

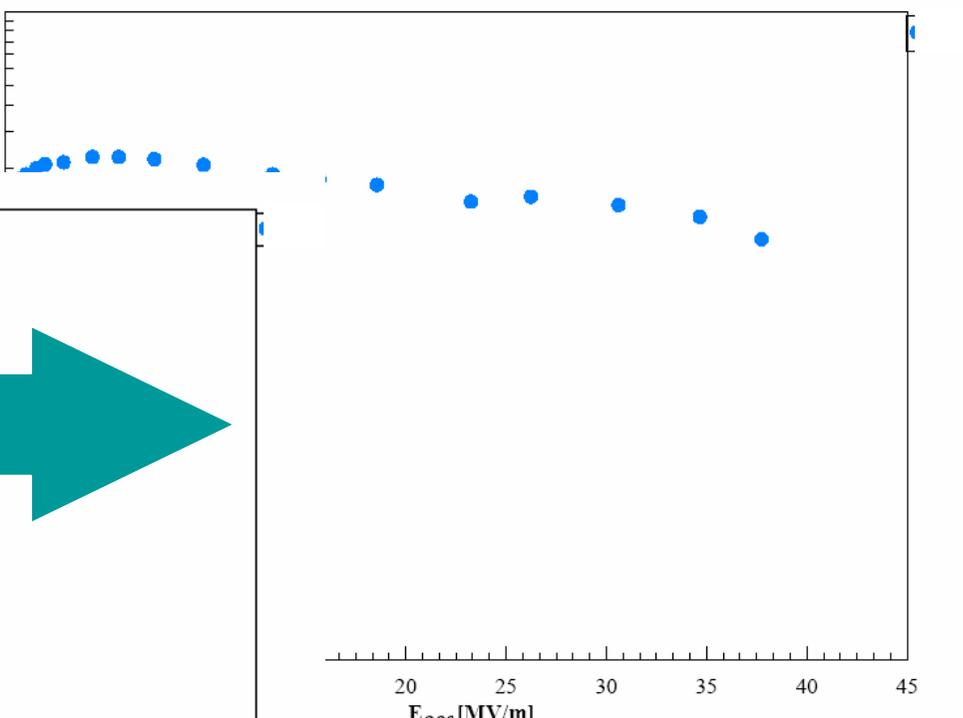
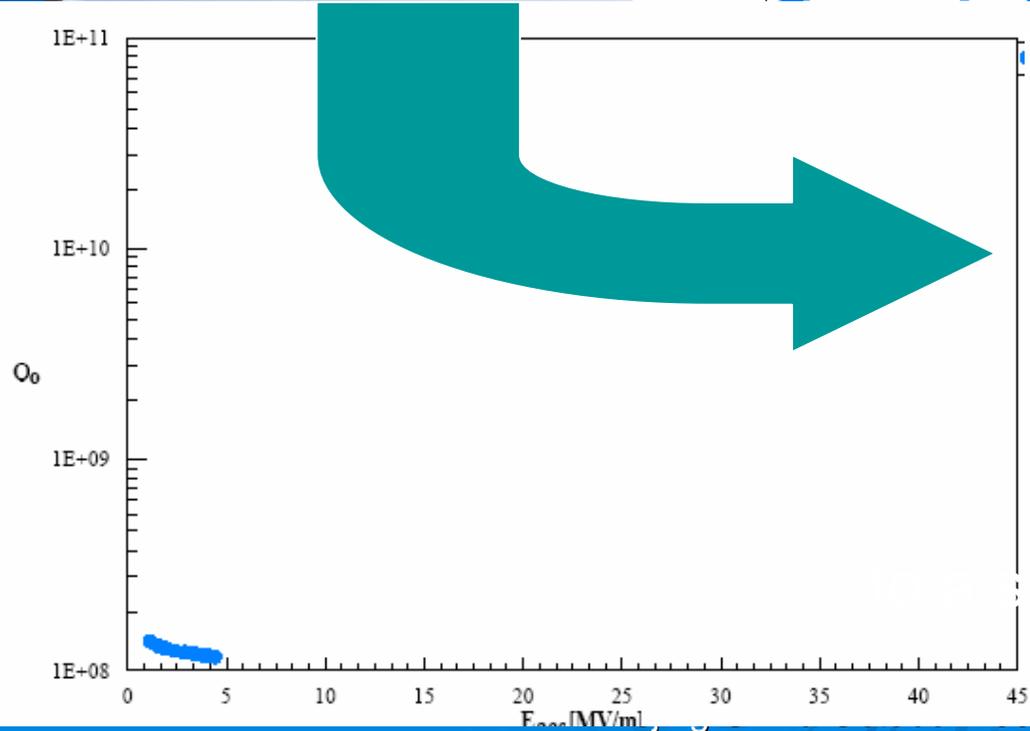
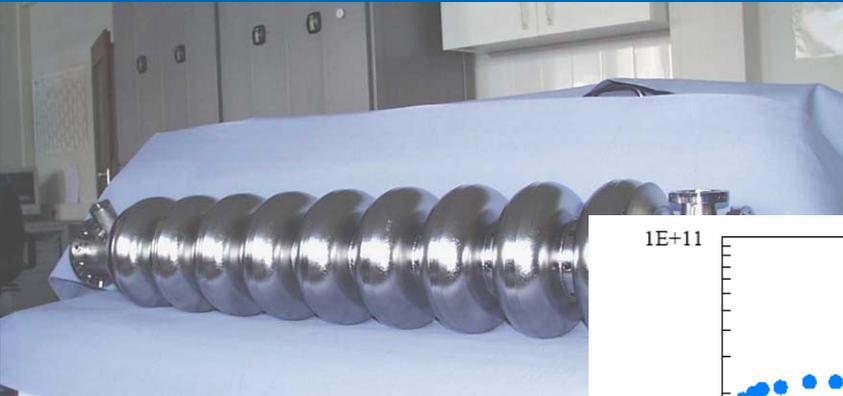
# Tutorial on Cavity Preparation SRF 2007 Workshop Beijing

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# Subtitle how to come from a Niobium Cavity



super conducting resonator

Some background information to understand  
what is the difference ?

1) Surface preparation methods

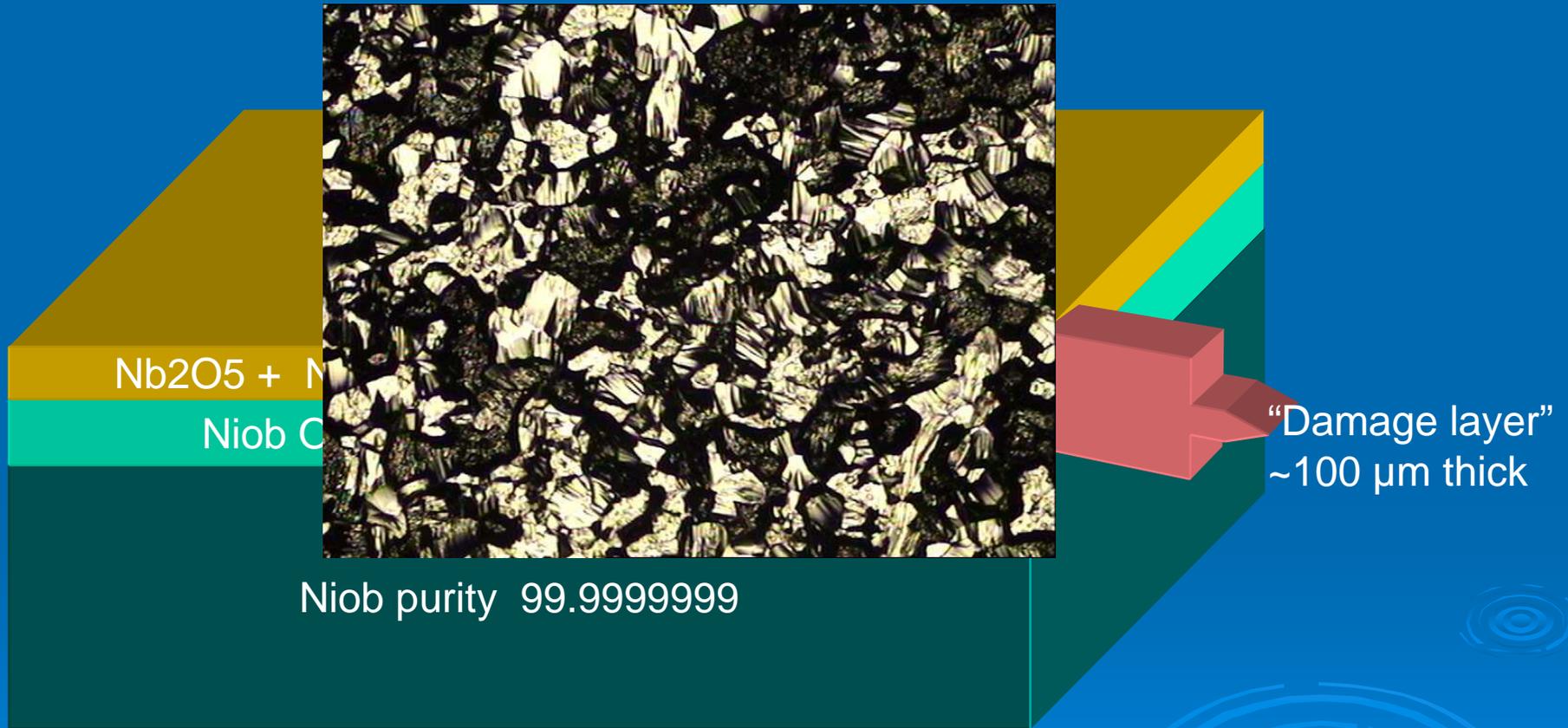
2) Infrastructure for Cavity preparation

