Cavity Processing: EP/BCP, heat treatments, baking and clean room techniques

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GANIL
SRF13 Tutorials, September 2013.

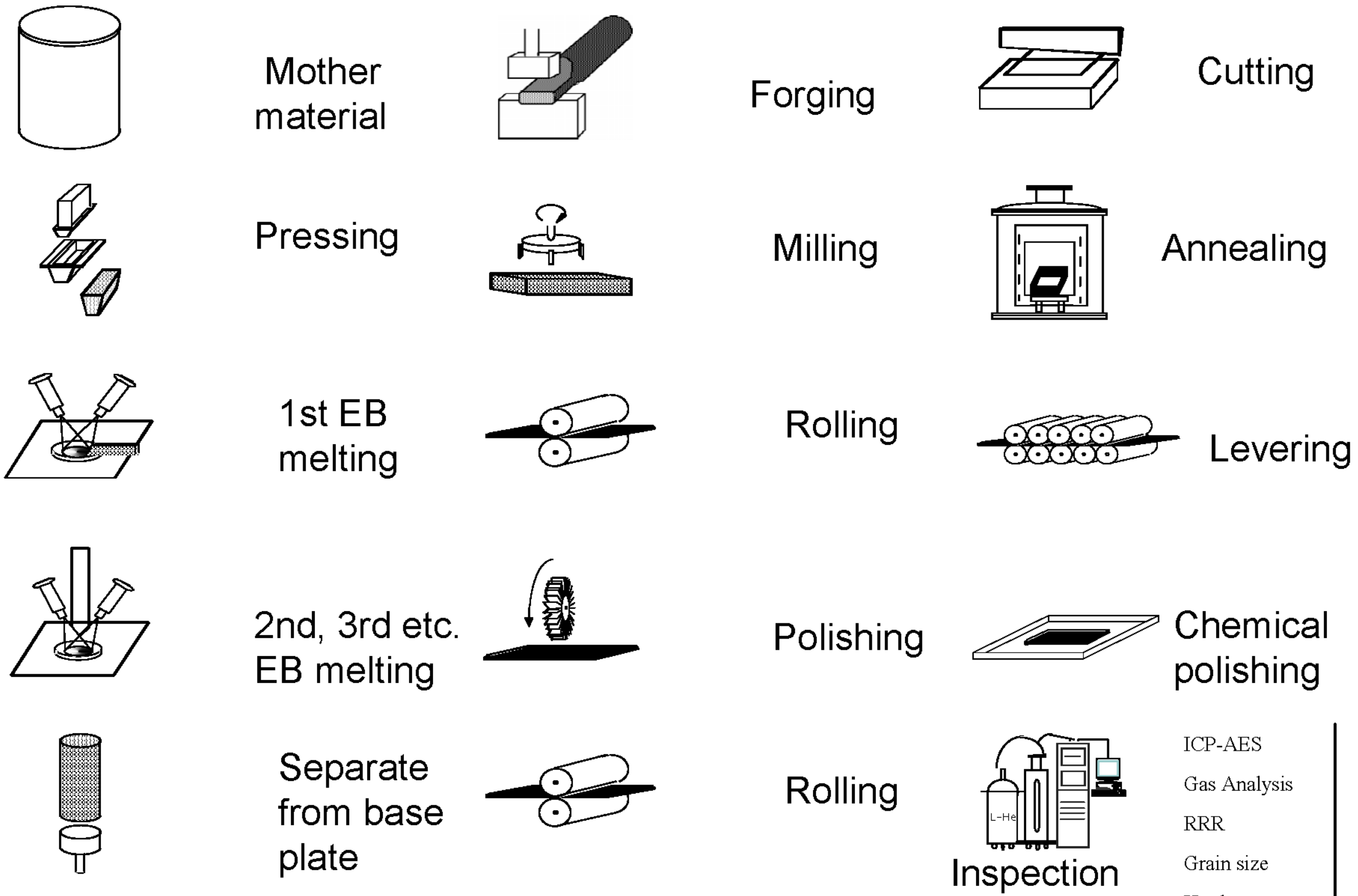
Niobium

Niobium (Nb) (Tc=9.2 K; superheating field of approx. 240 mT) is the favorite material for the fabrication of superconducting RF cavities.

- chemically inert (pentoxide layer)
- easily machined and deep drawn
- available as bulk and sheet material
- majority of superconducting RF cavities worldwide are formed from Nb sheet material

Nb sheet production

Fabrication of Nb sheets at Tokyo Denkai



Mother material

Forging

Cutting

Pressing

Milling

Annealing

1st EB melting

Rolling

Levering

2nd, 3rd etc. EB melting

Polishing

Chemical polishing

Separate from base plate

Rolling

Inspection

ICP-AES

Gas Analysis

RRR

Grain size

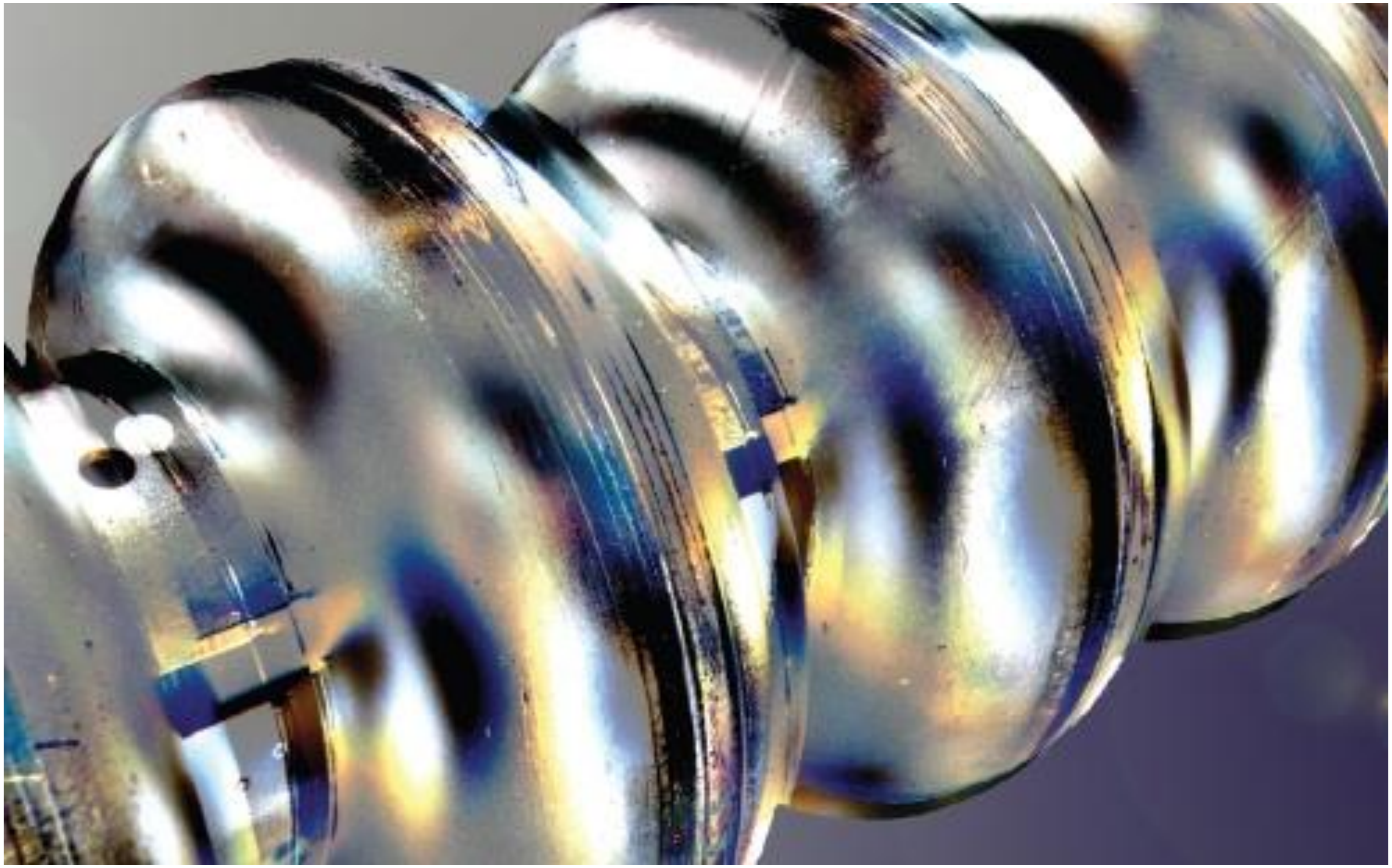
Hardness

Tensile tests

In the final sheet the purity of niobium should be not inferior as in the ingot

W. Singer. Tutorial 4a2. 13th International Workshop on RF Superconductivity, October 15-19, 2007, Beijing, China

Cleanroom technology for SRF applications

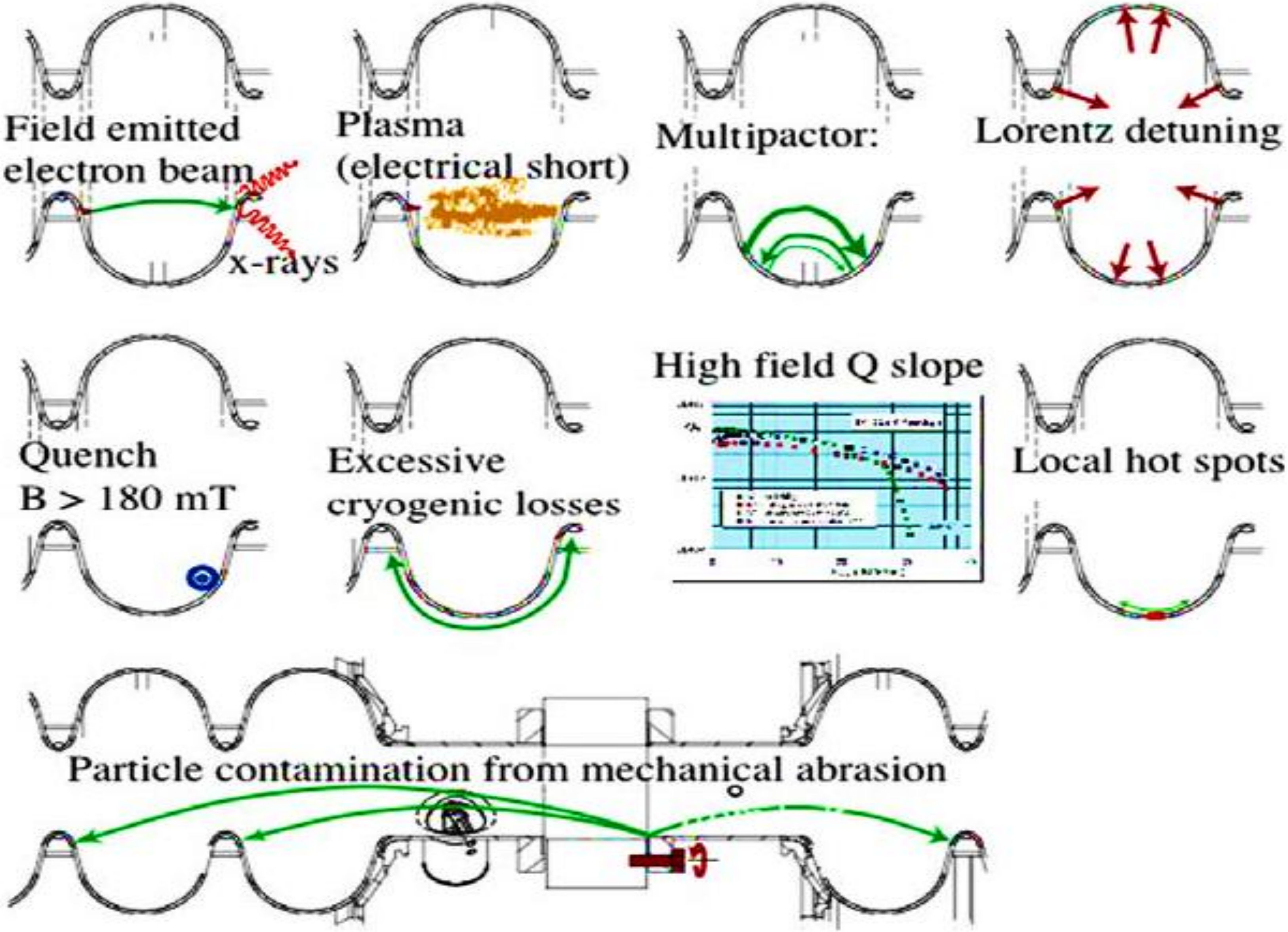


SC cavities production: a long chain, but…

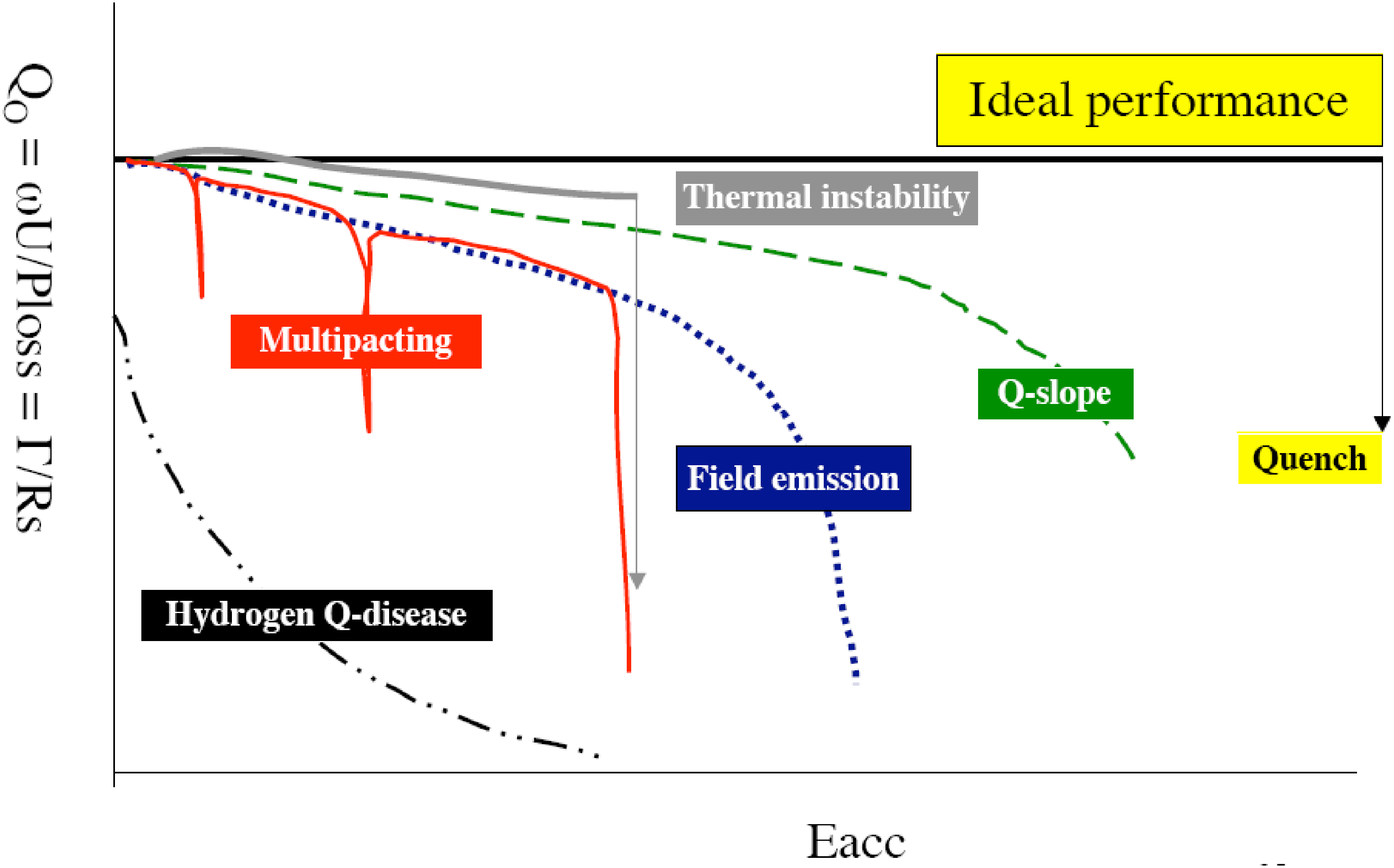
>A chain is as strong as its weakest link !!!

- **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

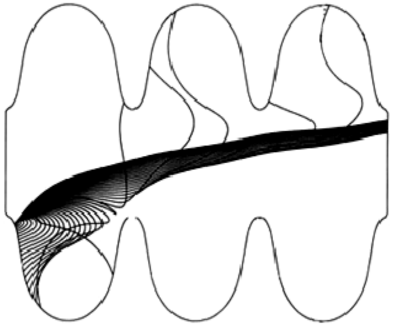
Present picture of field emission: observations

- **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
- **Sulfur** contamination after electropolishing process
- Modified Fowler-Nordheim's law :

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

- 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

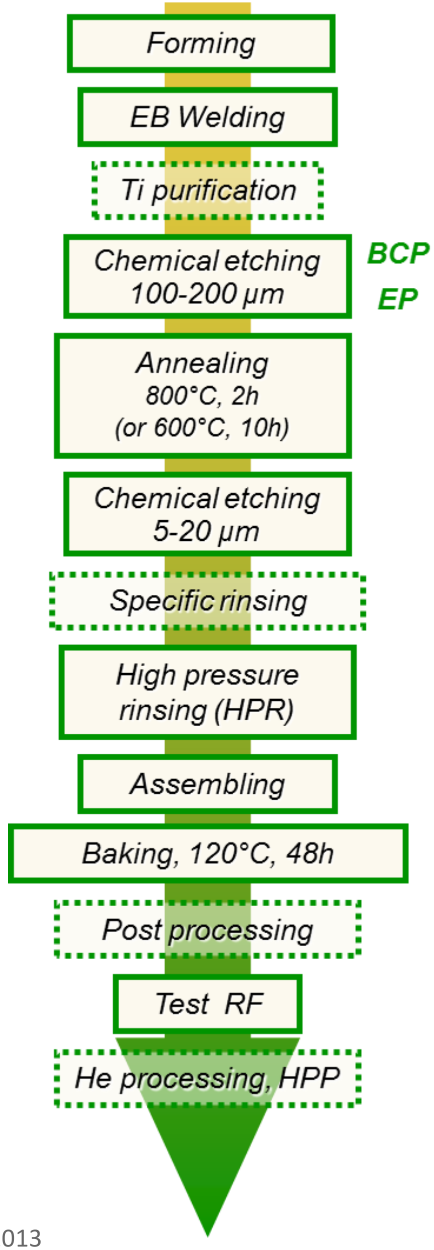


- **Field Emission is critical,** more for multi-cell cavities for high Rf acc. fields
- P. Kneisel + B. Lewis, SRF Workshop 1995:

  "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**

Cavities’ general fabrication scheme



	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*

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Process environment

The cleanliness of environment must match to process

- Do not make **excesses in both directions**:
  - **too clean** environment for a **“dirty”** step of the procedure
  - steps that **have to be “clean”** done in a non adequate **“dirty” area**.
- What could be the effect?
  - Procedure failure and/or many defect (for instance FE)
  - High investment without results (!maintenance!)

Can be both !

Some examples

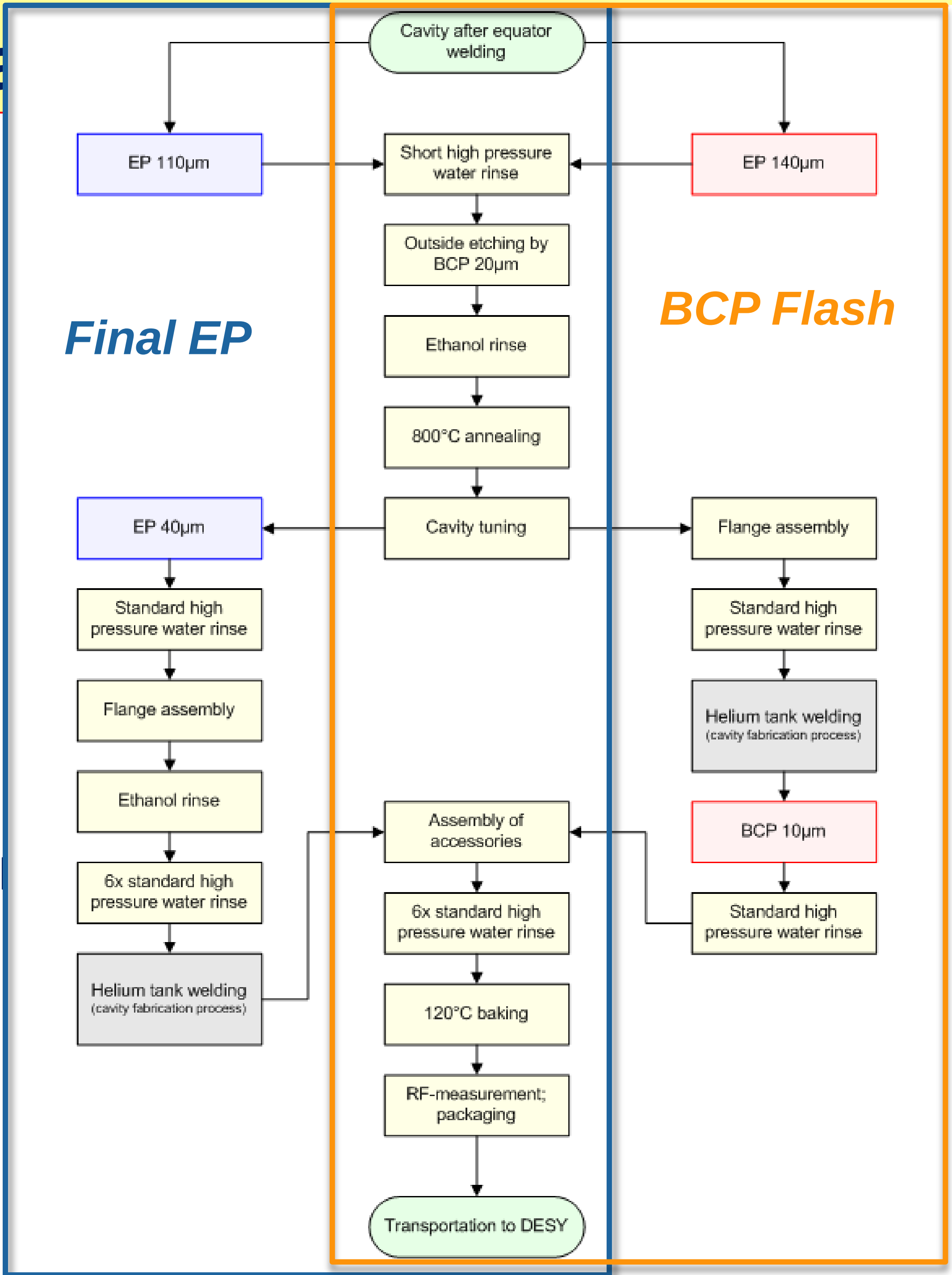
- Cavity fabrication => Workshop, Lab, cleanroom
- Furnaces, tuning apparatus => Lab
- He-tank welding => Lab, workshop
- 120 °C bake => Lab, with vac conn in ISO 5
- Cleaning facilities => Lab, with airlocks Lab to ISO 4
- Chemical treatment => Lab / ISO 6/7
- HPR, assembly, final leak check => ISO 4/5

Nb surface preparation: some general rules

- **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 a step in which 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 surface prepa

- European XFEL surface preparation schemes:
- EP scheme (similar to ILC):
    - 110 µm + 40 µm removal
    - He-tank welding **after** final surface treatment
- “BCP Flash” scheme:
    - 140 µm EP + 10 µm BCP
    - He-tank welding **before** final surface treatment
    - Results in less handling + preparation steps



Final EP

BCP Flash

Cavity after equator welding

EP 110µm

Short high pressure water rinse

EP 140µm

Outside etching by BCP 20µm

Ethanol rinse

800°C annealing

Cavity tuning

EP 40µm

Standard high pressure water rinse

Flange assembly

Ethanol rinse

6x standard high pressure water rinse

Helium tank welding (cavity fabrication process)

Flange assembly

Standard high pressure water rinse

Helium tank welding (cavity fabrication process)

BCP 10µm

Standard high pressure water rinse

Assembly of accessories

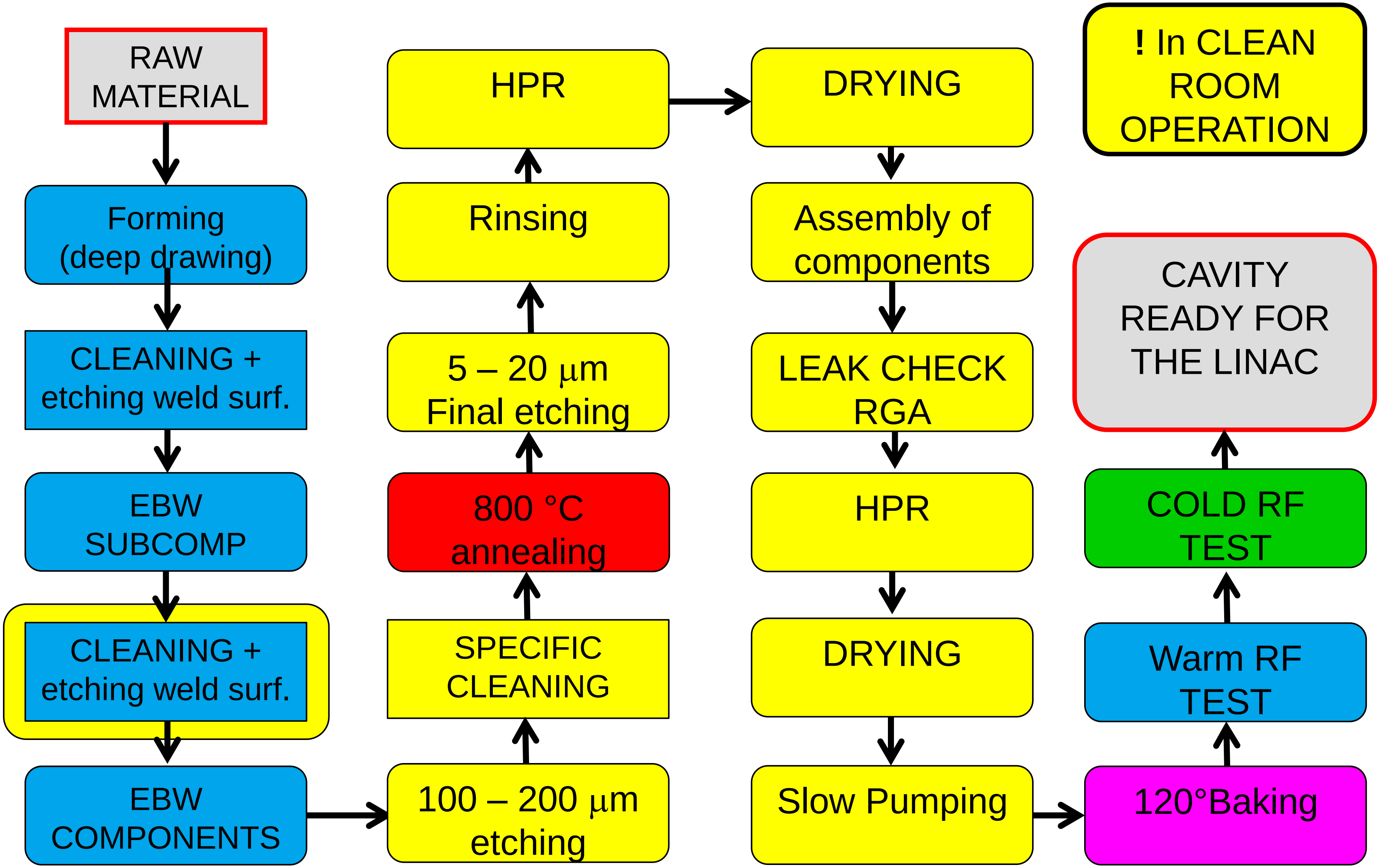
6x standard high pressure water rinse

120°C baking

RF-measurement; packaging

Transportation to DESY

From raw material to RF cavity for EXFEL



RAW MATERIAL

Forming (deep drawing)

CLEANING + etching weld surf.

EBW SUBCOMP

CLEANING + etching weld surf.

EBW COMPONENTS

100 – 200 μm etching

SPECIFIC CLEANING

800 °C annealing

5 – 20 μm Final etching

Rinsing

HPR

DRYING

Assembly of components

LEAK CHECK RGA

HPR

DRYING

Slow Pumping

120°Baking

Warm RF TEST

COLD RF TEST

CAVITY READY FOR THE LINAC

! In CLEAN ROOM OPERATION

Outlook

1. Cleaning and rinsing
2. Ultra pure water production
3. High pressure rinsing
4. Ultra pure gases
5. Preparation of the inner surface for RF: grinding, tumbling, BCP, EP
6. HF acid safety rules
7. Ethanol rinsing
8. High temperature (800 °C) dehydrogenation treatment
9. 120 °C final bake out
10. Vacuum
11. Clean room

Cleaning and rinsing

- All **water** based treatments, **similar to UHV cleaning**, using **detergents** (for EXFEL Tickopur **TR33**, 1 % to 3 % in UPW)
  - Seldom in case of strong surface contamination, e.g. after deep drawing, **solvents as acetone or ethanol** could be used for a first rough degreasing
- First step (as for ISO7 entering) of **small component**s: dish washer machine, hot water (t < 60 °C) + detergent
- First step (as for clean room ISO7 entering) of **large components** as cavities: car wash (high pressure UPW rinsing) with or w/o detergent
- Fine cleaning (as for **ISO4 entering**): **using detergents and US**

Cleaning process: surfactants

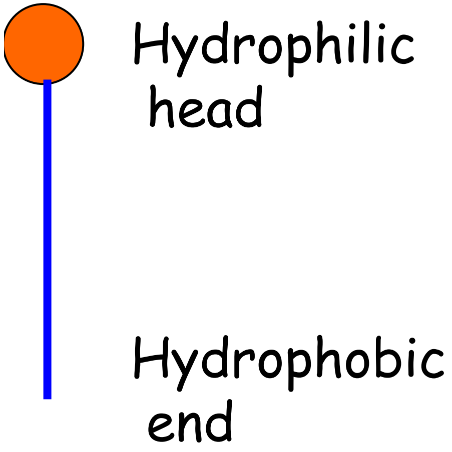
A detergent can wet any surface, it is a **Surfactant**. Amphiphilic molecule with polar head and non polar tail, soluble in water and organic solvents, can incorporate the hydrophobic material which can thus be dissolved (formation of micelles).

Surfactant: agents that reduce liquid surface tension favoring the wettability of surfaces or the miscibility of different liquids.

Detergent attack on soil

lifted, suspended and dispersed

hydrophillic and hydrophobic ends



Water based cleaning procedure: comments

- **Generally more effective than solvents**: solvents only diluted contaminants. **But solvents can diluted also silicone grease**!
- Can be used for non-porous materials and parts of simple shape, **which can be properly rinsed/dried**
- **pH is not neutral**, usually >> 7, surface can be oxidized, surface oxides and some alloys (brazing, NEG) can be slightly etched. Acid water based detergent (citric acid) are also available.
- **Silicones and Fomblin** (perfluoropolyether) are **NOT GREASE** and it is hard to remove using water based solution!
- **Time and ultrasound power is function of contamination amount, part shape, etc.**
- Bath quality **must be monitored** (conductivity, pH, concentration of detergent to be effective) and have to be renovated frequently.

Water break test

- **Immersion of components in UPW, NOT surfactants residuals on pieces!**
- **No surfactant residuals in UPW.**
- Piece must be completely wetted and the water film **MUST NOT BREAK** in any place
- High sensitivity for hydrocarbon contamination (hydrophobic contaminants)
- **Norm: ASTM F22 - 02(2007) Standard Test Method for Hydrophobic Surface Films by the Water-Break Test. Pieces are contaminated with a standard solution of fat material dissolved in acetone.**

Water break test

Not efficient cleaning

After good US cleaning procedure

Large components rough cleaning : car wash

Before entering clean room

- 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)

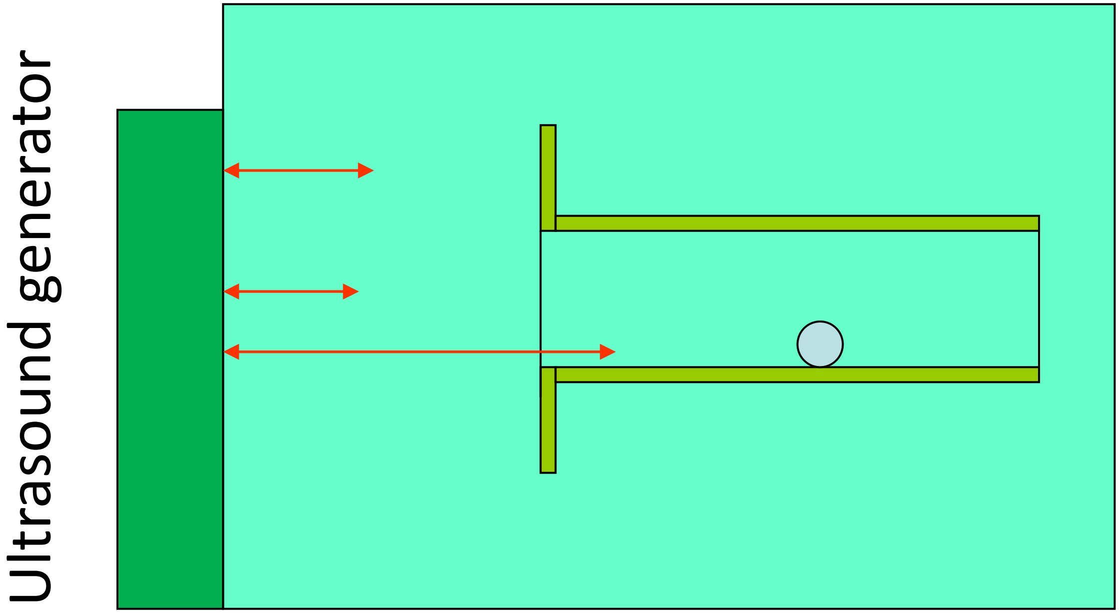
Small components rough cleaning: dish washer

- **Water: UPW (ρ >10 MΩ·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

Fine cleaning and rinsing: US bath + UPW rinse

- **Fine cleaning** (i.e. for EXFEL): ultrasonic cleaning + UPW rinsing
- Ultra pure water (18 MΩ·cm) + alkaline detergent (Tickopur), concentration 1 - 3%, @ 45 °C – 60 °C, duration about 20 min: **it removes grease, particulates, residues from former treatments**
- **Ultrasound basin**, some hundreds of liters, **US power 10 W/liter**
- **Component MUST be fully immersed, with no air bubbles inside. NO partial or two step (top and bottom) cleaning accepted!**
- **Rinsing with UPW, continuous flow**
- Detergent for large production should be **changed** many times a week (XFEL: once / 1 – 2 days)
- In many cases automatic (PLC controlled) process are used

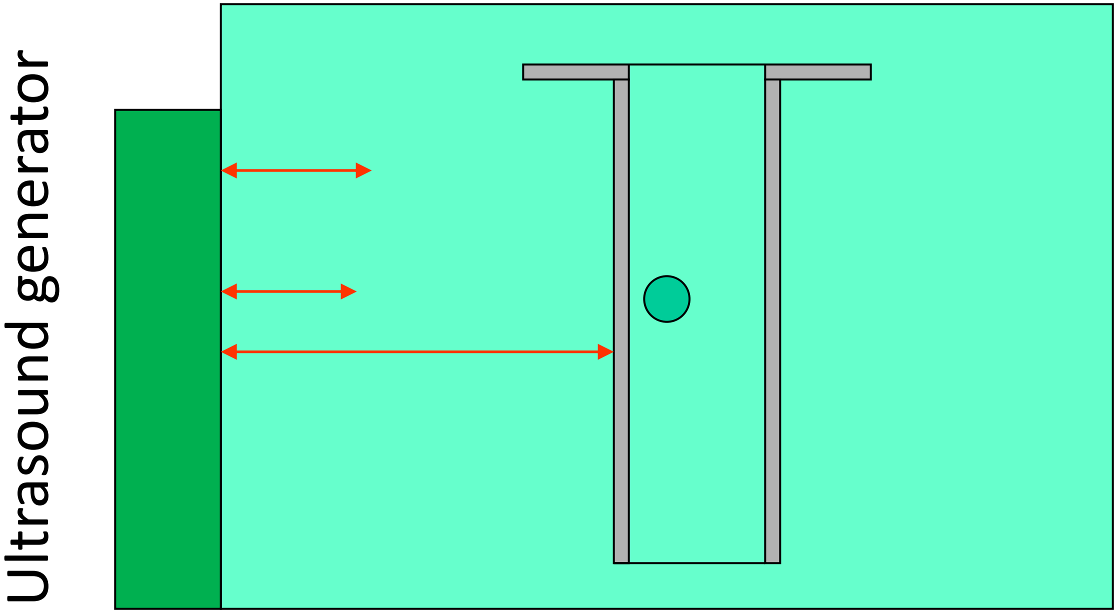
Ultrasonic cleaning



Standing waves transfer energy to the particle

A. Matheisen - Milan, Adv. Vac Course, 08.11.2005

Ultrasonic cleaning

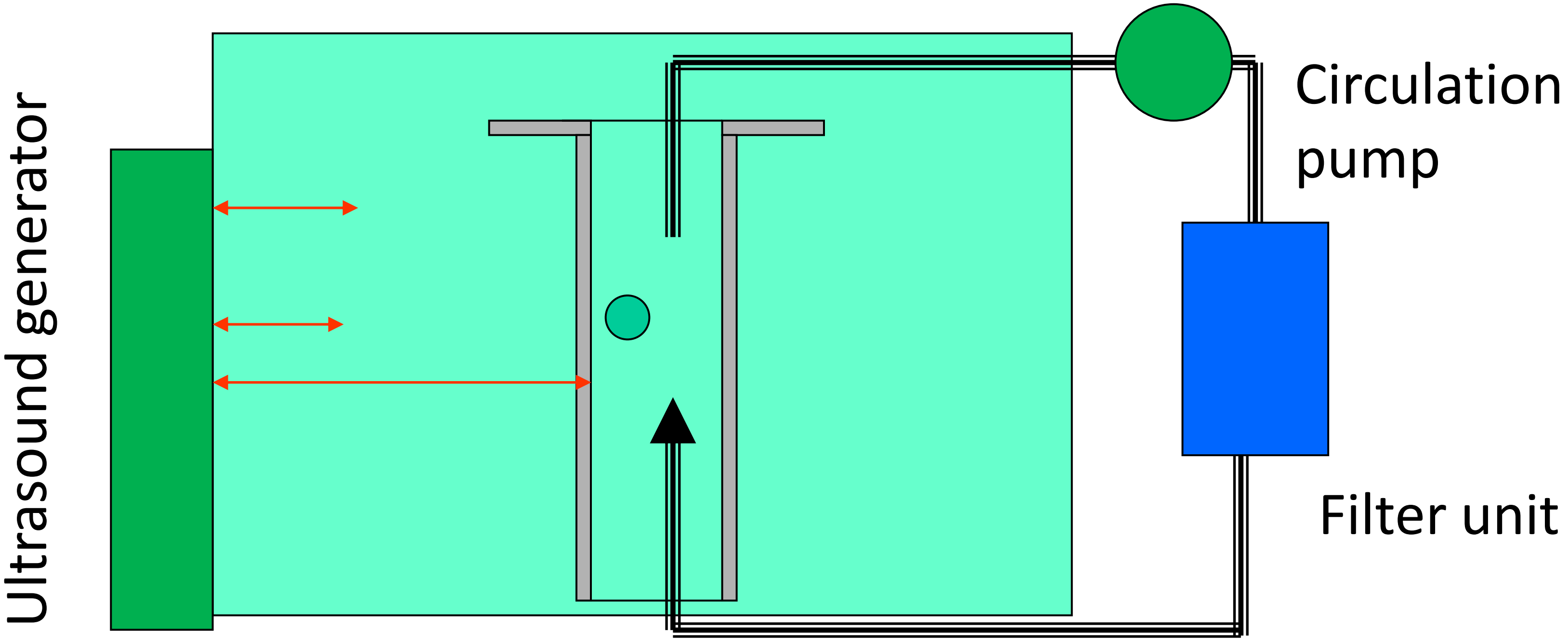


When US stops, particles can remain in the components.

Therefore…

A. Matheisen - Milan, Adv. Vac Course, 08.11.2005

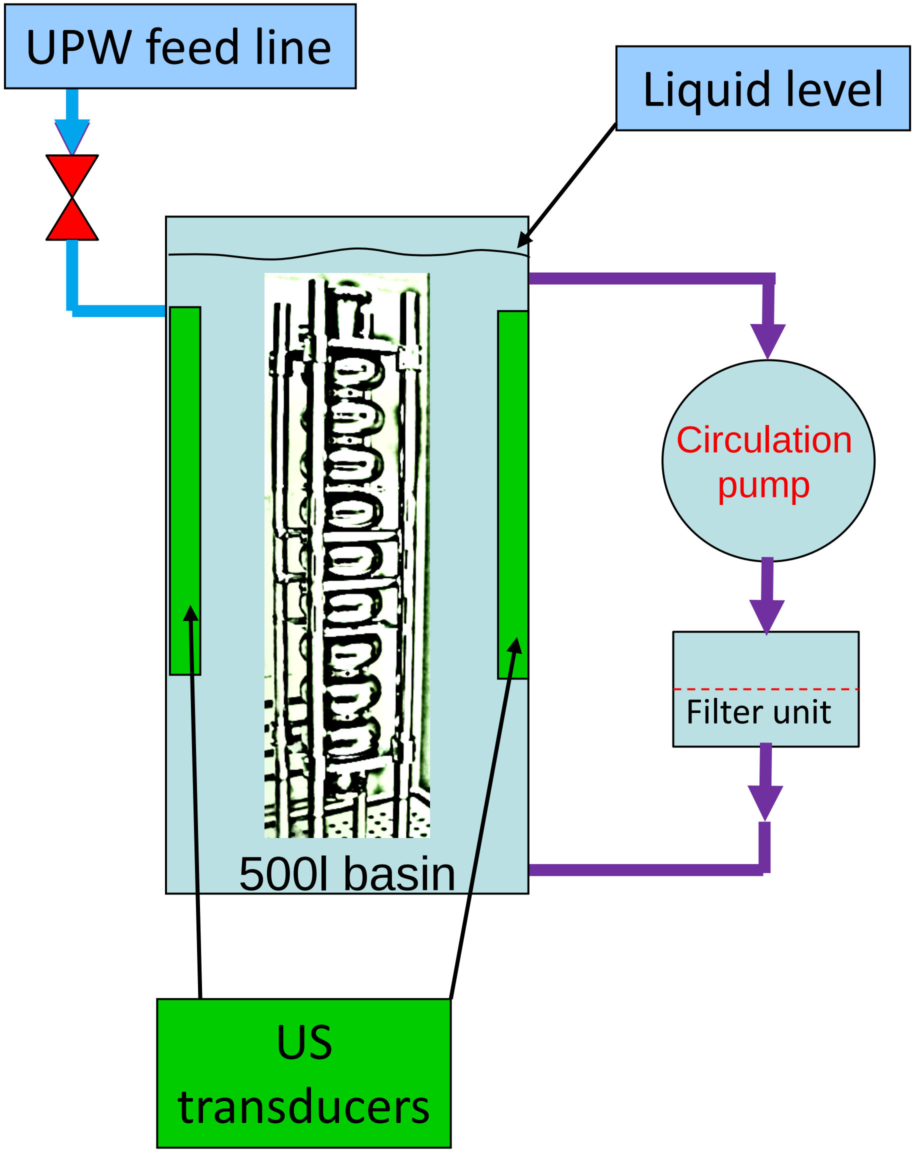
Ultrasonic cleaning



In Addition to ultrasound you **need to wash out the particulates**

A. Matheisen - Milan, Adv. Vac Course, 08.11.2005

US cleaning: summary



Ultra Clean water (≥ 18 MΩ cm)
+detergent (Tickopur)
concentration 1% to 3% @Temperature 45°C

Specifications

Ultrasound power: 10 W/liter
Circulation: system with 2 μm inline filter
Volume: 500 l
Circulation: 40 l/min

US cycle

5 Min circulation and warm up of item
5 Min US sound + circulation
5 Min US sound NO circulation
5 Min circulation

Effect of US-agitation

- Verification of the effect of ultrasonic agitation (thin Al foil)

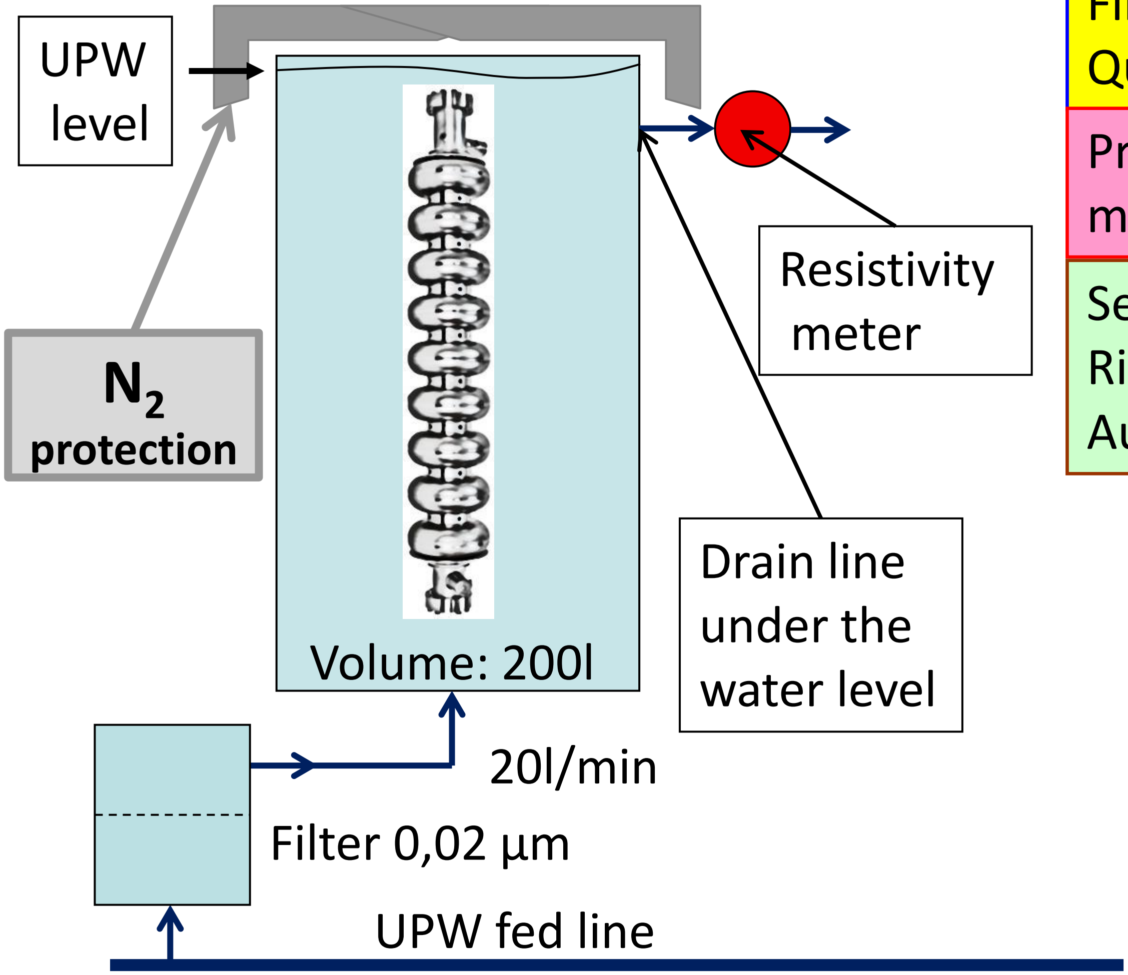
Rinsing with UPW for large components

- **So called “resistivity rinsing”: rinsing with UPW to eliminate “any” residuals of detergent or BCP / EP acid**
- Use of UPW, filtered (≤ 0.04 μm), with resistivity 18.2 MΩ·cm
- **Capping with Ultra Pure Nitrogen** needed to avoid resistivity limit due to air **CO_2** absorption that change output water resistivity.
- **Resistivity meter under water level at the exit line**
- **Water flow: about 20 l/min (typ duration for XFEL cavity: 30 minutes, that is 600 liters of UPW).**
- In some case **two bath are used in sequence** to speed up procedure (and reduce total UPW consumption). **First bath removes** the major part of the detergent, **the second one with N_2 capping makes the final rinsing (EZ).**
- Process is automatically stopped when resistivity is OK

Rinsing with UPW: “resistivity rinsing”

~~Clean room air~~

~~CO_2 gas solved in UPW => ρ **limited**~~



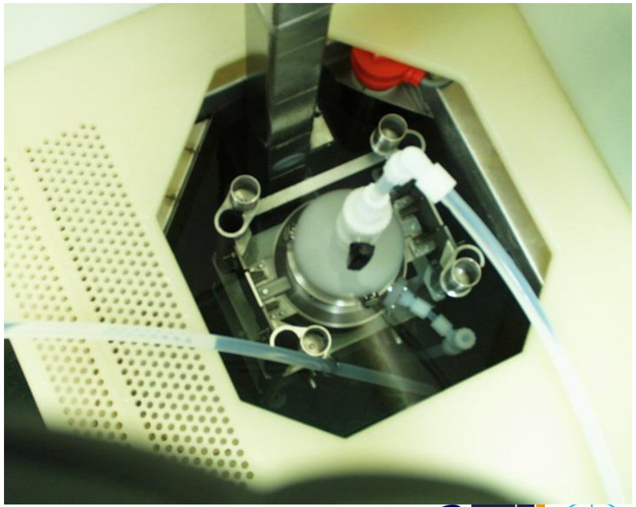
Primer water: 18.2 MΩ·cm
Filtration level: 0.04 – 0.02 µm
Quantity: 20 l/min renewed

Process steering: resistance measured at the basin exit

Sequence
Rinse manually by water jet
Automatic rinse to 12.4/18 MΩ·cm

From A. Matheisen High Gradient WSK Argonne 10.2008

Cleaning and rinsing @ DESY



Cleaning checks

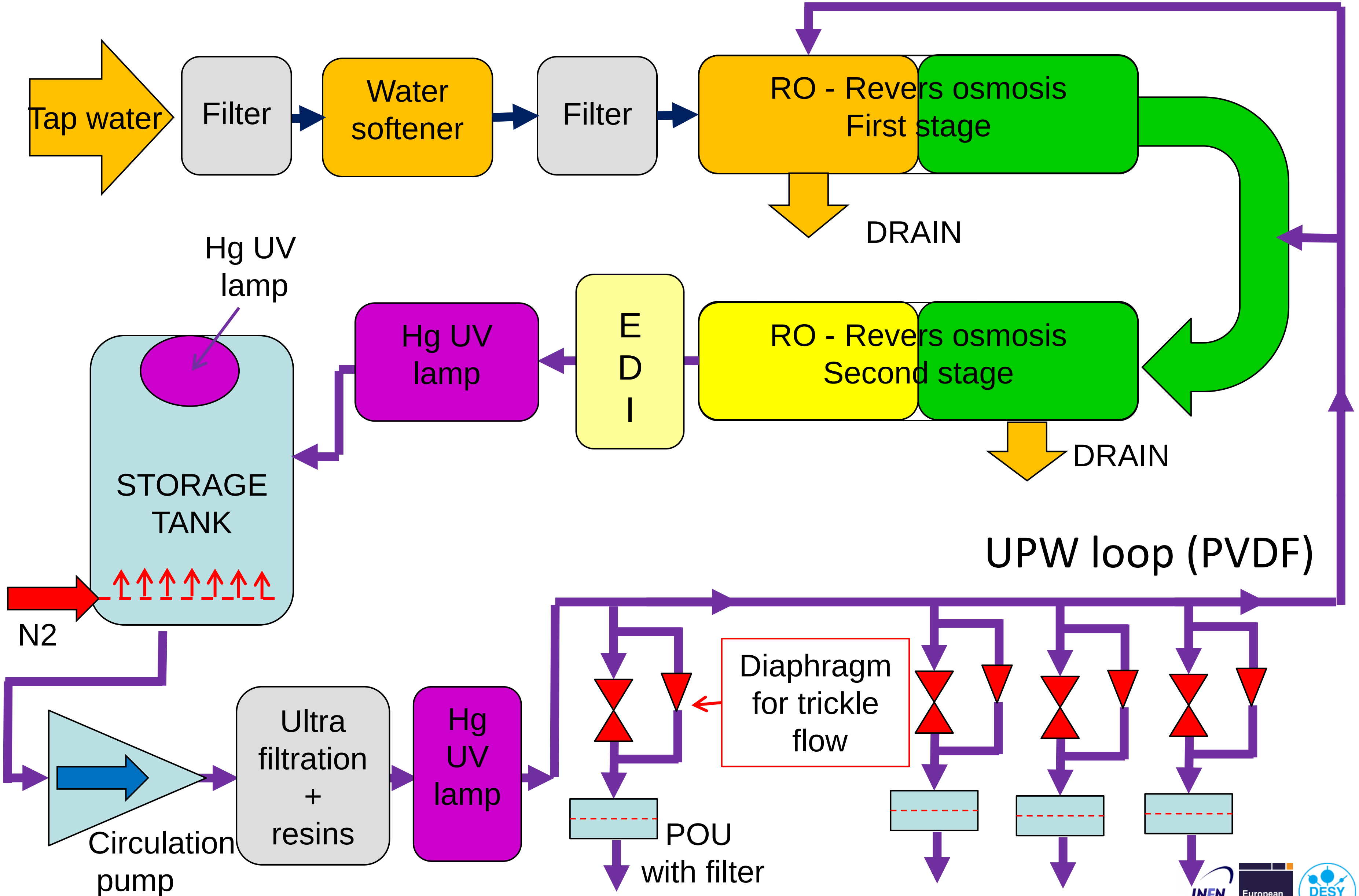
- Checks
  - Optical
  - Wipe tests
  - Particles (blowing)
  - Water break test or “Drip run test” for degreasing effect
  - Residual gas analysis

UPW

UPW: Ultra Pure Water

- **Thousands of liters** per hour of UPW are needed for EXFEL SC cavities production (**EZ** and **RI** production capability > **3000 l/h)**, but **peak throughput request is much more** (concurrent HPRs and rinsing) and **buffer** systems (**thousand of liters**) are installed.
- Water is used for US (cleaning), for rinsing, for HPR. **Two loops** with different quality are generally used: **18 MΩ·cm and 12 MΩ·cm**
- General rules:
  - Keep it **moving** all the time, **small flow in all POU**
  - **No dead legs**: continuous flow through the **whole system**
  - **Sudden flow changes** (opening / closing valves) cause particles

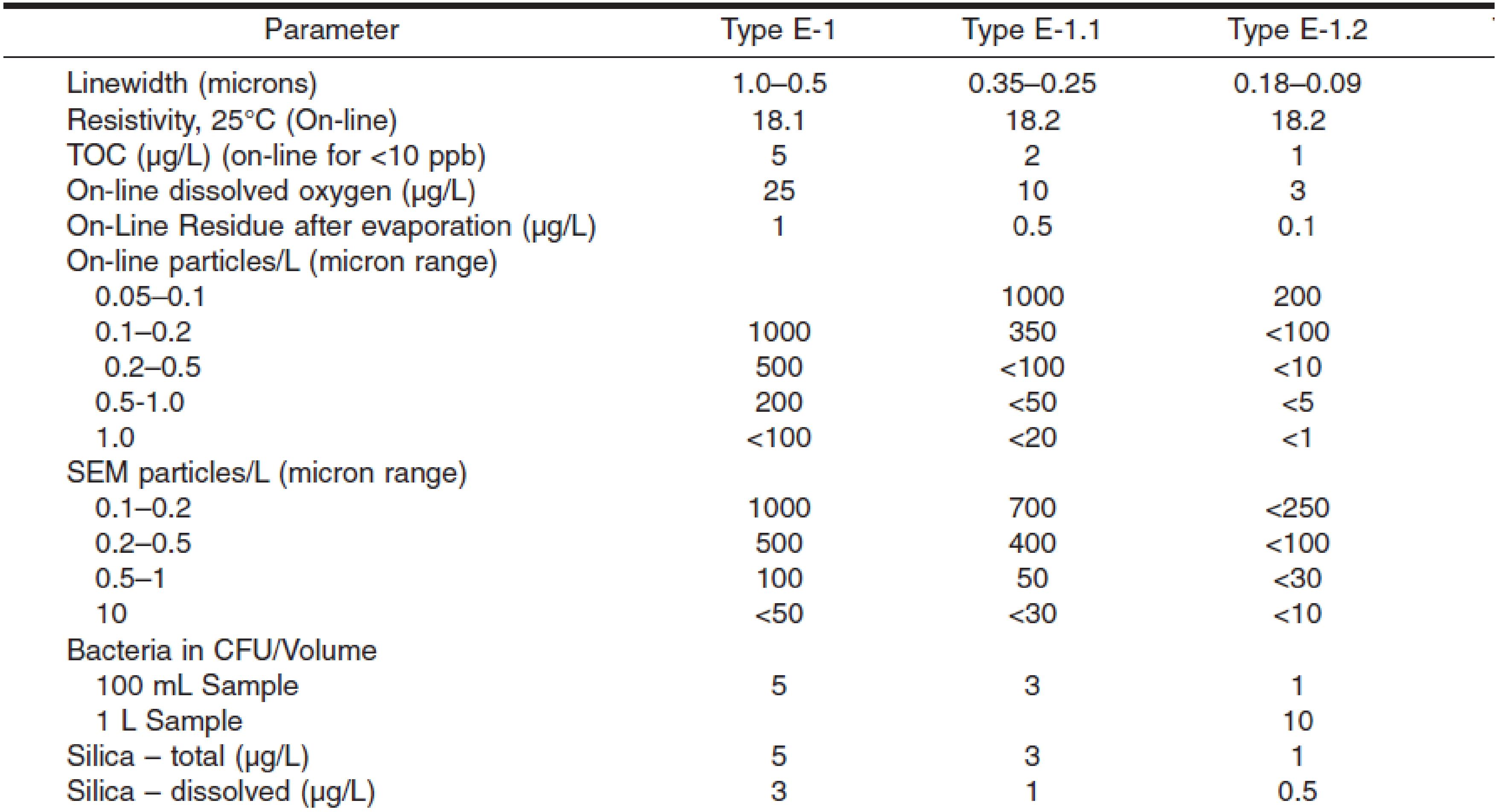
UPW typical production scheme



Ultra pure water

- Standards (ASTM, EN-ISO, …) for electronic-grade applications
- **Specifications for**
    - Resistivity (ions and metals)
    - TOC (**T**otal **O**rganic **C**arbon), material derived from decaying vegetation, bacterial growth, and metabolic activities **of living organisms** or chemicals
    - Particles
    - Bacteria
    - Foreign elements

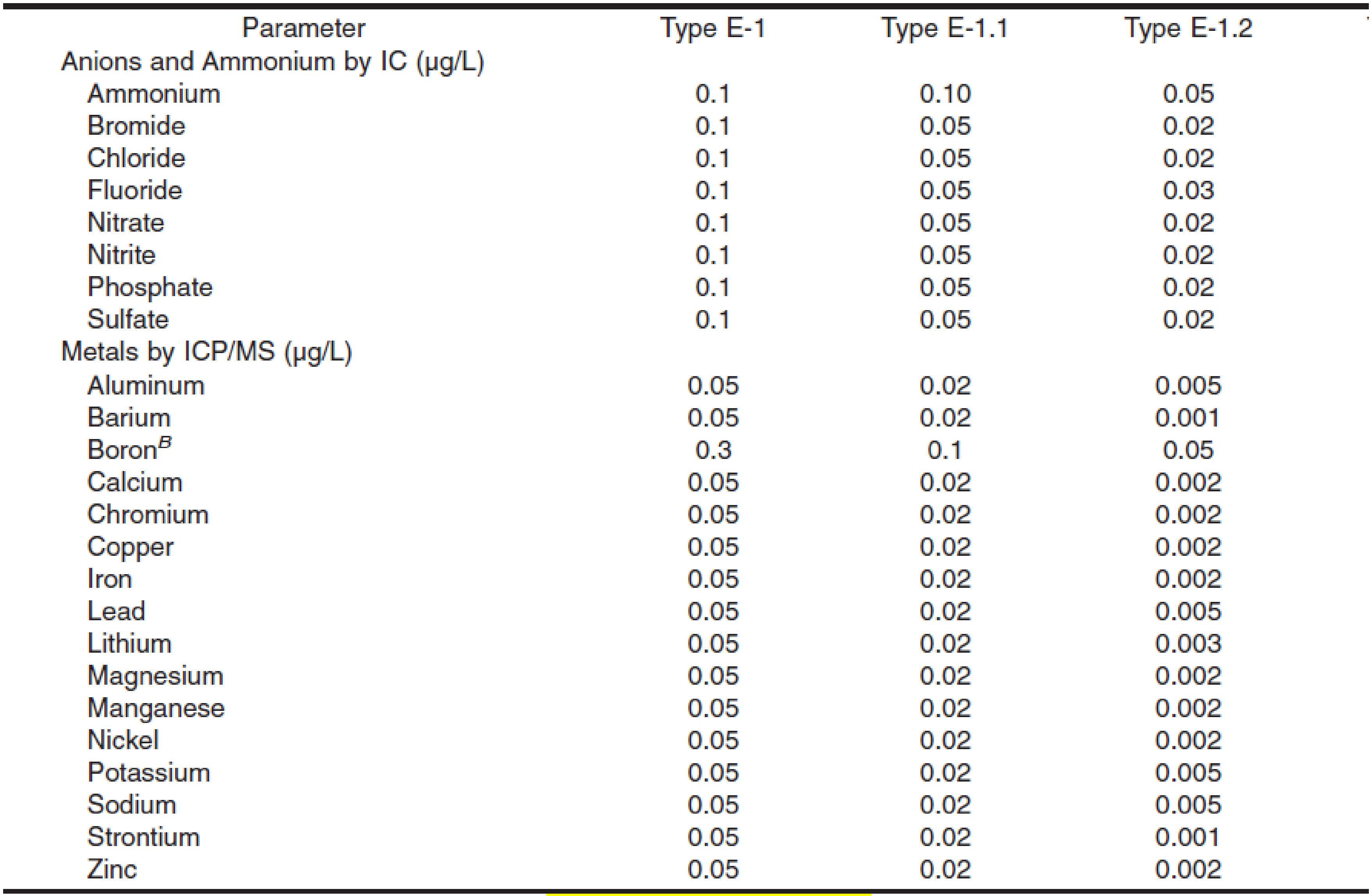
Ultra pure water: type E-1.2



Parameter	Type E-1	Type E-1.1	Type E-1.2
Linewidth (microns)	1.0–0.5	0.35–0.25	0.18–0.09
Resistivity, 25°C (On-line)	18.1	18.2	18.2
TOC (µg/L) (on-line for <10 ppb)	5	2	1
On-line dissolved oxygen (µg/L)	25	10	3
On-Line Residue after evaporation (µg/L)	1	0.5	0.1
On-line particles/L (micron range)			
0.05–0.1		1000	200
0.1–0.2	1000	350	<100
0.2–0.5	500	<100	<10
0.5-1.0	200	<50	<5
1.0	<100	<20	<1
SEM particles/L (micron range)			
0.1–0.2	1000	700	<250
0.2–0.5	500	400	<100
0.5–1	100	50	<30
10	<50	<30	<10
Bacteria in CFU/Volume			
100 mL Sample	5	3	1
1 L Sample			10
Silica – total (µg/L)	5	3	1
Silica – dissolved (µg/L)	3	1	0.5

from
ASTM D 5127-07

Ultra pure water: ions and metals



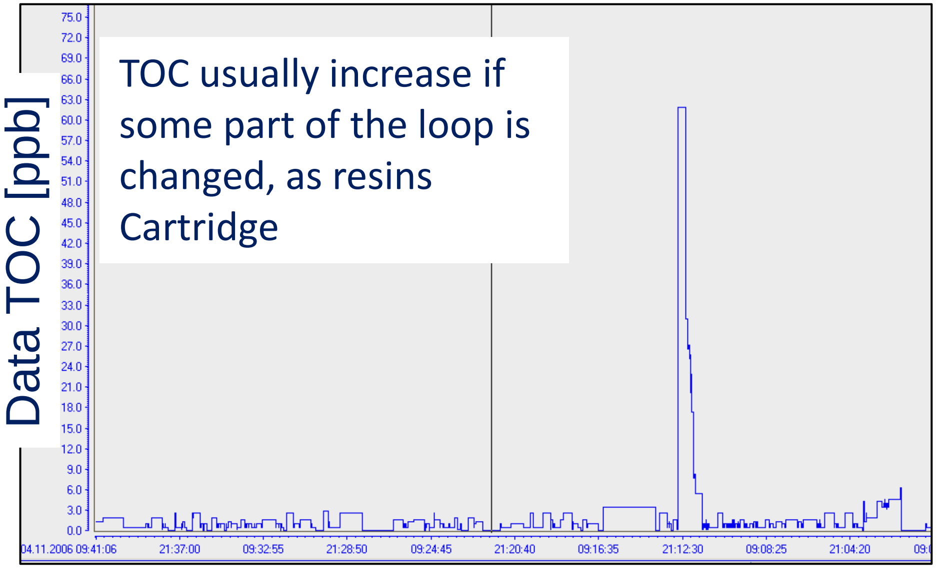
Parameter	Type E-1	Type E-1.1	Type E-1.2
Anions and Ammonium by IC (µg/L)			
Ammonium	0.1	0.10	0.05
Bromide	0.1	0.05	0.02
Chloride	0.1	0.05	0.02
Fluoride	0.1	0.05	0.03
Nitrate	0.1	0.05	0.02
Nitrite	0.1	0.05	0.02
Phosphate	0.1	0.05	0.02
Sulfate	0.1	0.05	0.02
Metals by ICP/MS (µg/L)			
Aluminum	0.05	0.02	0.005
Barium	0.05	0.02	0.001
Boron[B]	0.3	0.1	0.05
Calcium	0.05	0.02	0.002
Chromium	0.05	0.02	0.002
Copper	0.05	0.02	0.002
Iron	0.05	0.02	0.002
Lead	0.05	0.02	0.005
Lithium	0.05	0.02	0.003
Magnesium	0.05	0.02	0.002
Manganese	0.05	0.02	0.002
Nickel	0.05	0.02	0.002
Potassium	0.05	0.02	0.005
Sodium	0.05	0.02	0.005
Strontium	0.05	0.02	0.001
Zinc	0.05	0.02	0.002

from
ASTM D 5127-07

UPW: on line & off line quality check

- **Conductivity**: ion concentration, indicated how the water is pure with respect to "salts", ions, metals, anions, etc
- **TOC**: Total Organic (oxidisable) carbon. Good values are below 10 ppb (part per billion). **Organic carbon, but for instance lubricant oils are hard to be oxidized and therefore are not easily measured with TOC monitor.**
- **Bacteria: colonies count**. Usually sampling and incubation on Petri capsule. Special funnels with incorporated Petri capsule, Agar, dye, etc. are available on the market (Millipore Merck). Must be left in the incubator (38 °C) for 2 – 3 days
- **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).
- **Hydrocarbon (oils):** they **MUST** not be present. OFFLINE extraction method (with hexane) and gas chromatographic analysis.

