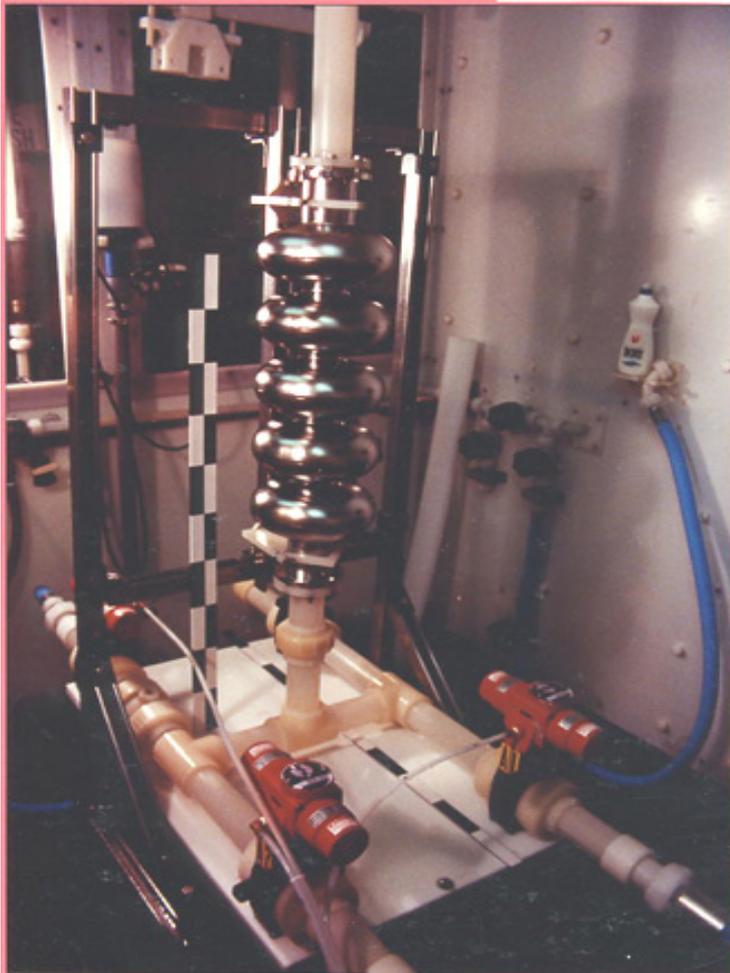
- Bulk & Final chemistry
 - Bulk removal of (100-200 μm)
 - Final removal of (5-20 μm) to remove any additional damage from QA steps and produce a fresh surface

Implementation:

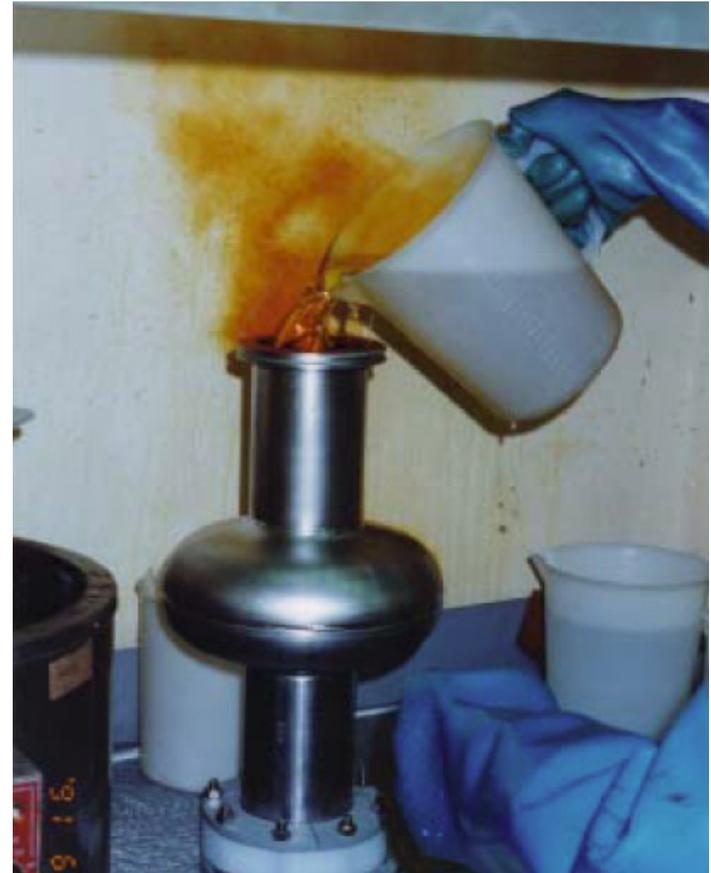
- Cavity held vertically
- Closed loop flow through style process, some gravity fed system designs
- Etch rate 2x on iris then equator, if no stirring mechanism
- Temperature gradient causes increased etching from one end to the other
- Manually connected to the cavity but process usually automated



Chemical Etching Setups

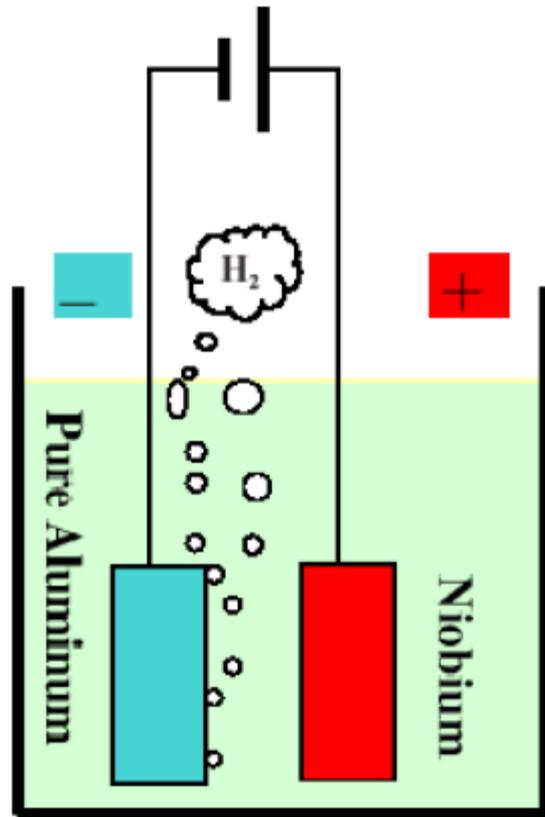


Old system for CEBAF cavities



BCP of single cell cavity under
chemical flow hood

Chemistry of Nb Electropolishing

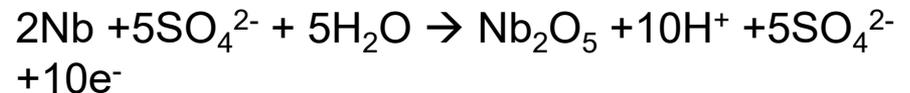


Electropolishing (EP) of Niobium:

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

Basic reactions:

Oxidation



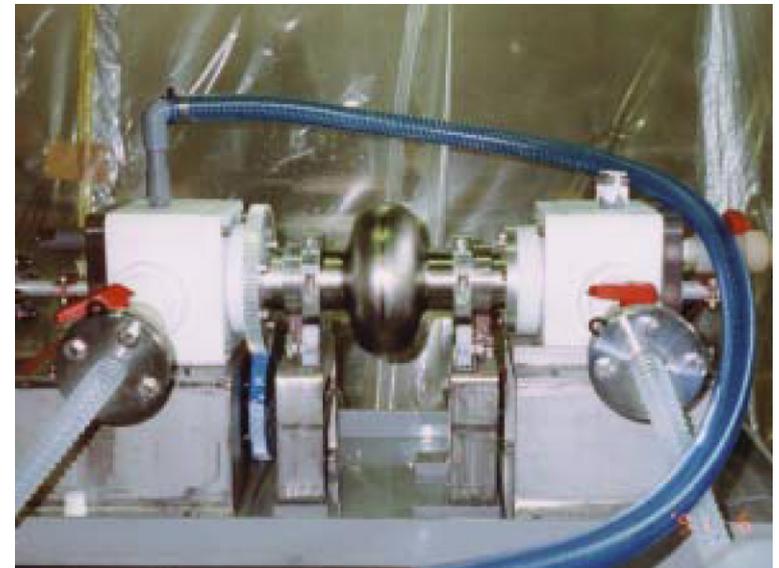
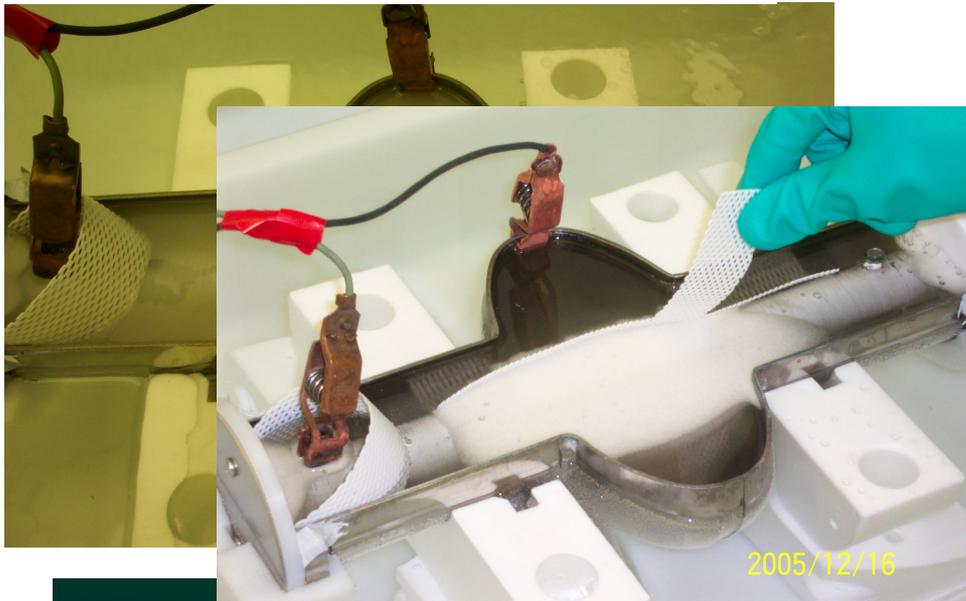
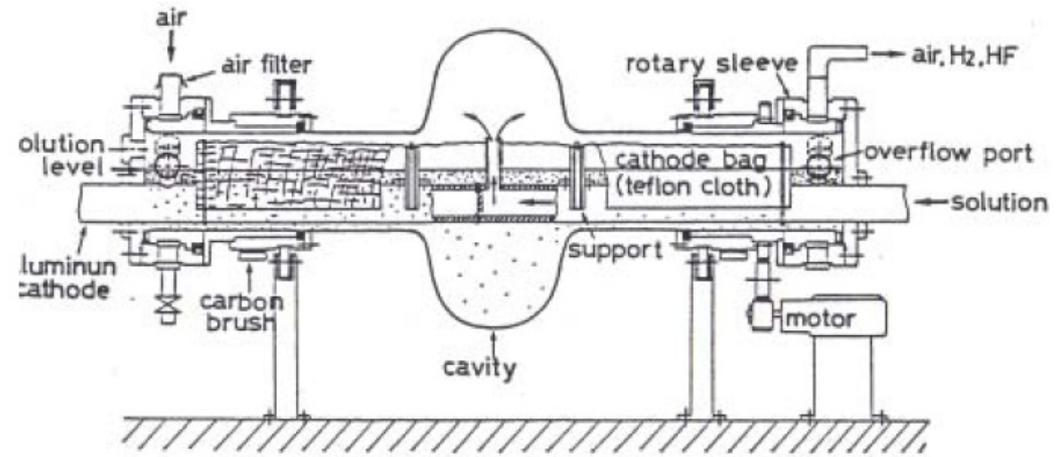
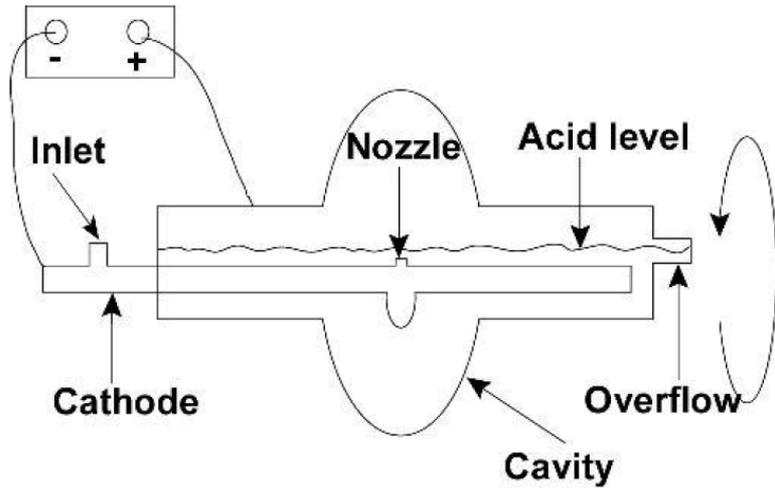
Reduction



Acid:
 $\text{H}_2\text{SO}_4 (>93\%): \text{HF}(46\%) = 10:1 \text{ V/V}$

9:1

EP Systems: Single-Cell



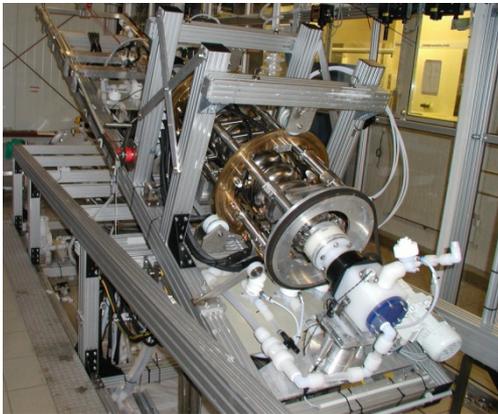
EP Systems for Multi-cell Cavities



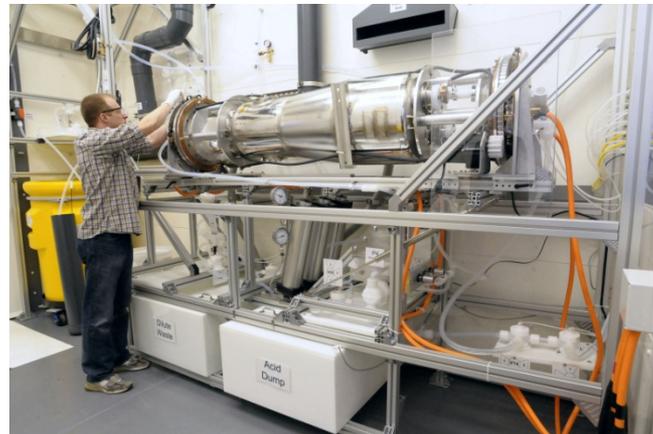
Nomura Plating and KEK



JLAB EP machine with active cooling



DESY EP machine



ANL low-beta cavity EP machine



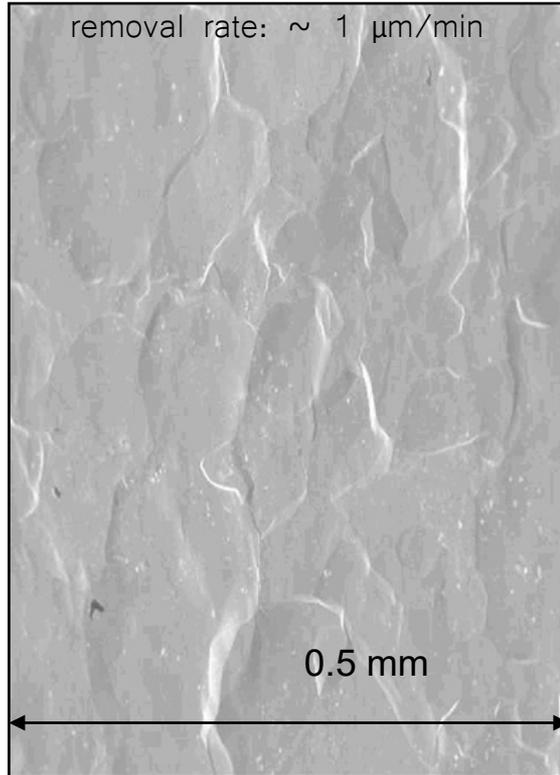
Cornell vertical EP machine

Polishing Techniques: BCP, EP

Buffered Chemical Polishing (BCP)