3) Handling and Know how on cavity preparation

# 1) Surface preparation methods

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# Virtual micro cut of a Niobium sheet



# Removal of “damage” layer

## Method's

Mechanically by

Grinding  
barrel polishing (tumbling)

Chemically by

chemical etching ( buffered chemical polishing [BCP])

Chemical polishing ( electro polishing [ EP])

+ + different new solution under development

For instance

*see EU JARY1 program WP 5*

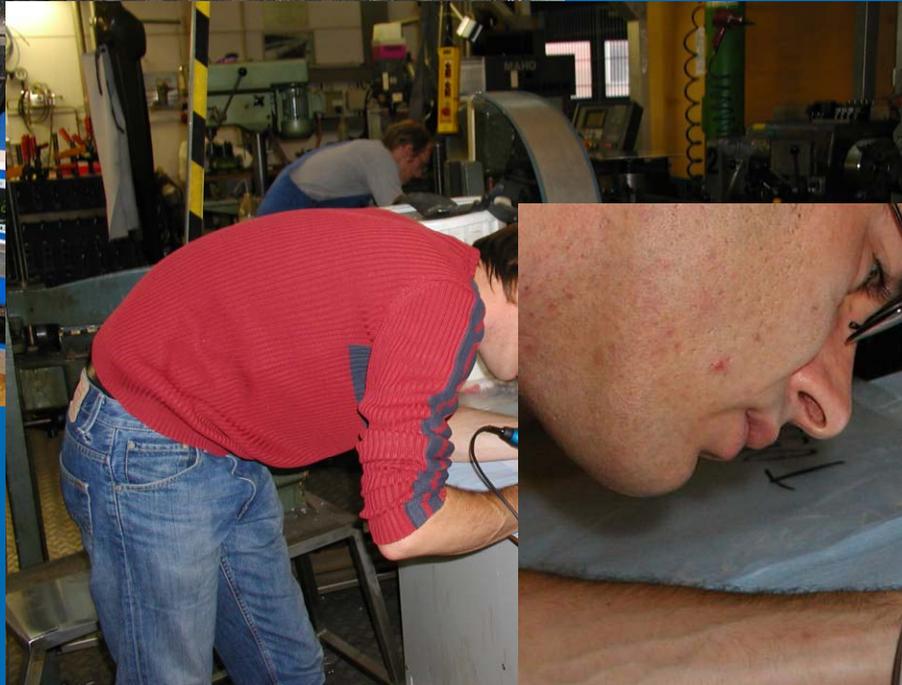
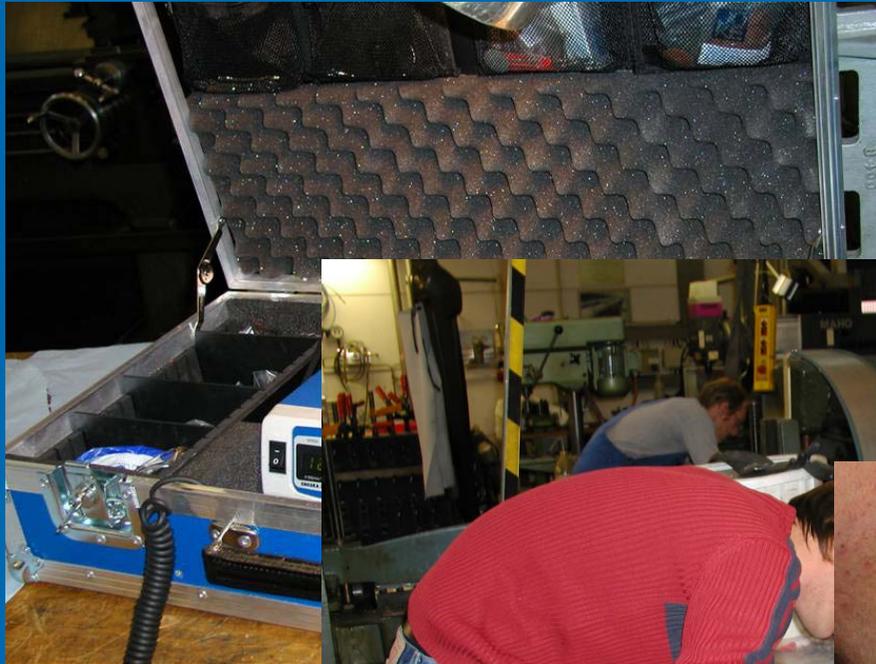
*Accel / Poligrad*

*Electro bright*

## Mechanical removal of damage layer

### Grinding

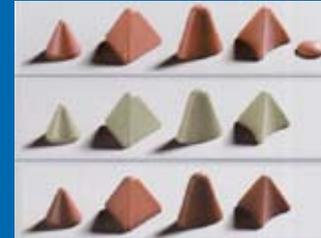
- + Simple handling
- + low cost standard mechanic
- + Mostly in use for removal of local defects non uniform abrasion !
- Abrasives need to be qualified on s.c. Cavities!!
- Remain of C; Si ; glue; scratch size
- Produces a new damage layer of about 40  $\mu\text{m}$  thickness!!!



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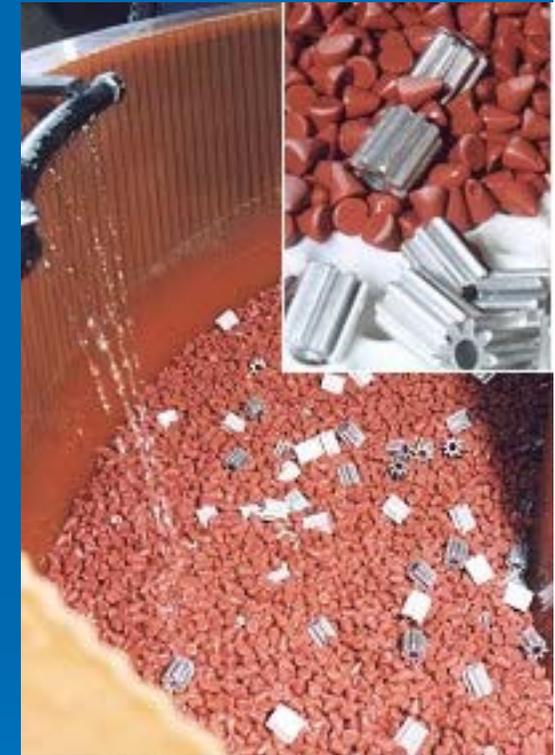
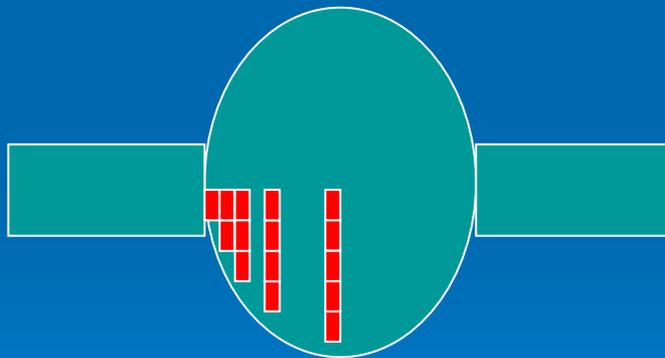
# Tumbling

Material : "Stones" made in different shape and material



Application: Global  
Effect: Smoothing 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)

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



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# Chemical removal of “damage” layer

Two types chemical removal are most commonly in use

## BCP

### **Buffered chemical polishing**

(Mixture of Hydrofluoric acid; Nitric acid and Phosphoric acid)

## EP

### **Electropolishing**

(Mixture of Hydrofluoric acid and Sulfuric acid)

Receipt:

BCP Acid

Mixed by volume from

1:1 HF(49%) /HNO<sub>3</sub>(70%)

to (1:1:2 HF(49%):HNO<sub>3</sub>(70%) :H<sub>3</sub>PO<sub>4</sub>85%)

removal rate

1:1 at 20C >20 μm/min Removal Rate

1:1:2 t 20 C 1μm/min

**Mixture is self exiting ! Spontaneous reaction with Nb!!**

EP Acid

Mixed by volume from

1:8 HF(45%) /H<sub>2</sub>SO<sub>4</sub> (96%)

to 1:10 (HF( 45%)/H<sub>2</sub>SO<sub>4</sub> (96%)

(+ H<sub>2</sub>O due to hygroscopic reaction of H<sub>2</sub>SO<sub>4</sub>!)