UPW: TOC monitor in line & bacteria count offline

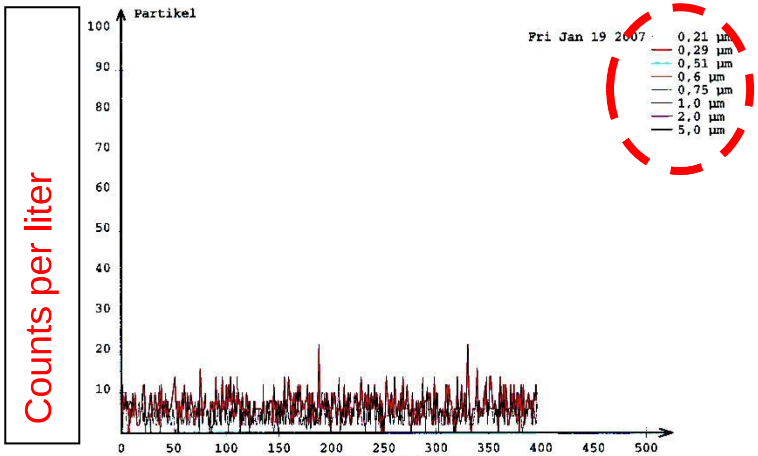


TOC usually increase if some part of the loop is changed, as resins Cartridge

- Test sample of 125 bacteria colonies per liter
- According to chip industry (UPW standard) 100/l are Ok
- DESY had experience of bad results at 50/l
- Standard for **full functional DESY system 1-5 / liter**