HF:HNO₃:H₃PO₄ in volume ratio 1:1:2

removal rate: ~ 1 μm/min

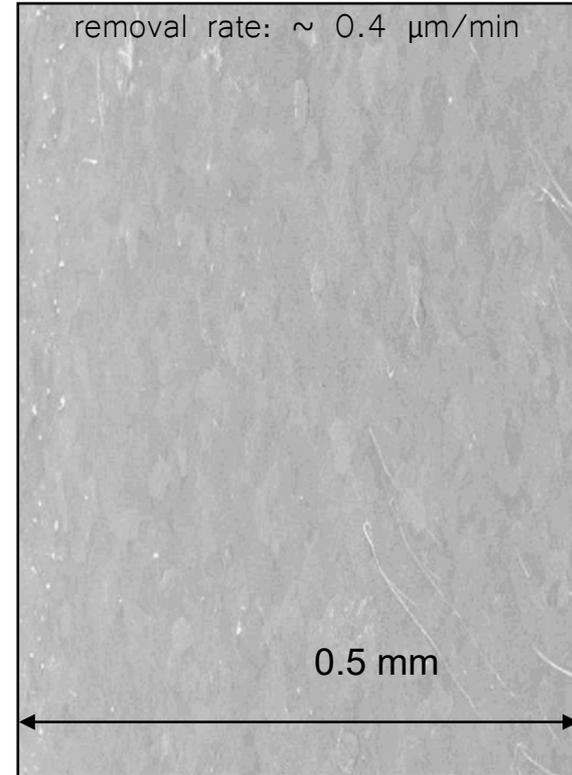


BCP Surface
(1 μm roughness)

Electropolishing Polishing (EP)

HF : H₂SO₄ in volume ratio 1:9

removal rate: ~ 0.4 μm/min



EP Surface
(0.1 μm roughness)

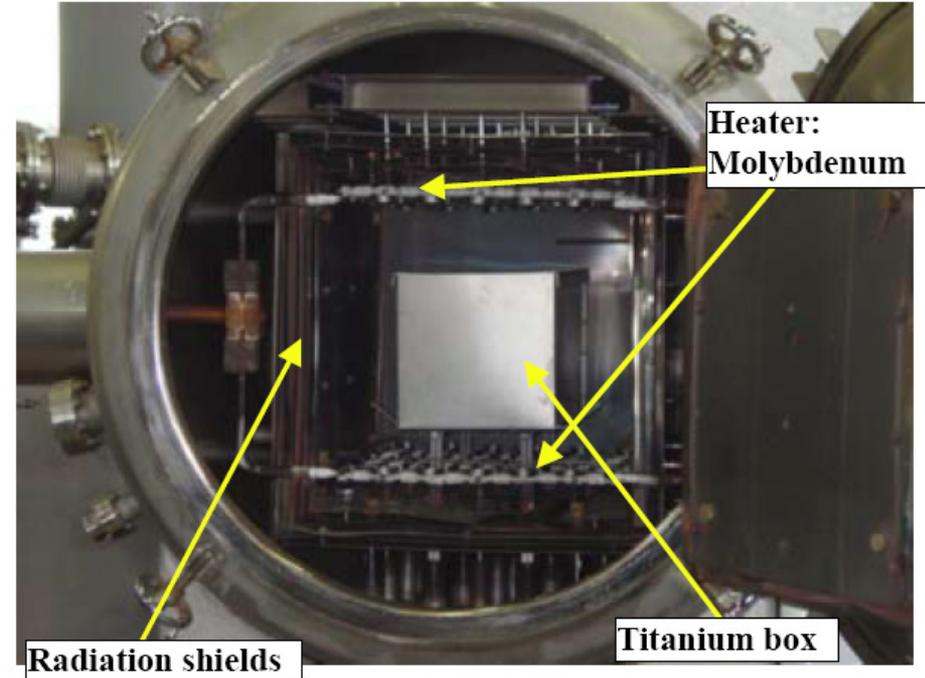
Heat Treatment for H-degassing

- H absorption occurs during chemical and/or mechanical material removal
- Reduce bulk H concentration in Nb to avoid Q-disease
- The heat treatment also “stress-relieves” the Nb
- Different parameters at different labs (600-900 °C)

High Temperature Vacuum Furnace



Heat Treatment Furnace at JLab up to
1250 °C, $P \leq 10^{-6}$ Torr

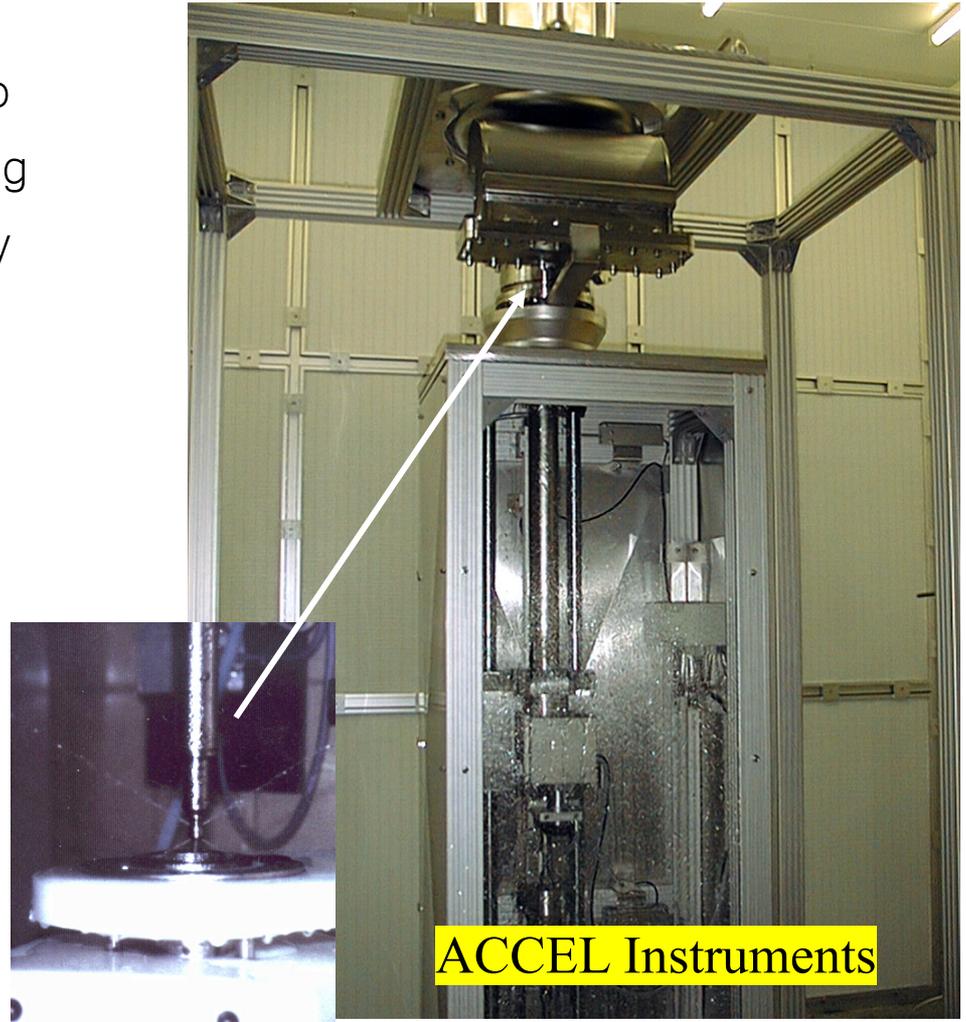
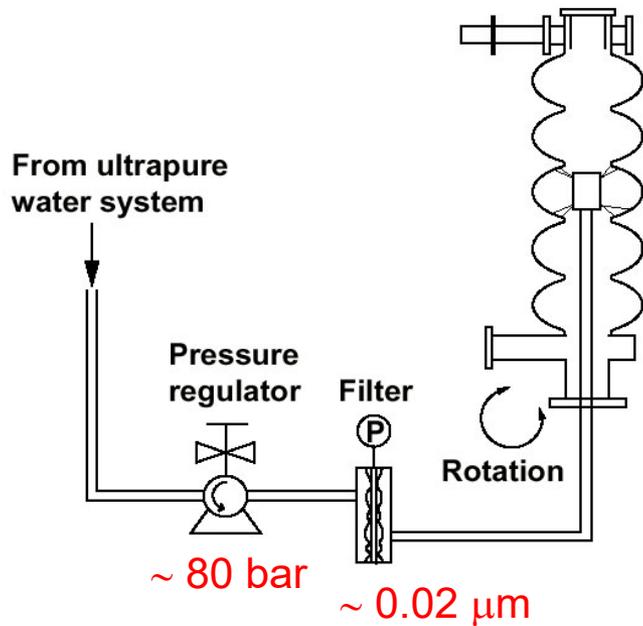