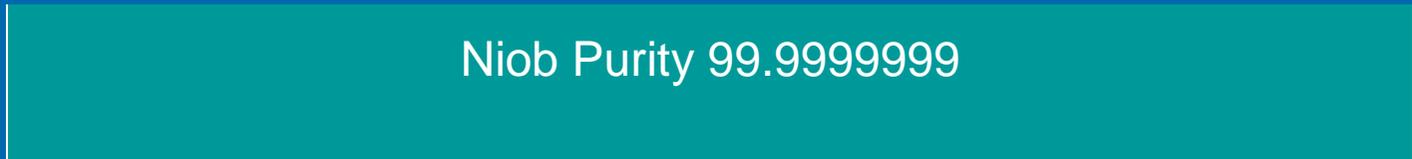
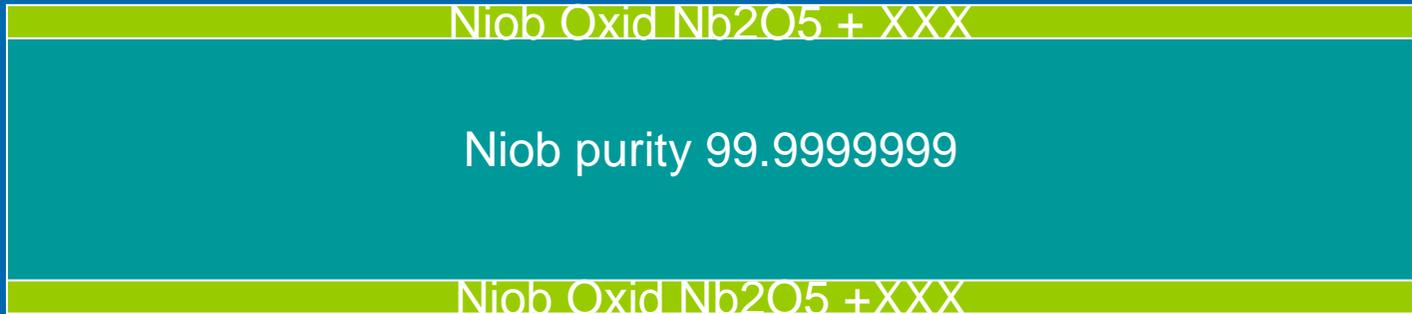
removal rate with 17 V applied

1:9 at 20C 0,3-0,5 μm/min

1:10 a t 20 C 0,3 – 0,4 μm/min

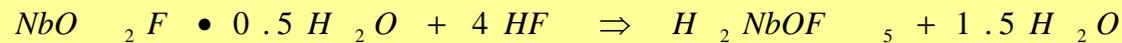
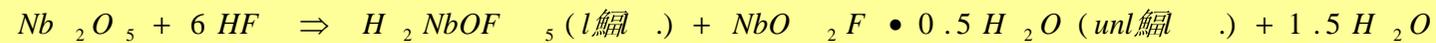
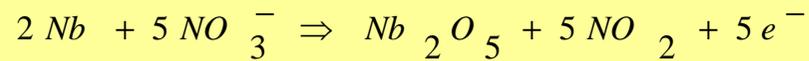
**No reaction on Niobium without voltage applied!**

# Removal of niobium by chemical reactions

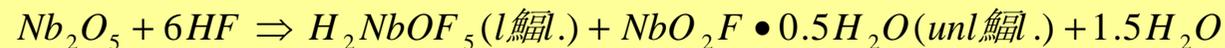
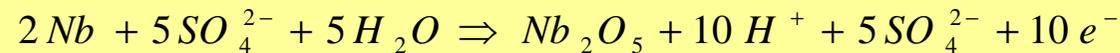


## Chemistry

BCP ( buffered chemical polishing )  
Mixture by volume 1/1/2 HF/HNO3/H3PO4



EP (electro chemical polishing)  
Mixture by volume 1/9 HF/H2SO4



## 2) Infrastructure for Cavity Preparation

Super conduction surface will be covered by normal conducting material after standard wet surface treatments  
(residues from chemical reaction and particulates)

- Large areas of n. c. Material reduce the Q value
- Particulates are origin of field emission

→ Need of particle and residue free

→ surface cleaning; Ultra pure water (UPW)

→ particle free storage and working atmosphere (Cleanroom)

→ and particle free handling adapted tools and processes

# Particulates on surfaces

Are there

Typical materials?

Non !

All air born substances of the world perform particulates

Typical size?

No!

Every size from ideal ball geometry to multi complex geometries

Common behavior?

Yes !

transportation in air make them flow every where  
well know forces making them stick on surfaces

Other sources of particles and particle motion

Mechanical vibration

**\*\* Hitting, banging, "tapping"  
(tightening bolts with wrench!!!)**

**\*\* Closing a valve quickly**

Thermal cycling.

One cannot avoid cycling to 2 K!

Particles can be dislodged by thermal stress differences.

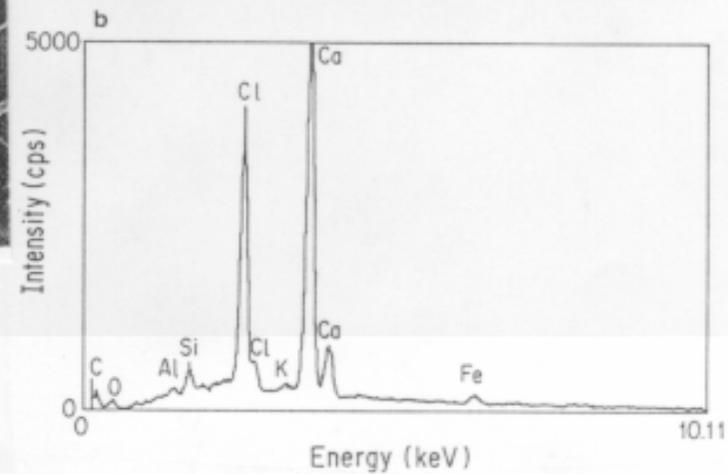
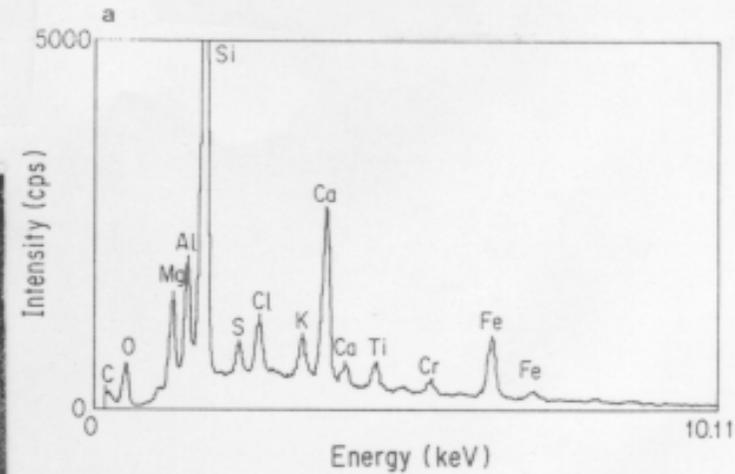
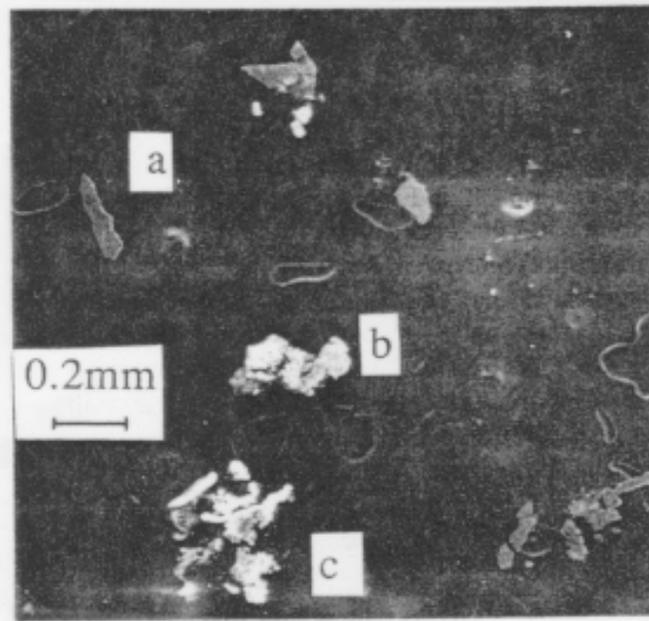
Example:

Particles found in Tristan reactor:

Environment (rock, concrete, paint)

Aluminum alloys (6063, 2219)

Pumps: Ion—TiO, TiN; NEG—ZrO



# Forces that make attractive potential to particulates facing a surface

Diameter d

$Q$

$h$

$z_0$

$-Q$

$$F = \frac{g^2}{16 \pi \epsilon_0 h^2}$$

Coulomb forces

Diameter d

$h$

$z_0$

$$F \sim \frac{d}{z_0^2}$$

Van der Waals forces

Diameter d

$\Phi_{W1}$

$\Phi_{W2}$

$h$

$z_0$

$$F \sim \frac{d (\Phi_{W1} - \Phi_{W2})^2}{z_0}$$

Electrostatic forces

Diameter d

$h$

$z_0$

$$F \sim d$$

Knudsen forces

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

Ultrasonic cleaning

Discharge of static loads



Ionized Gases (air N2)

Reduce surface tension



Alcohol / Detergents

Rinsing



High Pressure Rinsing

Enforced gas flow



air guns (N2 ; Ar)

**Problem speed on surface = 0**

oder aufgrund der Distanz von der Plattenkante  $x$

$$Re_x = \frac{U_o x}{\nu} \quad (7.16b)$$

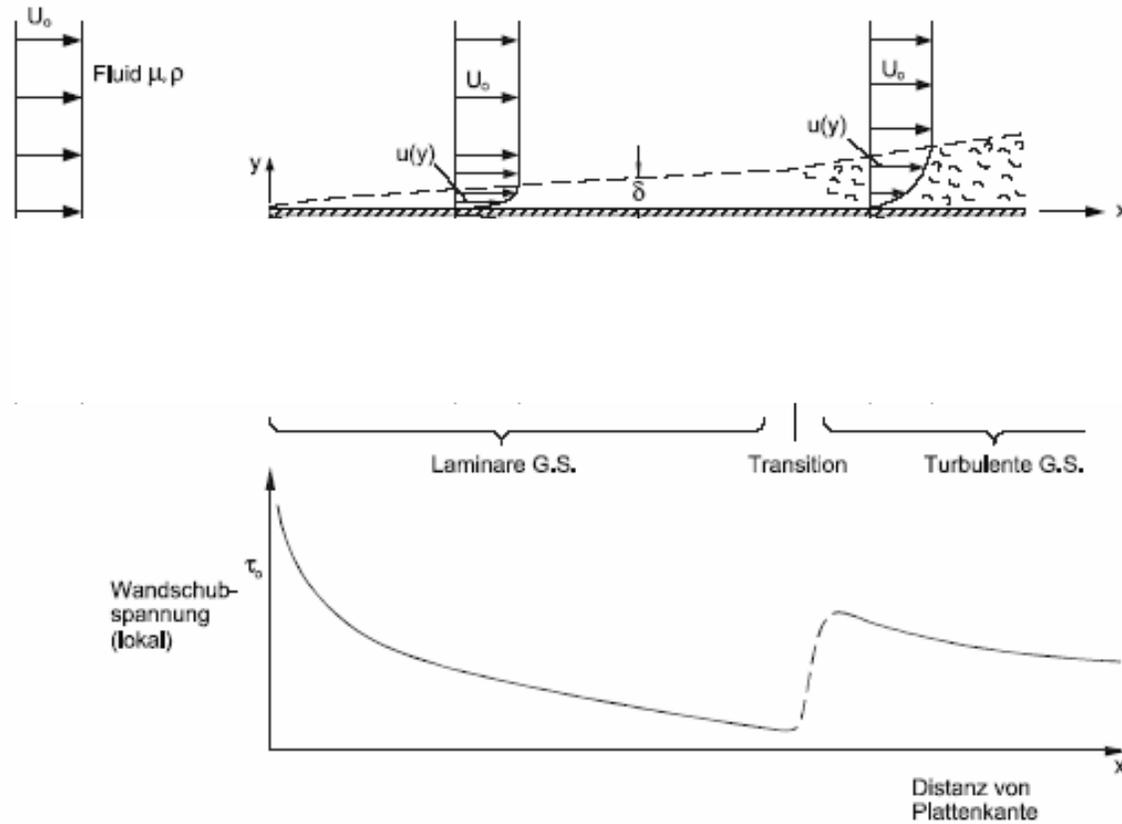
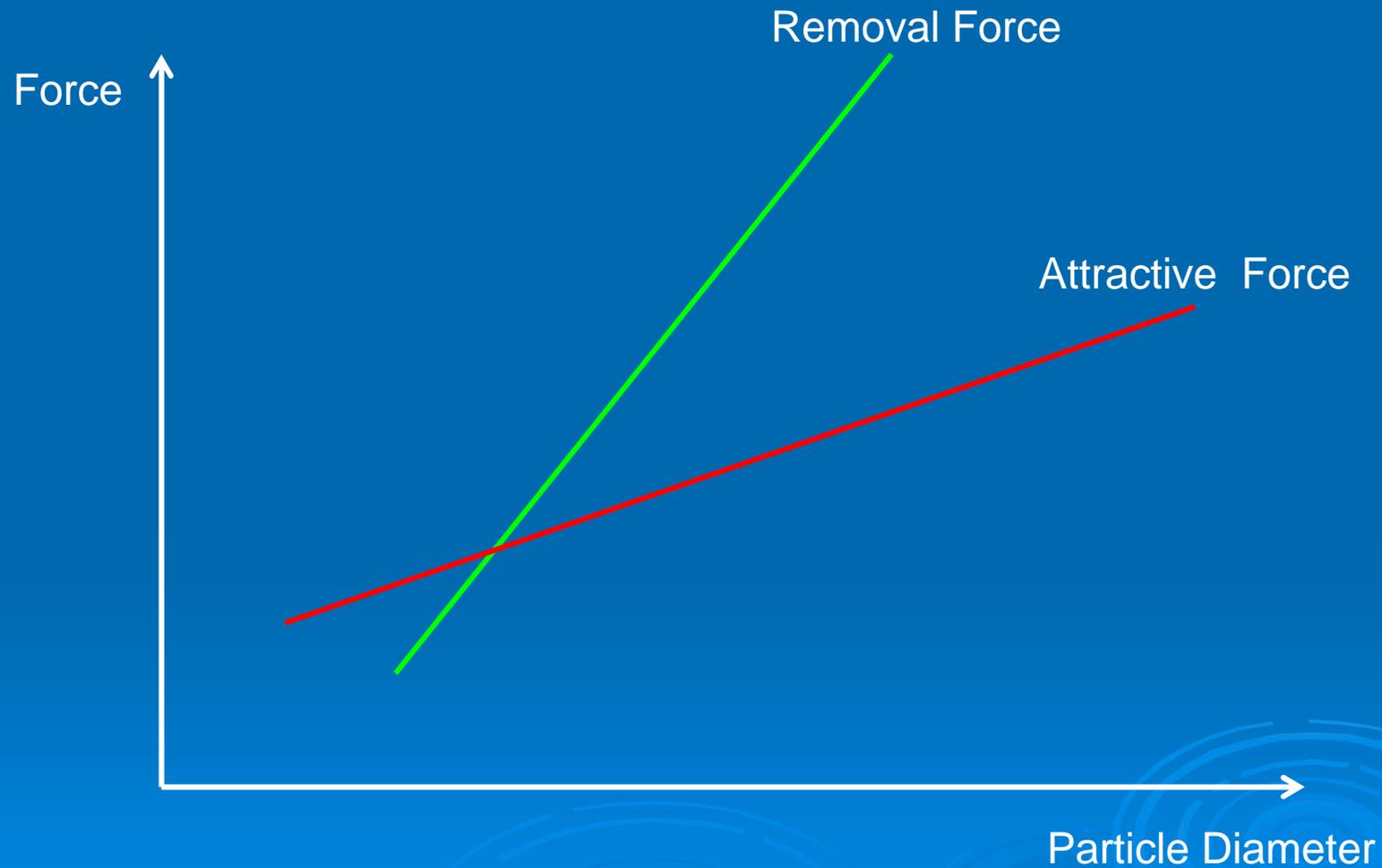
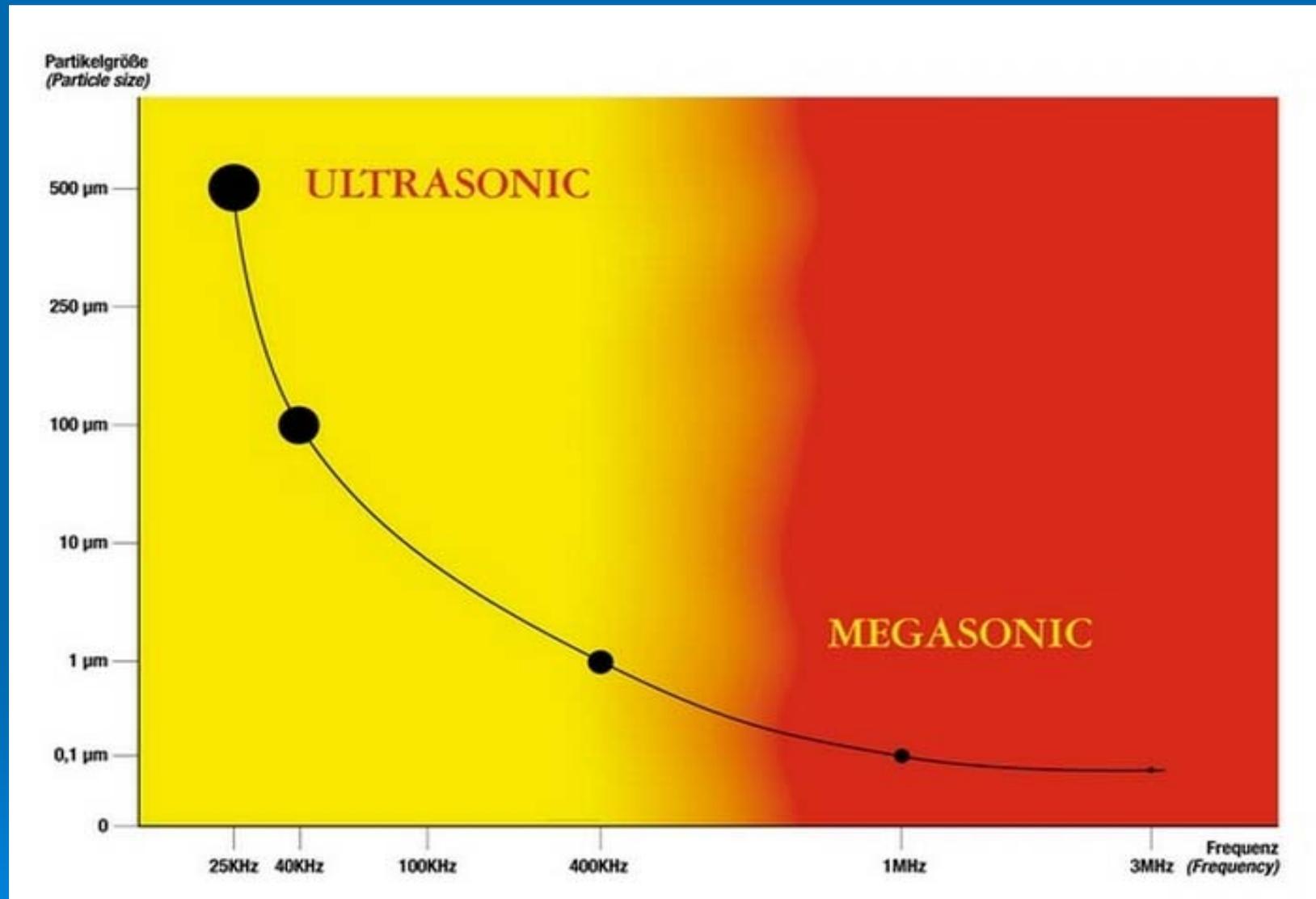