DESY Data

UPW: liquid particle counting



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Messdatei:            D:\DCC\MESSUNG\190107BB.DBF
Messgerät:            Hiac 8000
Messbeginn:           Fri Jan 19 11:30:01 2007
Sample/Delay Time: 600/1
Messproben gesamt: 0
Kommentar: Partikelmessung an HD-Innenlanze
Parrallel mit TOC
```

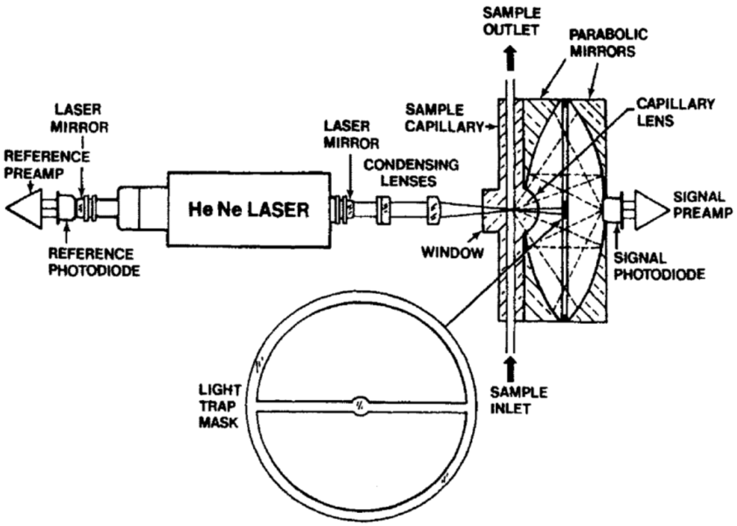


Figure 17: Integrated micro-optical liquid volumetric (IMOLV) sensor optical system.

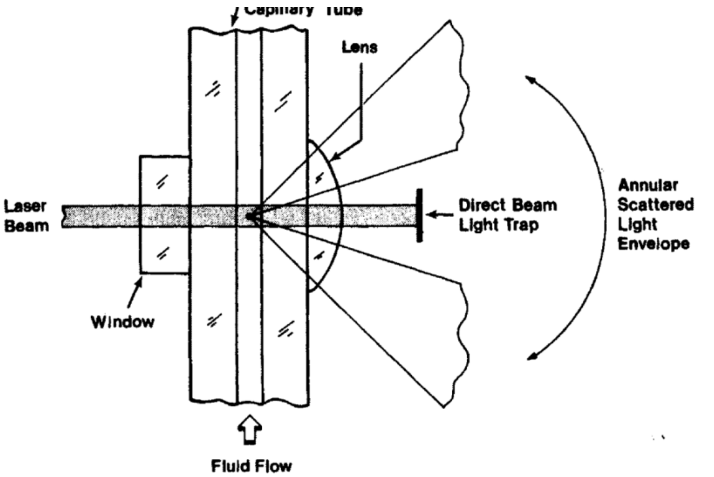


Figure 16: Integrated micro-optical liquid volumetric (IMOLV) sensor cell construction. This cell allows for the collection of light from 10° to 60°.

Industrial and laboratory UPW plants

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

HPR: High pressure rinsing with UPW

Cavity inner surface rinsing with high pressure water jets

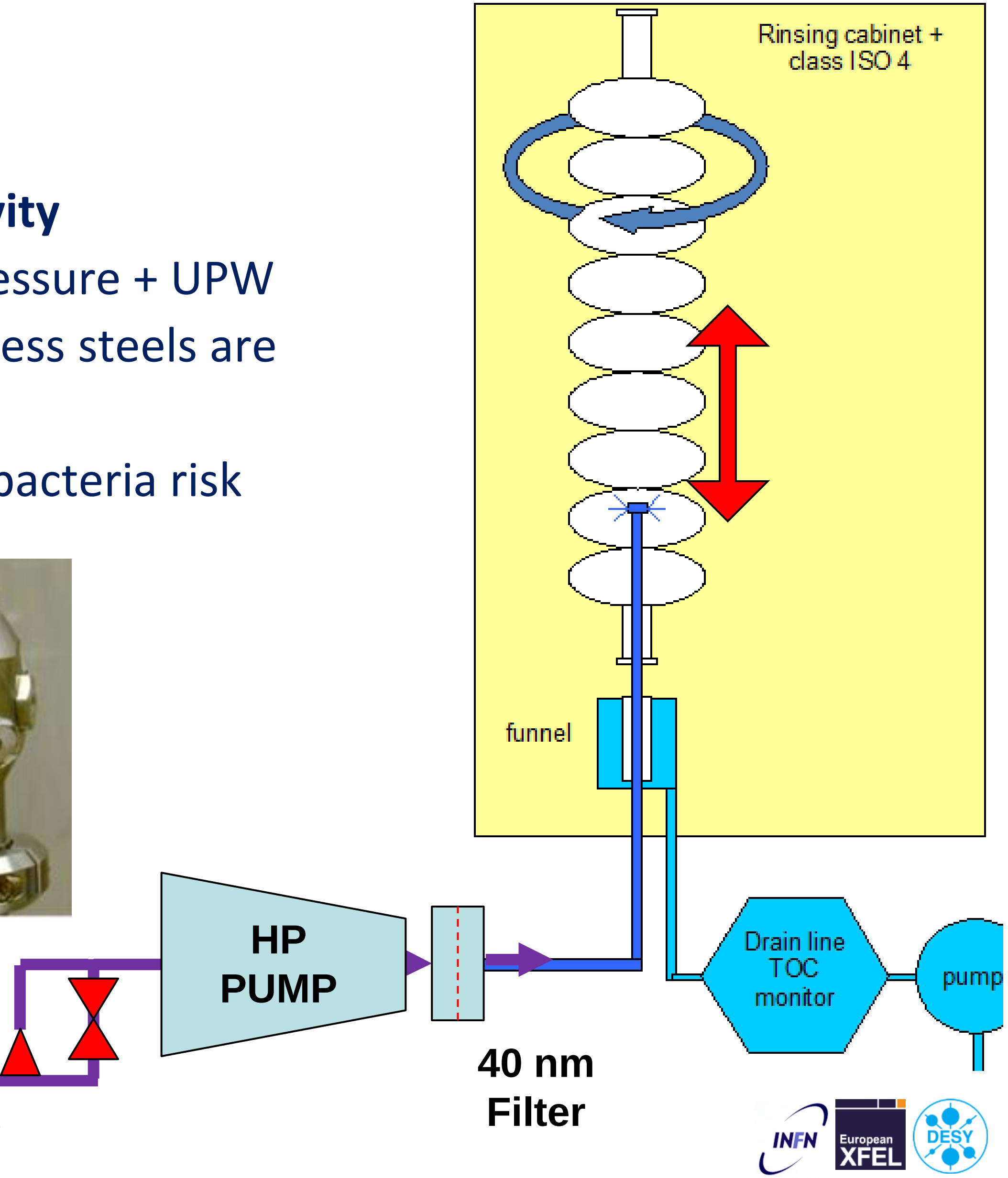
- HPR is the **final cleaning** step of the cavity preparation process
- Removal of **last treatment residuals**, **dust**, particles, etc.
- **Water jet must be moved continuously**: if jet impacts stably in one point Nb **surface can be damaged.**
- **Continuous motion of the cavity respect jets** (drawing a **spiral behavior** that cover completely the Nb surface)
- Typical pressure: **100 bar**
- **Water quality: E-1.2** (ASTM), **40 nm** filter **after HP pump!** No flex line should be used after the filter, to reduce particle generation.
- **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.

HPR system: set up

- Set-up
  - **HPR stand in ISO 4 / 5**
  - Nozzles: **sapphire nozzles**
  - **No moving parts inside the cavity**
  - adequate materials for high pressure + UPW lines + **HPR pump**: not all stainless steels are OK (316, electropolished, etc.)
  - **Trickle flow** all time to reduce bacteria risk

Sapphire nozzles

Nozzle head with sapphire nozzles

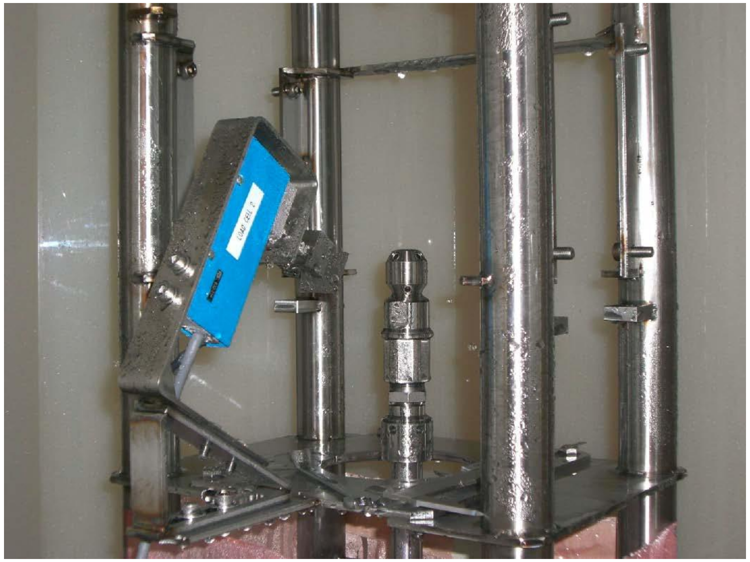


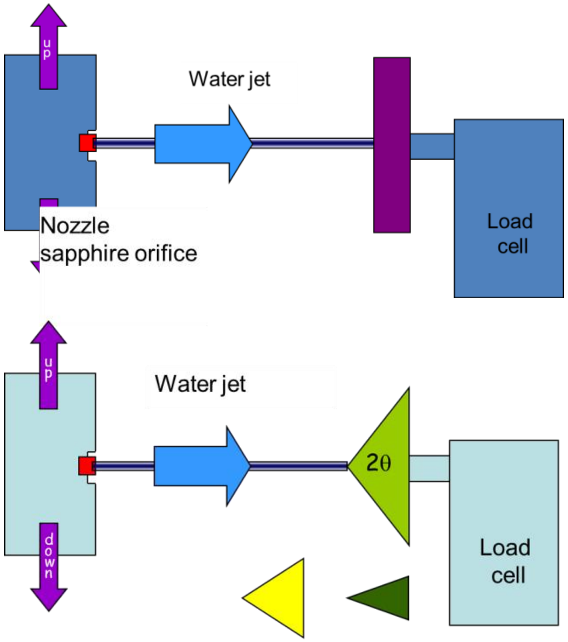
Trickle flow regulator

40 nm Filter

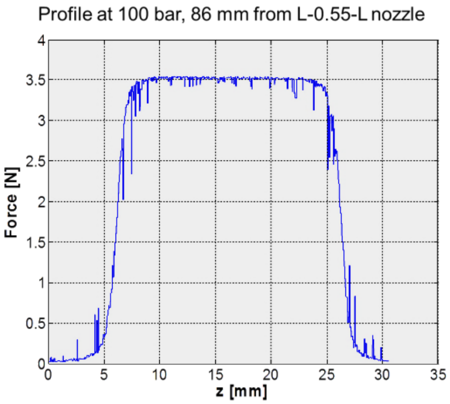
UPW HPR stand loop

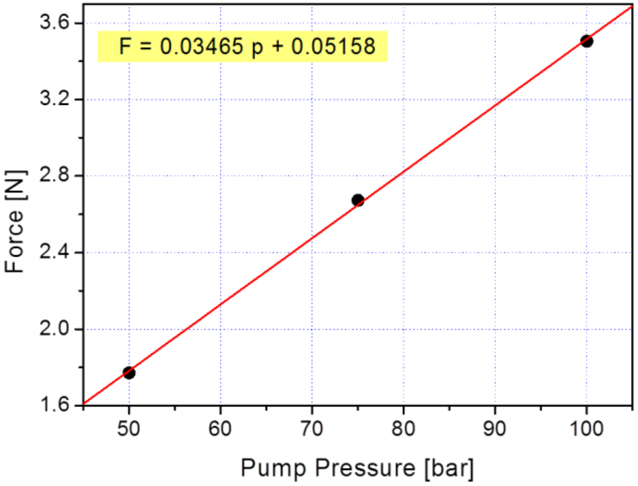
Water jet diagnostics using load cell (INFN – LASA)

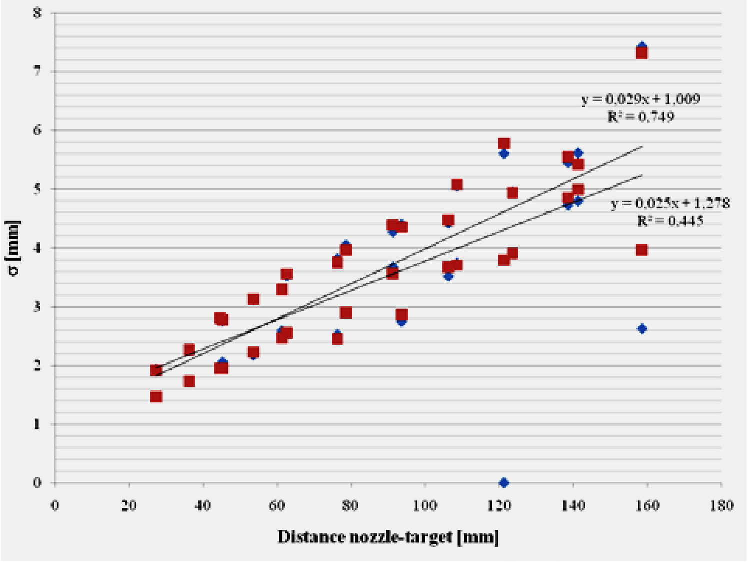




Force transfer to a non normal surface

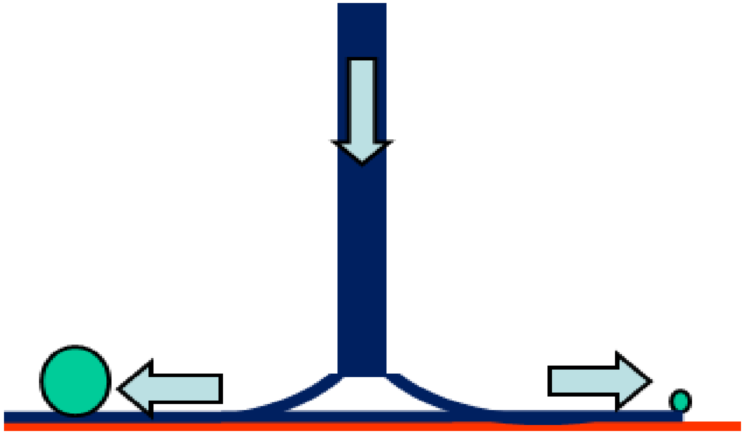


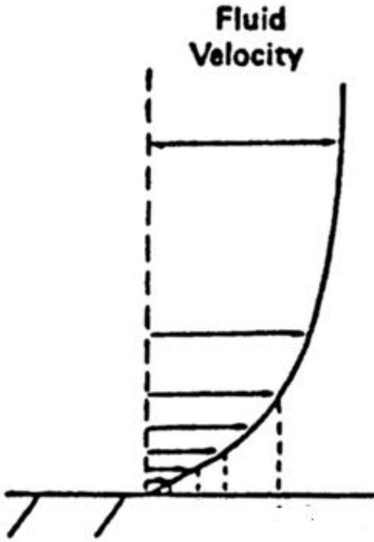


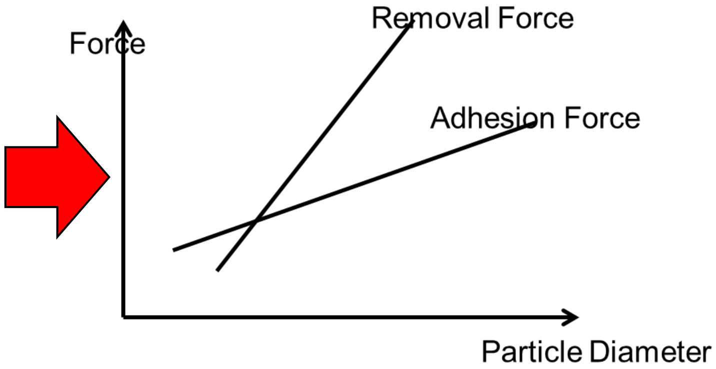


HPR: how it works (cnt)

- **Direct effects** of high speed water on particles
- Particles are removed if the **force of the jet and high speed water on the particle is > adhesion force of particles**
- **But water speed on the surface is nearly zero!**
- Investigations and models indicates an **important role of droplets**, that can generate higher pressure (they are small!)(Claire Antoine)
- **Large particle are more easily removed**.

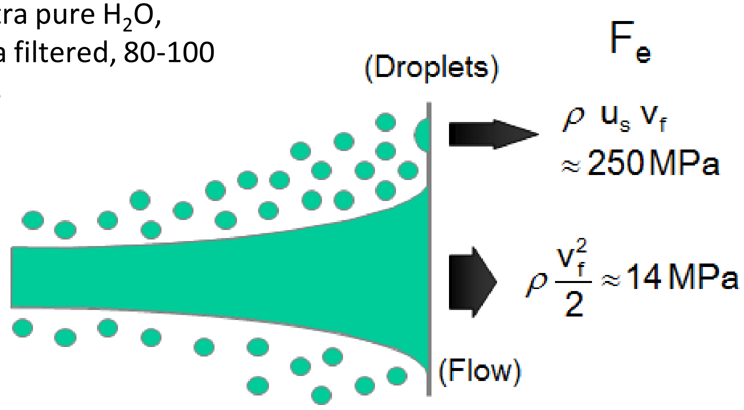


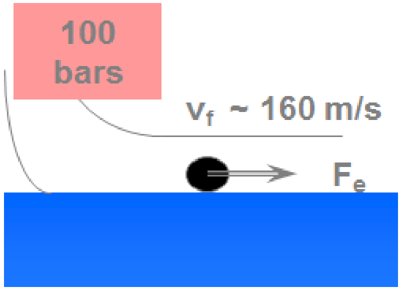




High pressure rinsing (HPR): how it works

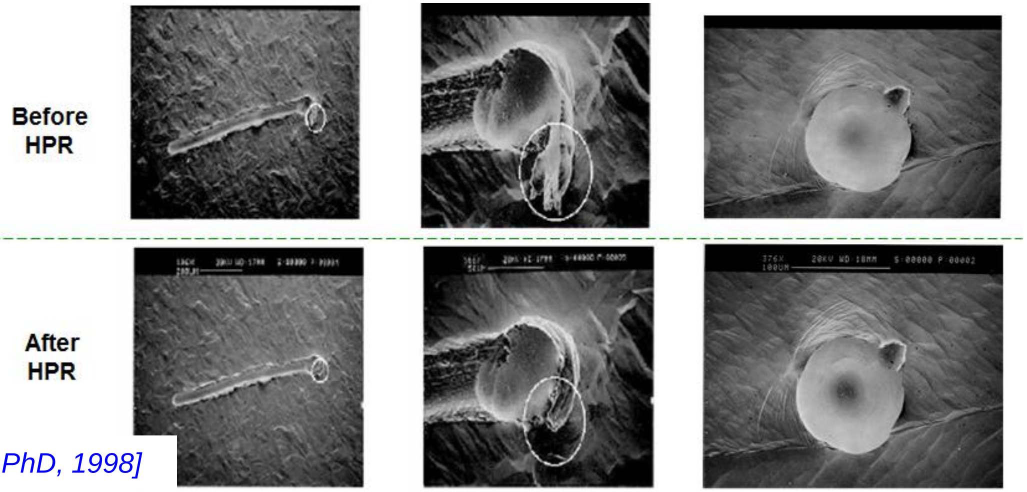
>ultra pure H_2O, ultra filtered, 80-100 bars





Size (μm)	F_e (N)	F_{ad} (N)
0.1	10^{-9}	10^{-9}
1	10^{-6}	10^{-8}
10	10^{-4}	10^{-6}

Particles are displaced when $F_e > F_{ad}$

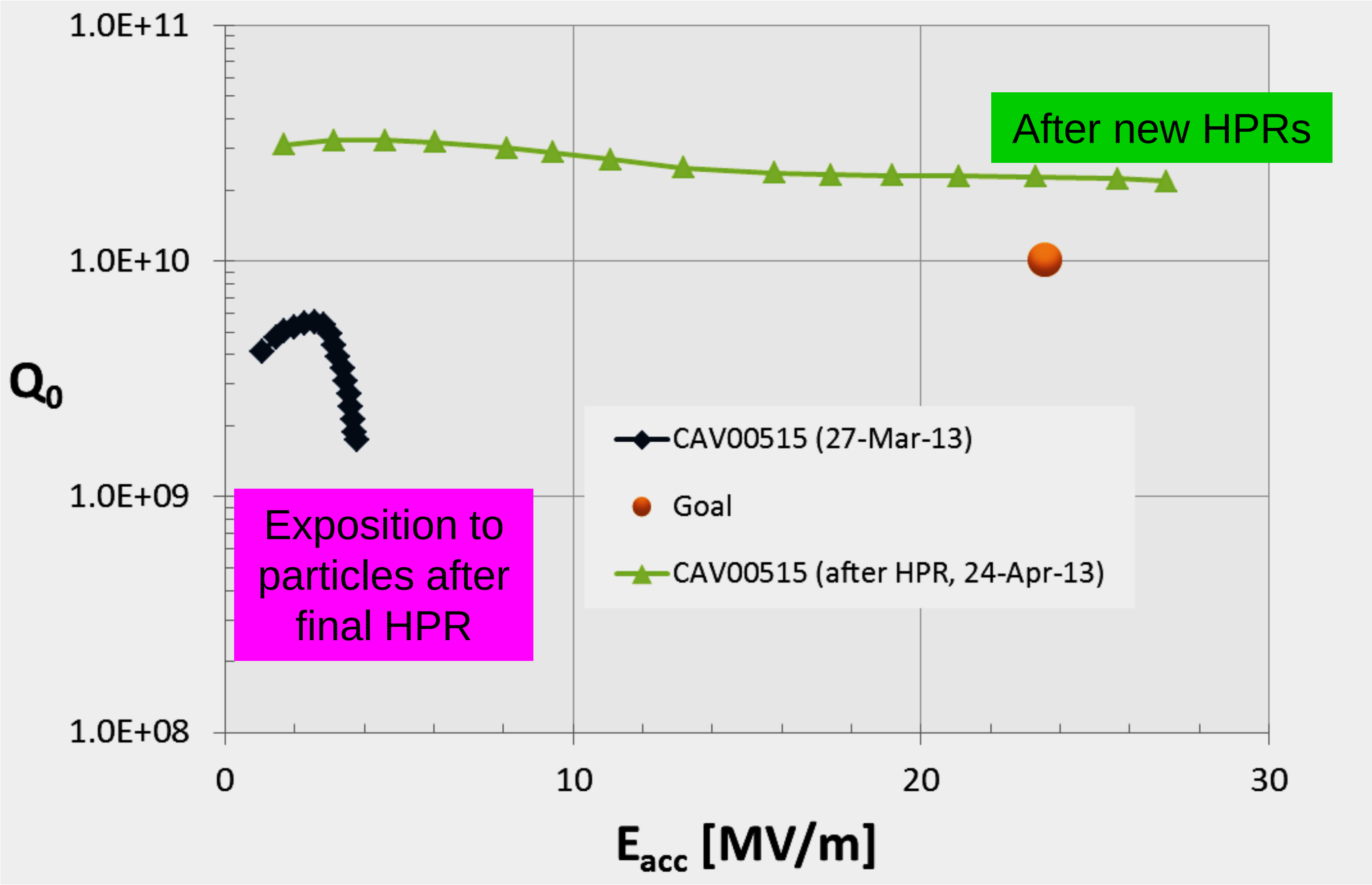


[M. Luong, PhD, 1998]

- **NB :**
  - Similar action : dry ice processing (developed @ DESY)
  - Surface microhardness ↑ (=> density of dislocations ↑)

See also *[C. Reece 2008]* https://indico.desy.de/getFile.py/access?sessionId=5&resId=0&materialId=3&confId=401

HPR effect on a particle contaminated cavity

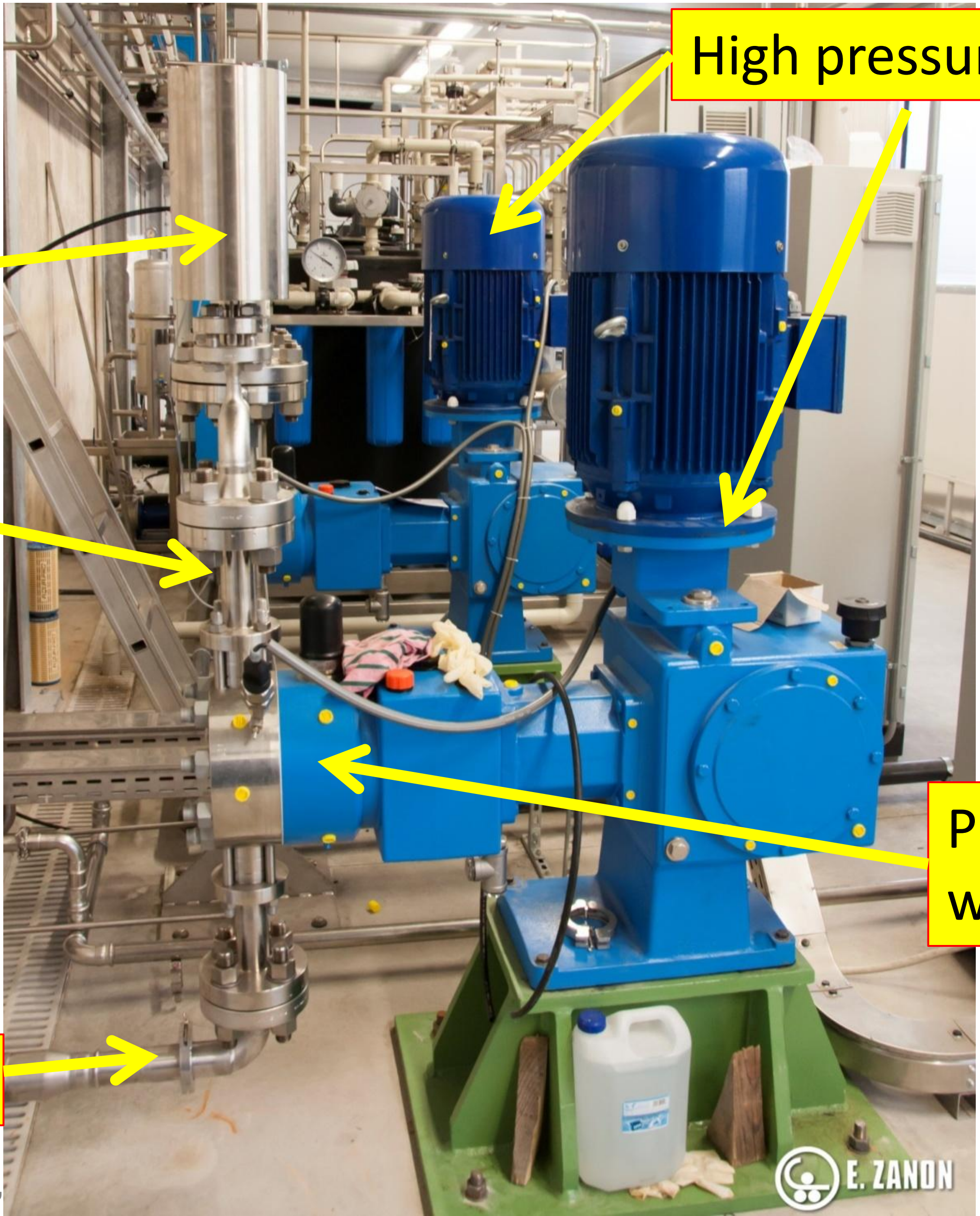


Cavity had a **problem** in the 120°C treatment (vacuum system power failure) that **produced a rapid change of pressure (a bump) in the cavity during last pumpdown**, with particle movement in the system. Consequences are clearly visible (**dark blue curve**). **After HPR**, with **no further chemical etching**, cavity performances are completely recovered.

HPR pump: car wash pump (low-cost…)

- **Oil lubricated**, **multi piston** pumps,
- **No physical separation** (no membrane) between oil lubricated part and UPW.
- Could work but there are **risk of small lubricant contamination** (oil, hydrocarbon based)
- **Not a long lifetime system**: service needed frequently

HPR UPW pumps at the industry



High pressure UPW pumps

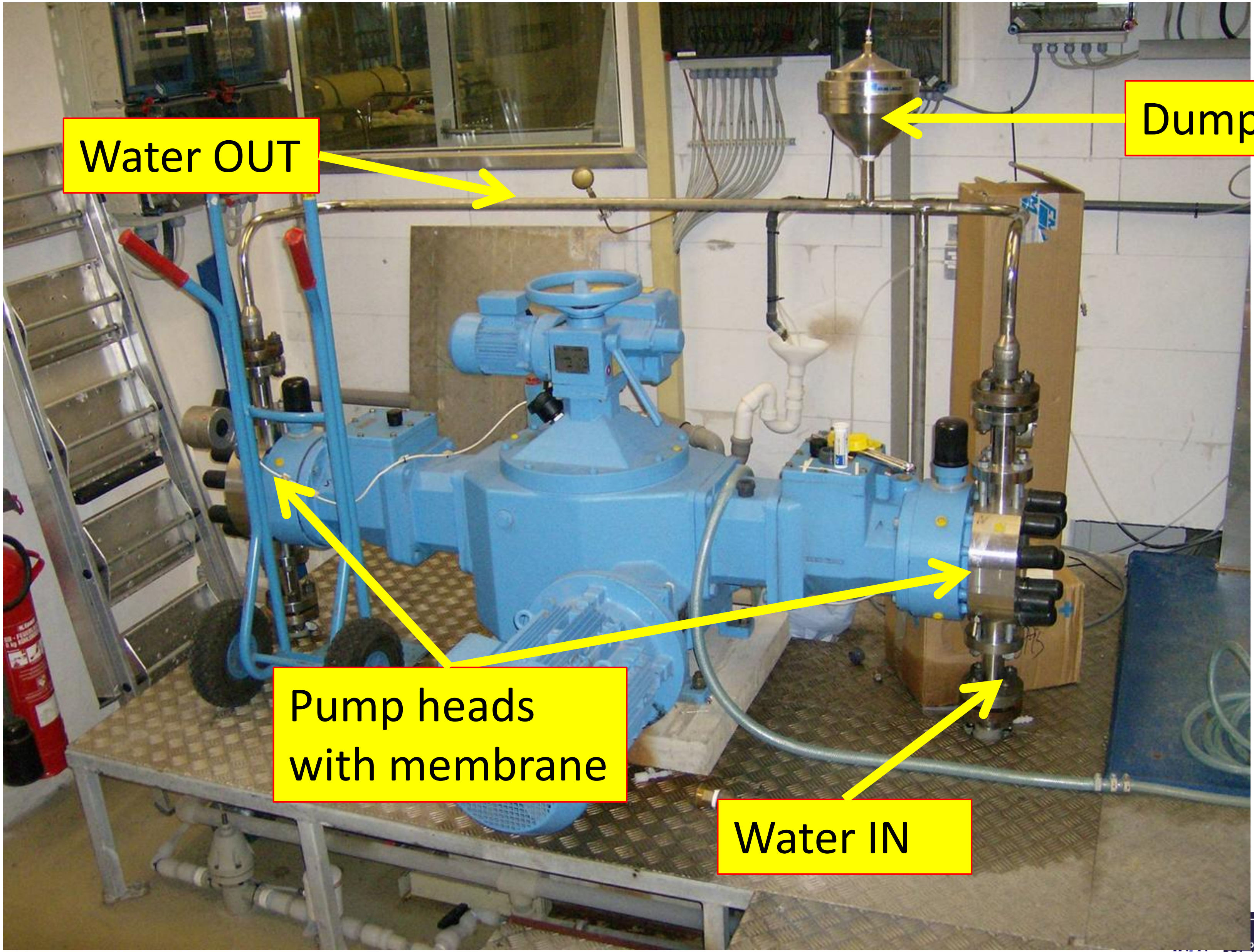
Dumper

Water OUT

Pump head with membrane

Water IN

HPR UPW pumps at DESY


Water OUT

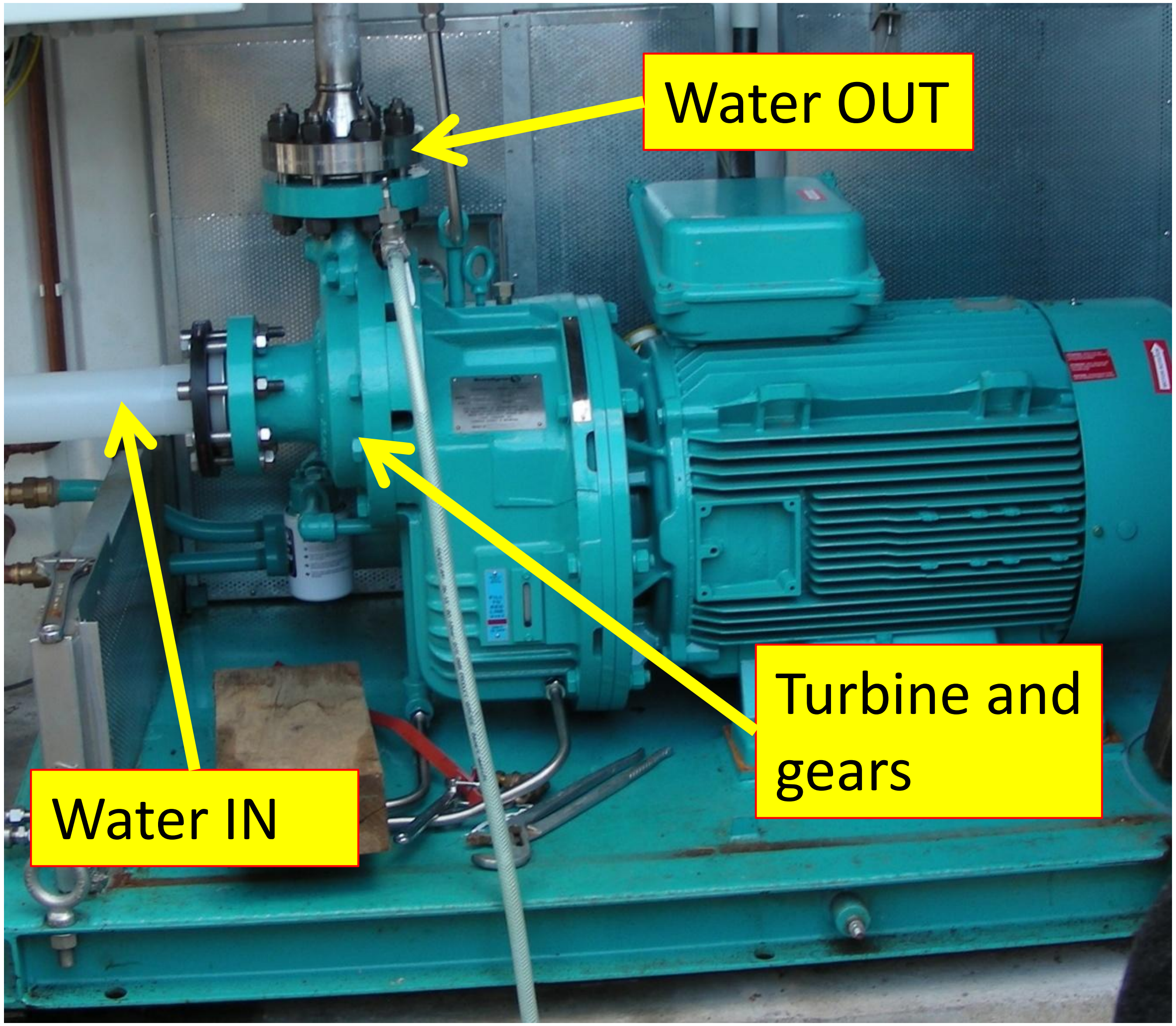
Dumper

Pump heads with membrane

Water IN

HPR: High throughput turbine pump at DESY

- **QUALIFIED at DESY**
- Single stage
- No dumper
- High throughput (but for **one HPR** part of flow must be **recirculated**)
- Can feed many HPR station
- High reliability



High pressure ultra pure water rinsing (HPR)

- **Process**
  - UPW with p = (80 - 150) bar
  - **repeated** inside rinsing

    i) after final surface treatment (1x)

    ii) after final assembly of cavity, **> 3 x,**

    for XFEL 6 x
  - well adjusted amount of water

    => too much water => circulating wave, no direct draining

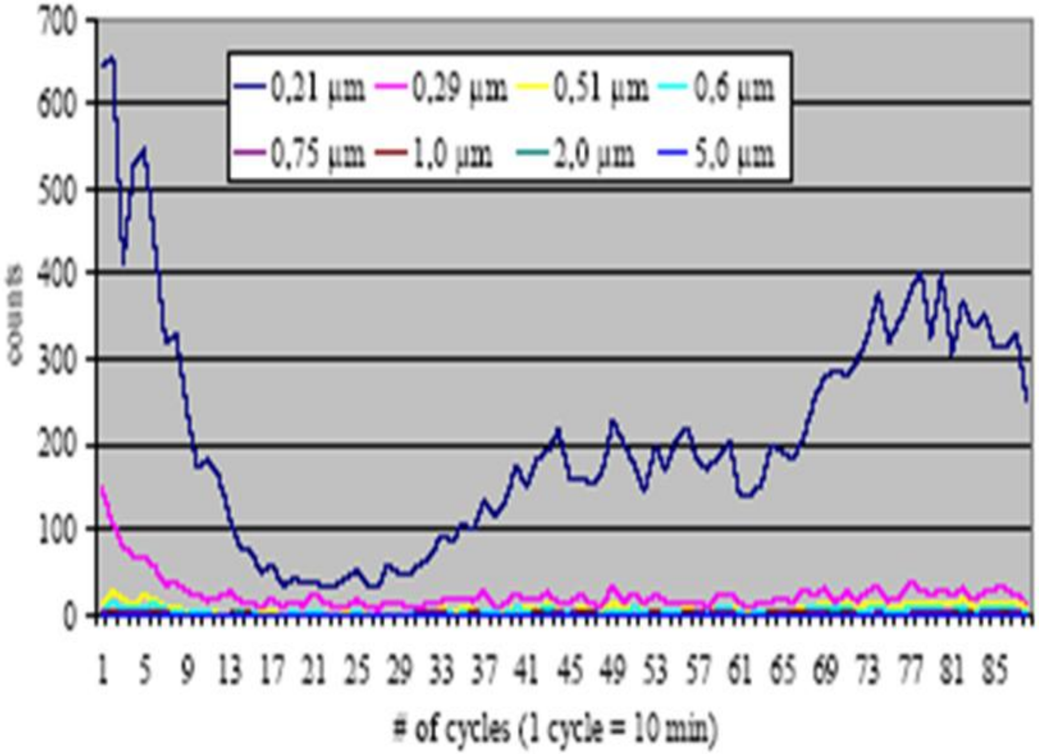
    => a "plastic" transparent cavity / dummy is very useful !

QC of high pressure ultra pure water rinsing

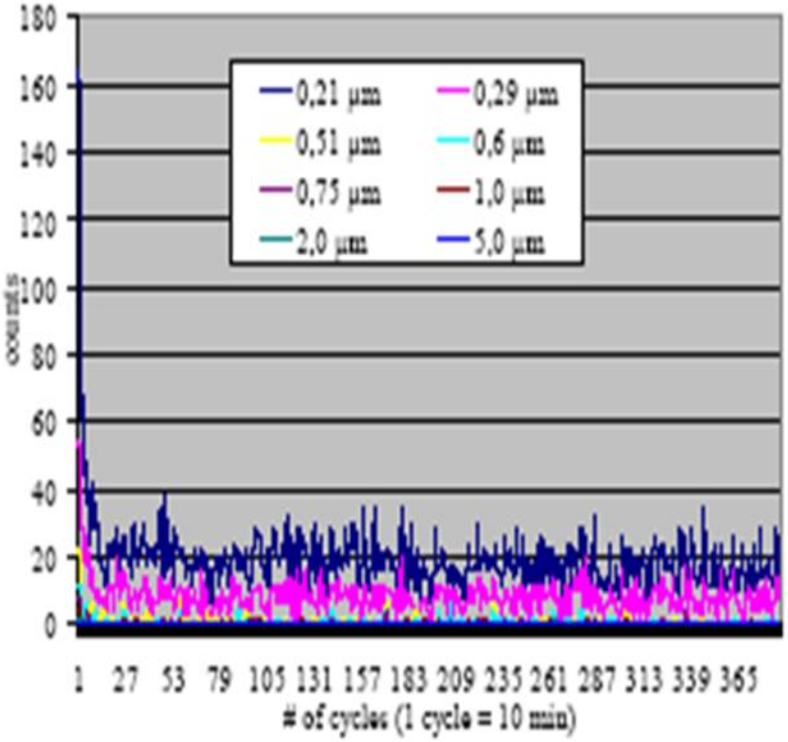
- **Examples of QC at HPR systems** (DESY, XFEL cavities production@ companies)
- Check of Point-of-use supply water quality:
  - **UPW conductivity**
  - **Particles**: online particle counter
  - Particles: off line sampling & identification(SEM optical microscope)
  - **TOC: online monitoring)**
  - **TOC drain line: sampling, after maintenance**
  - **Bacteria (=> offline)**

HPR: Particle counting at the jet exit

Particle counting of the HPR water at the jet exit (POU) after the replacement of the 40 nm filter. Particles number go down with time.



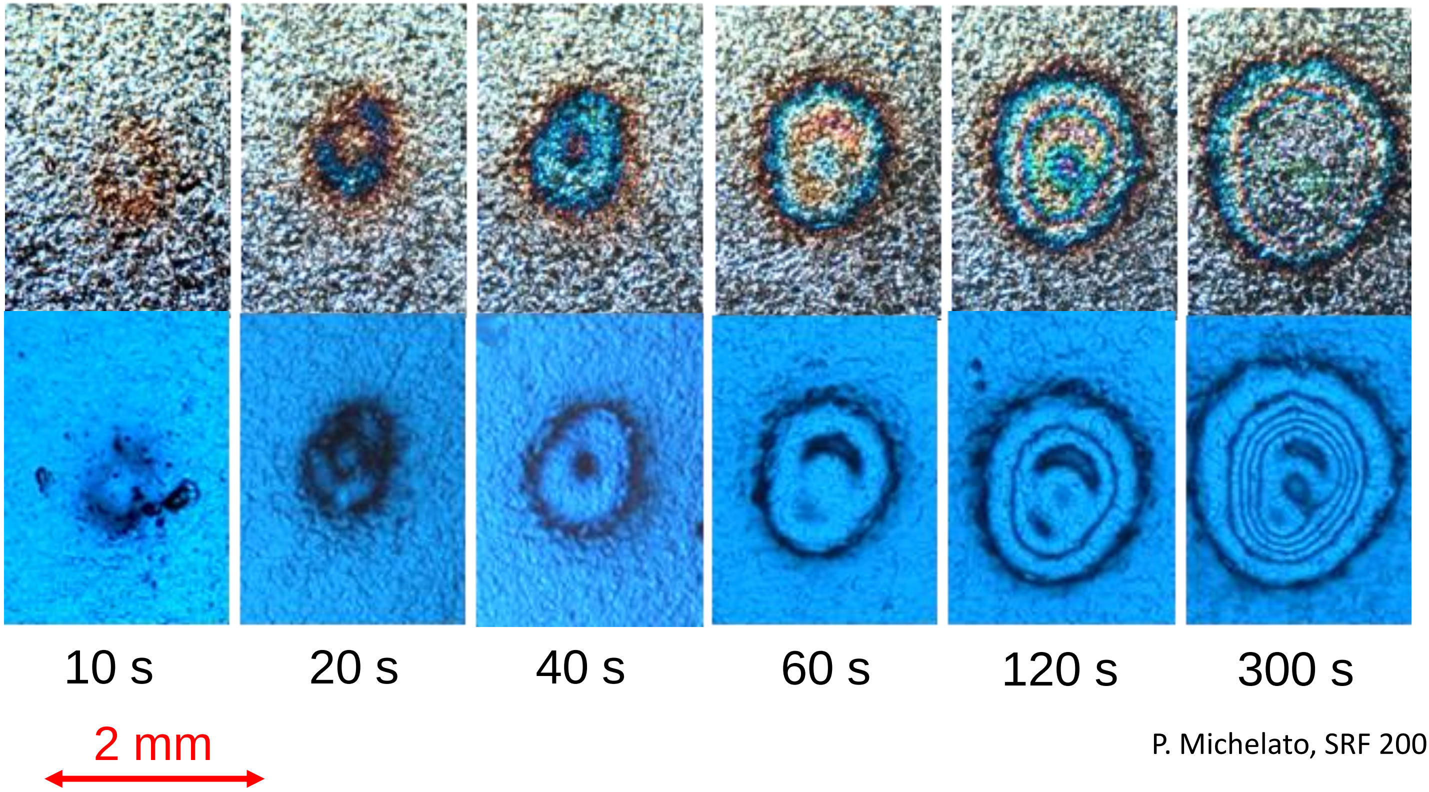
Particle counting after filter change: total sampling time: 16 hours



Particle counting after filter change, starting after 72 hours of rinsing

HPR: stationary water jet damages

P = 100 bar, distance = 35 mm (XFEL iris), nozzle diam. 0.55 mm



P. Michelato, SRF 2005

See also:
https://indico.desy.de/getFile.py/access?sessionId=5&resId=0&materialId=3&confId=401

HPR systems at DESY

Rinsing cabinet of “old“ DESY HPR system

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

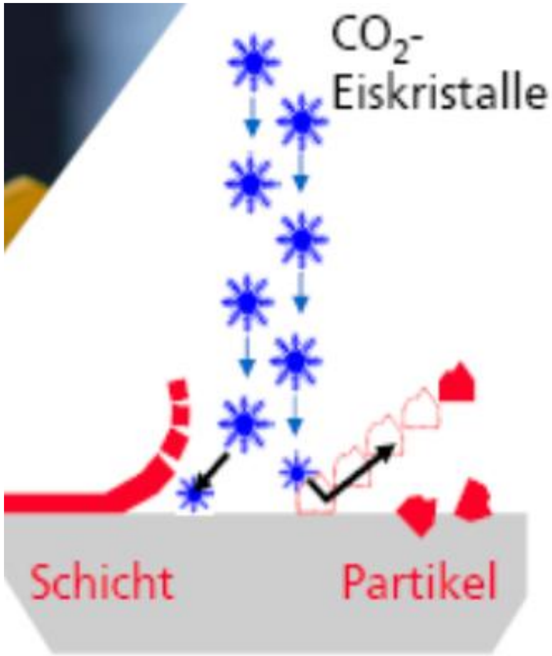
CEA HPR system

HPR: for EXFEL at the industry: EZ and RI

Cleaning alternative approaches: CO_2 snow cl.

Dry Ice Cleaning (CO_2 Snow)

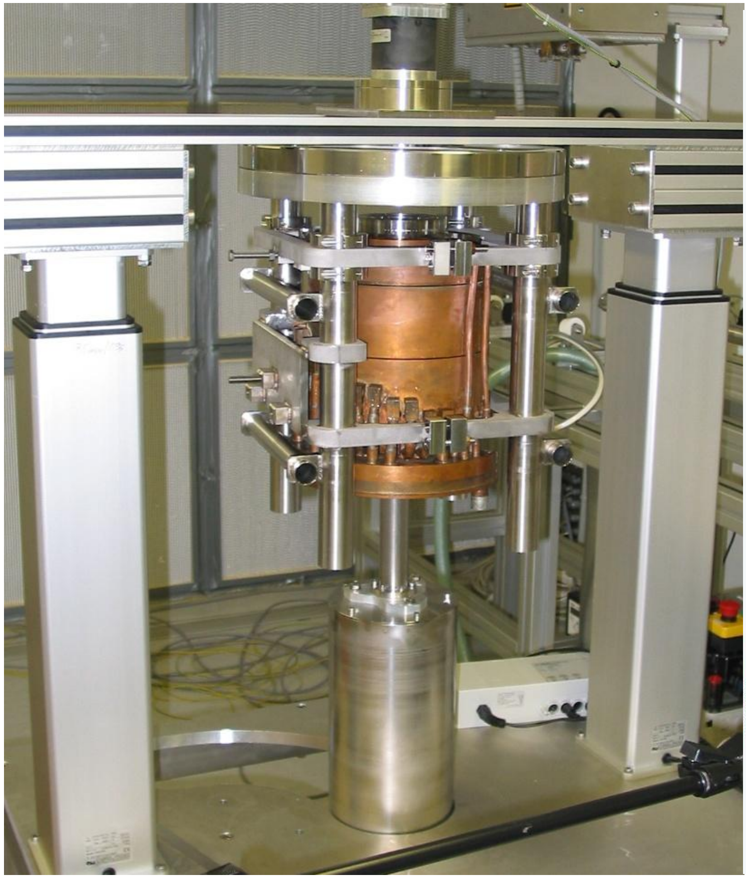
- Additional **final** cleaning technique for particles + film contaminations
- Mechanical, thermal + chemical cleaning forces
- Local, dry, without residues

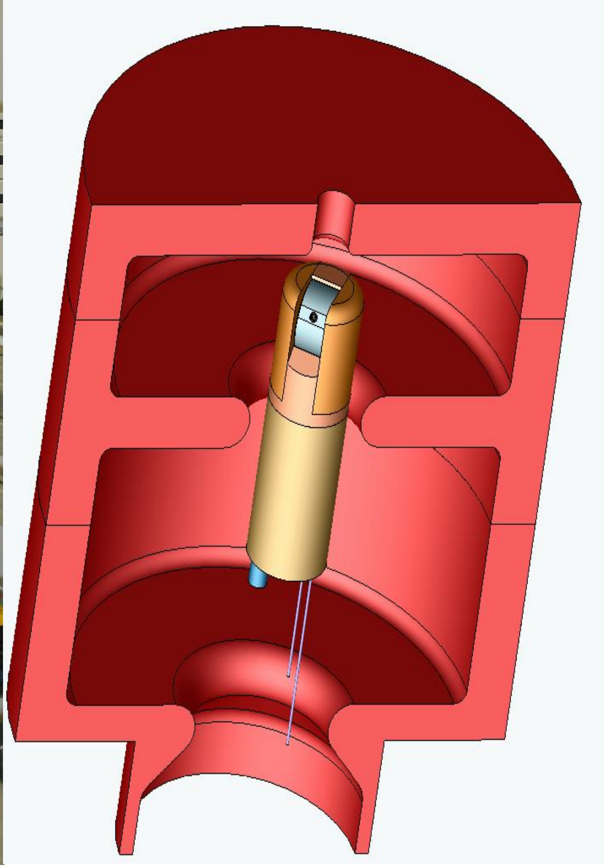


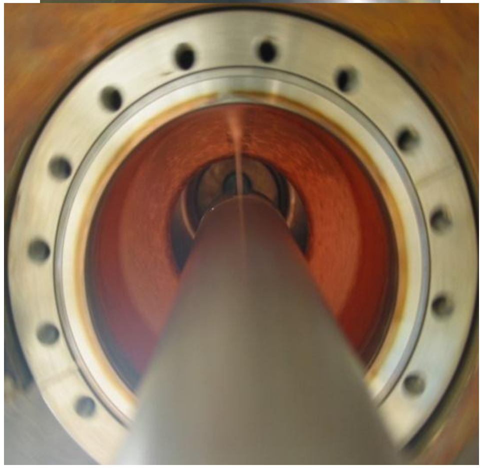
Detlef Rentsche

Alternative approaches: Dry-Ice Cleaning

- Successfully applied to 1-3-cell Nb cavities, but transfer to 9-cell cavities still missing
- Routinely applied to copper gun cavities at 1.3 GHz + 3.9 GHz (“Regae”)







Ultra pure gases: N_2 and Ar

- **Ultra pure gases** and supply systems are **commercially available**
- **Either pressurized or liquefied** (**my preference** is for **liquefied**, no risk of “dirty” bottles)!
- Specification of
  - **Purity** (volume min 99.9995% => 5.5, usual 6.0, 1 ppm)
  - **Minor components** (=> depending on supplier!!!)
- **Particle filtration** in supply system (POU)
- Available POU purifier for water, hydrocarbon and oxygen

Ultra pure gases

- Available large volume treatable filters and purifier based on getter materials. For instance oxysorb / hydrosorb (Messers-Griesheim GmBH), SAES Getters absorbers/purifier for various contaminants, etc
- **Critical**: **Connections** at pressure bottles or dewars and **gas transfer lines major source of contamination**!
- **Critical:** transfer line **certification** **(sensors), mainly for hydrocarbons. Solution: use qualified companies.**

Ultra pure gases: gas purifier

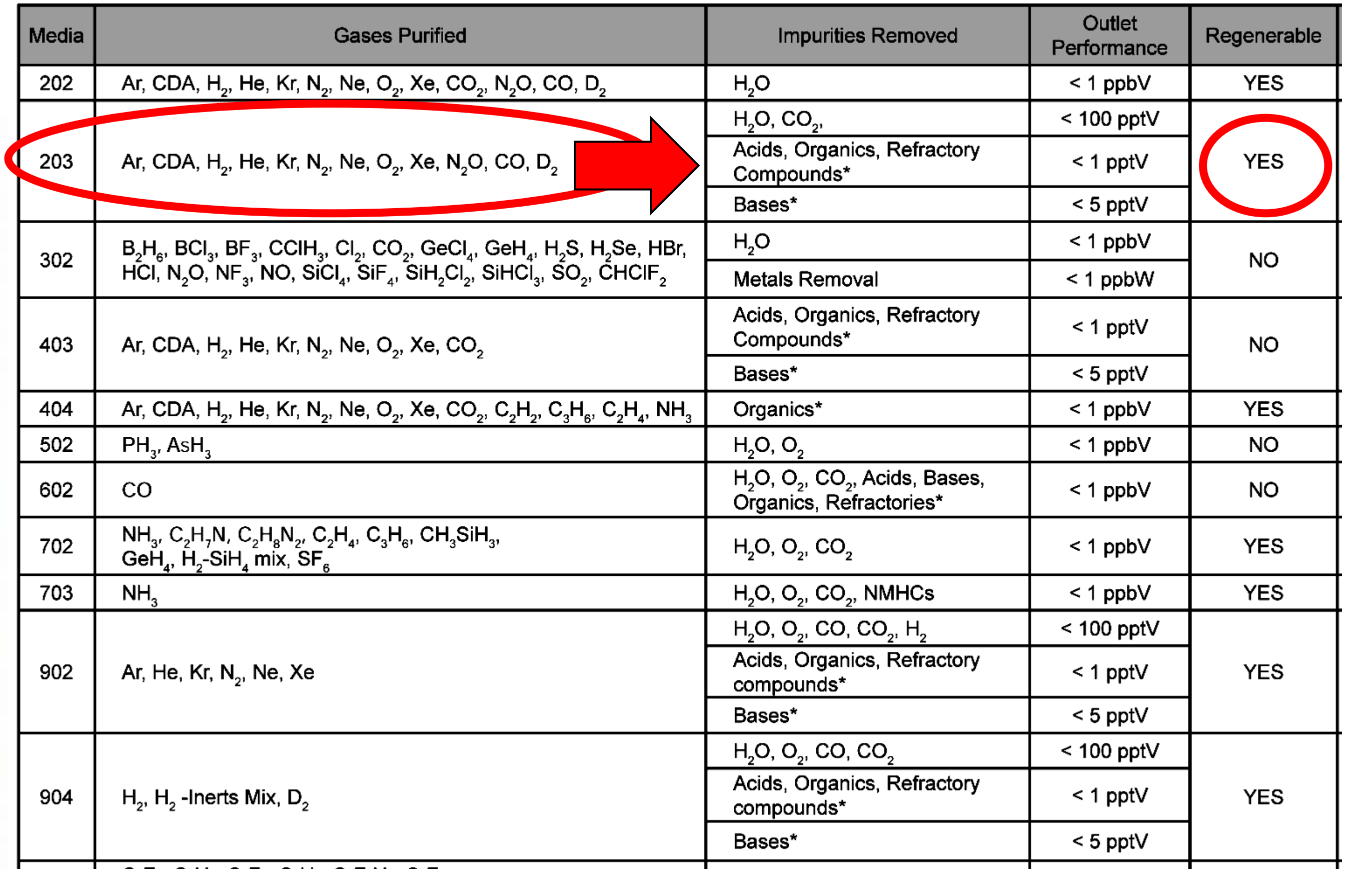
SAES Pure Gas, Inc.
The Technology of Pure Gas
4175 Santa Fe Road, San Luis Obispo, CA 93401
Tel: 1 (805) 541-9299 | Fax: 1 (805) 541-9399

MICRO TORR® Specifications

MC1500

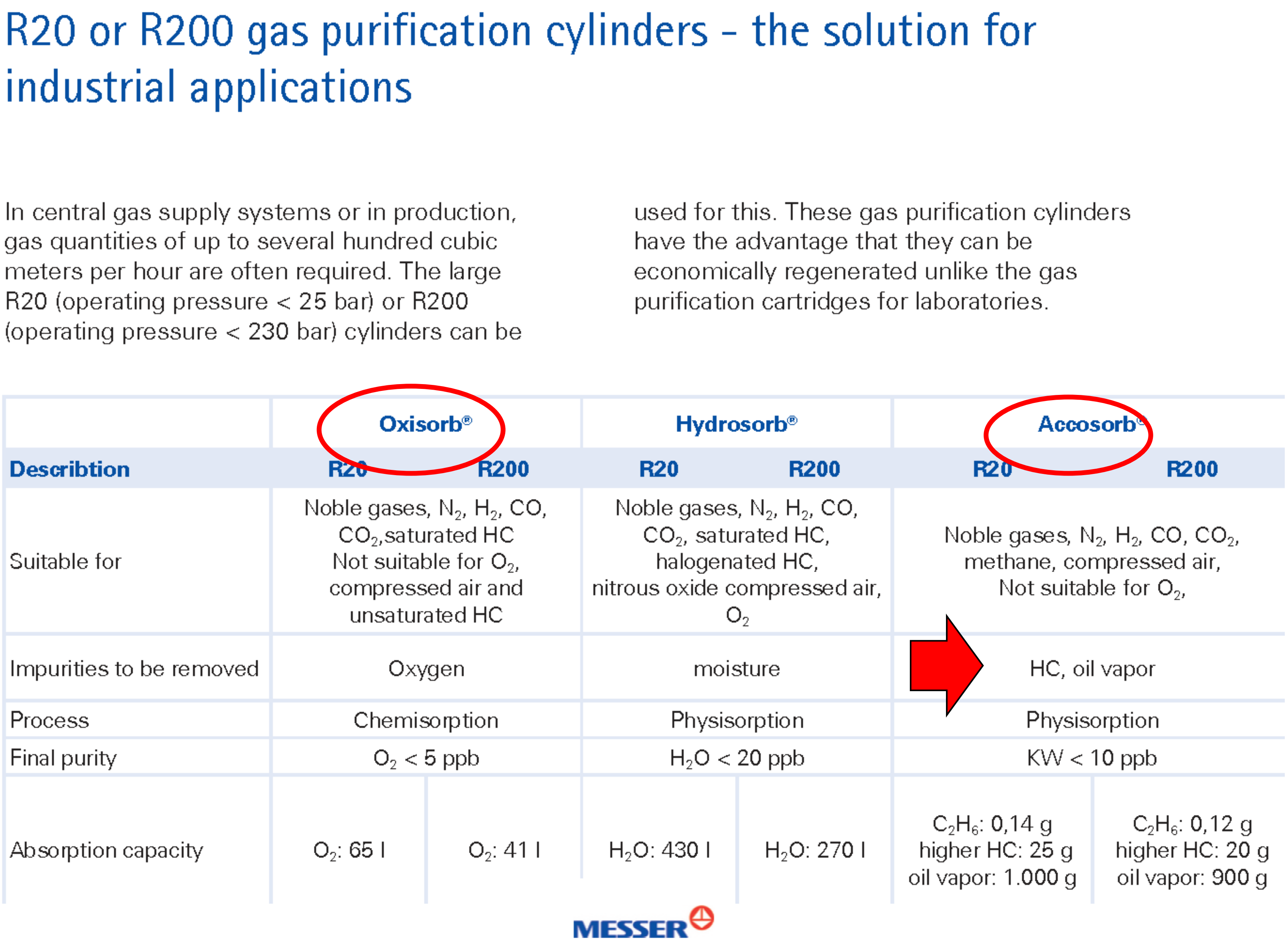
MC1500

MC1500 Purification and Removal Capabilities



Media	Gases Purified	Impurities Removed	Outlet Performance	Regenerable
202	Ar, CDA, H_2, He, Kr, N_2, Ne, O_2, Xe, CO_2, N_2O, CO, D_2	H_2O	< 1 ppbV	YES
203	Ar, CDA, H_2, He, Kr, N_2, Ne, O_2, Xe, N_2O, CO, D_2	H_2O, CO_2,	< 100 pptV	YES
		Acids, Organics, Refractory Compounds*	< 1 pptV	
		Bases*	< 5 pptV	
302	B_2H_6, BCl_3, BF_3, $CClH_3$, Cl_2, CO_2, $GeCl_4$, GeH_4, H_2S, H_2Se, HBr, HCl, N_2O, NF_3, NO, $SiCl_4$, SiF_4, SiH_2Cl_2, $SiHCl_3$, SO_2, $CHClF_2$	H_2O	< 1 ppbV	NO
		Metals Removal	< 1 ppbW	
403	Ar, CDA, H_2, He, Kr, N_2, Ne, O_2, Xe, CO_2	Acids, Organics, Refractory Compounds*	< 1 pptV	NO
		Bases*	< 5 pptV	
404	Ar, CDA, H_2, He, Kr, N_2, Ne, O_2, Xe, CO_2, C_2H_2, C_3H_6, C_2H_4, NH_3	Organics*	< 1 ppbV	YES
502	PH_3, AsH_3	H_2O, O_2	< 1 ppbV	NO
602	CO	H_2O, O_2, CO_2, Acids, Bases, Organics, Refractories*	< 1 ppbV	NO
702	NH_3, C_2H_7N, $C_2H_8N_2$, C_2H_4, C_3H_6, CH_3SiH_3, GeH_4, H_2-SiH_4 mix, SF_6	H_2O, O_2, CO_2	< 1 ppbV	YES
703	NH_3	H_2O, O_2, CO_2, NMHCs	< 1 ppbV	YES