Vacuum furnace in KEK : Temp.= 1300 °C max,
Vac. = 1xE-6 torr

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

High Pressure Rinsing (HPR)

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



High Pressure Rinsing (HPR)

Before

After



High Pressure Water Rinse Systems



KEK system



JLab HPR cabinet

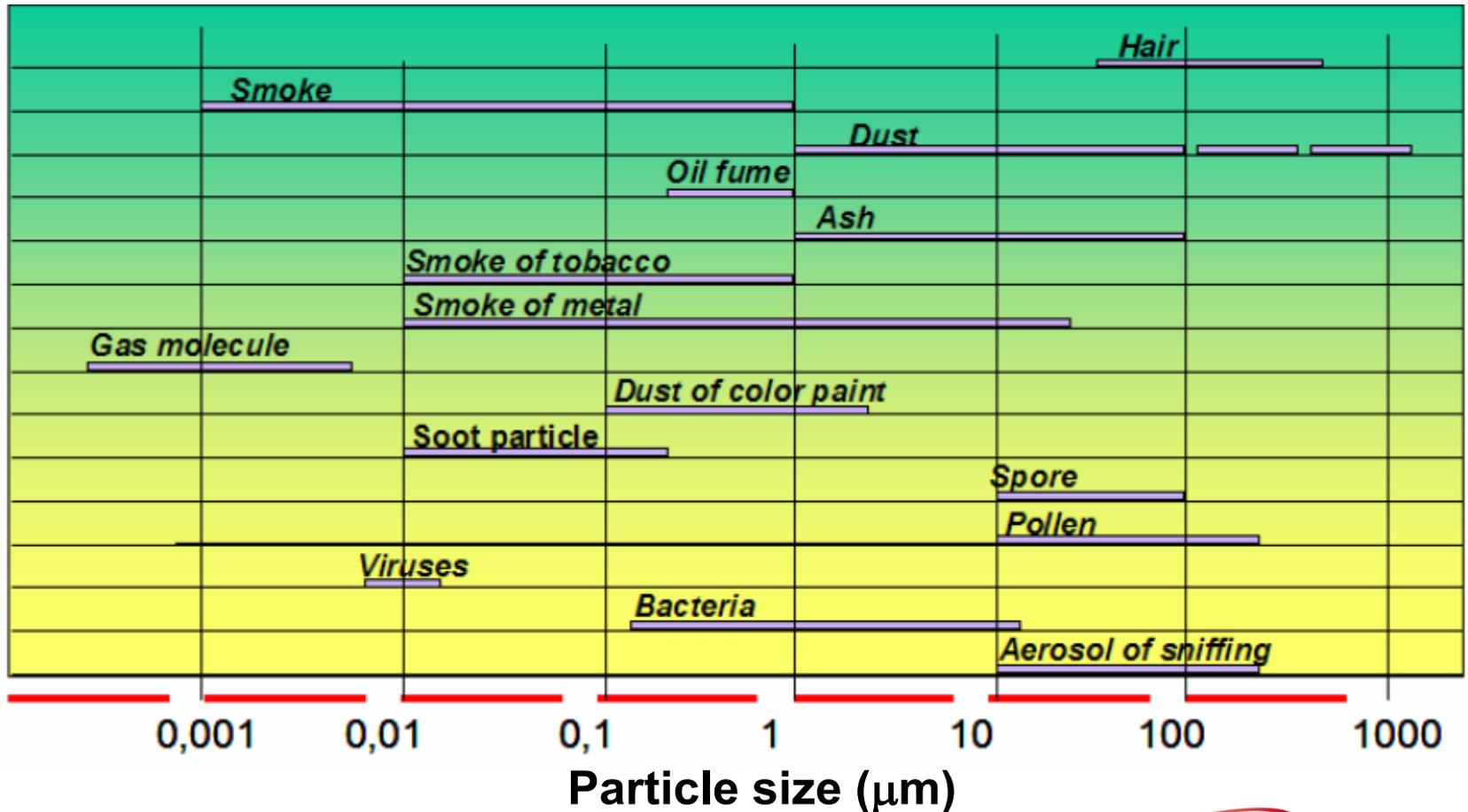


Nozzles of various designs

DESY system

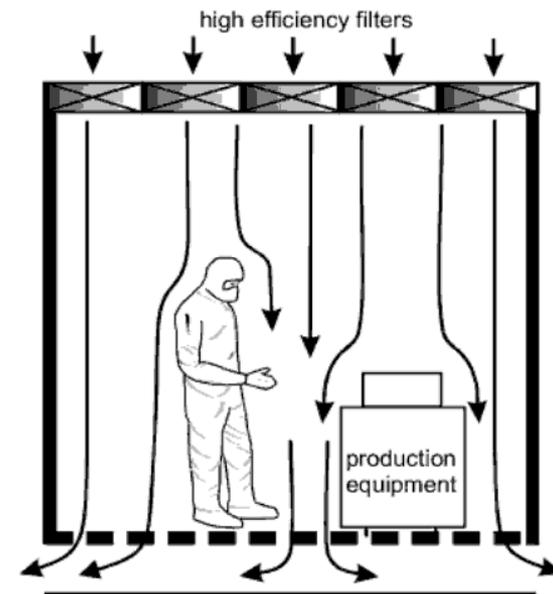
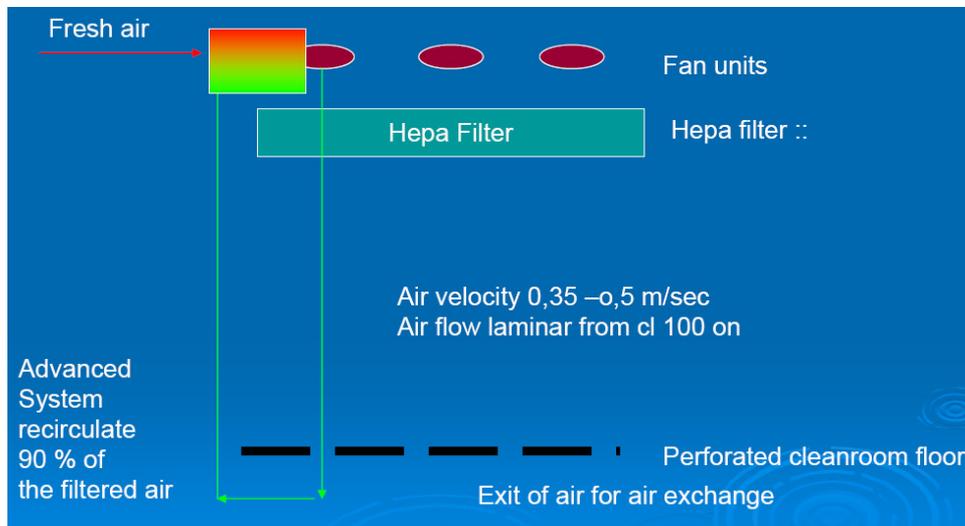
Particulates in Air

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



Cleanroom Technology

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



Cleanroom Classification

**ISO
Classification
number**

Maximum concentration limits (particles/m³ of air) for particles equal to and larger than the considered sizes shown below

	$\geq 0.1\mu\text{m}$	$\geq 0.2\mu\text{m}$	$\geq 0.3\mu\text{m}$	$\geq 0.5\mu\text{m}$	$\geq 1\mu\text{m}$	$\geq 5.0\mu\text{m}$
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

ISO 14644-1 Classes
FS 209 Classes

Class 3 Class 4 Class 5 Class 6 Class 7 Class 8
Class 1 Class 10 Class 100 Class 1000 Class 10,000 Class 100,000

↑
Cavity
assembly

↑
Cleanroom for
SRF

Room air ISO 9
Class 1 million



People in Cleanrooms

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



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6/22-24, 2023, MSU, East Lansing, MI

Contamination Prevention

Micro-particle contamination is the leading cause of field emission. This stresses the importance of cleanliness in all final treatment and assembly procedures.

