EASISchool 3
Superconductivity and its applications

SRF Cavity Fabrication

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Outline

- SRF Cavity fabrication, a long chain …
- From EM design to fabrication
- A fabrication cycle
- From raw Nb to sheets
- From sheets to cavity
- Cavity treatments

Fabrication activities at LASA

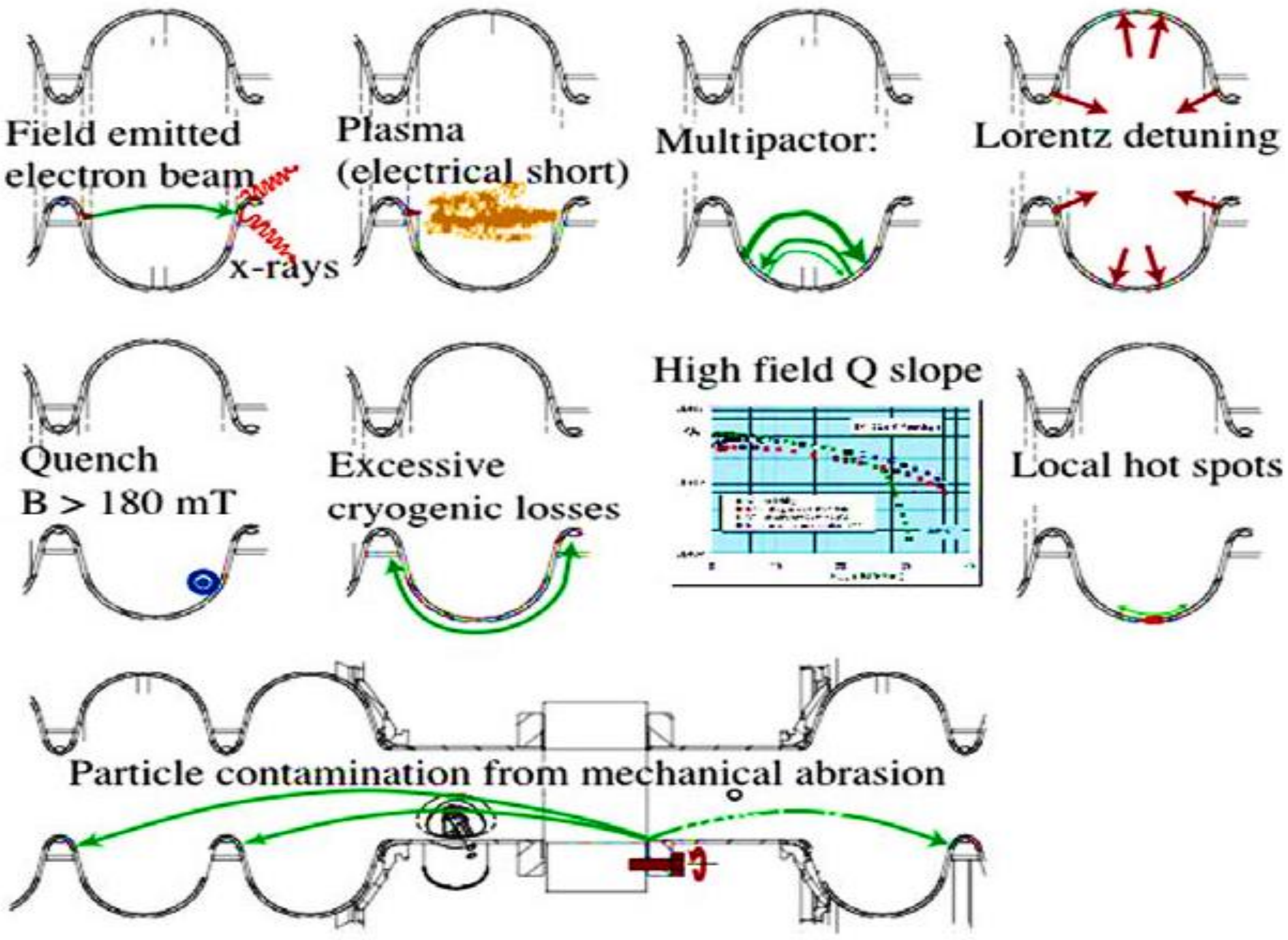
- INFN LASA has a long experience on cavity design and, in collaboration with industry, in fabrication of cavities
  - We have significantly contributed to the design of the **SNS cavities at 805 MHz**
  - **TRASCO cavities** were design at LASA and fabricated at **704.4 MHz**
  - LASA was deeply involved in the mass production of the **800 cavities for XFEL at 1.3 GHz**
  - **20 3.9 GHz** cavities for the third harmonic module of **XFEL** were designed at LASA and fabricated
  - **38 704.4 MHz** cavities for the Medium Beta Section of the **European Spallation Source**
  - **2 650 MHz** prototype cavities for the Low Beta Section of **PIP-II** in preparation for the 38 cavities in-kind contribution

SC cavities production: a long chain, but…

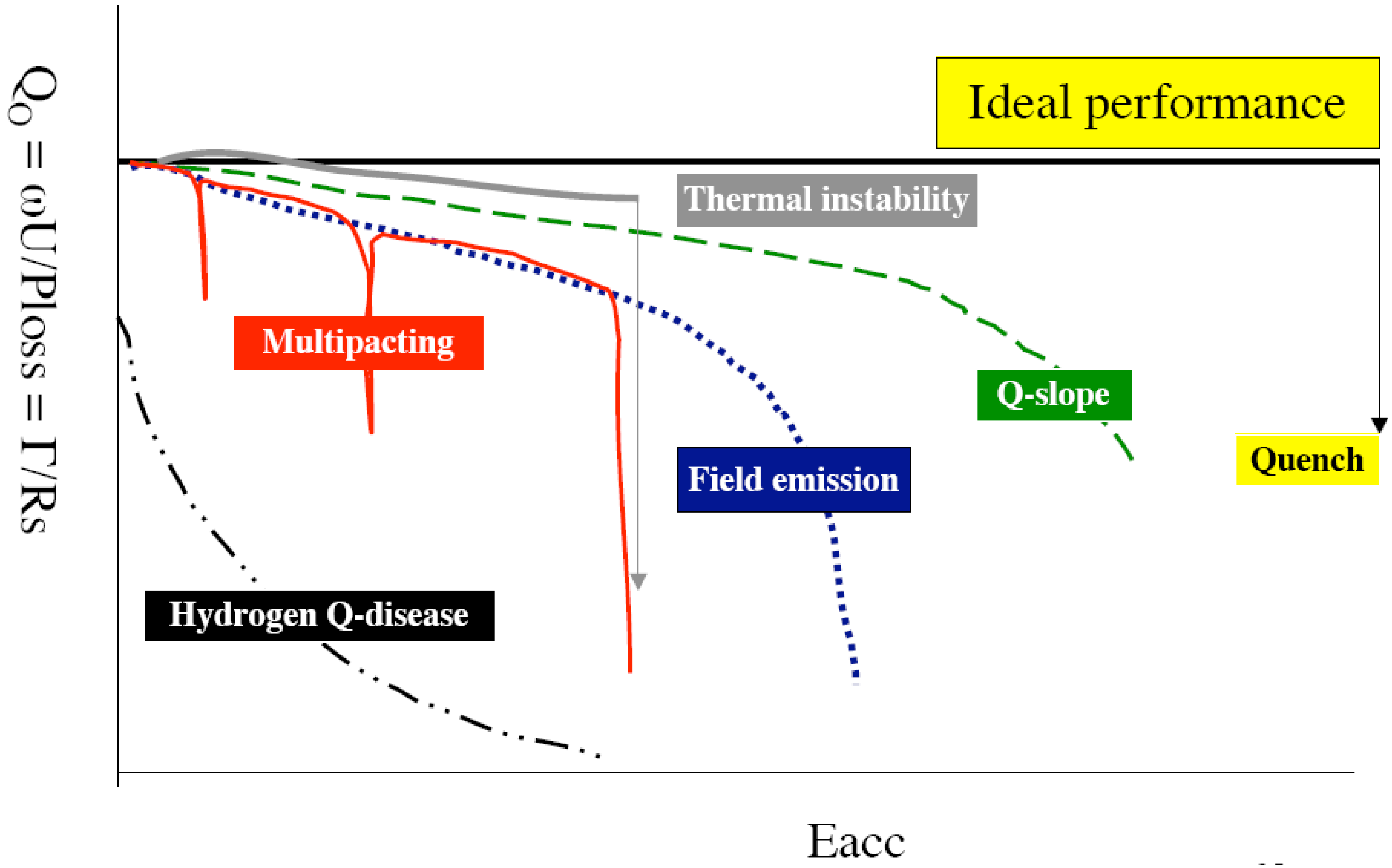
>A chain is as strong as its weakest link !!!

- **Chain of**
  - **- Material**
  - **- Fabrication**
  - **- Surface Preparation**
    incl. cleanroom, media, procedures, human factor
  - **- Vacuum**
  - **- Quality assurance**
- **For high gradient / low loss SRF cavities all aspects have to be fulfilled**

SC cavities may have various “illness”



Anomalous loss mechanism



Some general statements

- Anomalous loss mechanisms:
  - Quench (local thermal instability)
    => material + fabrication (=> cleanliness)
  - Field emission
    => Cleanliness of surface treatment, assembly, handling + vacuum
  - Q-drop (without field emission) + Q-slope => ?
  - Multipacting
    => Cavity shape + RF surface condition
  - Hydrogen Q-disease
    => Chemical surface treatment
  - Increased residual surface resistance
    => Cleanliness of surface treatment, assembly, handling + vacuum

Present picture of field emission: observations

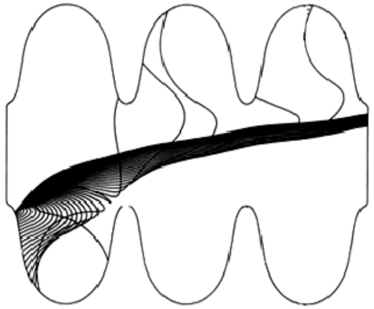
- Metallic (conducting) particles or “scratches” of irregular shape; typical size: 0.5 - 20 µm
- Only 5% - 10% of the particles emit
- Hydrocarbon contamination of the vacuum system
- Sulphur contamination after electropolishing process
- Modified Fowler-Nordheim’s law :

$$I \approx A_{FN} \frac{(\beta_{FN} E)^2}{\Phi} \exp\left(-\frac{C\,\Phi^{\frac{3}{2}}}{\beta_{FN} E}\right)$$

typical β-values between 50 and 500 for SRF cavities

A_{FN} (FN emission area) not directly correlated to physical size of emitter

Some general statements: field emission



- Field Emission is critical, more for multi-cell cavities for high RF accelerating fields
- **P. Kneisel + B. Lewis, SRF Workshop 1995**:
  "**Progress** towards routinely achieving higher gradients for future applications of RF-superconductivity **goes hand in hand with shifting the onset of field emission** loading towards higher fields."
- "It is generally accepted that the field emission behavior of a niobium cavity reflects the level of cleanliness of the superconducting surfaces subject to the RF-fields."

Improved clean preparation techniques allow an increased field emission onset

From EM design to fabrication

Few considerations

- From the EM point of view, the cavity is **design at the operative conditions** (usually 2 K, in vacuum, with tuner).
- When you **fabricate** the cavity, you are at **room temperature, in air** and the cavity needs to be **treated** before being operational
- You need then to consider
  - Thermal shrinkage from 2 K to 300 K (geometry, frequency)
  - Pressure effect (frequency)
  - Dielectric constant effect (frequency)
  - Over-metal for chemical treatment (geometry, frequency)
  - Pre-tuning (frequency)

P.S. When you fabricate a cavity, be sure that the design couples with the feasibility of the processes you are going to apply

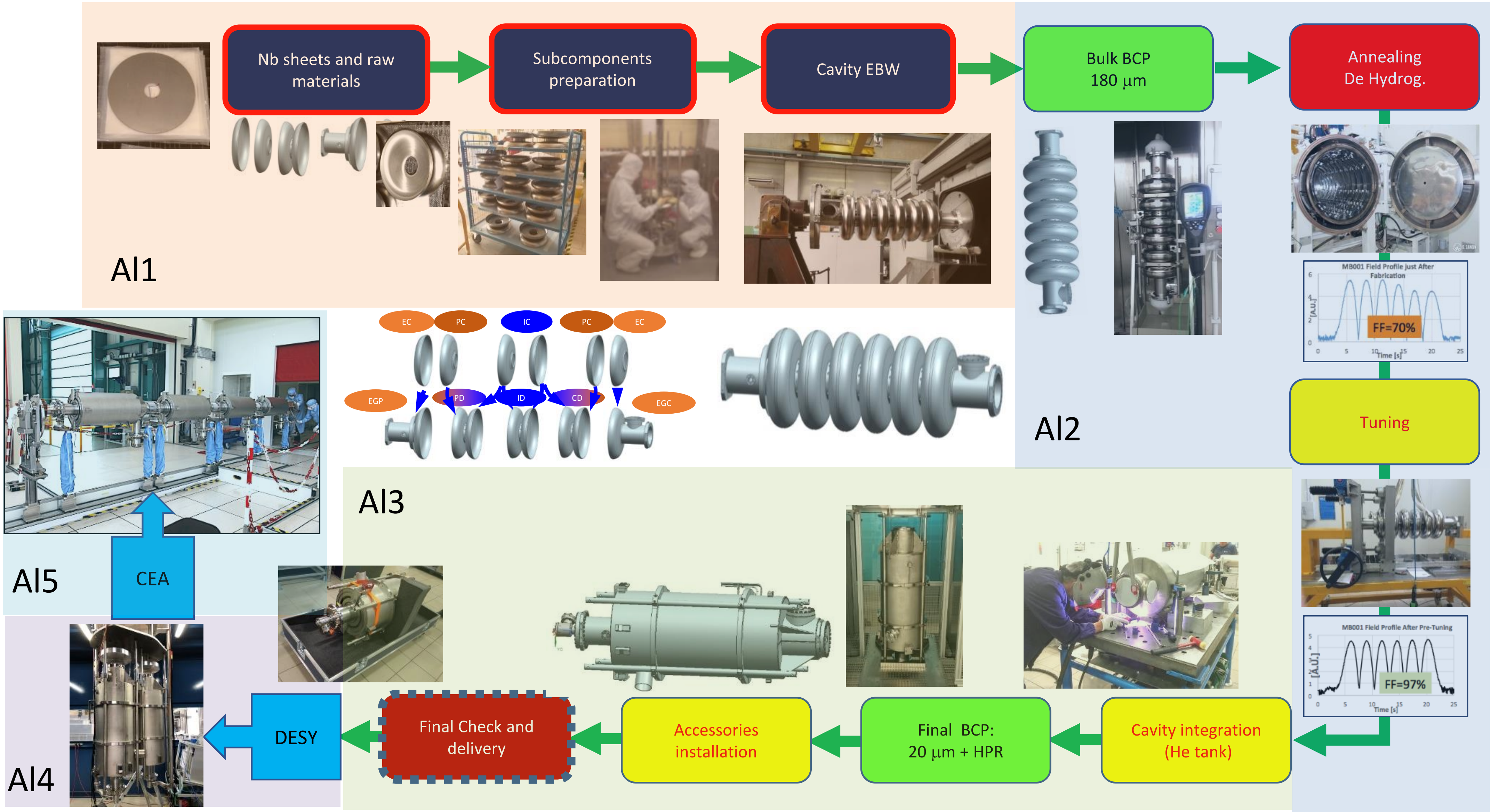
The ESS example

Step	Df [MHz]	Cavity Frequency [MHz]	Comment
Goal Frequency		704.420 MHz	2 K in vacuum
Pre-load for tuner	-0.100 MHz	704.320 MHz	Unloaded cavity at cold
Room Temperature	-1.028 MHz	703.292 MHz	Shrinkage from 2 K to 300 K [a]
In Air	-0.234 MHz	703.058 MHz	Dielectric constant [b]
Etching	+0.480 MHz	703.538 MHz	Before Chemistry (150 μm) [c]
Weld Seam	+0.000 MHz	703.538 MHz	Weld Seam perturbation [d]

[a] Integral shrinkage from 2 K to 300 K tabulated
[b] Inversely proportional to square root of dielectric constant
[c] Estimated from Slater's perturbation on cavity inner surface
[d] Estimated from direct measurement. More significant for high frequency cavities

A fabrication cycle

The ESS fabrication cycle

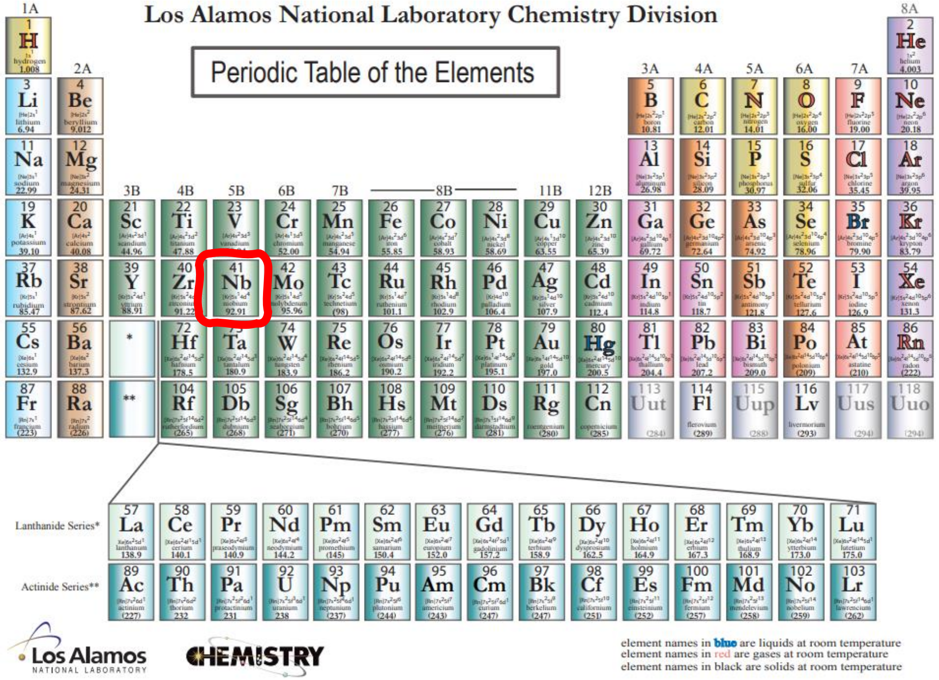


From raw Nb to cavity

Niobium

- Niobium is ***THE*** material for fabrication of supercondiucting cavities
  - Critical temperature T_c = 9.25 K
  - High critical field (H_c(0 K) ≅ 240 mT)
  - Chemically inert (surface covered by Niobium pentoxide Nb_2O_5)
  - Easily machined and deep drawn
  - Available as bulk and sheets of any size and different shapes

Highest between pure metals



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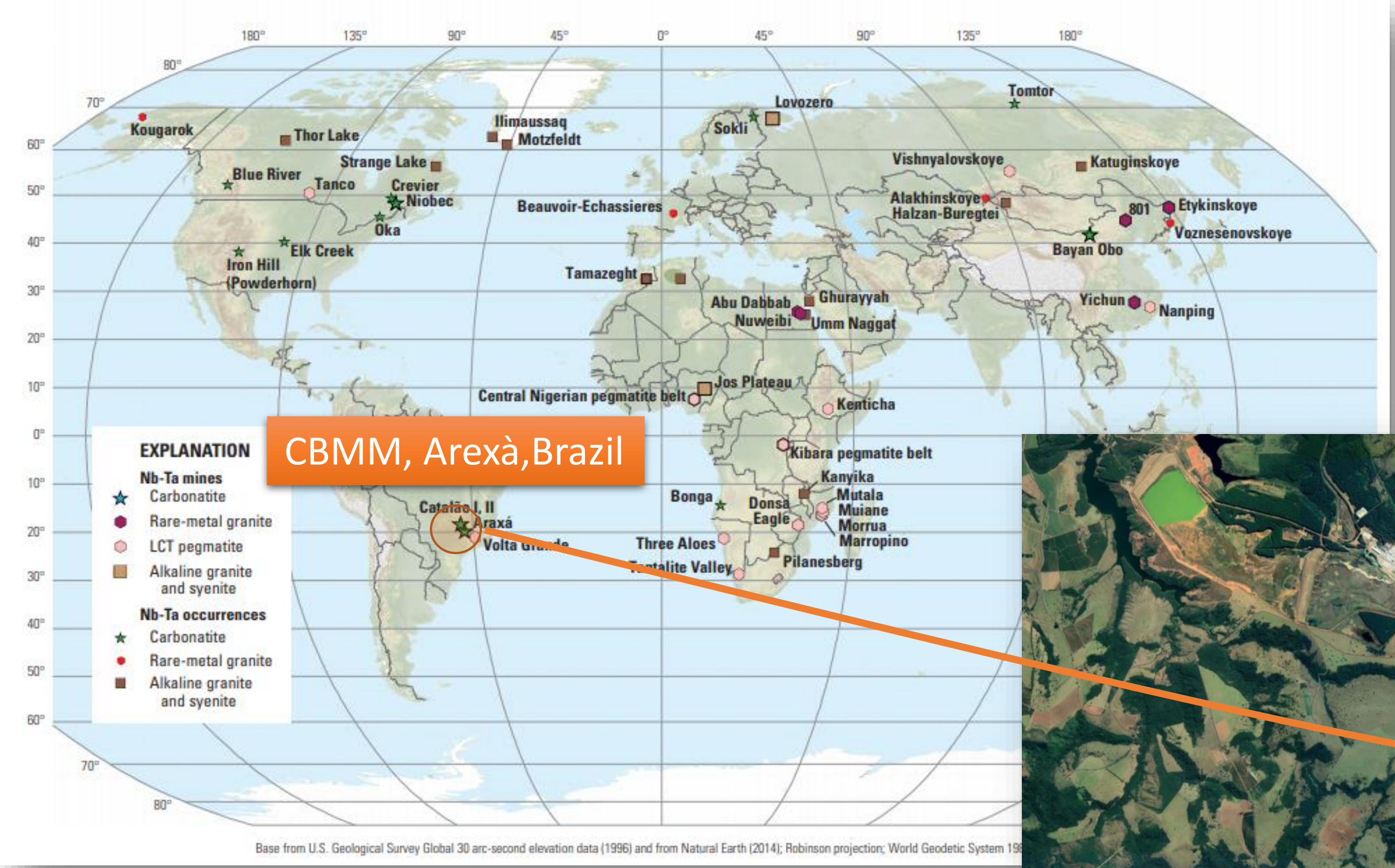
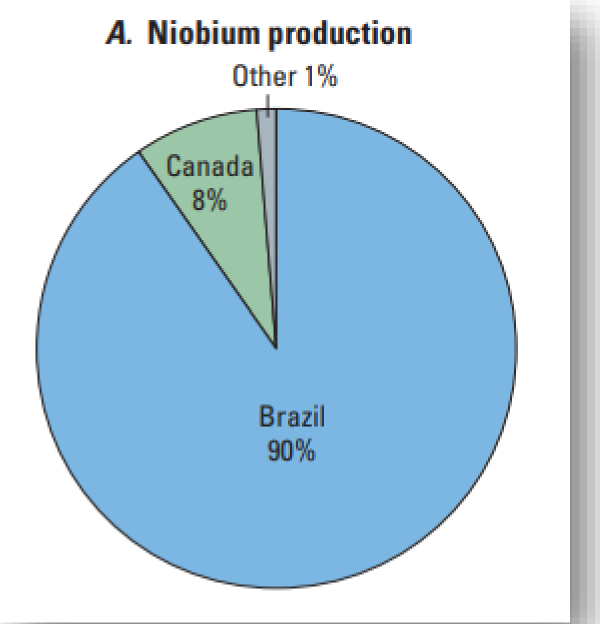
Niobium Production



Base from U.S. Geological Survey Global 30 arc-second elevation data (1996) and from Natural Earth (2014); Robinson projection; World Geodetic System 19



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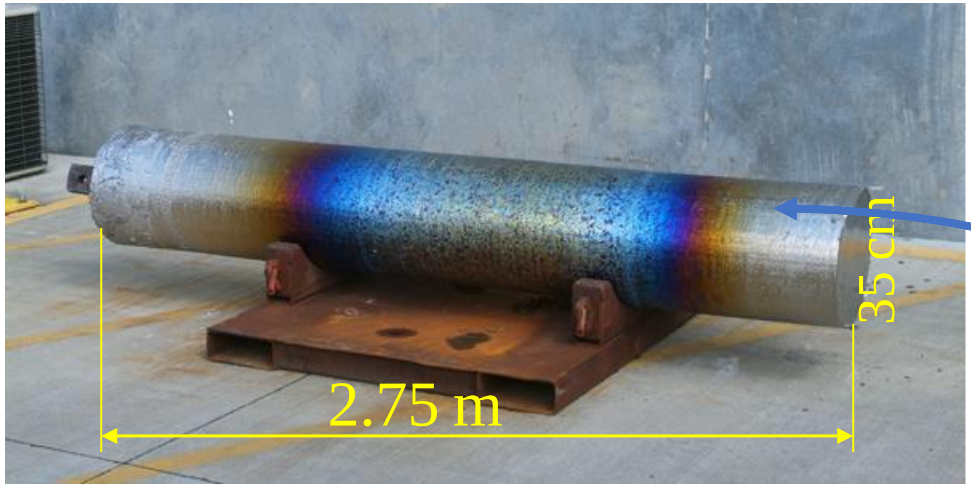
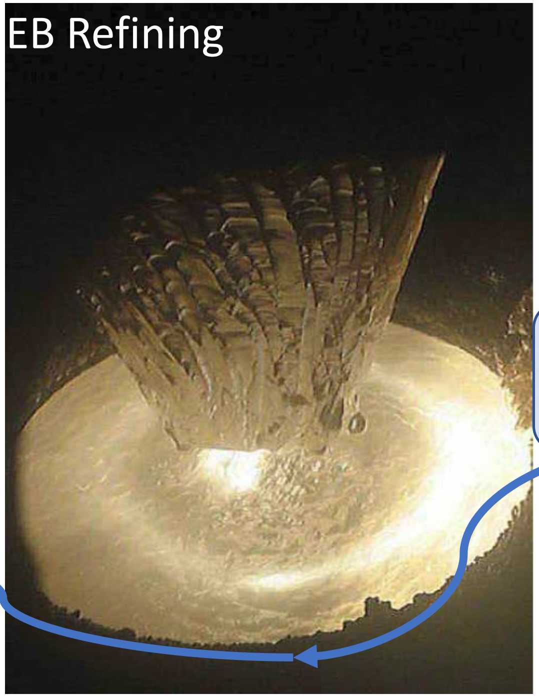
Niobium Production

- The leading use of niobium (about 75 %) is in the production of **high strength steel** alloys used in pipelines, transportation infrastructure, and structural applications.
- Niobium is primarily **derived from the complex oxide minerals** of the pyrochlore group and carbonatites, **usually together with Tantalum**
- The **estimated global reserves** appear more than sufficient to meet global demand for the foreseeable future, possibly the next **500 years**

Niobium Prodution Process

- The ore (Pyroclore in this case) is treated and **refined** at different stages untill it reaches the **purity necessary** for the Electron Beam Refining.
- The **Electron Beam** process **reduces the impurites** present in the incoming Niobium





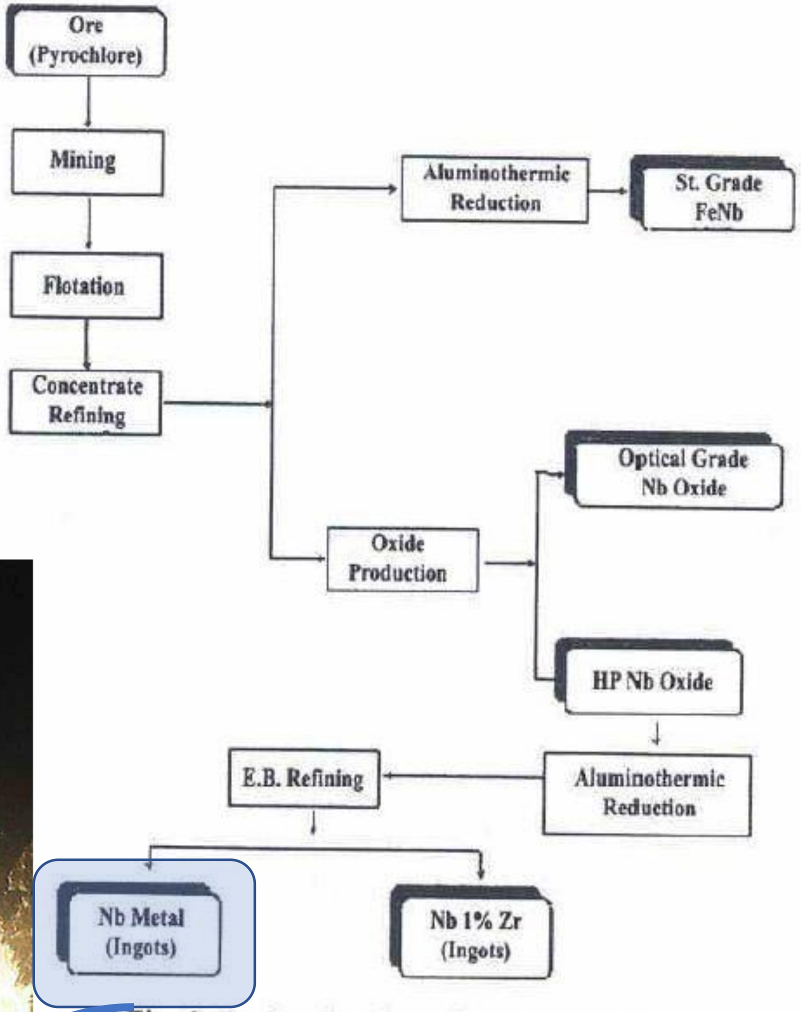
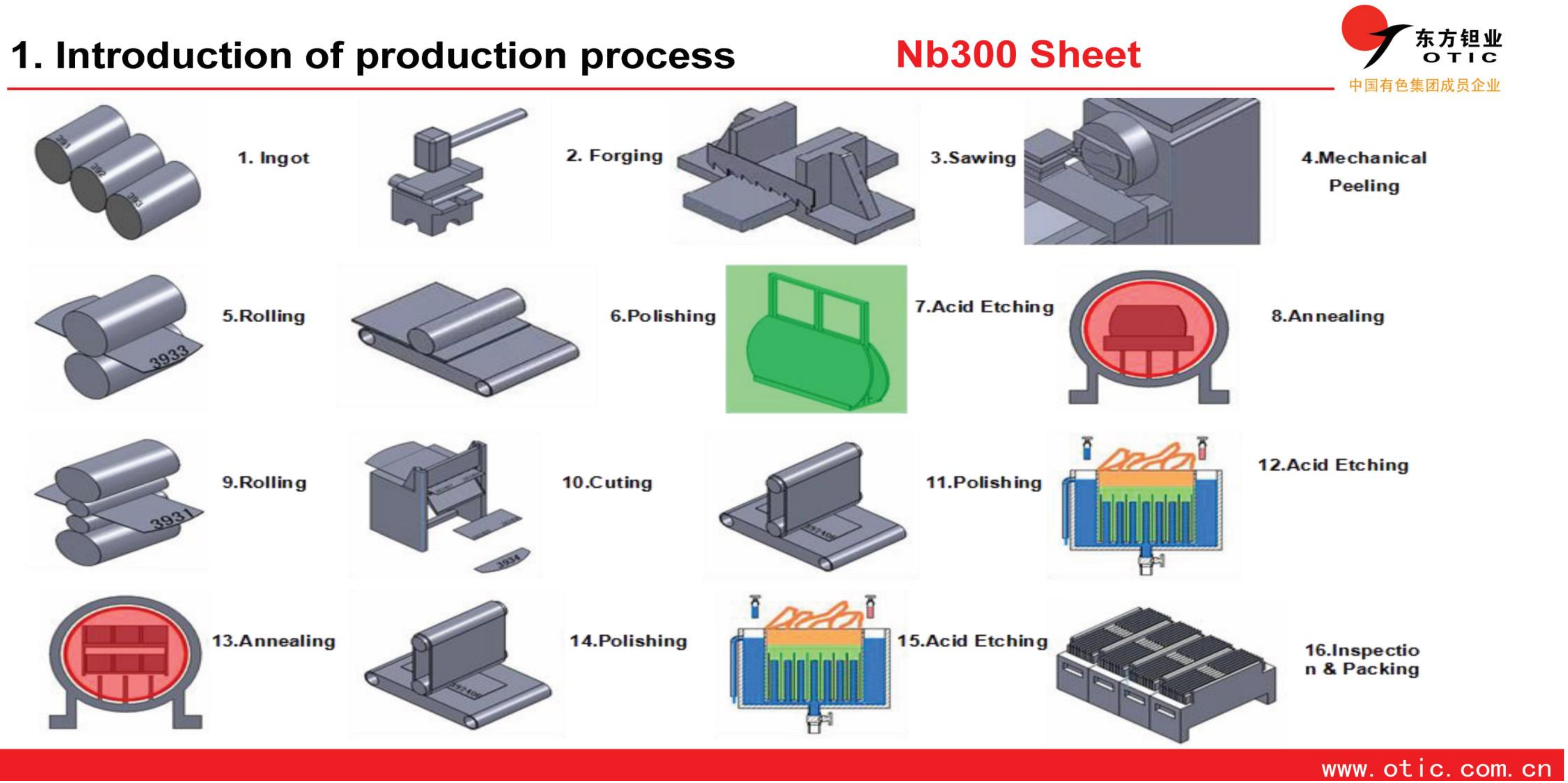


Fig. 3: Production flow chart at CBMM.