902	Ar, He, Kr, N_2, Ne, Xe	H_2O, O_2, CO, CO_2, H_2	< 100 pptV	YES
		Acids, Organics, Refractory compounds*	< 1 pptV	
		Bases*	< 5 pptV	
904	H_2, H_2 -Inerts Mix, D_2	H_2O, O_2, CO, CO_2	< 100 pptV	YES
		Acids, Organics, Refractory compounds*	< 1 pptV	
		Bases*	< 5 pptV	

Ultra pure gases: gas purifier



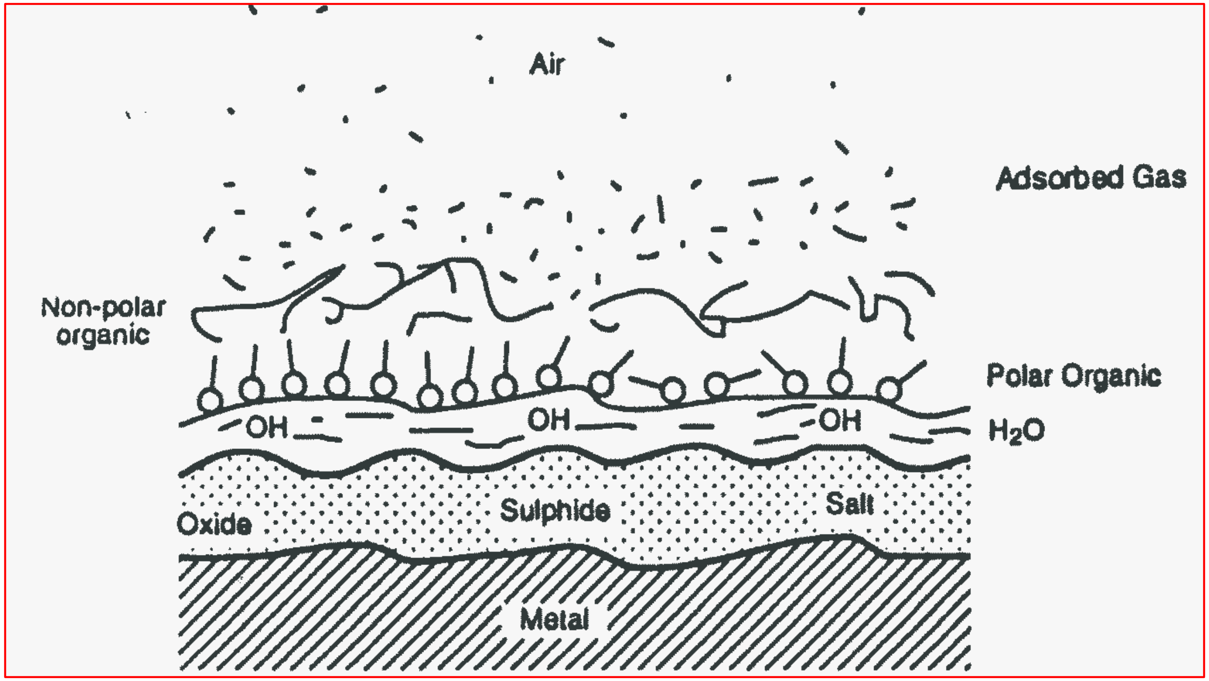
R20 or R200 gas purification cylinders – the solution for industrial applications

In central gas supply systems or in production, gas quantities of up to several hundred cubic meters per hour are often required. The large R20 (operating pressure < 25 bar) or R200 (operating pressure < 230 bar) cylinders can be used for this. These gas purification cylinders have the advantage that they can be economically regenerated unlike the gas purification cartridges for laboratories.

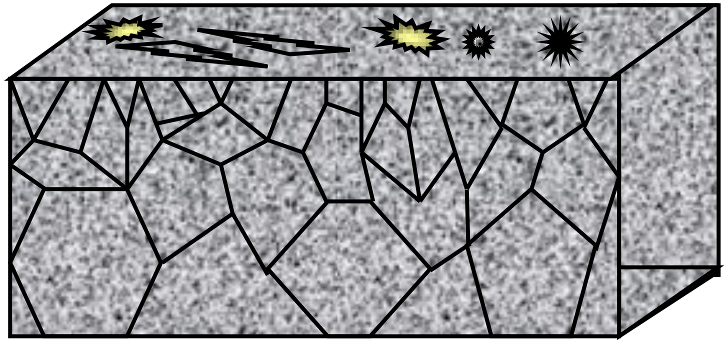
	Oxisorb®		**Hydrosorb®**		**Accosorb®**	
Describtion	**R20**	**R200**	**R20**	**R200**	**R20**	**R200**
Suitable for	Noble gases, N_2, H_2, CO, CO_2,saturated HC Not suitable for O_2, compressed air and unsaturated HC		Noble gases, N_2, H_2, CO, CO_2, saturated HC, halogenated HC, nitrous oxide compressed air, O_2		Noble gases, N_2, H_2, CO, CO_2, methane, compressed air, Not suitable for O_2,	
Impurities to be removed	Oxygen		moisture		HC, oil vapor	
Process	Chemisorption		Physisorption		Physisorption	
Final purity	O_2 < 5 ppb		H_2O < 20 ppb		KW < 10 ppb	
Absorption capacity	O_2: 65 l	O_2: 41 l	H_2O: 430 l	H_2O: 270 l	C_2H_6: 0,14 g higher HC: 25 g oil vapor: 1.000 g	C_2H_6: 0,12 g higher HC: 20 g oil vapor: 900 g

MESSER

Prepare inner surface for the RF field



“Contaminated” **metal** surface exposed to air, moisture, water, oils. Scratches, dust particle are also on the surface.

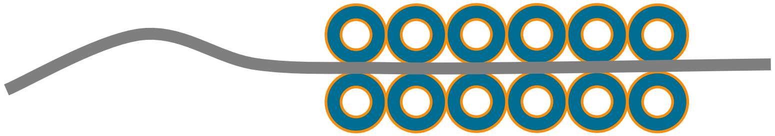


- The Nb surface present also a **damage layer**: due to rolling, forming, machining.
- Broken grains, smashed grains, residual strain are present.

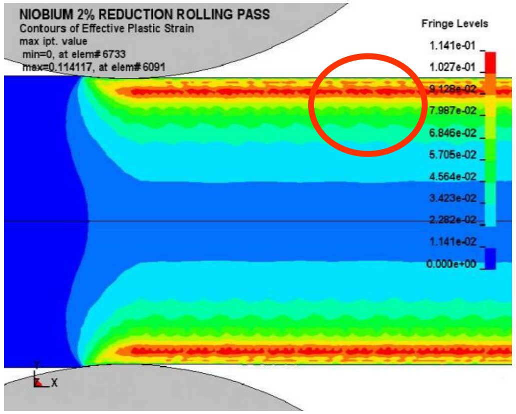
WHY SURFACE POLISHING?

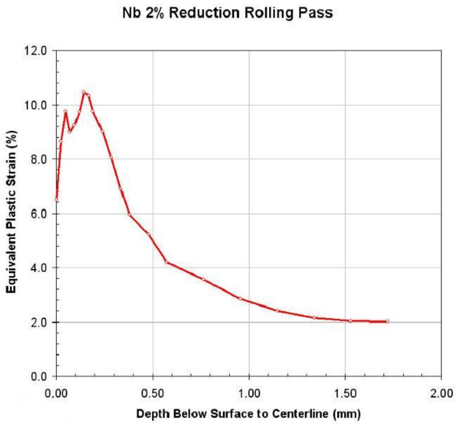
=>DAMAGE LAYER

After rolling sheets undergo a skin pass for planarity



[R. Crook et al, Black Laboratory]



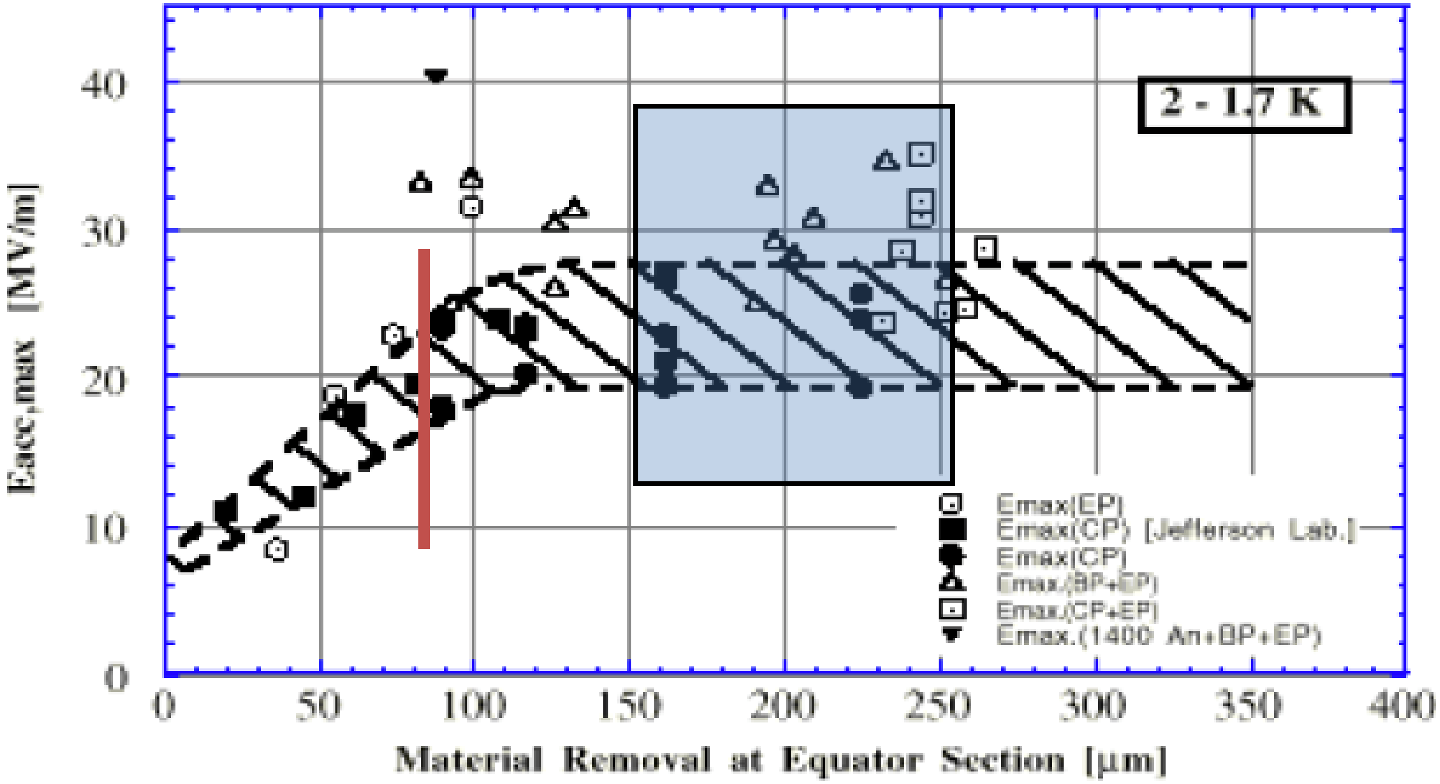


Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls (Courtesy Non-Linear Engineering, L.L.C.). Stress is concentrated in the near-surface region (~300 µm). Localized strain exceeds the average by a factor of 5

- Damage layer = deformed grains + high density of dislocations + (foreign atoms)
- Rolling leaves a damage layer ~2-300 µm with a texture resistant to recrystallization, i.e. same order of magnitude than the necessary etching of material.
- Further damage (dislocations !) probably brought by deep drawing and thermal strain during welding
- Interesting trails :
  - look at remaining stress after forming/welding,
  - chemical mechanical polishing

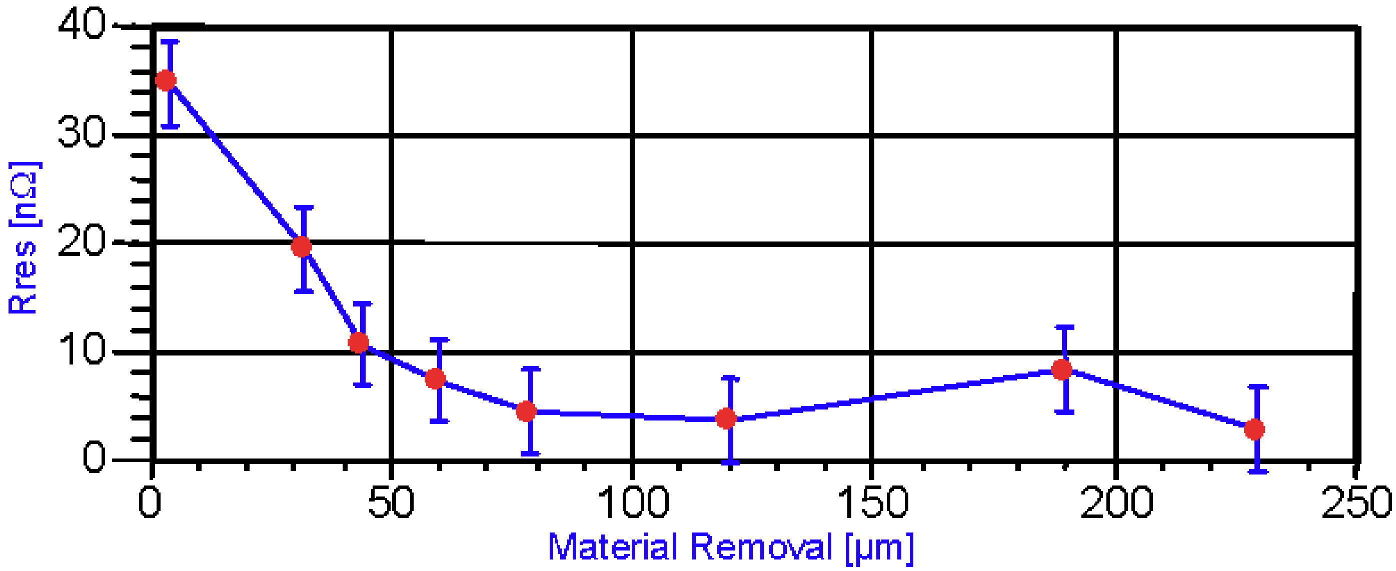
Nb removal

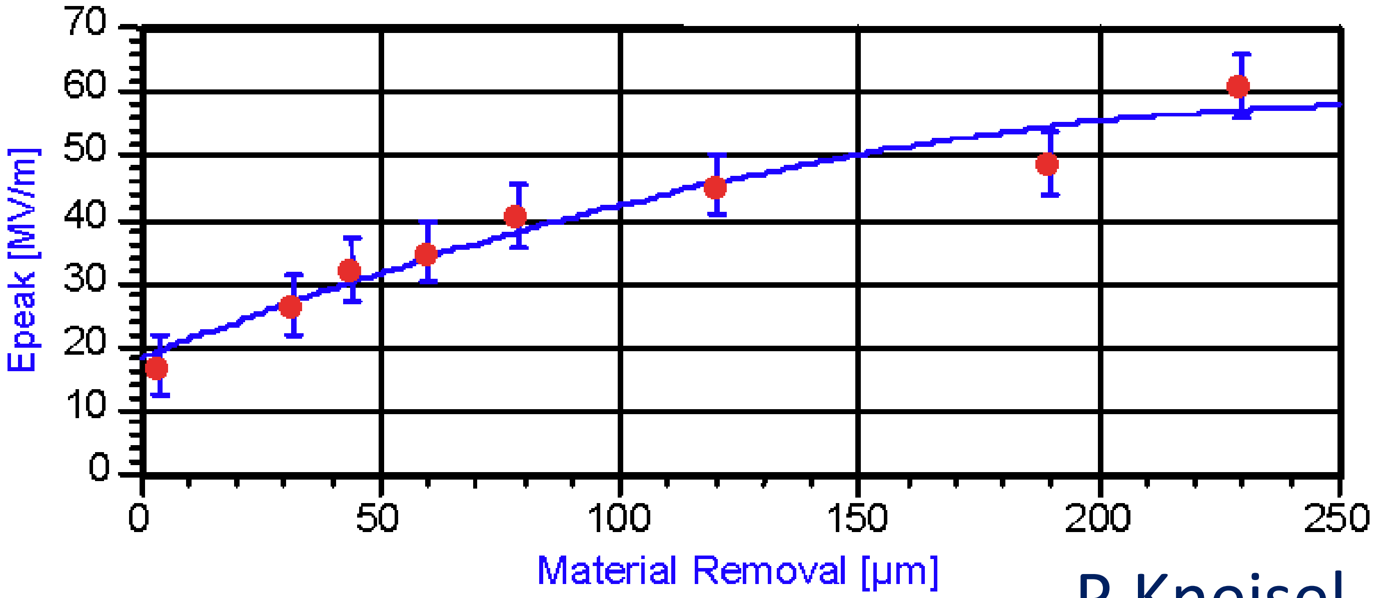
- After deep drawing, EBW and other machining, a removal of the Nb damaged layer (100 – 200 μm) is needed.



K. Saito

Nb removal. E_{peak} and R_{res} vs. material removal





P.Kneisel

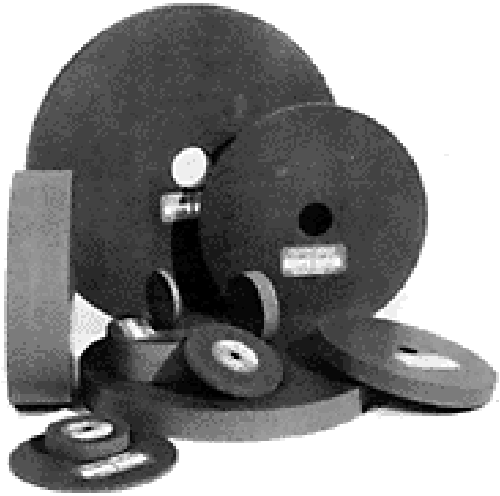
Nb damaged layer removal techniques

- Nb damaged layer to be removed: (100 – 200 μm)
- Two different strategy:
  - **Mechanical removal**
    - Grinding (local)
    - Tumbling
  - **Chemical removal**
    - BCP (Buffered Chemical Polishing)
    - EP (Electropolishing)

Mechanical removal of damaged layer

Grinding

- **Simple handling**
- **Low cost** standard
- Grinding mostly in use for removal of local defects: **non uniform abrasion** !
- Abrasives need to be **qualified** on superconductive cavities!
- **Remain of abrasive** (**SiC, WC, Al_2O_3, etc**), glue: grain size of **abrasive particle** must be **below** the next **etching step removal**
- Produces a **new damage** layer that **must be etched.**



Rubberized Abrasives

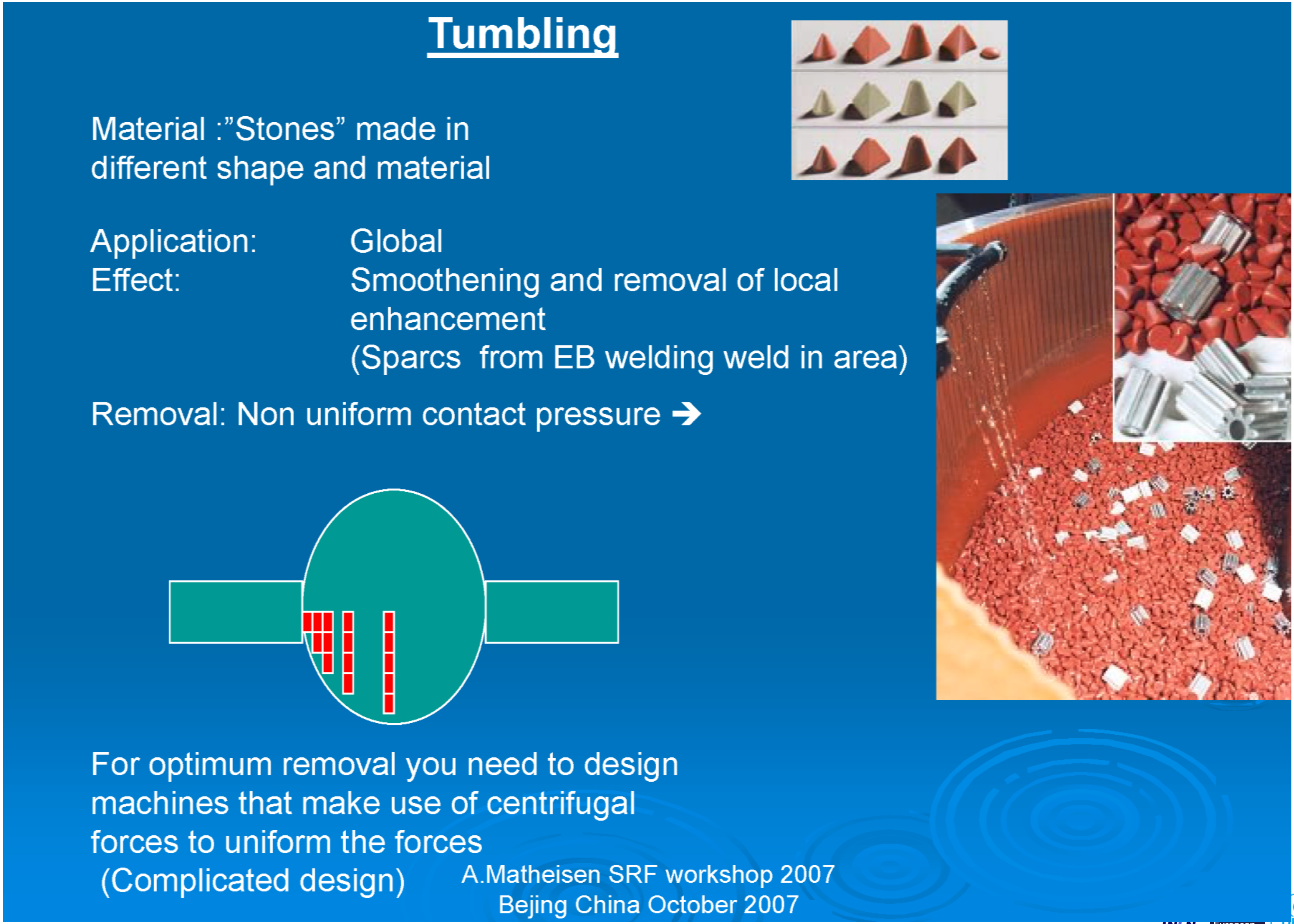
Not heat up Nb surface!

Mechanical removal of damaged layer

Tumbling

- **Simple** standard mechanic, but **tumbling machine** cost is some **hundreds of k€**
- Abrasives need to be qualified on superconductive cavities!
- **Remain of abrasive** (**SiC, WC, Al_2O_3, etc**): grain size of **abrasive particle** must be **below** the next **etching step removal**
- Produces a **new damage** layer of about **40 µm** thickness that **must be etched.**

Mechanical removal of the damaged layer



Tumbling

Material :”Stones” made in different shape and material

Application:	Global
Effect:	Smoothening and removal of local enhancement (Sparcs from EB welding weld in area)

Removal: Non uniform contact pressure ➔

For optimum removal you need to design machines that make use of centrifugal forces to uniform the forces
(Complicated design)

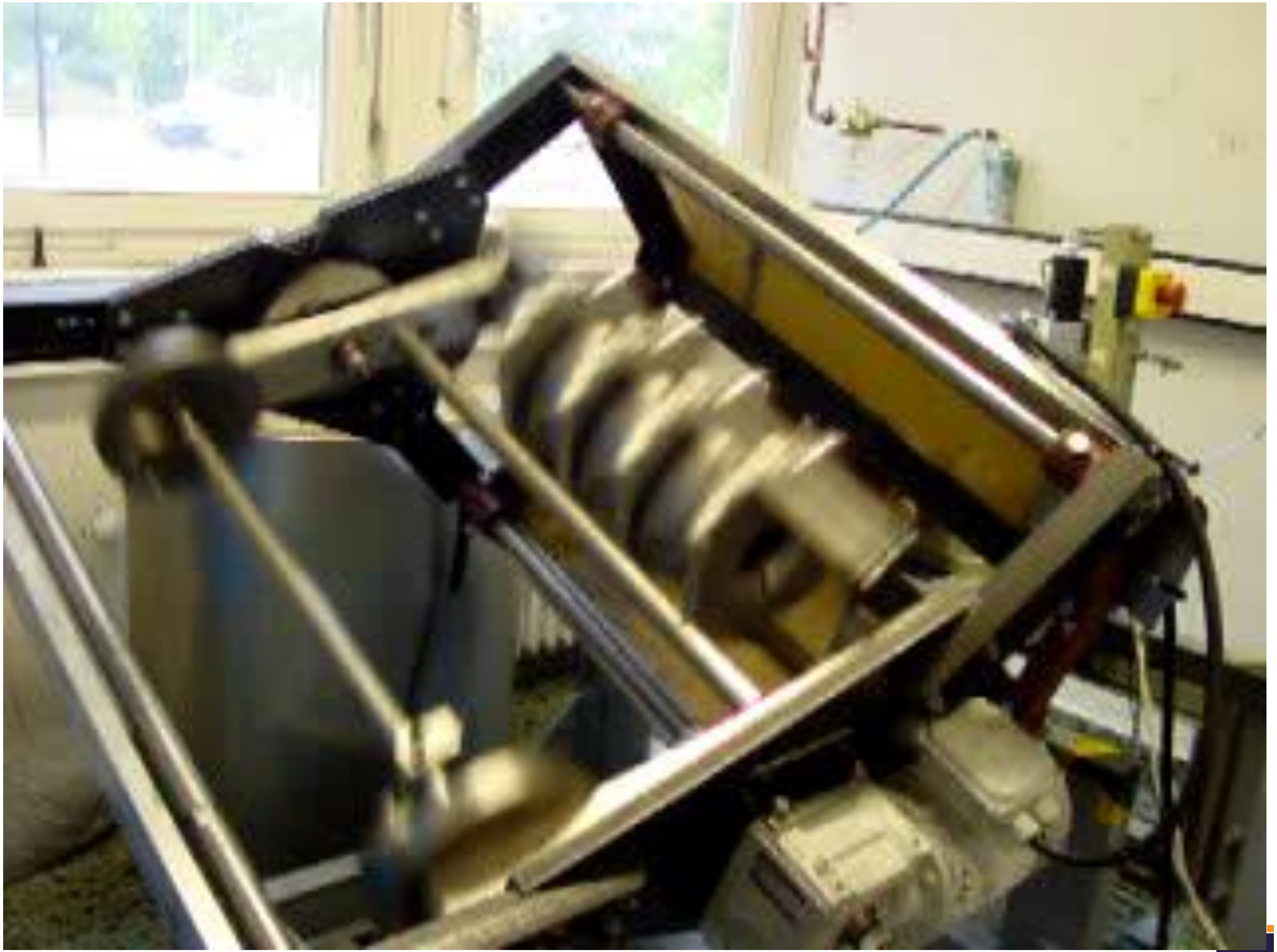
A.Matheisen SRF workshop 2007
Bejing China October 2007

Mechanical removal of the damaged layer

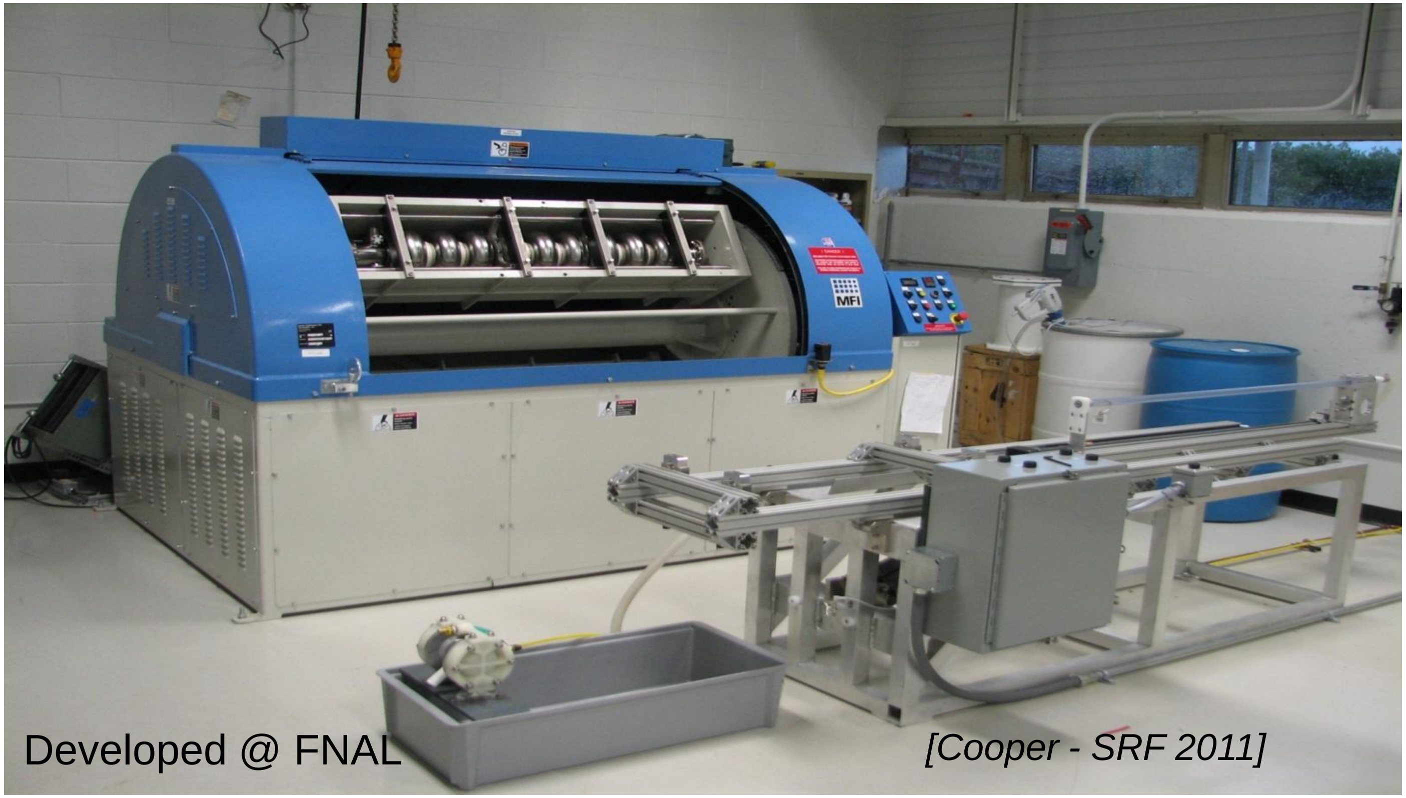
Example of a tumbling machine
Designed an manufactured by DESY group MPL Waldemar Singer

A.Matheisen SRF workshop 2007
Bejing China October 2007

Mechanical removal of the damaged layer



Mechanical removal of the damaged layer



Developed @ FNAL

[Cooper - SRF 2011]

Individual Barrels rotate 115 RPM in opposite direction to main shaft

Abrasive of different shape & roughness to obtain mirror-like surface

BCP & EP: (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).**

BCP & EP: (electro) chemical Nb removal

Two types of chemical removal process are commonly used

- **BCP: Buffered Chemical Polishing**
- Mixture of Hydrofluoric acid (**HF**), Nitric acid (**HNO_3**) and Phosphoric Acid (**H_3PO_4**)

and / or

- **EP: Electropolishing**
- Mixture of Hydrofluoric acid (HF) and Sulfuric Acid (H_2SO_4) + **electrical current**

BCP: Buffered Chemical Polishing

- Mixture of concentrated Hydrofluoric acid (**HF, 40%**), Nitric acid (**HNO_3**, 70%) and Phosphoric Acid (**H_3PO_4, 85%**)
- Different ratios used (HF, HNO_3, H_3PO_4), usually HF: 1, HNO_3: 1 **H_3PO_4 from 0 to >10**. **1:1:2 more frequently used.**
- 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

$$2NO_2 \Rightarrow N_2O_4$$

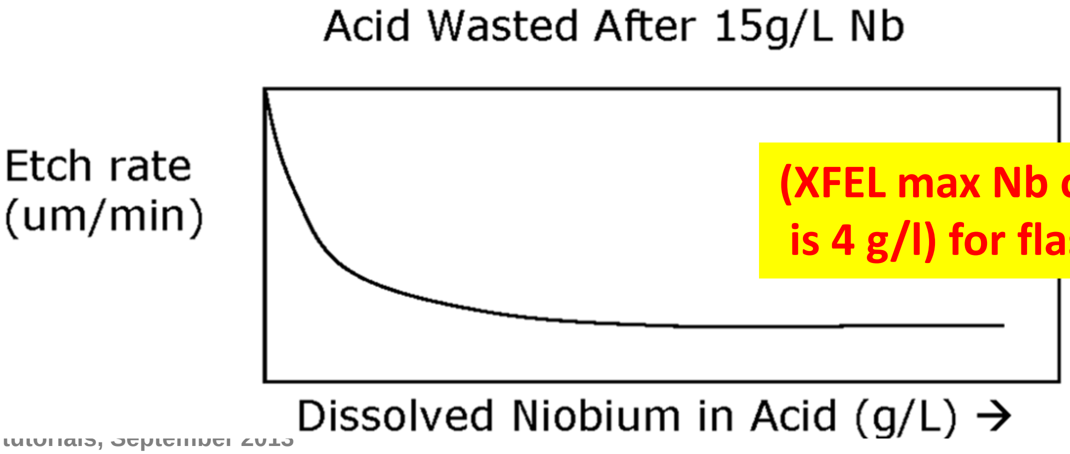
Nb Oxide Dissolving

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

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

BCP systems

- **Close loop flow system**, in some case gravity fed system design
- **Reagent grade** (pure) chemicals are needed.
- **BCP mixture** can be bought (Honeywell, microelectronics industry).
- **BCP MUST be mixed before the use**: it tends to stratify.
- **Etching rate** for 1:1:2: about **1 μm/min,** with fresh acid.
- **Reaction speed** is proportional to the **temperature**.
- BCP mixture compatible materials: **PTFE, PVDF, VITON,** etc
- **Acid with high Nb** concentration have **reduce corrosion speed**.



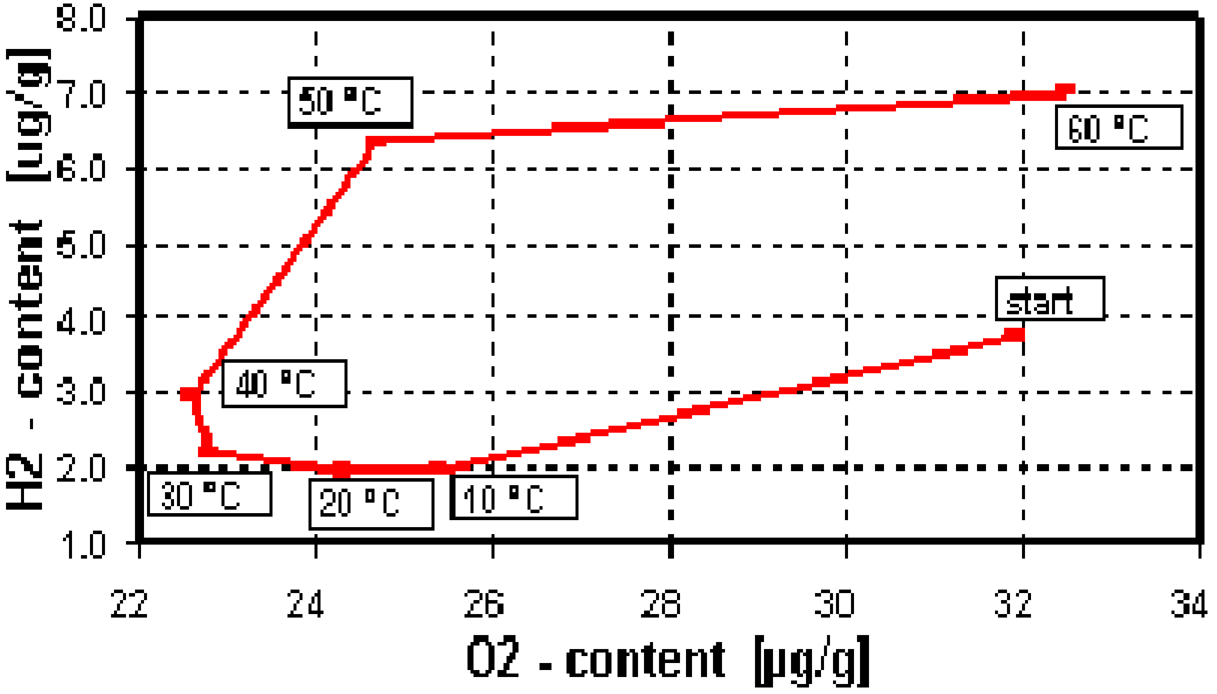
(XFEL max Nb content is 4 g/l) for flash BCP

BCP systems

- **All components** in the acid mixture circuit **MUST be resistant** to acid attack: pumps, flow meters, valves, tubes and flex connections, etc.
- 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 H_2 in Nb

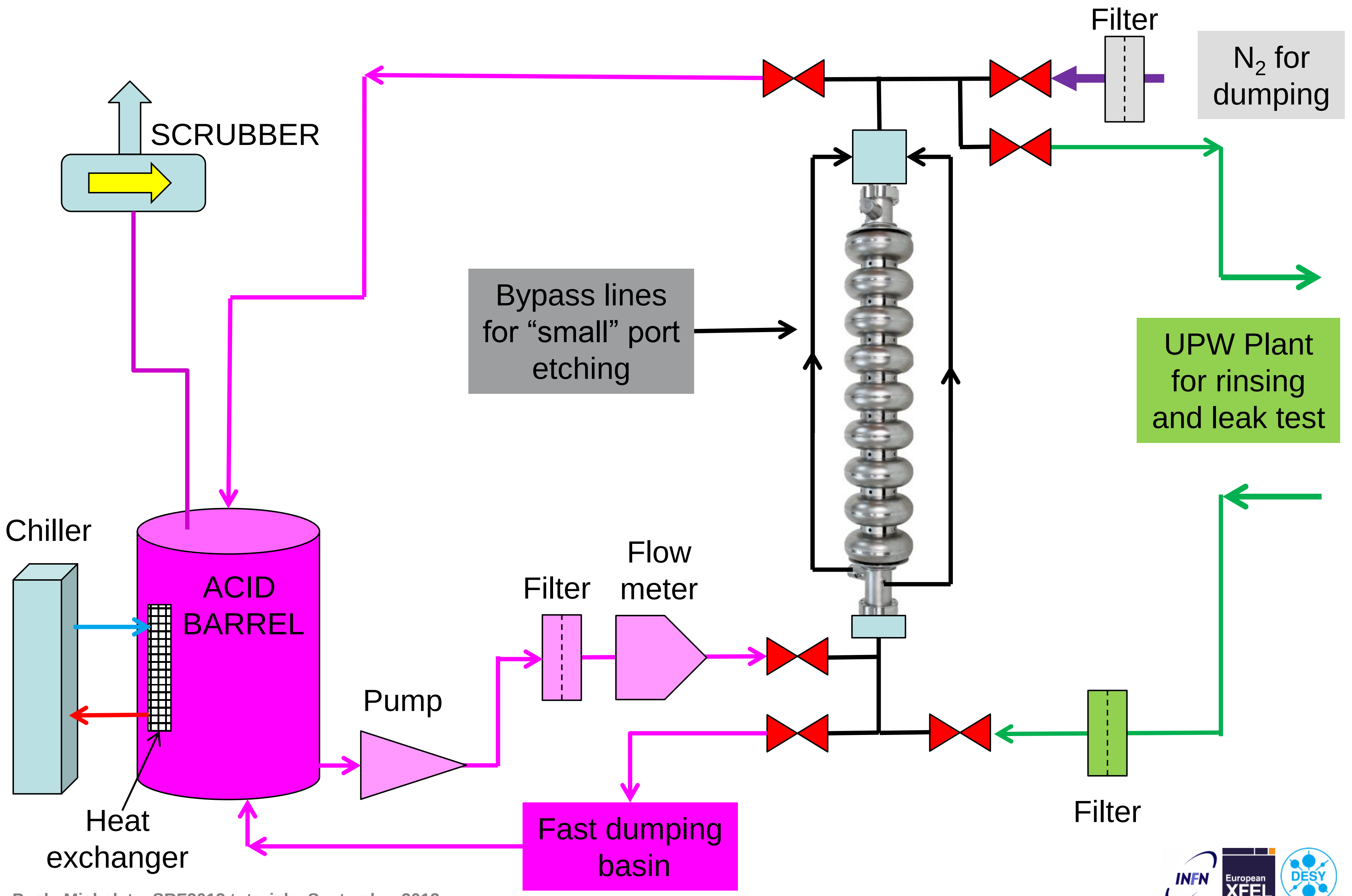
- **Chemical (wet)** treatments could **increase H_2 and O_2 in Nb**
- Effect depends on mixture and bath temperature
- For BCP mixture **must be used cold**, max temperature below 20°C, **also during transients** (cavity filling)



Gas content of Nb at different etching temperatures (Schölz).

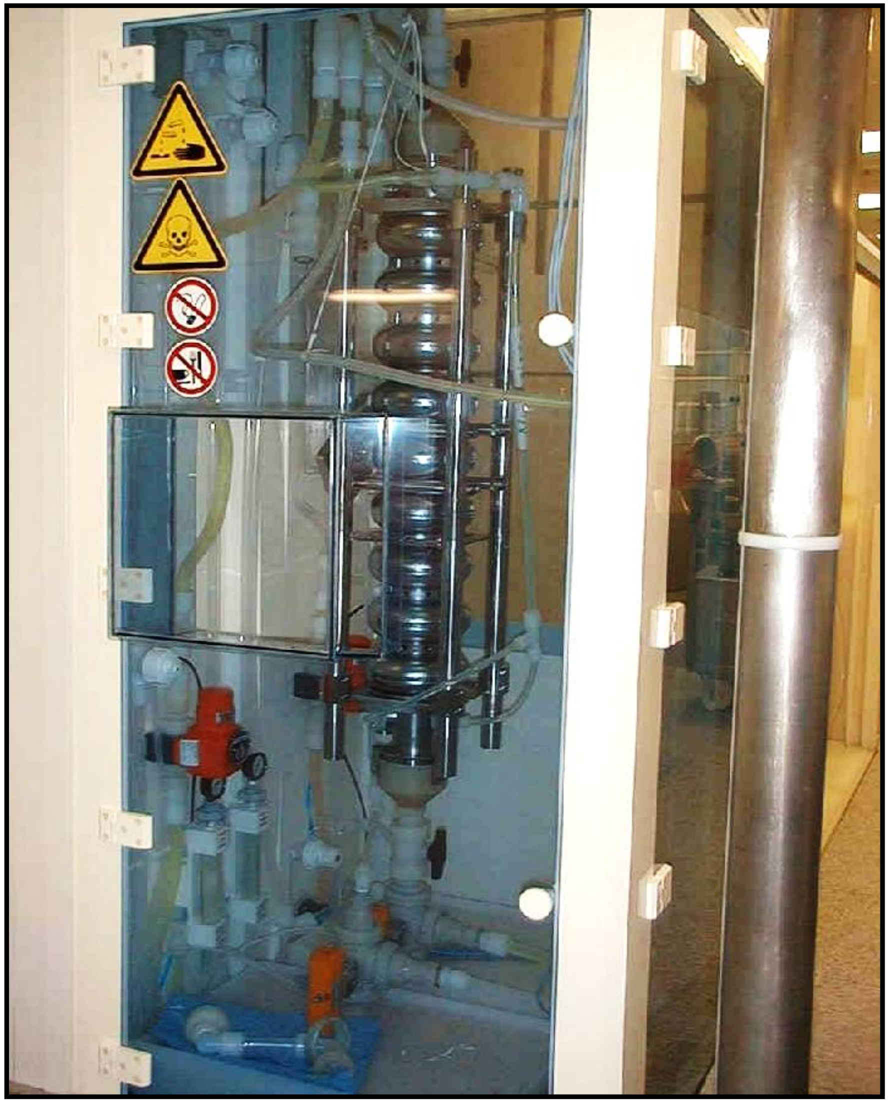
Etching time 20 min, BCP 1:1:4

BCP systems: typical plant



BCP cabinets in the labs


BCP Cabinet JLab

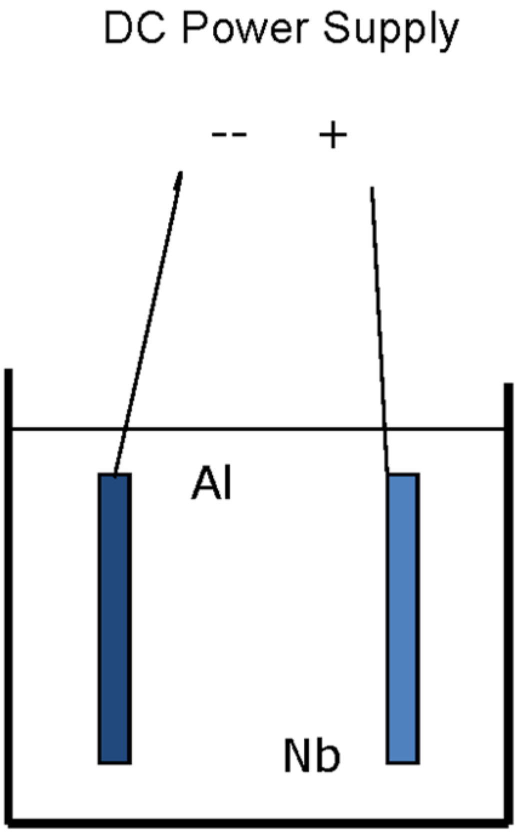

DESY BCP

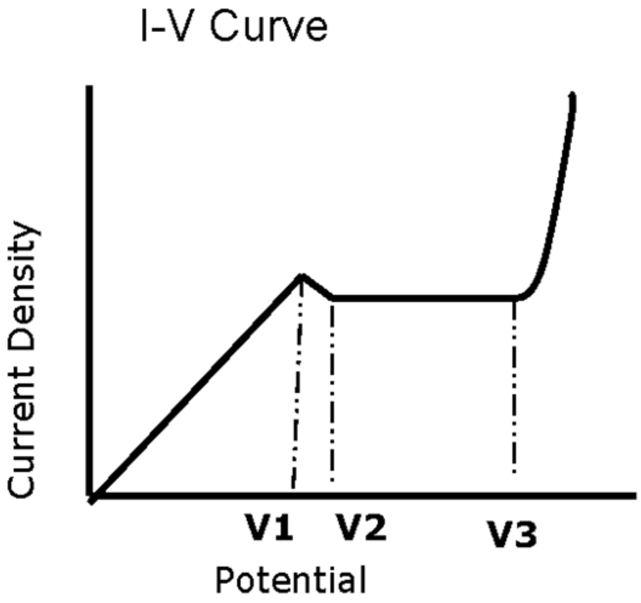
XFEL BCP for small components @ EZ

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

XFEL inner BCP systems at industry

Basic concepts for EP



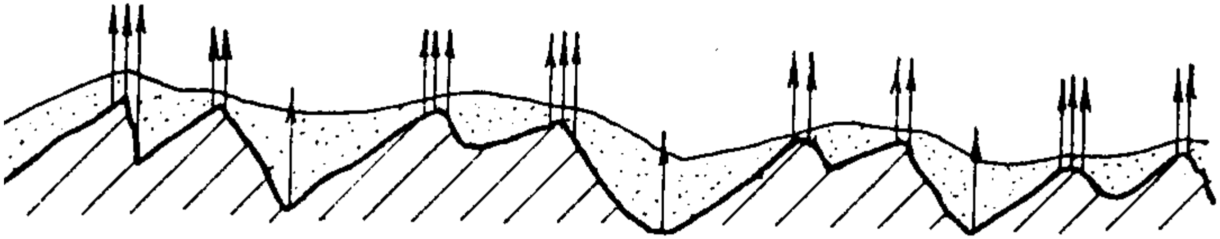


- **0-V1- Concentration Polarization occurs, active dilution of niobium, electrolyte resistance**
- **V2-V3 – Limiting Current Density, viscous layer on niobium surface**
- **>V3 Additional Cathodic Processes Occur, oxygen gas generated**

EP: KEK recipe. HF + H_2SO_4, 1:9

Electrolyte	10% HF (49%), 90% H_2SO_4 (96%)
Voltage	ca. 25 V
Current density	30-100 mA/cm^2
Temperature	30-35°C
Removal rate	0.5 µm/min

Successful experience both at constant current and constant voltage.



Schematic of **viscous layer** of electrolyte forming near the Nb anode **The current at this layer experienced higher resistivity.** The current density becomes **higher at protrusions and they dissolve faster**

EP: Electropolishing

- Mixture of concentrated **HF (49%**) and Sulfuric Acid (**H_2SO_4, 96%**)
- Usual ratio is 1:9. **1 part of HF, 9 parts of H_2SO_4**
- Reaction is not self exiting: **no current, no etching**.
- Etching rate (@ 17 V, constant voltage): 0.3 0.5 μm/min (50% of BCP 1:1:2)
- During process other reactions take place and **SULFUR** is produced

Oxidation

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

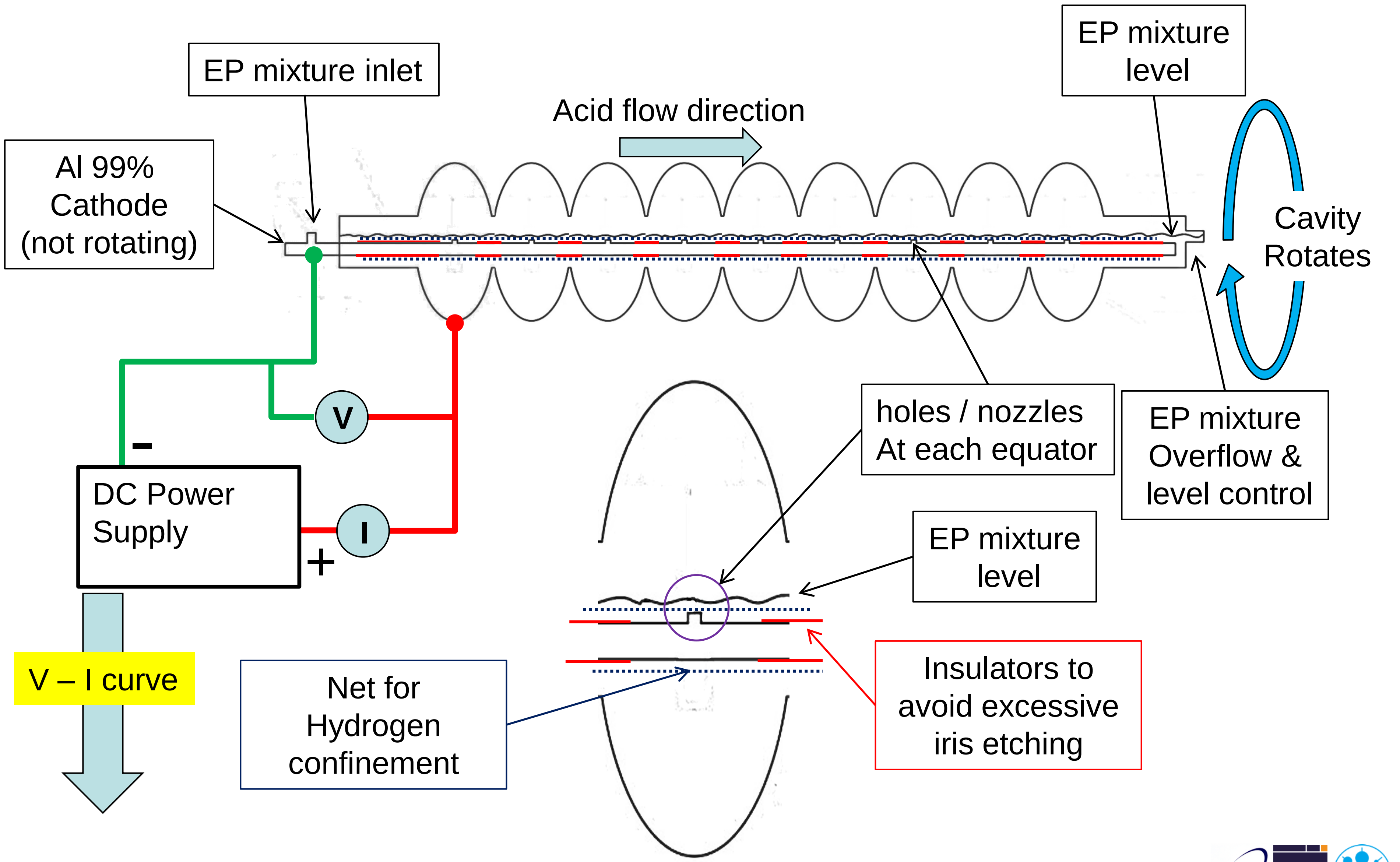
Hydrogen!

Nb Oxide Dissolving (as for BCP)

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

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