Sources of contamination:

- Processing chemicals (filtered!)
- High purity water ($>18\text{ M}\Omega\text{-cm}$, $<0.02\ \mu\text{m}$ filter)
- Clean room environment (entrance, class 10)
- Particulates on equipment, tooling, hardware, clothing, gloves, ...

Contamination control:

- Stringent control of processes and procedures
- In-line monitoring of particulate levels in air and liquids
- Scheduled maintenance
- “Blow-off” with filtered N_2 , monitored by particle counter
- Use of appropriate hardware (e.g. bolts..)
- Clever designs (e.g. gaskets, clamp rings, fixtures...)
- Consistent use of “best practices” through whole assembly process



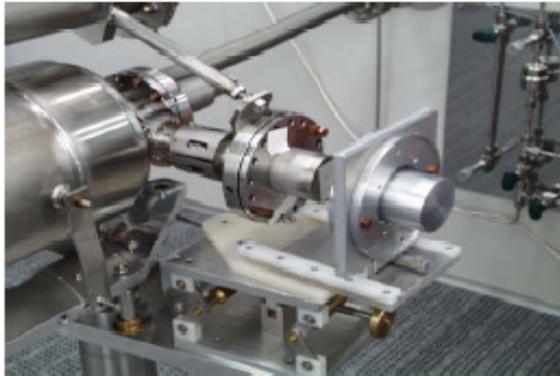
Clean Room Assembly



JLAB clean room assembly inside class-100 area for critical step

Cavity String and Cryomodule Assembly

Handling and assembly II



Detlef Reschke – SRF Workshop Cornell, 10. Juli 2005

25



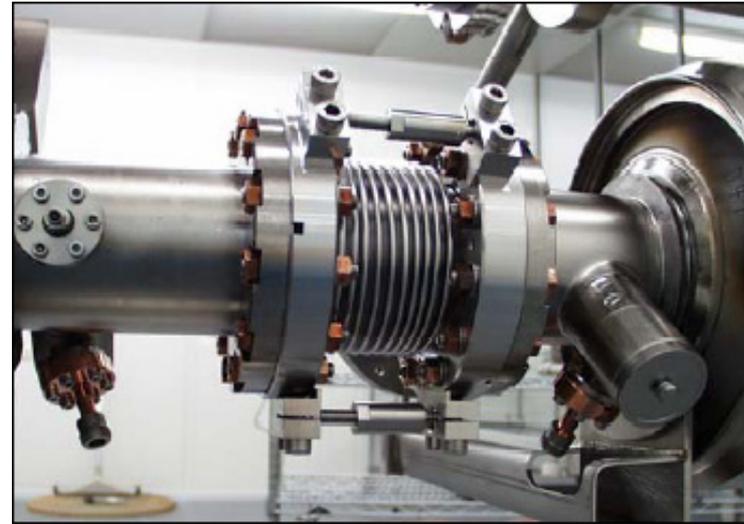
DESY clean room assembly



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6/22-24, 2023, MSU, East Lansing, MI



Cavity String and Cryomodule Assembly



The inter-cavity connection is done in class 10 cleanrooms



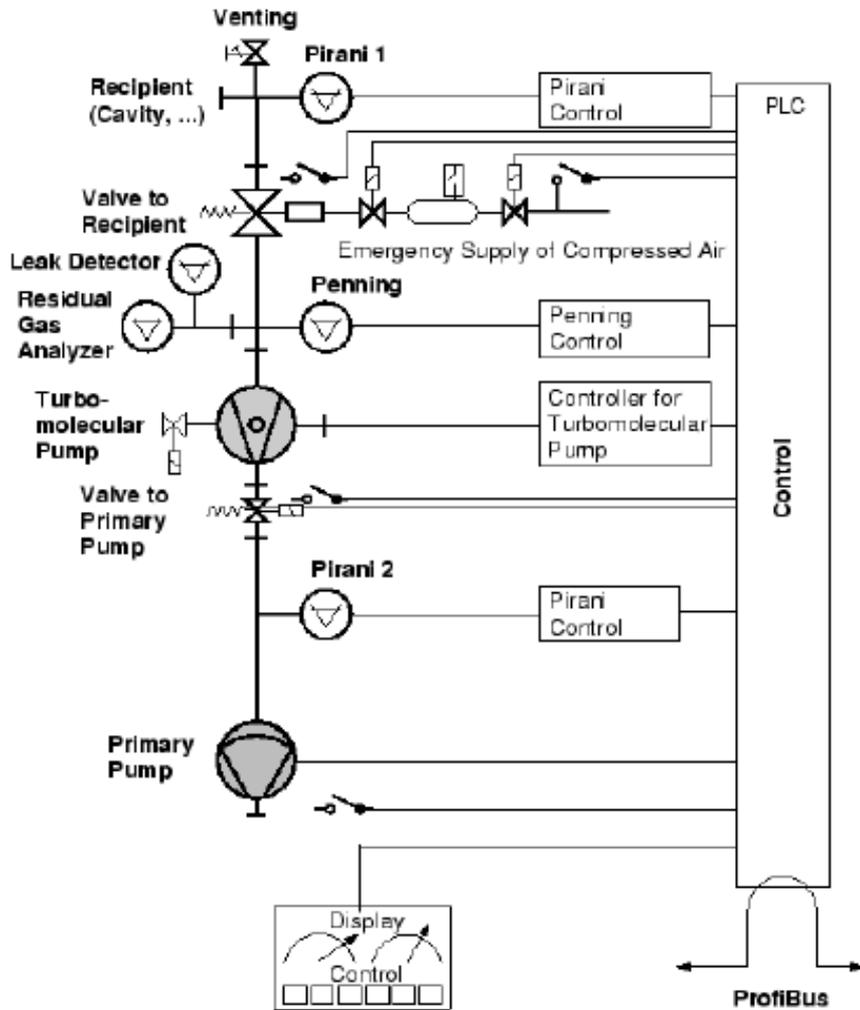
ANL QWR String Assembly



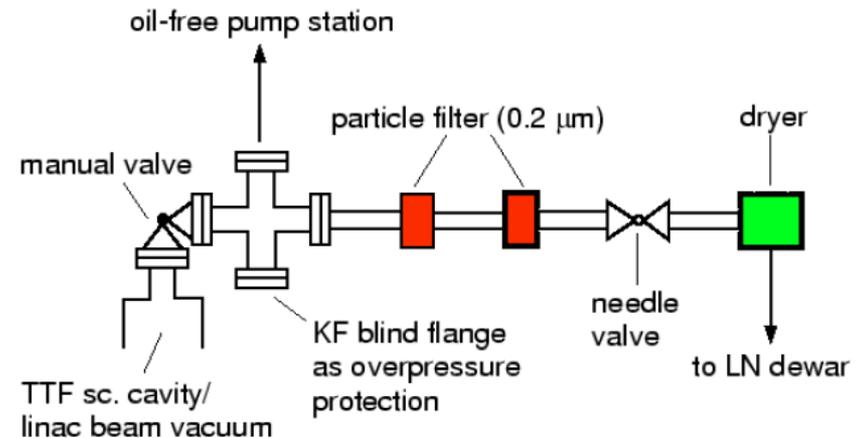
Clean Room at DESY



Clean Evacuation



- Oil-free pump stations with leak check and residual gas analyzer
- Laminar venting with pure, particle filtered N_2 or Ar