Niobium sheets production



Nb Technical Specifications (typical)

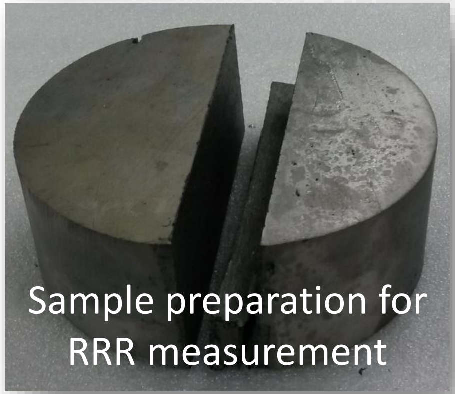
Concentration of impurities in wt.ppm				Mechanical properties	
Ta*	≤ 500	H*	≤ 2	Yield strength**, $\sigma_{0,2}$	$50<\sigma_{0,2}<100$ N/mm² (Mpa)
W*	≤ 70	N*	≤ 10	Tensile strength**	> 100 N/mm² (Mpa)
Ti*	≤ 50	O*	≤ 10	Elongation at break**	30 %
Fe*	≤ 30	C*	≤ 10	Vickers hardness** HV 10	≤ 60
Mo*	≤ 50	RRR*	≥ 300	Absence of foreign material inclusions*	Proven by scanning
Ni*	≤ 30	Recrystal. degree. Grain size* ,** ?	≈ 50 µm	Texture *, ** ?	

* - relevant for performance

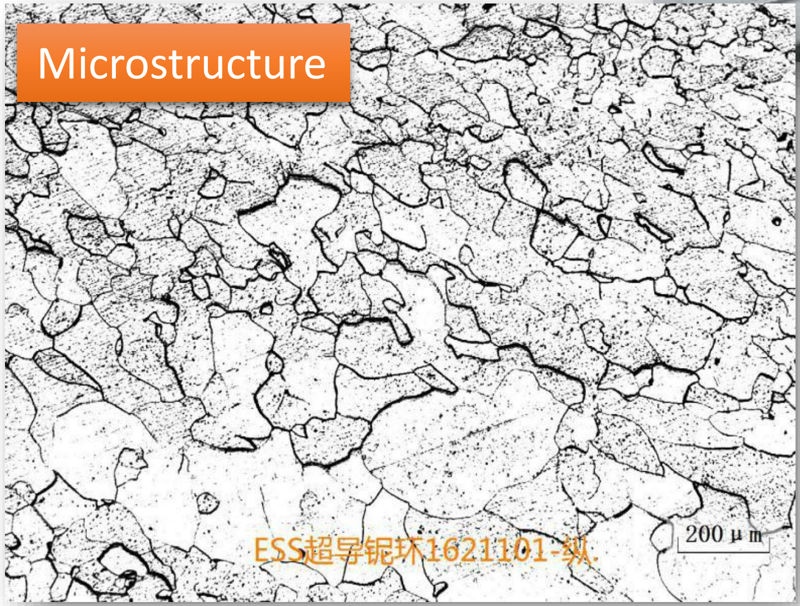
** - relevant for successful fabrication

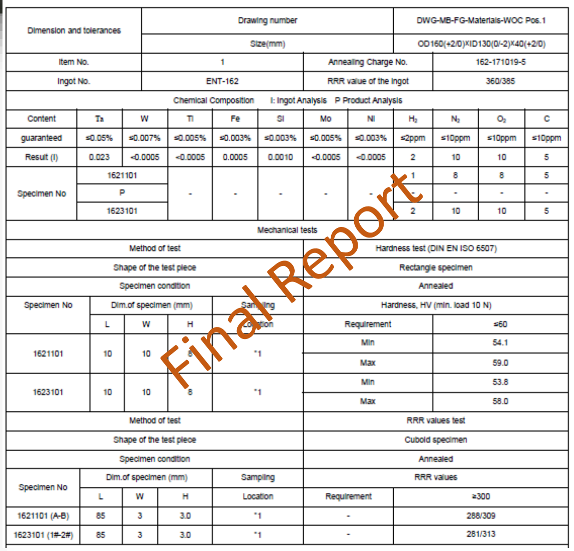
Nb sheets QC at productor premises

- **Ingot**
  - RRR
  - Mechanical Properties
  - Gas analysis
  - Crystallography
- **All sheets**
  - Visual Inspection
    - Defects (i.e. scratches)
    - Delamination
  - “Rust” test
    - Coarse check for Fe inclusions



Sample preparation for RRR measurement





Dimension and tolerances	Drawing number		DWG-MB-FG-Materials-WOC Pos.1
	Size(mm)		OD160(+2/0)×ID130(0/-2)×40(+2/0)
Item No.	1	Annealing Charge No.	162-171019-5
Ingot No.	ENT-162	RRR value of the ingot	360/385

Chemical Composition I: Ingot Analysis P Product Analysis

Content		Ta	W	Ti	Fe	Si	Mo	Ni	H_2	N_2	O_2	C
guaranteed		≤0.05%	≤0.007%	≤0.005%	≤0.003%	≤0.003%	≤0.005%	≤0.003%	≤2ppm	≤10ppm	≤10ppm	≤10ppm
Result (I)		0.023	<0.0005	<0.0005	0.0005	0.0010	<0.0005	<0.0005	2	10	10	5
Specimen No	1621101			-	-	-	-	-	1	8	8	5
	P								-	-	-	-
	1623101								2	10	10	5

Mechanical tests

Method of test					Hardness test (DIN EN ISO 6507)	
Shape of the test piece					Rectangle specimen	
Specimen condition					Annealed	
Specimen No	Dim.of specimen (mm)			Sampling	Hardness, HV (min. load 10 N)	
	L	W	H	Location	Requirement	≤60
1621101	10	10	8	*1	Min	54.1
					Max	59.0
1623101	10	10	8	*1	Min	53.8
					Max	58.0
Method of test					RRR values test	
Shape of the test piece					Cuboid specimen	
Specimen condition					Annealed	
Specimen No	Dim.of specimen (mm)			Sampling	RRR values	
	L	W	H	Location	Requirement	≥300
1621101 (A-B)	85	3	3.0	*1	-	288/309
1623101 (1#-2#)	85	3	3.0	*1	-	281/313

Residual Resistance Ratio (RRR)

- Electrical resistivity of metals at low temperatures is related to the impurity concentrations. The residual resistivity at T = 0 K is caused mainly by scattering of electrons by impurities.

Residual Resistivity Ratio

$$RRR = \frac{\rho(295\ K)}{\rho(4.2\ K)}$$

- **RRR** depends on **impurity content** in the material (typical RRR for cavity is around 300)
- **RRR** is linked to the Nb **thermal conductivity** by

$$\lambda(4.2\ K) \approx 0.25\ RRR\ [\frac{W}{m\ K}]$$

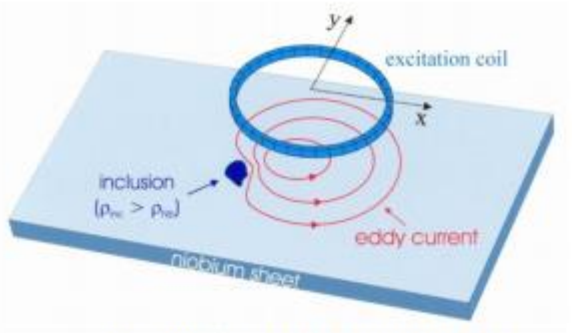
Nb Eddy Current Scanning

When an **AC current flows** in a coil in close **proximity** to a **conducting surface** the magnetic field of the coil will **induce circulating (eddy) currents** in that surface.

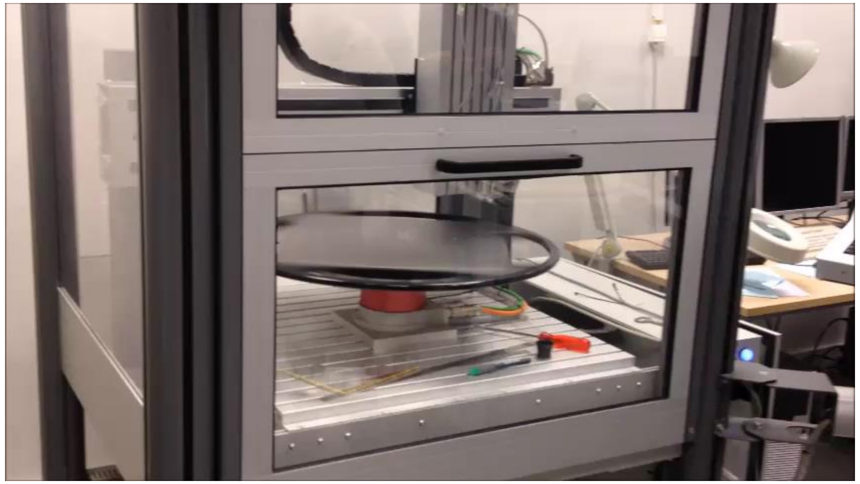
The **magnitude and phase** of the eddy currents will **affect the loading** on the coil and thus its impedance. If there is **a deep crack in the surface** immediately underneath the coil, it will **interrupt or reduce the eddy current** flow, thus decreasing the loading on the coil and increasing its effective impedance.

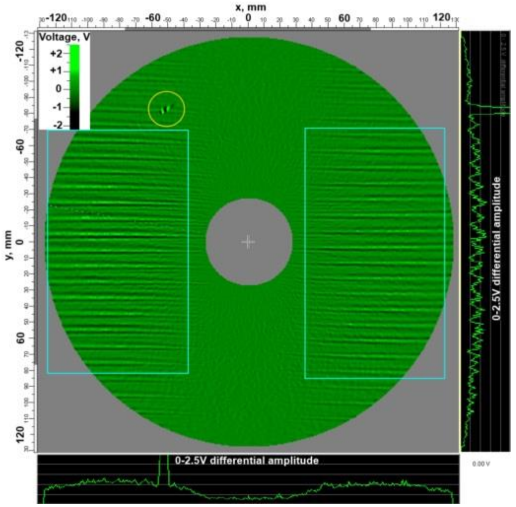
The operating frequency is between 100 kHz to few MHz and can span from the surface down into the materials for **some hundreds of microns**.

DESY Eddy Current principle

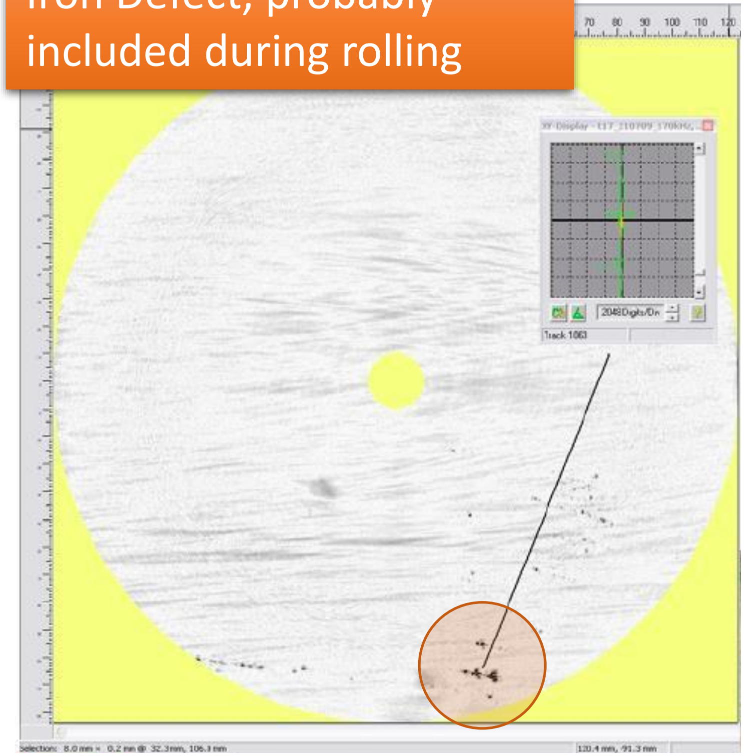


Principle of eddy current measurement





Iron Defect, probably included during rolling



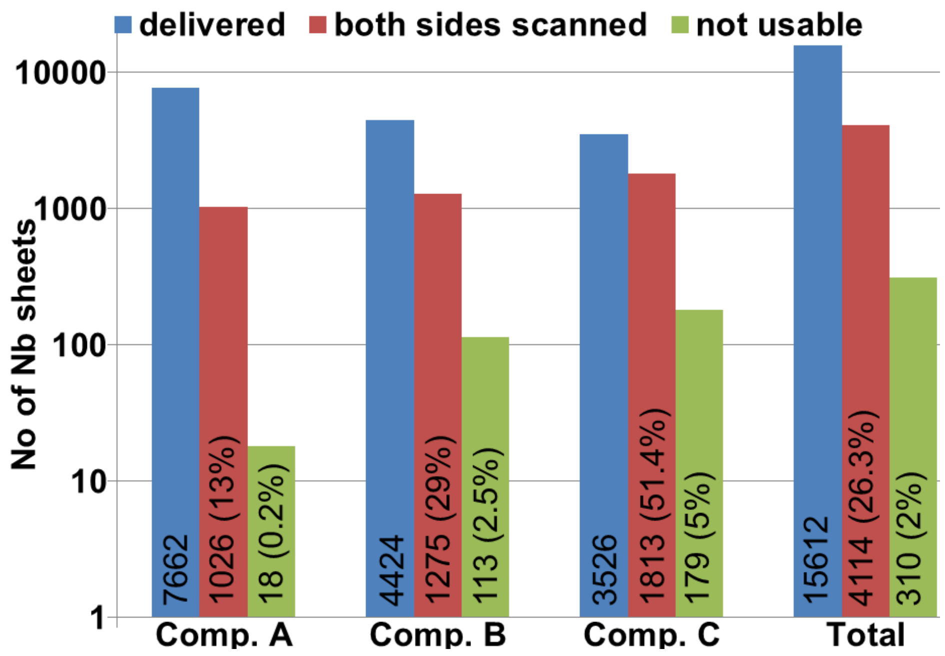
Eddy Current results

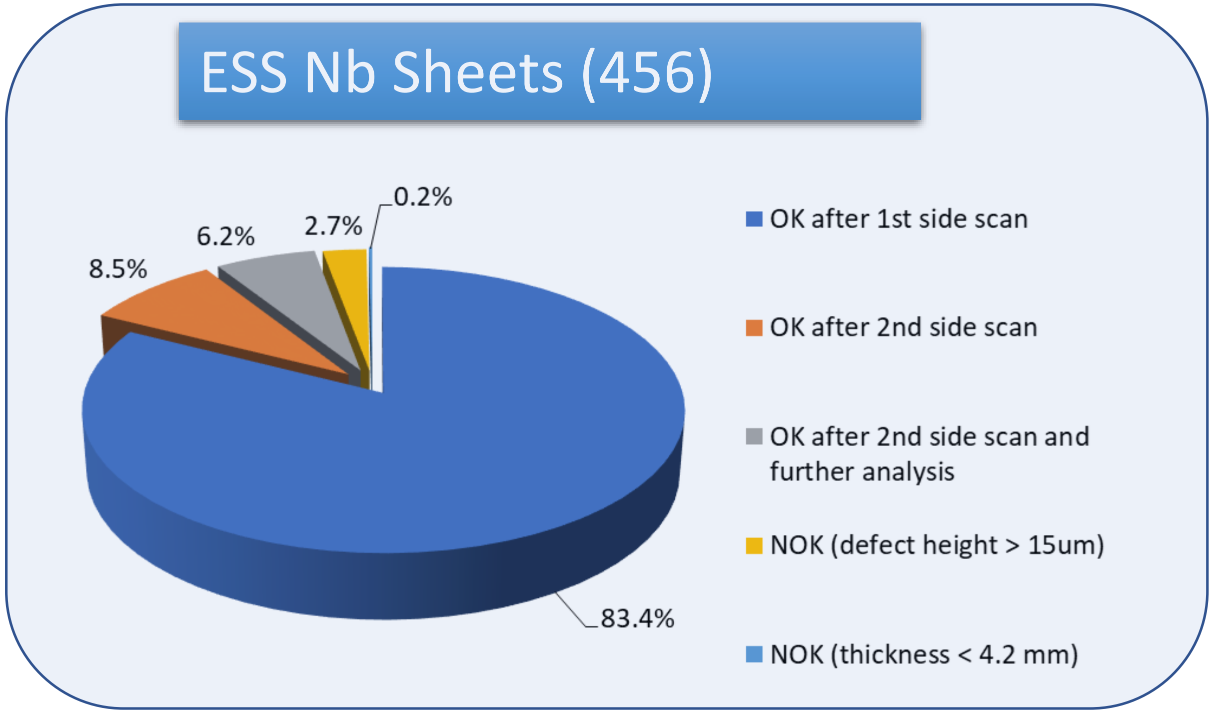
European XFEL

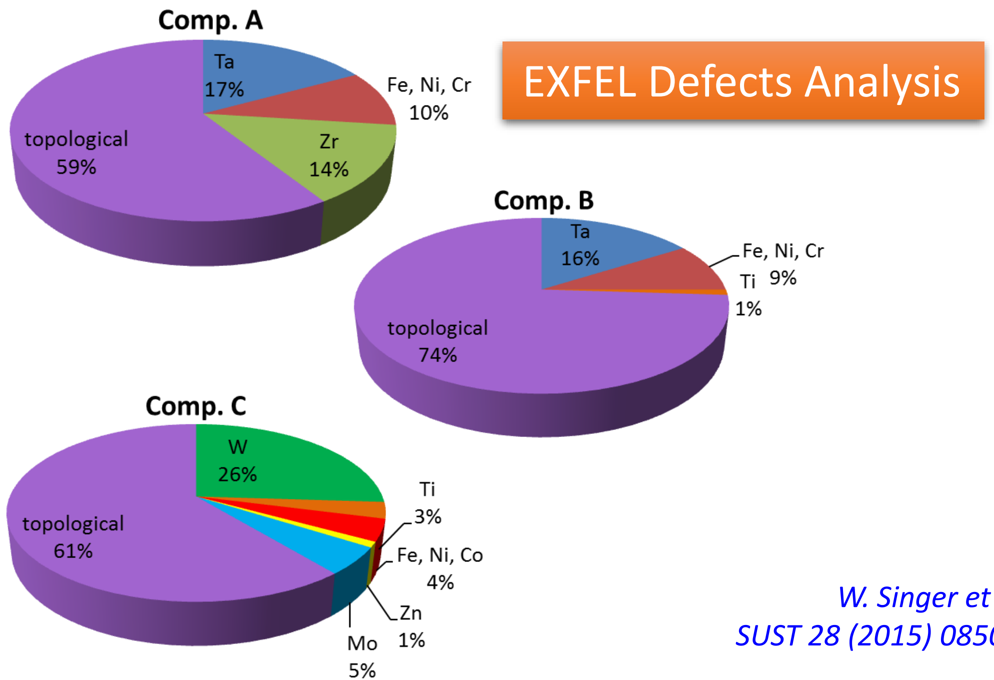
On the 15612 sheets

26% scanned on both sides

2% rejected (foreign material > 100 μm, delamination, etc.)



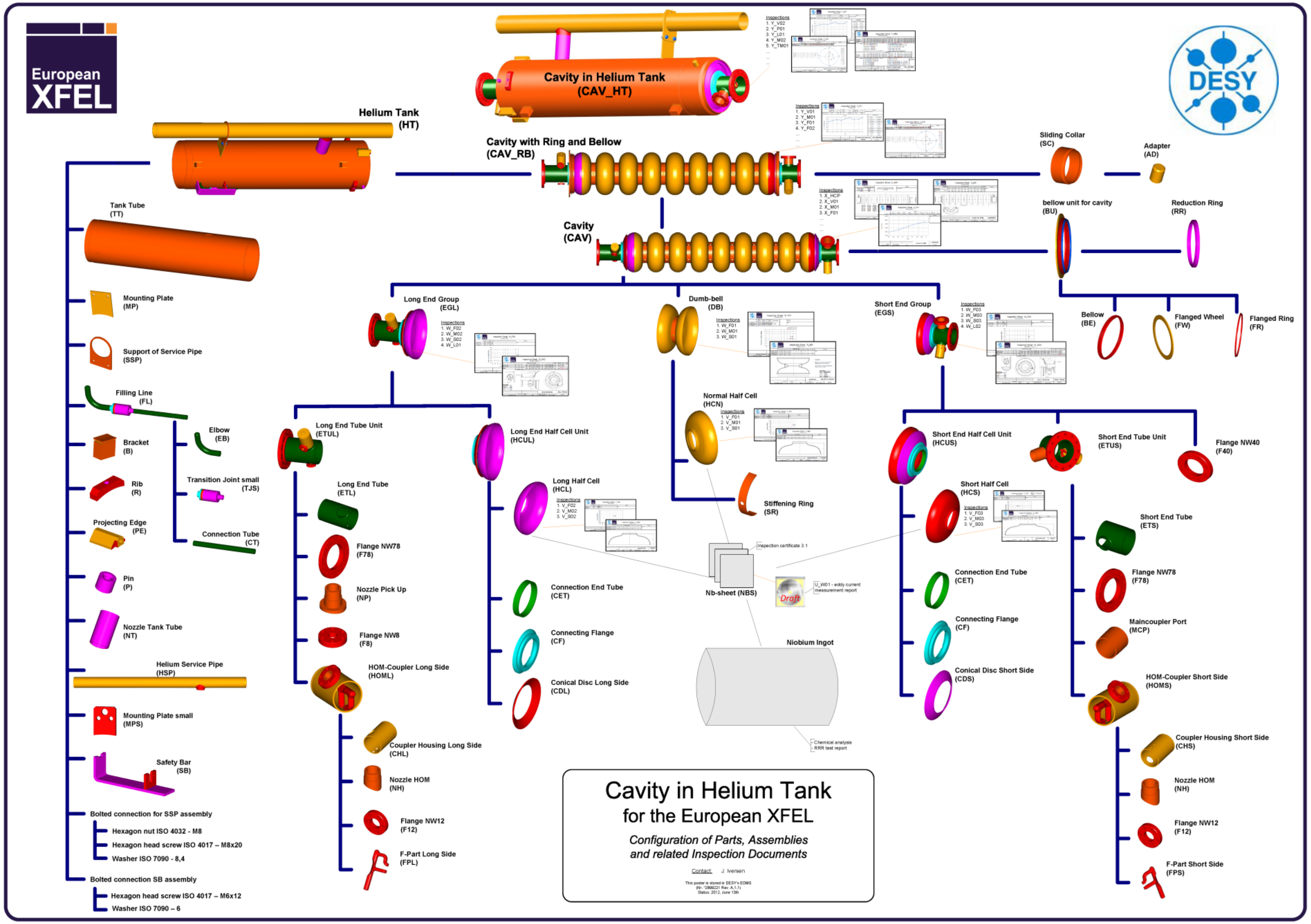




W. Singer et al., SUST 28 (2015) 085014

From sheets to cavity

Cavity components



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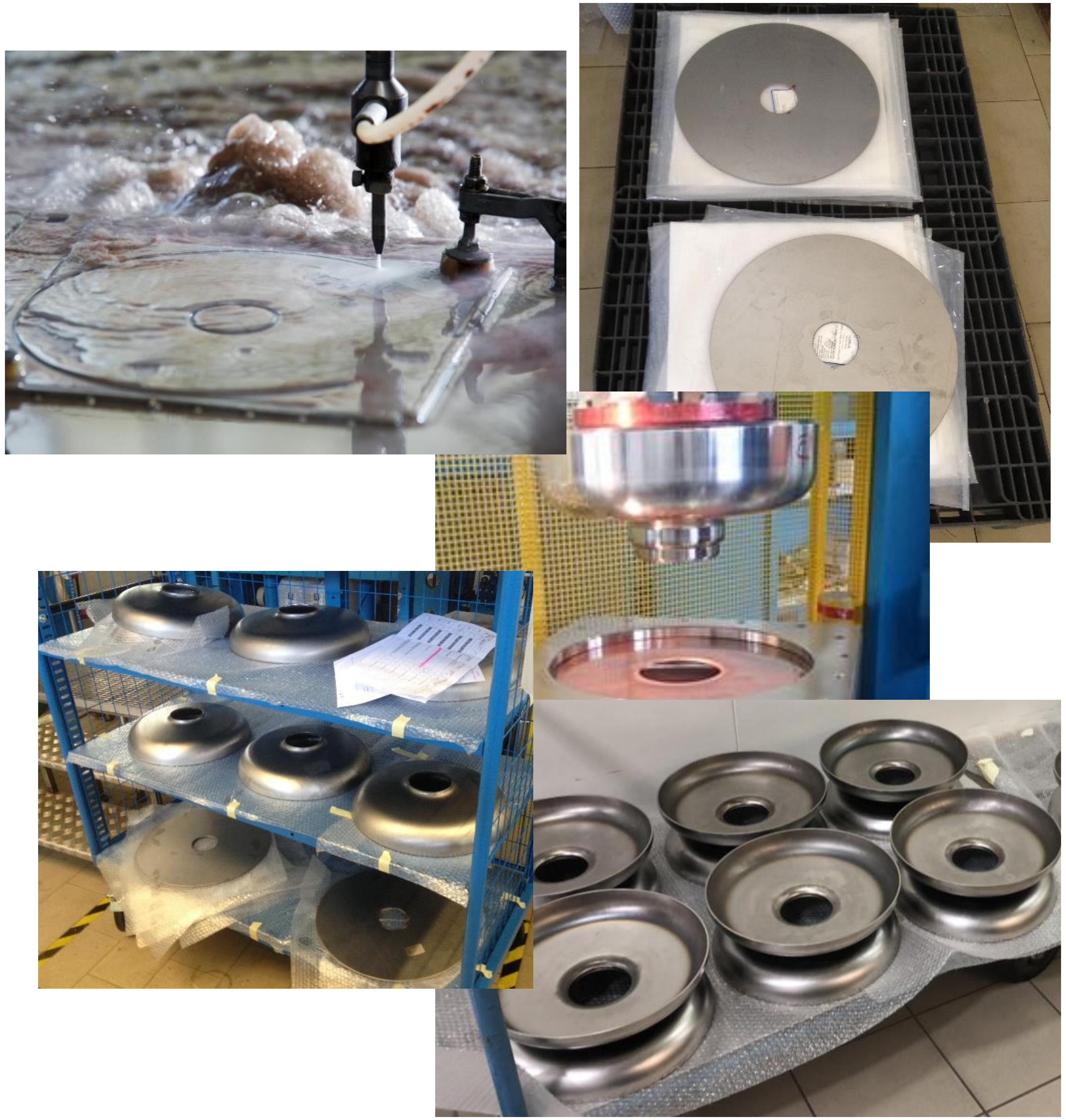
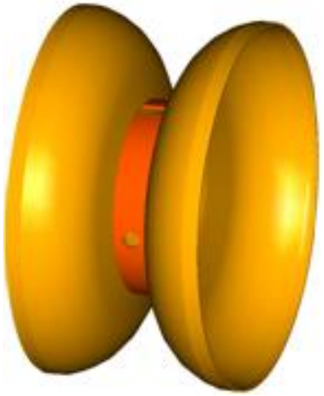
Dumb-bell Fabrication (an example)





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

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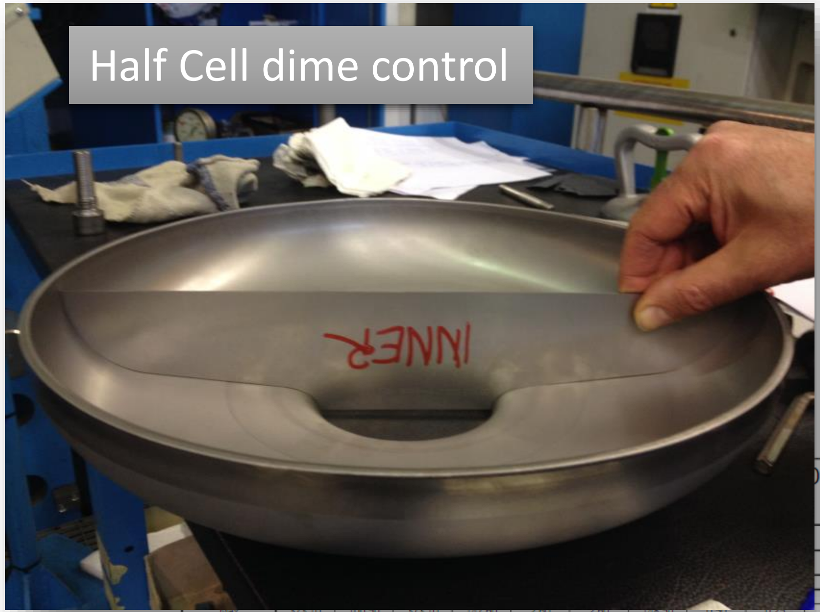
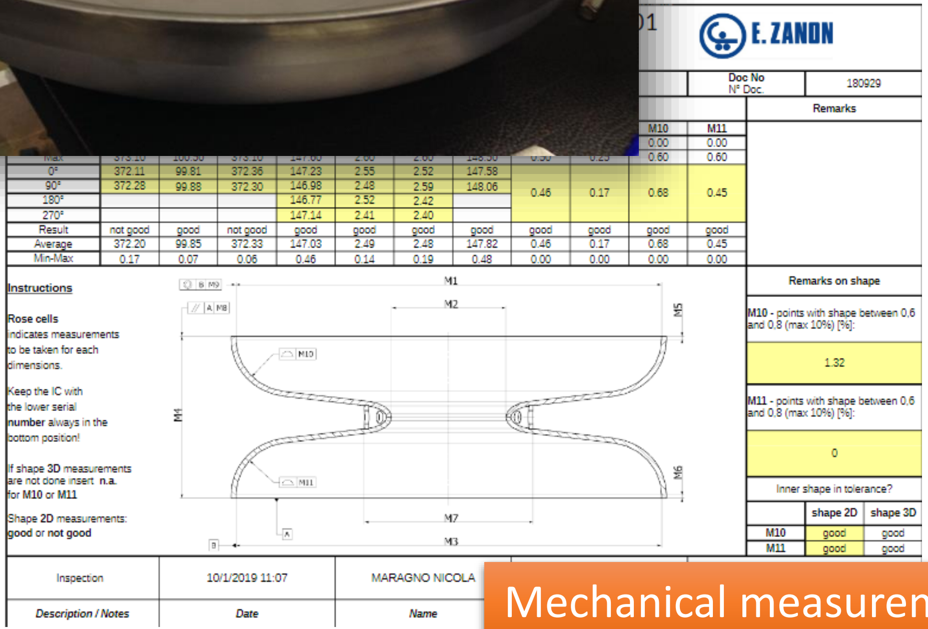
Mechanical QC



Half Cell dime control

3D profile



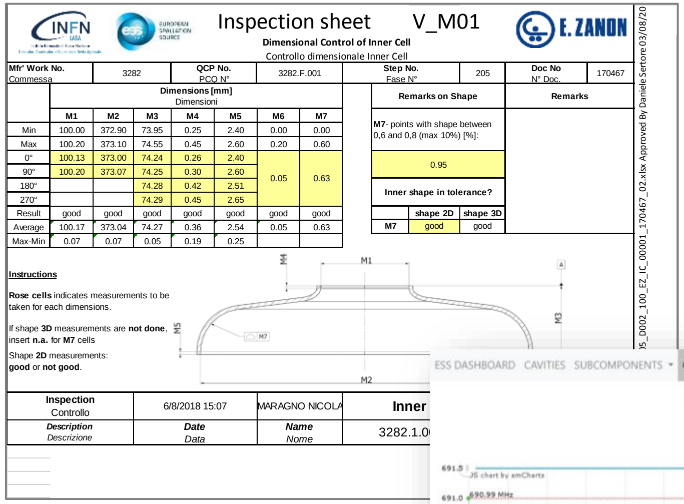
Mechanical measurements

Visual Inspection

Frequency QC

Measure frequency and length to determine how much to **trim** at the equator to obtain **target frequency and length** of the cavity fully welded

Co.Co.De for
HC and DB RF measurements



Inspection sheet V_M01

Dimensional Control of Inner Cell
Controllo dimensionale Inner Cell

Mfr' Work No. Commessa	3282	QCP No. PCO N°	3282.F.001	Step No. Fase N°	205	Doc No N° Doc.	170467

	Dimensions [mm] Dimensioni						
	M1	M2	M3	M4	M5	M6	M7
Min	100.00	372.90	73.95	0.25	2.40	0.00	0.00
Max	100.20	373.10	74.55	0.45	2.60	0.20	0.60
0°	100.13	373.00	74.24	0.26	2.40	0.05	0.63
90°	100.20	373.07	74.25	0.30	2.60		
180°			74.28	0.42	2.51		
270°			74.29	0.45	2.65		
Result	good	good	good	good	good	good	good
Average	100.17	373.04	74.27	0.36	2.54	0.05	0.63
Max-Min	0.07	0.07	0.05	0.19	0.25		

Remarks on Shape			Remarks
M7- points with shape between 0,6 and 0,8 (max 10%) [%]:			
0.95			
Inner shape in tolerance?			
	shape 2D	shape 3D	
M7	good	good	

Instructions

Rose cells indicates measurements to be taken for each dimensions.