Basic concepts for EP for cavities



EP mixture inlet

Acid flow direction

EP mixture level

Al 99% Cathode (not rotating)

Cavity Rotates

holes / nozzles At each equator

EP mixture Overflow & level control

DC Power Supply

V – I curve

EP mixture level

Net for Hydrogen confinement

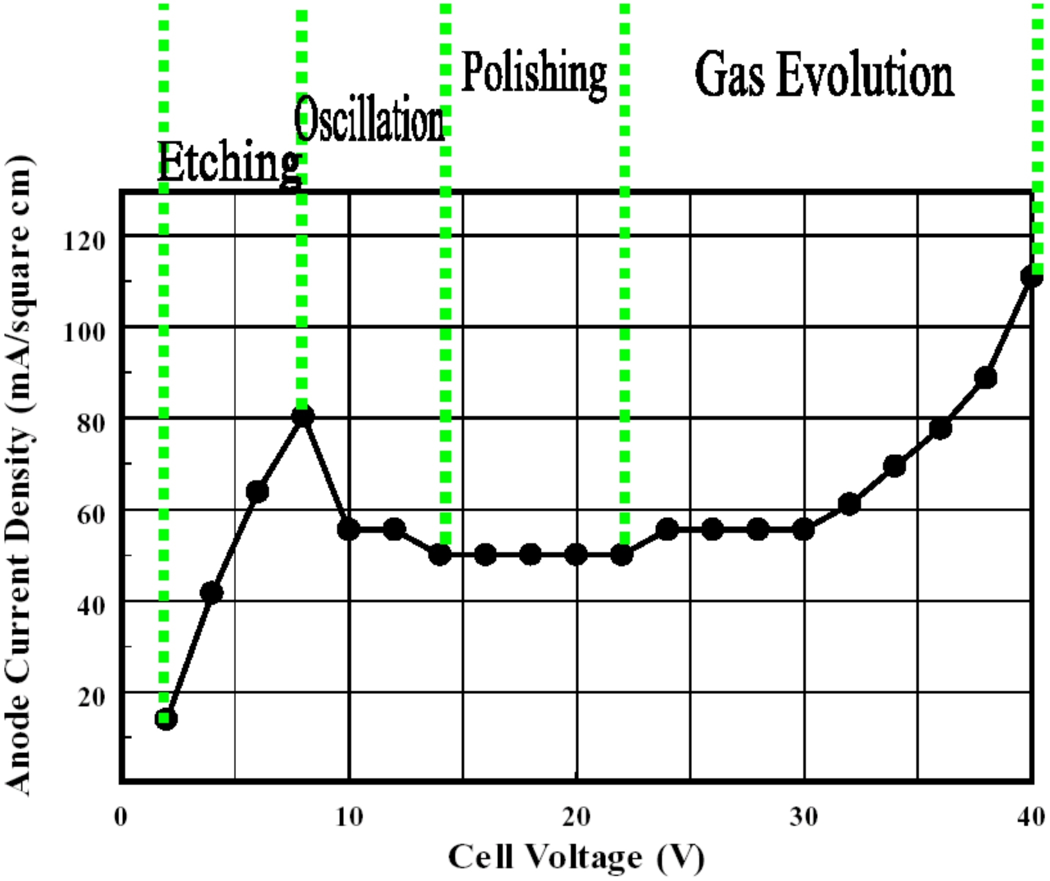
Insulators to avoid excessive iris etching

EP of Nb cavities

- **Cavity (or electrode) is rotating**
- **Close loop flow system**
- **EP mixture** can be bought at **microelectronics industry** (Honeywell)
- **EP mixture compatible materials: PTFE, PVDF, VITON, etc**
- All components **MUST** be resistant to acid attack: pumps, flow meters, valves, tubes, flex connections, etc.
- Final etching have to use **fresh acid**: max Nb concentration is **5 g/l**
- **Cavity** generally held in **horizontal position**, but successful experience for vertical EP are done **(CEA, Cornell)**
- Usually **etching rate on iris is 2 x the equator one**
- **Temperature gradient** causes **increased etching** from one end to the other
- **Used both for bulk removal and final etching: for EXFEL only for final etching of half of cavities (RI)**

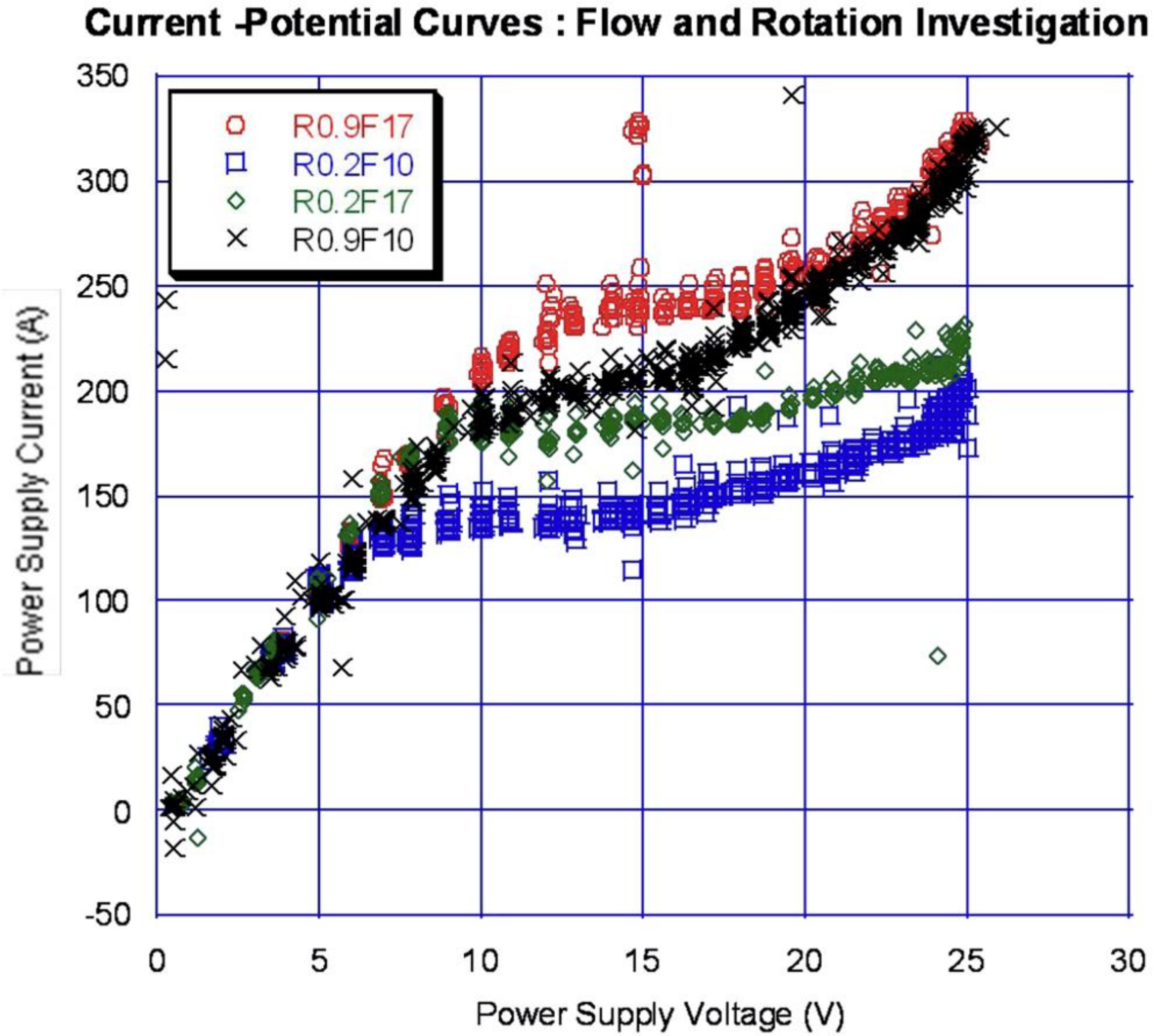
EP: KEK recipe. HF + H_2SO_4

I – V curve that we expect to have, BUT

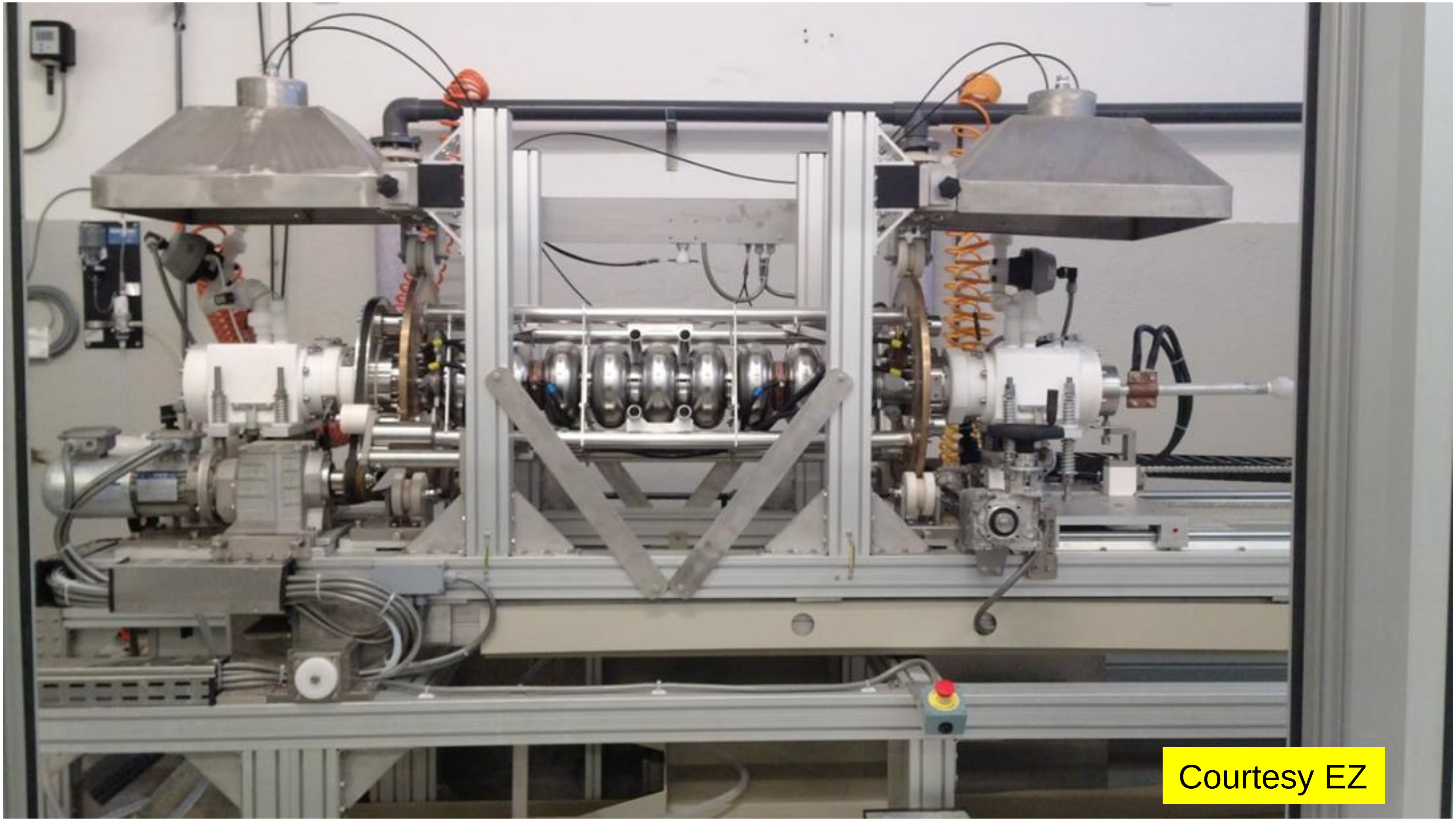


Nb EP: I-V curve not easy to interpret

Cavity is rotating, and viscous layer is disturbed.

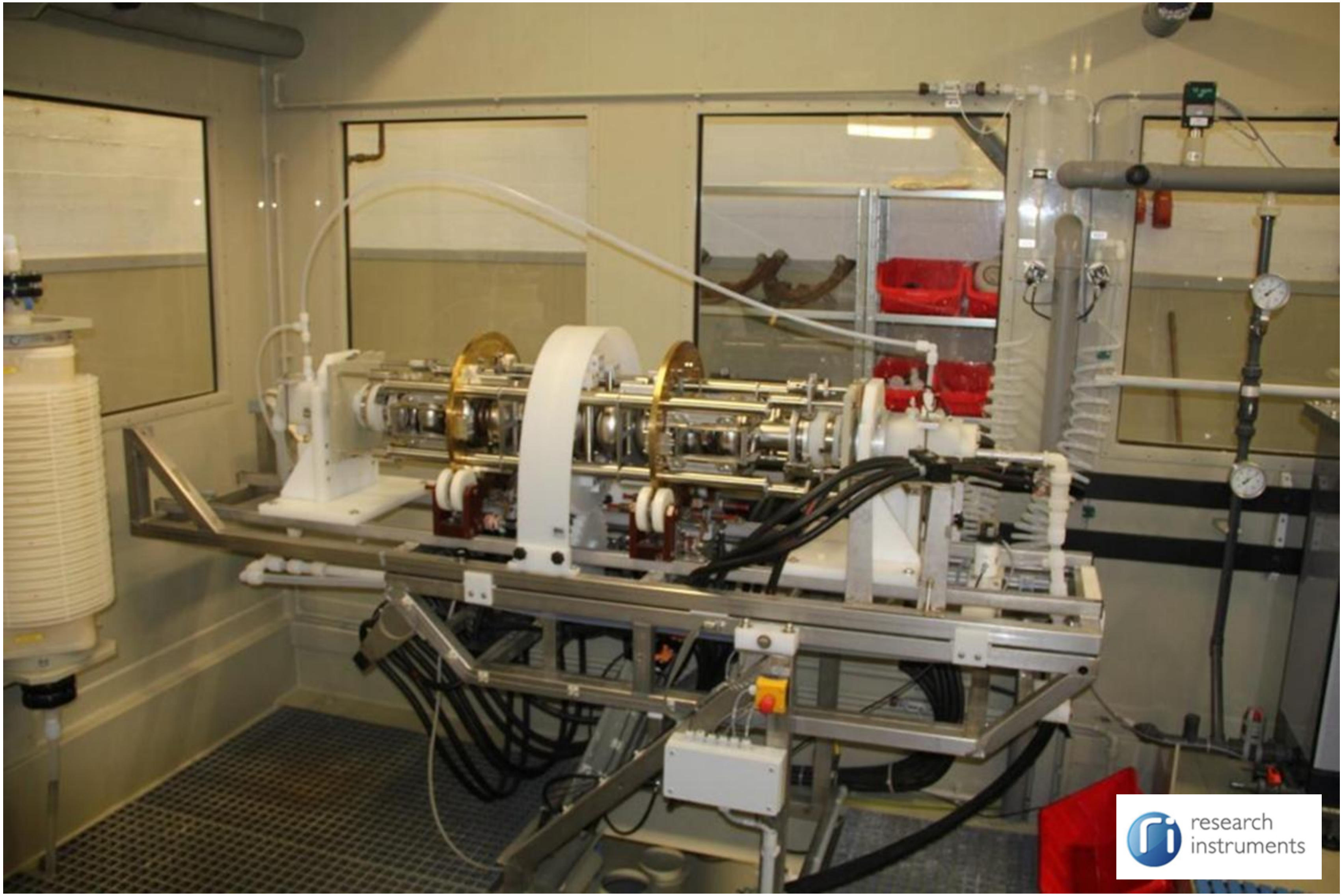


EP system at Ettore Zanon (EZ)

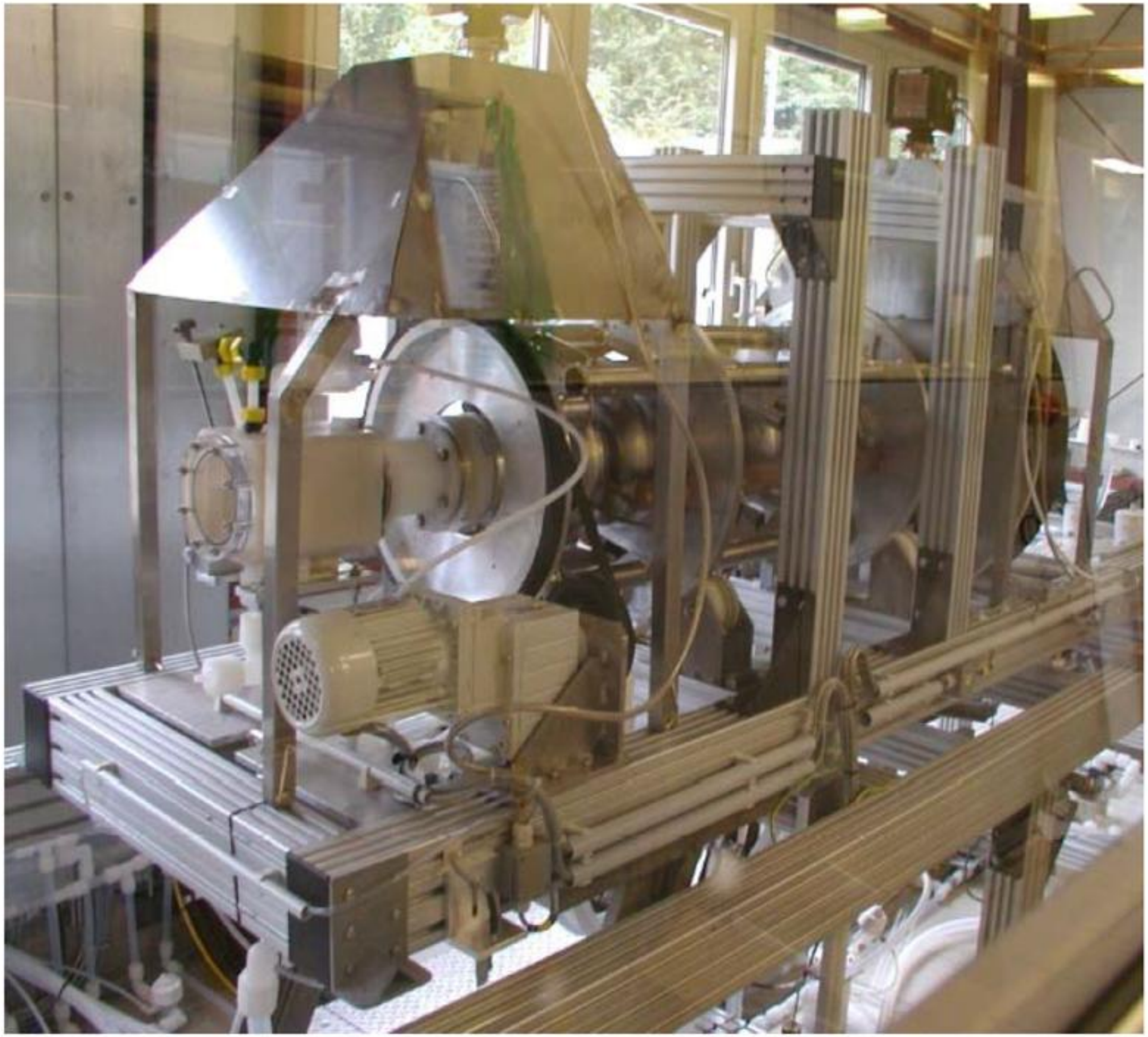


Courtesy EZ

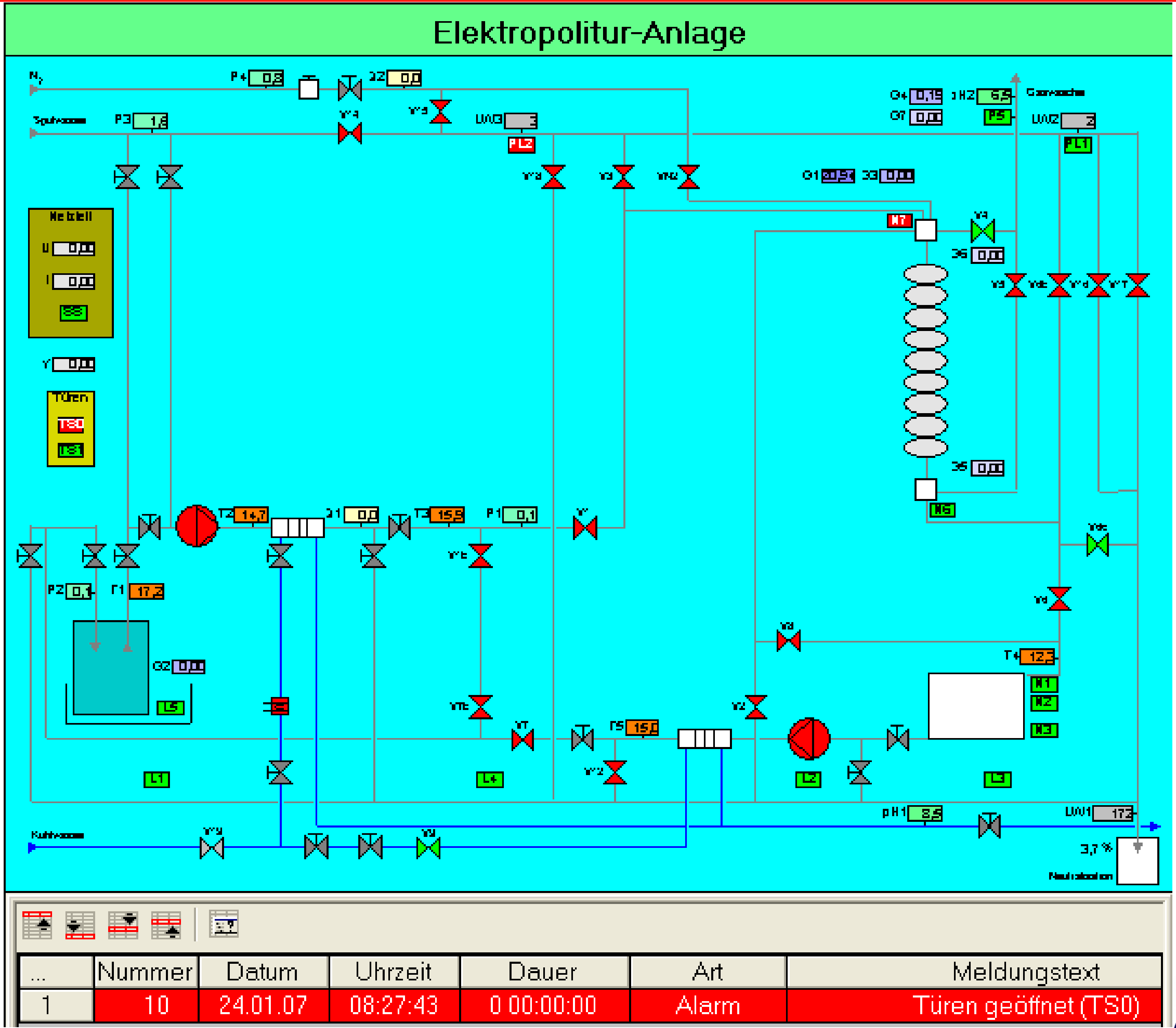
EP system at RI



EP system: DESY



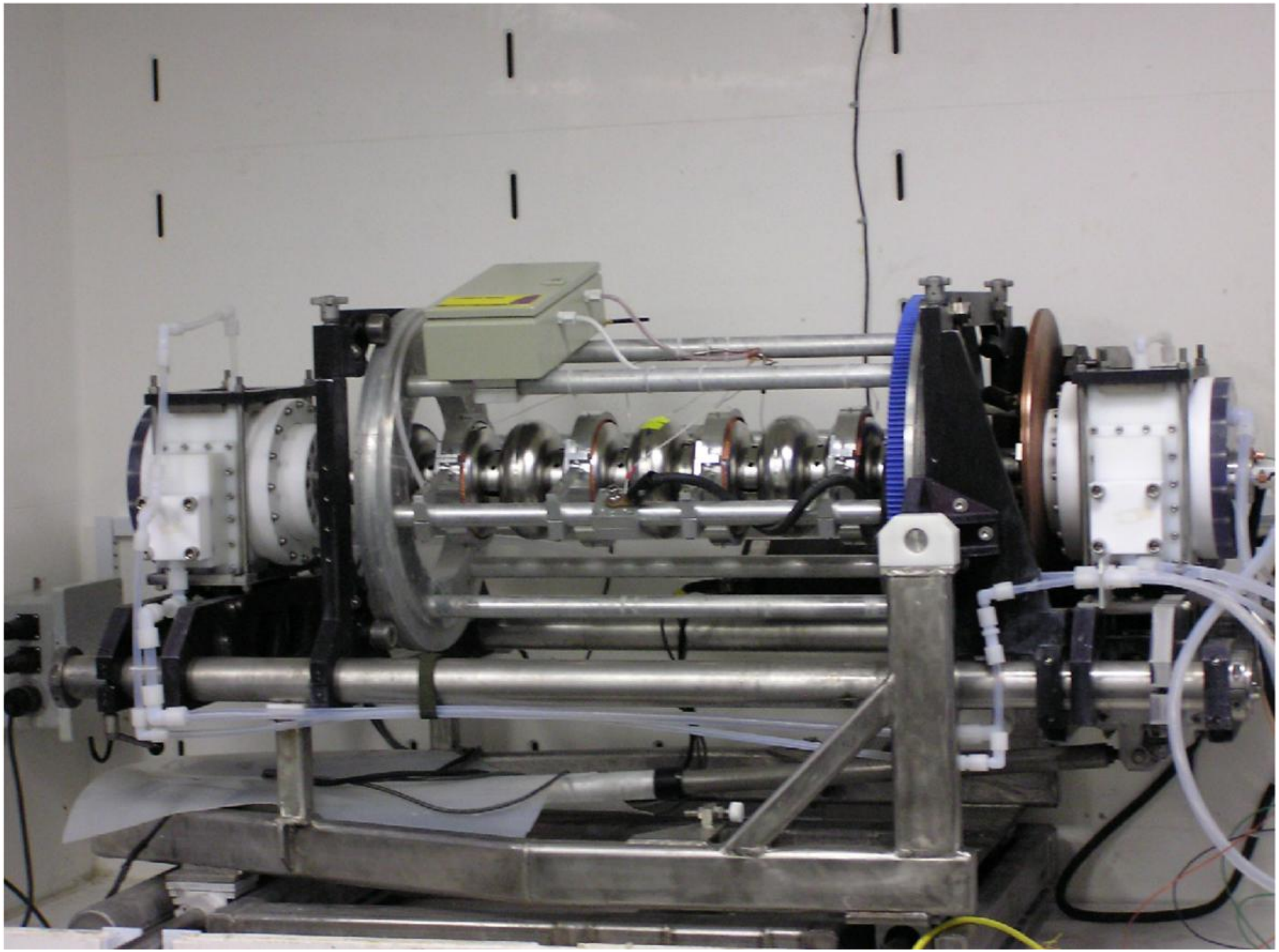
DESY EP control system



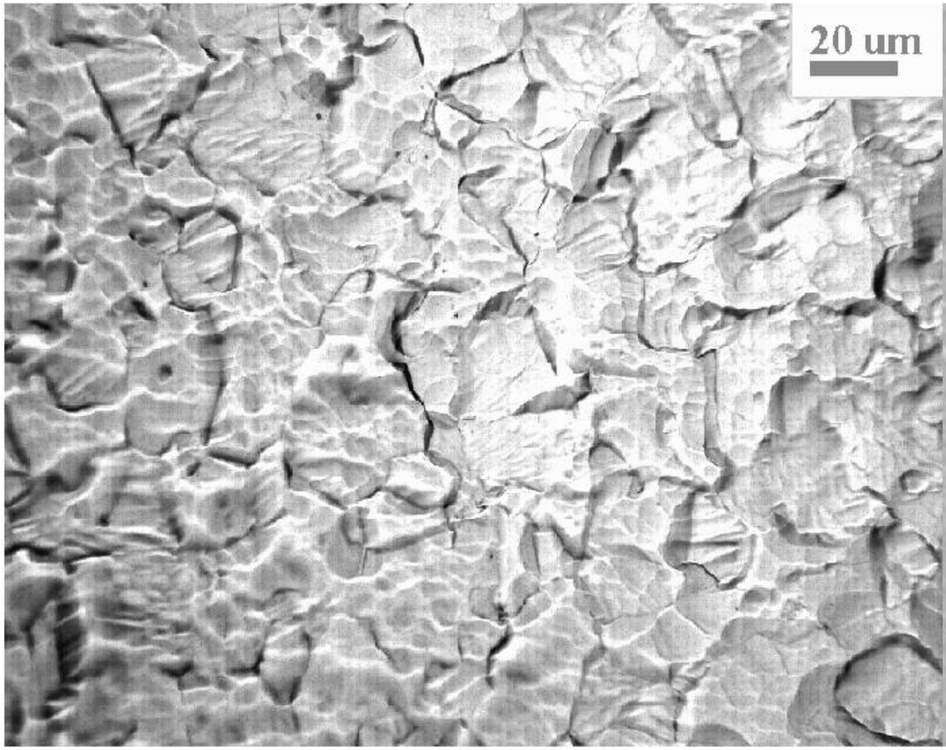
...	Nummer	Datum	Uhrzeit	Dauer	Art	Meldungstext
1	10	24.01.07	08:27:43	0 00:00:00	Alarm	Türen geöffnet (TS0)

Paolo Michelato, SRF2013 tutorials, September 2013

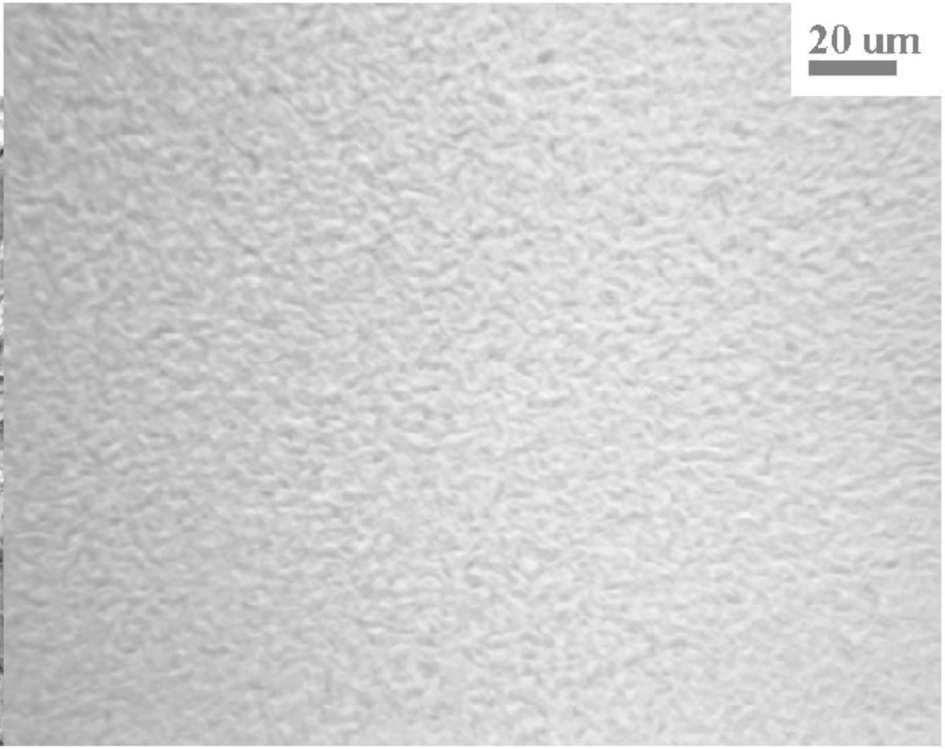
EP system: JLAB



EP vs. BCP niobium surface



Niobium surface after BCP

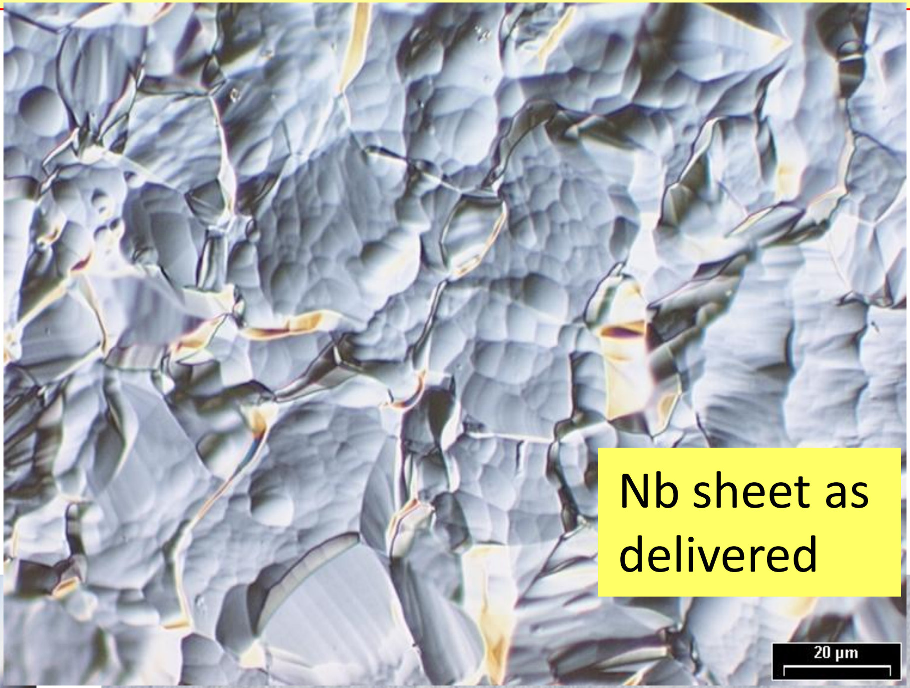


Niobium surface after EP

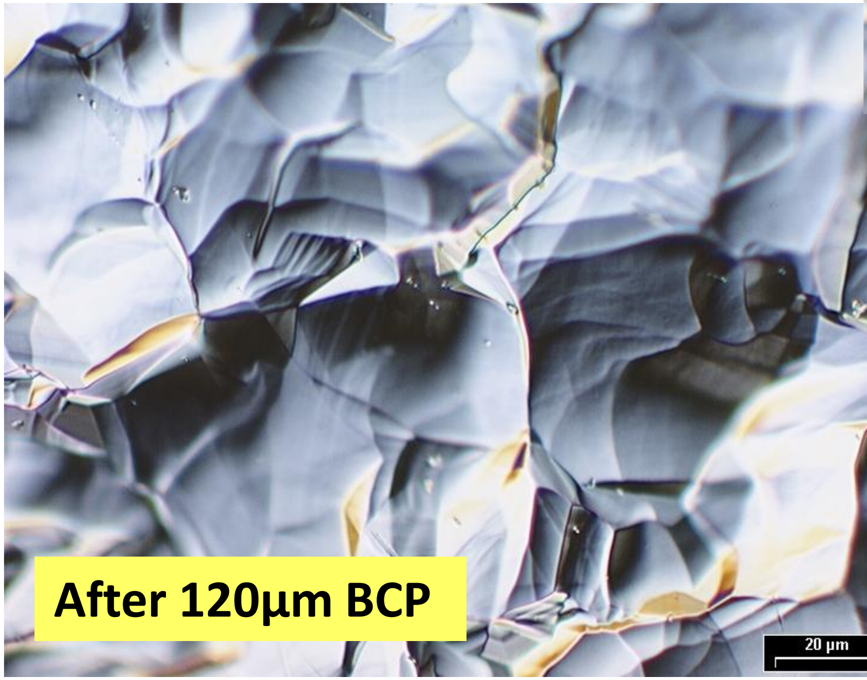
j_mammosser_srf_cavity_preparation_and_limitations_2009-2

EP vs BCP final surface roughthness

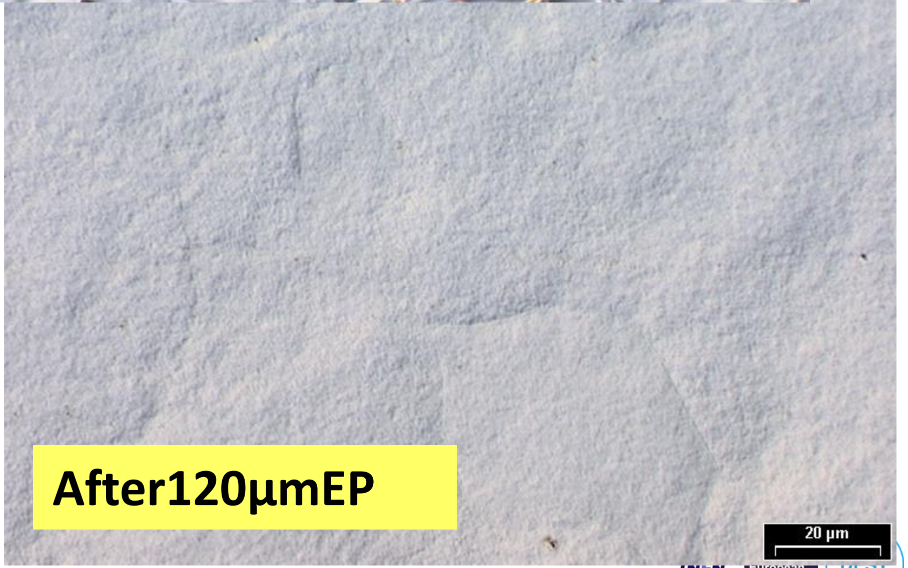
The main difference between BCP and EP:-smoothening of grain boundaries.



Nb sheet as delivered



After 120µm BCP



After120µmEP

Comment on surface processing (BCP vs. EP)

BCP

- **Pros**
  - Easy to handle, middle stirring is necessary
  - Fast etching rate
  - Very reproducible
- **Cons**
  - It is not “polishing”, it is “etching” : all crystalline defects are preferentially attacked (etching pits, etching figures)
  - Grains with various orientation are not etched at the same rate => roughness !
  - Except a few cases, $E_{acc}^{max} \sim$ 25-30 MV/m for a full BCP treated cavity.

(for EXFEL results up to now are encouraging, > 30 MV/m with flash BCP, 10 mm after bulk EP)

- **Caution :**
  - Don’t process at T higher than 25° C
  - Risk of runaway

Comment on surface processing (BCP vs EP) II

EP (Electropolishing)

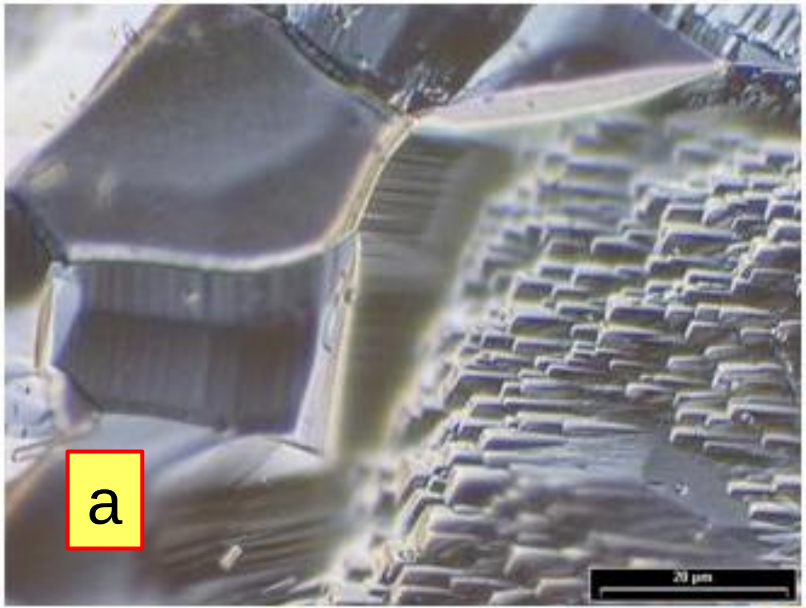
- **Pros** (when idealistic conditions, i.e. viscous layer present)
  - **It is really "polishing"**, no sensitive to crystallographic defects. => smooth surface
  - **Should not be sensitive to the cathode-anode** distance => same etching rate everywhere **(but on EXFEL cavities factor about 2 iris / equator)**
  - Gives **(but not always!)** the best ever E_{acc}^{max}~ 45 MV/m (TESLA shape => ~180mT)
- **Cons**
  - Idealistic conditions are not possible to reach in most of our processing conditions
  - **Very sensitive to stirring condition, temperature, aging of the mixture**
  - **Not very reproducible**
  - Safety issues (**acid mixture sensitive to water**, H_2 evolution…)
- **Caution :**
  - If T ↑ : etching rate ↑ but pitting risk ↑, H loading ↑, HF evolution ↑
  - If V ↑ : etching rate ↑ but pitting risk ↑, S generation ↑, sensitivity to Cathode/Anode distance ↑

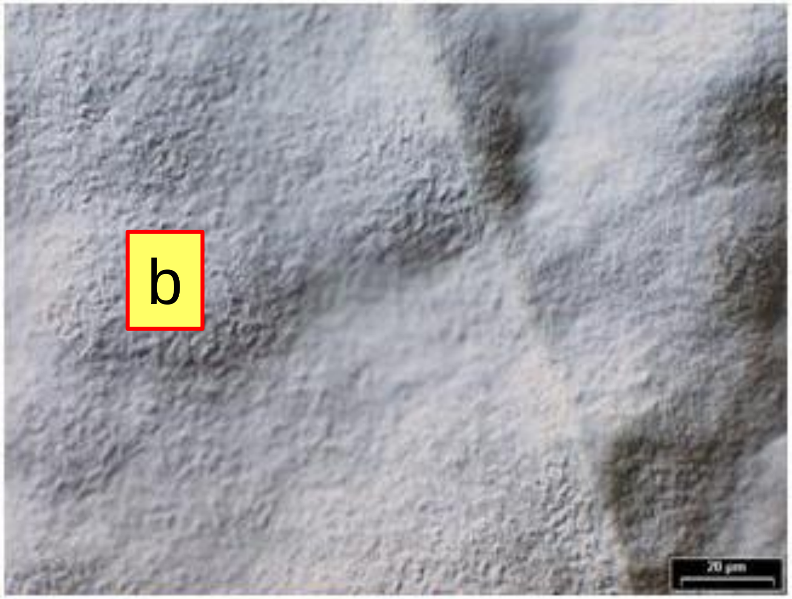
http://ilc-dms.fnal.gov/Members/tajima/References/Antoine_EP_tutorial_01JUN2006.ppt/file_view

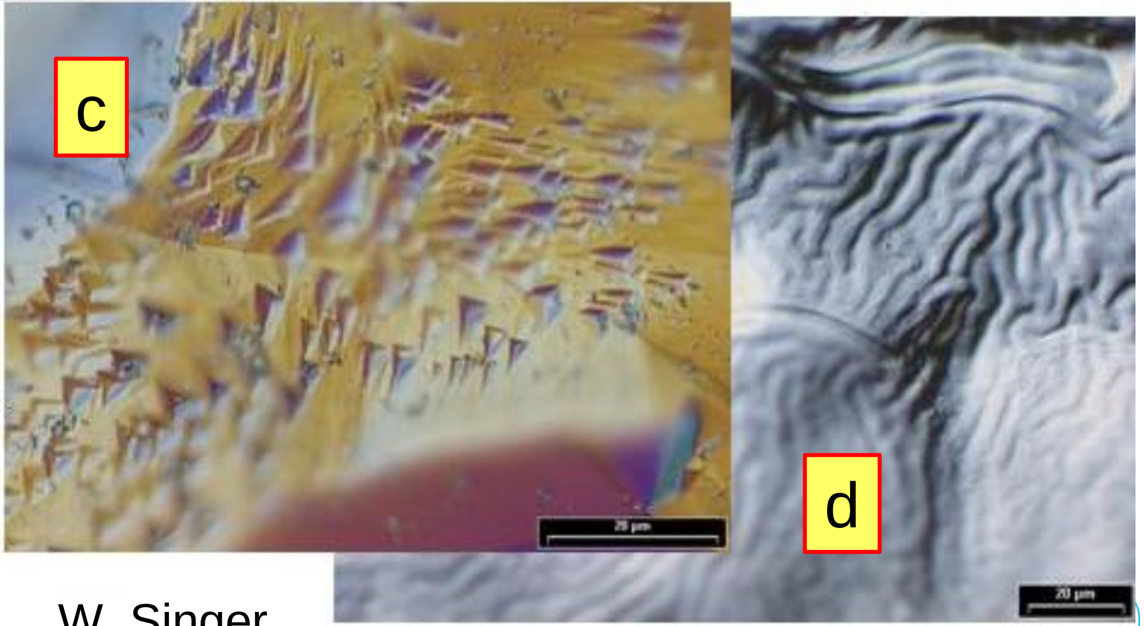
(BCP + EP) comments

- **no cleaning**, but surface removal
- Typically: “main” treatment: 100 – 140 µm
  final removal 10 - 40 µm
- **no (or really weak) removal of e.g. grease, plastics**
- closed system with integrated DI-/pure water rinsing
- acid quality: “pro analysi” or better
- Systems are well established, but environmental and safety critical
- EP requires ethanol rinse (or special detergent) in order to remove sulphur contamination

EP: surface aspect







a) etching area
b) oscillation area
c) gas evolution area
d) dirty electrolyte

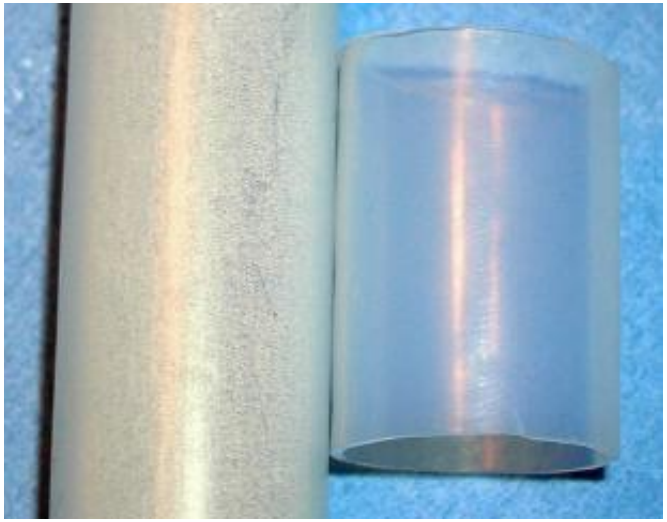
W. Singer

HF safety and use

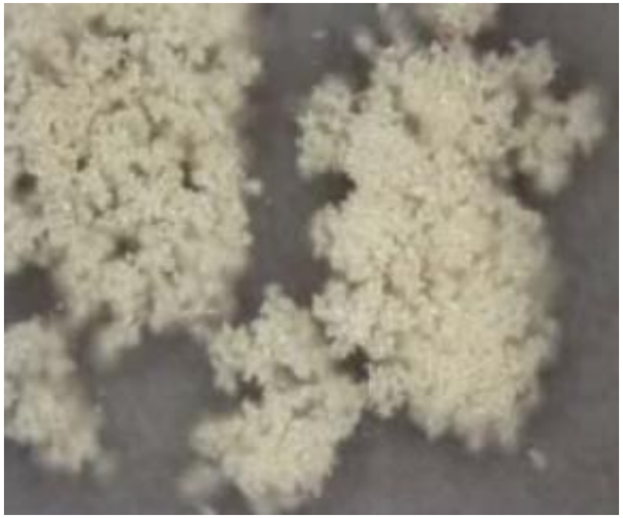
Info on the appendix 1

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

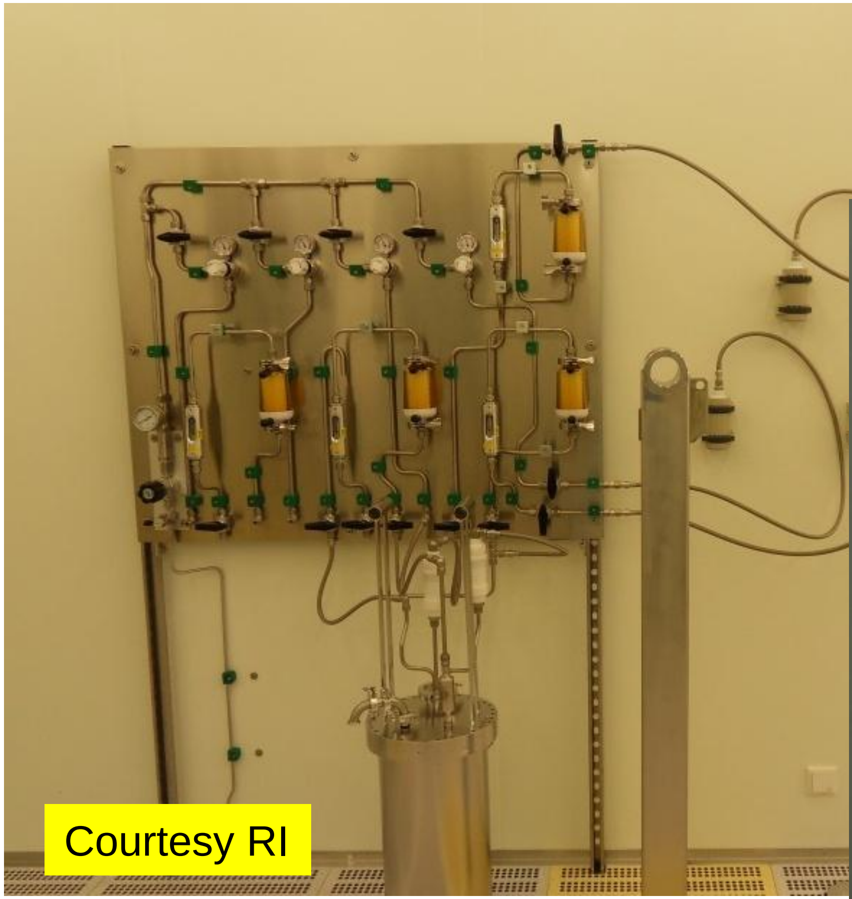


PVDF tube before and after ethanol cleaning



Sulphur removed from a PVDF tube

Ethanol rinse for EXFEL @ the industries: RI

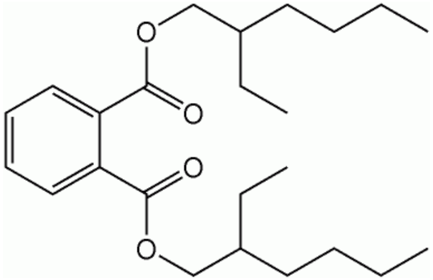


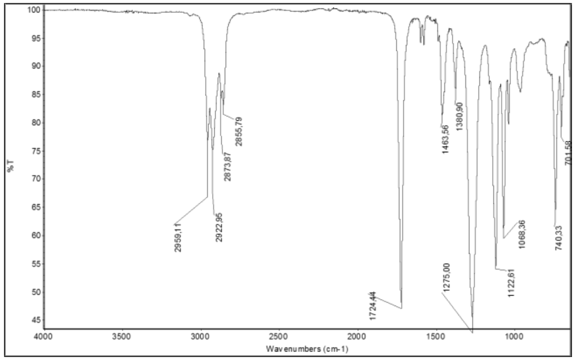
Courtesy RI

Courtesy EZ

Ethanol rinsing: comments

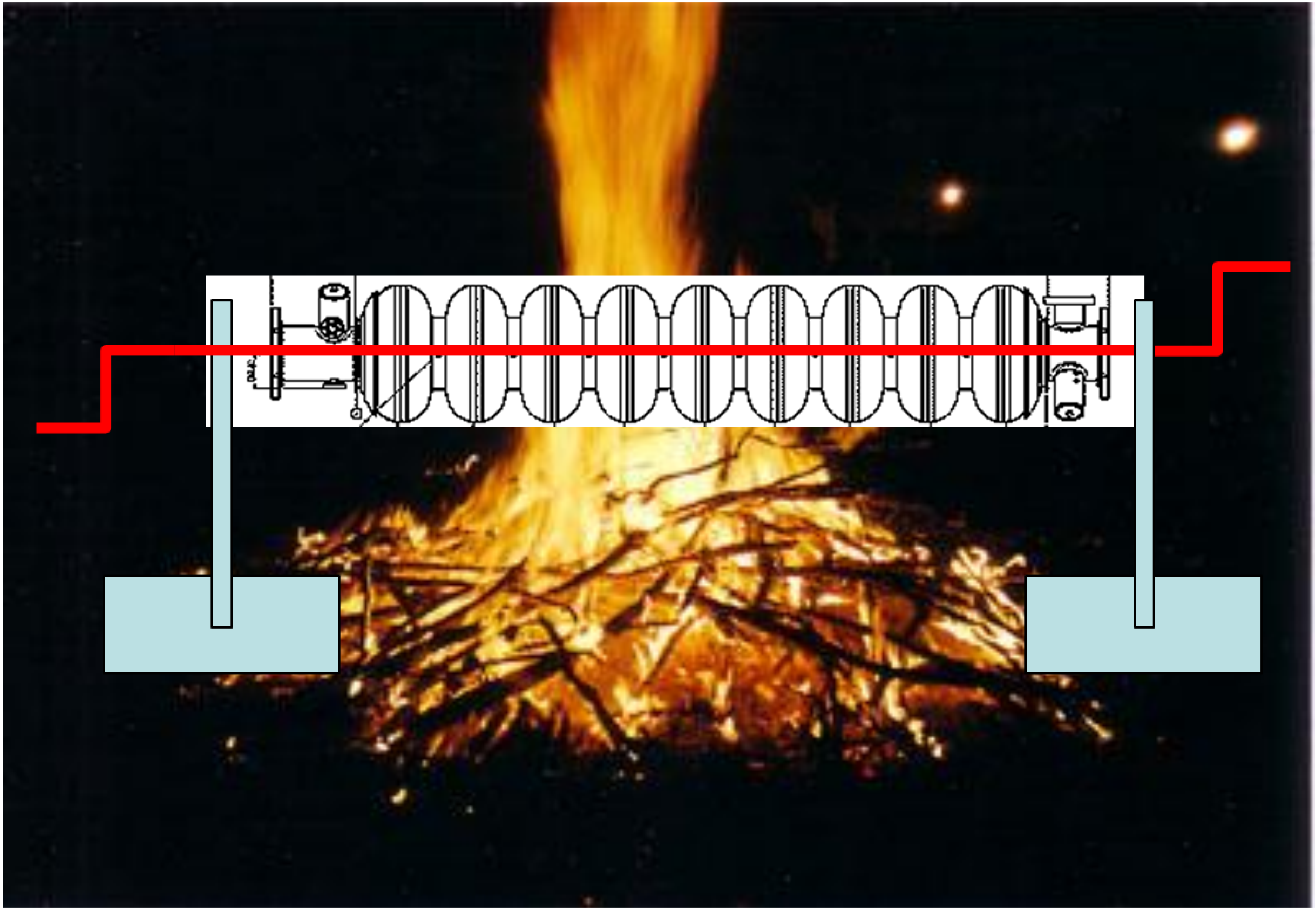
- **Pure ethanol** must be used, and **NOT denaturized** one, or at least ethanol must have **very low not evaporating** residuals (ppm!).
- **One charge** can be used for **many "ethanol rinsing**" operations, it have not to be renovated each time.
- Take care that all the components in the Ethanol circuit, filters, tubes, gaskets, etc, have to be fully compatible for **LONG TIME** exposition to ethanol to avoid any ethanol contamination
- For instance **PVC tubing and PVC parts can loose some plasticizer** during the long staying in contact with Ethanol and contaminate it.
- **DEHP** (Bis(2-ethylhexyl) phthalate) was identified with FTIR **in the ethanol rinsing system that used PVC for tubing**.





FTIR Spectrum of ethanol residuals

800 deg heat treatment



High temperature (800°C) Nb firing: why is needed

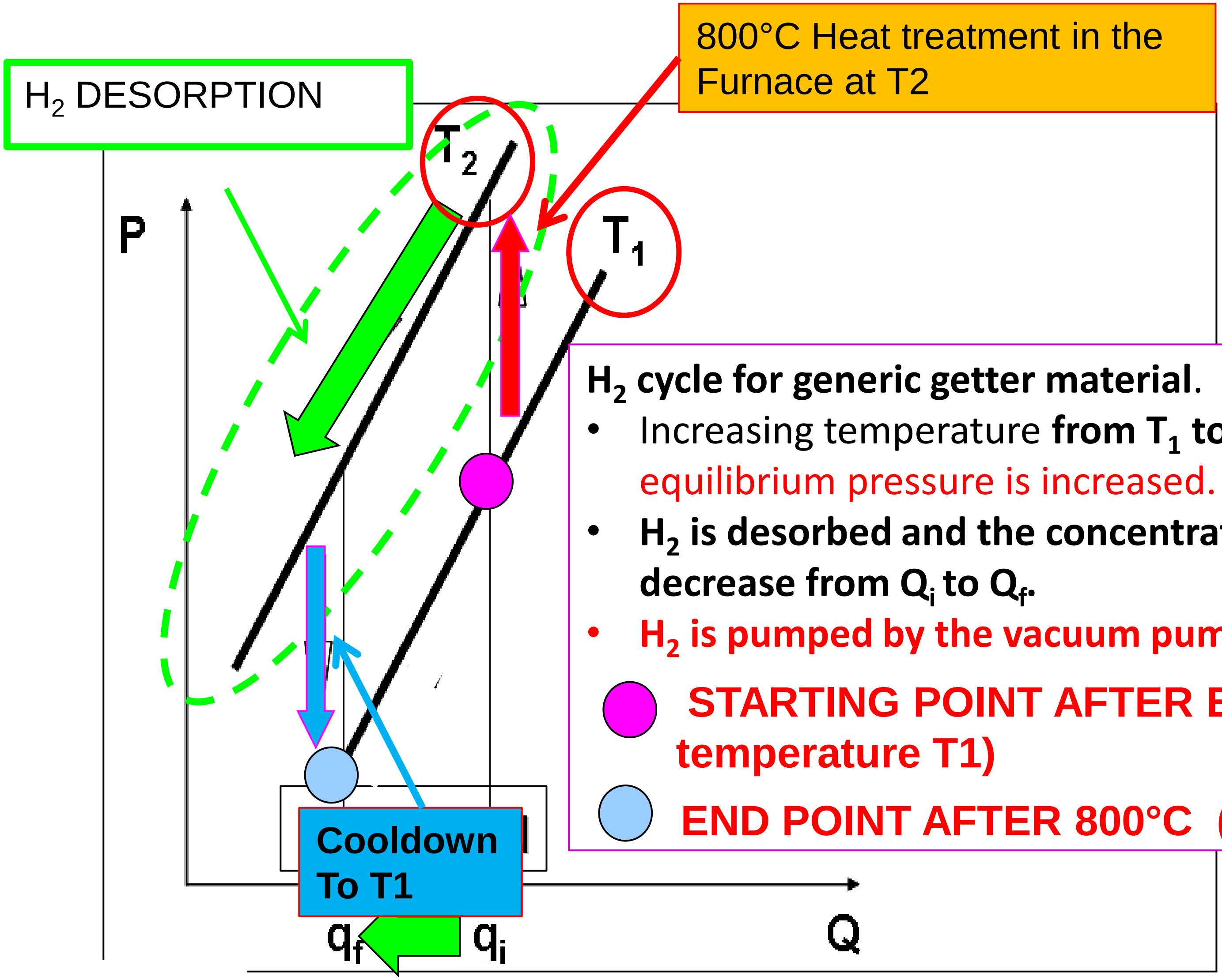
- Hydrogen is diffused in the bulk during the various etching treatments. See **R. E. Ricker and G. R. Myneni,** J. Res. Natl. Inst. Stand. Technol. **115**, 353-371 (2010), Evaluation of the Propensity of Niobium to Absorb Hydrogen During Fabrication of Superconducting Radio Frequency Cavities for Particle 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 temperatur**e, maintaining a **low H_2 partial pressure**, **H_2 is desorbed from the bulk (Nb)**

High temperature Nb firing



H_2 DESORPTION

800°C Heat treatment in the Furnace at T2

Cooldown To T1

H_2 cycle for generic getter material.

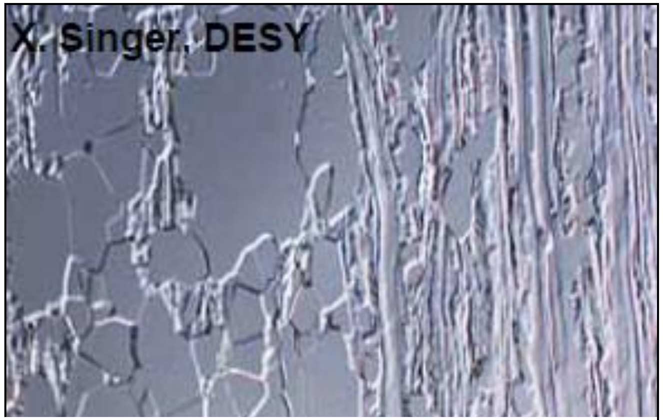
- Increasing temperature **from T_1 to T_2**, the H_2 equilibrium pressure is increased.
- **H_2 is desorbed and the concentration in the bulk decrease from Q_i to Q_f.**
- **H_2 is pumped by the vacuum pumps**

● **STARTING POINT AFTER EP (at room temperature T1)**

● **END POINT AFTER 800°C (at T1)**

High temperature Nb firing

- **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 more soft and this facilitate the cavity tuning process**



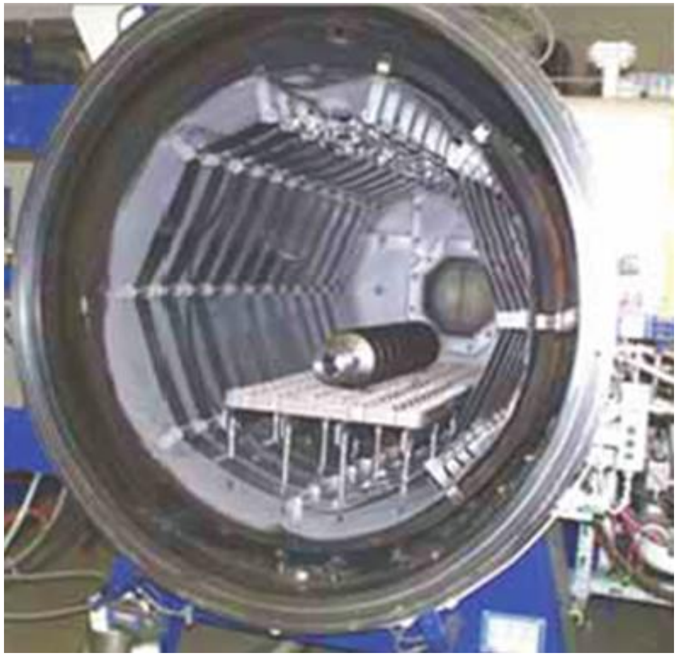
Not completely recrystallized Nb

Completely recrystallized Nb

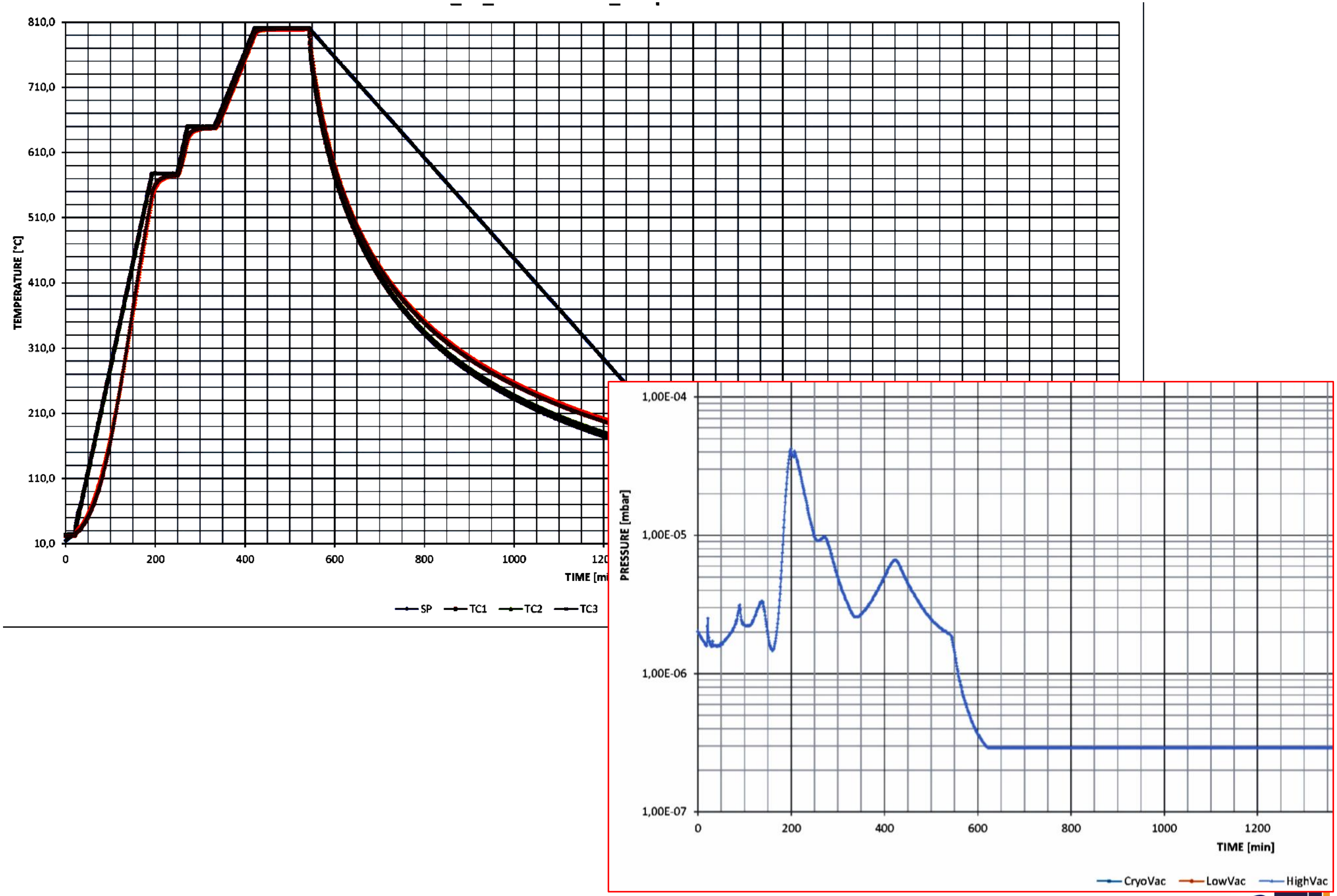
Firing process and furnaces

Furnace:

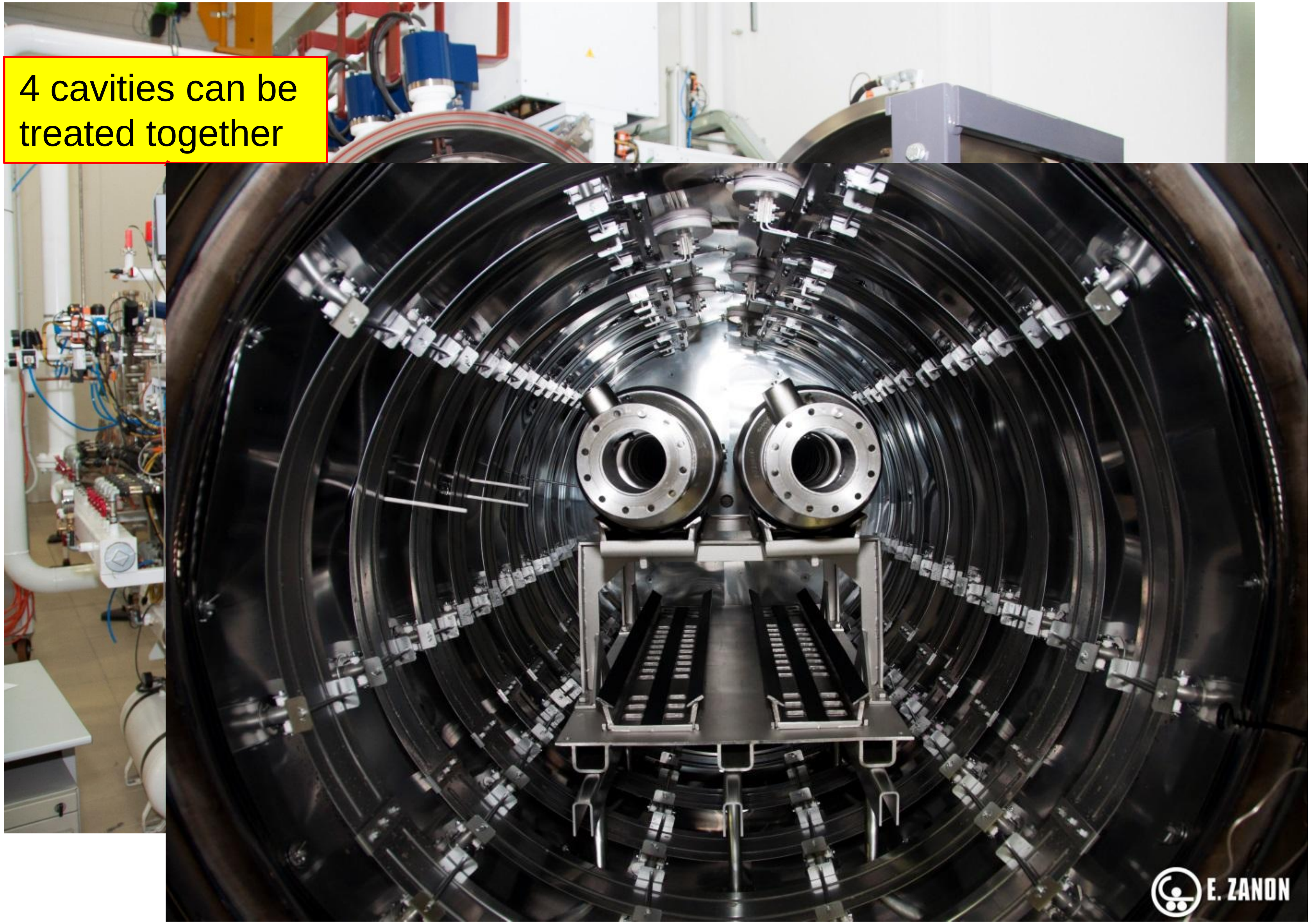
- Operative temperature for treatment: 800 °C
- Cleaning cycle temp.: 1100 °C
- Metal chamber water cooled, Mo heaters
- Oil free vacuum system (dry screw pump + roots + cryopump)
- Pressure at 800°C: < $1\text{x}10^{-5}$ mbar
- Residual gas analyzer for cleanliness check and H_2 partial pressure measurement



Typical XFEL 800°C thermal cycle and pressures



Furnace at the industry: EZ furnace

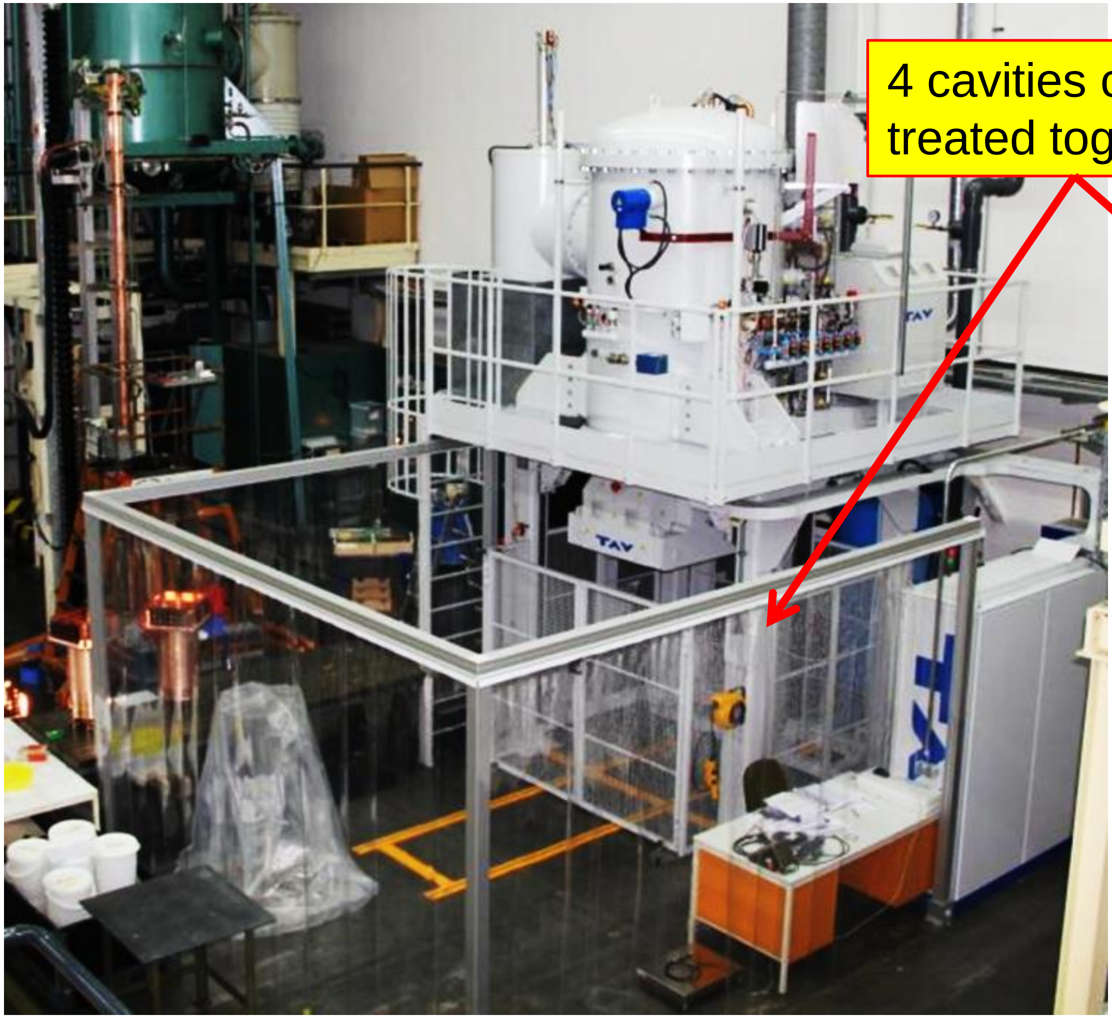


4 cavities can be treated together

800 deg heat treatment furnace at RI

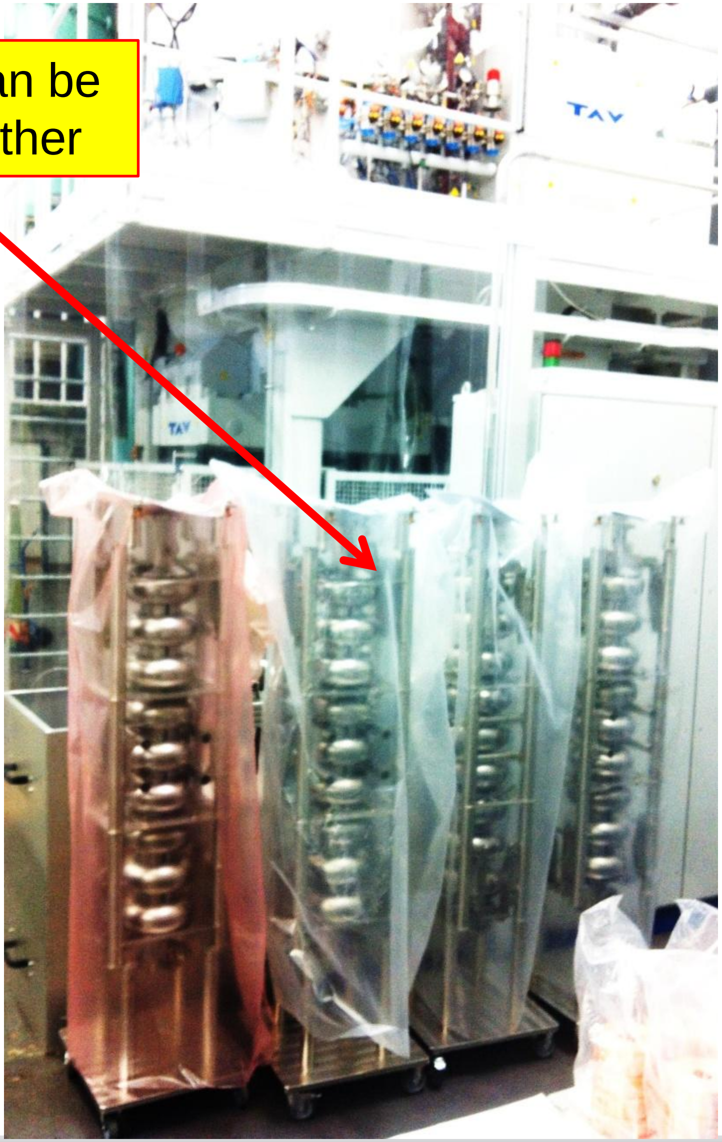
research
instruments

RI infrastructure for XFEL



800 C annealing furnace for hydrogen degassing, hydrogen enters the niobium during the electropolishing process

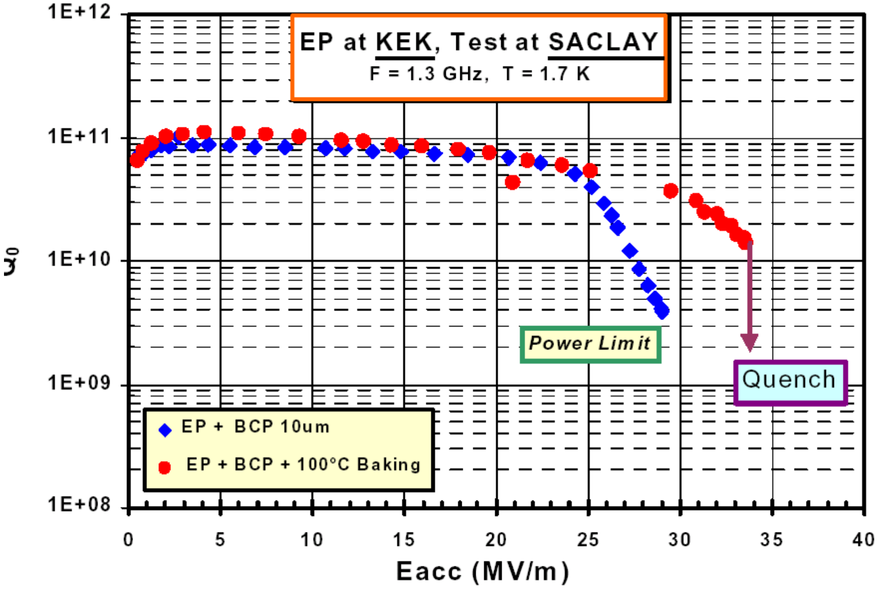
4 cavities can be treated together



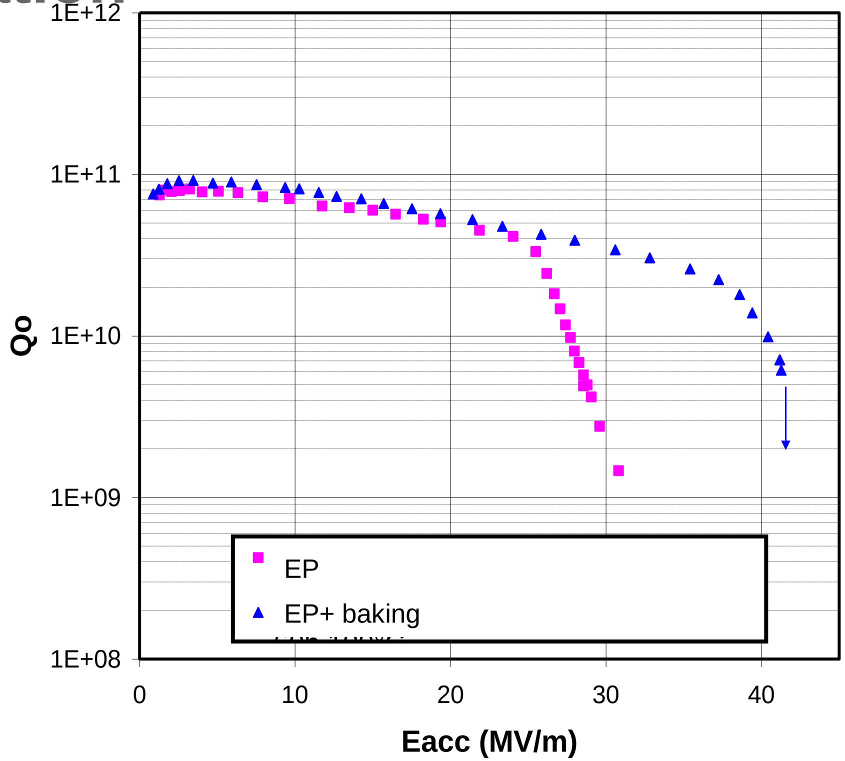
120°C heat treatment @ EZ

WHY BAKING ?

- **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**



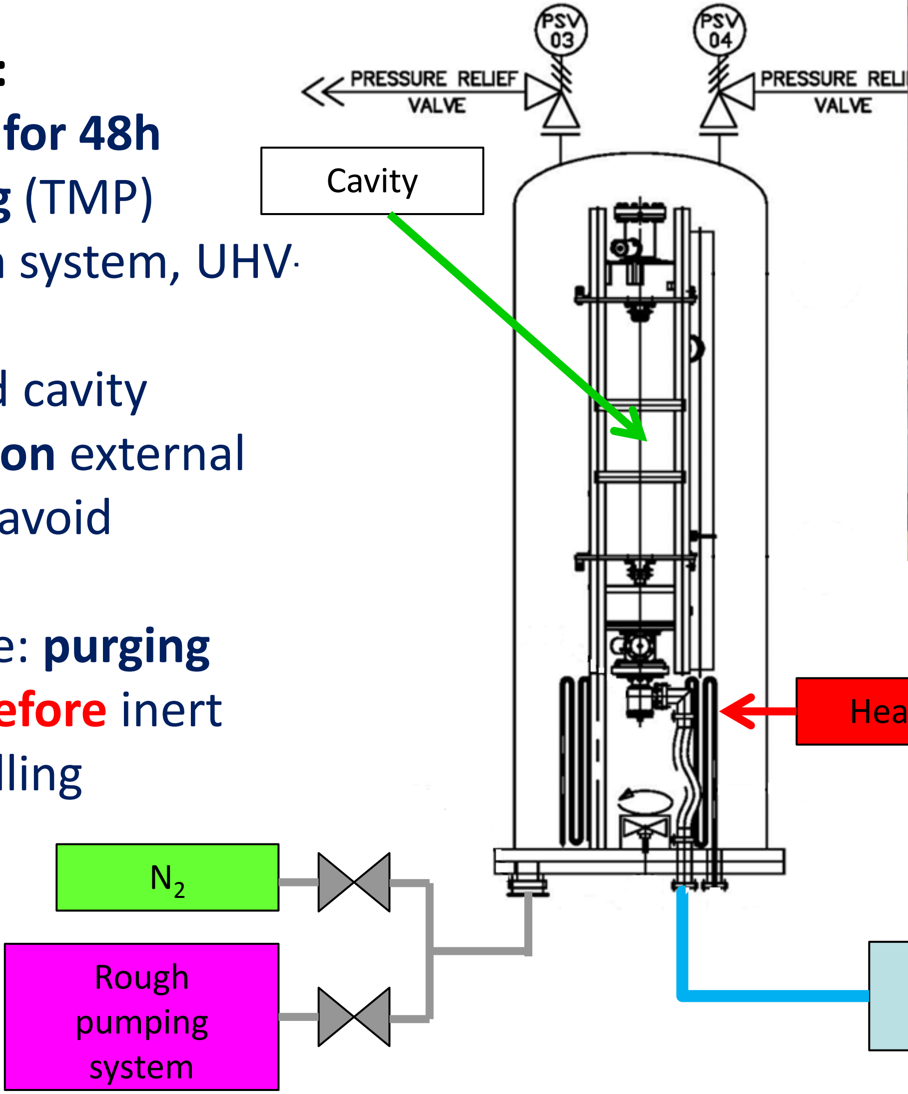
Collaboration KEK / CEA Saclay, August 1999



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



120°C backing @ EZ and RI

Courtesy RI

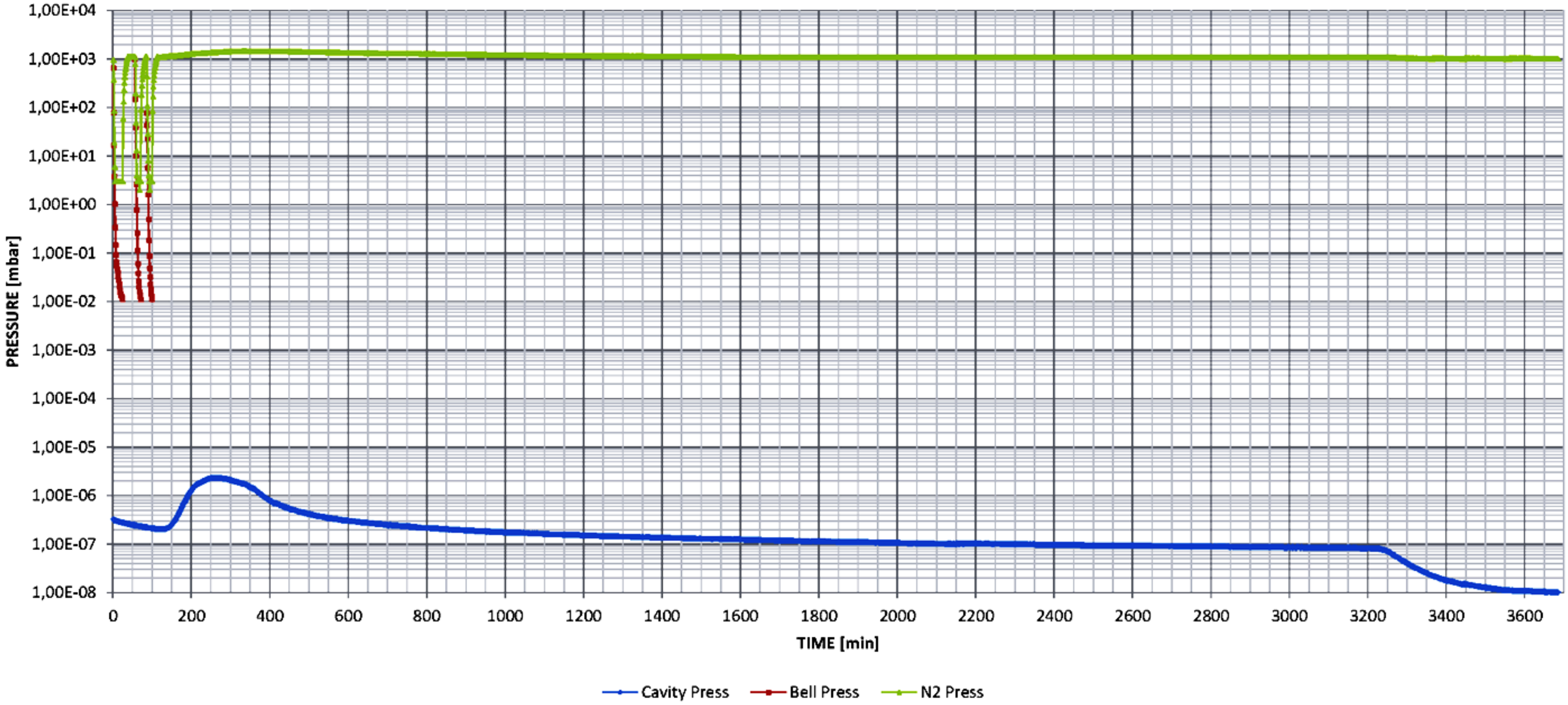
Courtesy EZ

Courtesy RI

Courtesy EZ

Similar architecture, one UHV pump for more than one cavity (2 or 3).

120 °C baking: pressure in the jar and in the cavity



Vacuum

See also appendix 2

SPSV: slow pumping slow venting

- Motivation of automated venting + pumping
  - **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 $\Delta p > 1$ 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: slow pumping slow venting (cnt)

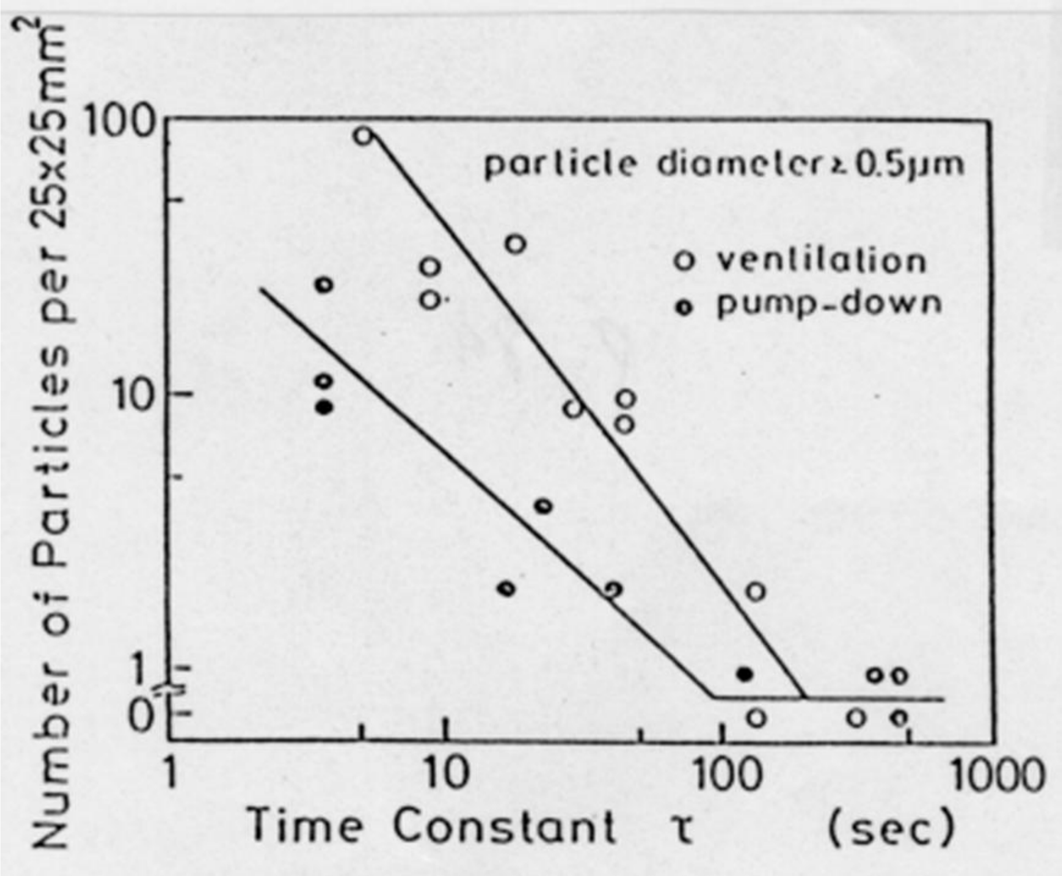
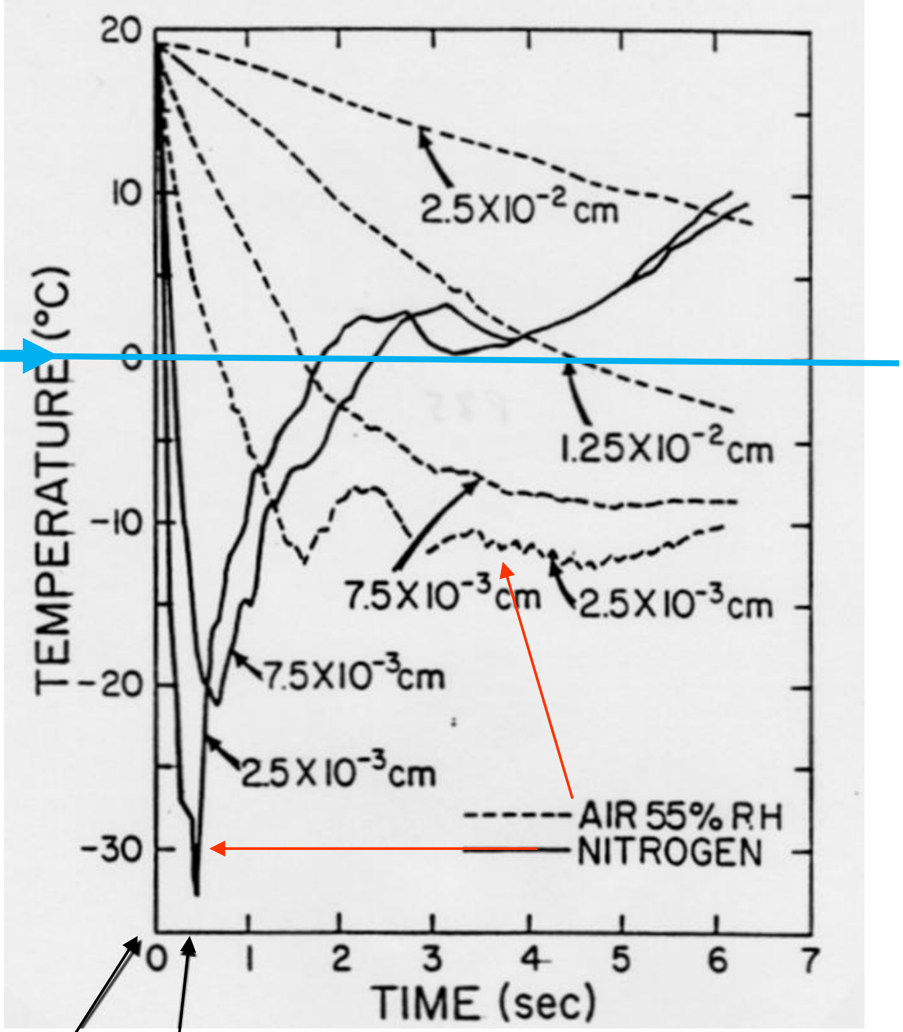


Fig.3 Increment in number of dust particles after each ventilation and pumpdown, is plotted versus the time constant τ.

Both rapid venting and rapid pumping introduce particle motion.