Abb. 7.5: Grenzschichtverhalten entlang einer dünnen Platte ohne Druckgradienten,  $dp/dx = 0$ .

But what to do when particulates already rest on a superconduction surface?  
Apply forces larger than the active ones (static; v.d. Waals etc.) to remove them



# Surface cleaning with sound waves in ultra pure water



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Preparation of flange surface:

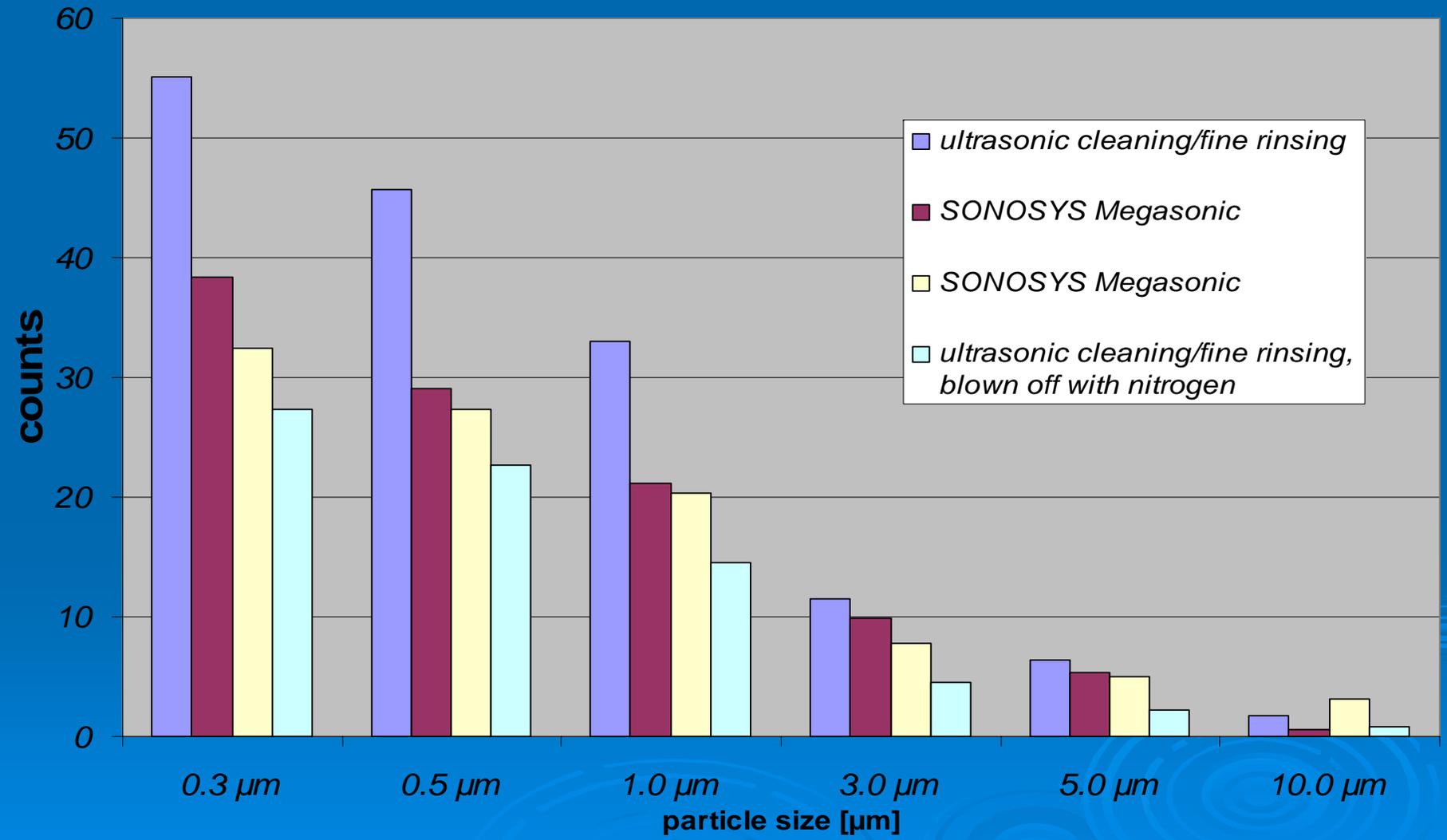
- a) - 20 Min US cleaning within DESY US basin  
- Automatic rinsing with DESY UPW rinsing basin  
( $R \geq 12 \text{ MOhmcm}$ )  
- Drying in class 100
  