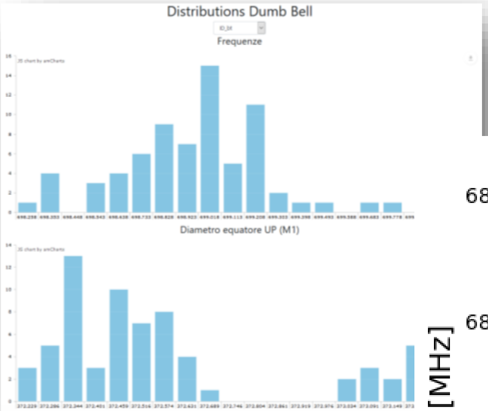
If shape **3D** measurements are **not done**, insert **n.a.** for **M7** cells

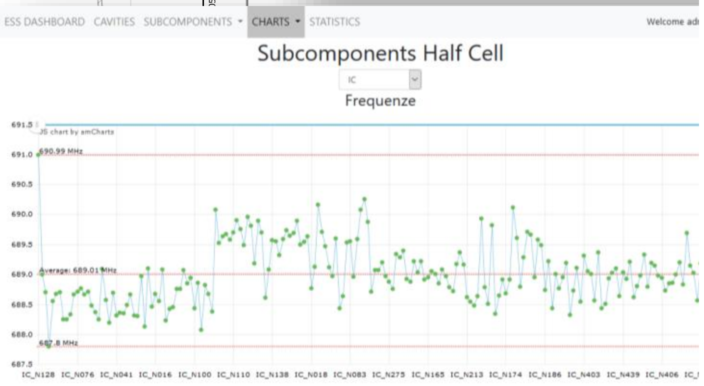
Shape **2D** measurements:
good or **not good**.

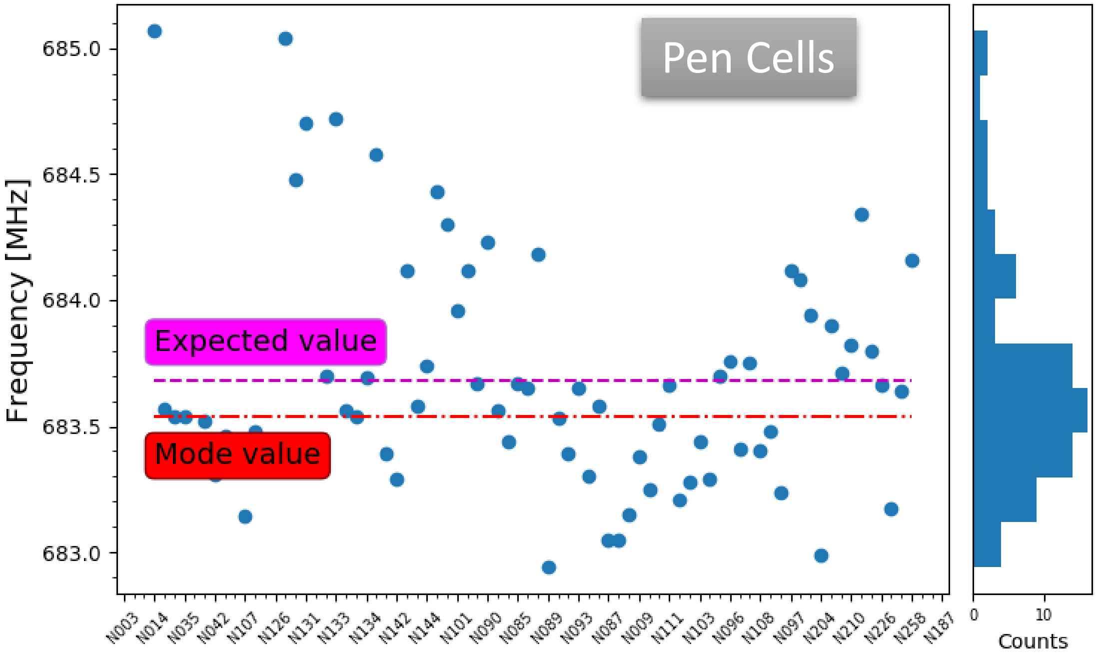
M1 M2 M3 M4 M5 M7 A

Inspection Controllo	6/8/2018 15:07	MARAGNO NICOLA	Inner
Description *Descrizione*	*Date* *Data*	*Name* *Nome*	3282.1.0

05_D002_100_EZ_IC_00001_170467_02.xlsx Approved By Daniele Sertore 03/08/20







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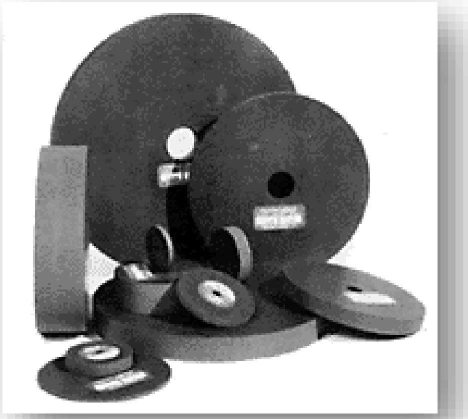
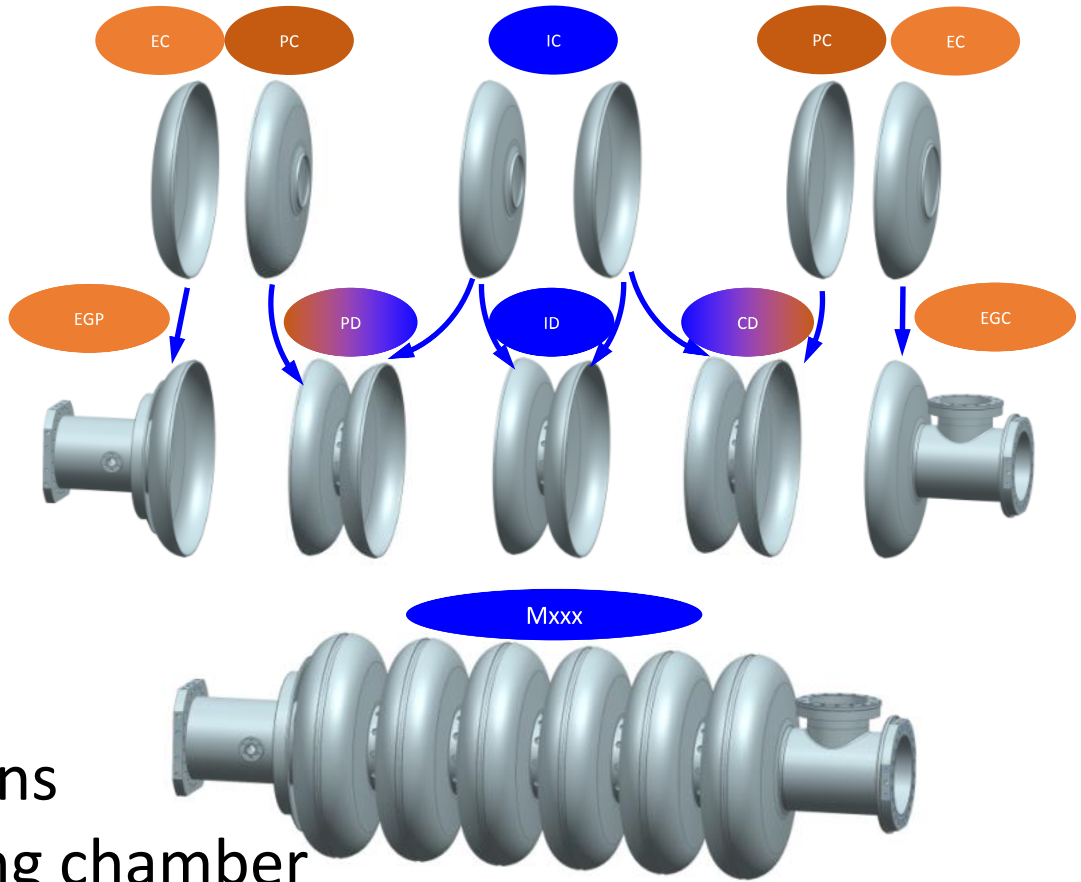
Mechanical Grinding

- **Mechanical grinding** of visible **local defects** (deeper than 15 μm for EXFEL) with aluminum oxide grinding discs or rubberized abrasive (CRATEX®)



Rubberized abrasive

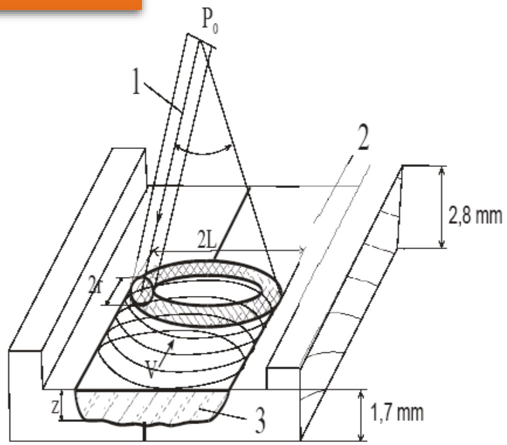
Cavity parts EB welding



- Degreasing and rinsing of parts
- Drying under clean condition
- Chemical etching at the welding area (equator)
- Careful and intensive rinsing with Ultra Pure Water
- Dry under clean conditions
- Install parts to fixture under clean conditions
- Install parts into Electron Beam (EB) welding chamber (**no contamination on the weld area allowed**)
- Pump down to vacuum in the EBW chamber in the 10^{-5} mbar range
- Welding and cool down of Nb to T < 150 °C, then venting with N_2
- Leak check of welds

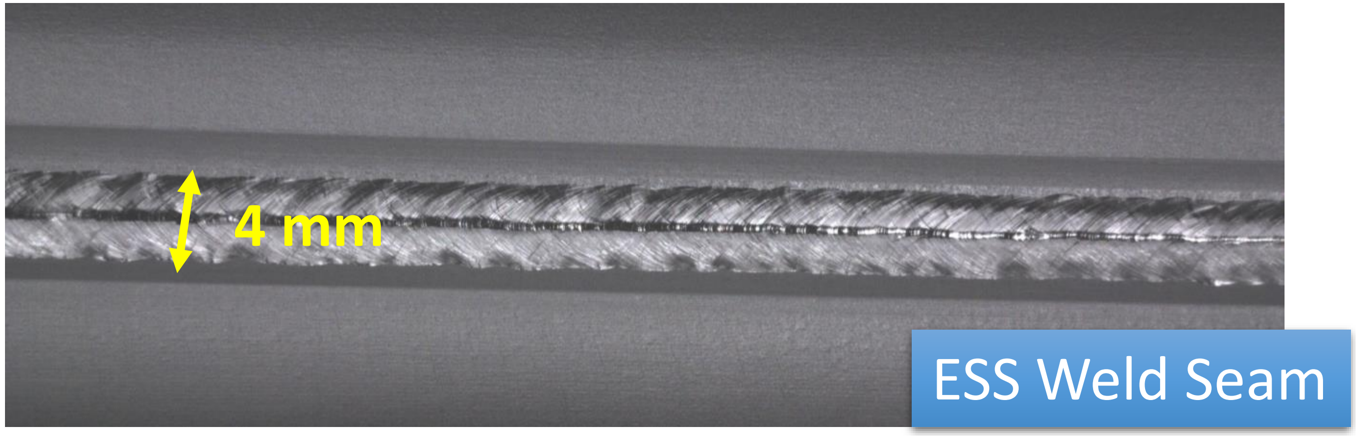
Electron Beam Welding

- **Welding under good vacuum**, 10^{-5} mbar range
- Broad welding seam
  – Operate with defocussed beam
  – Smooth underbead
- **Overlap at end of welding** to avoid accumulation of impurities
- **Wait to cool down before opening chamber**



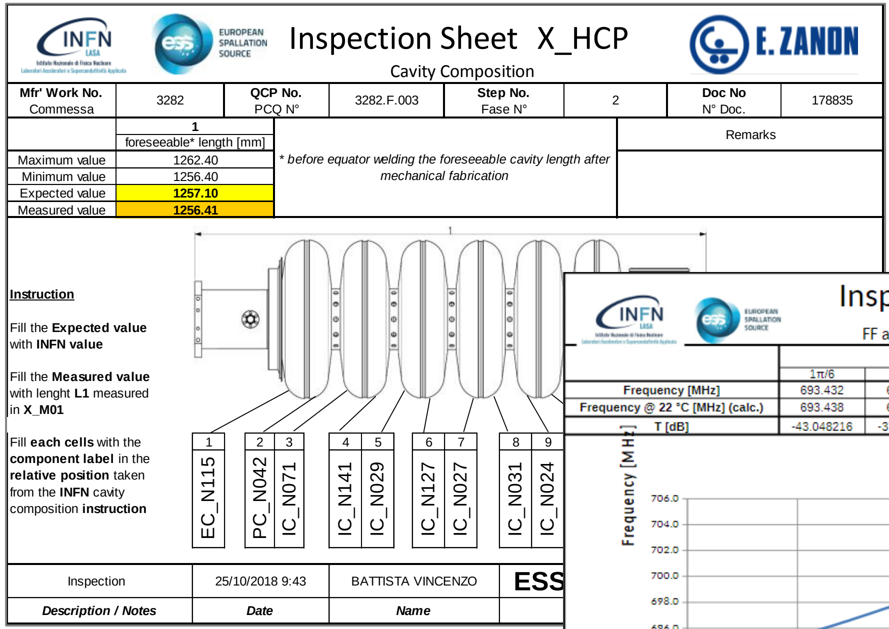
Welding Scheme (circular raster)

1. Electron beam (P_0-power of the beam, r-spot radius on the surface, L-scanning amplitude, V-velocity of the beam movement)
2. Nb sheet
3. melting zone (z-depth of the melting zone)



Mechanical and frequency QC

- After EBW, the cavity is mechanical measured, inner inspected and the frequency is controlled



INFN LASA · ESS European Spallation Source · E. ZANON

Inspection Sheet X_HCP

Cavity Composition

Mfr' Work No. Commessa	3282	QCP No. PCQ N°	3282.F.003	Step No. Fase N°	2	Doc No N° Doc.	178835

	1 foreseeable* length [mm]
Maximum value	1262.40
Minimum value	1256.40
Expected value	1257.10
Measured value	1256.41

* before equator welding the foreseeable cavity length after mechanical fabrication

Remarks

Instruction

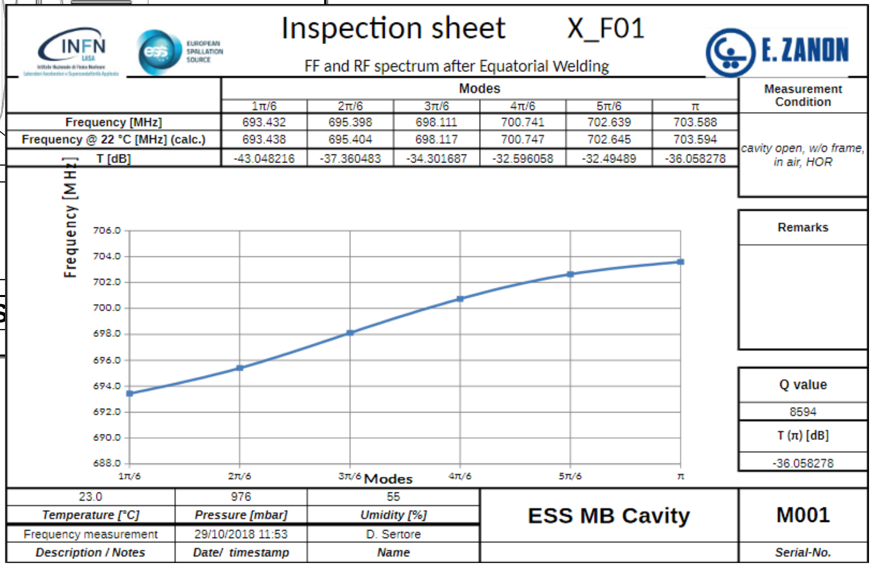
Fill the **Expected value** with **INFN value**

Fill the **Measured value** with lenght **L1** measured in **X_M01**

Fill **each cells** with the **component label** in the **relative position** taken from the **INFN** cavity composition **instruction**

1	2	3	4	5	6	7	8	9
EC_N115	PC_N042	IC_N071	IC_N141	IC_N029	IC_N127	IC_N027	IC_N031	IC_N024

Inspection	25/10/2018 9:43	BATTISTA VINCENZO	ESS
Description / Notes	**Date**	**Name**	



INFN LASA · ESS European Spallation Source · E. ZANON

Inspection sheet X_F01

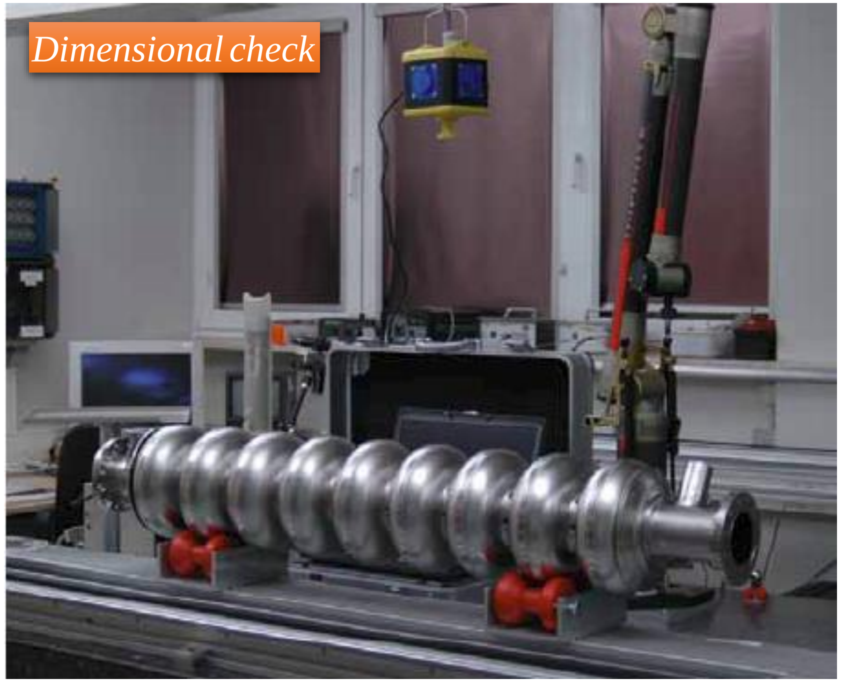
FF and RF spectrum after Equatorial Welding

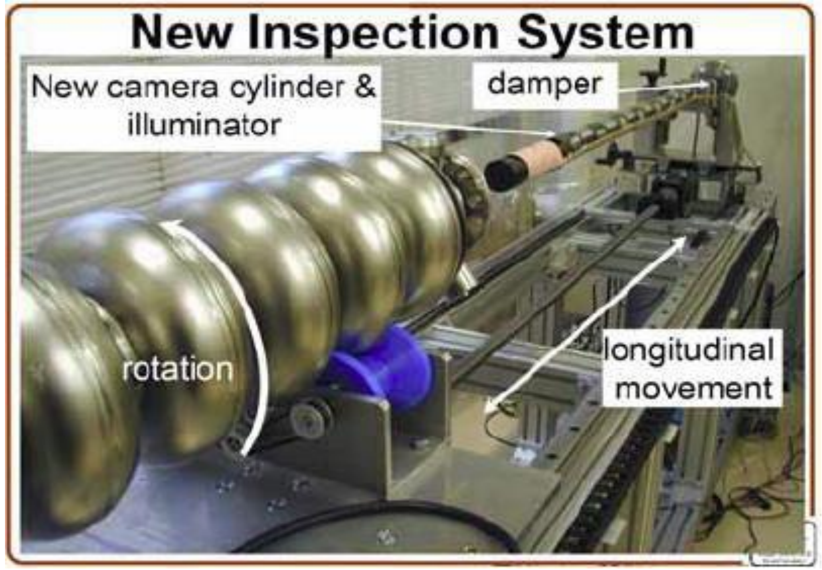
	Modes						Measurement Condition
	1π/6	2π/6	3π/6	4π/6	5π/6	π	
Frequency [MHz]	693.432	695.398	698.111	700.741	702.639	703.588	cavity open, w/o frame, in air, HOR
Frequency @ 22 °C [MHz] (calc.)	693.438	695.404	698.117	700.747	702.645	703.594	
T [dB]	-43.048216	-37.360483	-34.301687	-32.596058	-32.49489	-36.058278	

Remarks

Q value
8594
T (π) [dB]
-36.058278

23.0	976	55	ESS MB Cavity	M001
Temperature [°C]	*Pressure [mbar]*	*Umidity [%]*		
Frequency measurement	29/10/2018 11:53	D. Sertore		
Description / Notes	**Date/ timestamp**	**Name**		**Serial-No.**





Cavity treatment

Overview

Step	WHY	COMMENTS
Forming		
EB Welding	Clean welding	Nb = getter material. If RRR/ 10 @ welding => Q_0/10
Ti purification	RRR enhancement	RRR 300-400 now commercially available
Chemical etching 100-200 µm (BCP / EP)	Remove contamination and damage layer	Limitation : BCP ~ 30MV/m; EP => >40 mV/m but lack of reproducibility
Annealing 800°C, 2h (or 600°C, 10h)	Get rid of hydrogen	Source of H: wet processes H segregates near surface in form of hydrides (= bad SC)
Chemical etching 5-20 µm	Remove diffusion layer (O, C, N)	Diffusion layer < ~1µm in bulk, a little higher at Grain Boundaries
Specific rinsing	e.g. remove S particles due to EP	Under evaluation HF, H_2O_2, ethanol, degreasing,…
High pressure rinsing (HPR)	Get rid of dust particles	Not always enough (recontamination during assembly)
Assembling	Ancillaries : antennas, couplers , vacuum ports…	**In clean room, but recontamination still possible**
Baking, 120°C, 48h	Decrease high field losses (Q-drop)	Unknown mechanism, first 10 nm of the surface in concern.
Post processing	Get rid of "re-contamination" ?	Under evaluation: dry ice cleaning, plasma
Test RF	Cavity's performance	First naked cavity in vertical cryostat, then dressed in horizontal cryostat/ accelerating facility
He processing, HPP	Decrease field emission	RF power with/ without He to destroy field emitters (dust particles) NB field emission : principal practical problem in accelerators

A general consideration

Do not make Nb surface worst than before with the next treatment!

- **Do not apply treatments** that affect the Nb surface and **could not be "accepted" by the next step**
- If a **mistake is done**, **go back in the procedure** until the step where contaminant can be removed without contaminating the system
- **Chemical reactions** in many cases can **not be stopped** simply **removing acid (residuals, no cooling, …). Rinsing is needed!**
- **Do not contaminate US bathes with material that can not be diluted**, as for **silicone grease**, oil, etc. Moreover take care of **contaminants** that can **float** over the liquid surface!
- **Wet components are more "sensitive" for collecting particles.**
- Duration limit for a final treated cavity is about 24 hours (XFEL)
  Do not leave open cavities for longer time

Cavity preparation for SRF qualification

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - – 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

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- Clean evacuation
- Low-temperature baking

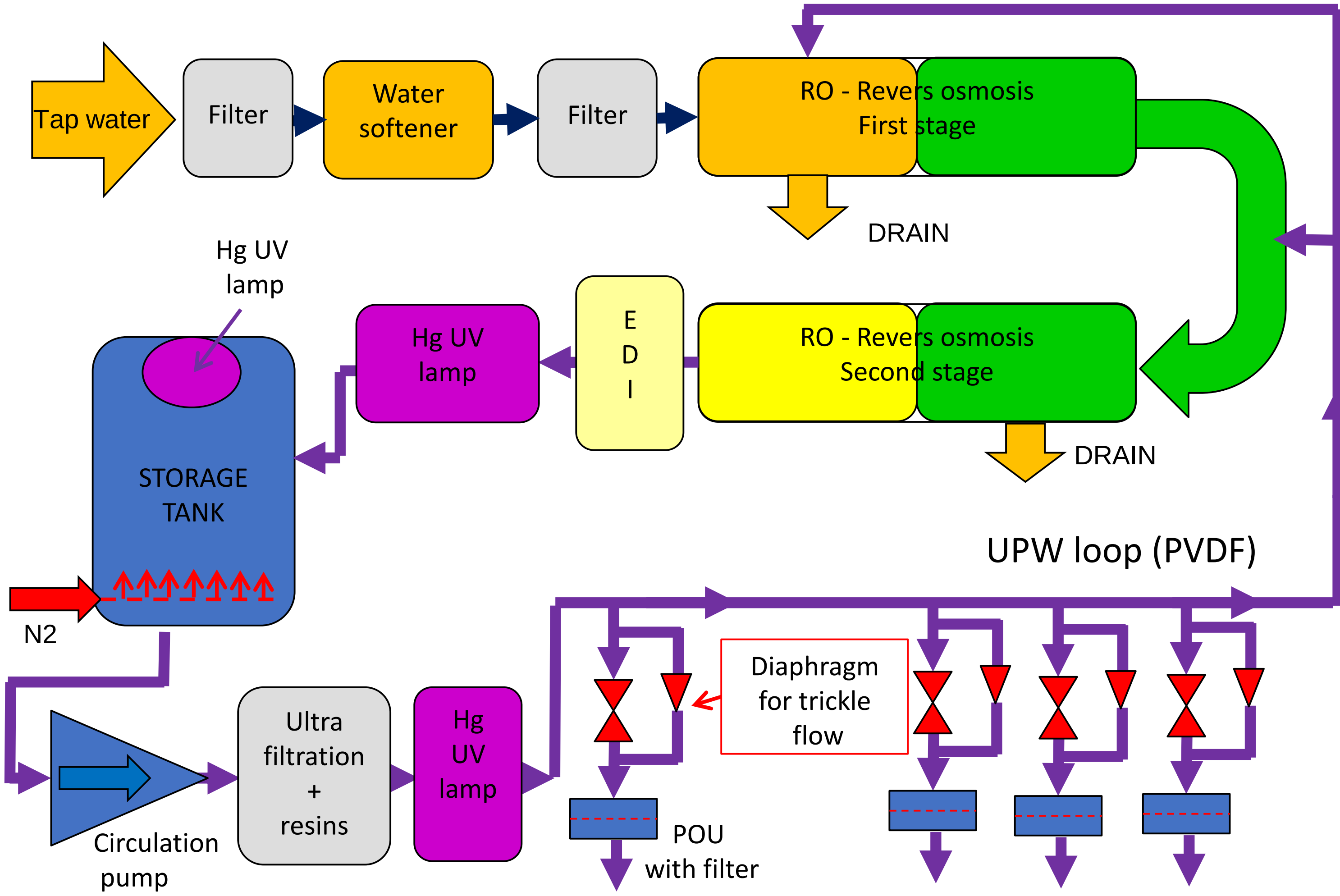
Degreasing and surface preparation

- After mechanical fabrication, **all contaminants** (fingerprints, oil, residuals from machining and QC) **must be removed**, similar to preparation of Ultra High Vacuum components.
- Typical process:
  - **Water rinsing** with specific detergent (Tikopur TR33, Micro -90, Liqui-Nox) usually 1-3%
    - For "dirty" component, alcohol and acetone could be used before
  - Water is usually **Ultra Pure Water** with 18 MΩ cm and filtered below 200 nm
  - Often in **HEPA filter environment**
- For entering **ISO7 clean room**, dishwashers are used for small components and car-wash for large components
- For entering **ISO4 clean room**, UltraSound is mandatory

Ultra Pure Water

Typical UPW plant

- UPW Specifications
  - Resistivity:
    18.2 MΩ cm
  - Total organic carbon (TOC):
    < 5 ppb
  - Particulate counts (> 0.3 μm/l):
    < 10
  - Bacteria counts:
    < 0.1 CFU/100 ml



The water quality is as for the semiconductor industry:
ASTM- D 5127-07 E-1.2

Ultra Pure Water QA

- **Conductivity**: ion concentration, indicates water purity with respect to “salts”, ions, metals, anions, etc.
- **TOC**: Total Organic (oxidizable) Carbon. Organic carbon, but for instance lubricant oils are hard to be oxidized and therefore are not easily measured with TOC monitor.
- **Particle count**: number of particles suspended or in flow in the water. Counting is done using devices (laser counter) similar with the ones used for clean room particle monitoring (for the air).
- **Bacteria**: colonies count. Usually sampling and incubation on Petri capsule.
- **Hydrocarbon** (oils): they **MUST NOT** be present. OFFLINE extraction method (with hexane) and gas chromatographic analysis.

Industrial and laboratory plant

Large system:

- Production: **3000 l/h**
- **Storage: 9000 l**
- Typ. TOC: 3 ppb

Small lab production plant:

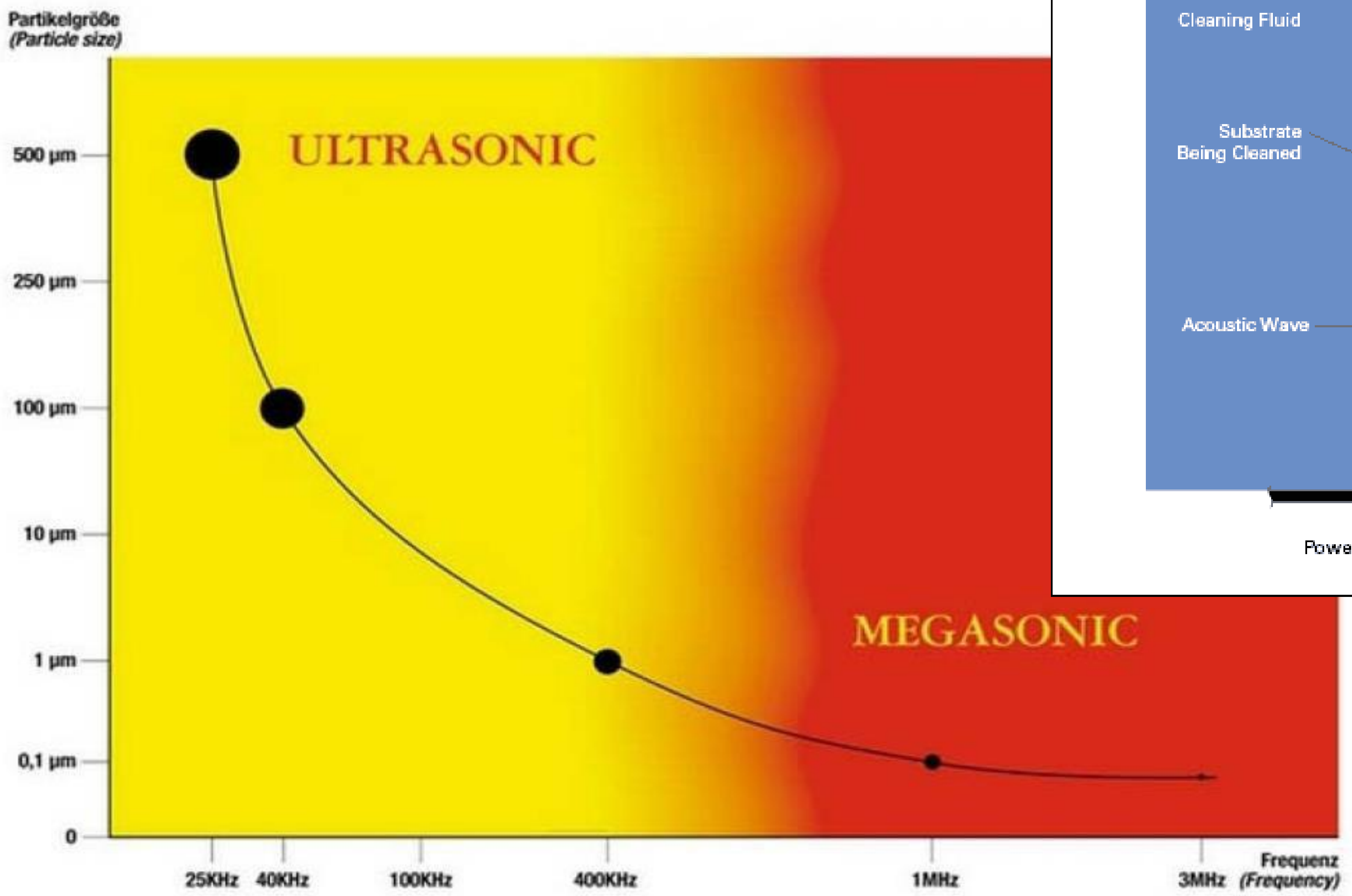
- Production: **170 l/h**
- **Storage: 6000 l**
- Typ. TOC: 3 ppb

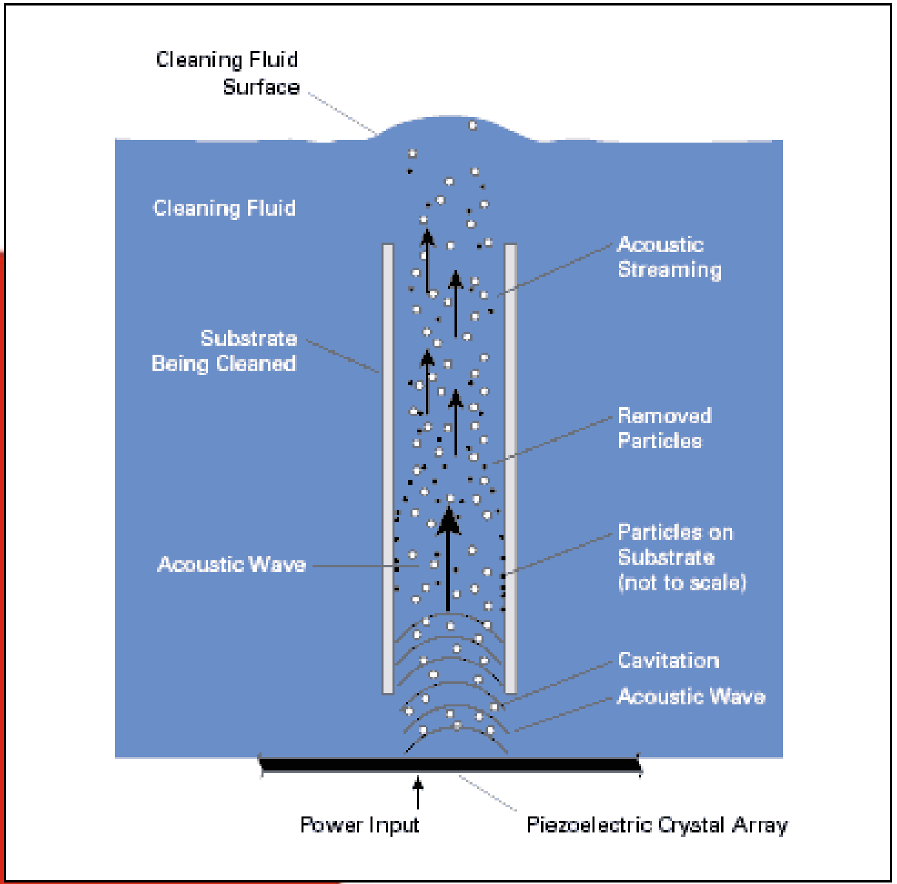
Ultrasonic Cleaning

- Immersion of components in DI water and detergent medium
- **Wave energy forms microscopic bubbles** on component surfaces
- **Bubbles collapse (cavitation)** on surface loosening particulate matter
- **Transducer** provides **high intensity ultrasonic fields** that set up standing waves. Higher frequencies lowers the distance between nodes which produce less dead zones with no cavitation
- Ultrasonic transducers are available in many different wave frequencies from 18 kHz to 120 kHz, **the higher the frequency the lower the wave intensity**

Cavities and all hardware components (Flanges, nuts & bolts…) have to be degreased with ultrasonic cleaning

US Cleaning efficiency





https://sonosys.de/en/ultrasonic-megasonic.html

Water Break Test on Nb Sample

- It’s a standard test for testing cleaning procedure with UPW and US

Not efficient cleaning

After good US cleaning procedure

ASTM F22 - 02(2007) Standard Test Method for Hydrophobic Surface Films by the Water-Break Test.