SPSV: slow pumping slow venting (cnt)

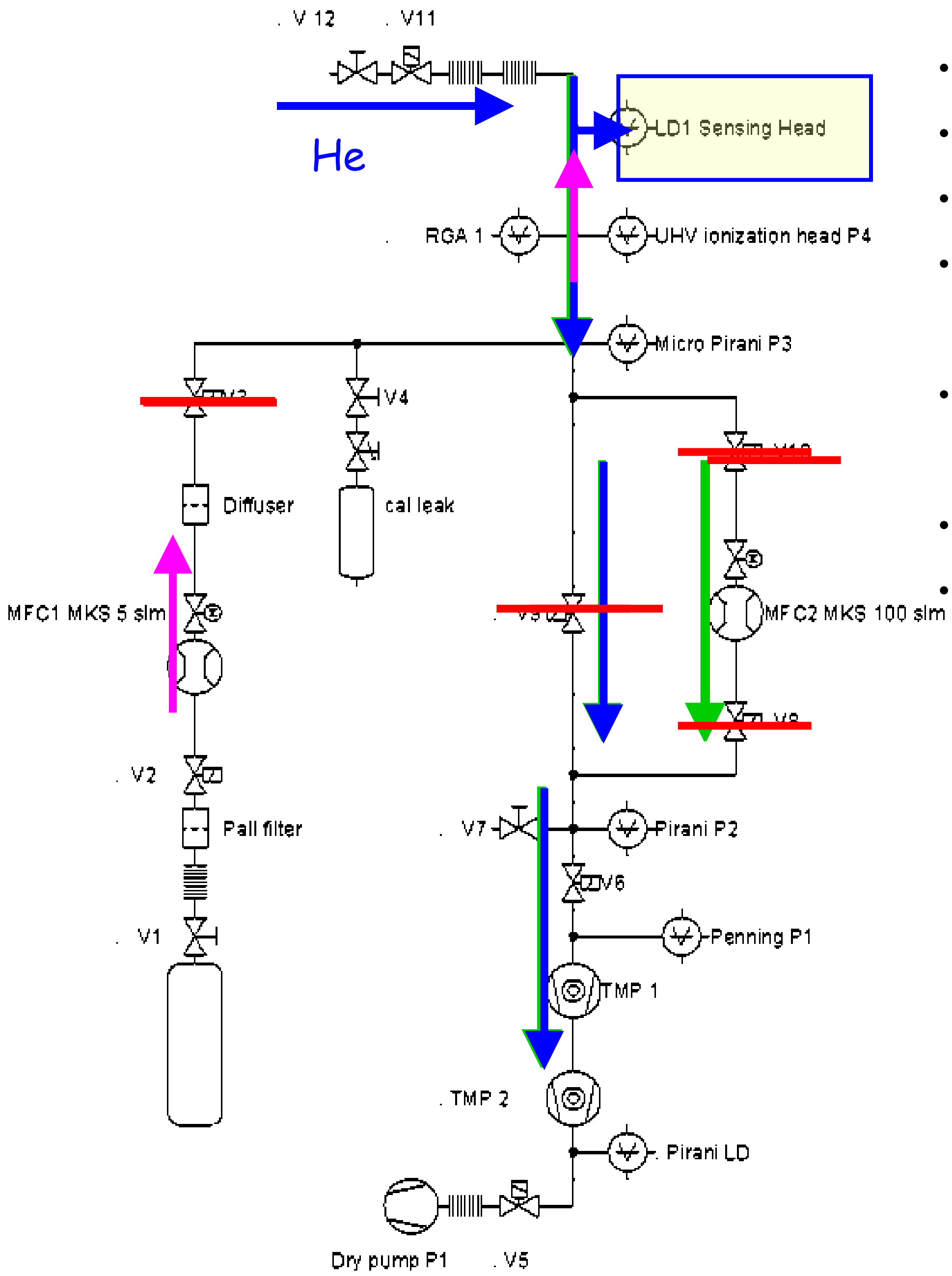
- **In vacuum temperature** measured with micro **thermocouples** (250 μm, 125 μm, 75 μm, 25 μm) during fast pumpdown.
- **Temperature is going down rapidly, well below 0 °C!**
- **Condensation** of water on particulates will arrive.
- **Sticking of particles on surfaces!**
- Movement of particles on the system!



760 Torr

650 Torr

INFN SPSV



- 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

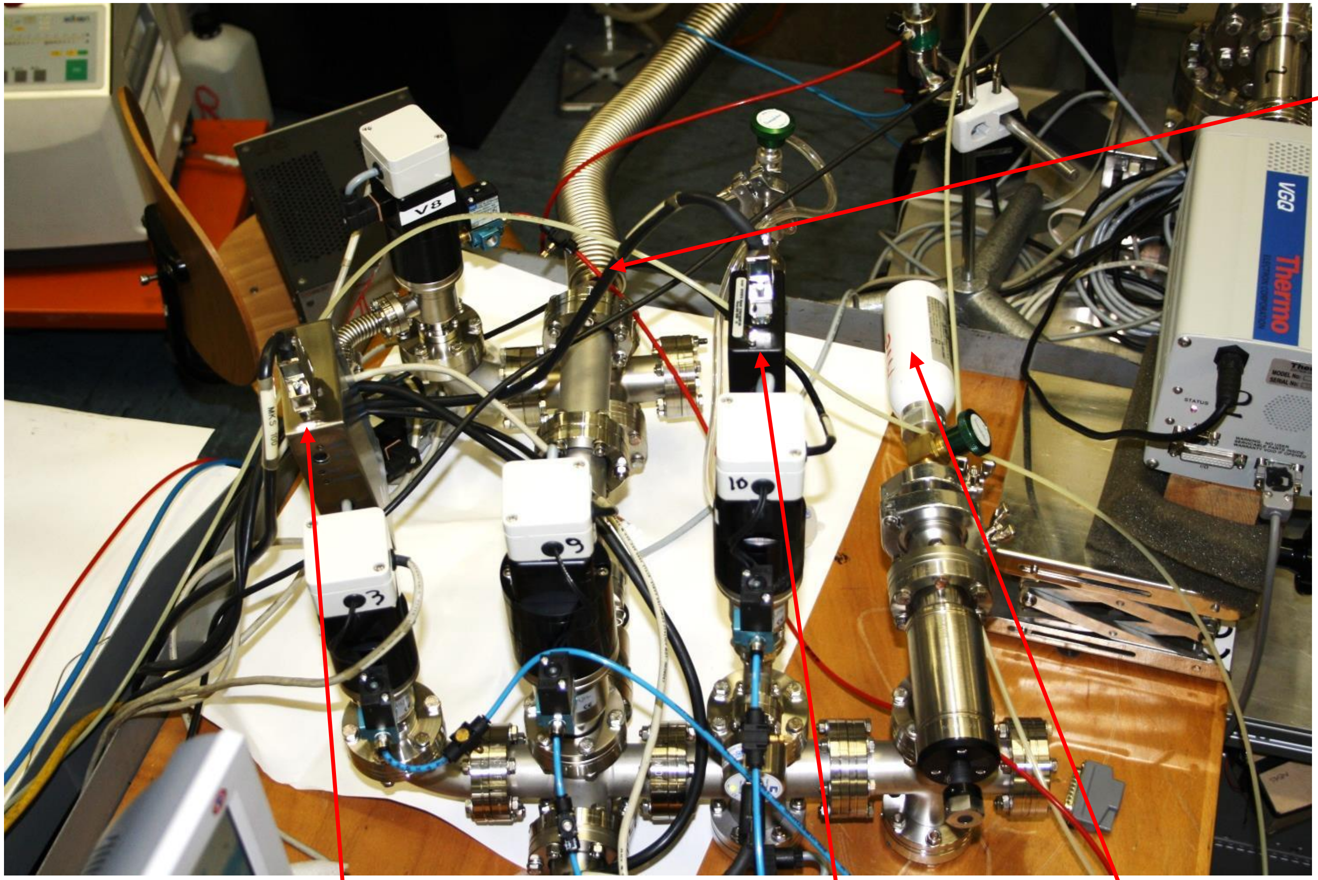
Pump down fm atmospheric pressure

Pumpdown at p< 1 mbar

Leak check

Slow venting

SPSV: INFN system



Connection to the TMP

Pumping line: controller and valves

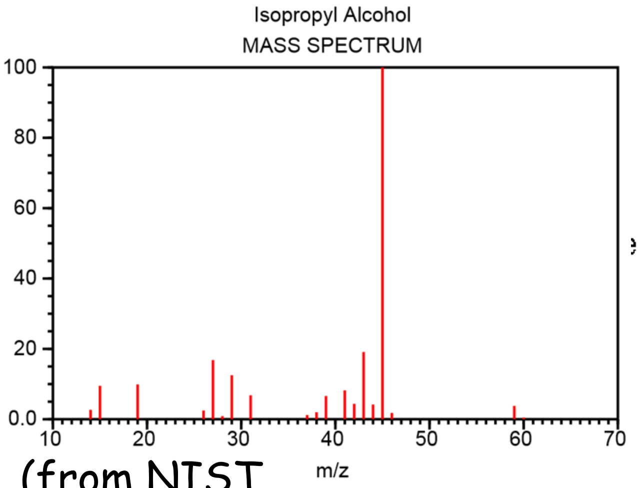
Cal. leak

Venting line: controller and valves

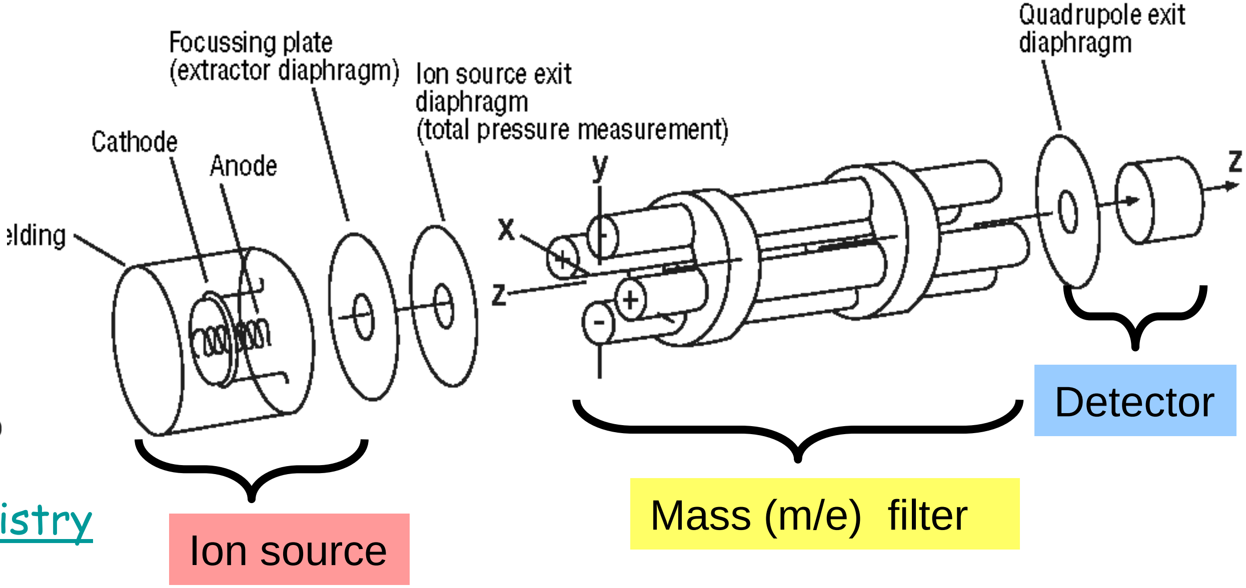
Residual Gas Analysis

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 fingerpring are available for some components (for instance for isopropanol, acetone, ethanol, etc.)
- Evaluation of **Hydrocarbon** content



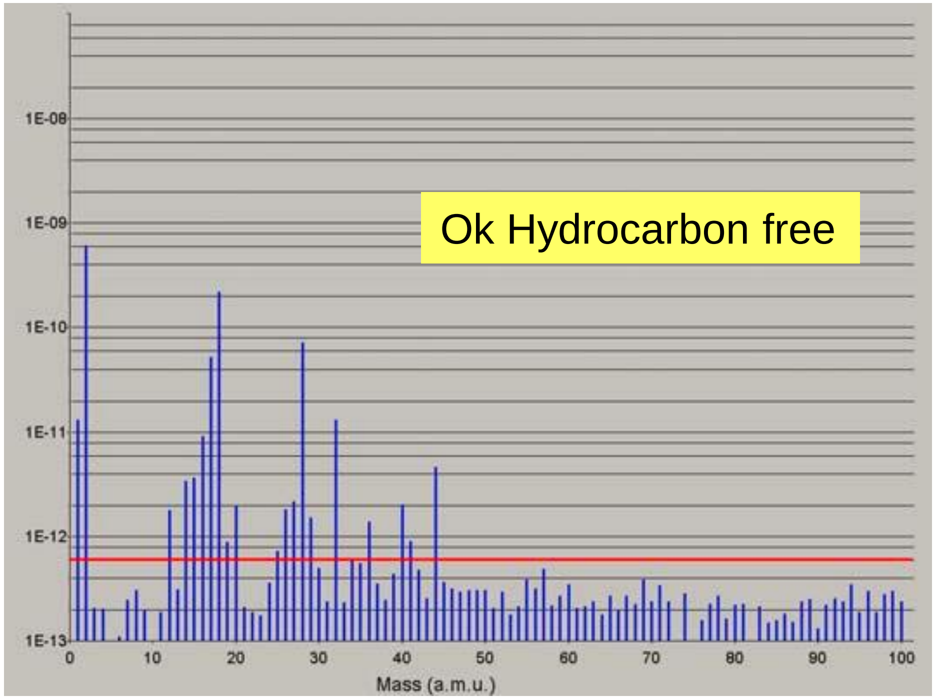
(from NIST, http://webbook.nist.gov/chemistry/name-ser.html).

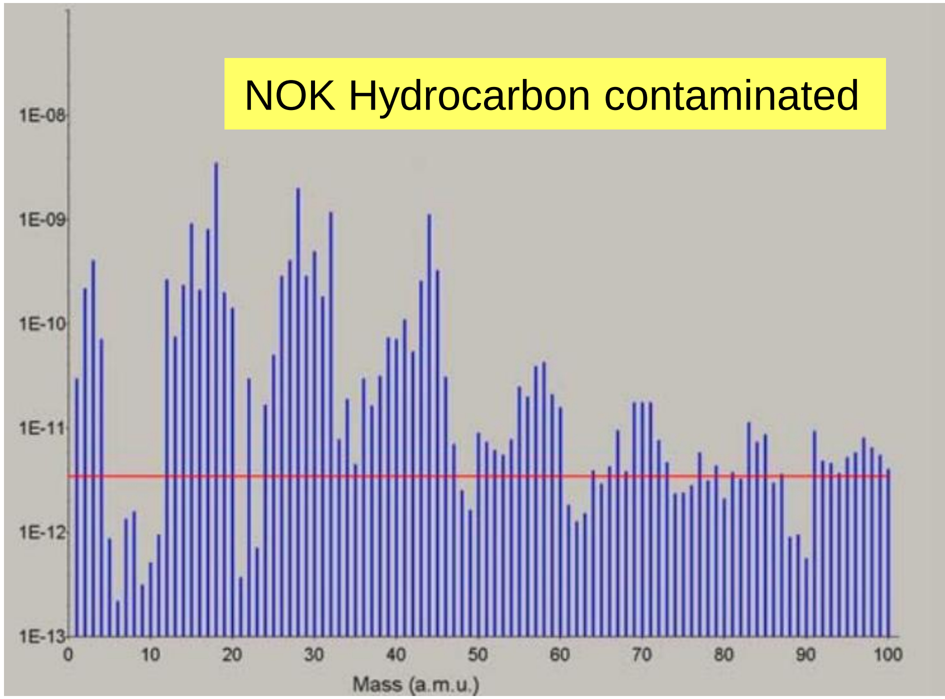


Definition of hydrocarbon free parts for EXFEL

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

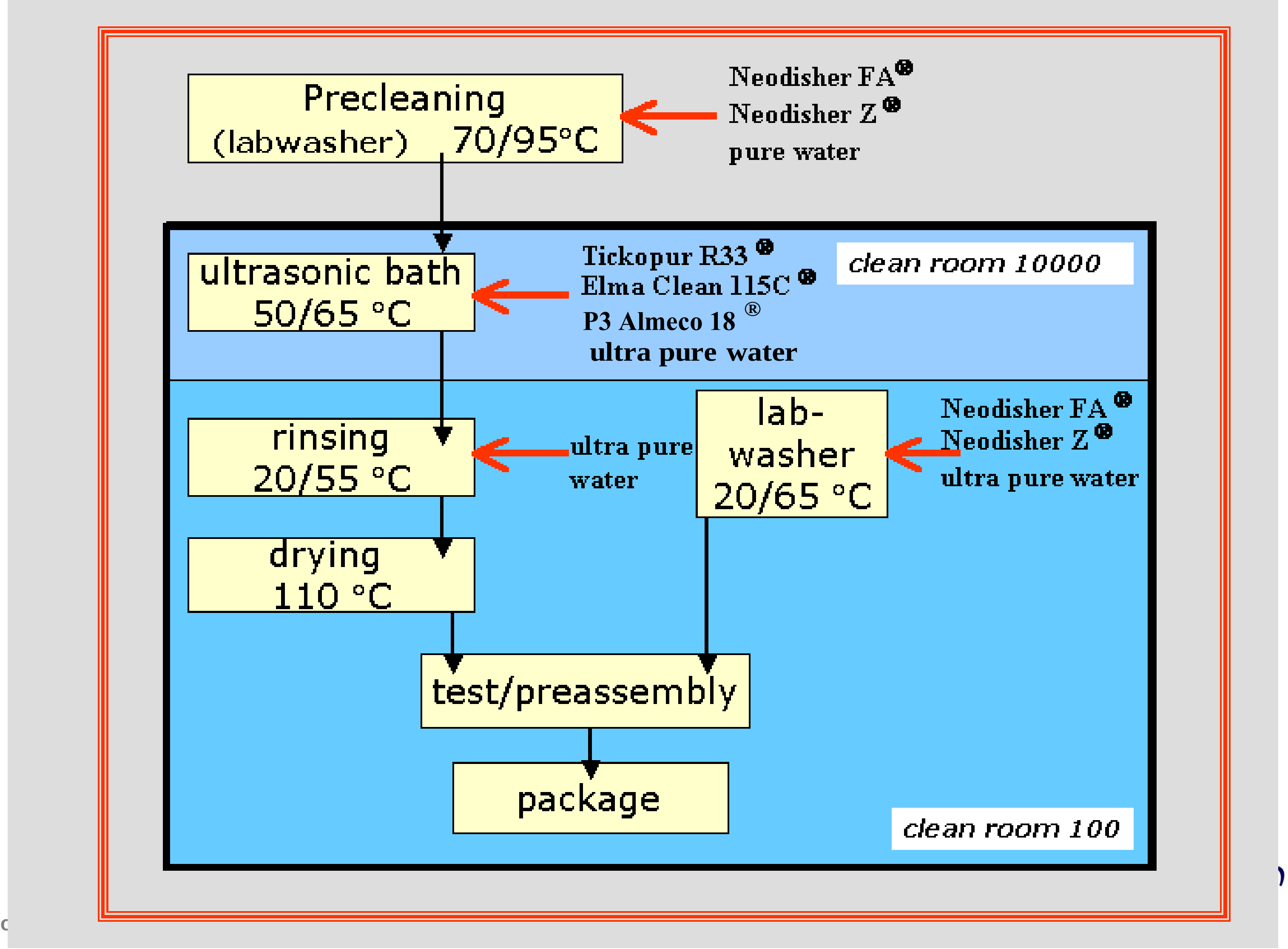
Components are considered free of hydrocarbons if in a leak-free system with a total pressure below 10^{-7} mbar the sum of the partial pressures of masses above mass 45 is less than 10^{-3} of the total pressure (1 : 1000).





Vacuum: Cleaning

- Cleaning of all vacuum components in the cavity environment to the same level as the cavity itself



Precleaning
(labwasher) 70/95°C

Neodisher FA®
Neodisher Z®
pure water

ultrasonic bath
50/65 °C

Tickopur R33®
Elma Clean 115C®
P3 Almeco 18®
ultra pure water

clean room 10000

rinsing
20/55 °C

ultra pure water

lab-washer
20/65 °C

Neodisher FA®
Neodisher Z®
ultra pure water

drying
110 °C

test/preassembly

package

clean room 100

Vacuum: Cleaning

- DESY: Separate cleanroom for cleaning of vacuum components

Cleanroom

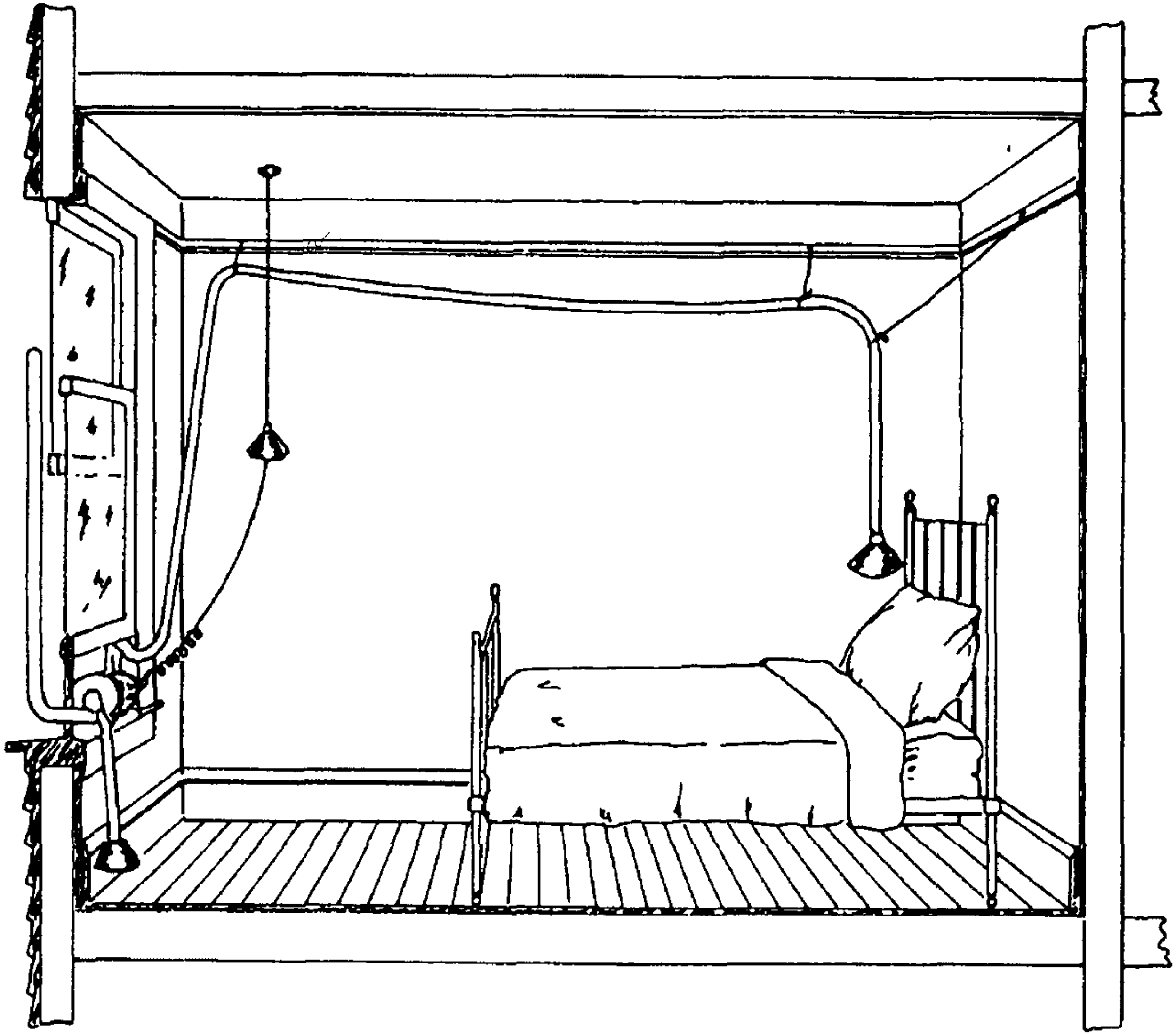
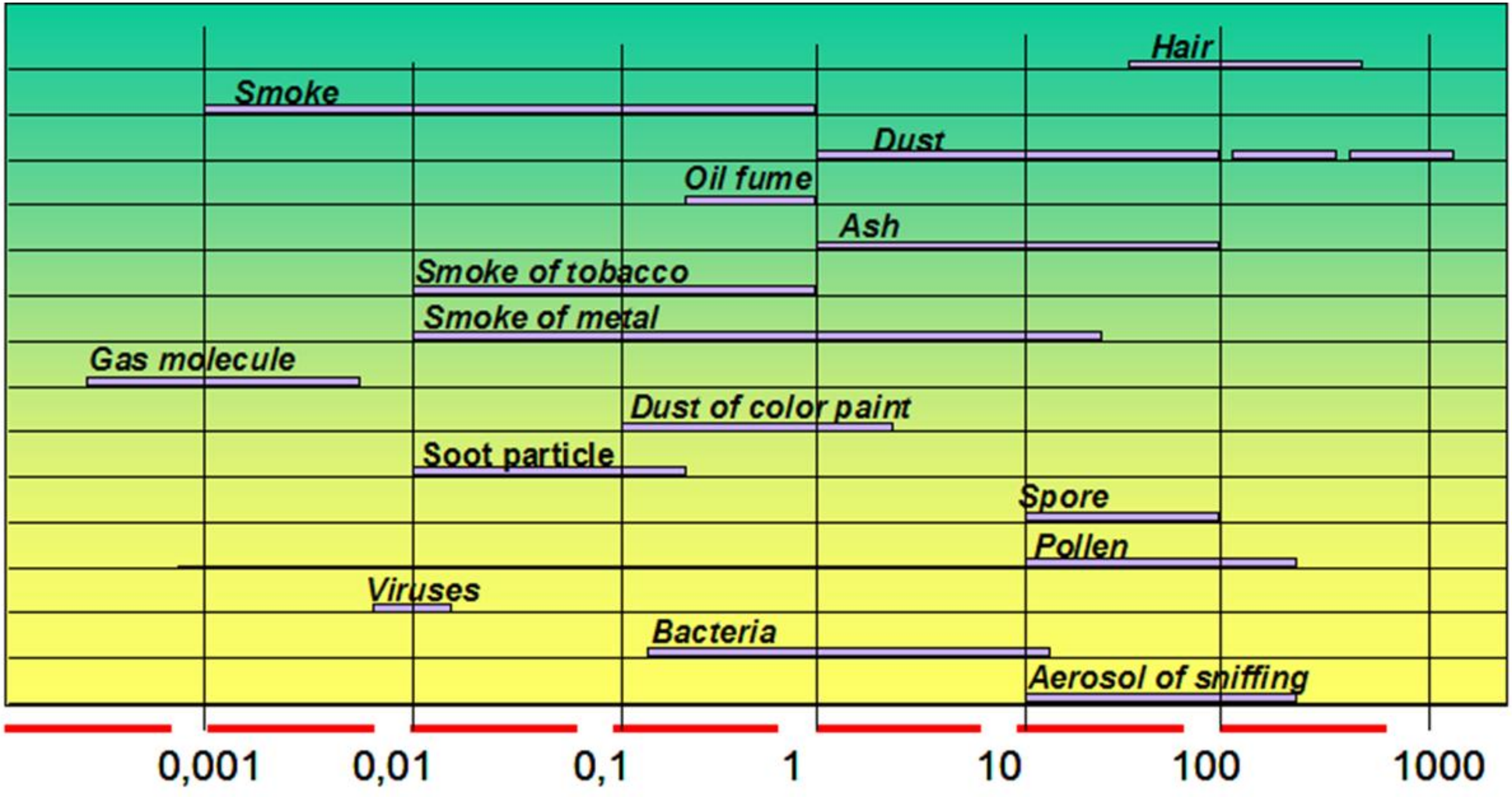


FIGURE 1.1. Ventilation of hospital room in the 1920s. A patient could inhale fresh air from the funnel. Foul air from the floor was extracted by another funnel.

What is a clean room? 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 mm 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.

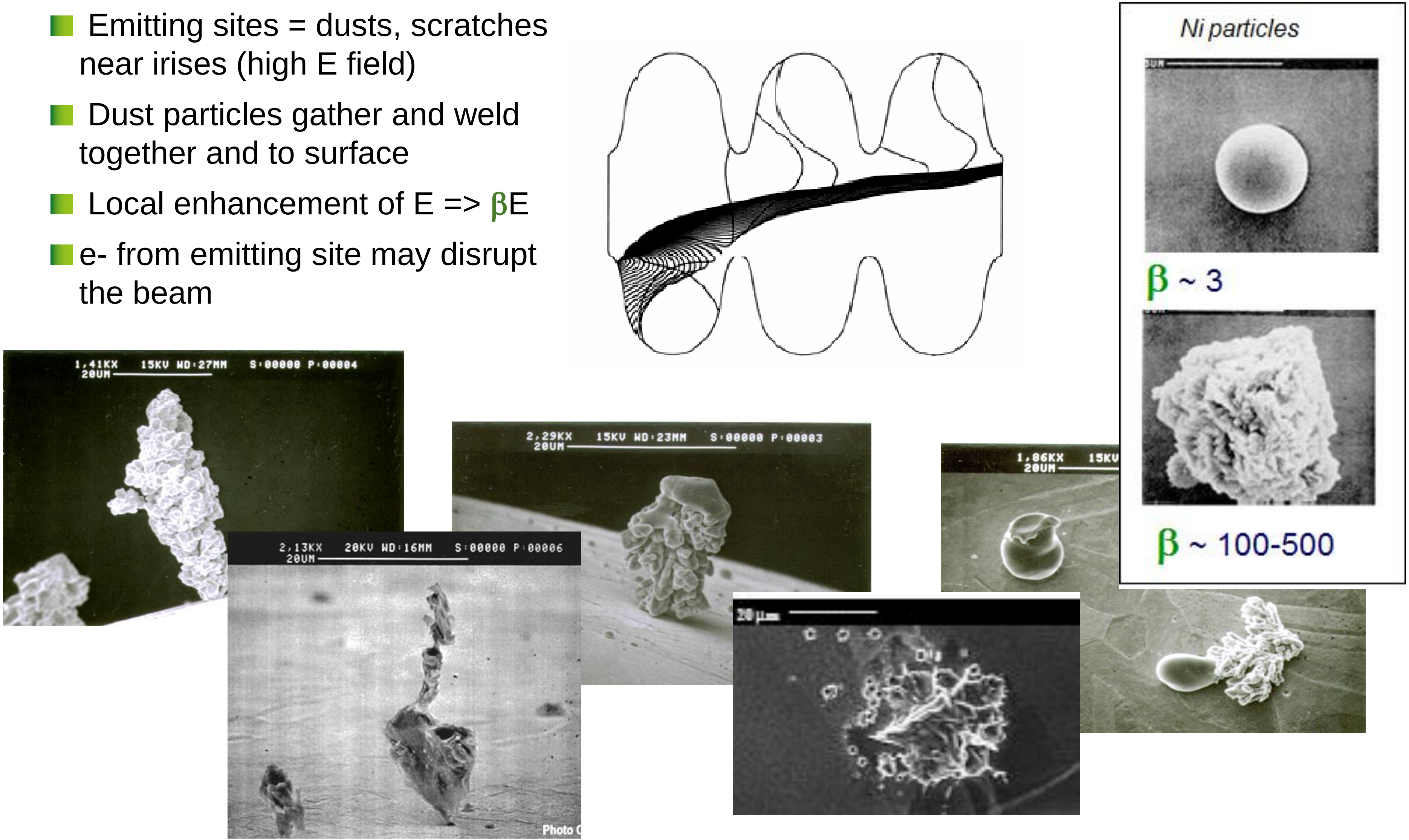
Particles: diameter and species



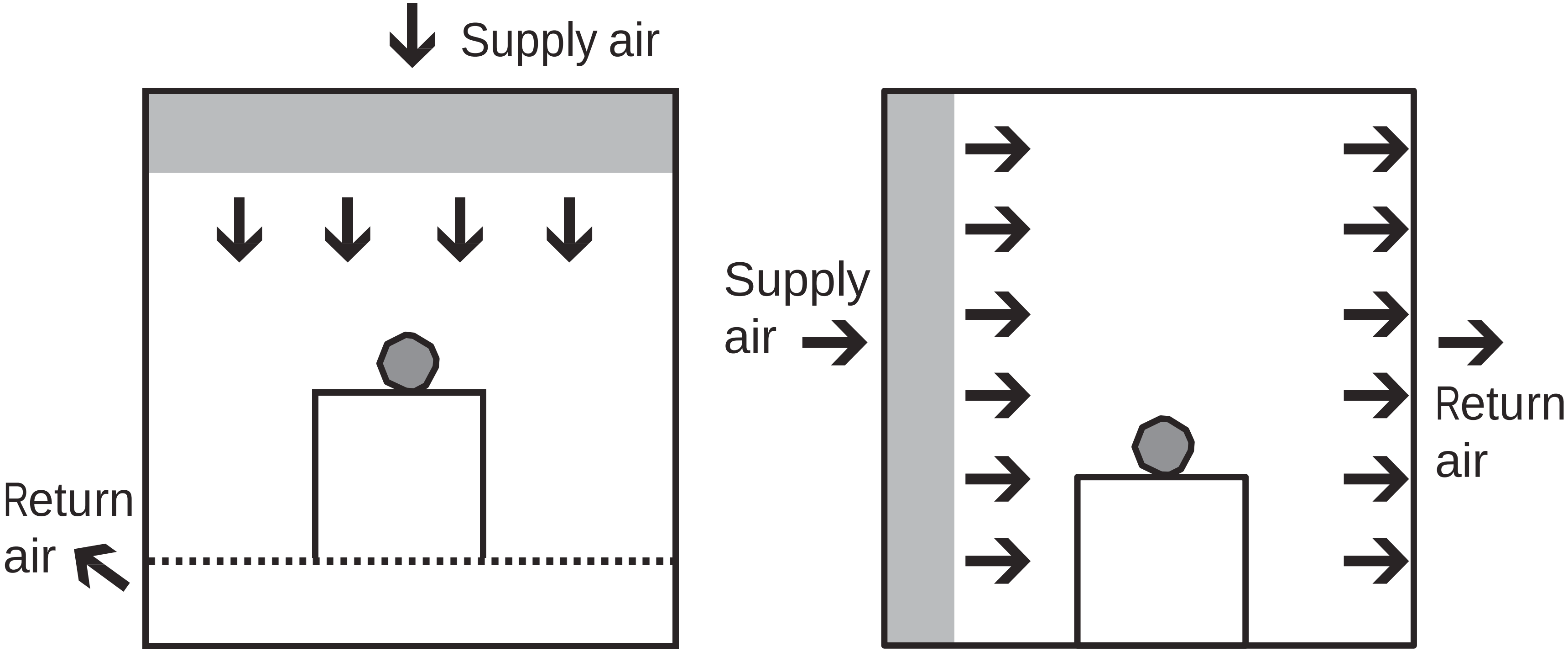
Humans typically emit 100000 to 300000 particles per minute sized 0.3 µm and larger, sitting or standing.

WHY DO WE NEED A CLEAN ROOM ?

- Emitting sites = dusts, scratches near irises (high E field)
- Dust particles gather and weld together and to surface
- Local enhancement of E => βE
- e- from emitting site may disrupt the beam



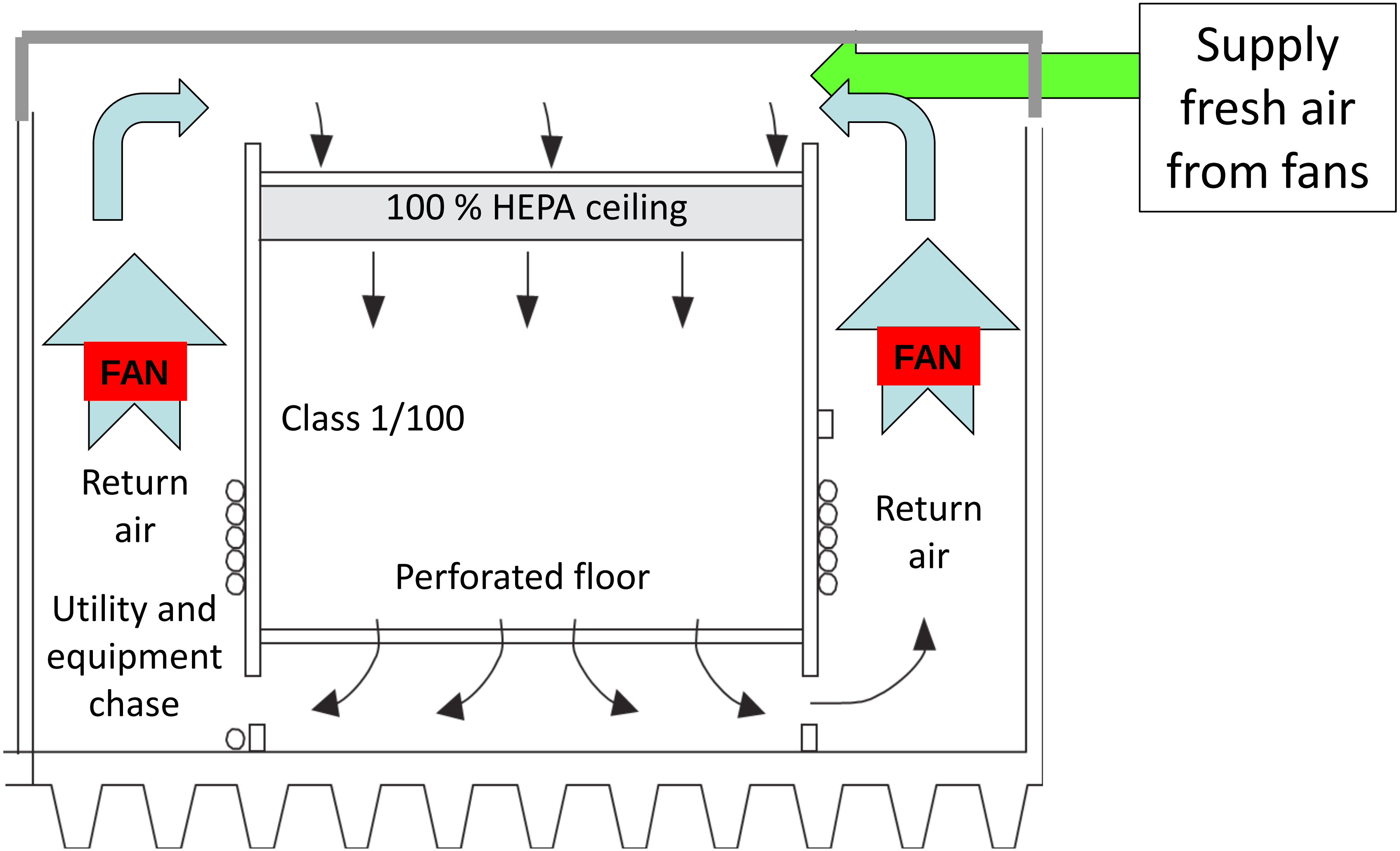
Cleanroom: horizontal and vertical flow



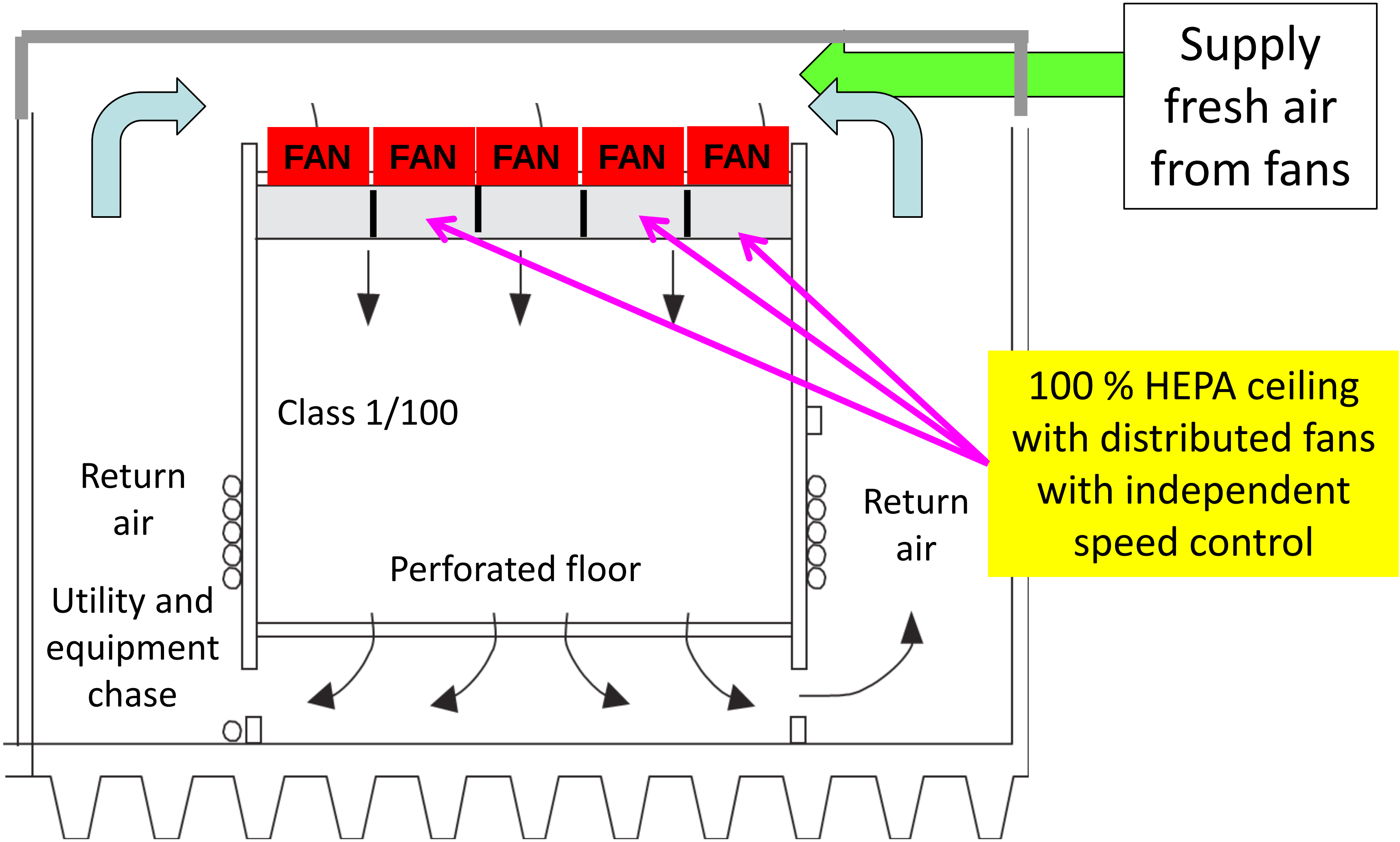
vertical

horizontal

Cleanroom: unidirectional flow, ducted



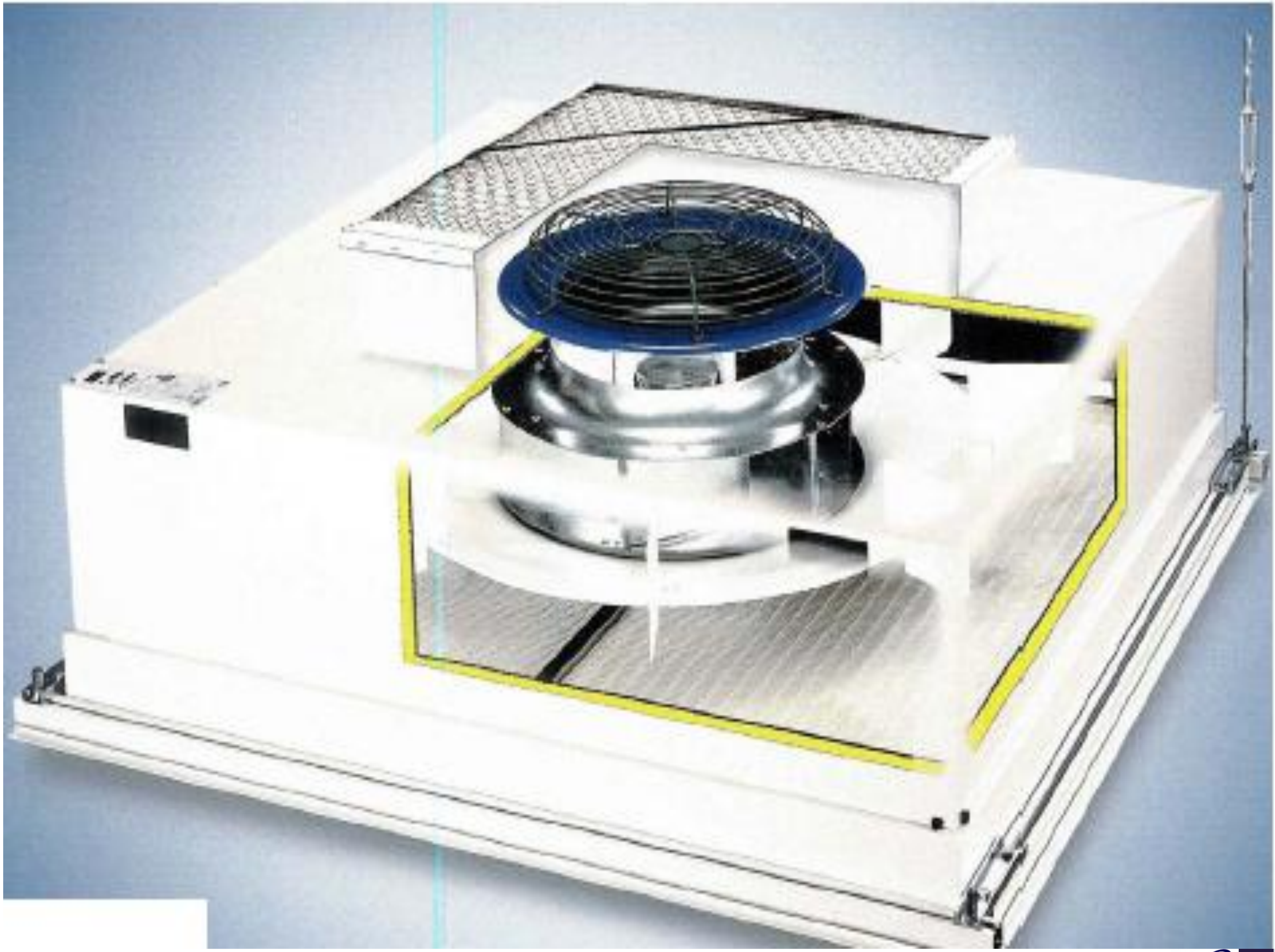
Cleanroom: unidirectional flow, ceiling fans



Supply
fresh air
from fans

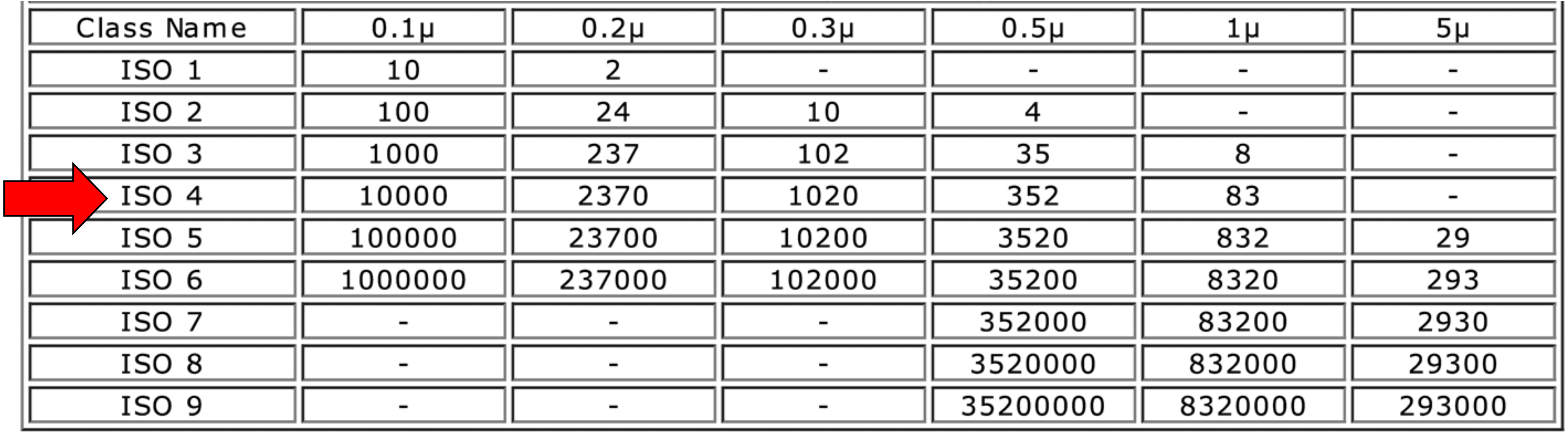
100 % HEPA ceiling
with distributed fans
with independent
speed control

Filters and fan units: distributed structure



Cleanliness according ISO 14644

Particles per m^3

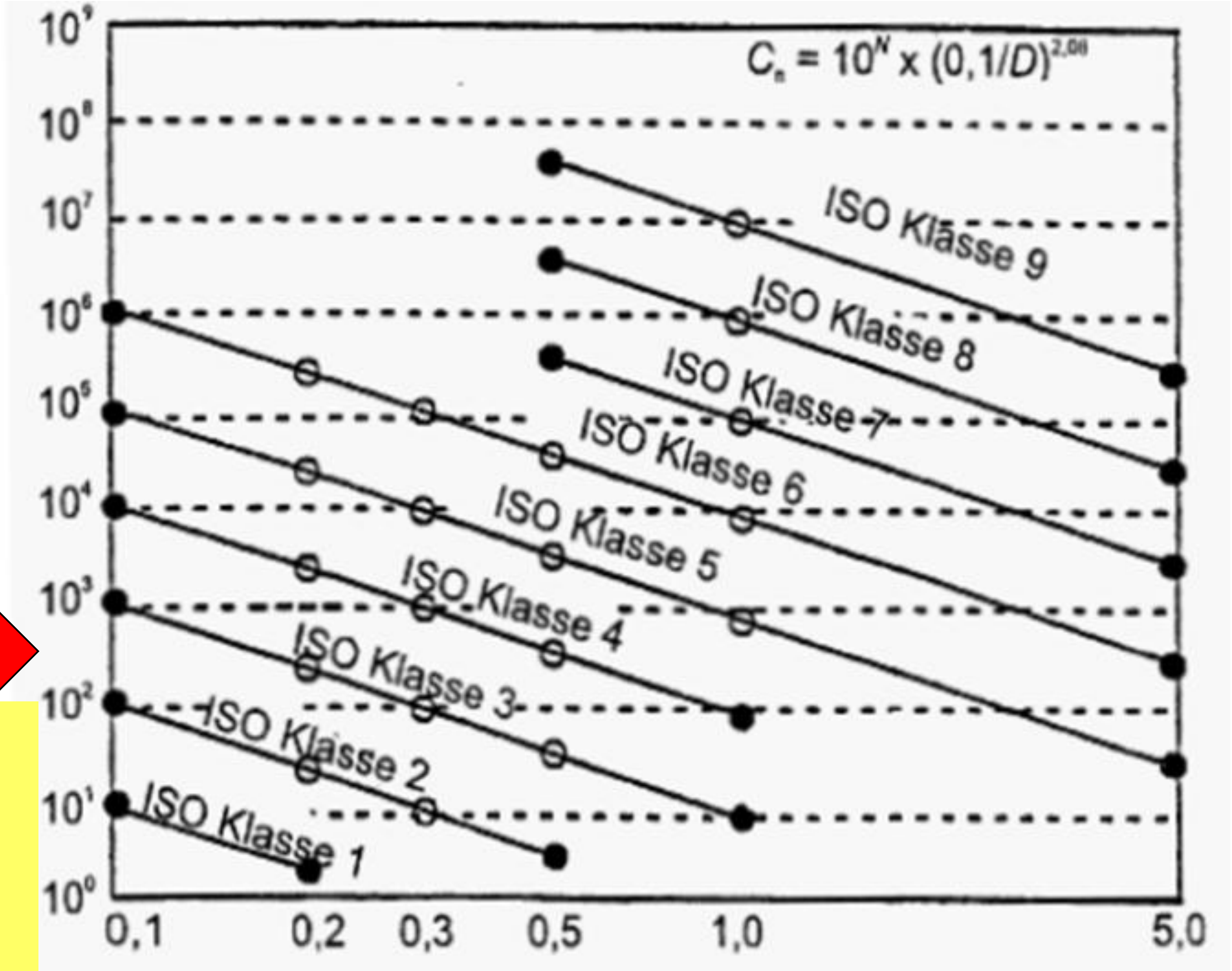


Class Name	0.1μ	0.2μ	0.3μ	0.5μ	1μ	5μ
ISO 1	10	2	-	-	-	-
ISO 2	100	24	10	4	-	-
ISO 3	1000	237	102	35	8	-
ISO 4	10000	2370	1020	352	83	-
ISO 5	100000	23700	10200	3520	832	29
ISO 6	1000000	237000	102000	35200	8320	293
ISO 7	-	-	-	352000	83200	2930
ISO 8	-	-	-	3520000	832000	29300
ISO 9	-	-	-	35200000	8320000	293000

ISO 4: SC cavities typical clean room for final treatments

Cleanliness according ISO 14644

Particle Concentration per m³



Particle size (µm)

SC cavities typical clean room for final treatments

Cleanroom: air velocity, air changes per hour

Class	Flow	Average air velocity	Air change per hour
M7 & M6.5 (Class 100,000)	**N, M**	0.005 - 0.041 m/s	4 – 48
M6 & M5.5 (Class 10,000)	**N, M**	0.051 - 0.076 m/s	60 – 90
M5 & M4.5 (Class 1,000)	**N, M**	0.127 - 0.203 m/s	*150 – 240*
M4 & M3.5 (Class 100)	**U, N, M**	0.254 - 0.457 m/s	*240* – 480
M3 & M2.5 (Class 10)	**U**	0.305 - 0.457 m/s	300 – 540
M2 & M1.5 (Class 1)	**U**	0.305 - 0.457 m/s	360 – 540
M1 & Cleaner	**U**	0.305 - 0.508 m/s	360 – 600

S.C. EXFEL

Flow: U = unidirectional; N = nonunidirectional; M = mixed

The different conditions for classifying the cleanroom

As built:

Condition where the installation is complete with all services connected and functioning but with no production equipment, materials or personnel present.

At rest:

Condition where the installation is complete with equipment installed and operating in a manner agreed upon by the customer and supplier, but with no personnel present.

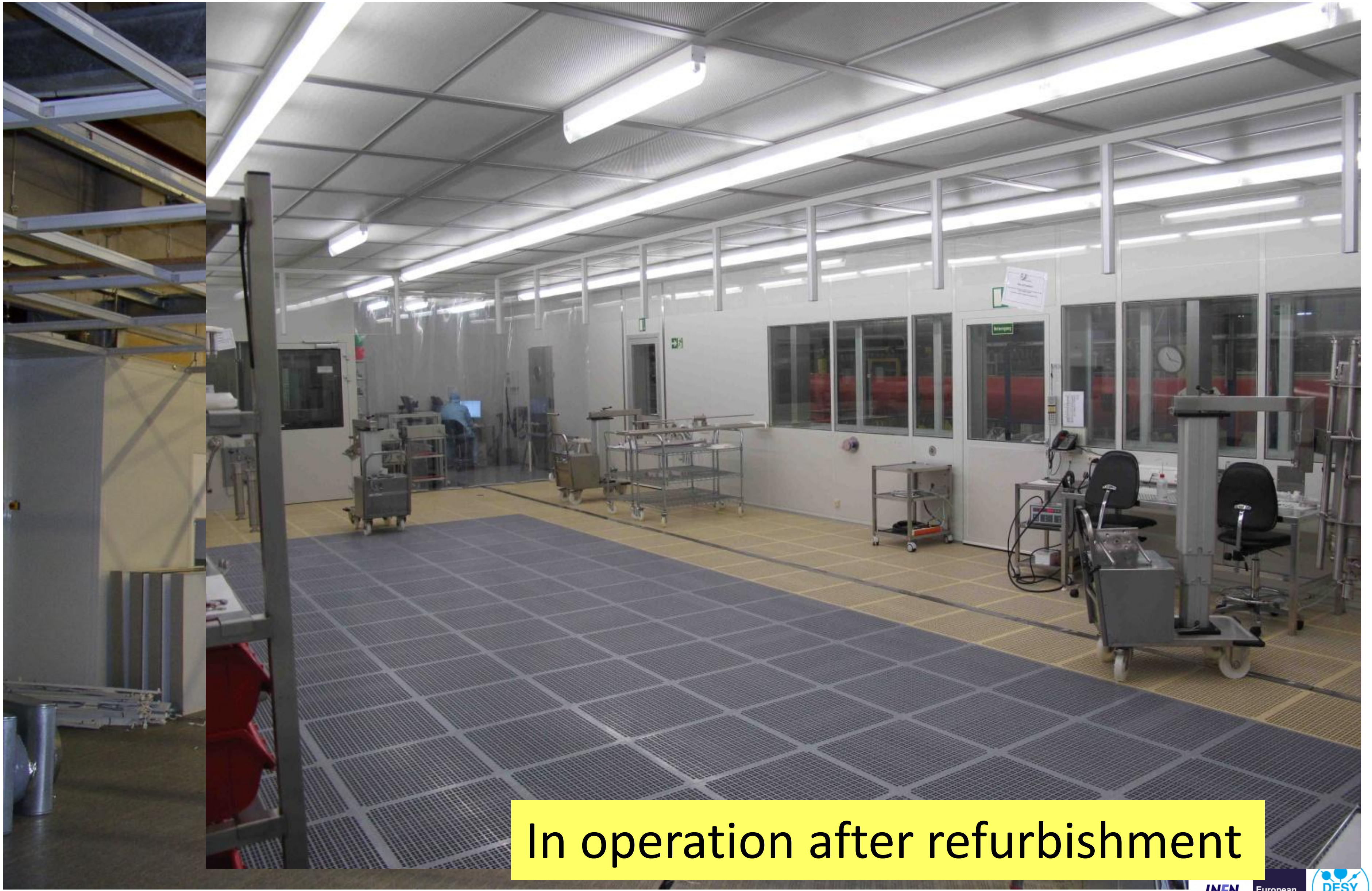
Operational:

Condition where the installation is functioning in the specified manner, with the specified number of personnel and working in the manner agreed upon.

Clean room ISO 4 ceiling @ EZ.

Filter and fan holders.

DESY Cleanroom refurbishment: roof structure



In operation after refurbishment

Requirements to SRF Cleanroom technology

- For cavity applications critical contaminations:

  - particles + (sharp) surface irregularities

  - hydro carbons

  - sulfur (EP process)

- **Task:** Define and install the

  appropriate clean environment + procedures + media

  → cleaning of cavity + all auxiliaries

  → “dust free” assembly

  → pumping & venting without recontamination (particles, HCarb)

  → documentation

- **BUT: Compared to semi-conductor industry we poorly know the surface conditions**

- for review of contamination and cleaning mechanisms see:

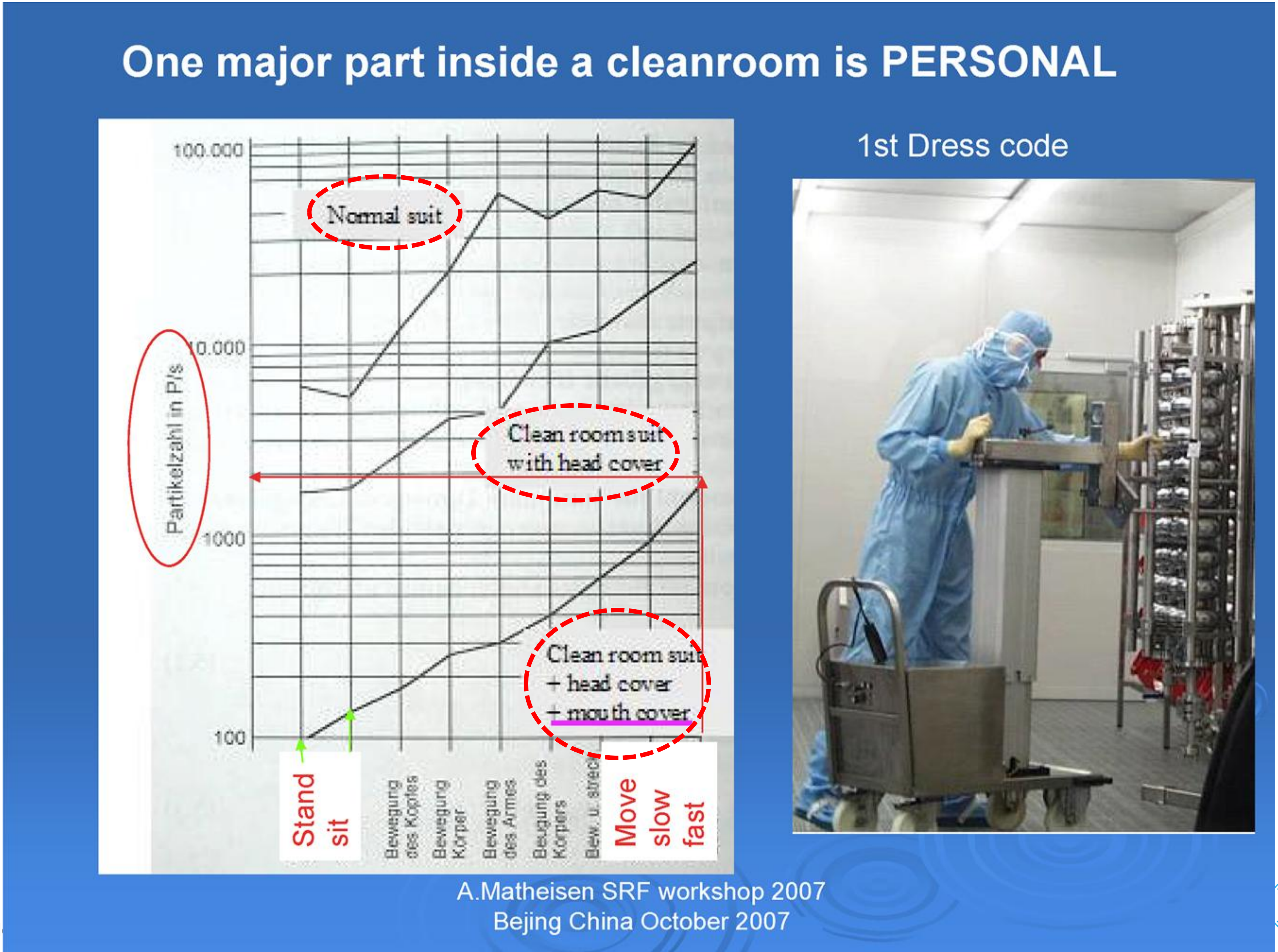
  P. Kneisel, B. Lewis, SRF workshop 1995

Cleanroom: contamination source

The largest cause of **contamination** in a cleanroom is **personnel.**

Action	Particles generated / minute (0.3 µm or larger)
Sitting or standing, no movement	10^5
Sitting or standing, light head, hand and forearm movements	$5\text{x}10^5$
Sitting or standing, average body or arm movement, toe tapping	10^6
Changing positions, sitting to standing	$2.5\text{x}10^6$
Slow walking (3.2 km/h)	$5\text{x}10^6$
Average walking (5.6 km/h)	$7.5\text{x}10^6$
Fast walking (8 km/h)	10^7
Climbing stairs	10^7

Particle generation by human activities



One major part inside a cleanroom is PERSONAL

1st Dress code

A.Matheisen SRF workshop 2007
Bejing China October 2007

Cleanroom: particle transport and generation

Unavoidable sources of particles and particle motion:

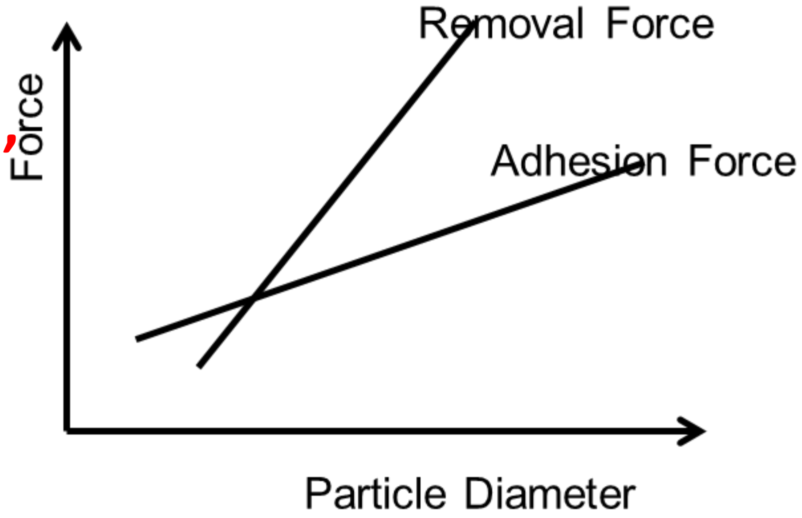
- **Mechanical vibration**: Hitting, banging, tightening bolts with wrench, closing a valve quickly.
- **Rubber gloves** release organic on contact with surfaces. (Norms available).
- **Thermal cycling**. One cannot avoid cycling to 2 K! Particles can be dislodged by thermal stress differences.

Particle transport agents

- **Kinetic:** carried by **air** or other fluid
- **Vectoring:** carried by **objects or people**
- **Electric:** moved by **electric fields**
- **Thermal:** moved by **convection** or thermophoresis

Few fundamentals of contamination in CR

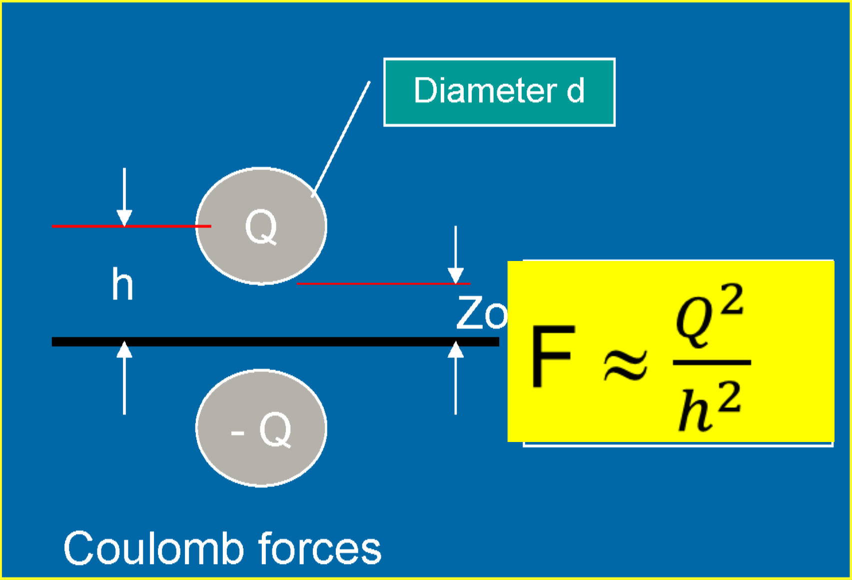
- **Cleaning: Overcome adhesion forces of particles**
- **The adhesion forces** depend on:
  - **material, roughness, electrical charge, hardness of particle & surface**
  - **size and shape** of the particle
  - **temperature & humidity** of the surrounding environment



- Adhesion forces between particulates and a surface:
  - **van der Waals** (often dominating, in air stronger than in liquids)
  - **capillary** (for hydrophilic materials)
  - **electrical double layer** (in liquids)
  - **electrostatic**
- **Care! In our case particle size and shape: no typical size**

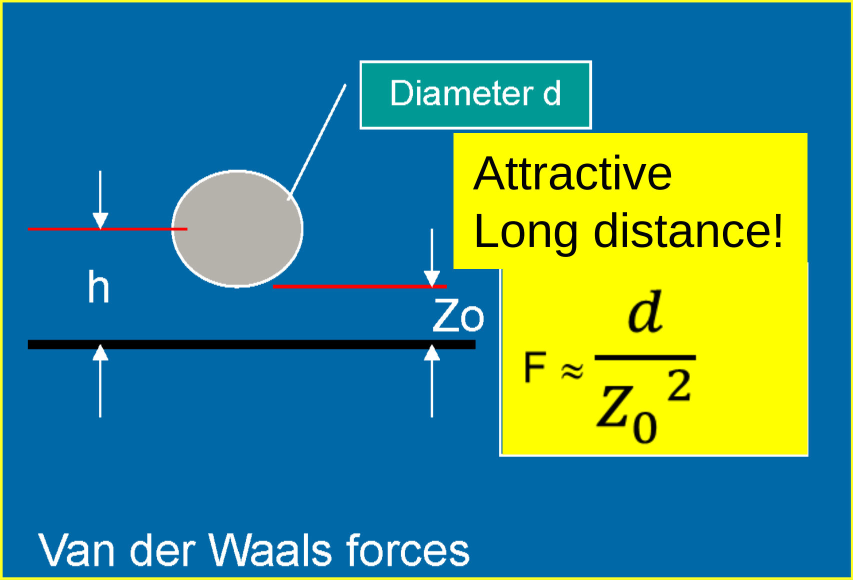
Cleanroom: particle adhesion on surfaces

Forces that make attractive potential to particulates facing a surface



$$F \approx \frac{Q^2}{h^2}$$

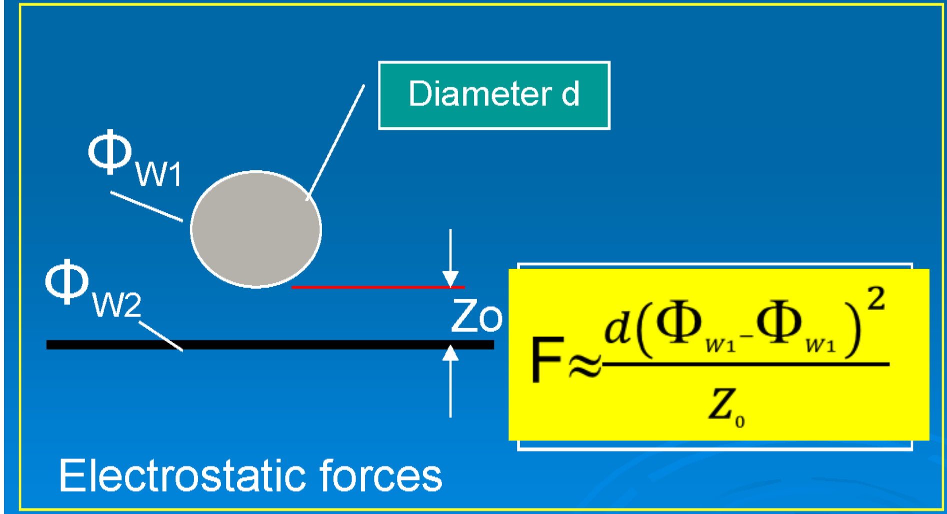
Coulomb forces



Attractive
Long distance!

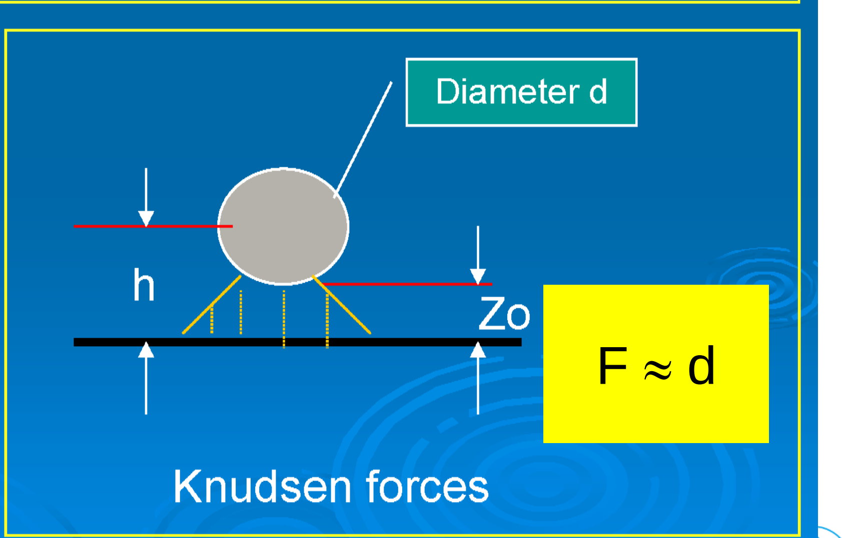
$$F \approx \frac{d}{Z_0{}^2}$$

Van der Waals forces



$$F \approx \frac{d(\Phi_{W1} - \Phi_{W1})^2}{Z_0}$$

Electrostatic forces

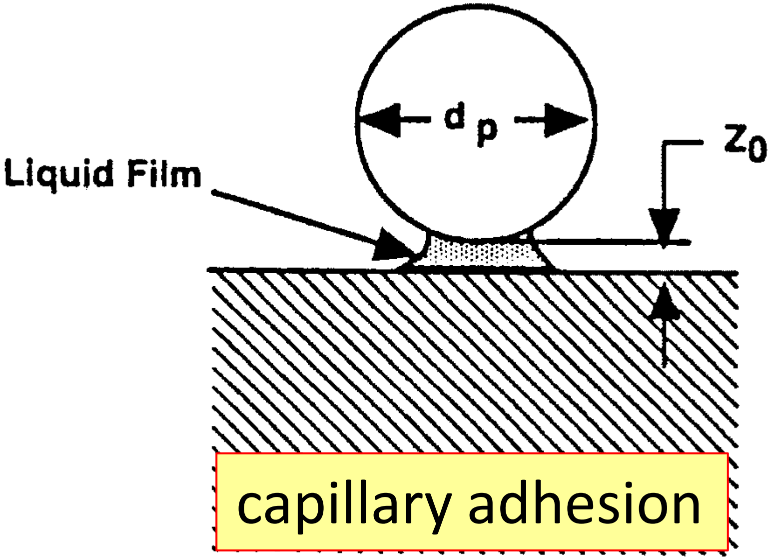


$$F \approx d$$

Knudsen forces

Cleanroom: particle adhesion on wet surfaces

- During drying cavity surface is **more and more** sensitive to particle contamination.
- Wet surface shows the so called “capillary adhesion”.
- Particles are much more “glued” to the surface then for dry items
- Therefore **cavity must dry before** any manipulation: no installation of accessories, flanges, etc. have to be done
- Great **care** must be taken also **walking close to wet cavities**
- Typical drying time for EXFEL: **between 9 and 18 hours**



$F_c = 2\pi d_p \gamma$

where γ is the liquid surface tension

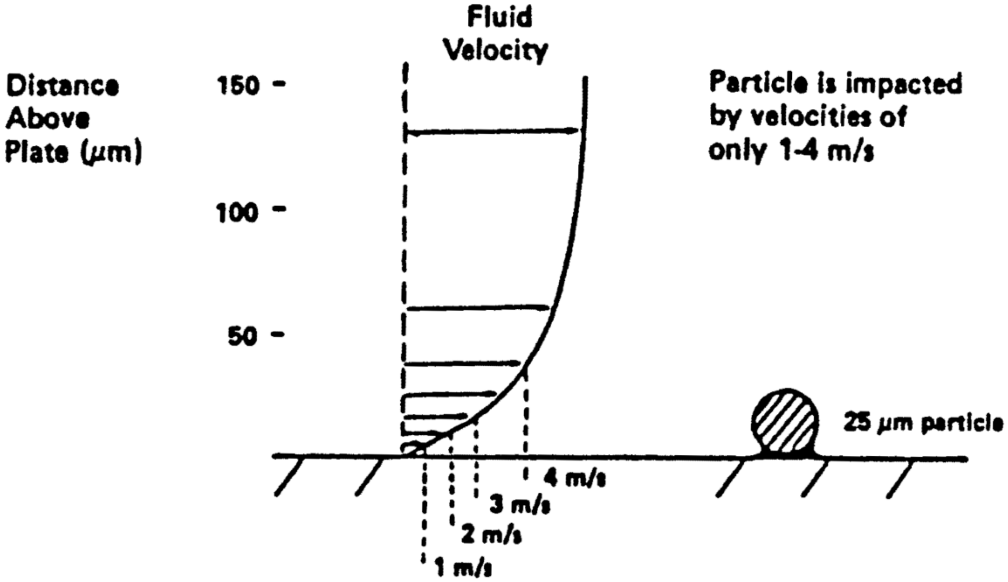
capillary adhesion

Cleanroom: particle on surfaces, how to remove?

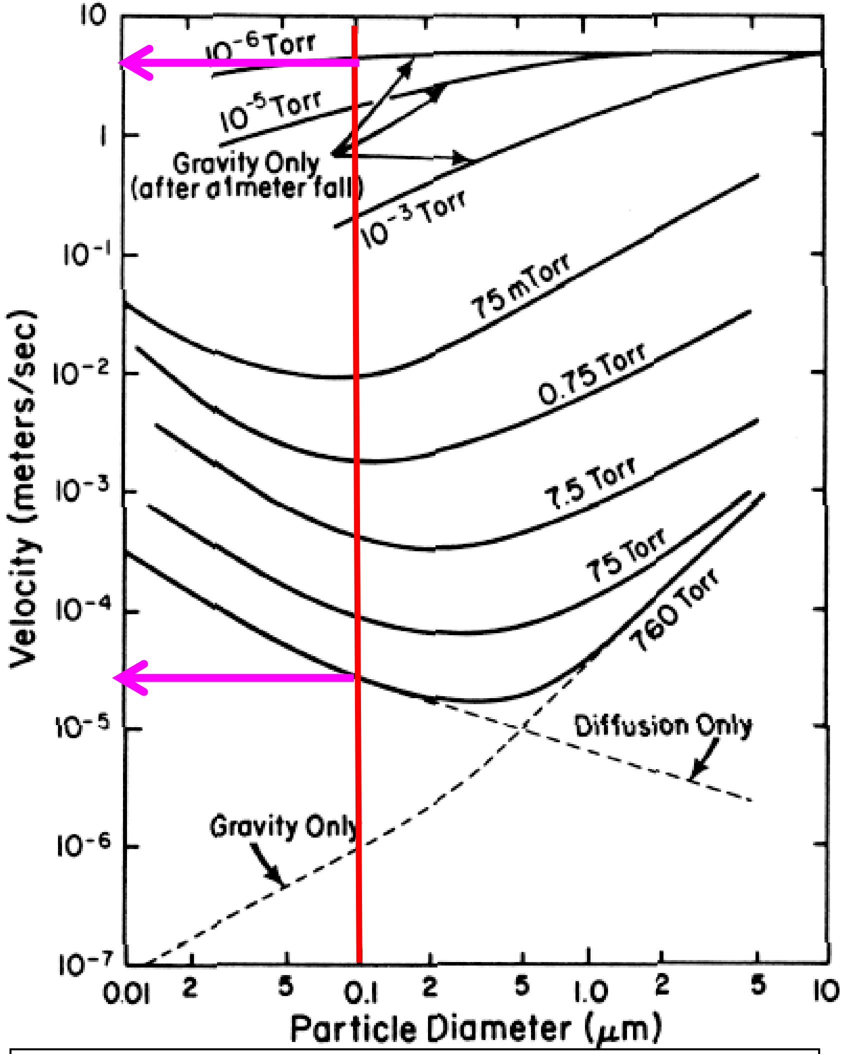