- b) - cleaning with hand held megasonic cell<sup>®</sup><sub>2</sub> at optimized distance of 20 cm between Object and Cell (Nozzle)  
Drying in class 100
  
- c) - 20 Min US cleaning within DESY US basin  
- Automatic rinsing with DESY UPW rinsing basin  
( $R \geq 12 \text{ MOhmcm}$ )  
- Drying in class 100  
Blowing of surface by ionized nitrogen of the pulsed gun

## Surface scanning to measure particle concentrations on surfaces



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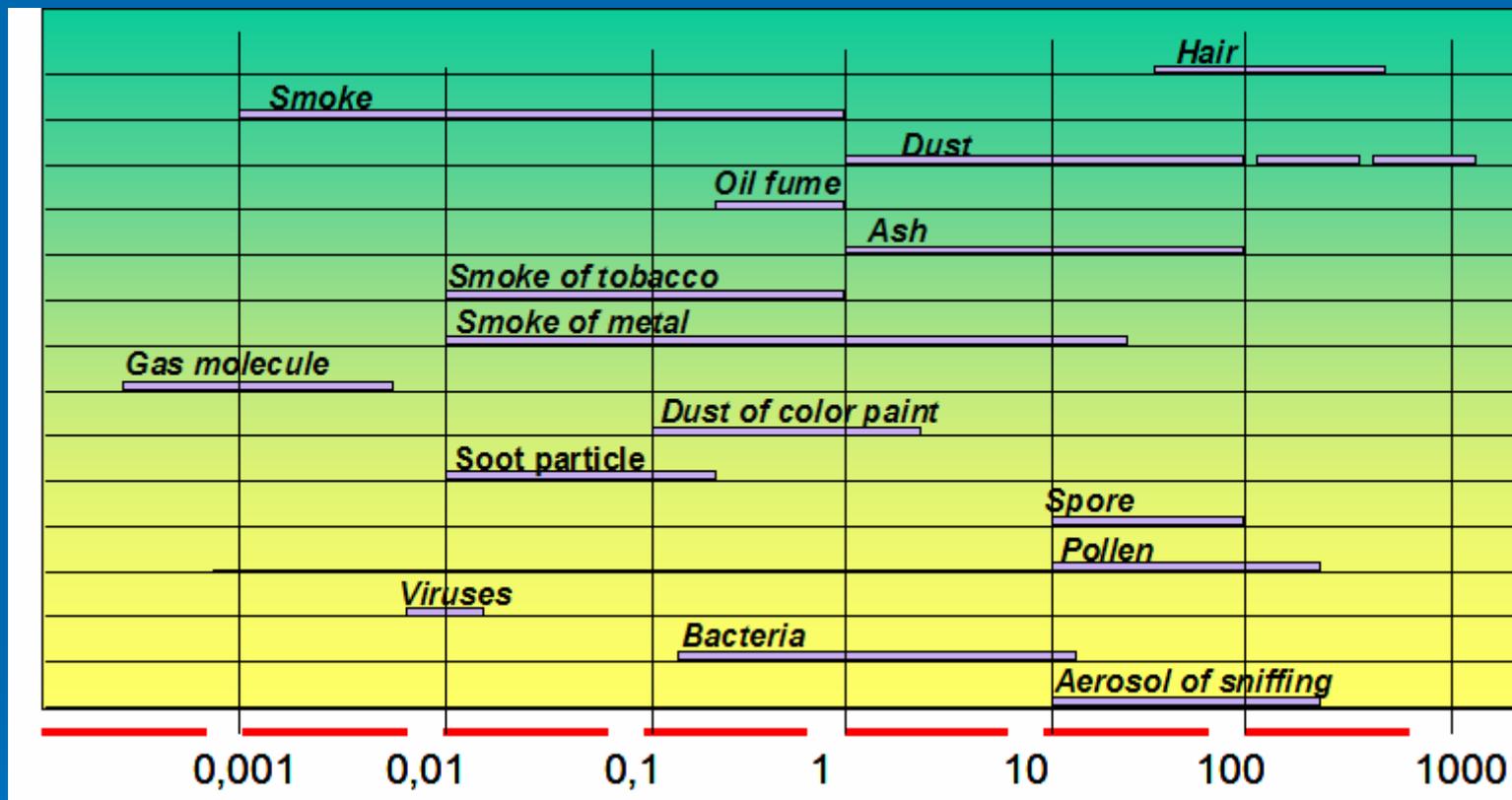
# Studies on most efficient cleaning



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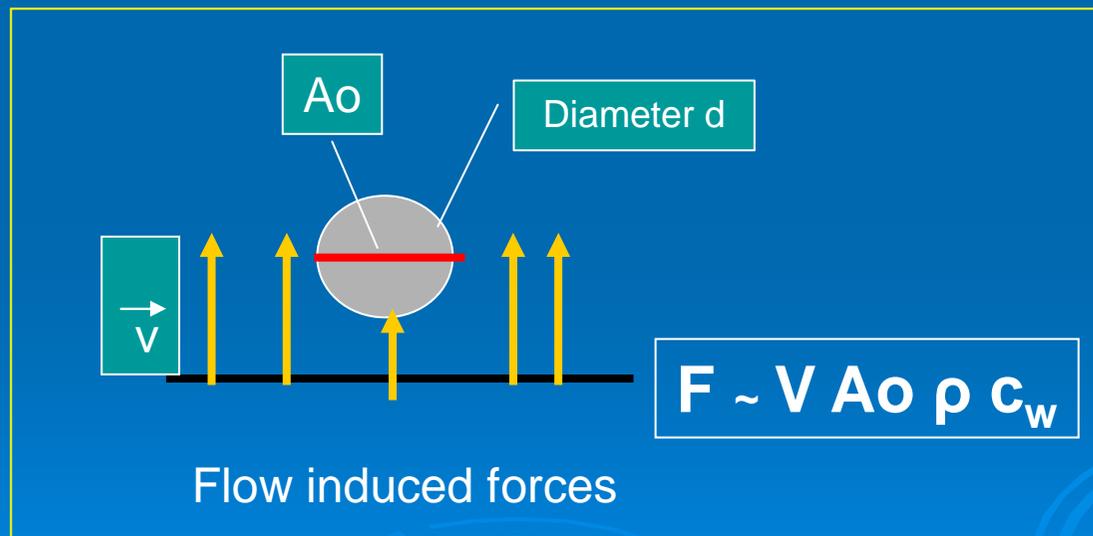
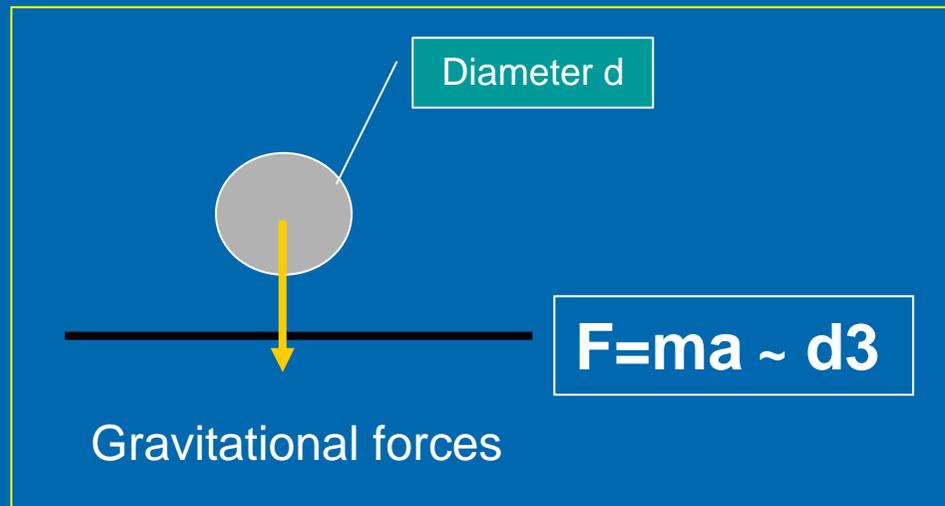
# Particulates in the air

Which particles do we find in our normal air ? ?