Superconductivity and its applications
EASISchool 3

Small parts cleaning

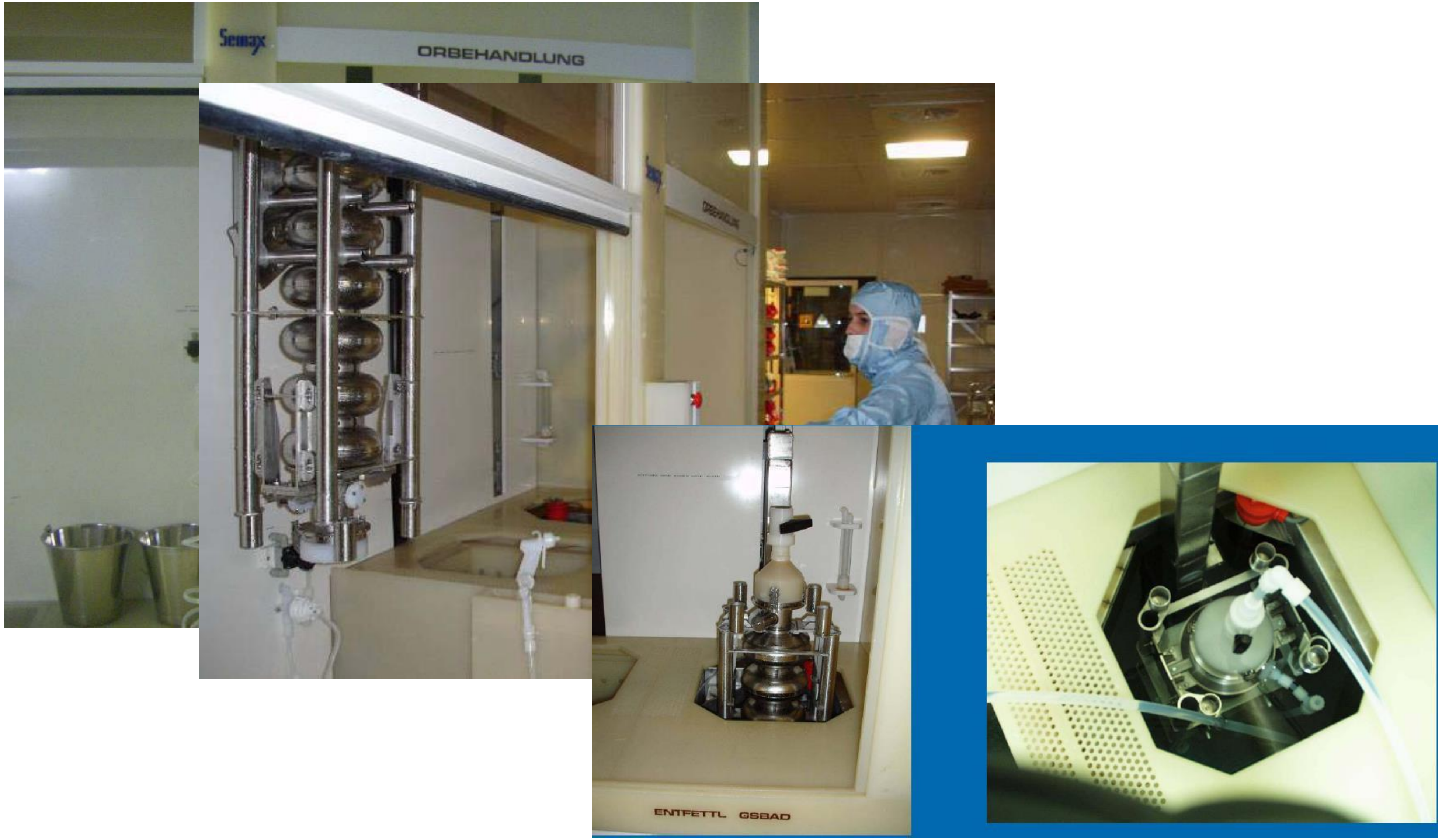
- **Water: UPW** (r >10 MW×cm)
- **Inline particle filter**
- **Standard dish washer machine** are successfully used, with a few corrosion problem (lifetime)
- **Special stainless-steel dish washer** for chemical and bio labs are available on the market (but expensive)
- **Detergent** can be the same used in US bath (foam!) as Tickopur
- **No additives**: salt or cleanser
- **Rapid cycling** (5 min) dish washer available

EASITrain

Large parts cleaning

- **UPW**(ρ >10 MΩ×cm)
- **Pressure**: 100 bar
- **Pump**: car wash piston pump
- **Detergent** as TICKOPUR R 33, but not used at companies for EXFEL
- **Automatic systems** with rotating table supporting the cavity frame
- **5 min spray** with detergent, if used
- **Water Rinse** (5 – 10 min)
- Widely used for **cleaning before entering** Clean Room

Ultrasonic tanks for cavity cleaning



DESY Courtesy

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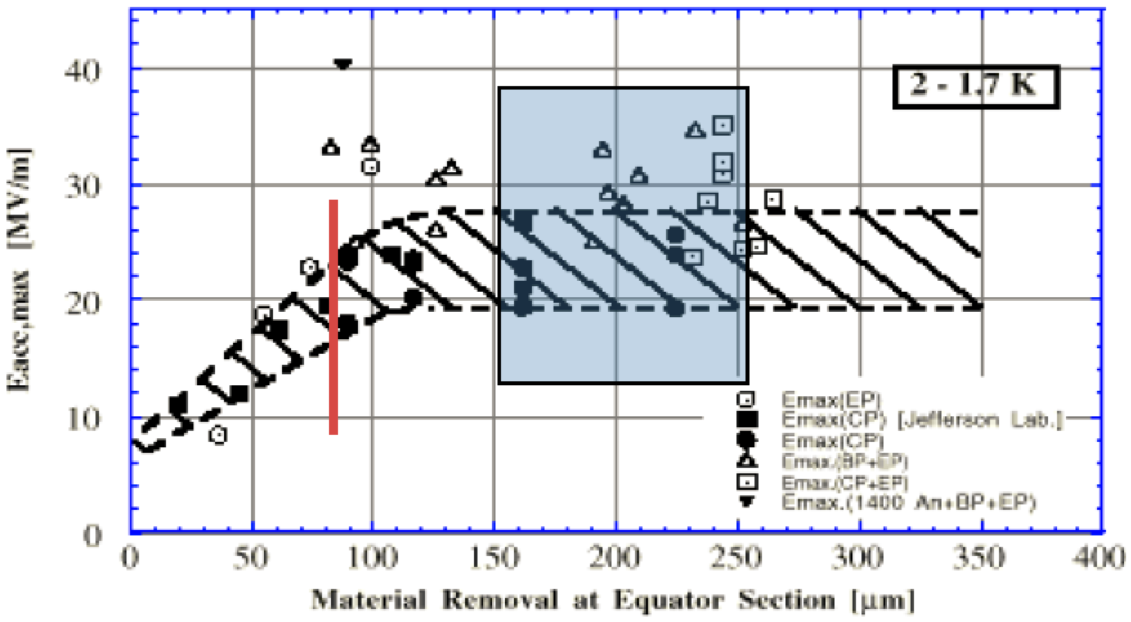
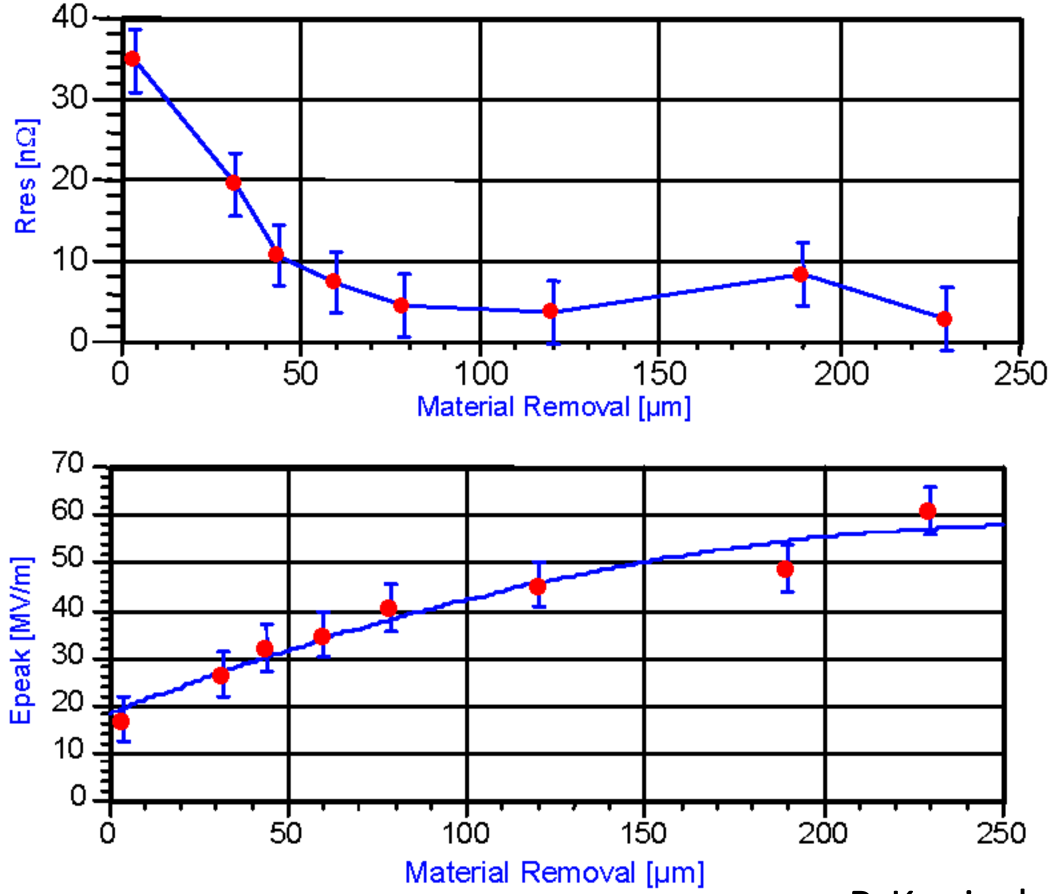
Removal of damage layer

- After all the mechanical operations, a **thin layer of about 200 μm** must be removed

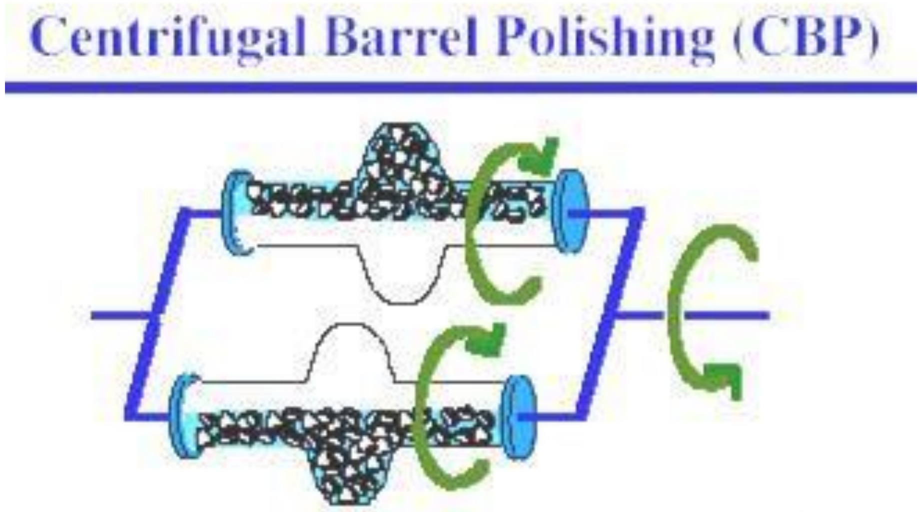


K. Saito



P. Kneisel

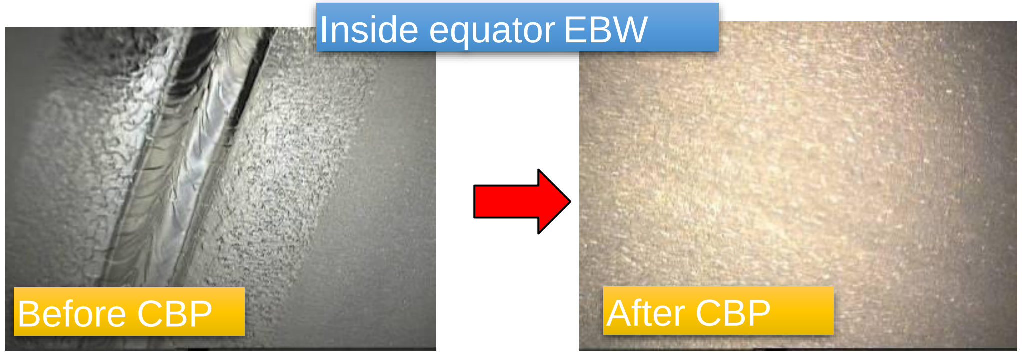
Centrifugal Barrel Polishing (CBP)



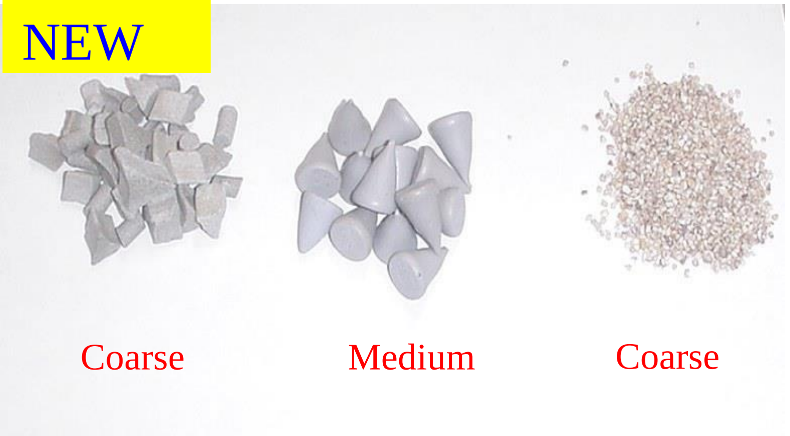
KEK 2001

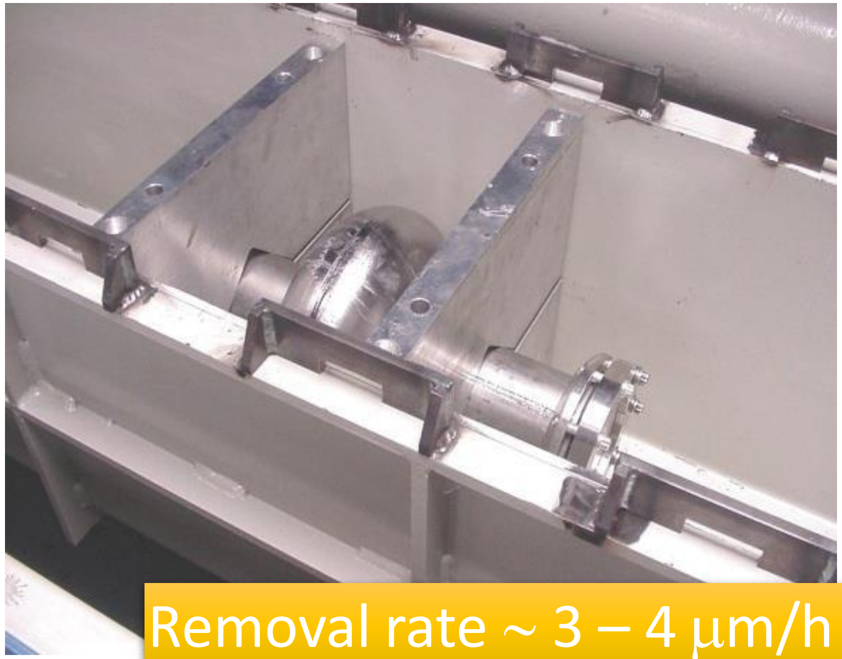
Implementation:

- **Plastic stones and liquid abrasive** added inside cavity and rotated
- **Stones rubbing on surface removes material** thus smoothing the surfaces (including weld areas)
- Benefit is **less overall chemistry** needed (80 μm) and smooth weld areas
- Removal of material **2x on equators** then irises. Average removal rate ≈ 5 μm/h



Barrel Polishing Machine @ JLAB





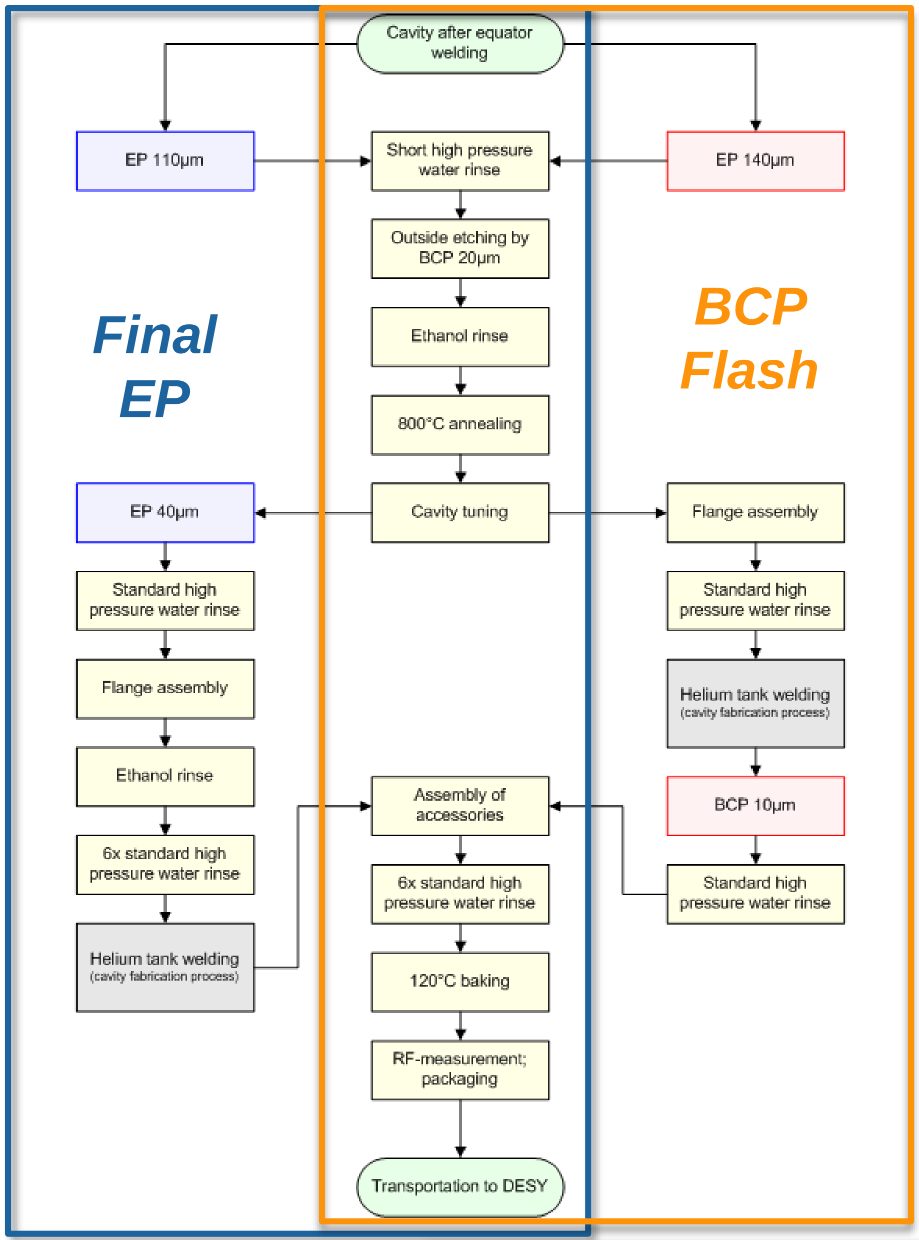
Removal rate ~ 3 – 4 μm/h

(Electro-)Chemical Nb removal

- **Nb is resistant to chemical attack**
  - **HNO_3:** oxidation of Nb surface and **passivation**, i.e. no more corrosion of the metal.
  - **HF**: **dissolve only Nb oxides**, but doesn't attack Nb itself
  - **HCl**: no attack
  - **H_2SO_4**: no attack
  - Strong alkaline solution (**NaOK, KOH, NH_4OH**): no attack
- **Two effects have to be coupled: Nb oxidation (e.g. HNO_3) and Nb oxides dissolution (HF).**

Buffer Chemical Polishing and Electro Polishing

- Buffer Chemical Polishing (BCP)
  - A mixture of Hydrofluoric (**HF**), Nitric Acid (**HNO_3**) and Phosphoric acid (**H_3PO_4**), usually in 1:1:1 or 1:1:2 ratio in volume
- Electro Polishing (EP)
  - A mixture of Hydrofluoric Acid (**HF**) and Sulfuric Acid (**H_2SO_4**) + electric current
- Sometimes, the **two processes** are used **together** to achieve better surface polishing (see EXFEL final steps)



EXFEL Cavity preparation

Buffered Chemical Process (BCP)

- Mixture of concentrated Hydrofluoric Acid (**HF, 40%**), Nitric acid (**HNO_3**, 70%) and Phosphoric Acid (**H_3PO_4, 85%**)
- H_3PO_4 doesn't participate the reaction: it act like a buffer slowing down the speed of the **exothermic reaction (self exiting!)**.
- **1:1:2, generally used, 1 μm/min @ 20 °C**

Oxidation

$$2Nb + 5NO_3^- \Rightarrow Nb_2O_5 + 5NO_2 + 5e^-$$

Brownish gas

$$2NO_2 \Rightarrow N_2O_4$$

Dissolve

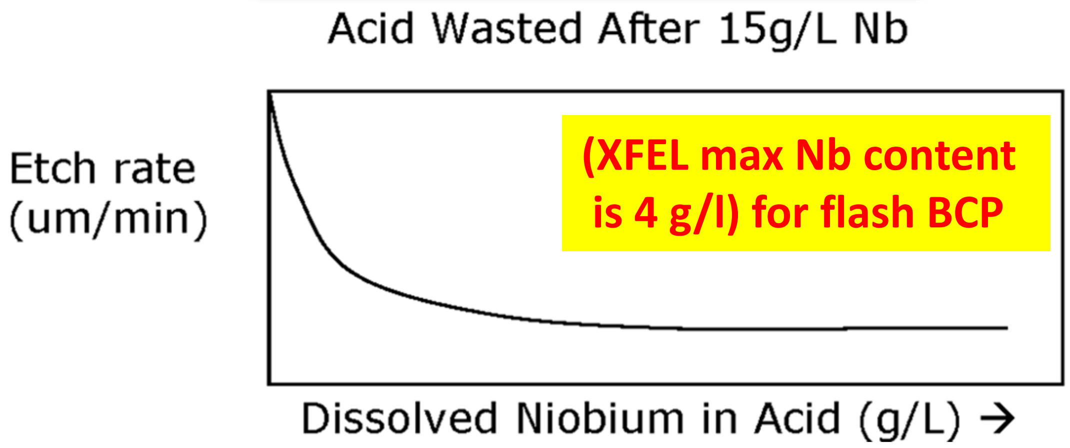
$$Nb_2O_5 + 6HF \Rightarrow H_2NbOF_5(\text{soluble}) + NbO_2F \cdot 0.5H_2O\ (\text{not soluble}) + 1.5H_2O$$

$$NbO_2F \cdot 0.5H_2O + 4HF \Rightarrow H_2NbOF_5 + 1.5H_2O$$

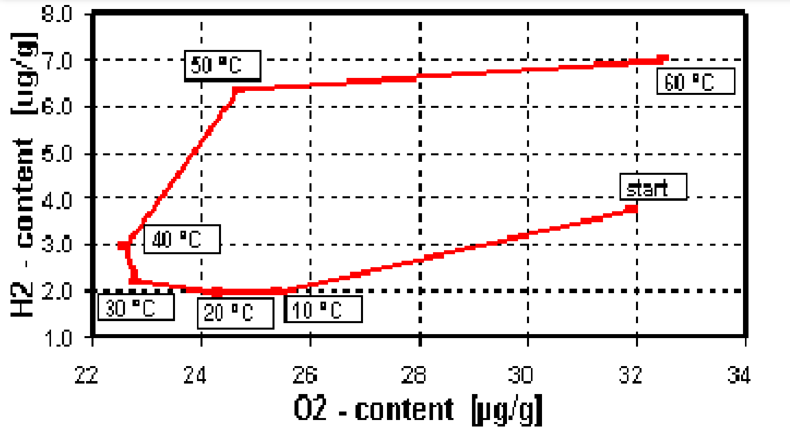
Use of BCP Process

- **1:1:1** still used for subcomponents due to high etching rate (~ 8 μm/min)
- **1:1:2** used for cavity treatment (~ 1 μm/min)
- BCP must **mixed before used** because it stratifies
- BCP is usually **cool down before and during etching** to mitigate temperature increase and hydrogen content (starts at 3-5 °C and ends around 20 °C)

Etching rate versus dissolved Nb

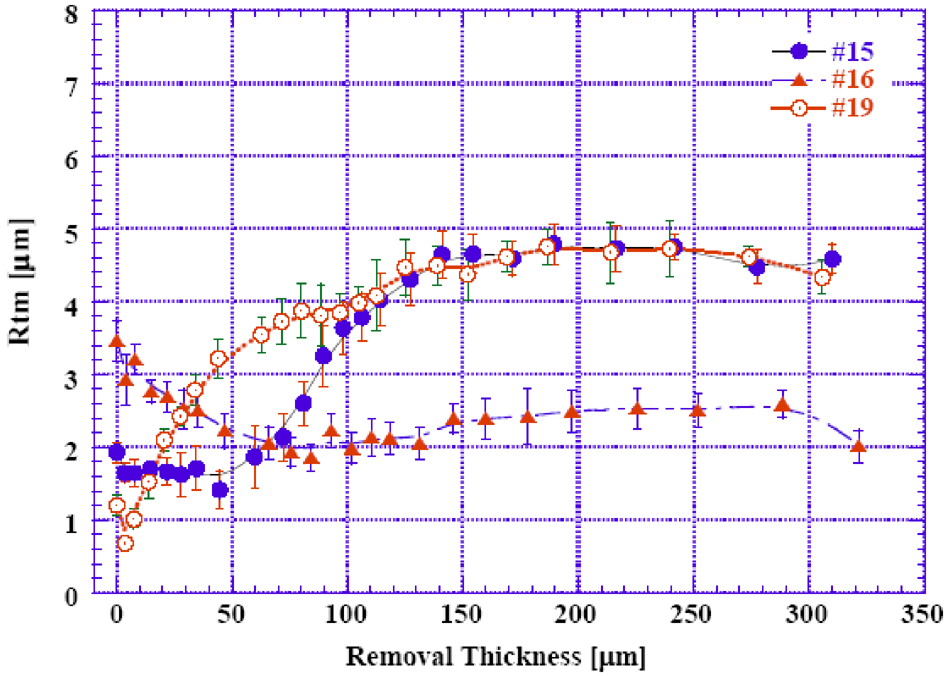


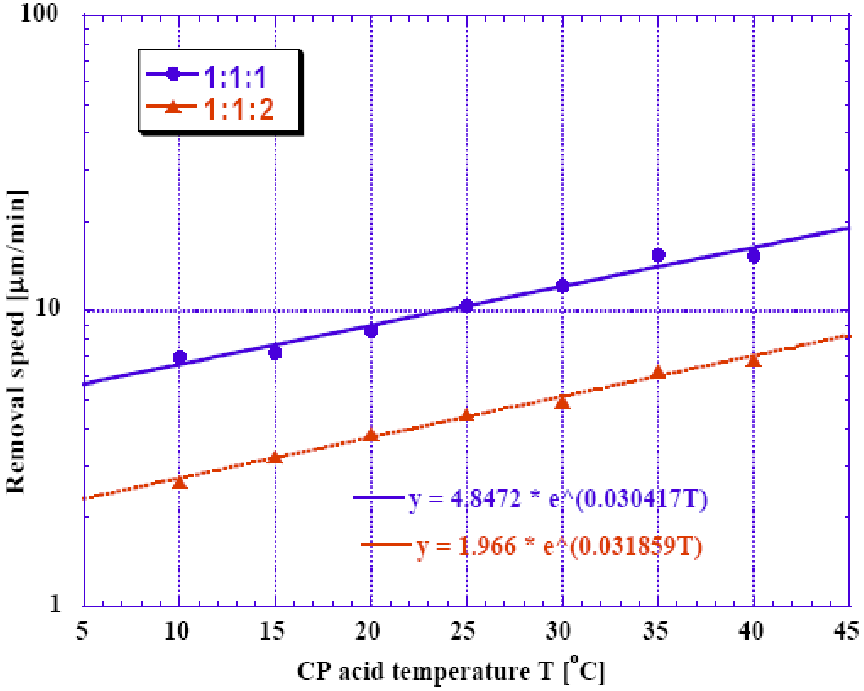
Hydrogen content vs BCP Temperature

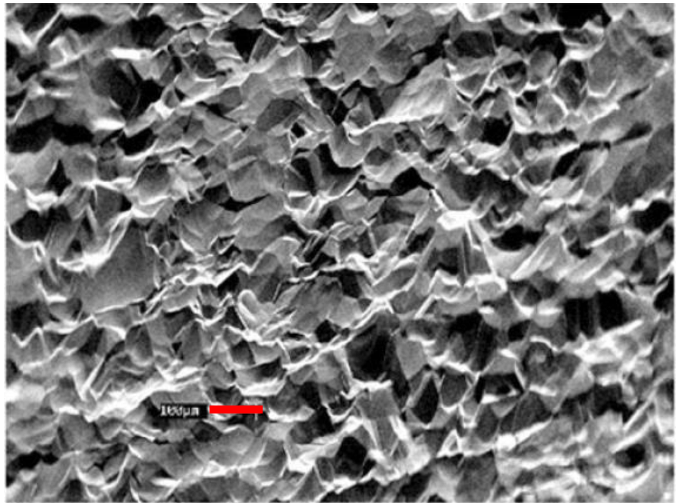


Gas content of Nb at different etching temperatures (Schölz). Etching time 20 min, BCP 1:1:4