How to introduce forces (energy) to particles on a surface?

Ultrasonic cleaning	
Discharge of static loads:	Ionized Gases (air, N_2)
Reduce surface tension:	Alcohol / Detergents
Rinsing:	High Pressure Rinsing
Enforced gas flow:	Air guns (N_2 ; Ar)

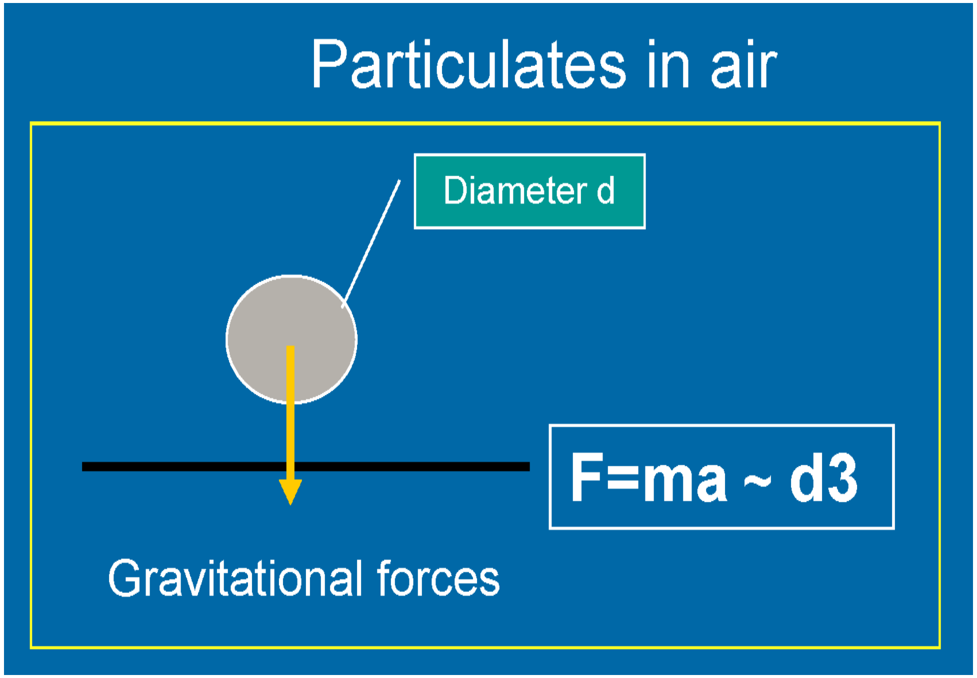
- **General problem: Problem: speed on surface = 0**



Particles and Surfaces (stationary) in Air



Settling (fall) velocity for particles at constant temperature and with no electric fields



Particulates in air

Diameter d

F=ma ~ d3

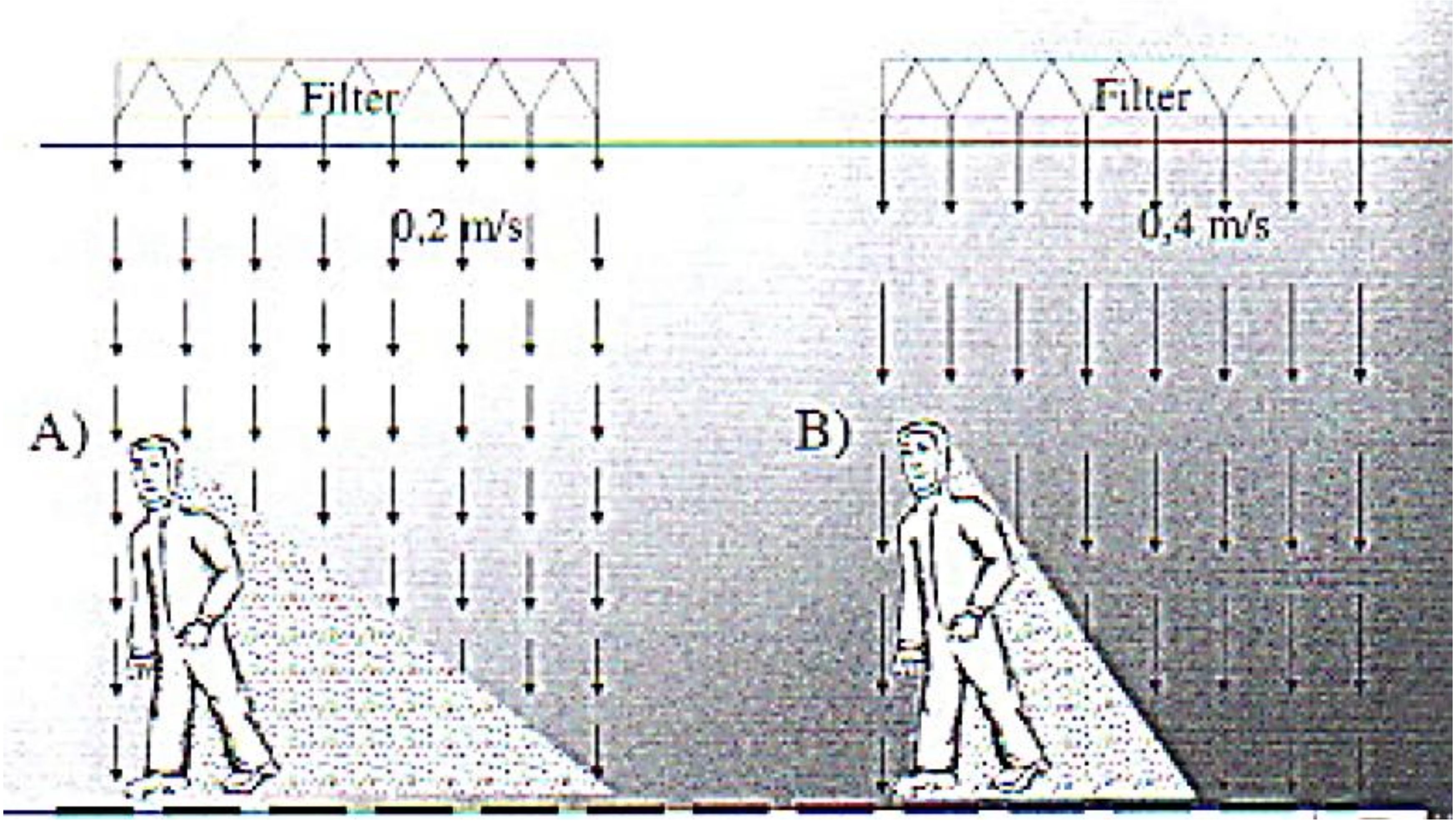
Gravitational forces

- Particles **bigger than 1 µm fall out** (sedimentation)
- Particles **from 0.1 - 1 µm fall very slowly**
- Particles **less than 0.1 µm remain suspended/diffuse**

From John O'Hanlon vacuum@dakotacom.net

Cleanroom: effect of laminar flow on particles

Particles generated by moving people displacement by laminar flow: laminar flow speed effect.



Non laminar wake caused by object in laminar flow

Cleanroom Technology Th. von Kahlden www.cci-vk.de

CCI - von Kahlden GmbH

Cleanroom: wrong and right way of walking

Behavior of personal inside a cleanroom

Wrong !!!!

Right !!!

A.Matheisen SRF workshop 2007
Bejing China October 2007

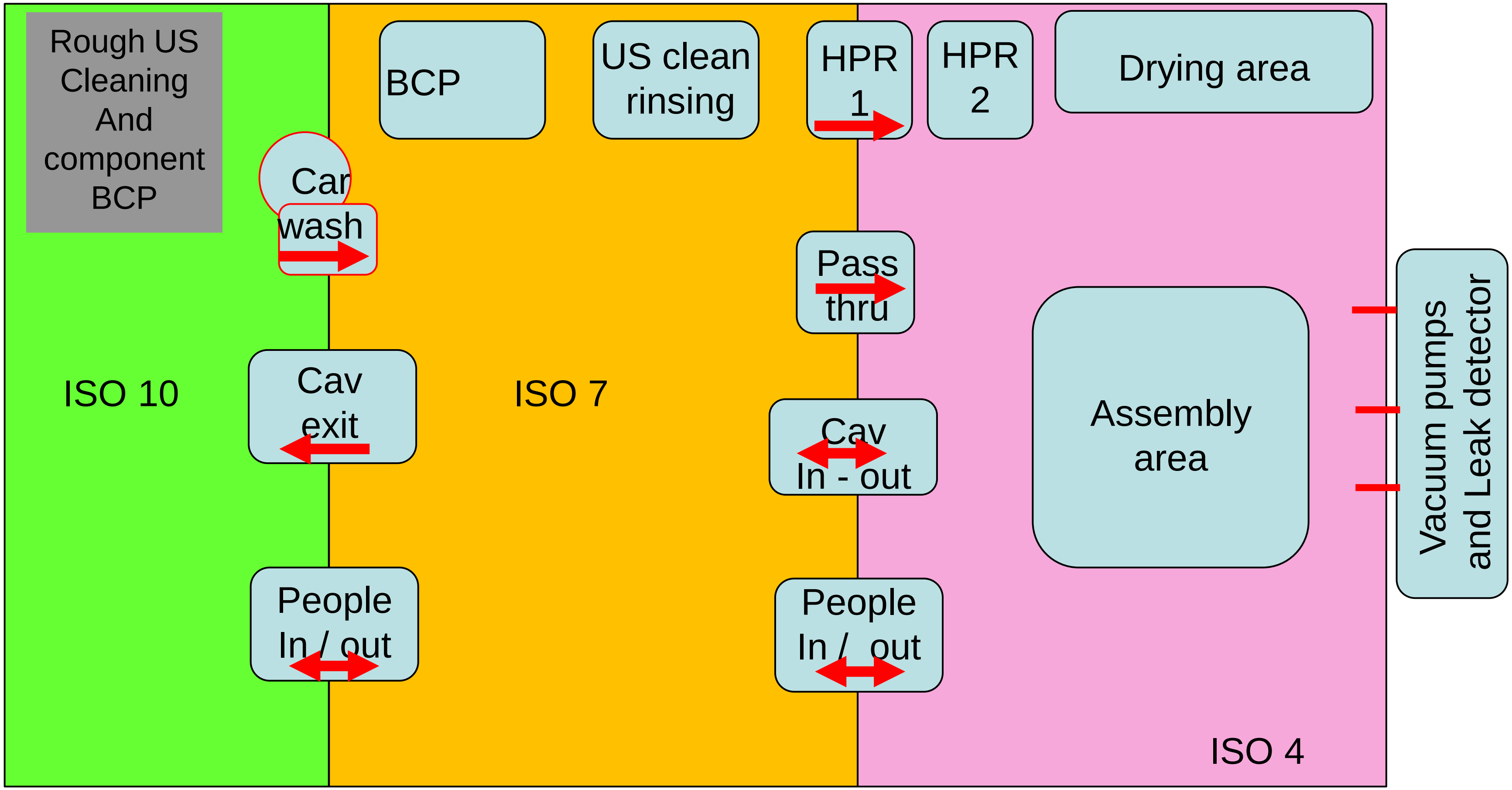
Cleanroom: opening a door in the correct way

Videos fm Axel Matheisen - DESY

Cleanroom: closing an open cavity

Videos fm Axel Matheisen - DESY

Sketch of a clean room for Serial production



Component transportation under water

Transfer of components after local BCP to the ISO7 clean room. After etching they are transferred under UPW. Specific attention must be paid to “floating contaminants” as oil, dust, hear, etc.

Key points for CR

- **Well cleaned components subcomponents** (flanges, **bolts, nuts**, gasket, external parts of cavities, valves, etc) using standardized procedure, if applicable, on all components.
  - **Blowing of components to assembled just in time**
- Correct items entering procedure, mainly based on the use of **WATER** rinsing
- **Precisely defined procedures**
- Well-trained and motivated personal
- Keep **duration** of actions at **open cavity** as **short** as possible
- **Check cleanliness**

Subcomponent preparation

Summary and consideration

- **Cavities that do not have "basic" problems as inclusions are mainly limited by FE**

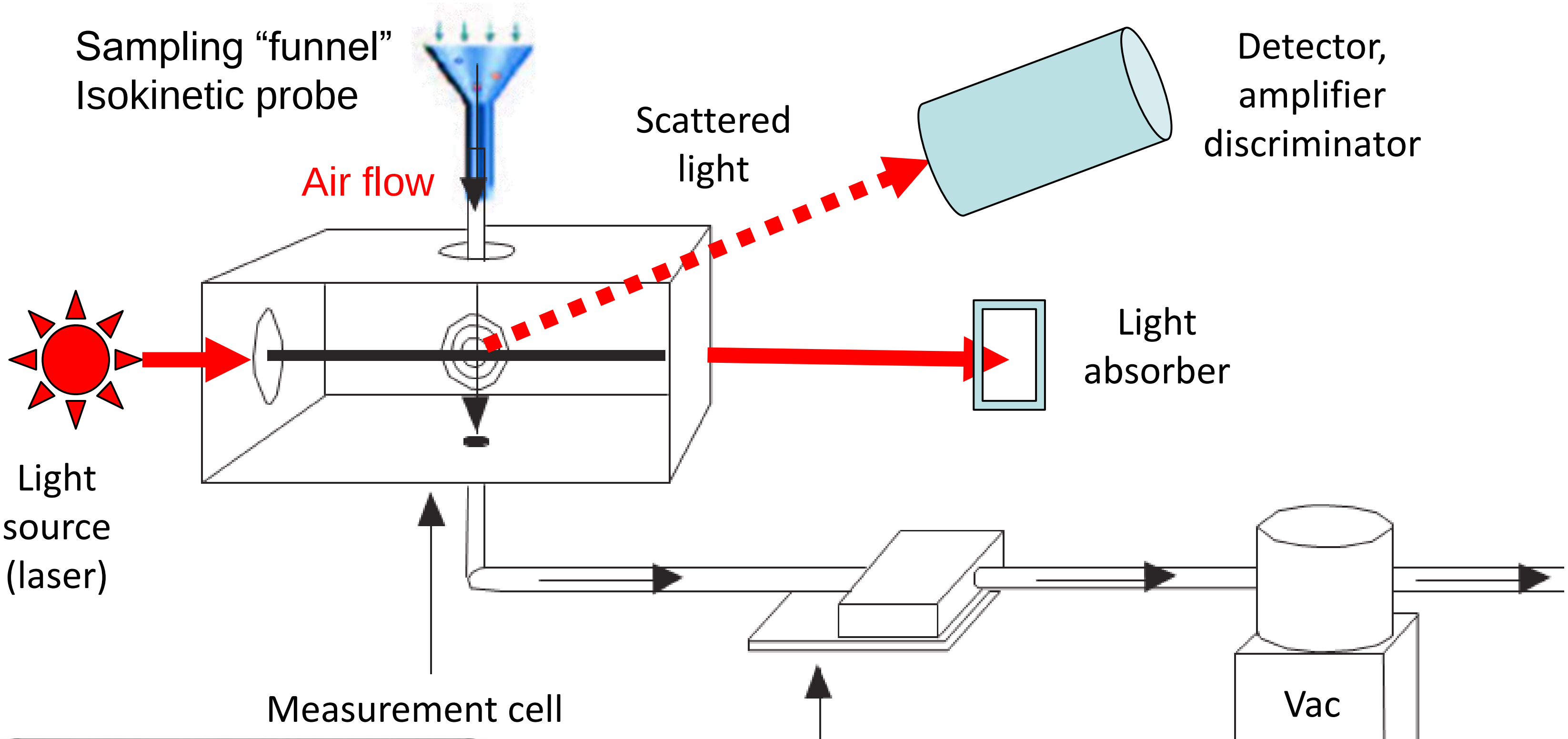
- Standard cleaning and assembly procedures allow high quality cavity performance, but:
  Field emission (= dark current) is still the **main limitation**, if usable gradients above 25 MV/m in multi-cell accelerator cavities are required

How is measured the cleanliness of the CR?

Measurement technology for Contamination

- Particles
  - In air => available
  - In liquids => available
  - On surfaces (flat) => online available for big particles
  - On surfaces (“complicated“) => online not available
  - Vacuum => available, but tricky
- (Airborne Molecular Contamination => partly available)

Principle of optical particle counters



Sampling “funnel”
Isokinetic probe

Air flow

Scattered light

Detector, amplifier discriminator

Light source (laser)

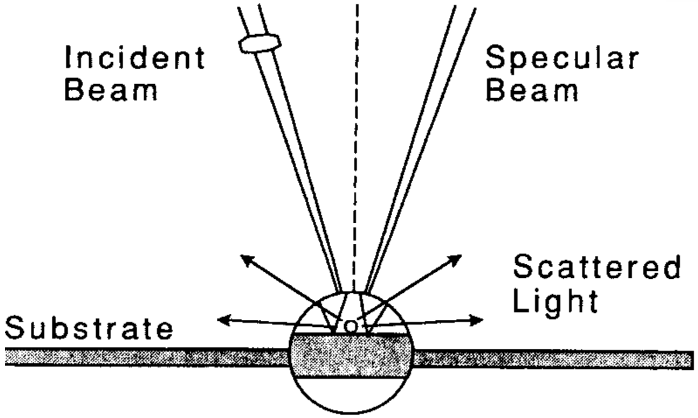
Light absorber

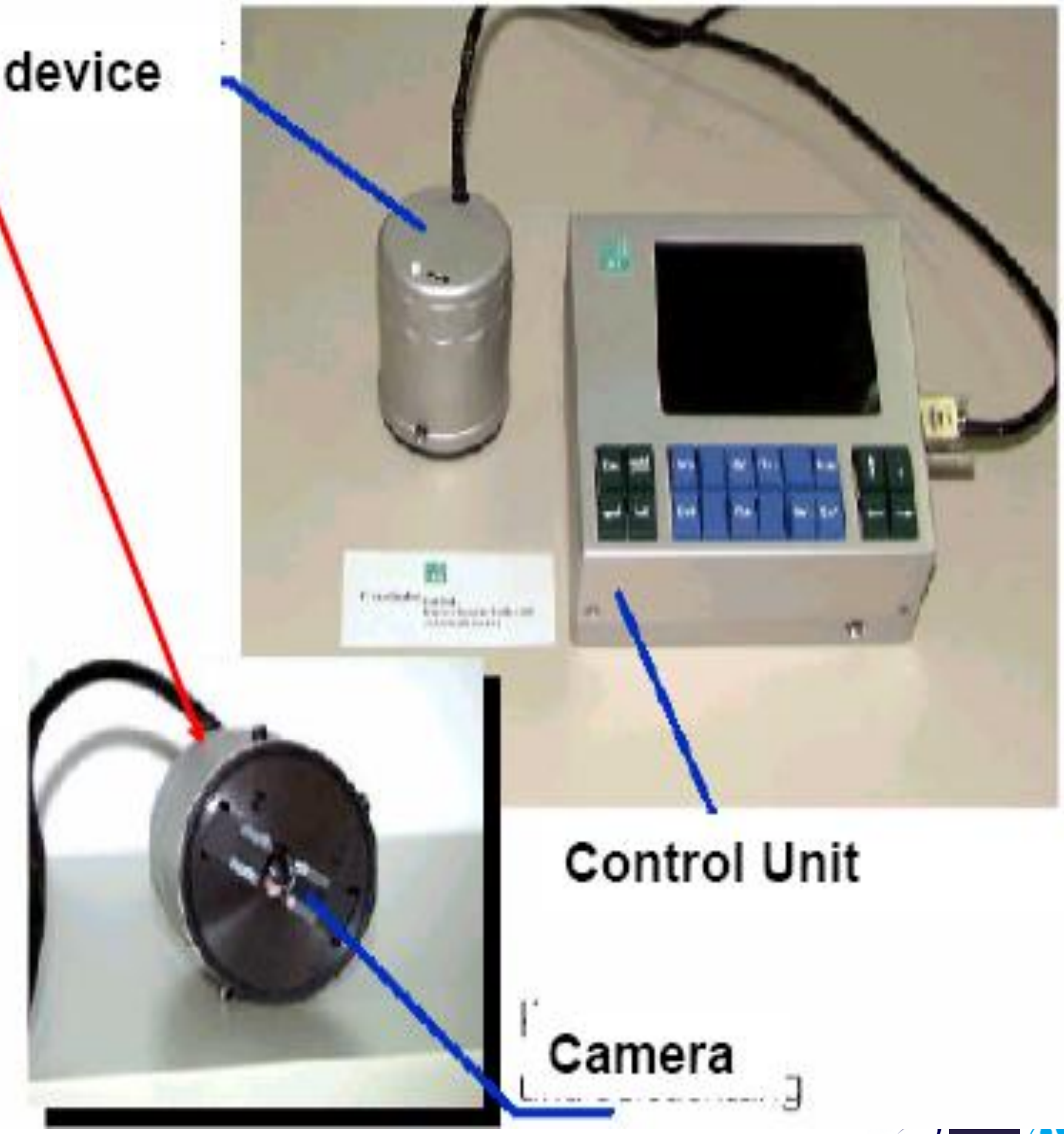
Measurement cell

Vac Pump

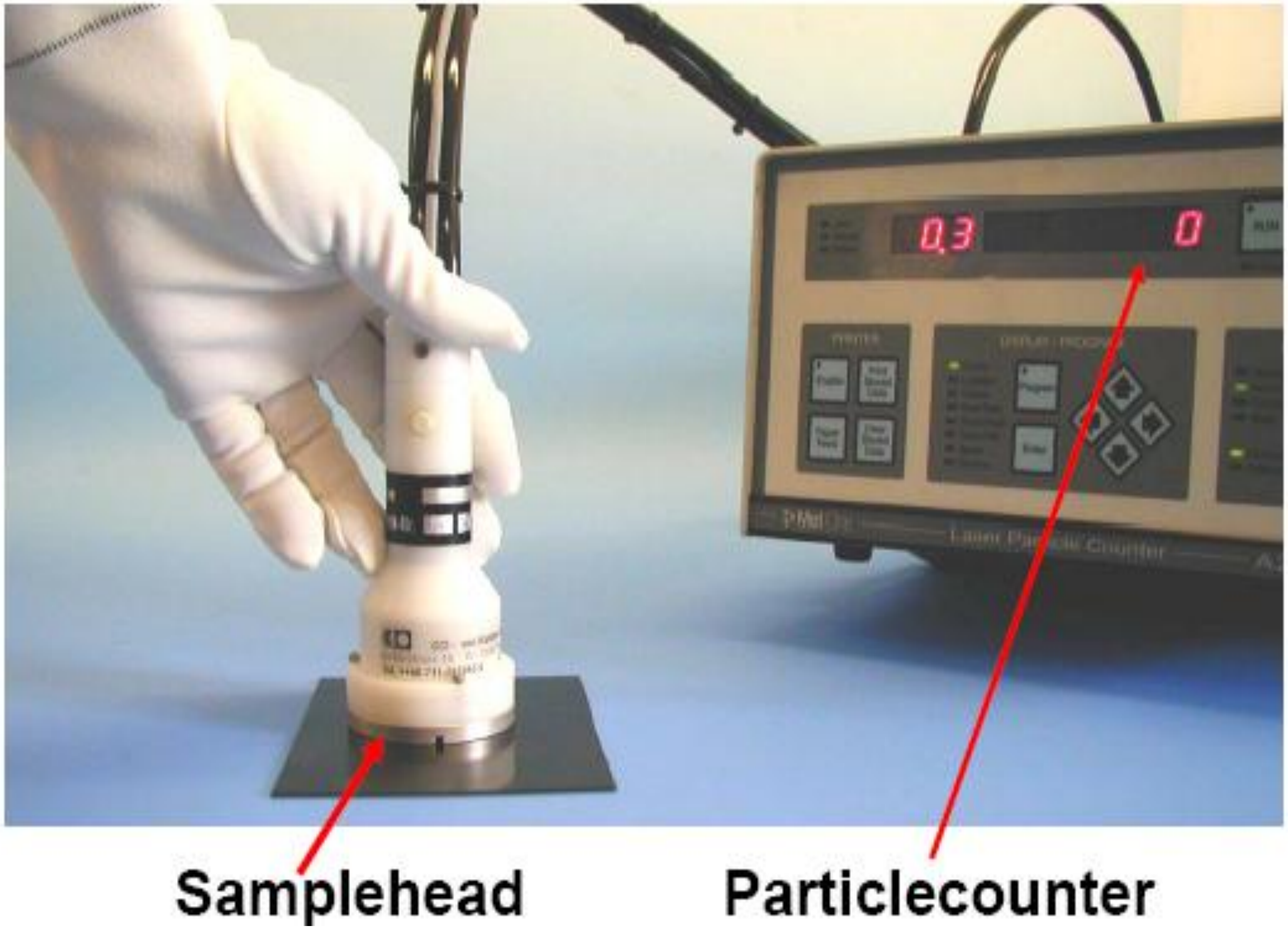
Flow controller

Particle measurement on surfaces: optical system





Particle collection and measurement from surfaces



Modified “standard” particle counter with special head for surface contamination measurements (pneumatic transfer of particles)

Literature

- http://cas.web.cern.ch/CAS/Spain-2006/PDFs/Taborelli.pdf
- NIST, http://webbook.nist.gov/chemistry/name-ser.html
- Paolo Chiggiato, CAS school on vacuum
- Ed by W. White, Cleanroom design
- W. Kern ed., Handbook of Semiconductor Wafer Cleaning Technology
- **A User's Guide to Vacuum Technology , O. Hanlon**
- Fundamentals vacuum tecnology, oerlikon Leybold Vacuum web side
- Festo, clean room technology
- C. Antoine, Erice School
- E. Ciapala et al., SRF Workshop 2001
- L.Gail, H.P.Hortig, Reinraumtechnik (in german), 2002
- P. Kneisel,B. Lewis, SRF Workshop, 1995
- P. Kneisel, Contamination Workshop Jlab, 1997
- H.Padamsee, J.Knobloch, T.Hays, RF Superconductivity f. Accelerators, 1998
- D.L. Tolliver, Handbook of Contamination Control in Microelectronics, 1988

- In general all **Proceedings of the SRF Workshops**

Acknowledgement and Comments

- Thanks to Axel Matheisen, Detlef Reschke and to all people of the companies EZ and RI for picture and info.

Final comments

- The big effort of EXFEL SC cavities construction **, besides the fact that is done at the industry,** therefore with specific time schedule and constrains, it would be the **"largest" occasion** that was never been available in the past to understand the **real effect of treatments on SC cavities performances.**
- Also **errors and mistakes** can be a profitable source of information: during industrial production the number of "**non standard**" cavities is reduces at the minimum, but still large, and **differences** are (hopefully) well known.

The end…

The end… of this long running

Thanks for the attention

Appendix 1: HF safety and use

HF use: safety rules

- **Hydrofluoric acid HF must be used with special care with respect to other oxygenated and not oxygenated acids.**
- It tend to **diffuse and destroy** human tissues and bones, reacting with calcium making calcium fluoride and producing an unbalancing of blood electrolytes.

- Main concern is for **HYPOCALCEMIA**: **can cause your heart to stop.↓ Magnesium (Mg), ↑ K+, ↑ Fluoride**
- Contact with dilute HF solutions **may not produce immediate pain**, but may result in severe burns without immediate treatment. **Symptoms may be delayed.**

HF use: safety rules

- Pain onset for diluted HF exposition (up to 20%) could be delayed up to one day. HF exposition with concentration 40% - 50%, produces effects within 8 hours

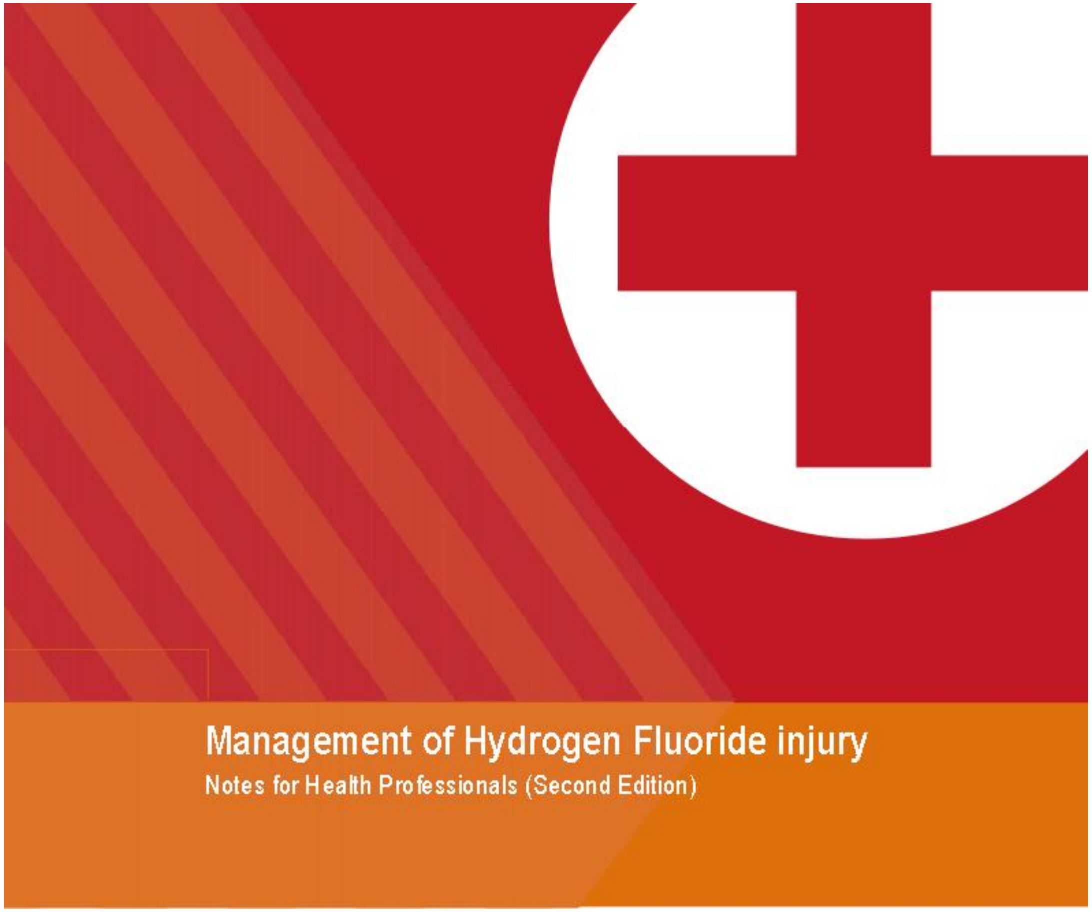
- **Large burns (> 25 sq inches, 160 cm2) could be fatal.**

- **Skin Exposure Rapid decontamination is critical to minimizing /preventing injury. First wash with water (5-15 min) then apply calcium gluconate first aid gel. Calcium gluconate binds HF and prevents it from penetrating deeper into tissues. This is critical.**

- http://www-esh.fnal.gov/CourseHandout_Mat/Hydrofluoric_Acid_Safety_Handout.pdf

HF use: safety rules

CTEF: Comité Tecnique Européen du Flor



Management of Hydrogen Fluoride injury

Notes for Health Professionals (Second Edition)

http://www.eurofluor.org/publications.html?cmd=download&pp=1&file=First_aid_UK.pdf

Hospital ER must be notified that an HF victim will be arriving for immediate treatment!

HF use: safety rules

Airborne Exposure, FNAL regulation

- Irritation to nose and throat at **3 parts per million (ppm)**
- Time-weighted average (**TWA**) of **0.5 ppm**
- Short-term exposure limit (**STEL**): **15 min @ 2 ppm**
- **30 ppm is considered immediately dangerous to life and health (IDLH)**

Therefore avoid any airborne exposition! Mask with filter / full body suits with compressed air connection (umbilical connection)

HF safety: standard protection equipment

Mask with large protection surface and helmet

TYCHEM F Suite

VITOJECT (Viton) gloves

HF safety: emergency equipment @ DESY

Service activities with high risk of HF exposition: full protection with umbilical air connection

BCP acid storage and plants

Appendix 2: Vacuum

Vacuum

- Few (basic) concepts and information
  - Physical quantities: pressure, volumetric flow, conductance, pumping speed, throughput
  - Pumping equation and equilibrium condition
  - Residual gas analysis
  - Hydrocarbon free vacuum
  - Leak detection
  - Vacuum cleaning, material, etc

Quantities

- **Quantity of gas (pV value),** (mbar × l). **$pV = nRT$**
- **Volumetric flow** (flow volume) **Q_v** (l/s, m_3/h, cm_3/s). The flow volume designates **the volume of the gas** which flows through a piping element within a unit of time, at the pressure and temperature prevailing at the particular moment.
- **Pumping speed S** (l/s, m_3/h, cm_3/s) The pumping speed is the volumetric flow through the pump's intake port. **$S = dV/dt$**
- **Pump throughput, pV flow Q_{pv} (mbar ls^{-1}):** is the product of the pressure and volume of a quantity of gas flowing through a piping element, divided by time **$Q_{pv} = d(pV)/dt$**
- **Pump down equation**

Gas that enters the pump

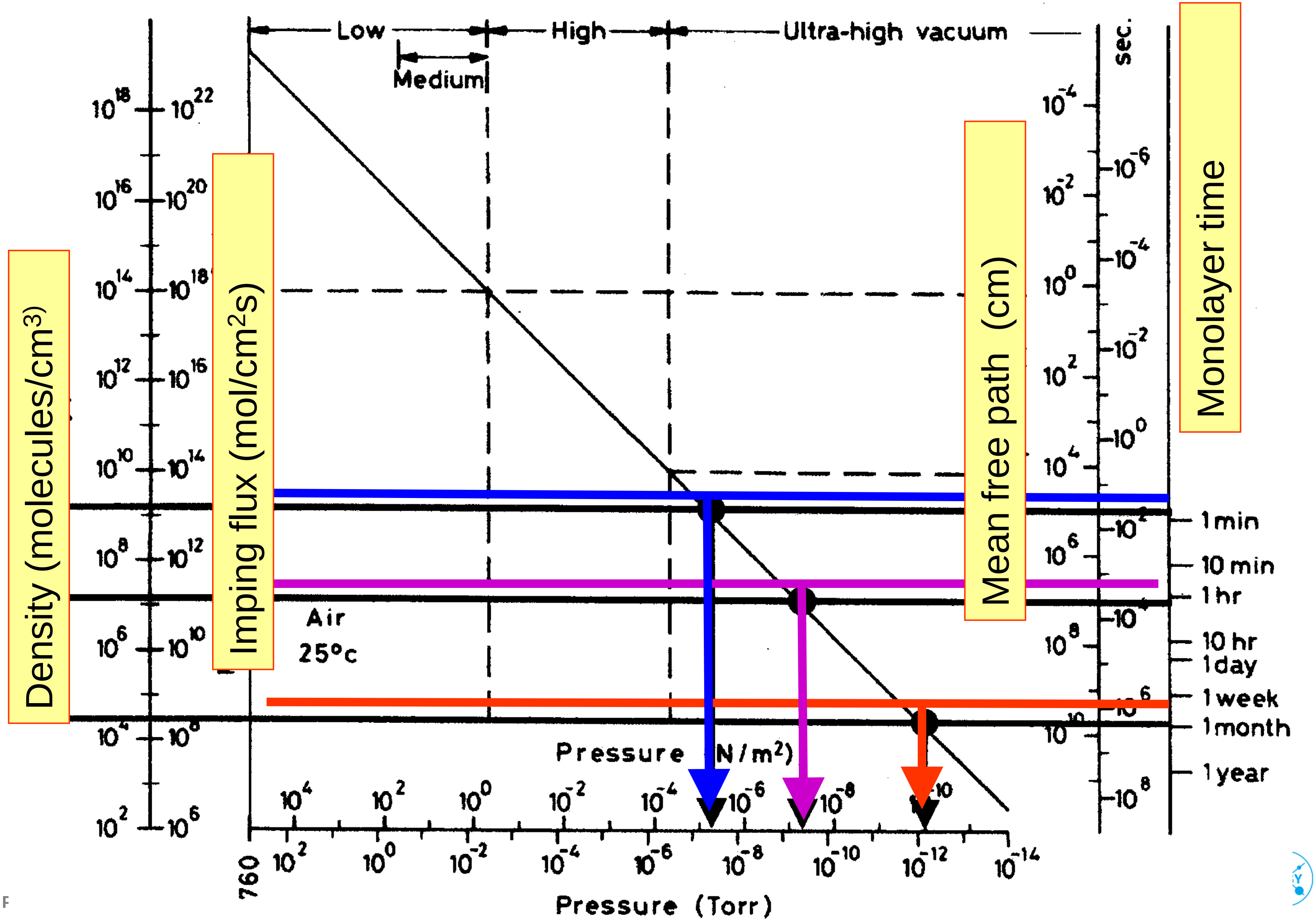
Gas that is pumped

Gas that is produced

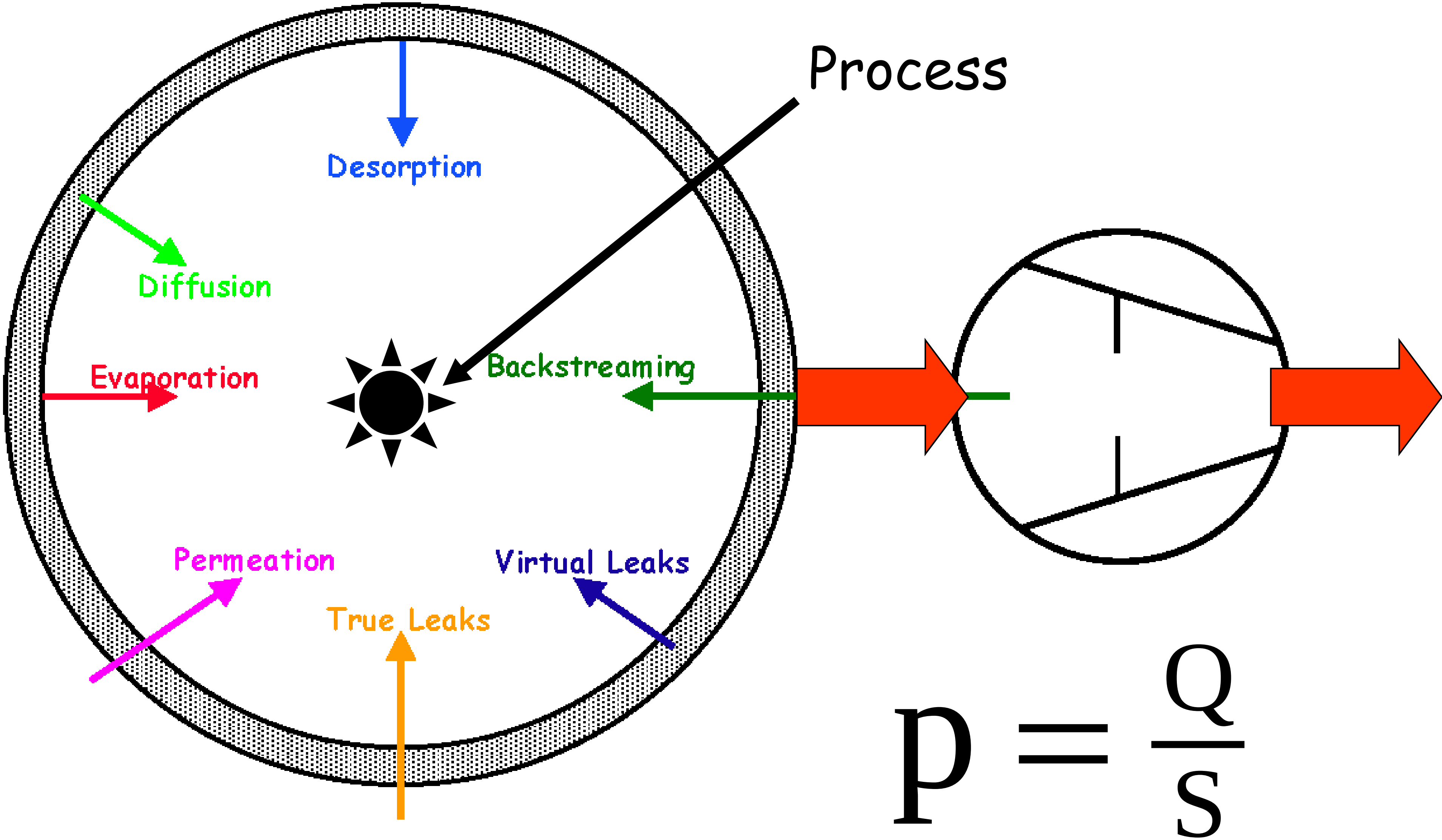
- In (quasi) stationay conditions, as for $dp/dt \approx 0$

$$Q_{tot} = S \cdot p$$

Fundamentals concepts



Equilibrium conditions



$$p = \frac{Q}{S}$$

Throughput (as the leak rate) and bubbling

Std cc/s = mbar l s-1	1 scc every	Bubbles (3 mm diam, water, 1 atm)
10-1	10 seconds	4 bubbles /second
10-2	100 seconds	25 bubbles / minutes
10-3	20 minutes	2 bubbles /minute
10-4	3 hours	1 bubble / 5 minutes
10-5	24 hours	can not be measured
10-7	3 months	can not be measured
10-9	30 years	can not be measured
10-10	300 years	can not be measured
10-11	3000 years	can not be measured

EXFEL specs for cavity and helium tank

Why Helium?

- **Non toxic**
- **Small molecules**: it enters more easily in small holes