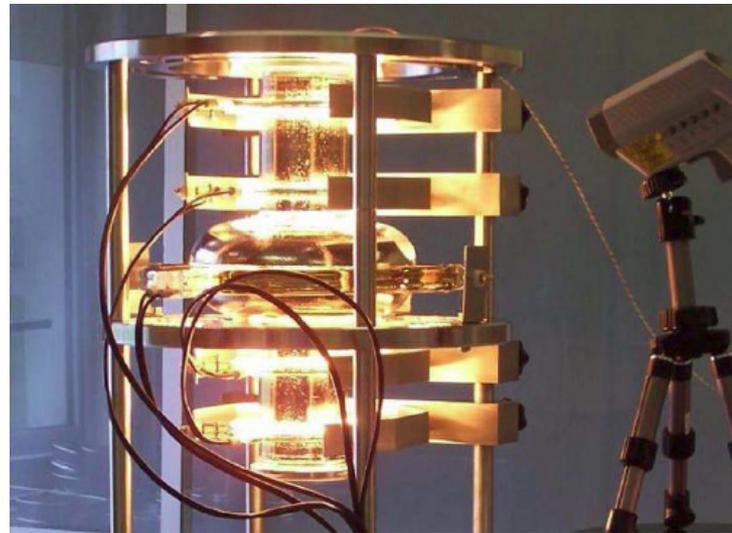


Low-Temperature Baking



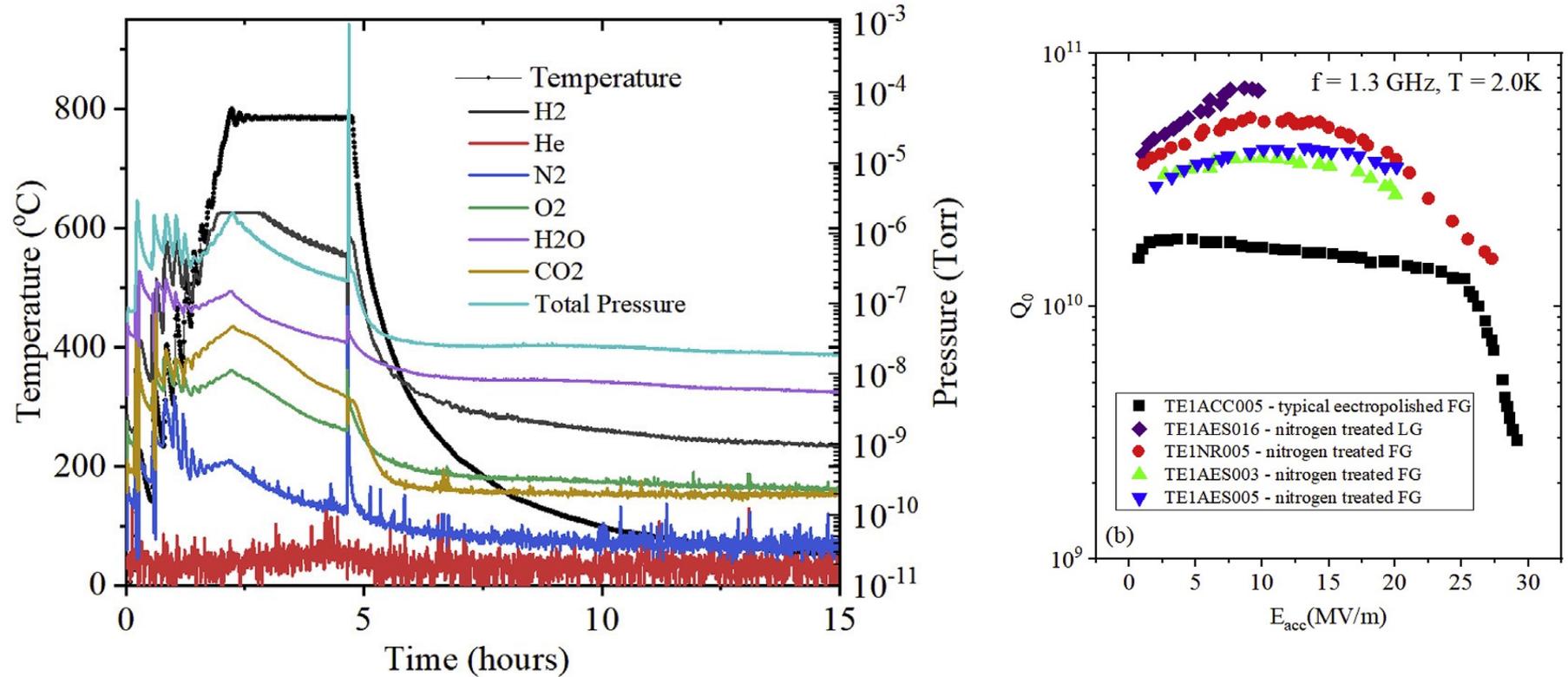
Hot N_2 gas uniformly heats up the cavity (JLab)

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



Benefit: eliminate high-field Q-slope (slides later)

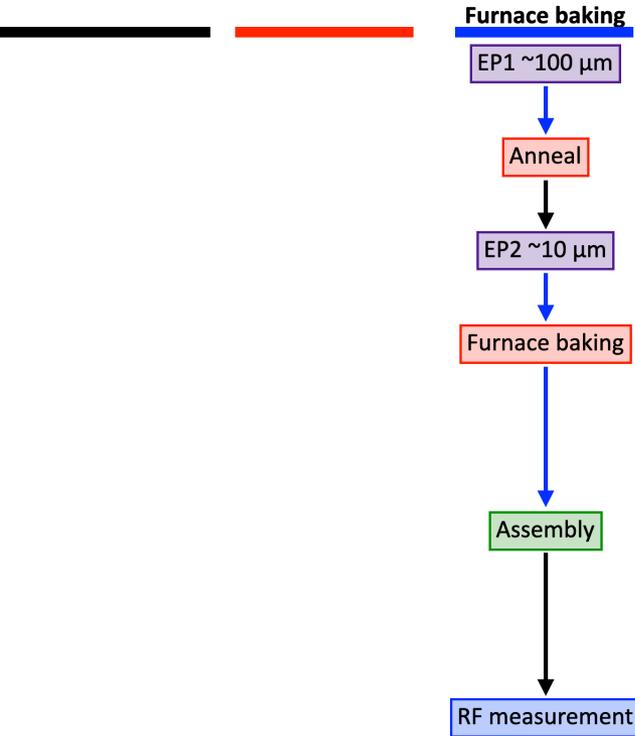
Treatments Raising Q0 – Nitrogen Doping



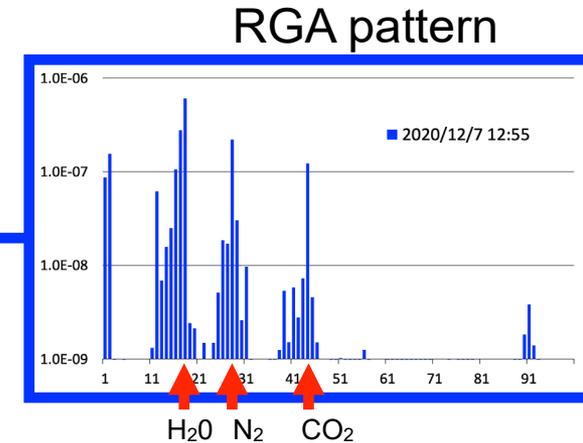
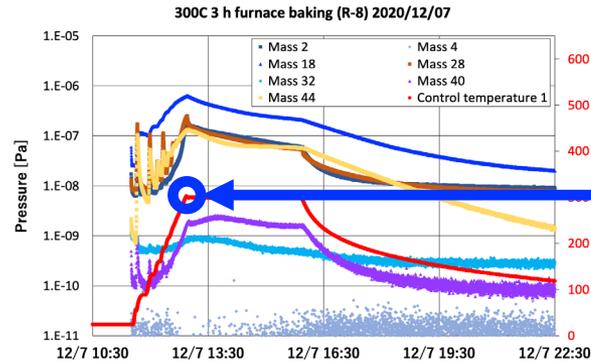
Typical temperature and pressure profile with residual gas analyzer during the nitrogen doping. The nitrogen was injected into the furnace at the end of 800 C for 2 min (N₂ pressure 25 mTorr) and the cavity was further annealed in UHV for 6 min before cooldown to room temperature. Post treated cavity surface was EP processed for a small removal of 5-10 μm .