BCP smoothing





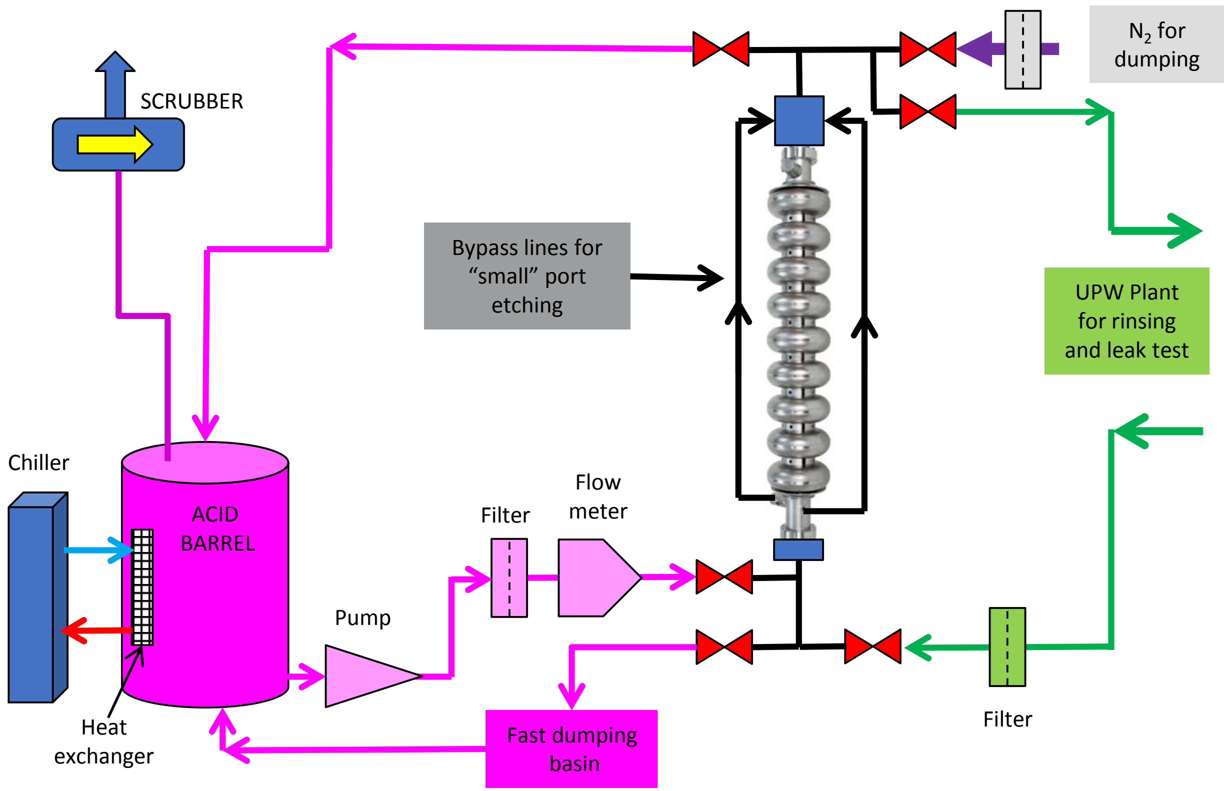


- **"Simple"** process
- **Roughness of 2 -5 µm** (100 x 100 μm^2 scale) after 100 µm etching
- **High etching rate**

Courtesy G. Ciovati

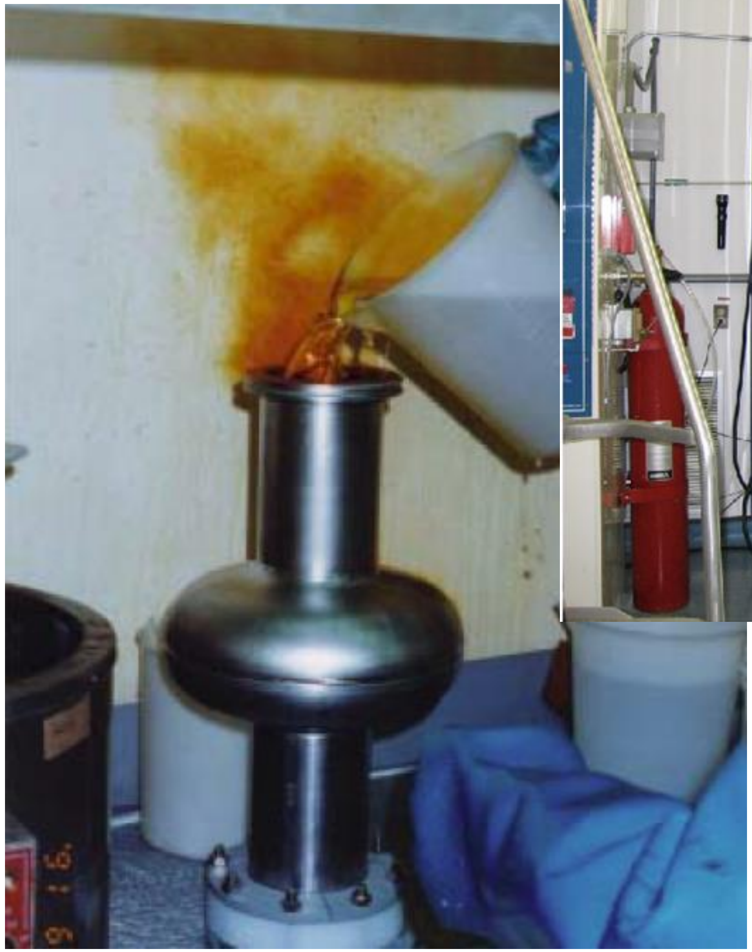
BCP Plant Layout

- **All components** in the acid mixture circuit **MUST be resistant** to acid attack
- Operative **temperature**: **below 20 °C,** to reduce hydrogen diffusion in Nb. Usually treatment starts at about **5°C ÷ 6 °C**
- **Exothermic reaction**: heat exchanger or cooled barrel is needed



- **Cavity held in vertical position**, acid flow from the bottom part
- **Temperature gradient** causes **increased** etching from one end to the other
- **Usually etching rate on iris is 2 x the equator one**
- Used both for **bulk removal and final etching**: for **EXFEL** only for **final etching of half of cavities**

BCP Plant in Operation in Labs



BCP Cabinet JLAB

DESY BCP

BCP Plant in Operation at Qualified Vendors

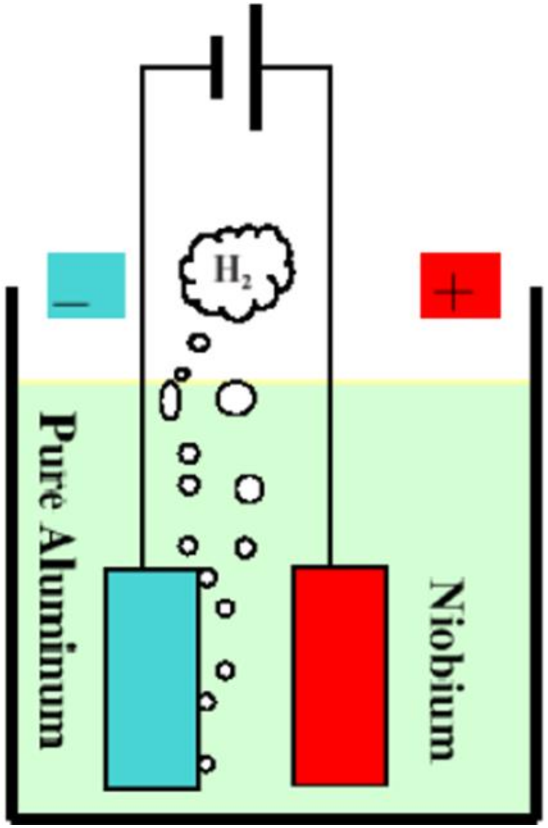
Automatic BCP system for subcomponents @ Ettore Zanon for EXFEL (etching + rinsing)

INFN

EASISchool 3
Superconductivity and its applications

Electropolishing (EP)

- A **constant voltage** is kept between an Aluminum electrodes and the cavity immerse in a mixture of Hydrofluoric Acid (**HF**, 49%) and Sulfuric Acid (**H_2SO_4**, 96%) in a ratio 1:9 (typical) in volume.
- **Reaction is not self sustained**: no current - no reaction



Oxidation

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

Dissolve

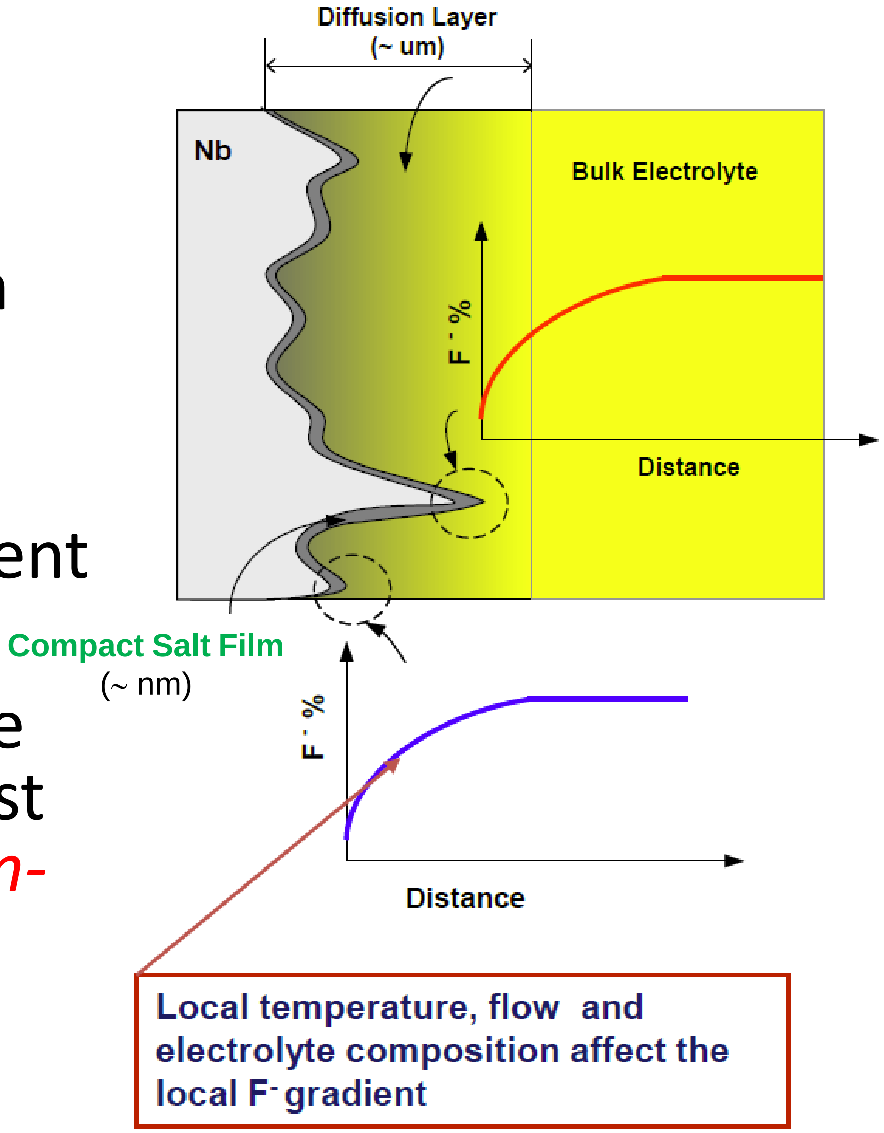
$$Nb_2O_5 + 6HF \Rightarrow H_2NbOF_5(\text{soluble}) + NbO_2F \cdot 0.5H_2O\ (\text{not soluble}) + 1.5H_2O$$

$$NbO_2F \cdot 0.5H_2O + 4HF \Rightarrow H_2NbOF_5 + 1.5H_2O$$

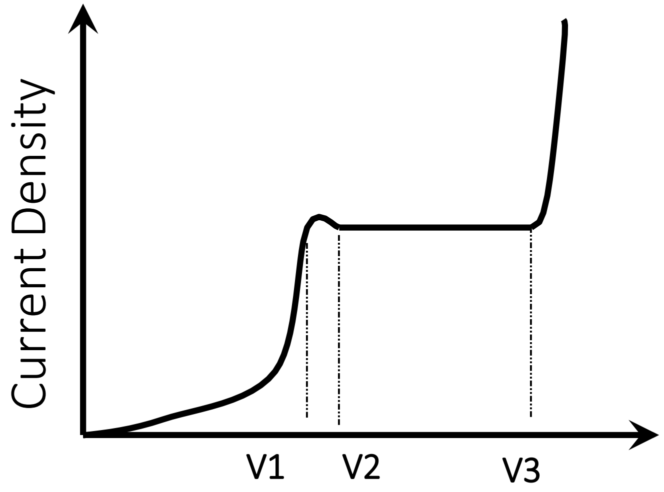
EASITrain

EP Mechanism

- Anodization of **Nb** in **H_2SO_4** forces growth of **Nb_2O_5**
- **F^-** dissolves **Nb_2O_5**
- These competing processes result in current flow and material removal
- Above a certain anodization potential, the **reaction rate plateaus**, limited by how fast fresh **F^-** can arrive at the surface (*diffusion-limited*)
- The diffusion coefficient sets a scale for optimum leveling effects



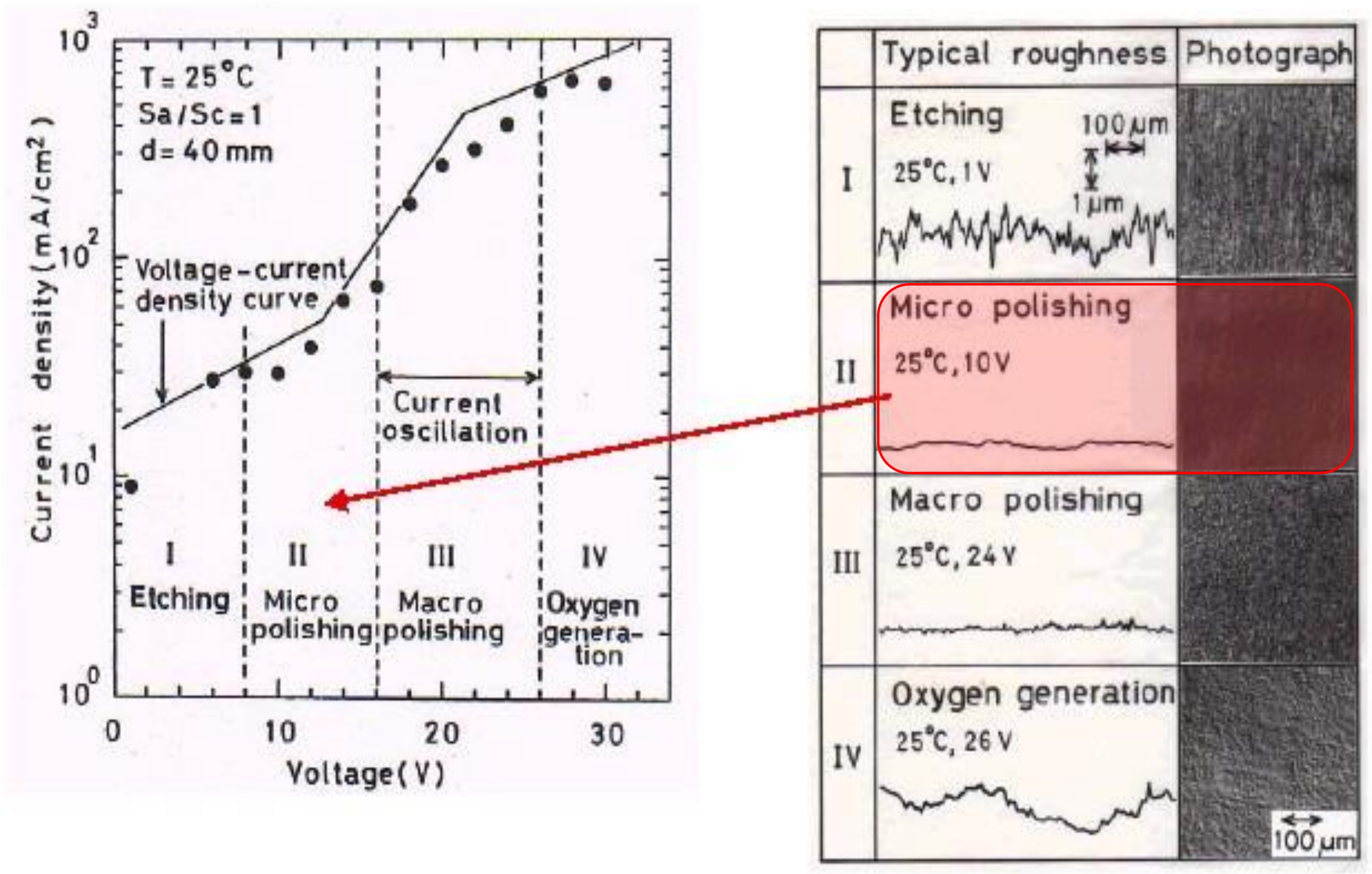
EP V vs J



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

Talk by K.Saito in JLAB on Oct. 2003

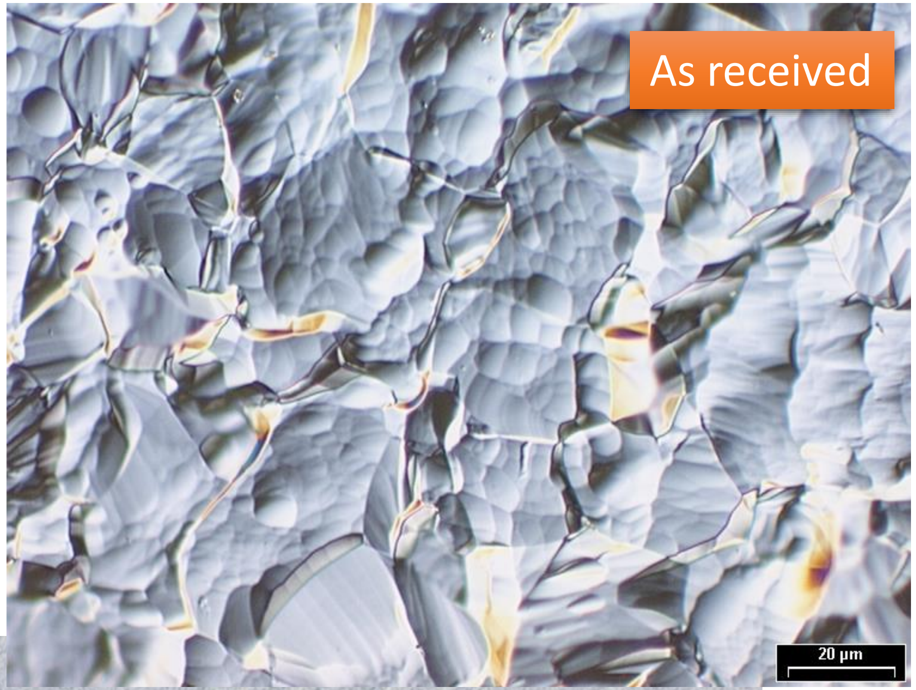
Micro and macro electropolishing in niobium EP

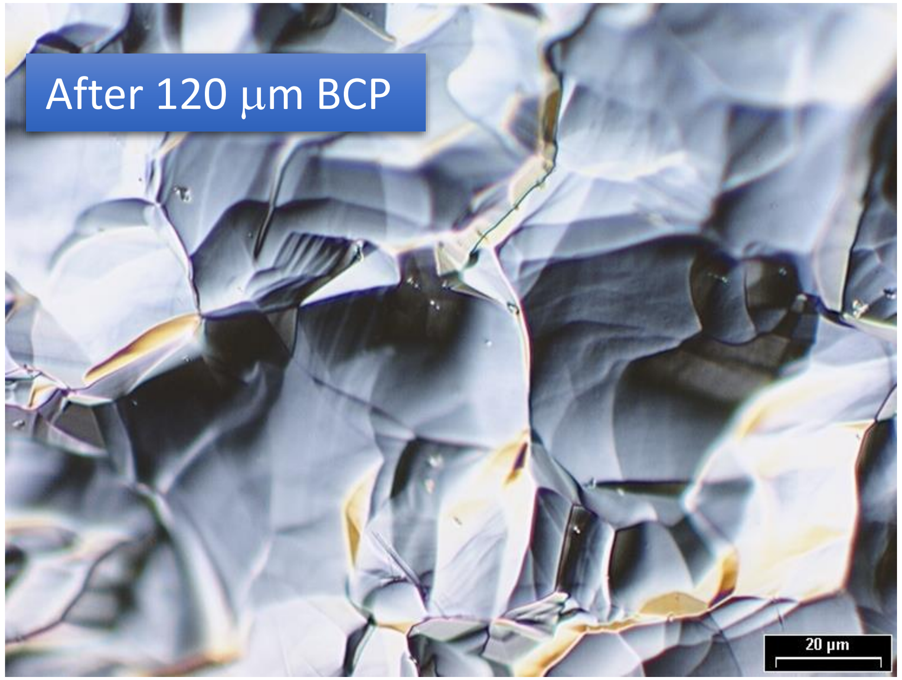


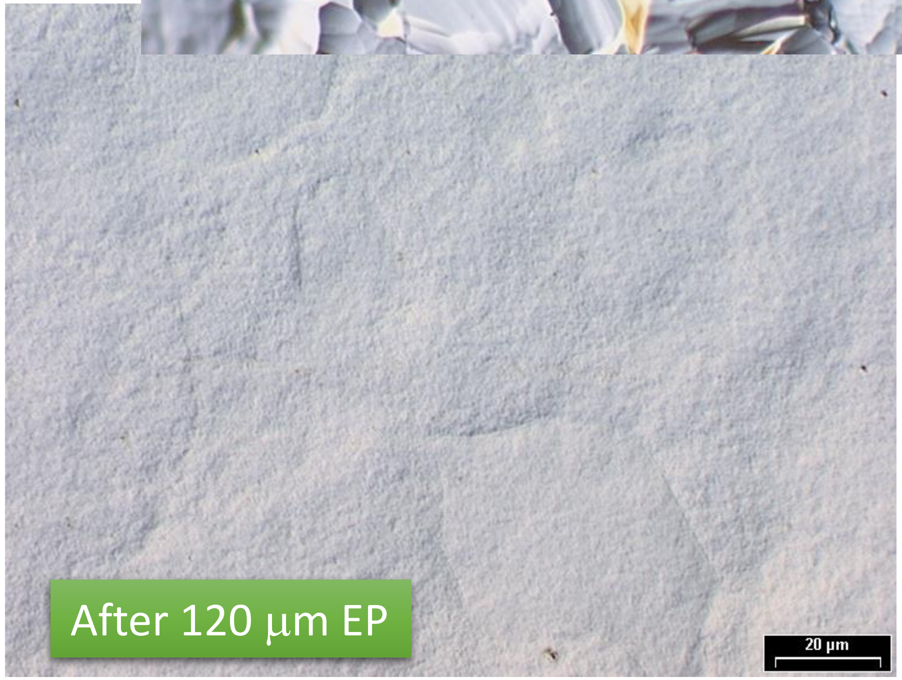
Kenji Saito, KEK, 1989

Surface Polishing BCP vs EP

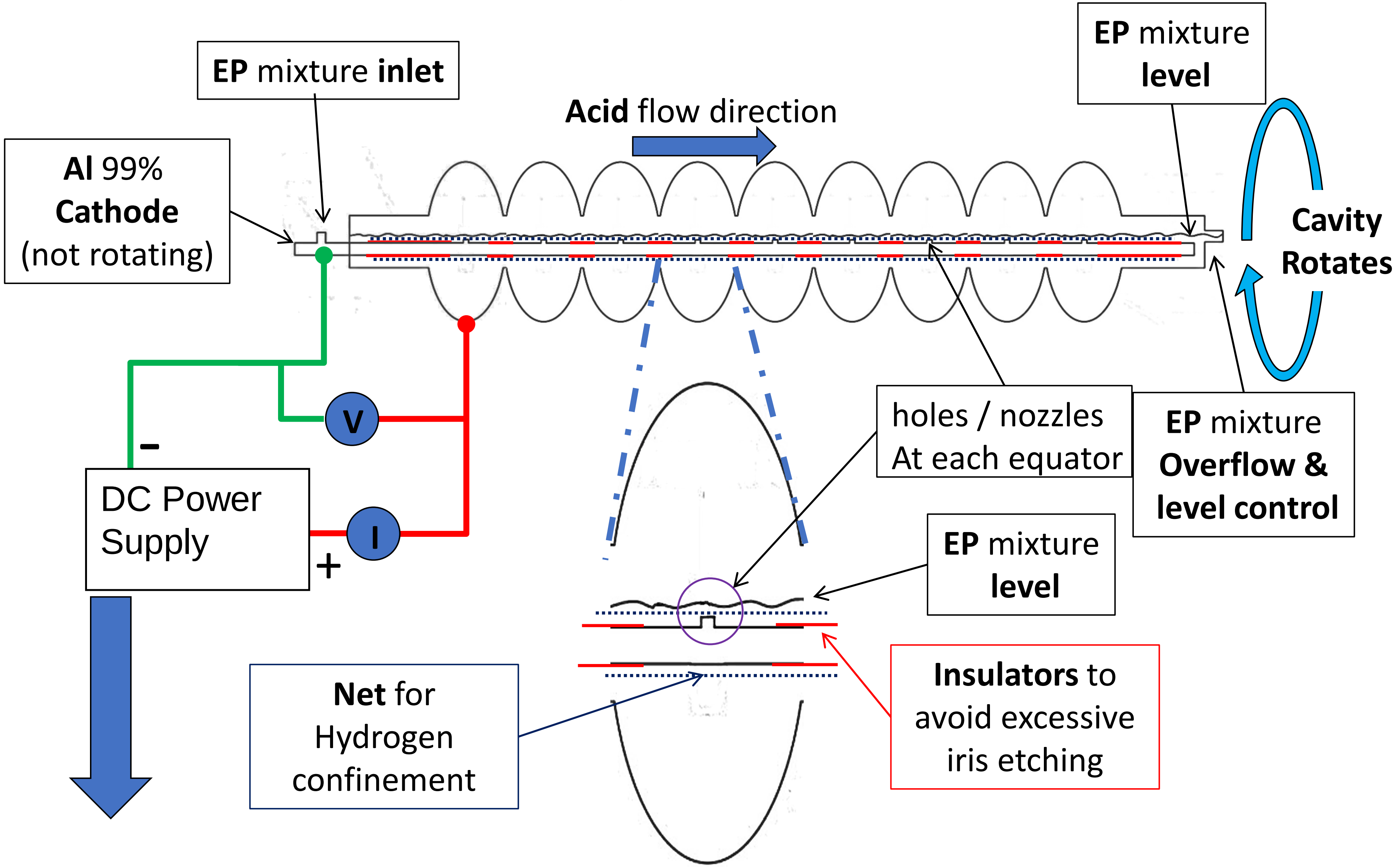
- The main **difference** between BCP and EP is **smoothening of grain boundaries**.







Basic EP for SRF cavity

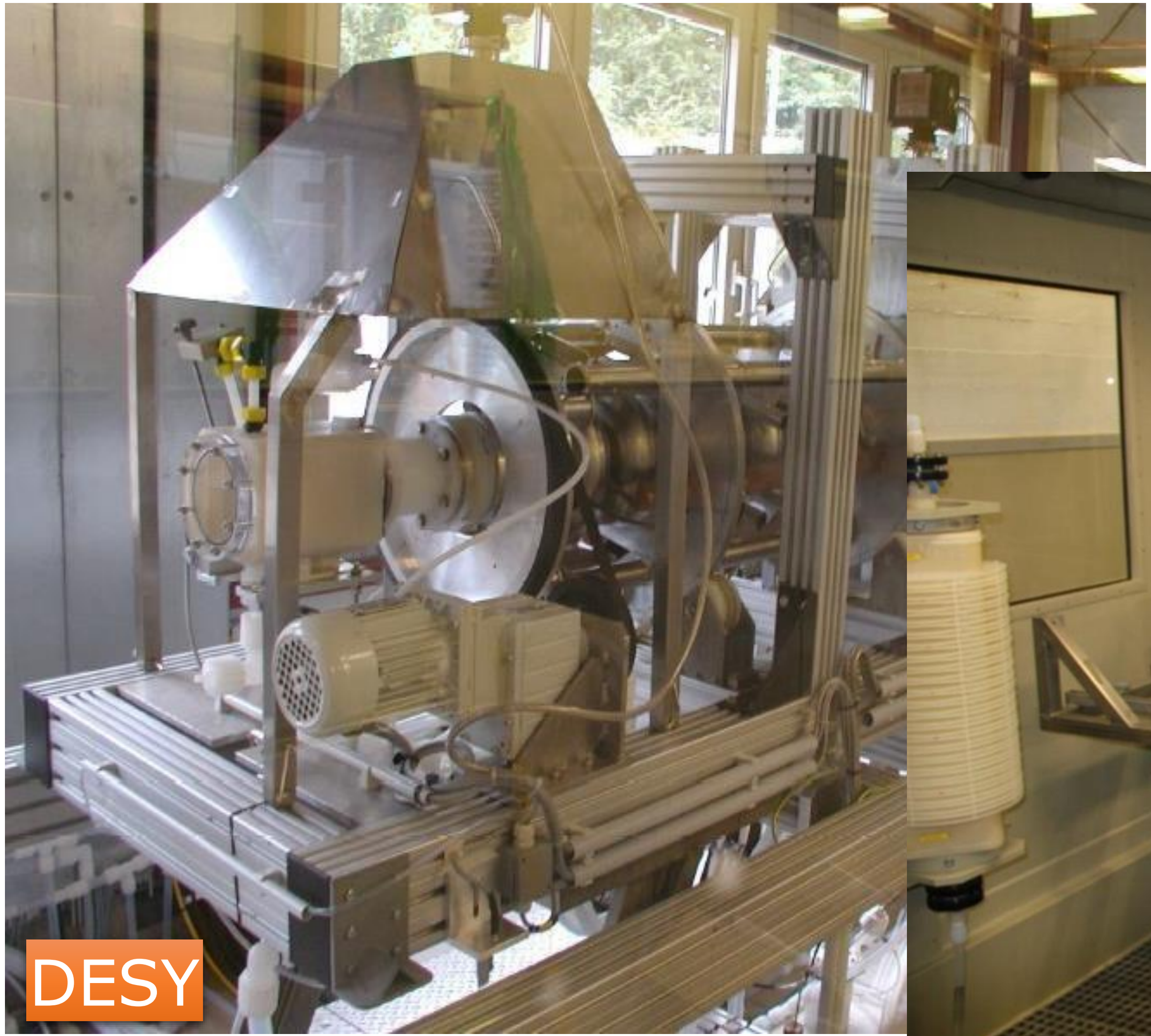


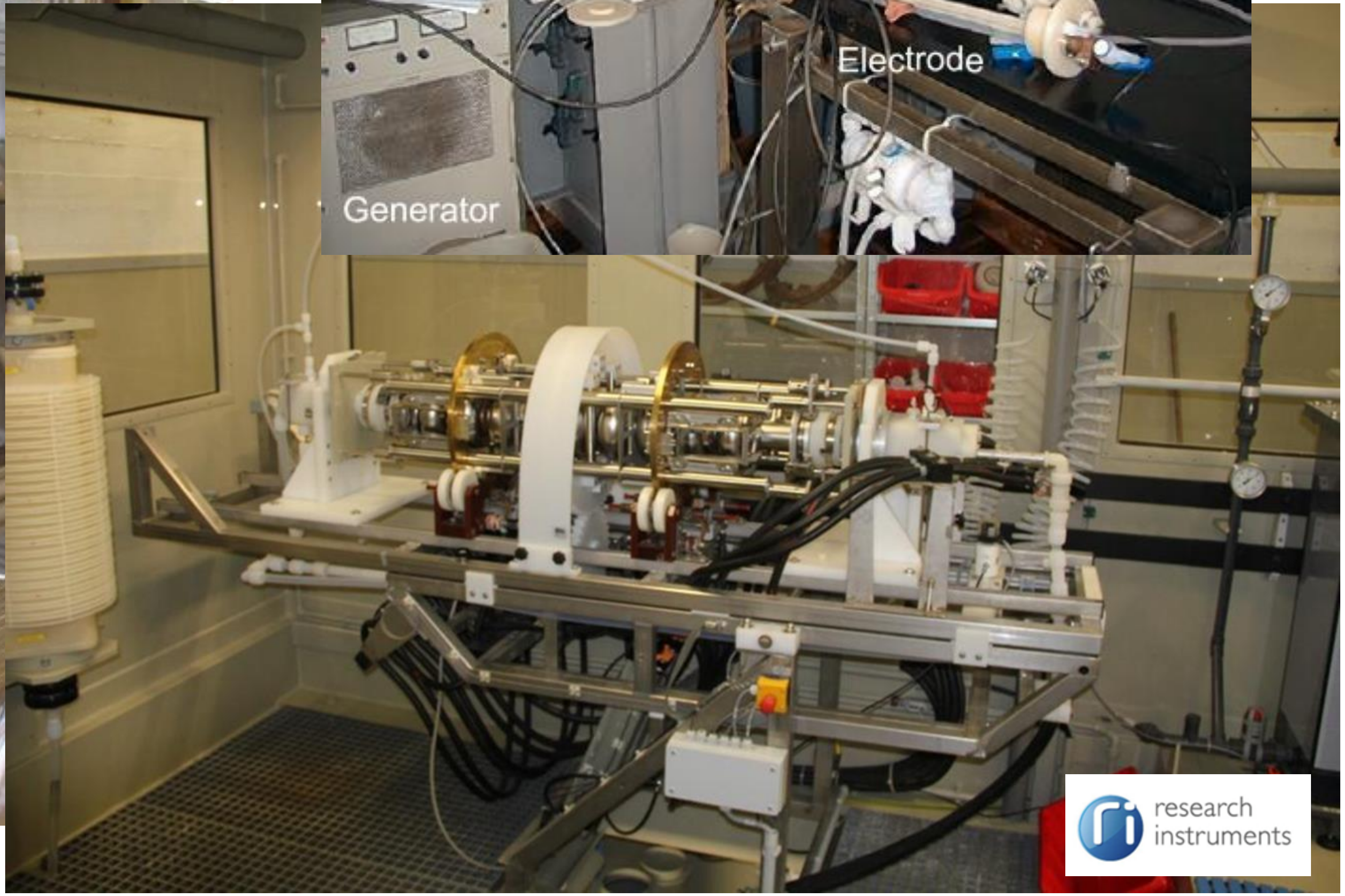
Electropolishing of SRF cavities

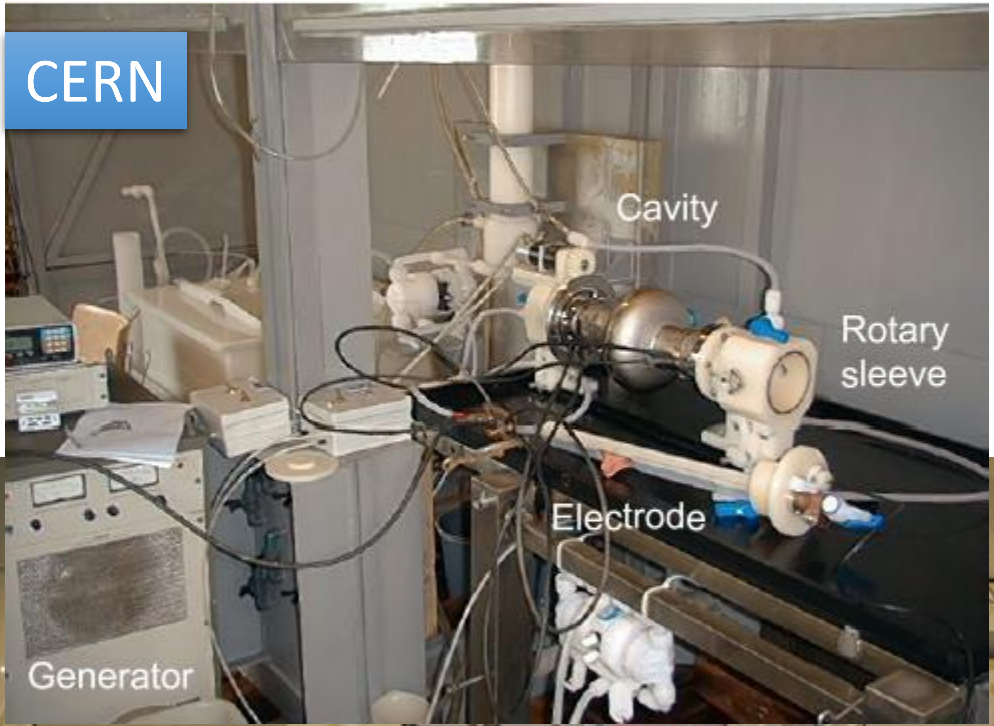
- Etching rate typical **0.3 ÷ 0.4 μm/min**
- Cavity (or electrode) is **rotating**
- It requires **ethanol rinsing** to remove Sulphur
- The current density (30-100 mA/cm^2) in the plateau region:
  - decreases linearly with lower HF/H_2SO_4 ratio
  - increases with increasing temperature
- **Temperature** during the process is maintained **between 25 – 35 °C**
- **Current oscillations often observed during polishing** (dynamic balance between oxide formation and dissolution). It's not a necessary condition for good surface finishing but indication of good processing parameters (temperature, voltage, agitation, HF concentration)

Finding the right balance among the processing parameters becomes complicated when polishing multi-cell cavities!