- **Noble gas**: no reaction with other materials, metals or gases
- Small amount in the atmosphere: **5 ppm, therefore low background**
- **No crazy price**: about 8-9 €/m^3

SPSV: slow pumping slow venting

- **Motivation of automated venting + pumping**
  - **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 $\Delta p > 1$ 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: slow pumping slow venting (cnt)

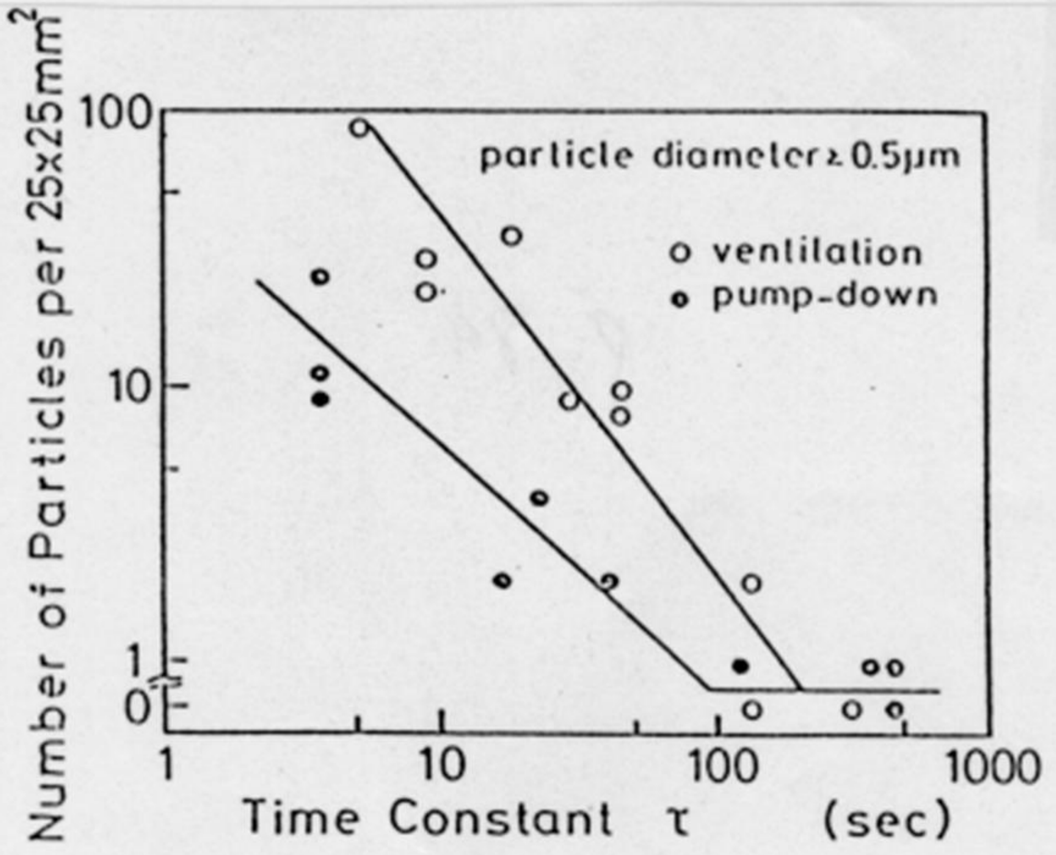
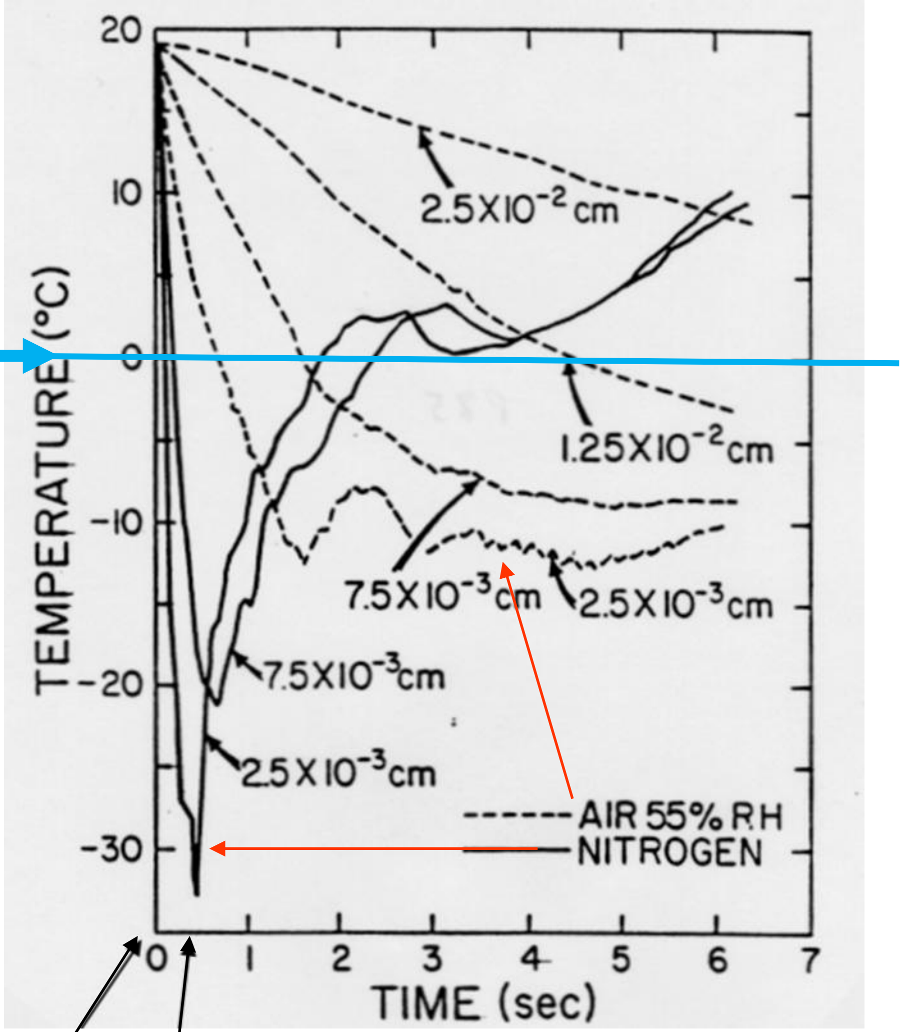


Fig.3 Increment in number of dust particles after each ventilation and pumpdown, is plotted versus the time constant τ.

Both rapid venting and rapid pumping introduce particle motion.

SPSV: slow pumping slow venting (cnt)

- **In vacuum temperature** measured with micro **thermocouples** (250 μm, 125 μm, 75 μm, 25 μm) during fast pumpdown.
- **Temperature is going down rapidly, well below 0 °C!**
- **Condensation** of water on particulates will arrive.
- **Sticking of particles on surfaces!**
- Movement of particles on the system!



760 Torr

650 Torr

SPSV: slow pumping slow venting (cnt)

For EXFEL cavities SPSV units had been capability to

- **Pump cavities**
- **Vent cavities**
- **Leak check cavities**

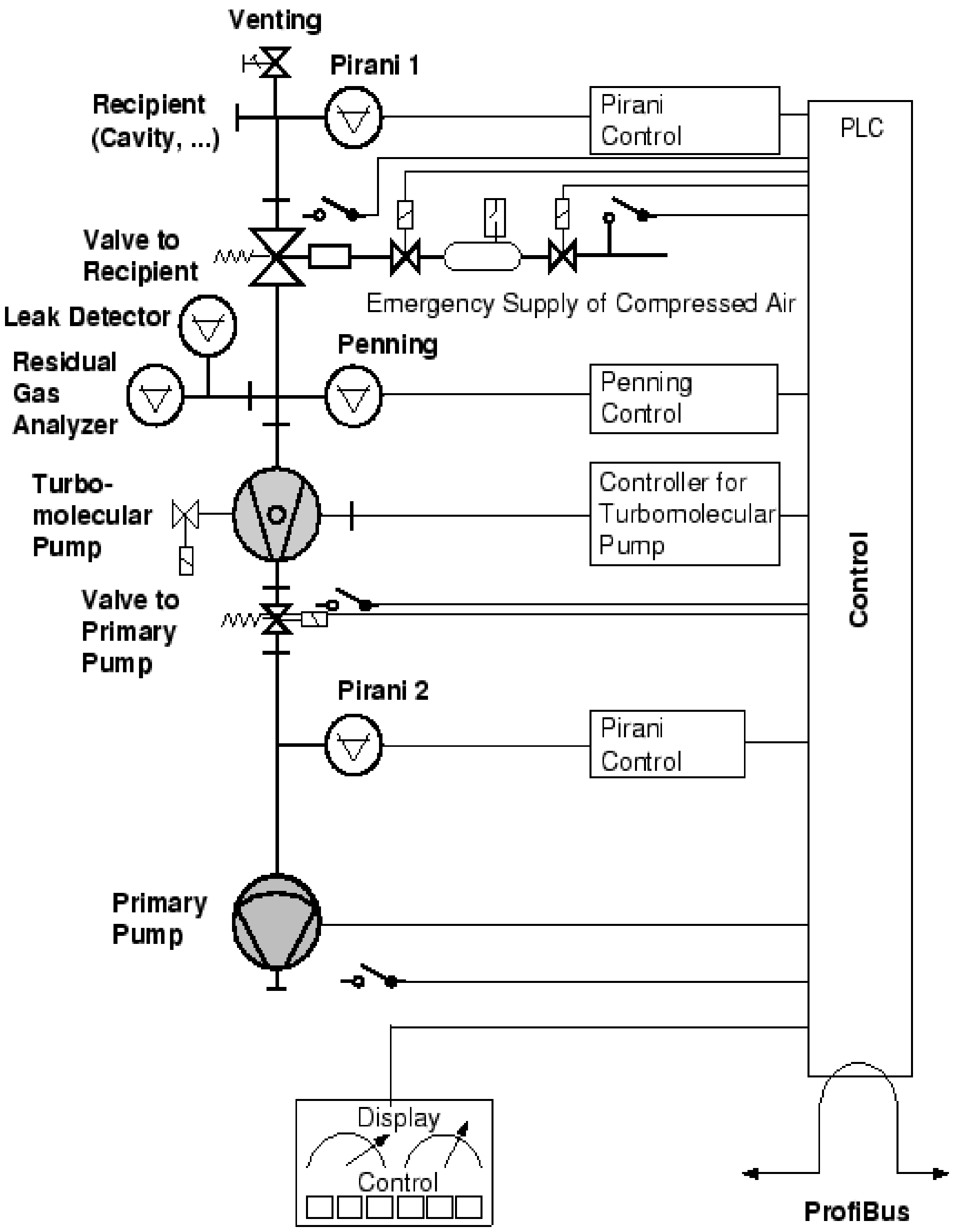
without contaminating them with hydrocarbon and particles.

System verified with

- RGA
- Particle counting

Then qualification done using an EXFEL Reference Cavitity (RCV).

Pumping systems, leak check and venting

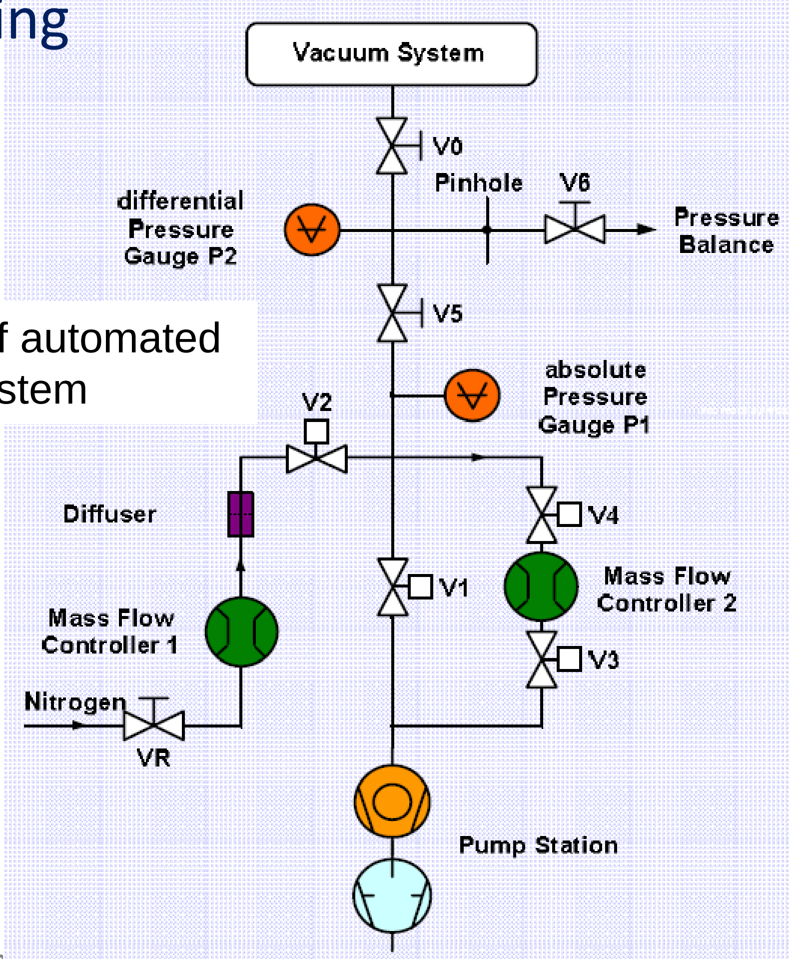


First generation DESY SPSV

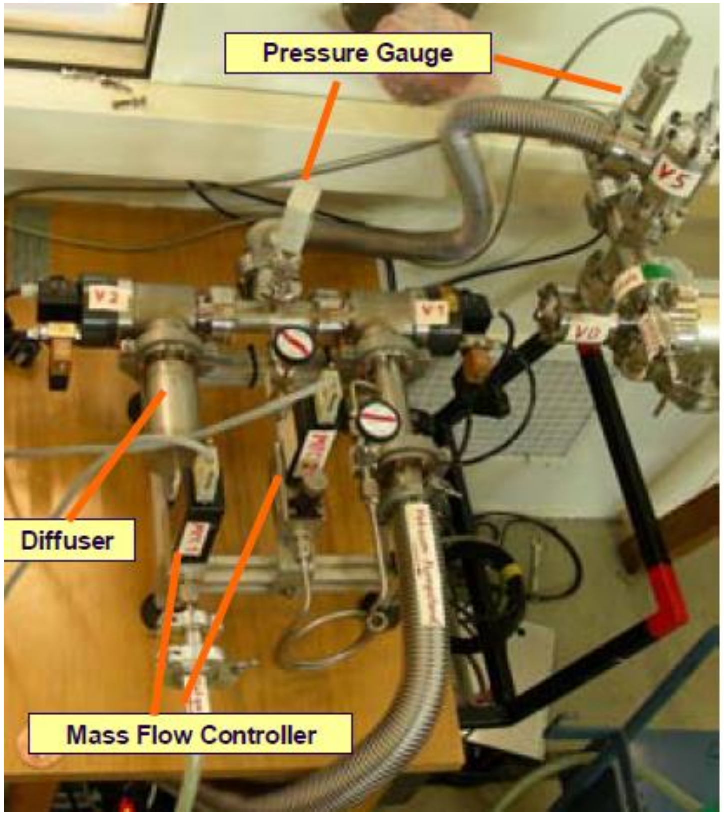
- leak check + venting (cleanroom cl.10)
  - oil-free pump stations with leak check and residual gas analyzer
  - laminar venting with pure, particle filtered N_2 or Ar

SPSV: slow pumping slow venting (ctd.)

- Set-up with diffuser, mass flow controller and precise pressure gauge successfully developed and tested since 2007
- Reduced process time for venting, increased (safe) process time for pumping

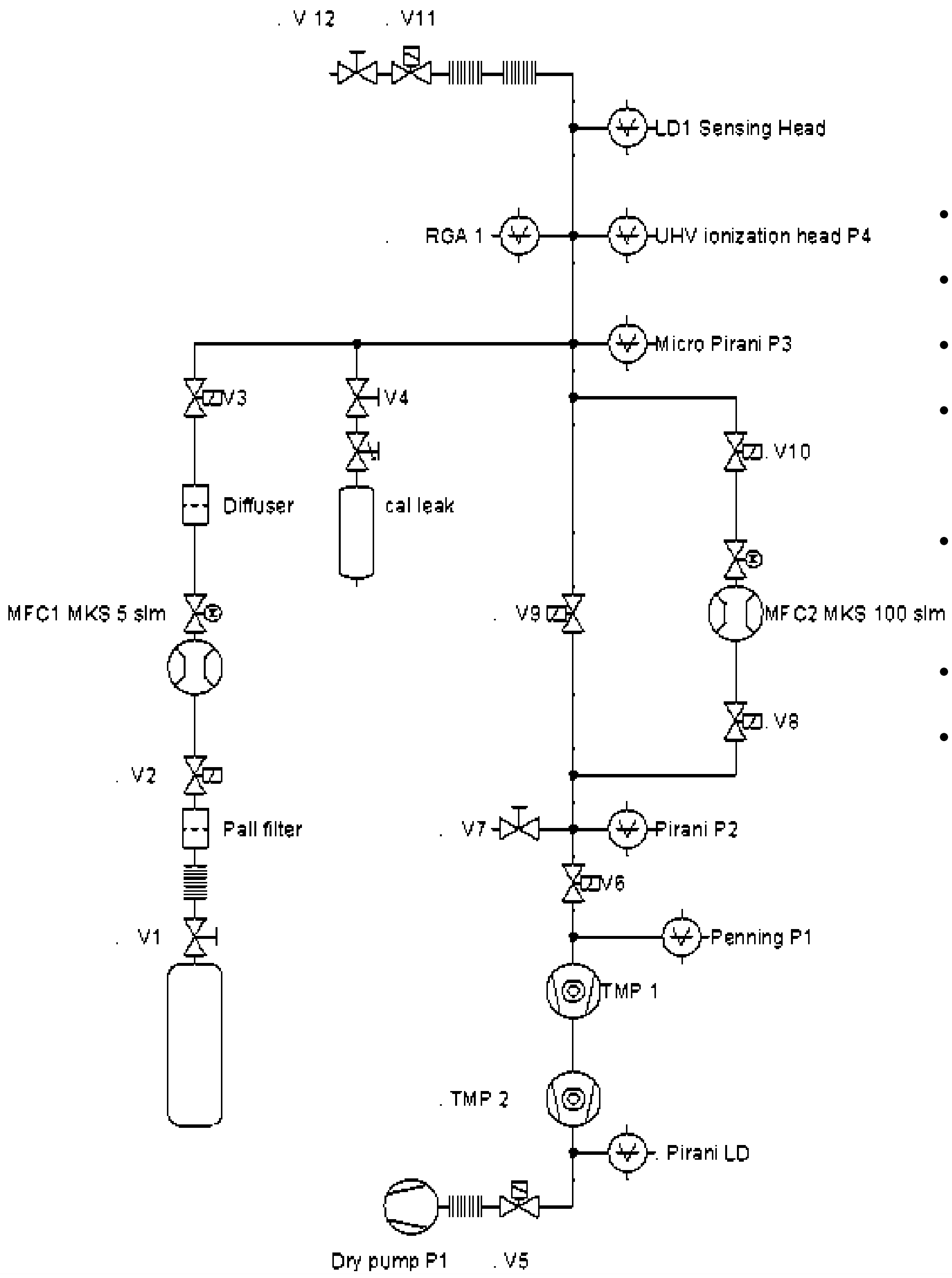


Scheme of automated venting system



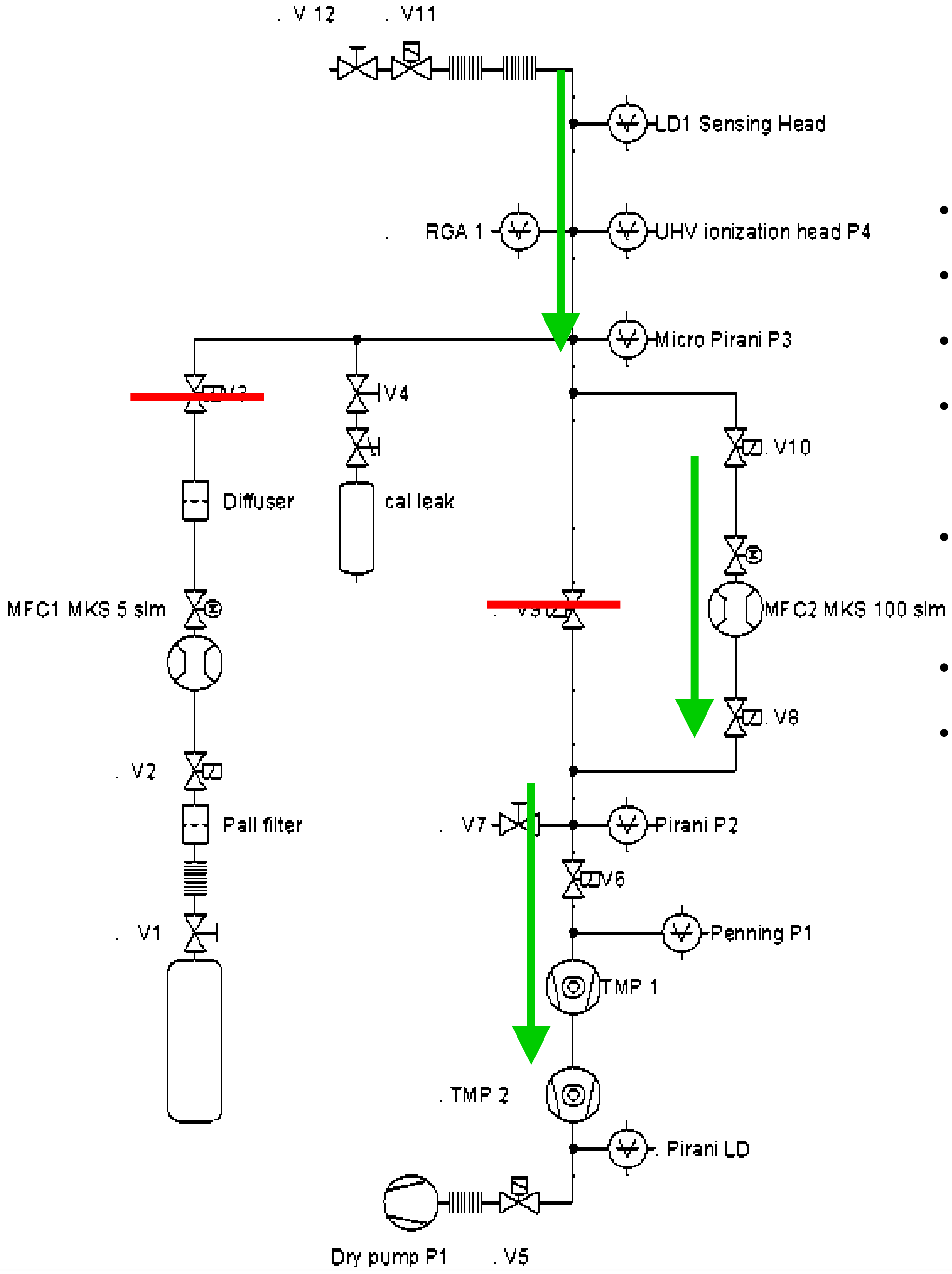
Test set-up of automated venting system

INFN SPSV



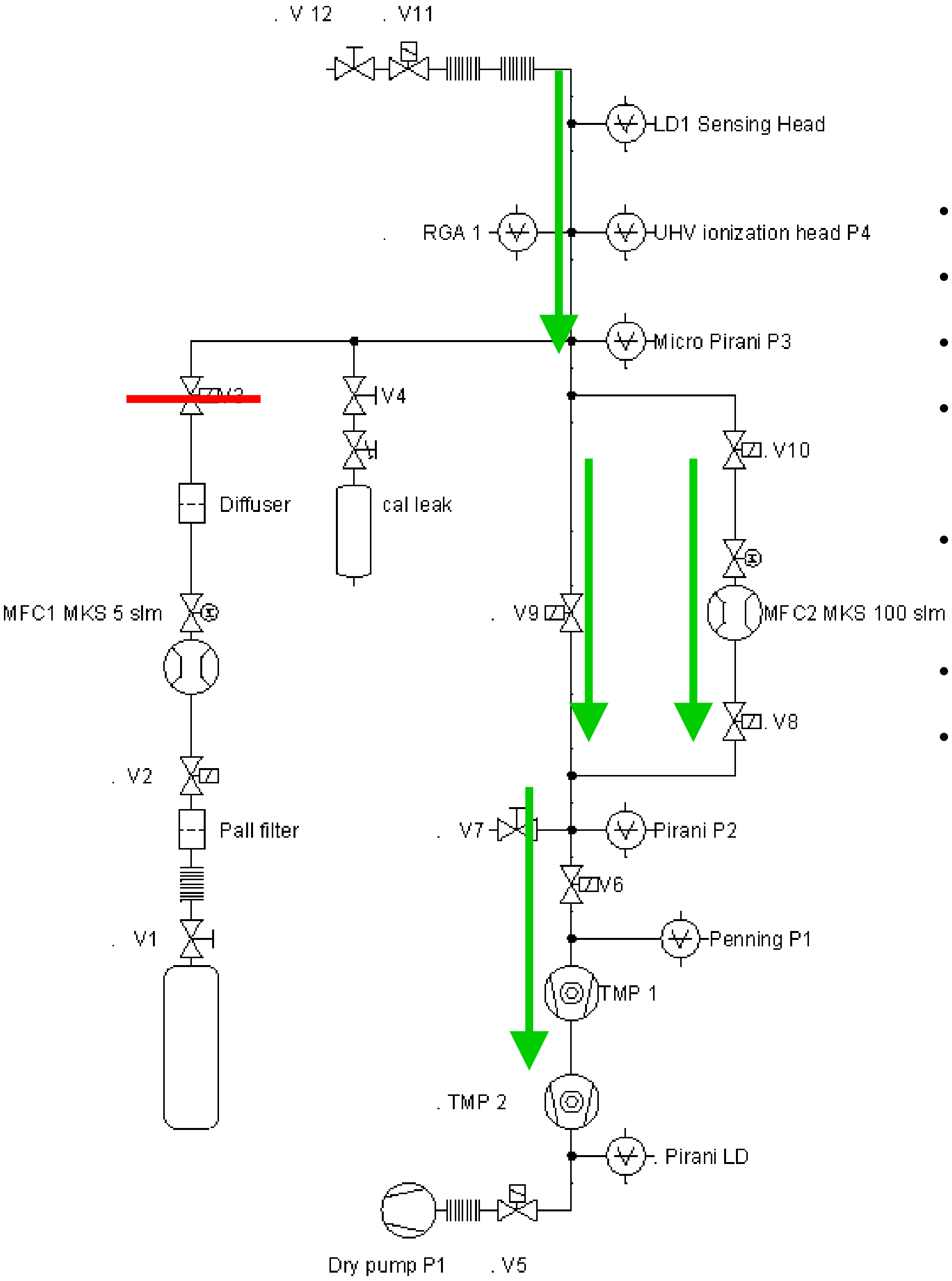
- Dry pump 1: scroll pump, Triscroll 600
- TMP1: Pfeiffer TMP
- TMP2: TMP of the LDS1000 Oerlikon LD
- LD1: LDS1000 Oerlikon Inficon sensing head
- V6, V8, V9, V10, V3: Varian Viton seal on the bonnet, metal seal on the body
- MFC1: MKS, 5 slm flow controller
- MFC2: MKS, 100 slm flow controller

SPSV: pump down fm atmospheric pressure



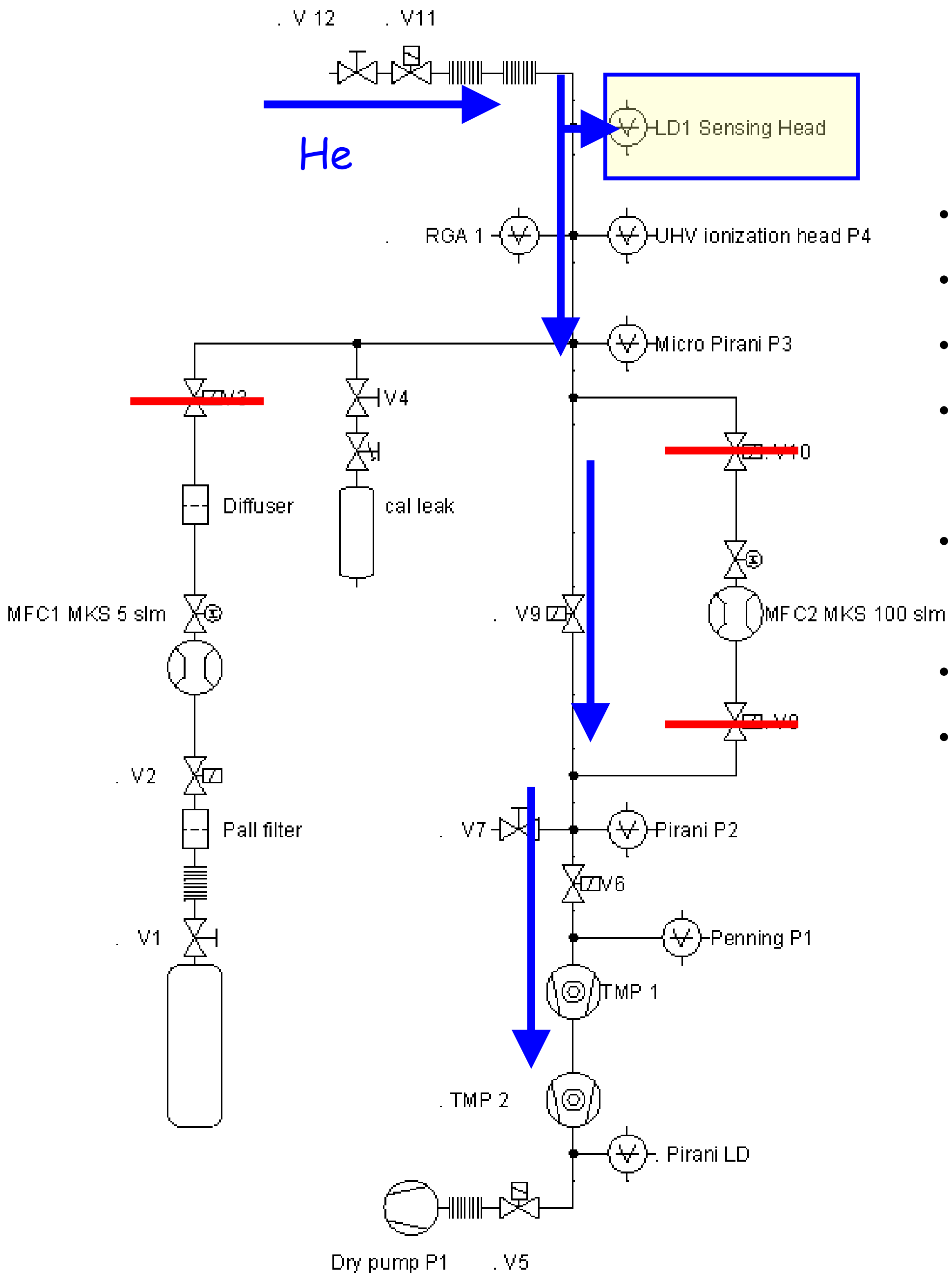
- Dry pump 1: scroll pump, Triscroll 600
- TMP1: Pfeiffer TMP
- TMP2: TMP of the LDS1000 Oerlikon LD
- LD1: LDS1000 Oerlikon Inficon sensing head
- V6, V8, V9, V10, V3: Varian Viton seal on the bonnet, metal seal on the body
- MFC1: MKS, 5 slm flow controller
- MFC2: MKS, 100 slm flow controller

SPSV: pumpdown at p< 1 mbar



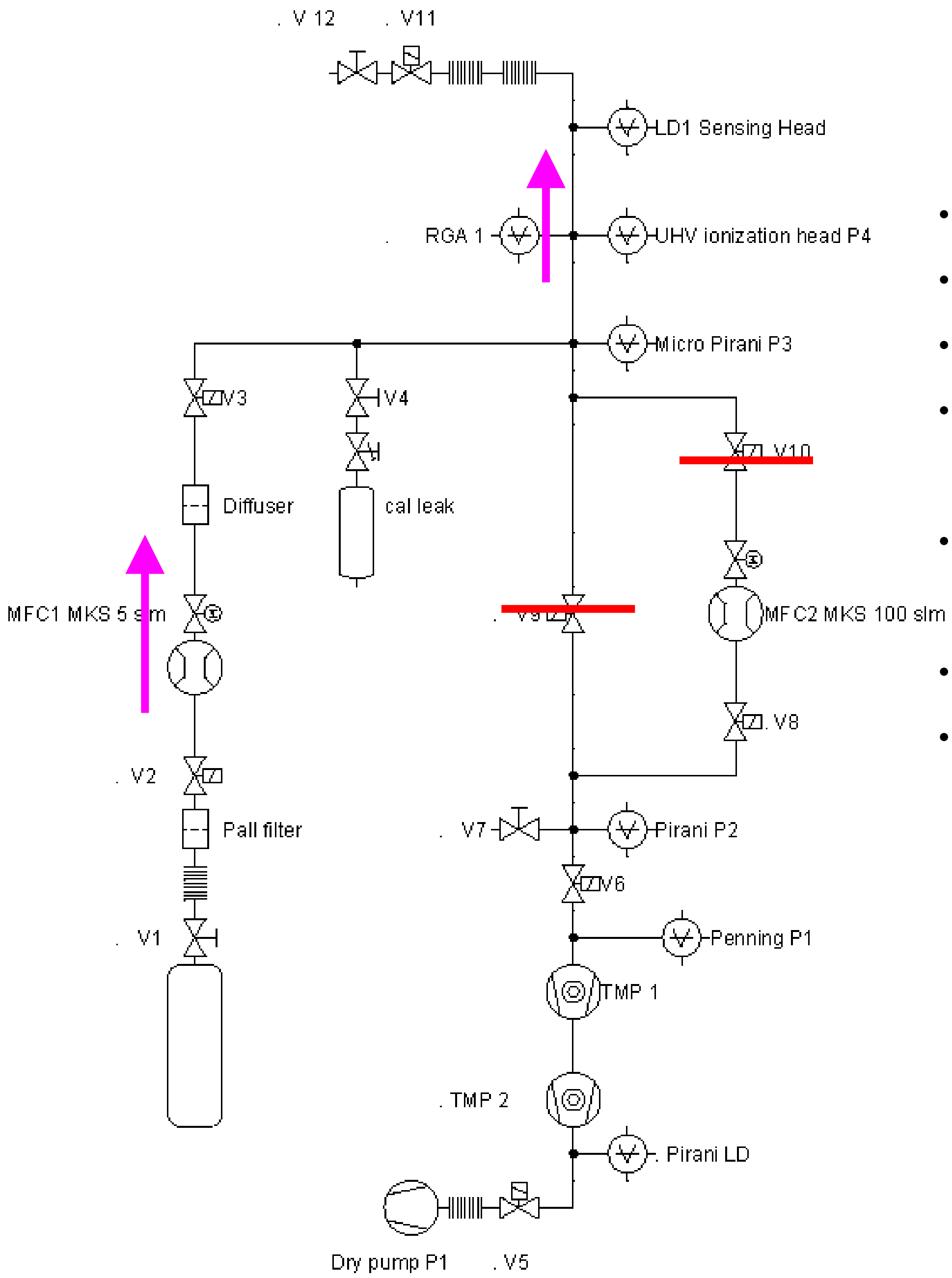
- Dry pump 1: scroll pump, Triscroll 600
- TMP1: Pfeiffer TMP
- TMP2: TMP of the LDS1000 Oerlikon LD
- LD1: LDS1000 Oerlikon Inficon sensing head
- V6, V8, V9, V10, V3: Varian Viton seal on the bonnet, metal seal on the body
- MFC1: MKS, 5 slm flow controller
- MFC2: MKS, 100 slm flow controller

SPSV: Leak check



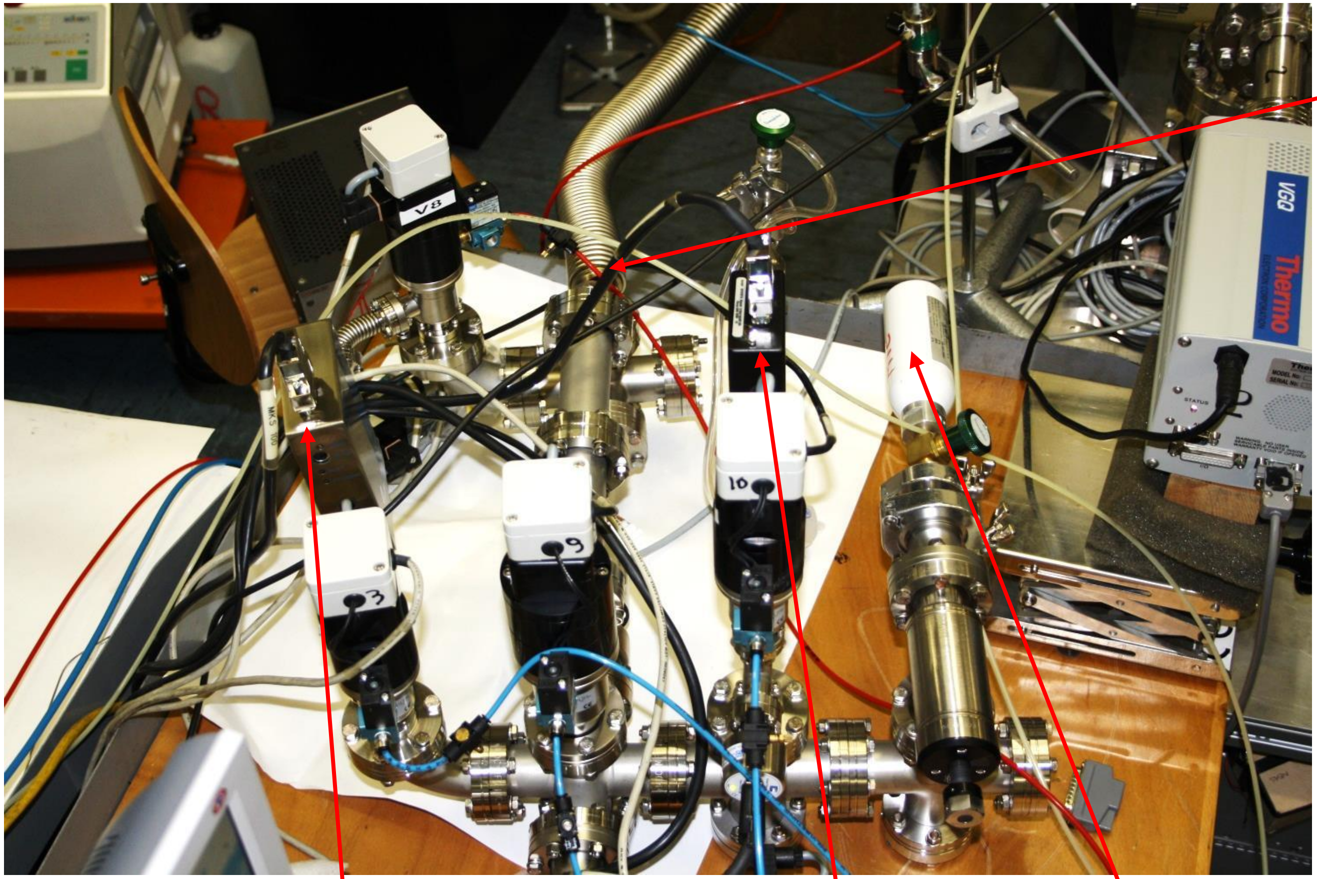
- Dry pump 1: scroll pump, Triscroll 600
- TMP1: Pfeiffer TMP
- TMP2: TMP of the LDS1000 Oerlikon LD
- LD1: LDS1000 Oerlikon Inficon sensing head
- V6, V8, V9, V10, V3: Varian Viton seal on the bonnet, metal seal on the body
- MFC1: MKS, 5 slm flow controller
- MFC2: MKS, 100 slm flow controller

SPSV: slow venting



- 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

SPSV: INFN system



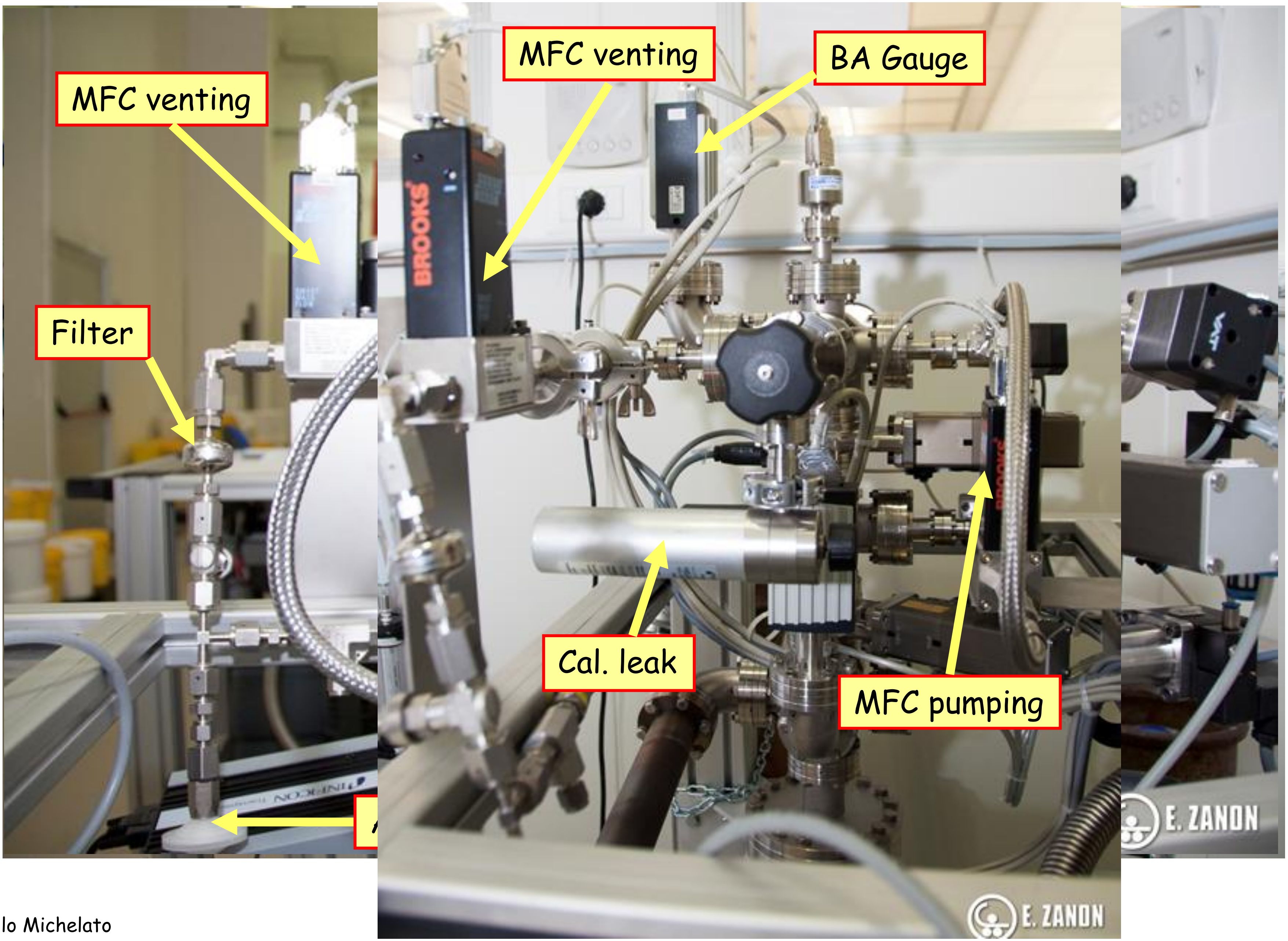
Connection to the TMP

Pumping line: controller and valves

Cal. leak

Venting line: controller and valves

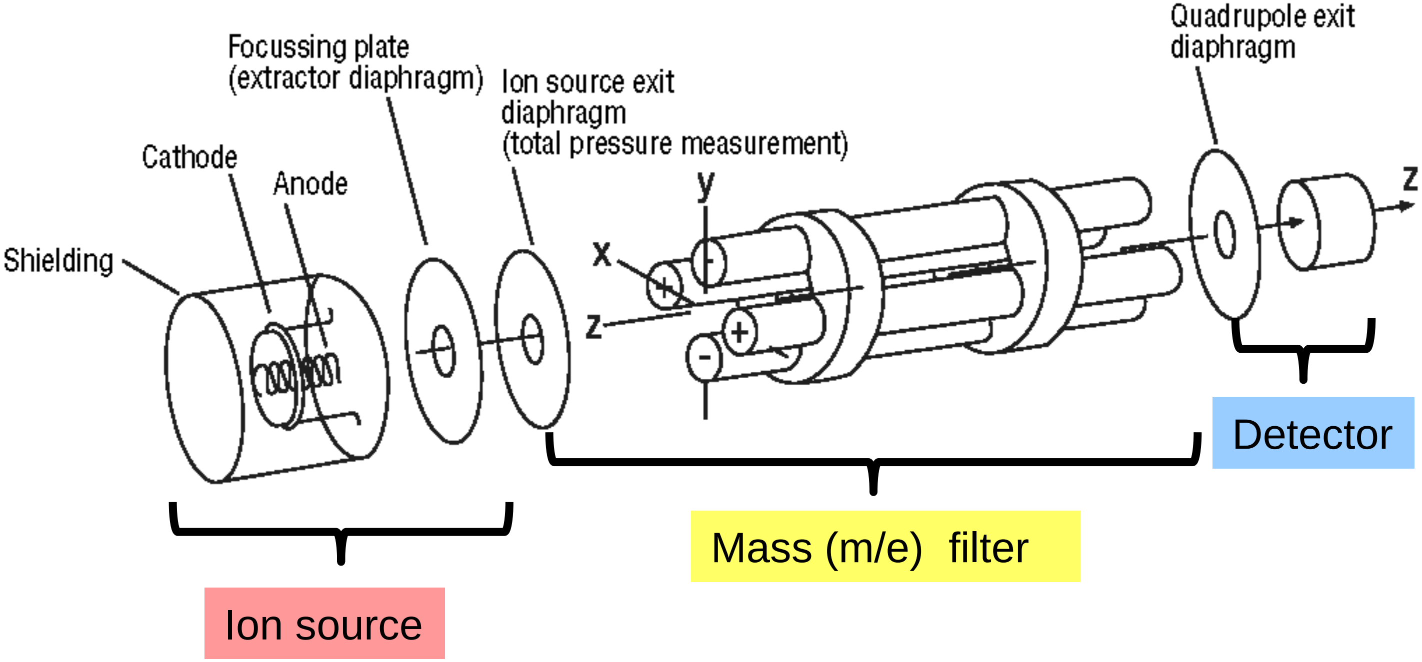
SPSV: EZ system



Residual Gas Analysis

Partial pressure measurement system used for:

- **Vacuum quality check**
- **Partial pressure** evaluation of various gases: e.g. for **discriminate water** desorption from **leaks and outgas**
- Impurities and contaminants evaluation
- Evaluation of **Hydrocarbon** content
- **Leak check**

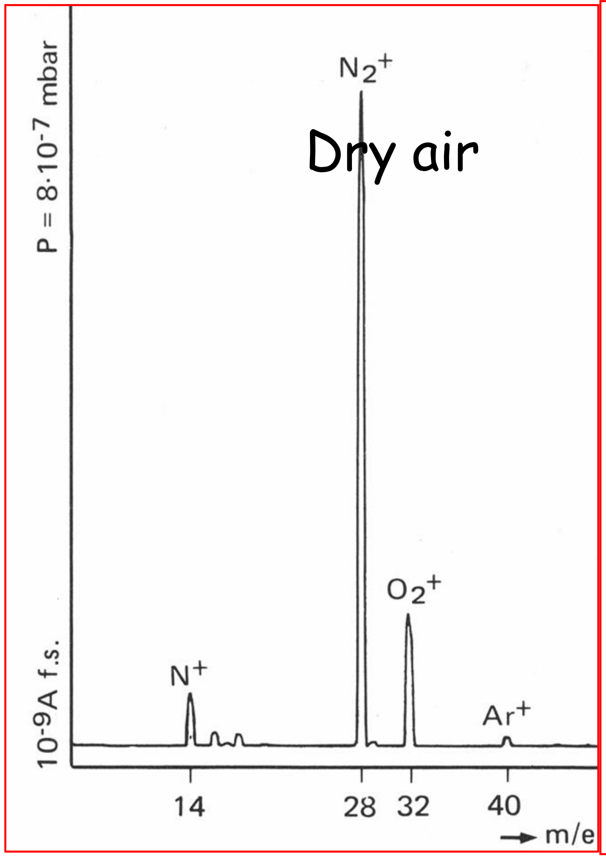


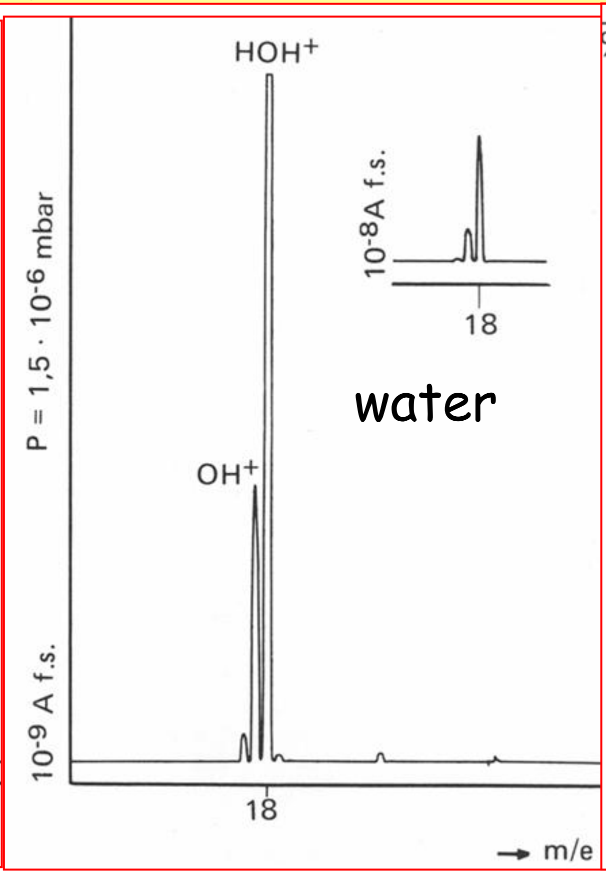
Detector

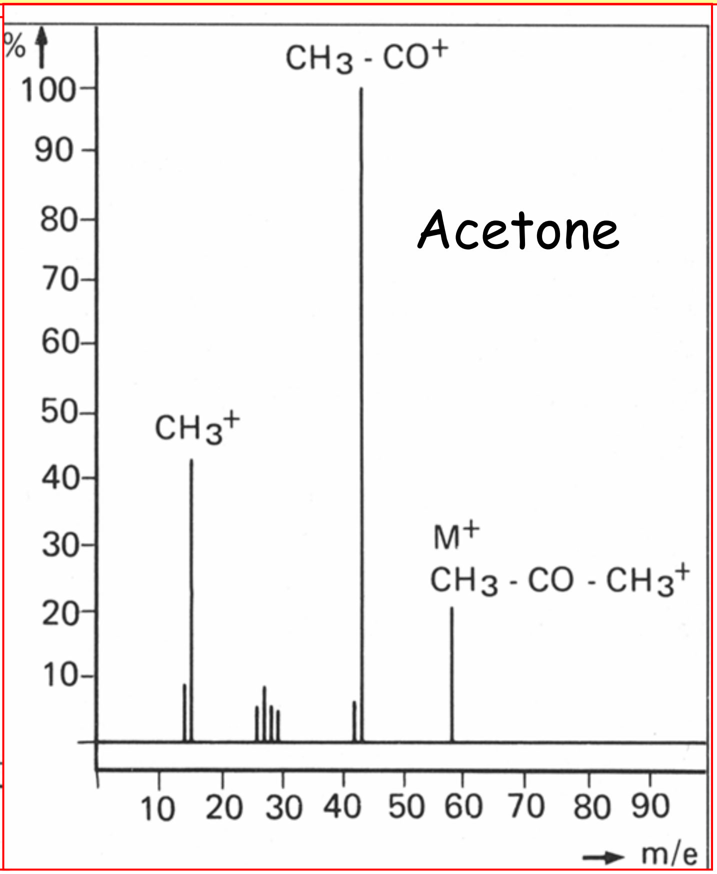
Mass (m/e) filter

Ion source

Some RGA: dry air, water, aceton





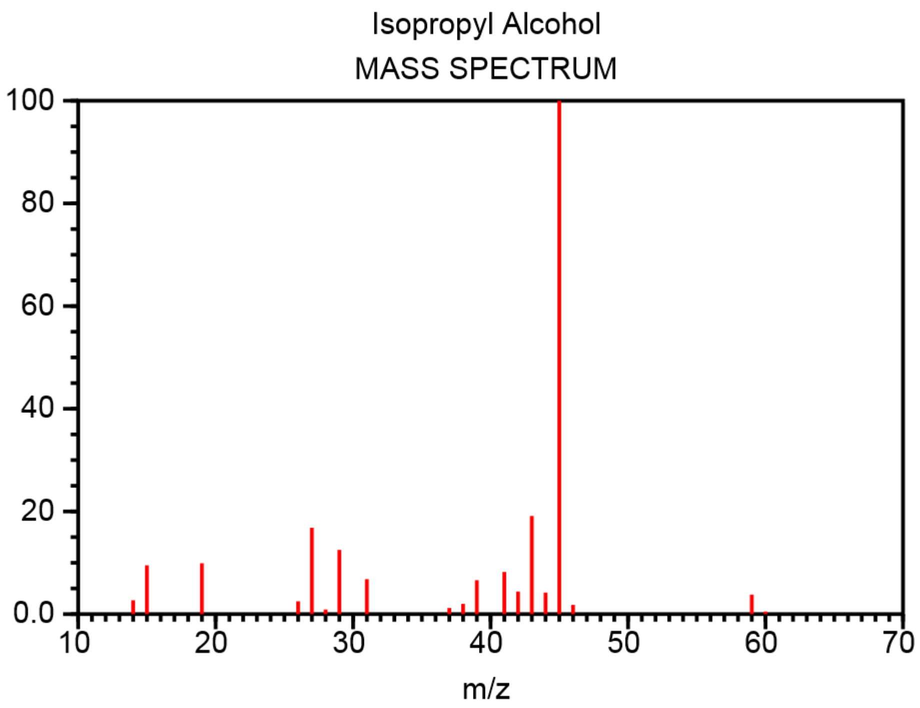


	m/e
N^+	14
N_2^+	28
O^+	16
O_2^+	32
Ar^+	40

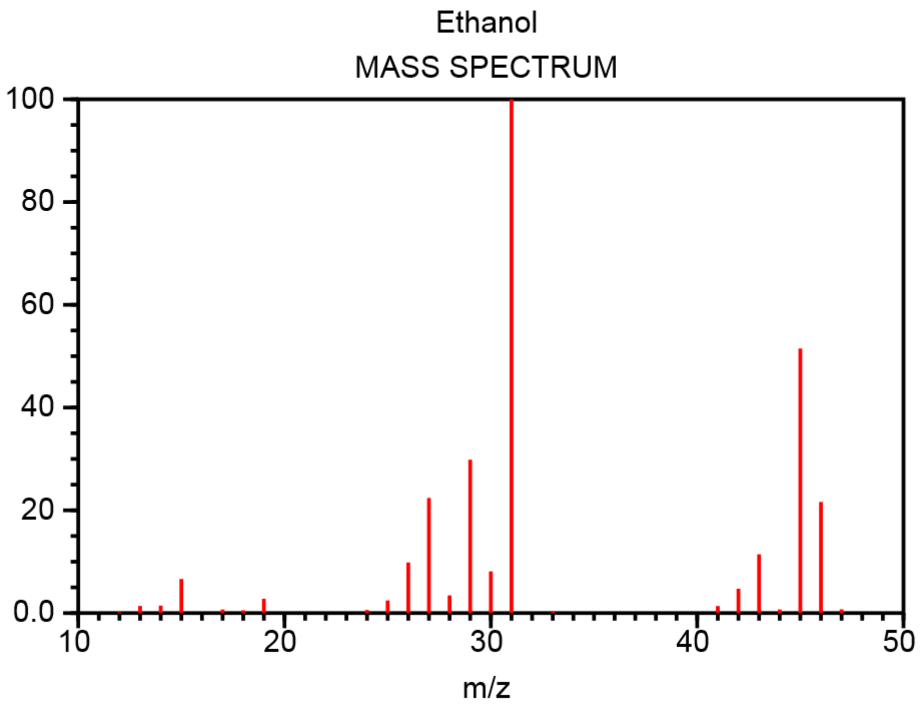
	m/e
O^+	16
OH^+	17
HOH^+	18

	m/e
CH_3^+	15
CH_3CO^+	43
$CH_3COCH_3^+$	58

RGA: isopropanol, ethanol



Isopropyl Alcohol



Ethanol

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

RGA spectra interpretation

F = Fragment
P = Parent Ion
DI = Doubly Ionized

AMU NO	CHEMICAL SYMBOL	SOURCES	
1.	H	Water F or Hydrogen F	
2.	H_2, D	Hydrogen, Deuterium (H^2)	
3.	HD, H^3	Hydrogen - Deuterium, Tritium (H^3)	
4.	He	Helium	
5.	–	No known elements	
6.	C^{++}	Doubly Ionized C^{12}	Rare
7.	N^{++}	DI N^{14}	Rare
8.	O^{++}	DI O^{16}	Rare
9.	–	No known elements	
10.	Ne + +	DI Ne^{20}	
11.	Ne + +	DI Ne^{22}	
12.	C	Carbon, Carbon Monoxide F, Carbon Dioxide F	
13.	CH, C^{13}	Methane F, Carbon isotope	
14.	N, CH_2	Nitrogen, Methane F or Note 1	
15.	CH_3	Methane F, Carbon Isotope	
16.	O, CH_4, NH_2	Oxygen or Carbon Monoxide F, Methane P. Ammonia F	
17.	OH. NH_3	Water F, Ammonia P	
18.	H_2O	Water P	
19.	F	Fluorine or Freon F	
20.	Ar + +, Ne, HF	DI Argon, Neon, ,.Ne, Hydrofluoric acid	
21.			
22.	Ne_{22}	Neon Isotope	
23.			
24.	C_2	See Note 1	
25.	C_2H	See Note 1	

RGA spectra interpretation

26.	C_2H_2, CN	See Note 1, Cyanide F
27.	C_2H_3, Al, HCN	See Note 1, Aluminum, Hydrogen Cyanide
28.	N_2, CO, C_2H_4, Si	Nitrogen, Carbon Monoxide, Ethylene P, Silicon
29.	CH_3C_2	Ethane F or Ethanol F or Isopropyl alcohol F
30.	C_2H_6, NO	Ethane P, Nitric Oxide
31.	P, CH_2OH, CF	Phosphorus, Methanol F, Ethanol F,
32.	O_2, CH_3OH, S	Oxygen, Methanol P, Sulfur
33.	HS	Hydrogen Sulfide F
34.	H_2S, S^{34}	Hydrogen Sulfide F’, Sulfur isotope
35.	Cl	Chlorine isotope, See Note 2
36.	HCl, Ar^{36}	Hydrochloric acid, Argon isotope
37.	Cl^{37}	Chlorine isotope, See Note 2
38.	HCl^{38}	Hydrochloric acid or See Note 2
39.	C_3H_3	See Note 3
40.	Ar, C_3H_4	Argon, See Note 1
41.	C_3H_5	See Note 1
42.	C H_6	See Note 1
43.	C_3H_7, CH_3CO	Note 1, Acetone F or Methyl Ethyl Ketone F
44.	CO_2, C_3H_8	Carbon dioxide, See Note 3
45.	CH_3CH_2O	Ethanol F or Isopropyl alcohol F
46.	CH_3CH_2OH	Ethanol P
47.	CCl^{35}	See Note 2
48.	$HCCl^{35}$, SO	See Note 2, sulfur Dioxide F
49.	CCl_{37}, SiOH	See Note 2, pump oil F
50.	CCl^{37}, CF_2, C_4H_2	See Note 2, Freon F, Note 3

* NOTE I

Fragments of several hydrocarbons, such as mechanical pump oil, diffusion pump oil, vacuum grease, cutting oil, and organic solvents.

NOTE 2

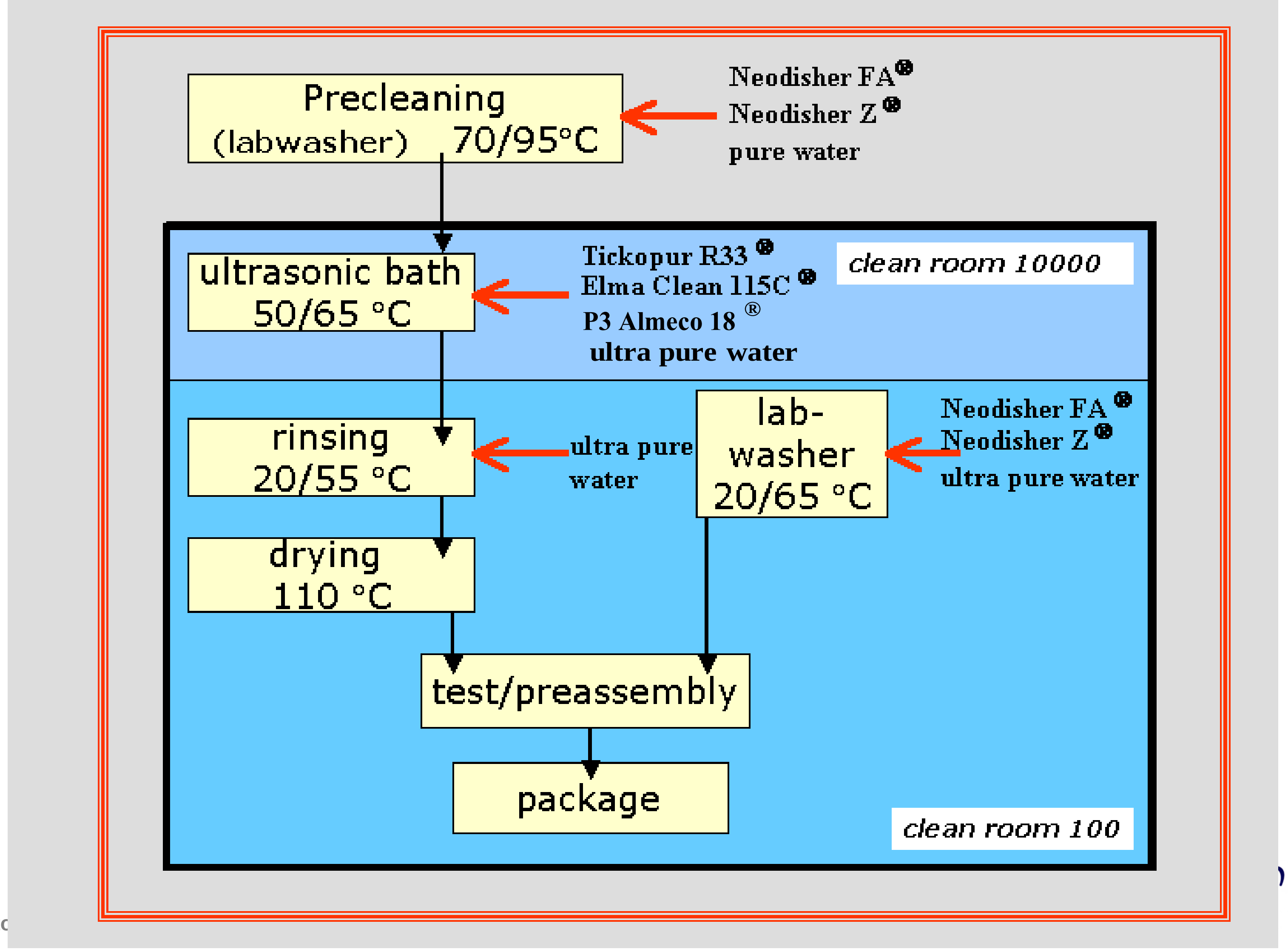
Fragments of several, chlorinated hydrocarbons, such as carbon tetrachloride, trichloroethylene and many Freons.

§ NOTE 3

Fragments from both straight chain hydrocarbons and benzene ring hydrocarbons.

Vacuum: Cleaning

- Cleaning of all vacuum components in the cavity environment to the same level as the cavity itself



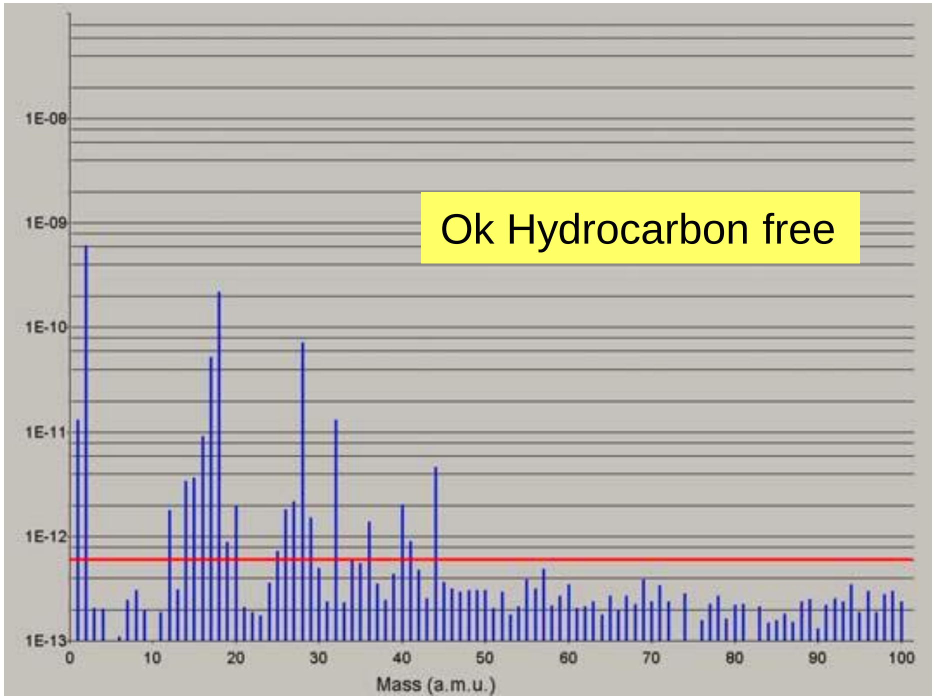
Vacuum: Cleaning

- DESY: Separate cleanroom for cleaning of vacuum components

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).

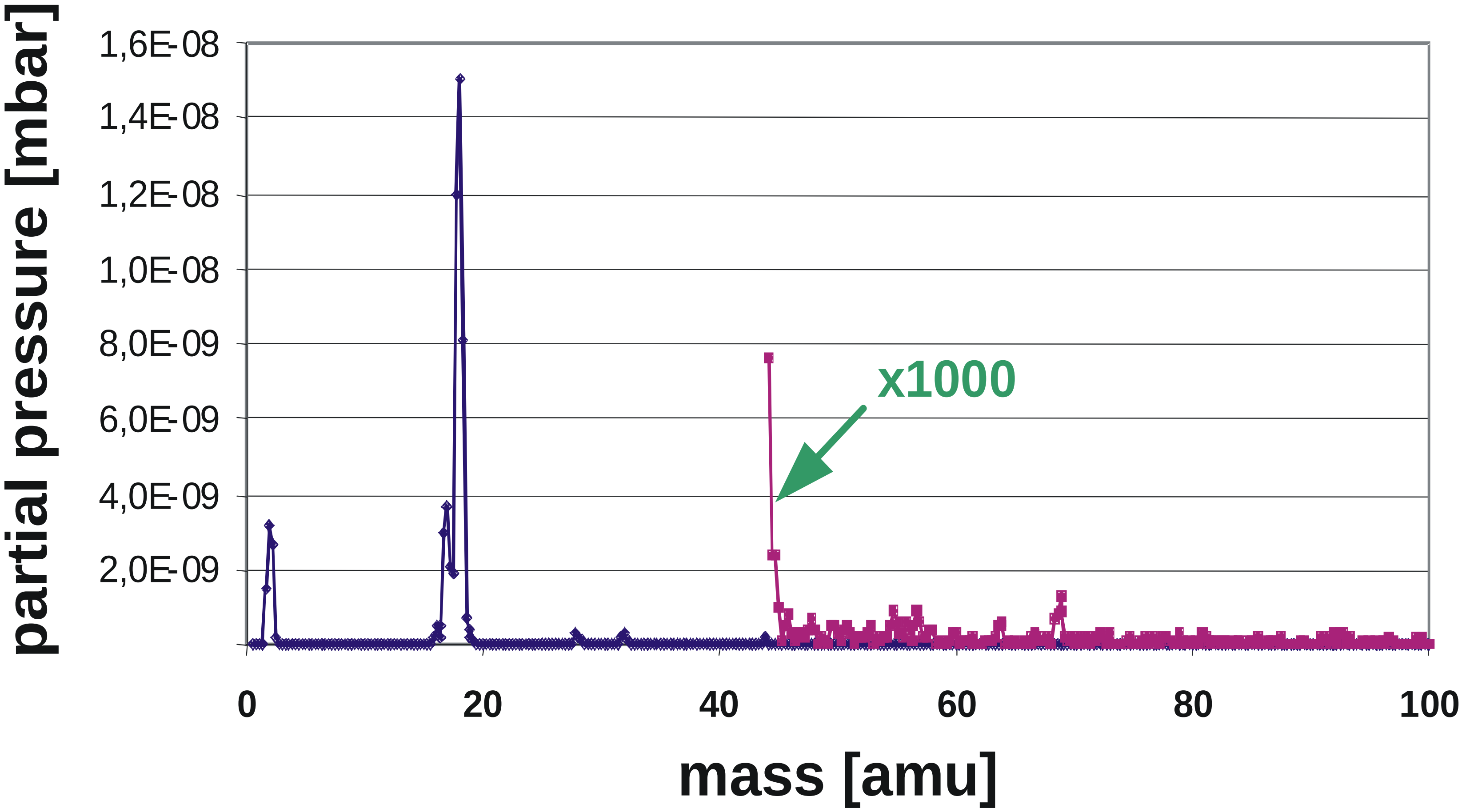


Ok Hydrocarbon free

NOK Hydrocarbon contaminated

Vacuum: Residual gas analysis

- **Residual gas analysis** (RGA): Check for hydrocarbon contamination
- Example: RGA of a vacuum chamber after cleaning

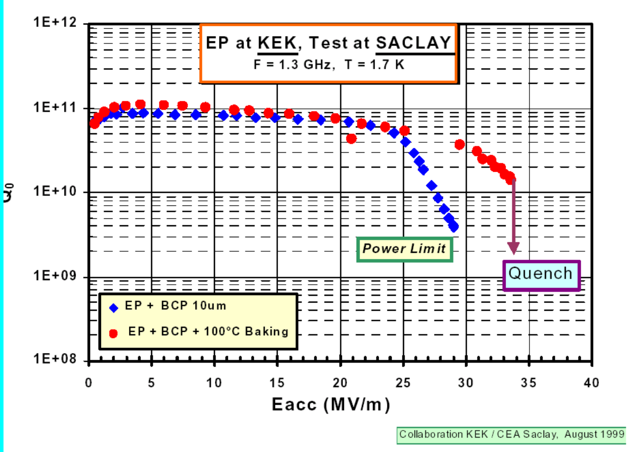


Final rinsing, after internal BCP

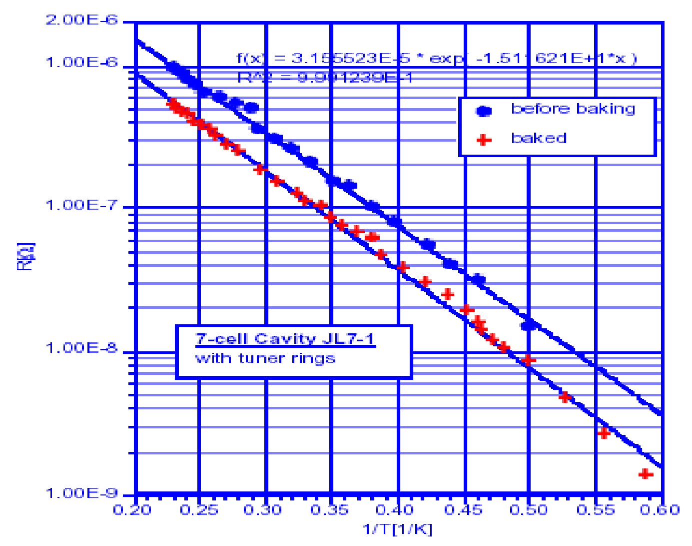
Resistivity rinsing after BCP

Appendix: 120 °C bake

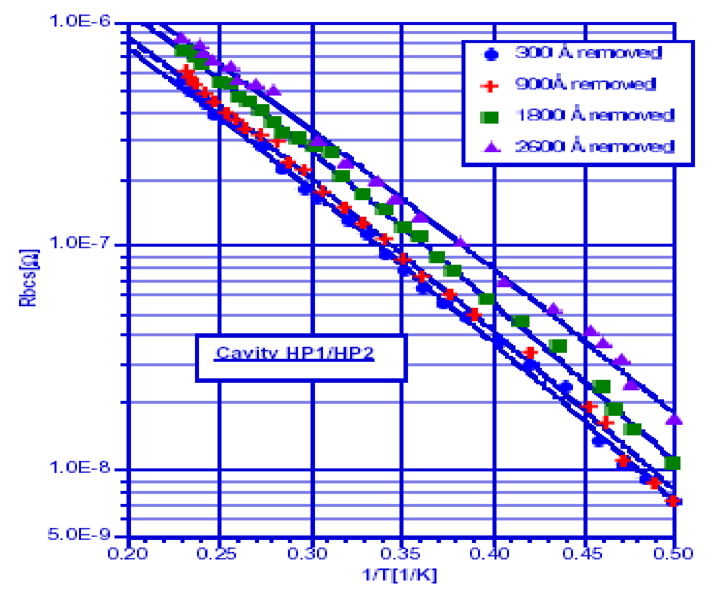
Nb baking



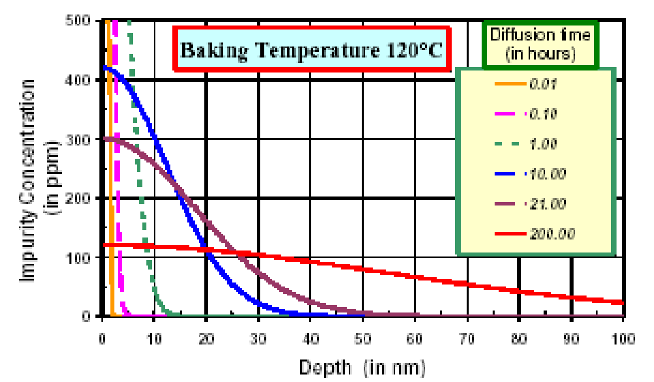
Q0 vs Eacc for the electropolished cavity before and after "in - situ" baking (Safa)



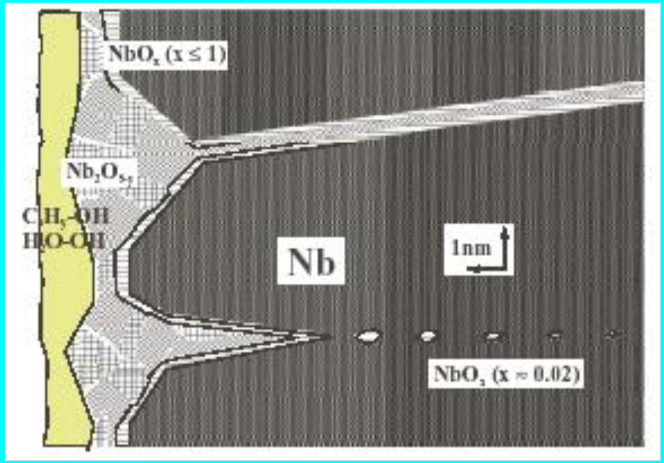
Temperature dependence of the BCS surface resistance before and after "in-situ" baking at 145° C (P. Kneisel).



Temperature dependence of the BCS surface resistance after several steps of material removal by oxipolishing (P. Kneisel)

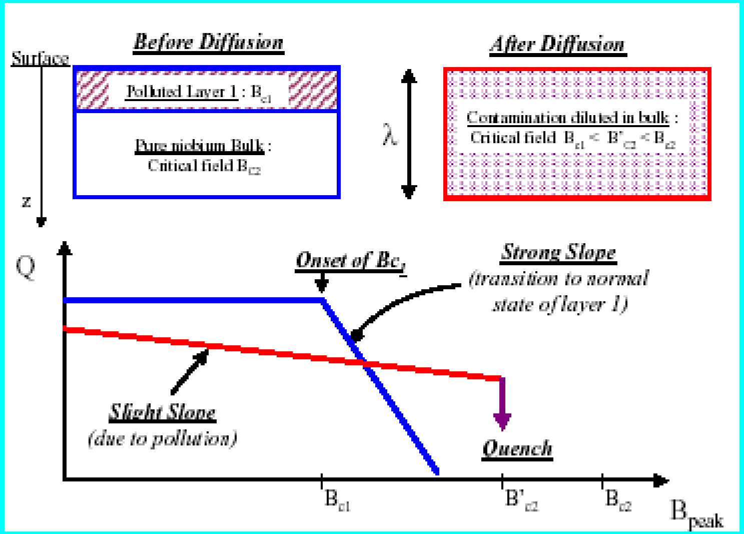


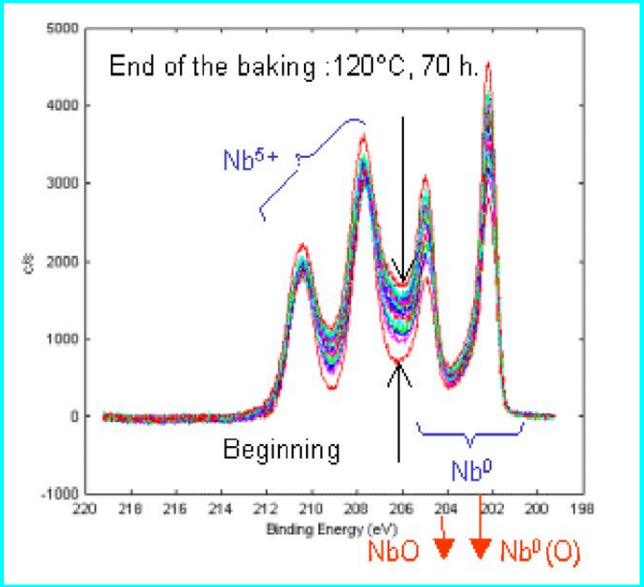
Diffusion of oxygen in the Nb.
After 200 hours at 120°C, oxygen will almost uniformly diffuse inside the material up to a depth > 50 nm (H.Safa).



Nb surface (Halbritter).

- layer of adsorbates (water, hydrocarbons and other gases)
- Dielectric oxide layer Nb_2O_5 (till ca.300°C)
- Layer of metallic oxide NbO (till ca.700°C)
- Sub oxides NbOx
- Nb bulk with impurity atoms





Nb spectra obtained by X-ray photoemission spectroscopy during a 3 days baking at 120°C. The 2 peaks at the right side correspond to Nb° , the 2 peaks at the left side correspond to Nb5+; the apparition of a 3rd component (Nb4+) can be inferred from the enlargement of the middle of the spectra (Antoine).

Main tendency:

Degradation of Nb pentoxide to NbO_2, dilution of oxygen in bulk Nb due to thermal diffusion. The details not yet well understood

One of possible explanations : After baking, the pollution is diluted up to a depth of the order of the London penetration (H.Safa).