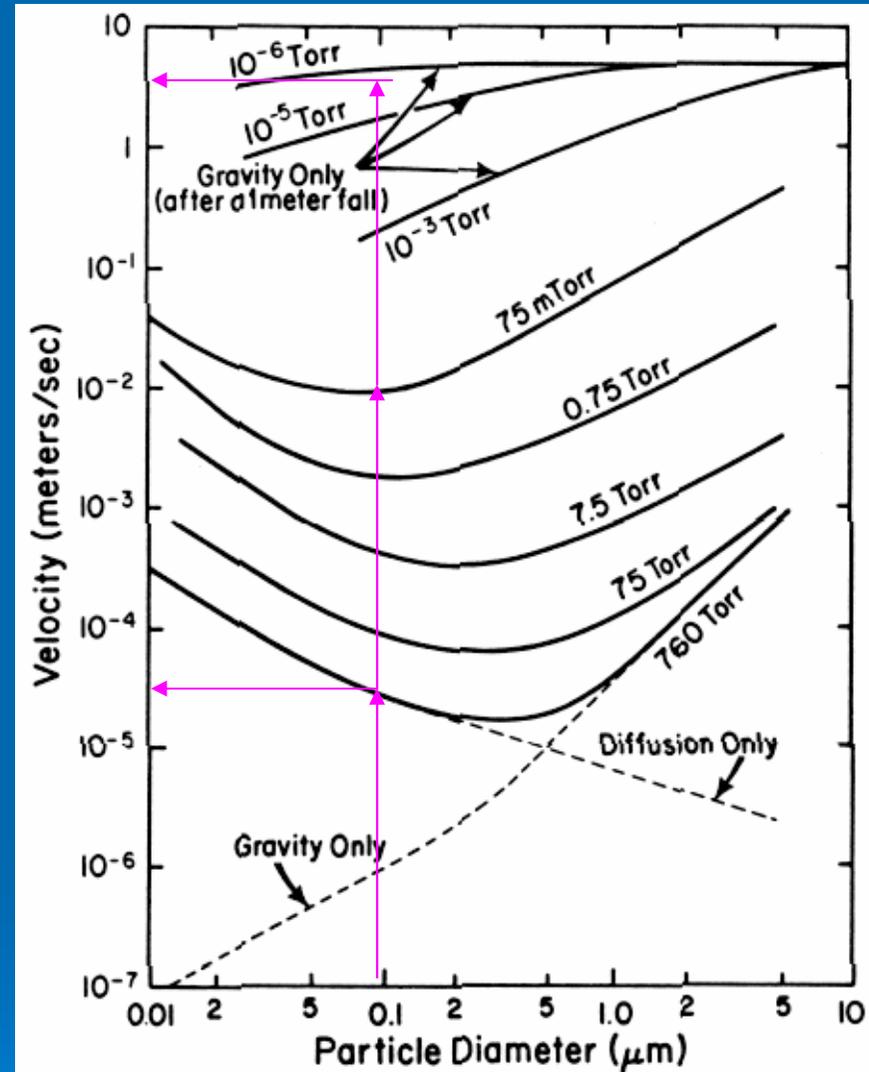


( Particle size ) [µm]

# Particulates in air

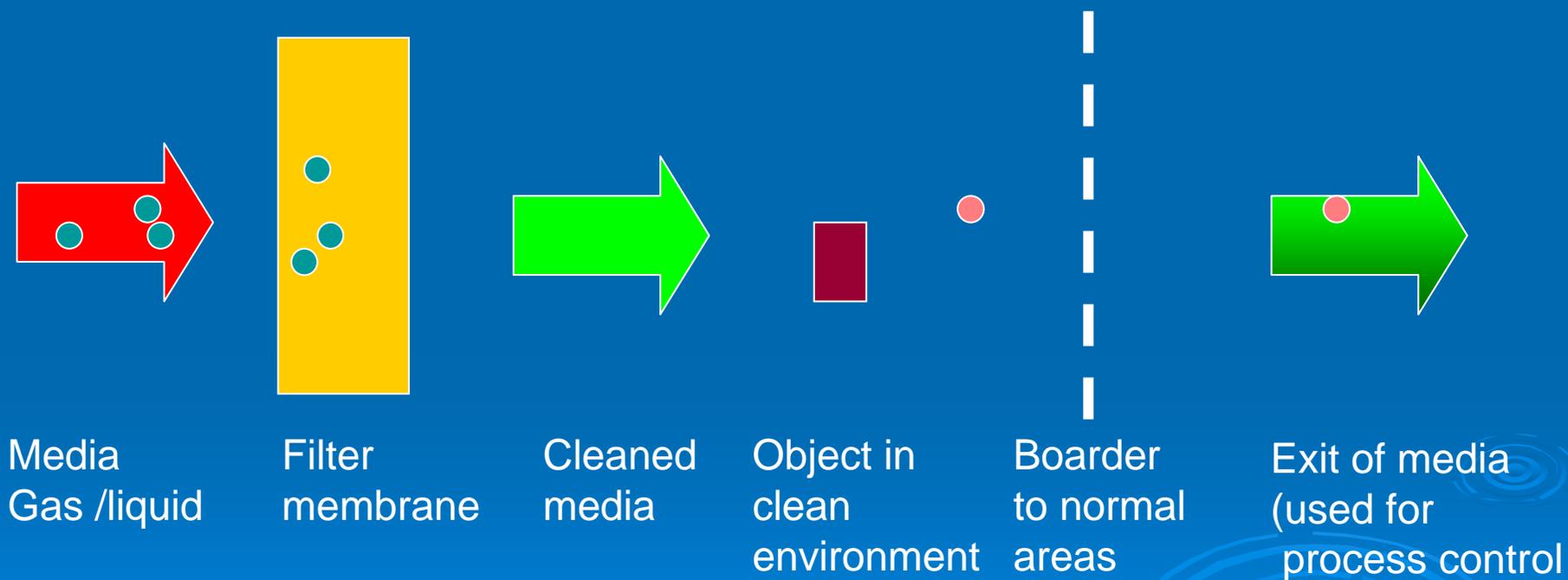


Example from  
Settling velocity for particles  
Gas :Air  
Temperature = room temperature  
Electric fields: non  
airflow: not enforced (normal air)