EP Systems

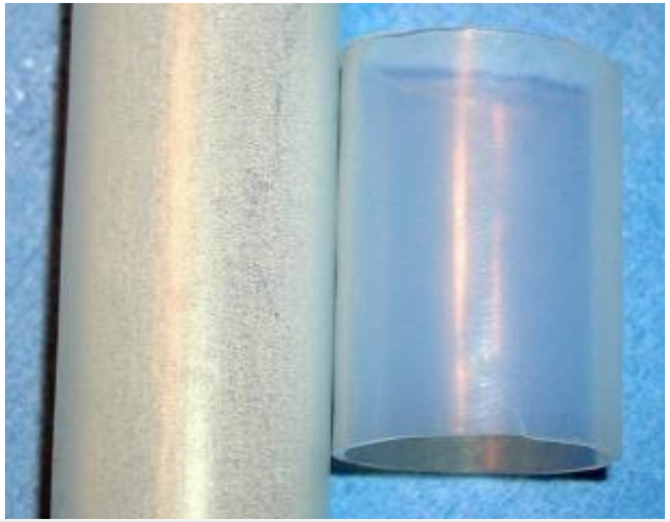

DESY


research
instruments


CERN
Cavity
Rotary
sleeve
Electrode
Generator

EP Ethanol Rinse

- **Motivation: during EP process sulfur is produced and can cause field emission**
- Sulfur segregates out of the acid as a **reaction with the Al electrode**, and is **deposited all over the system**, and also on the **Nb surface**
- Risk of **reaction with Nb during 800 °C** heat treatment: S must be removed before this step
- **Sulfur is insoluble in water**, but (slightly) soluble in ethanol
- Either ethanol rinse or cleaning with detergent + US necessary



PVDF tube before and after ethanol cleaning

Sulfur removed from a PVDF tube

BCP vs EP

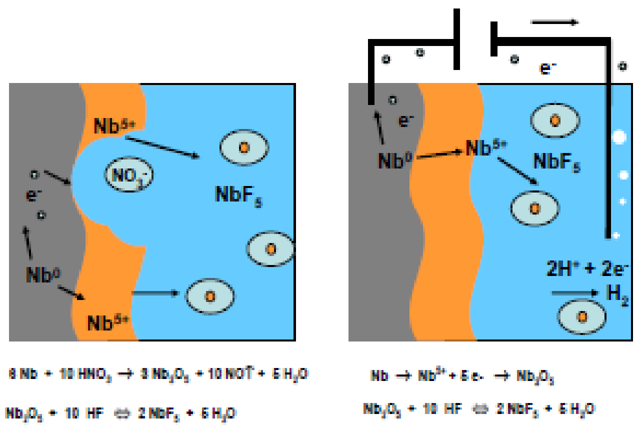


Fig. 6: A comparison between chemical polishing (left) and electropolishing (right). In both cases, niobium is oxidized into Nb^{5+}. In the case of chemical polishing, oxidation occurs because of the presence of a strong oxidant (NO_3^-) in the solution, while in electropolishing oxidation occurs because of the bias applied to the anode. Because of the presence of water, the stable form of Nb is Nb_2O_5; but HF decomposes the oxides into NbF_5, which is soluble in the solution.

- BCP
  - 2 Volumes of **H_3PO_4** (**buffer**, very viscous)
  - 1 Volume of **HNO_3** (**oxidant**, transforms Nb into Nb^{5+})
  - 1 Volume of **HF** (**complexant** of Nb^{5+}, dissolves the oxide layer formed by HNO_3 into NbF_5)
  - Pros
    - **Easy to handle**, middle stirring necessary
    - **Fast etching rate**
    - **Very reproducible**
  - Cons
    - **This is not "polishing" but "etching"**: all crystalline defects are preferentially attacked (etching pits, etching figures)
    - Grains with various orientations are not etched at the same rate, which **induced roughness**!
    - Except for a few cases E_{acc}^{max}~25-30 MV/
  - **Caution!**
    - Do not process at temperatures higher than 25 °C
    - Risk of runaway

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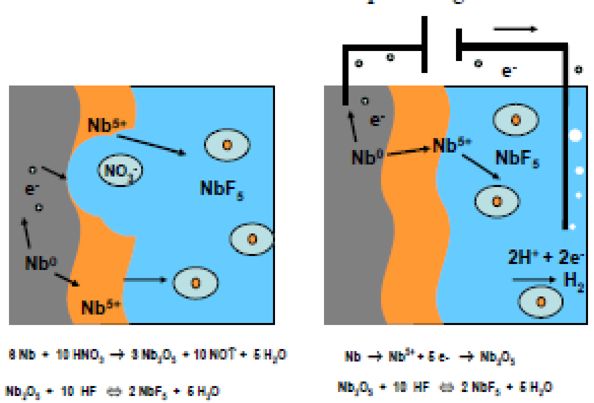
BCP vs EP



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- EP
  - 9 Volumes of **H_2SO_4** (**buffer**, very viscous)
  - 1 Volume of **HF** (**complexant** of Nb^{5+}, dissolves the c layer formed due to the high potential applied to Nb
  - Pros (Ideal condition, i.e. viscous layer present)
    - **This is really "polishing"**, not sensitive to crystallographic defects – it produces a smooth surface
    - Should not be sensitive to the cathode-anode distance - the **same etching rate everywhere**
    - It gives (but not always) **the best ever E_{acc}^{max}** ~ 45 MV/m (TESLA shape -> ~180 mT)
  - Cons
    - It is **not possible to reach an ideal state** in most of our processing conditions
    - **Very sensitive** to stirring condition, temperature, and aging of the mixture
    - **Not very reproducible**
    - **Safety issues** (acid mixture sensitive to water, H2 evolution, etc.)
  - **Caution!**
    - If T increases: the etching rate increases but there is also a risk of pitting, H loading and HF evolution
    - It V increases: the etching rate increases but there is also a risk of pitting, the generation of Sulphur particles and sensitivity to the cathode-anode distance

How to Achieve the Best SRF Performance: (Practical) Limitations and Possible Solutions1
C. Z. Antoine

Cavity preparation for SRF qualification

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication ($\approx$ 150÷200 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - Additional "cleaning" steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
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- Clean evacuation
- Low-temperature baking

Annealing – Cavity firing at high temperature

- H diffuses in the bulk during the various etching treatments.
  See **R. E. Ricker and G. R. Myneni,** J. Res. Natl. Inst. Stand. Technol. **115**, 353-371 (2010), Evaluation of the Propensity of Niobium to Absorb Hydrogen During Fabrication of Superconducting Radio Frequency Cavities for Particle Accelerators.
- Nb is an active metal with respect to various gases: it acts like a getter.
- Hydrogen makes a solid solution in Nb, H_2 equilibrium pressure is driven by Sievert Law

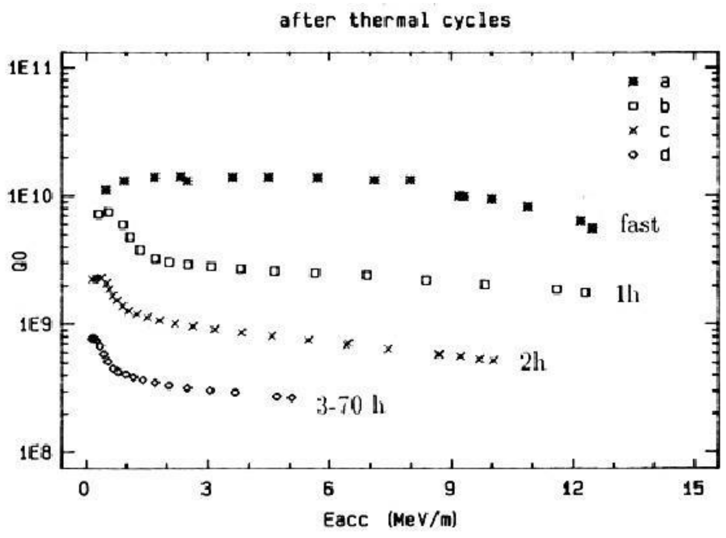
$$\ln p_{H2} = 2\ln Q_{Hbulk} + B/T + A$$

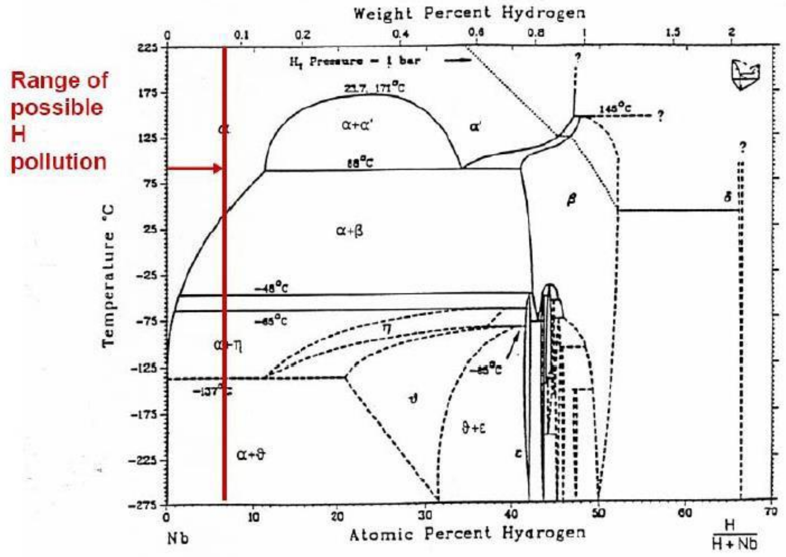
where Q_{HBulk} is the concentration of H_2 in the metal, B and A are constants.

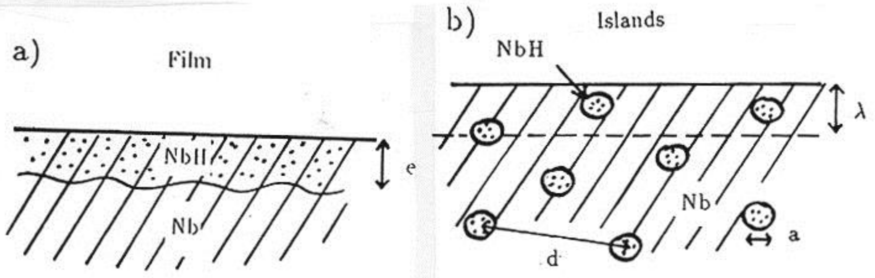
- Equilibrium pressure is **temperature dependent** and **increasing the temperature**, maintaining a **low H_2 partial pressure**, **H_2 is desorbed from the bulk (Nb)**

Hydrogen in Niobium Q "disease"

- Cavities that remain at **70-150 K for several hours** (or slow cool-down, < 1 K/min) experience **a sharp increase of residual resistance**
- **More severe** in cavities which have been heavily **chemically etched**
- **H is readily absorbed into Nb** where the oxide layer is removed (during chemical etching or mechanical grinding)
- **H has high diffusion rate in Nb**, even at low temperatures.
- **H precipitates** to form a hydride phase **with poor superconducting properties**: T_c=2.8 K, H_c=60 G
- At room temperature the required concentration to form a hydride is 103- 104 wppm
- At 150 K it is < 10 wppm

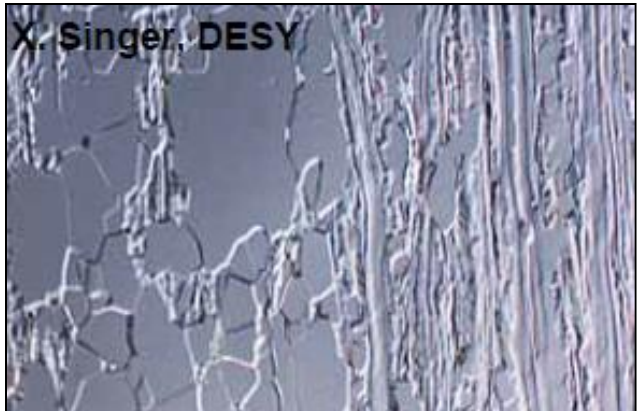






Annealining

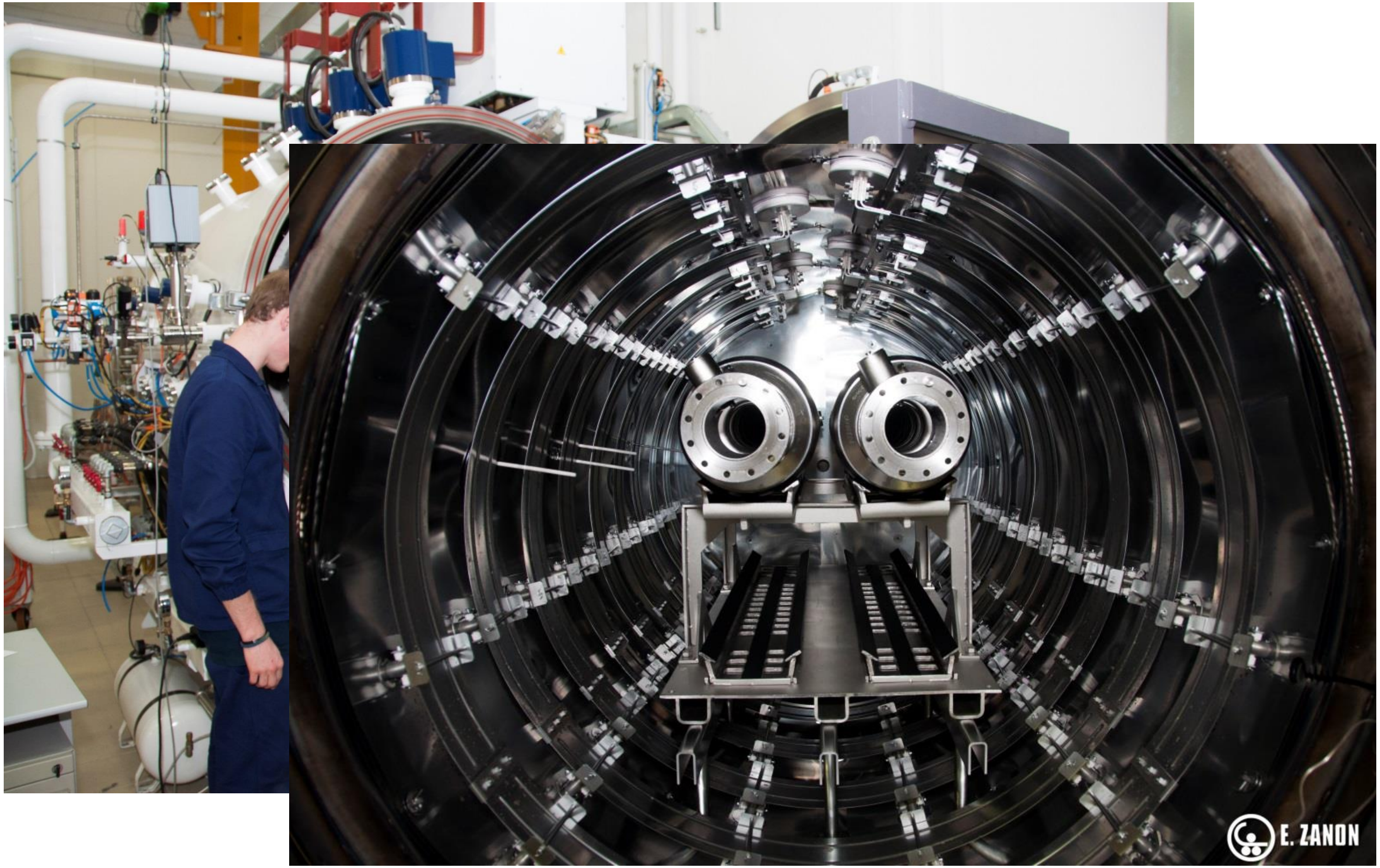
- **Hydrogen outgassing**
  => most efficient at 750°C – 800°C, **2h under good vacuum**
- **Recrystallization** (goal is close to 100% with highest RRR)
  - **Removing of defects and curing of dislocations**
  - **Nucleation of new grains and growing of new crystals**
  - Grain growth (depending on temperature and purity)
- **Nb becomes softer and this facilitate the cavity tuning process**
- Different parameters at different labs:
  - 600 °C/10 h at Jlab
  - 800 °C/2 h at DESY
  - 750 °C/3 h at KEK



No completely recrystallized Nb

Completely recrystallized Nb

EZ Furnace



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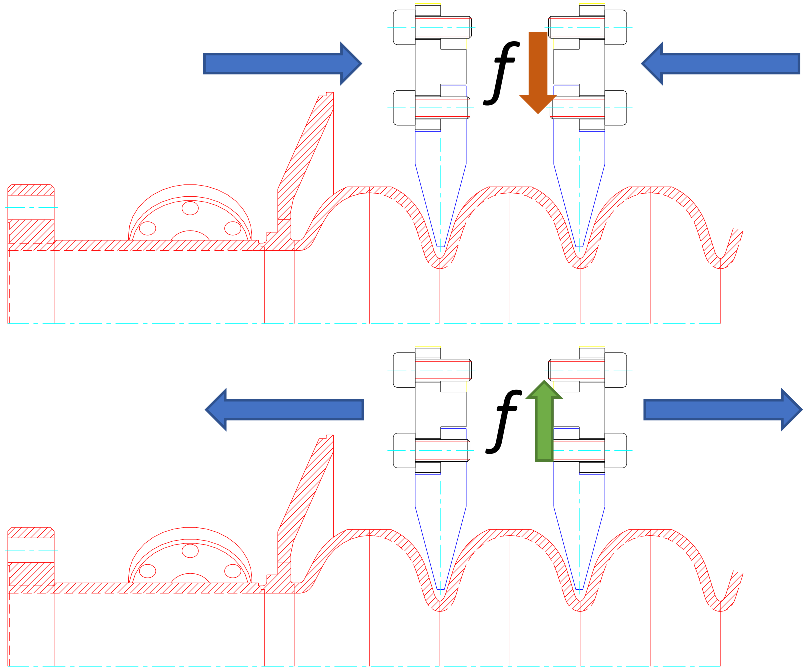
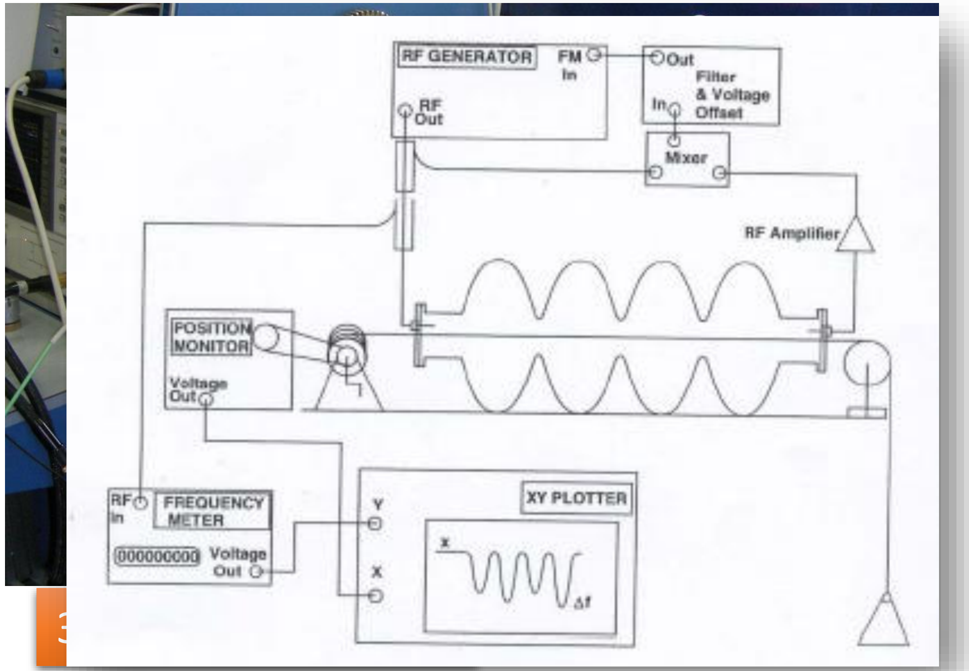
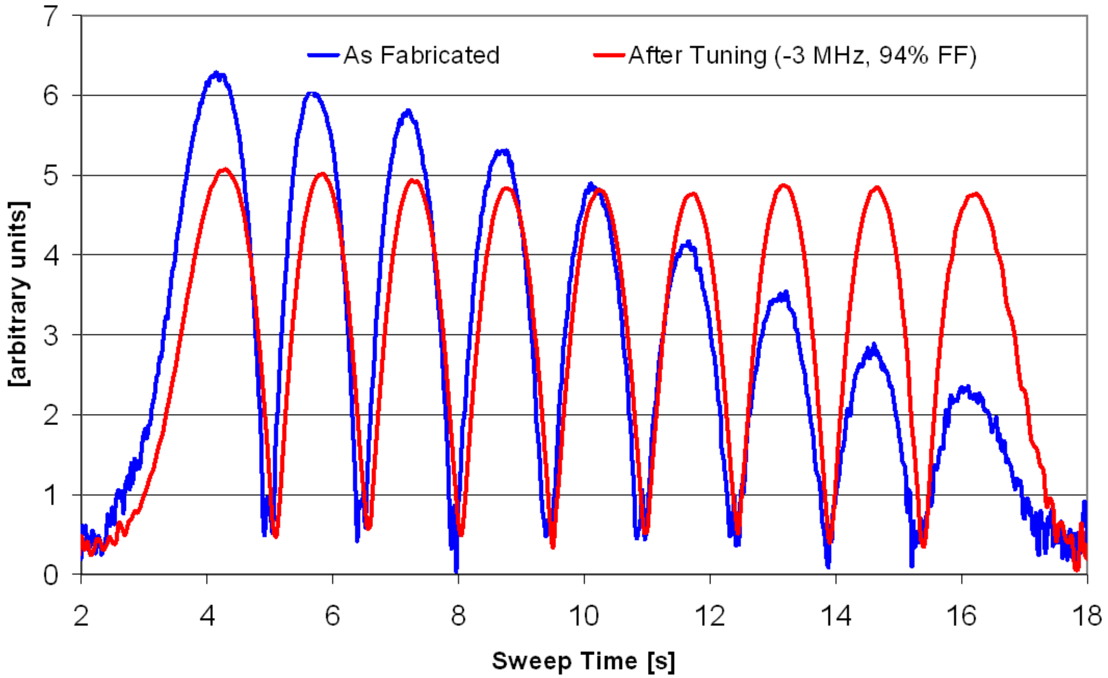
Frequency tuning



- After the treatments, the cavity needs to be tuned to the **right frequency** and **field flatness**.
- This operation is done by **tuning each single cell** to achieve proper field distribution





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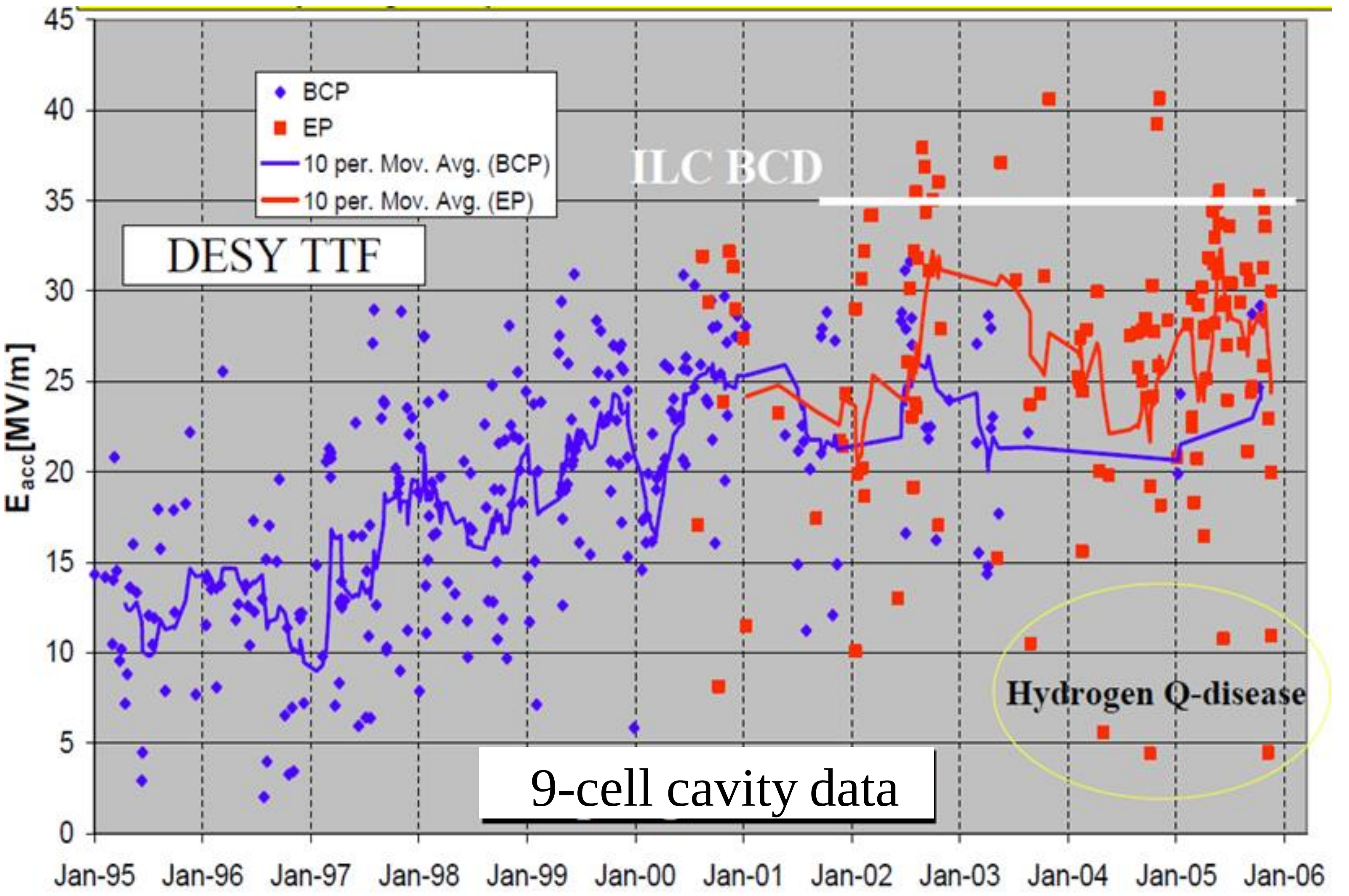
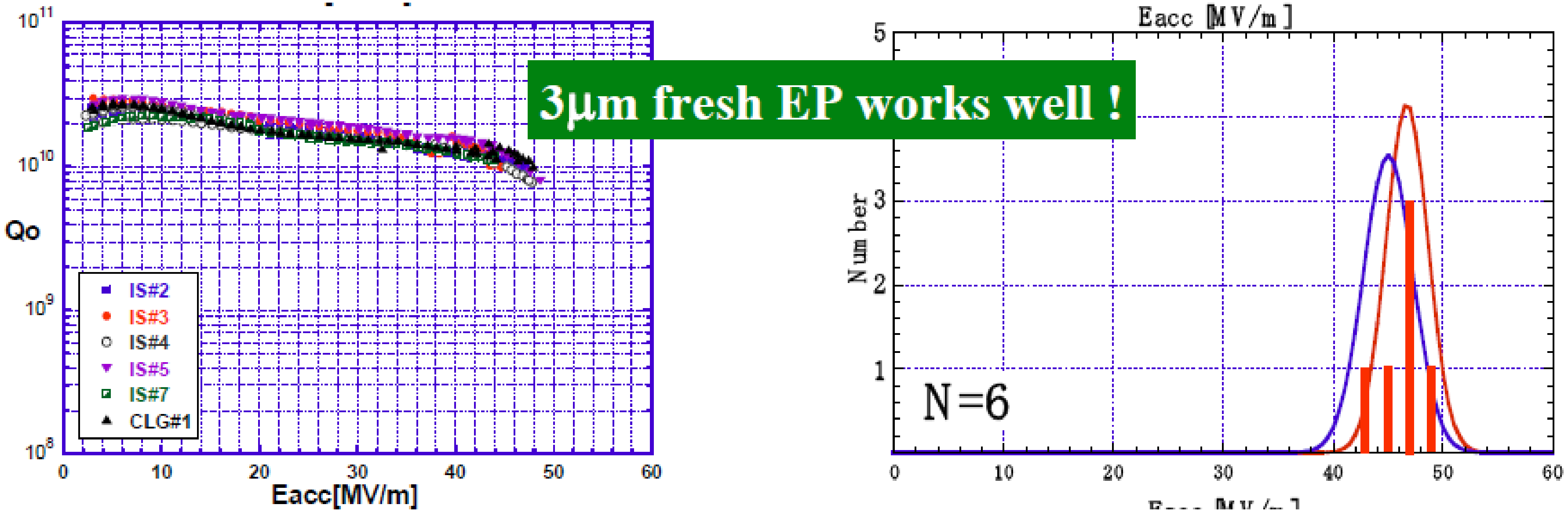
Post EP treatment

- The **early EP** treatment was **quite unstable** and with large scatter in the results
- A proper post EP treatment was found ….