Treatments Raising Q0 – Mid-T Bake v1

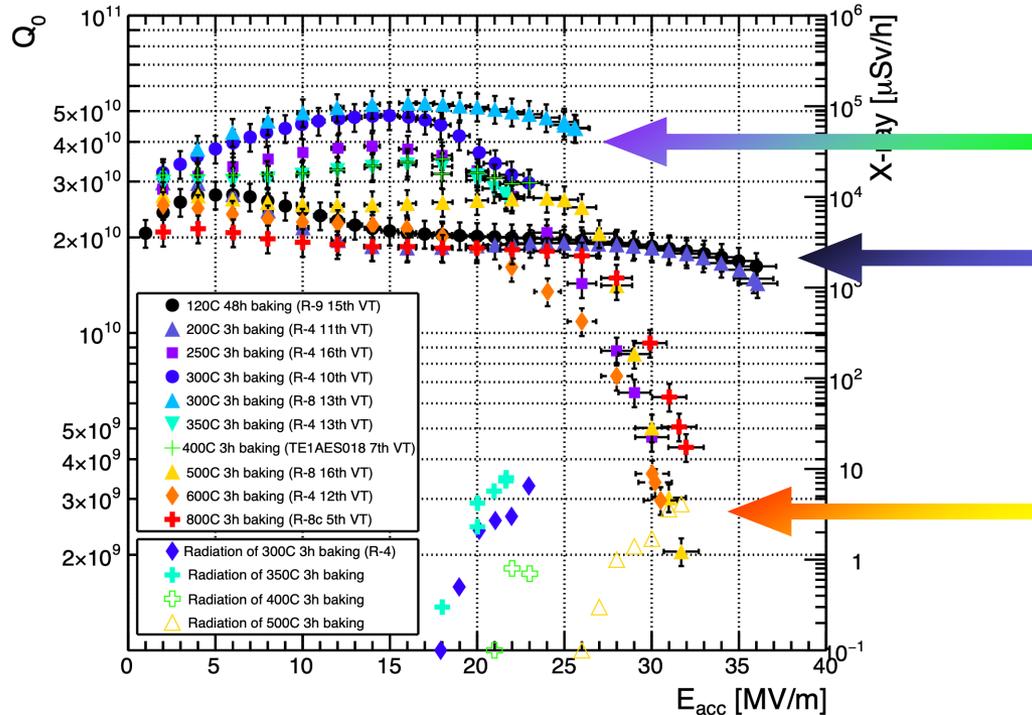
300 C x 3hr



→ w/o HPR
→ w/ HPR



Q(E_{acc}) Behaviors Baked at Various T



250 ~ 400°C 3 h

- Extremely high Q value and anti-Q slope are observed
- Highest Q value at 2.0 K is ~ 5E10 for 300°C baked cavity
- Magnetic field was trapped before 2 K measurement of 350°C baked cavity -> Q value is Essentially a bit higher

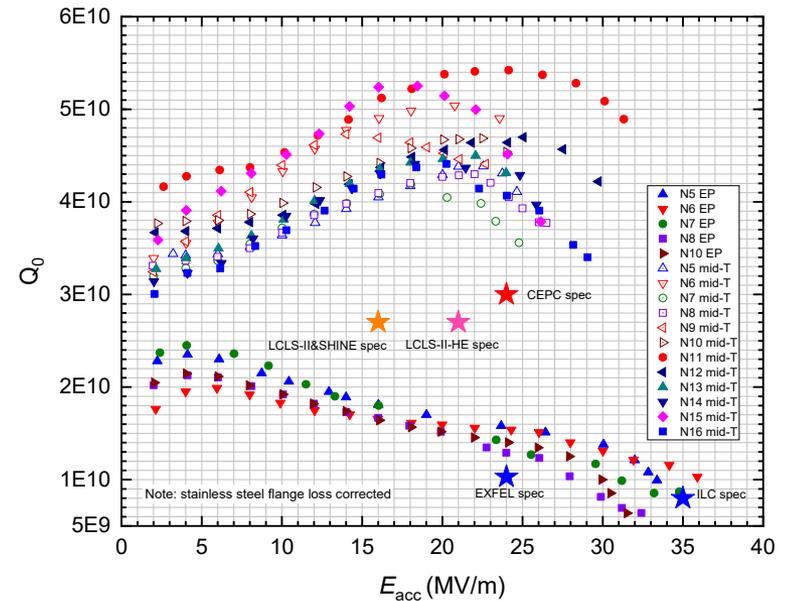
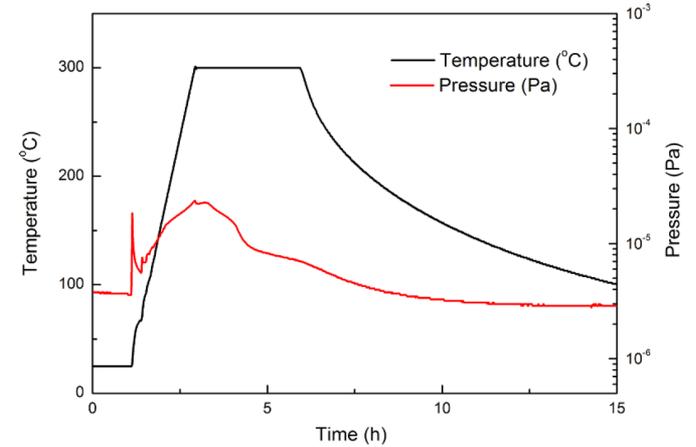
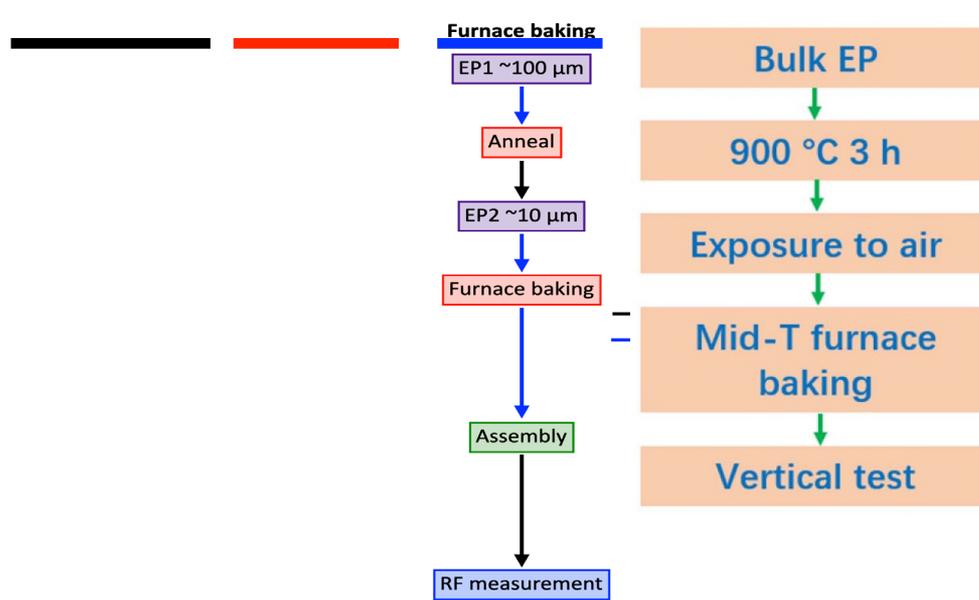
Standard recipe (120°C 48 h), 200°C 3 h