Post EP treatment

- Ethanol Rinse (DESY)
- "Flash" BCP (10 μm) (DESY)
- "Flash" EP (3 μm, fresh acid, no re-circulation) (KEK)
- Ultrasonic Degreasing with Micro-90 and hot water (JLab)



Courtesy G. Ciovati

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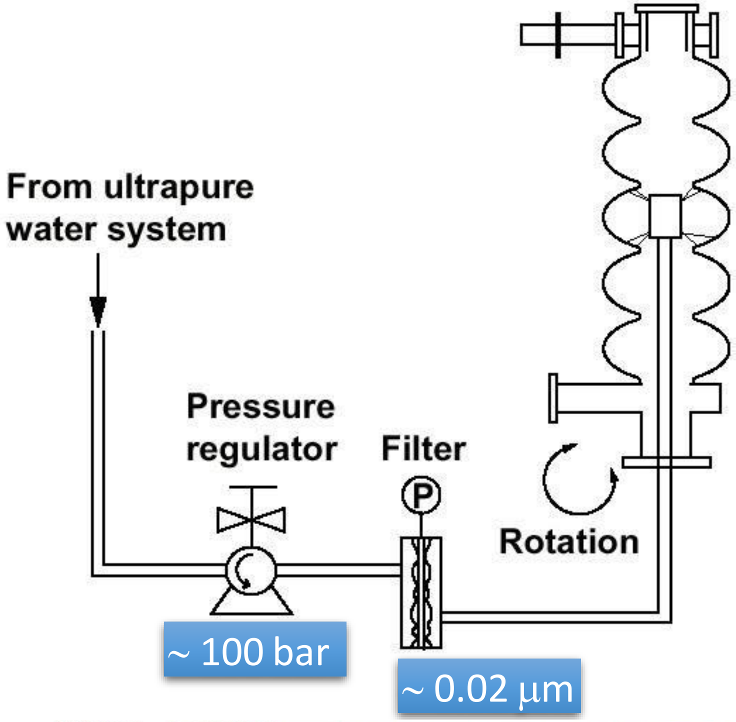
High Pressure Rinsing

- The **final step** in cavity assembly is the **Rinsing with a High Pressure UPW** jet to remove particulate from the handling and residual from chemical treatments
- Water jet **must be moved continuously**: if jet impacts stably in one-point Nb surface can be damaged
- Continuous **motion of the cavity respect jets** (drawing a spiral behavior that cover completely the Nb surface)
- Ultra pure (6.0) filtered (40 nm) **nitrogen protection** gas injection **coaxial** with water to reduce risk of particles entering
- **Cavity** must be **grounded** otherwise it will be electrically charged



Oxidation induced by fixed HPR jets

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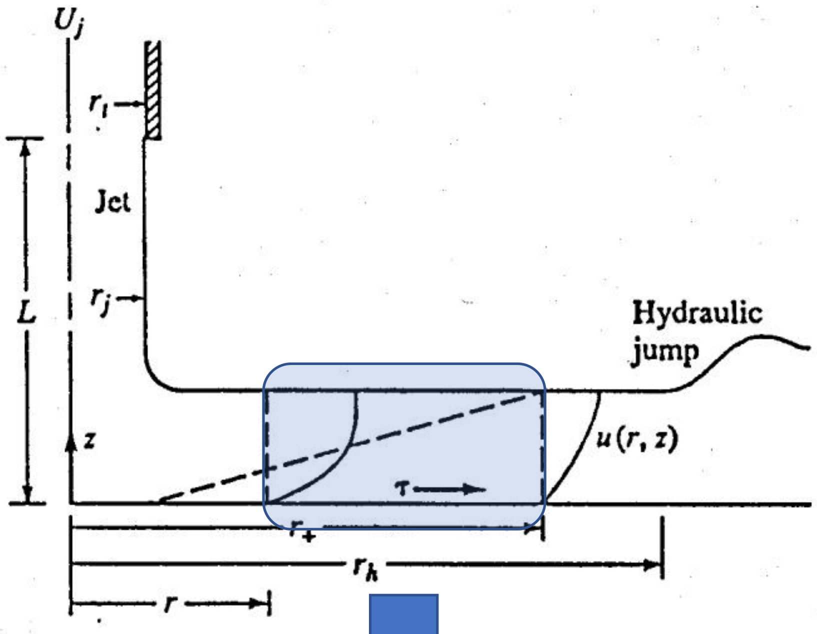
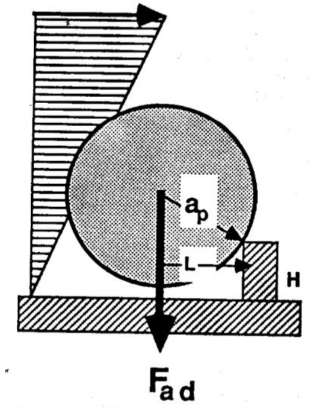
Superconductivity and its applications

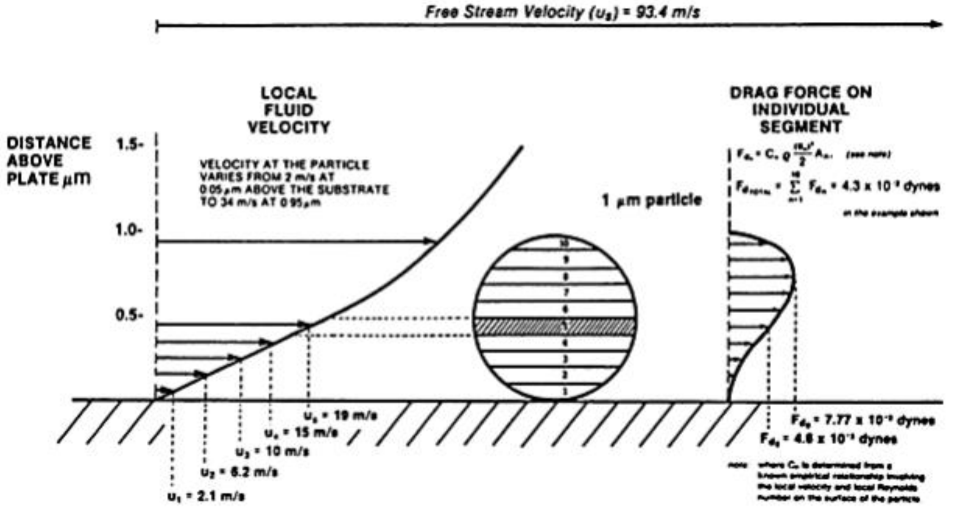
HPR Process

- Hydrodynamic model allows estimating the **shear stress τ of the water jet**, which depends on flow rate and pressure
- **Particle removal by rolling** if the water shear stress is greater than a critical shear stress τ_0, related to the particle size, adhesion force and surface roughness



$$\tau_0 = \frac{F_{ad}}{44\, a_p^2} \sqrt{2\frac{H}{a_p} + \left(\frac{H}{a_p}\right)^2}$$





https://doi.org/10.1063/1.4949242

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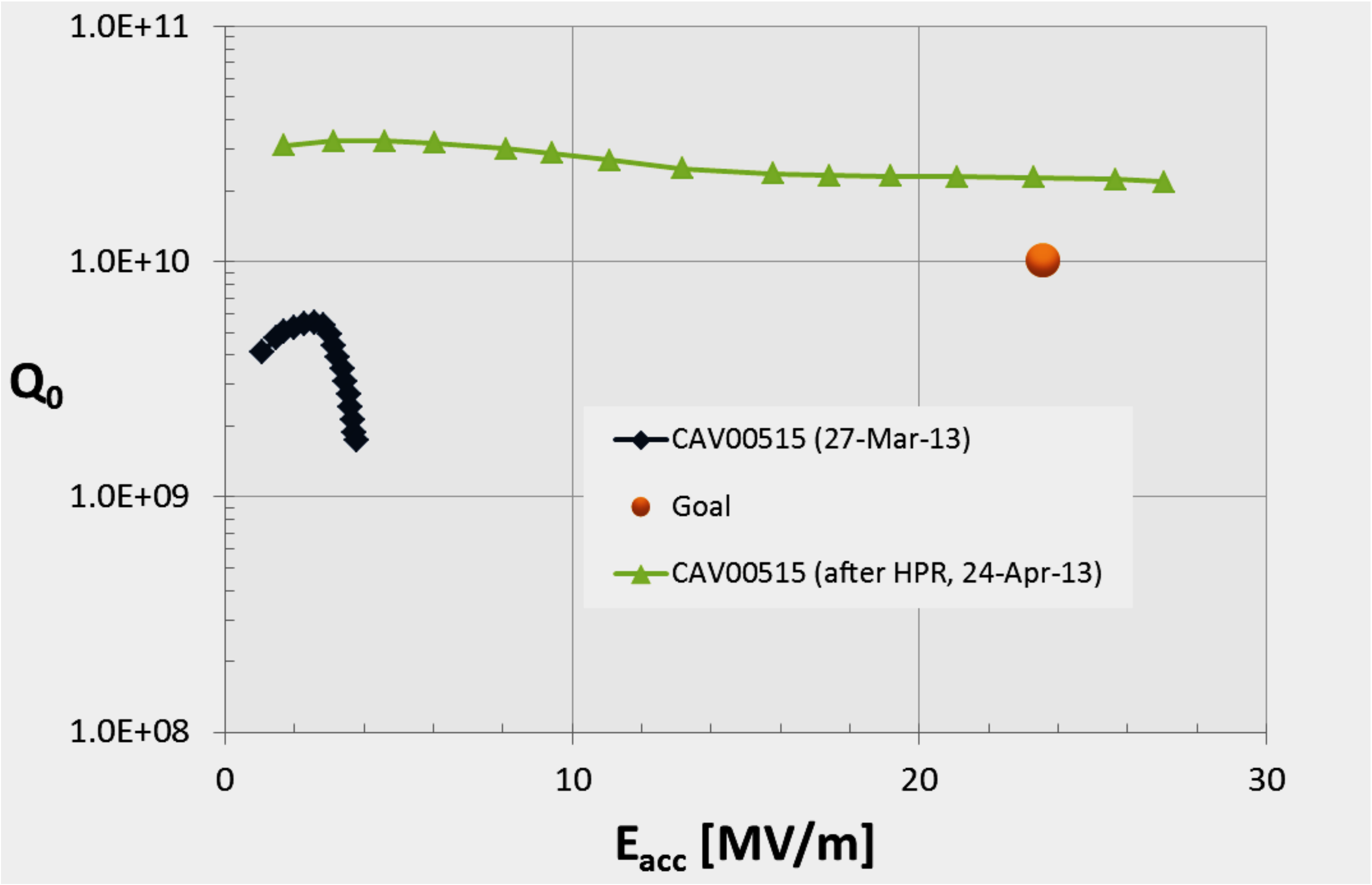
HPR Effect

- Cavity had a **problem** in the 120°C treatment (vacuum system power failure) that **produced a rapid change of pressure (a bump) in the cavity during last pump-down**, with particle movement in the system. Consequences are clearly visible (**dark blue curve**).

- **After HPR**, with **no further chemical etching**, cavity performances are completely recovered.



HPR Systems

Rinsing cabinet of
“old“ DESY HPR system

Rinsing cabinet of “new“ DESY
HPR system with “plastic” cavity

CEA HPR system

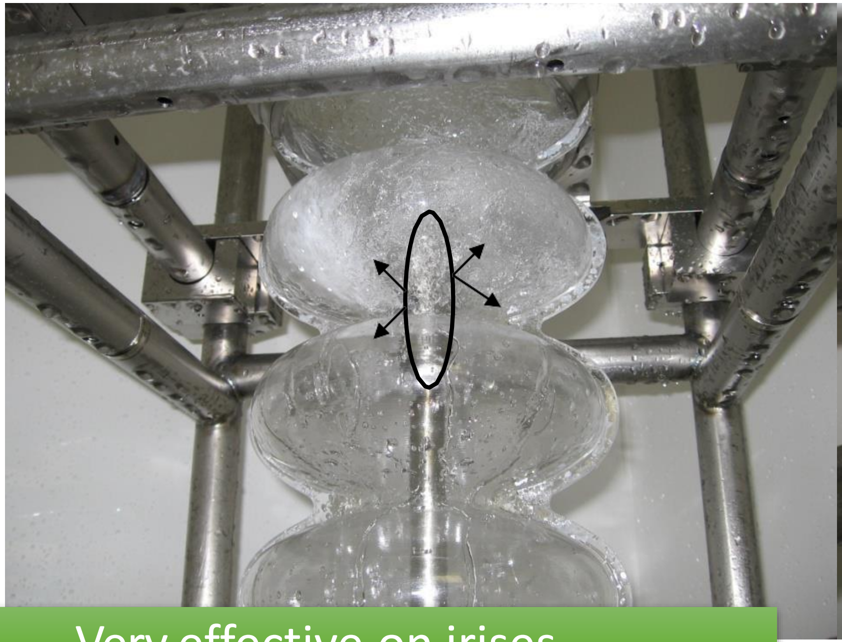
HPR QA

- **Examples of QC at HPR systems**
  (DESY, EXFEL cavities production@ companies)

- Check of Point-of-use supply water quality:
  - **UPW conductivity**
  - **Particles**: online particle counter
  - Particles: off-line sampling & identification (SEM optical microscope)
  - **TOC: online monitoring**
  - **TOC drain line: sampling, after maintenance**
  - **Bacteria (=> offline)**

HPR Spray Head Optimization

- For a given pump displacement **the nozzle opening diameter and number of nozzles sets the system pressure and flow rate**
- The **HPR spray head needs to be optimized for each cell geometry**!



Very effective on irises

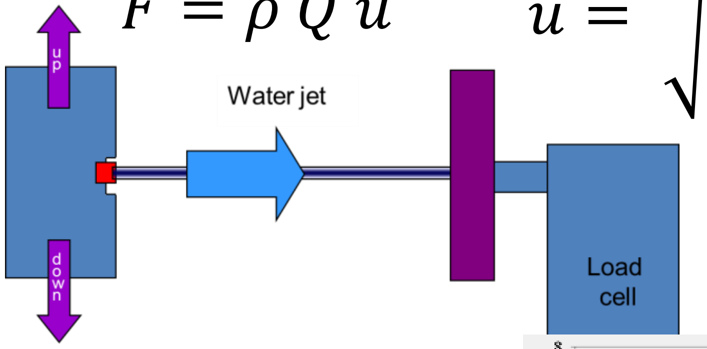
Equator fill with water ⟶ too high flow rate

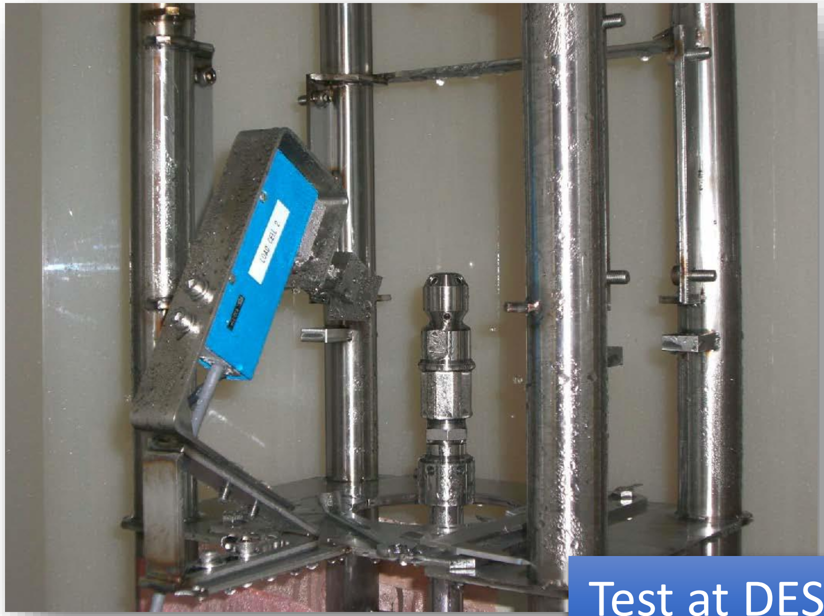
HPR Water Jet Characterization (INFN-LASA)

- Use a **load-cell to measure the jet force**

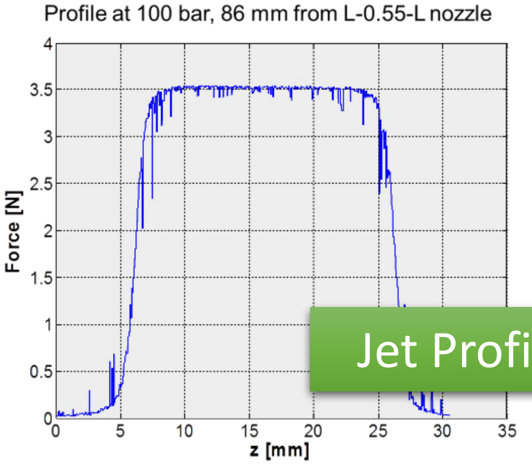
$$F = \rho\, Q\, u \qquad u = \sqrt{\frac{2\,p}{\rho}}$$

u = velocity
Q = flow
p = pressure
ρ = density

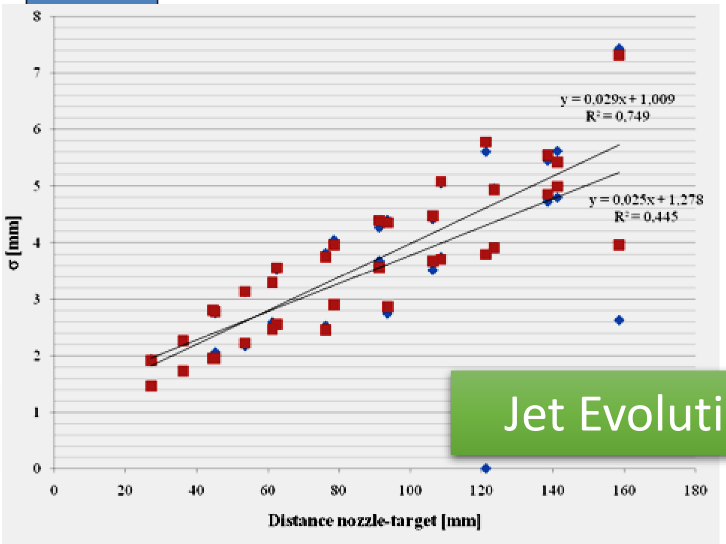




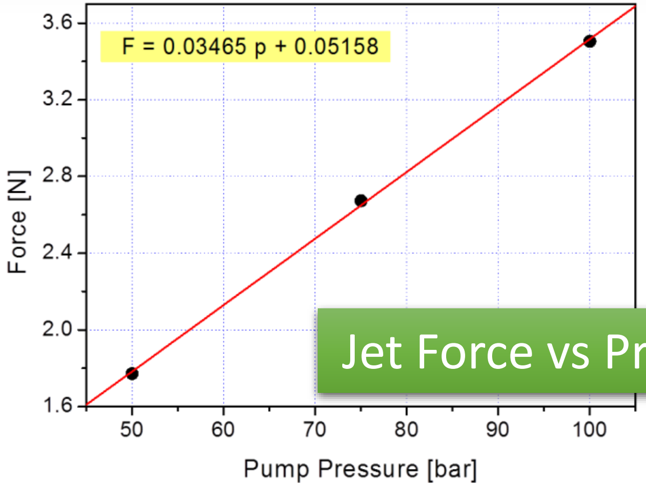
Test at DESY



Jet Profile



Jet Evolution



Jet Force vs Pressure

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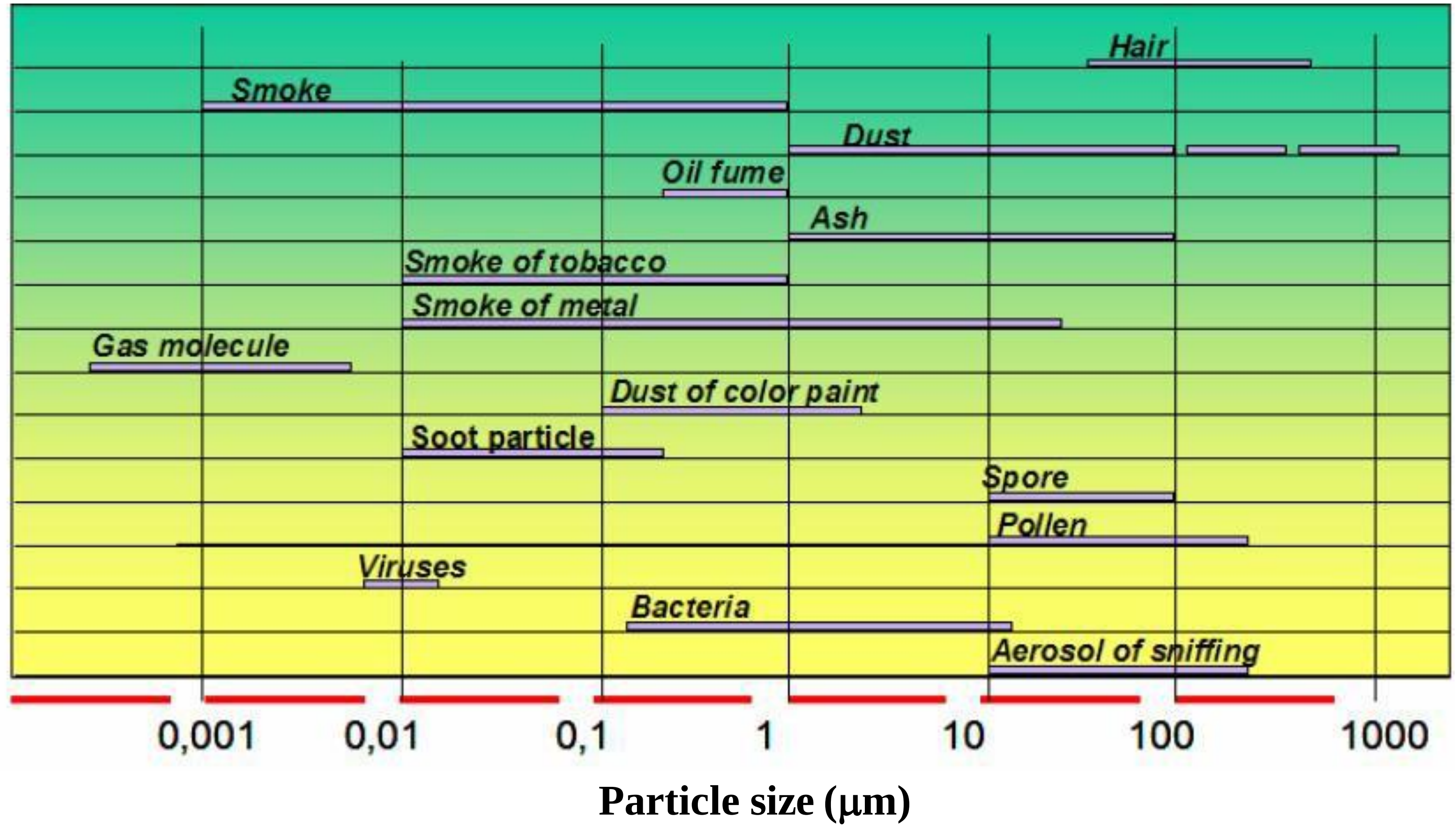
What is a cleanroom? The ISO 44644 definition

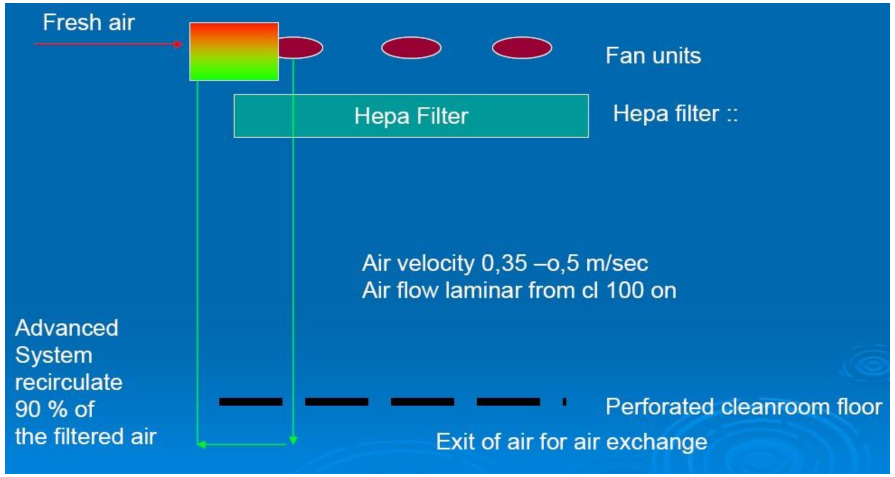
- "A room in which the **concentration of airborne particles** is **controlled**, and which is constructed and used in a manner to **minimize the introduction, generation and retention of particles** inside the room and in which other relevant **particles** inside the room and in which other relevant parameters, e.g. **temperature, humidity and pressure**, are **controlled as necessary**."
- **A cleanroom** is likely to have between **some tens of air changes per hour up to many hundreds of them.**
- A cleanroom uses filters that would normally be 99.97 % and more efficient in removing particles greater than 0.3 μm from the room air supply. These filters are known as **High Efficiency Particle Air (HEPA) filters, although Ultra Low Particle Air (ULPA) filters**, which have a higher efficiency, are used in microelectronic fabrication areas.

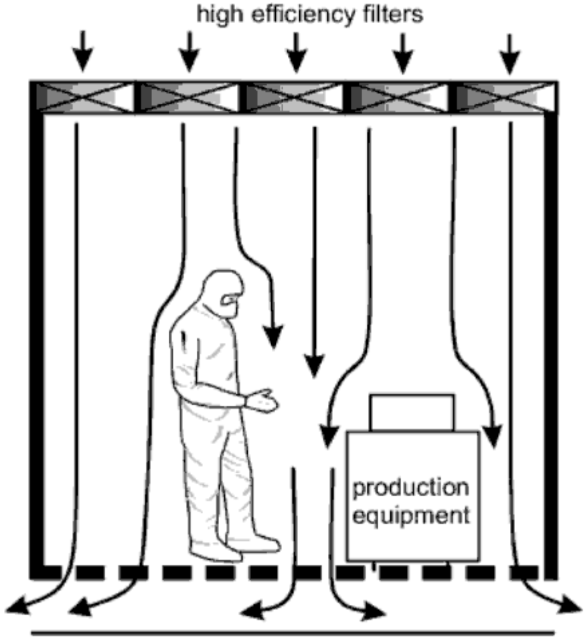
Particulate in air

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



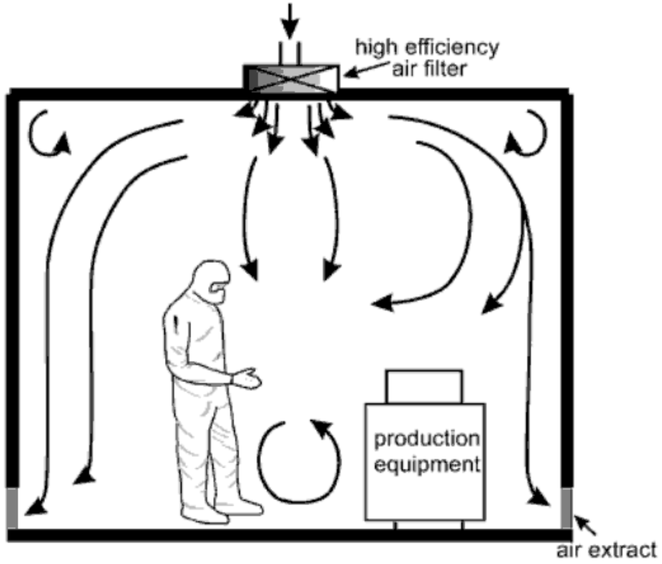
Type of cleanrooms

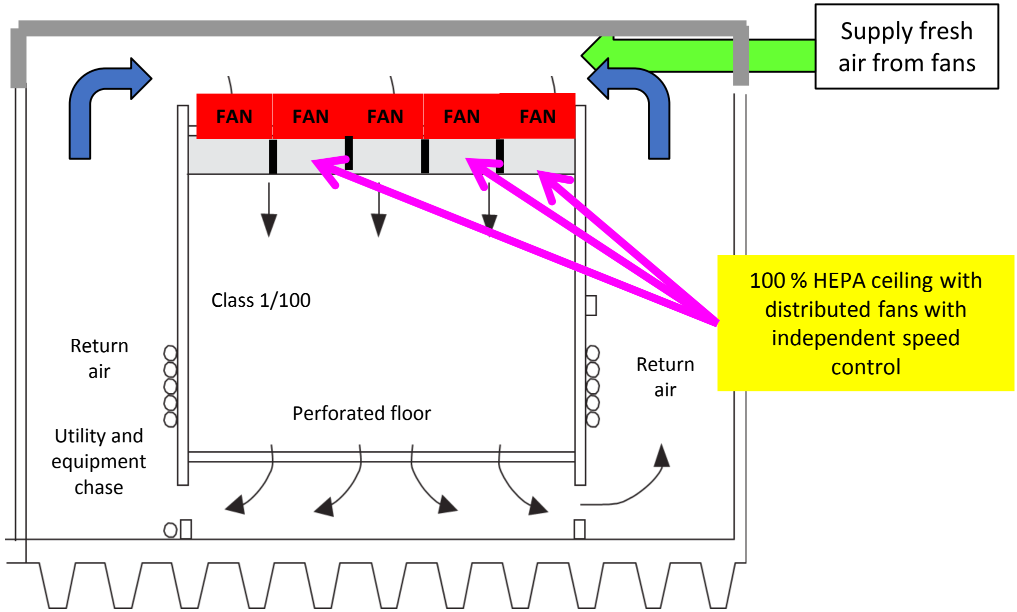




Unidirectional airflow type

Non-Unidirectional airflow type





Unidirectional airflow type with recirculation

Cleanroom classification

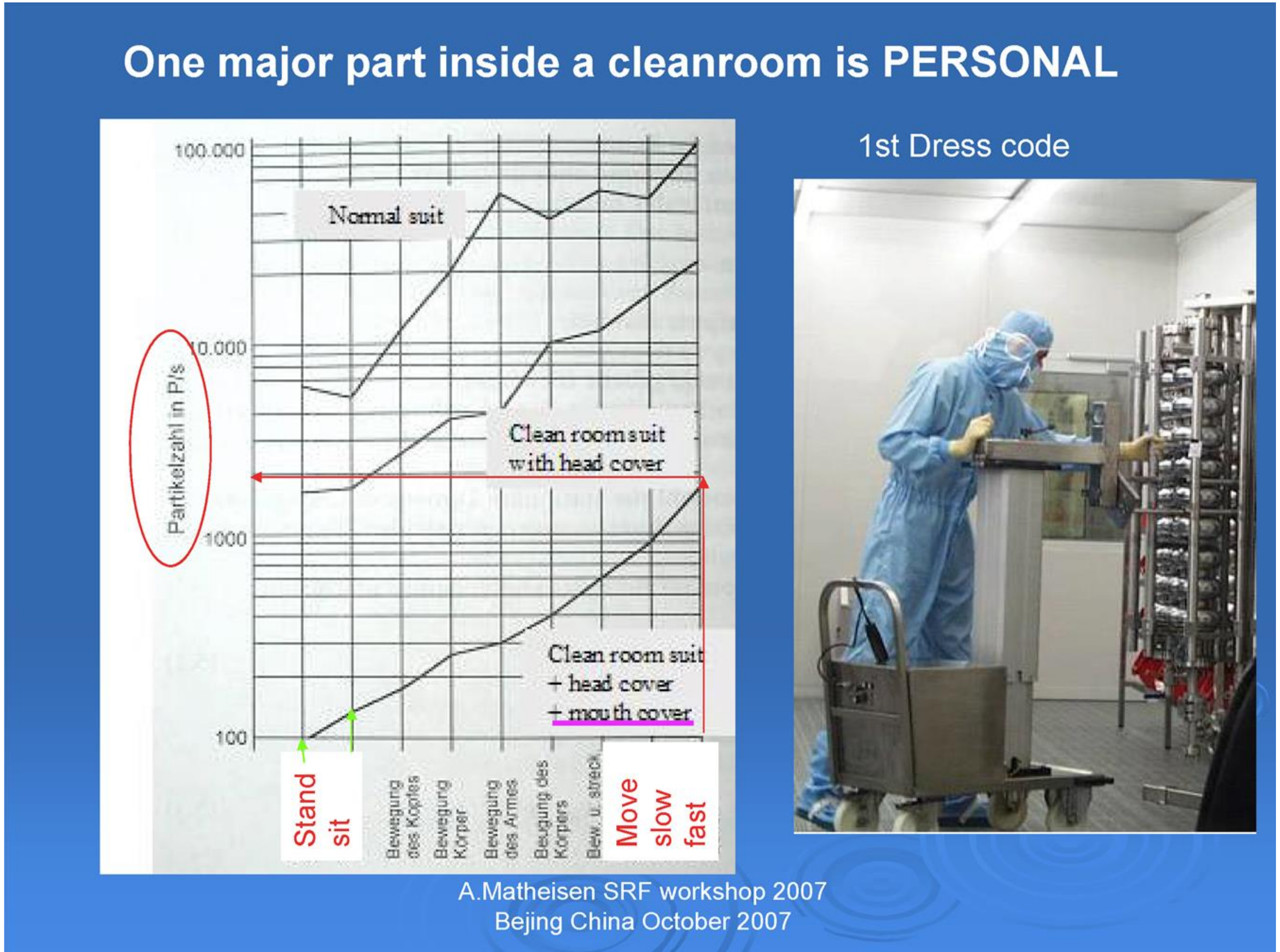
ISO Classification number	Maximum concentration limits (particles/m³ of air) for particles equal to and larger than the considered sizes shown below					
	>=0.1µm	>=0.2µm	>=0.3µm	>=0.5µm	>=1µm	>=5.0µm
ISO Class 1	10	2				
ISO Class 2	100	24	10	4		
ISO Class 3	1 000	237	102	35	8	
ISO Class 4	10 000	2 370	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

ISO 14644-1 Classes	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8
FS 209 Classes	Class1	Class 10	Class 100	Class 1000	Class 10, 000	Class 100, 000

Cavity assembly

Cleanroom for SRF

Human generated particle

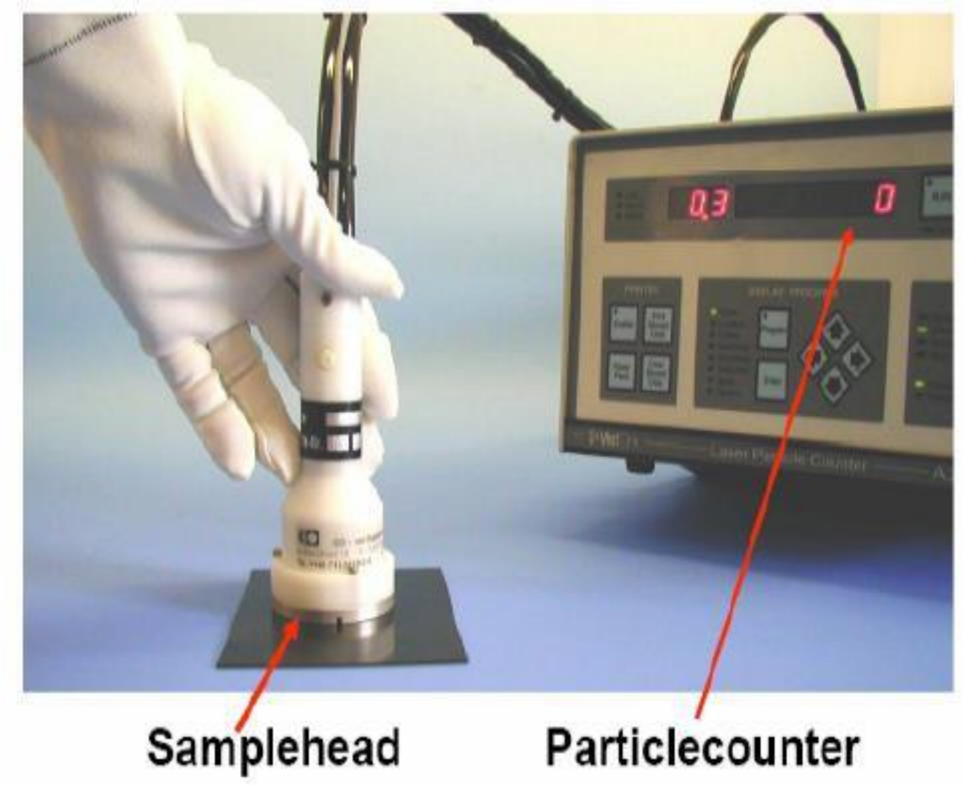


Clean room «dress» code

- **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.

Particle counters

- To ensure the proper cleanness, **all components** before installation need to be **washed, rinsed and particle counted**



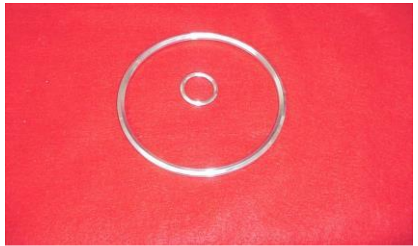
Cavity Assembly

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

- UHV Gasket
  - Present choice for SRF cavities:

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

AlMg-Gasket



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- Clean assembly
- **Clean evacuation**
- Low-temperature baking

Slow Pumping – Slow Venting

- **Avoid particle transport** from outside into the vacuum system **and avoid movement** of particles already in the vacuum system

- **For abs. pressure p > 1 mbar and differential pressure Δp > 1 mbar** (e.g. opening of valves, start pumping)
  => movement of particles observed

- **For abs. pressure p < 1 mbar => no movement of particles observed**

- **Manual dosing valves cannot safely avoid particle transport**

SPSV motivation

Both rapid venting and rapid pumping introduce particle motion

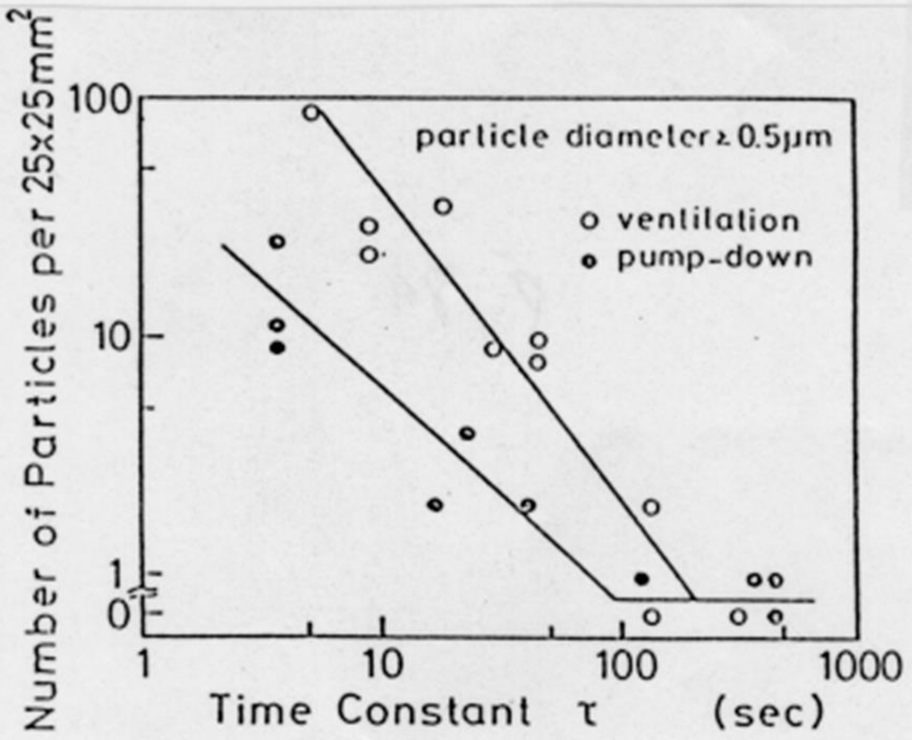

Fig.3 Increment in number of dust particles after each ventilation and pumpdown, is plotted versus the time constant τ.

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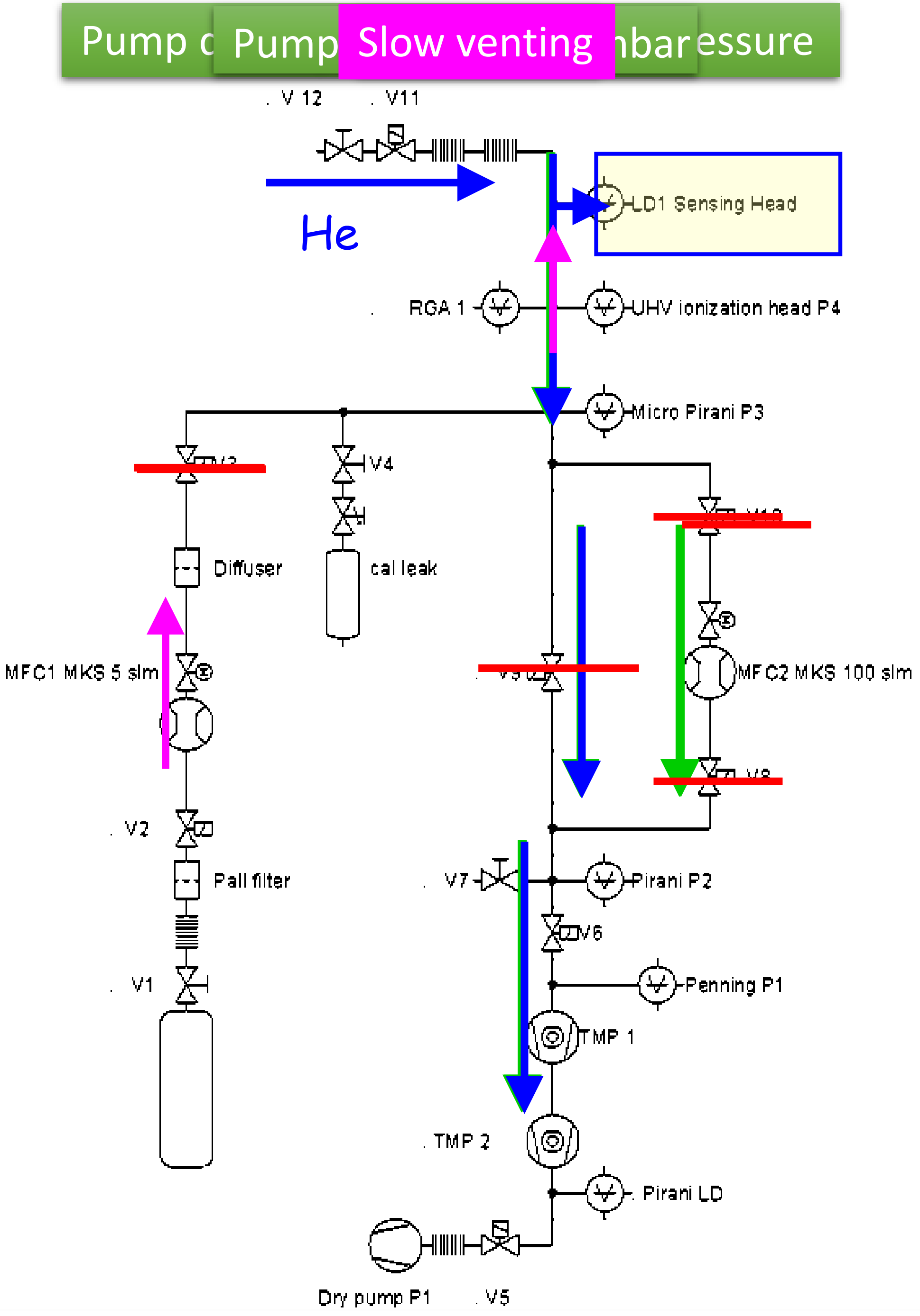
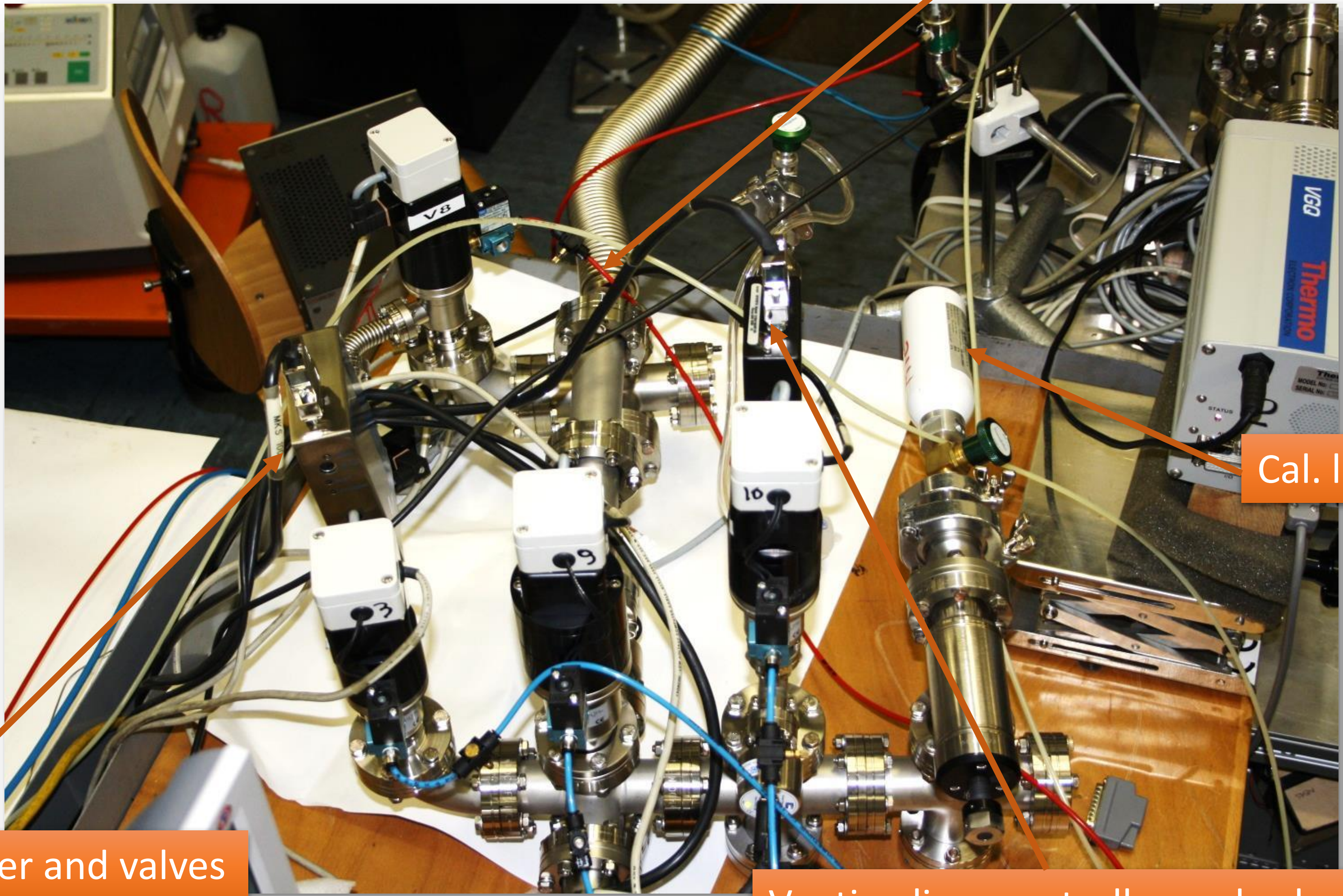
SPSV at INFN LASA

- Dry pump 1: scroll pump, Triscroll 600
- TMP1: Pfeiffer TMP
- TMP2: TMP of the LDS1000 Oerlikon LD
- LD1: LDS1000 Oerlikon Inficon sensing head
- V6, V8, V9, V10, V3: Varian Viton seal on the bonnet,
  metal seal on the body
- MFC1: MKS, 5 slm flow controller
- MFC2: MKS, 100 slm flow controller



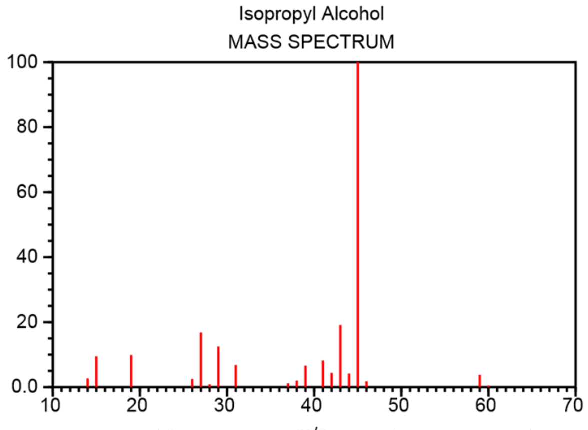
EASITrain

SPSV at LASA

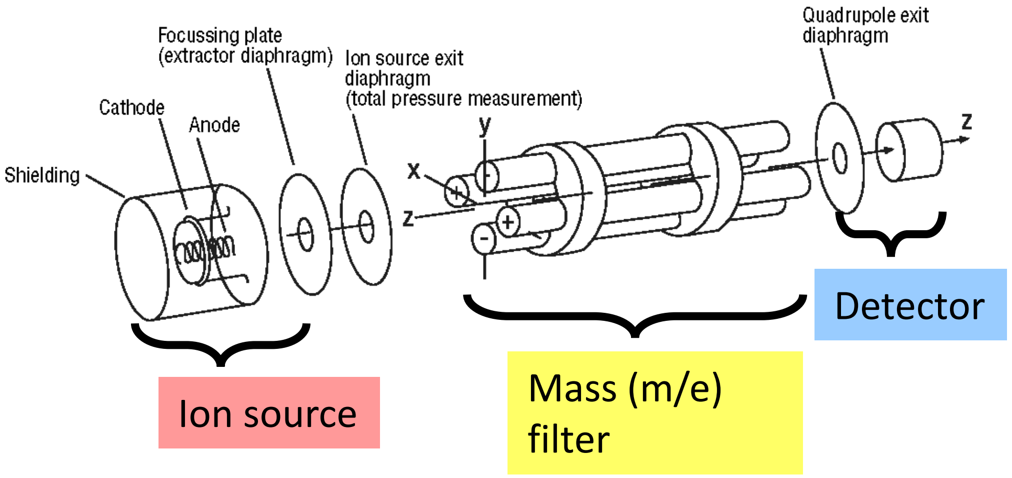

Connection to the TMP
Cal. leak
Pumping line: controller and valves
Venting line: controller and valves

Residual Gas Analysis (RGA)

- UHV partial pressure measurement system used for:
  - **Partial pressure evaluation of various gases**: e.g. for discriminate water desorption from leaks and outgas
  - **Vacuum quality check**
    - Leak check
    - Impurities and contaminants evaluation: specific fingerprint are available for some components (for instance for isopropanol, acetone, ethanol, etc.)
- Evaluation of Hydrocarbon content



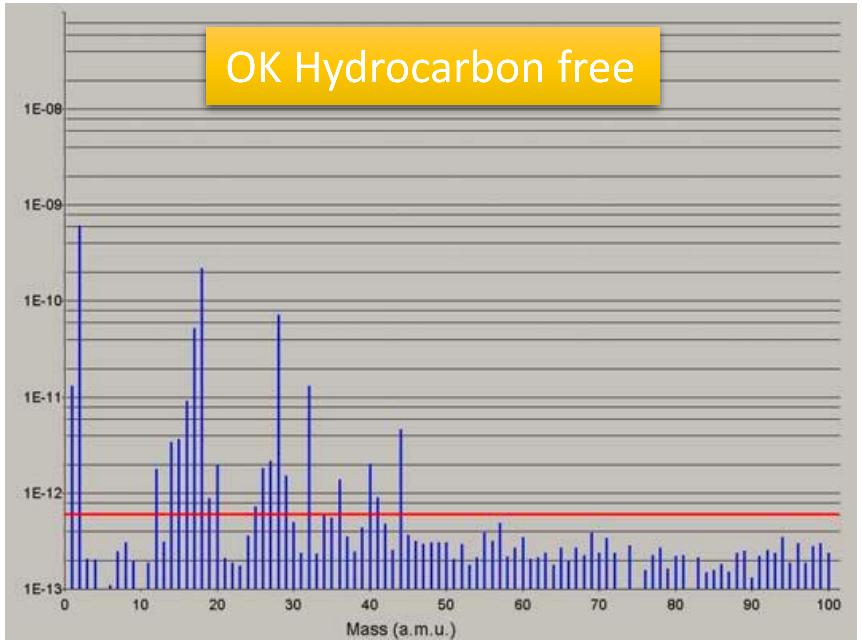
(from NIST, http://webbook.nist.gov/chemistry/name-ser.html).

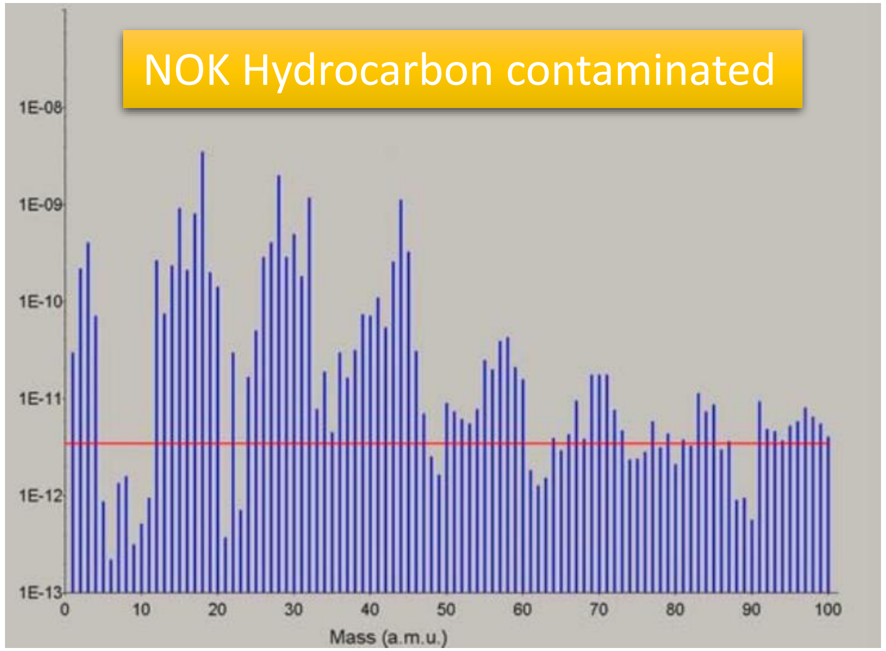


Definition of hydrocarbon free parts for EXFEL

- Appropriate proofs have to be performed using a sufficiently sensitive residual gas analyzer, usually equipped with a secondary electron multiplier (SEM).

- Components are considered **free of hydrocarbons if** in a leak-free system with a total pressure **below 10^{-7} mbar the sum of the partial pressures of masses above mass 45 is less than 10^{-3}** of the total pressure (**1 : 1000**).



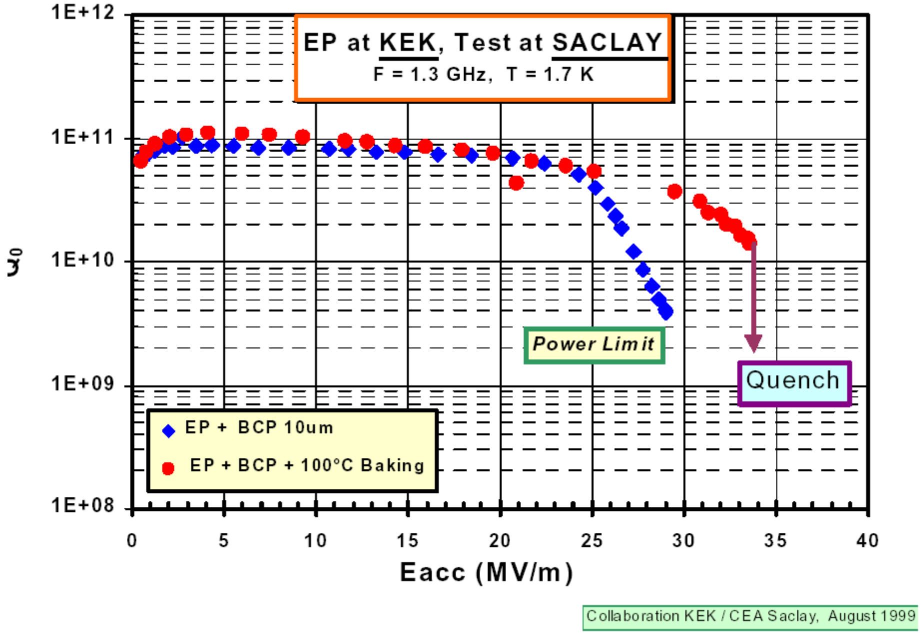


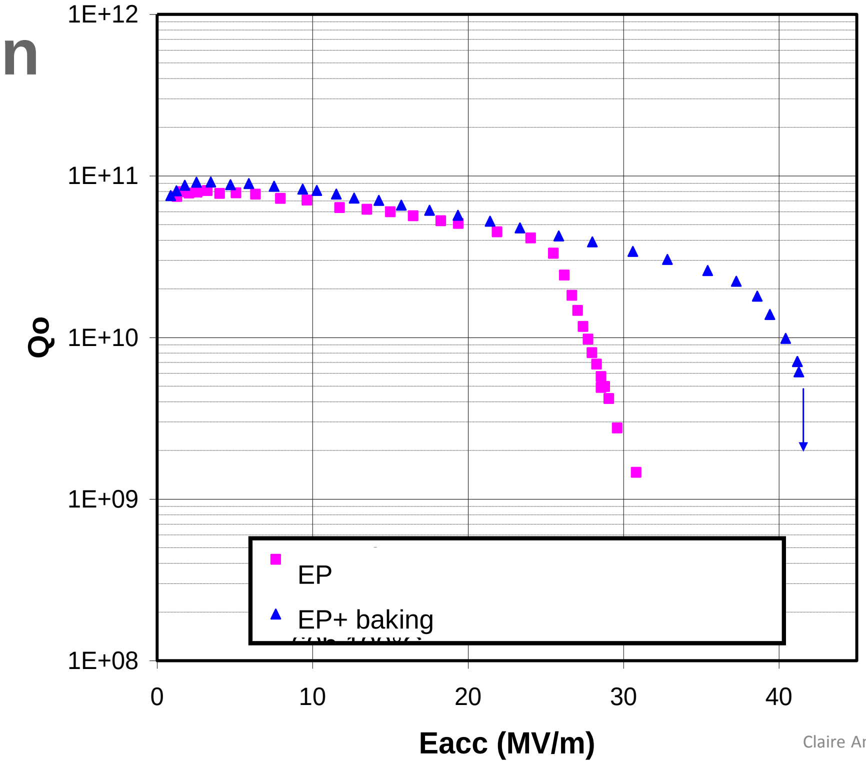
Cavity preparation for SRF qualification

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - 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

Why low temperature baking (120 °C)?

- **Baking:** shifts high field dissipation to higher field
  - Discovered at Saclay in 1998 (B. Visentin)
  - Low temperature treatment : 110-120°C, 48 H : few changes expected
  - **Dramatic effect on performances**
  - **Still resists full explanation**

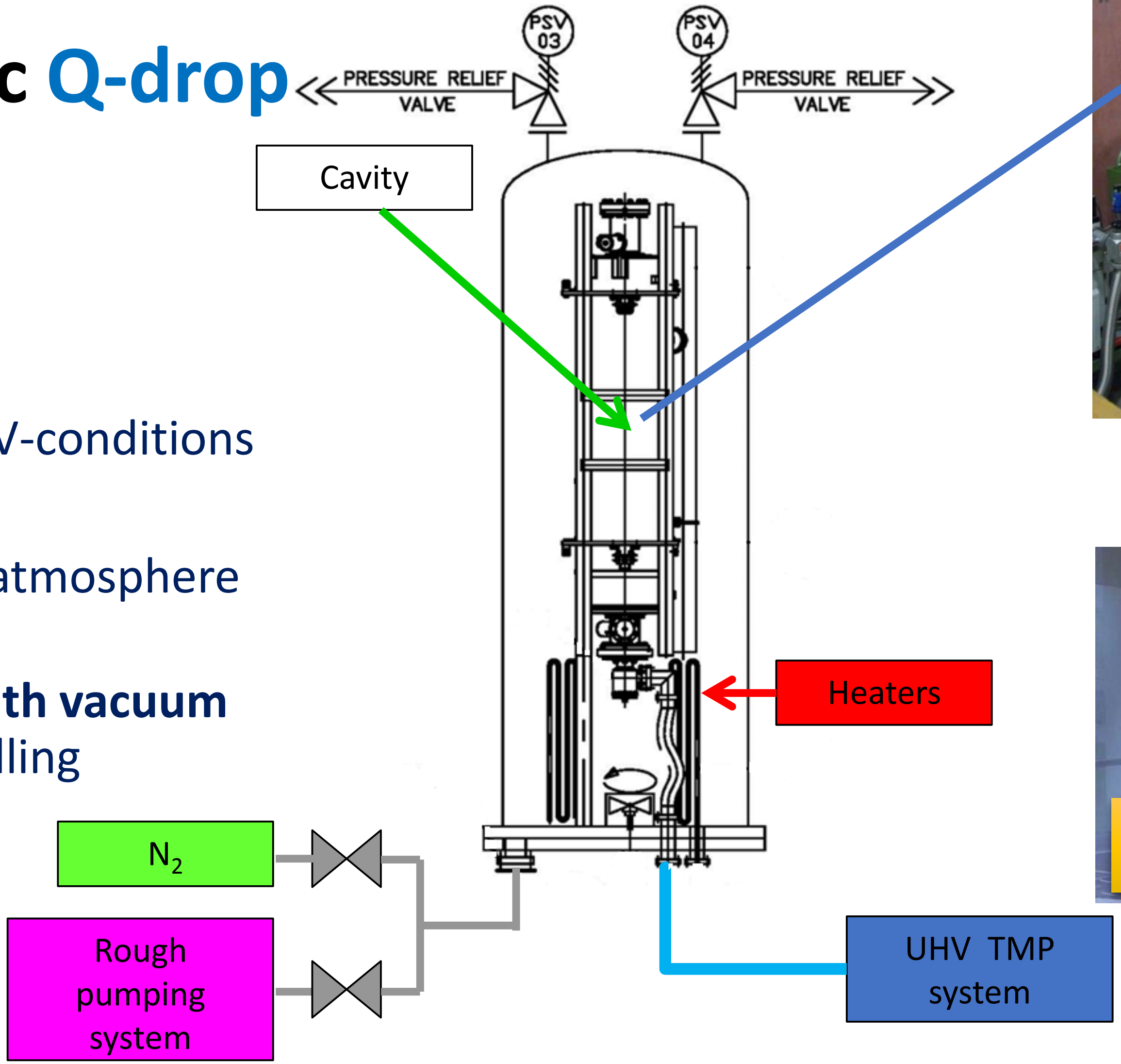




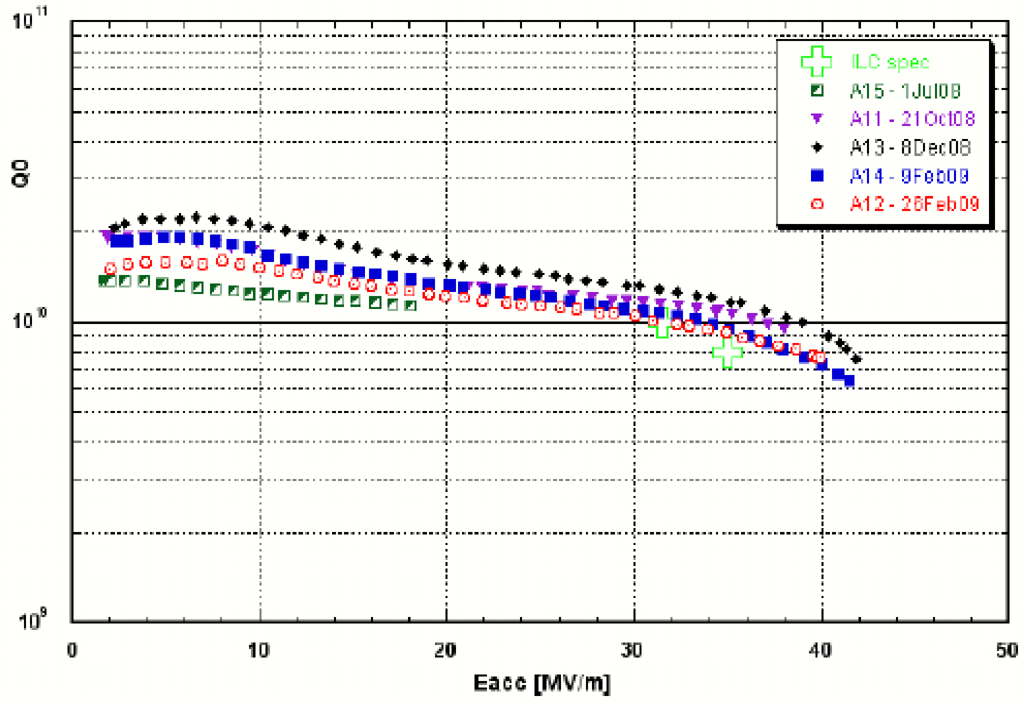
120 °C baking

Cure of characteristic Q-drop

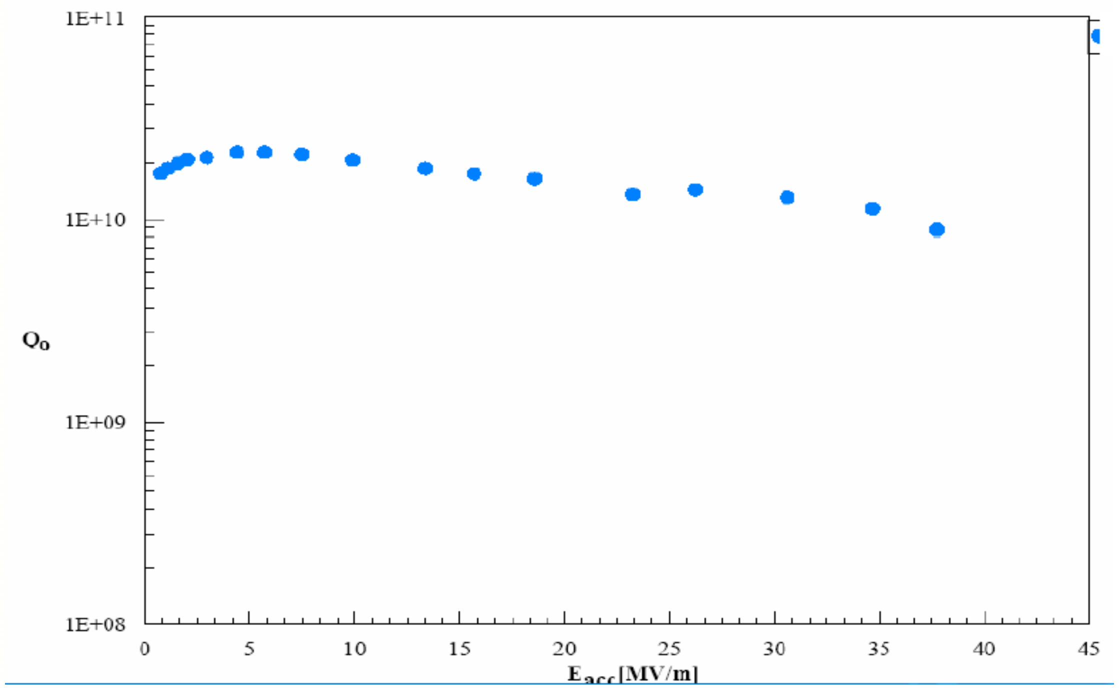
- **Standard recipe:**
  - **T = 110 - 125°C for 48h**
  - **Active pumping** (TMP)
  - Oil free vacuum system, UHV-conditions
  - Fully assembled cavity
  - **Nitrogen or argon** external atmosphere to avoid oxidation
  - External volume: **purging with vacuum before** inert gas (N_2 or Ar) filling



If everything went well you will get …



JLAB



DESY

$E_p \cong$ 80 MV/m, $B_p \cong$ 170 mT can be achieved in the vertical test of 9-cell ILC cavities (~ 1 m² of Nb surface)

Thanks!!

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- In general all **Proceedings of:**

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