- 200°C baked cavity follows the standard recipe (120°C 48h)
- Q-E behavior at low E_{acc} is slightly different

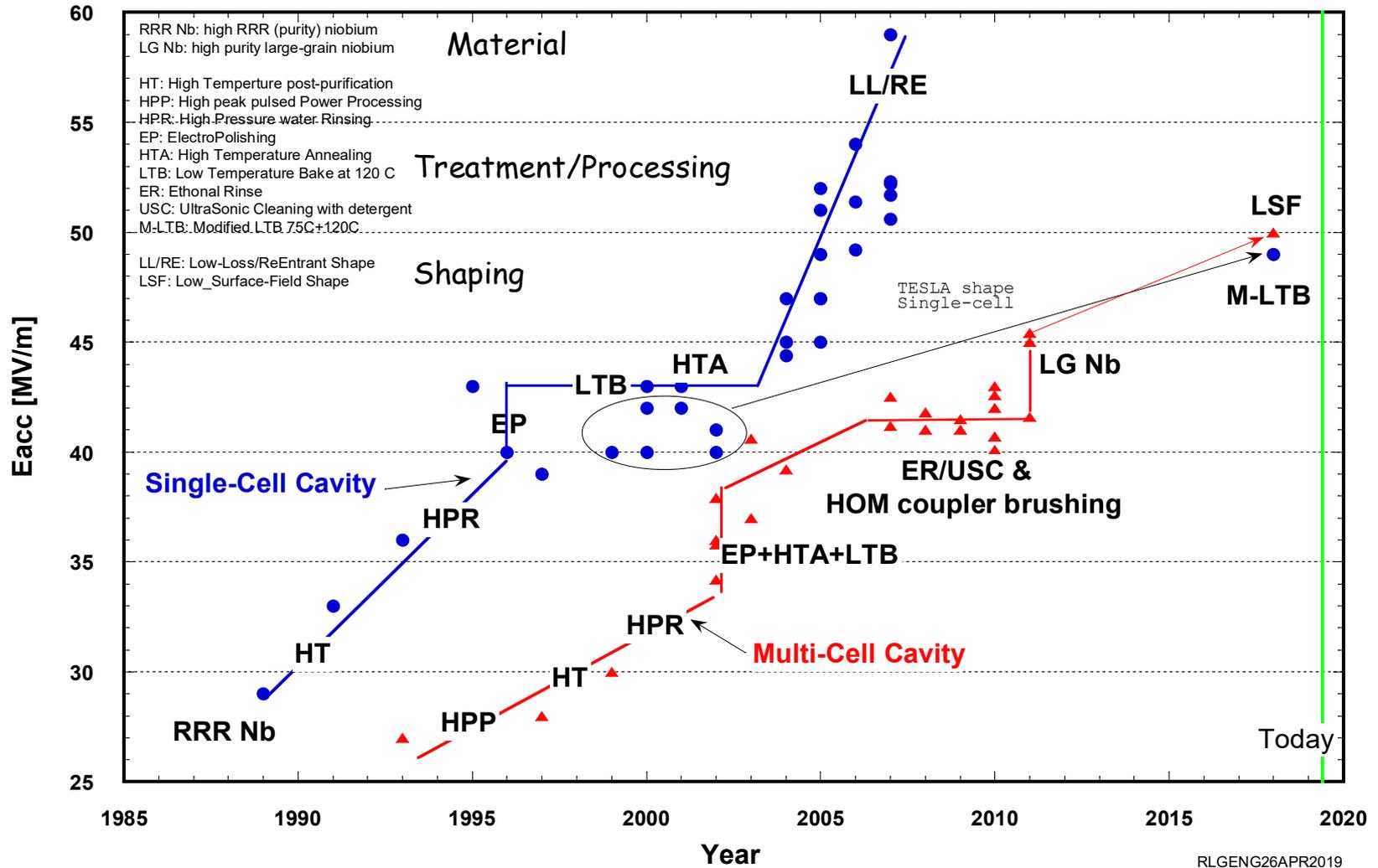
500 ~ 800°C 3 h

- High Q value wasn't observed
- HFQS occurred

Treatments Raising Q0 – Mid-T Bake v2



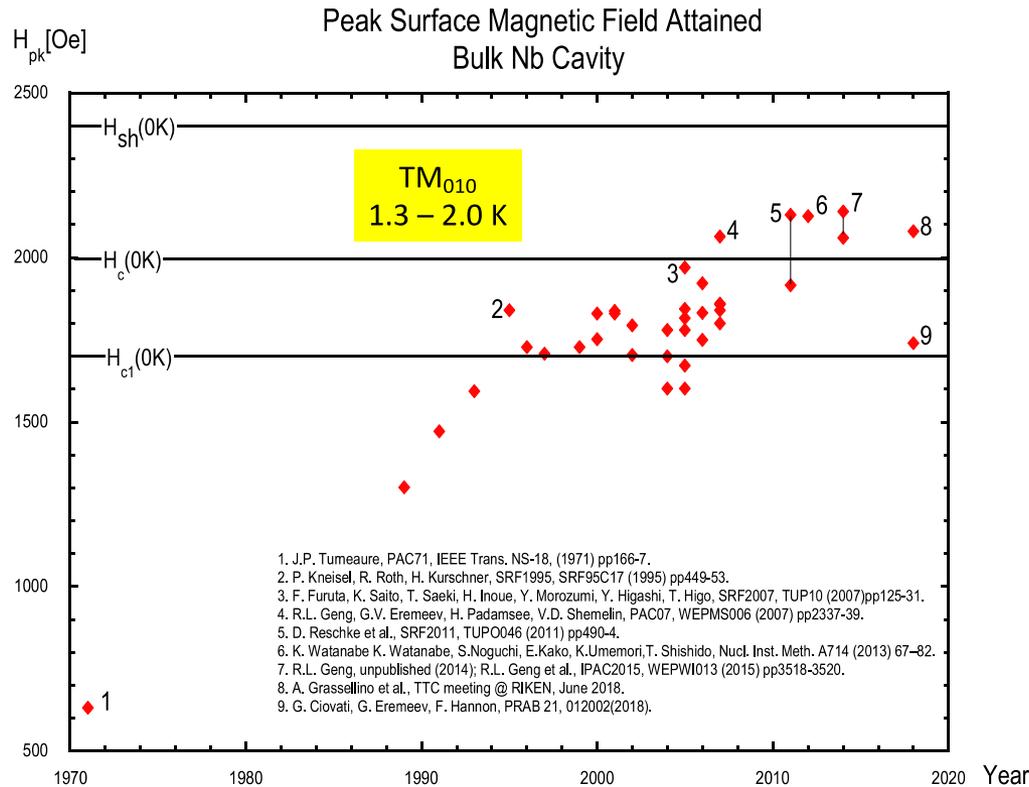
L-Band SRF Cavity Gradient Improvement and Underlying Technical Approaches



RLGENG26APR2019



Treatments Pushing Performance to Theoretical Limit



Data Point	material	Freq [GHz]	# of cells	Treatment
1	Nb	1.3	1	HT+LTB
2	Nb	1.3	1	BCP
3	Nb	1.3	1	EP+LTB
4	Nb	1.3	1	EP+LTB
5	LG Nb	1.3	9	EP+LTB
6	Nb	1.3	2	EP+LTB
7	LG Nb	1.5	1	EP+LTB
8	Nb	1.3	1	EP+M-LTB
9	Nb	3.0	1	EP+LTB

Nb: Fine-grain nNb

LG Nb: large grain ingot Nb

HT: UHV firing 1800 °C

CP: Buffered chemical Polishing (HNO₃+HF+H₂O)

EP: Electropolishing (HF+H₂O+H₂SO₄)

LTB: Low temperature bake 100-120 °C

M-LTB: Modified LTB (75 °C + 120 °C)

What You Have Learned

- Cavity fabrication methods from sheet Nb forming to electron beam welding.
- Cavity processing methods and techniques for high gradient high Q0 SRF cavities from surface chemical processing, furnace vacuum heat treatment, clean room assembly, and in-situ low-temperature bake.
- Nb cavity performance steady improvement over past 50 years.