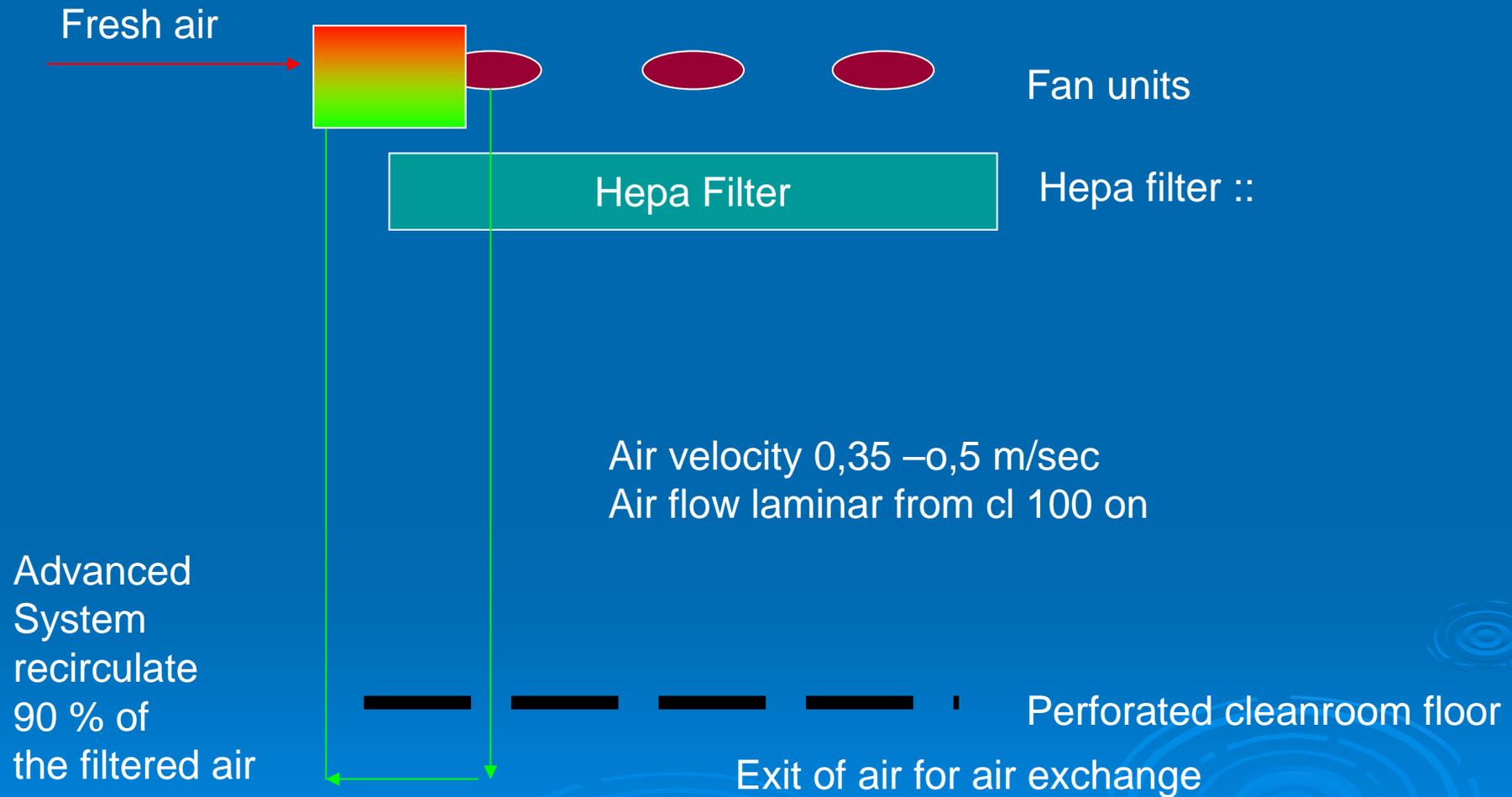


For super conduction cavities clean water and cleanroom technology is required to prevent air born particulates from settlement on surfaces

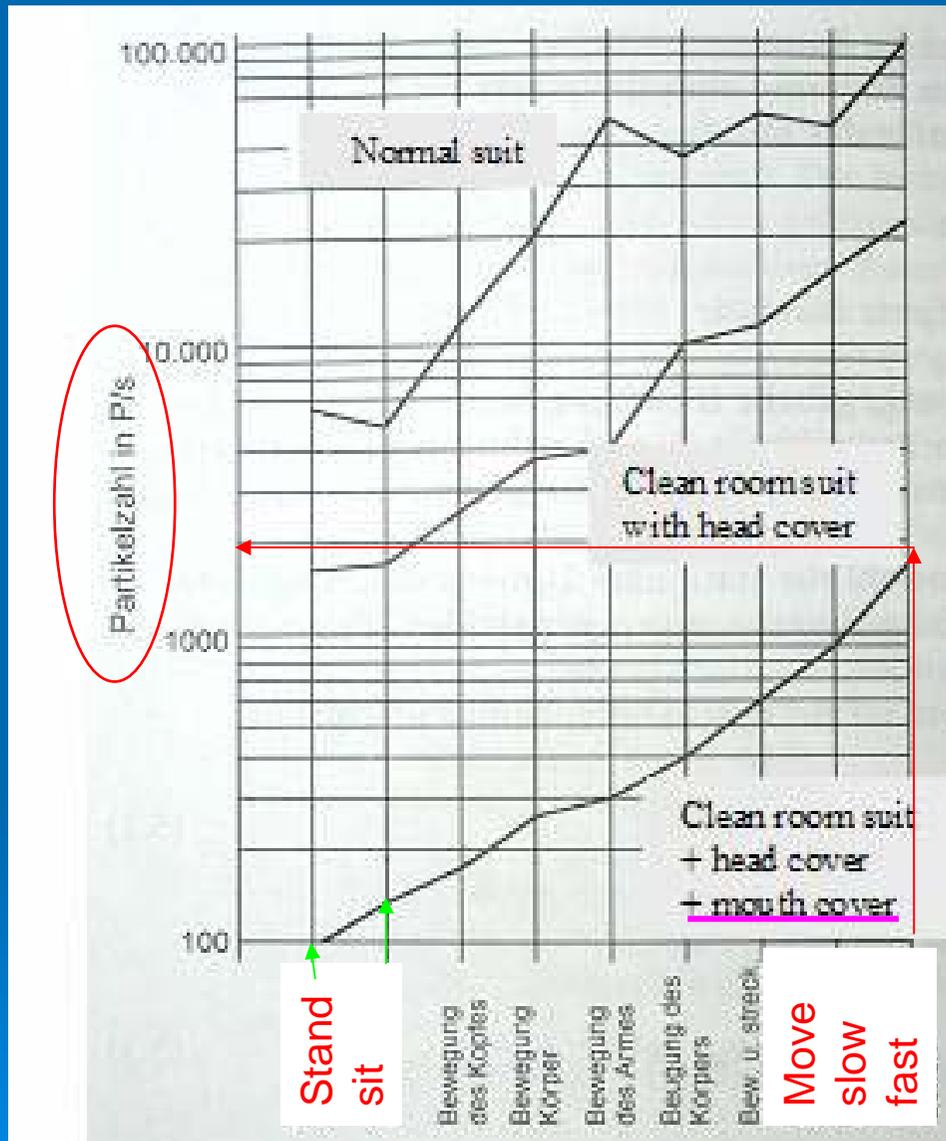
## Basics of cleanroom technology



## Basic function of a cleanroom



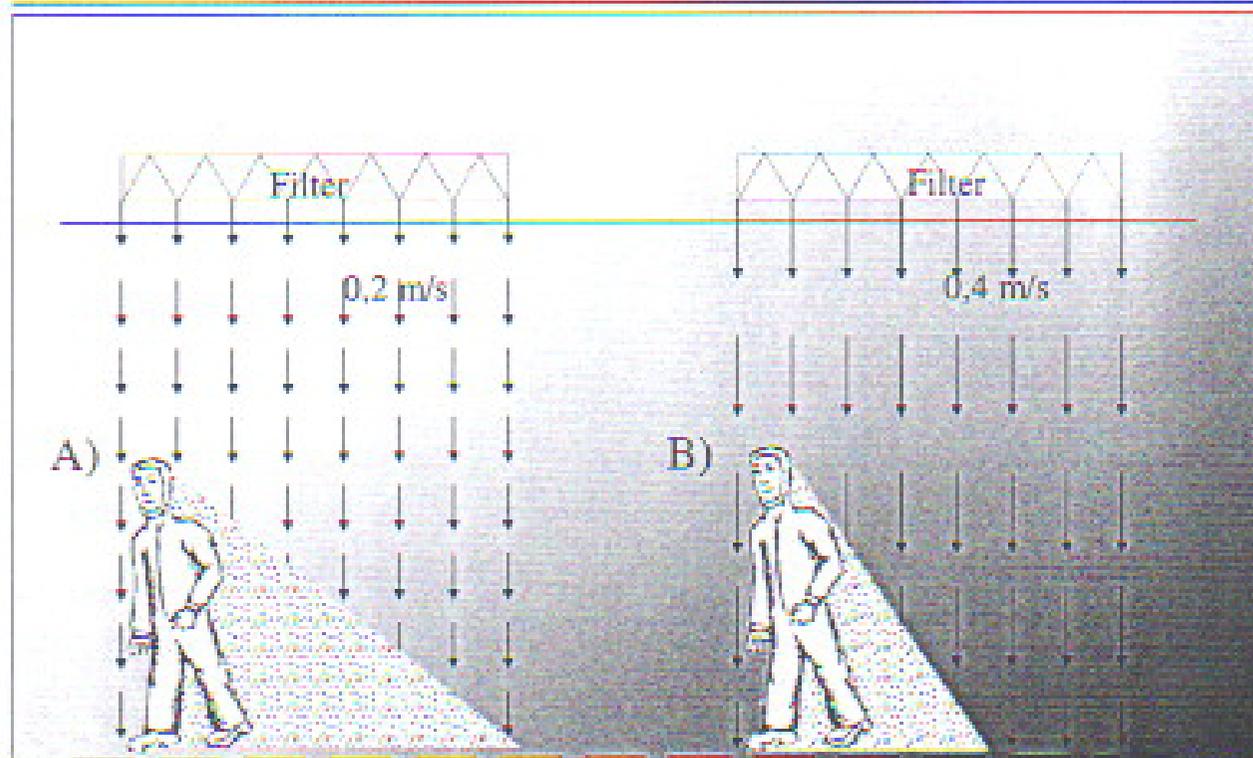
# One major part inside a cleanroom is PERSONAL



1st Dress code



## Partikelverschleppung durch Personen



Personal in Reinräumen Thomas von Kahlden info@cci-vk.de



# 3) Handling and know how on cavity preparation

Behavior of personal inside a cleanroom



**Wrong !!!!**



**Right !!!**

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**Open the door  
But right !**



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**Be careful where your hands and your body  
is close to an open cavity**



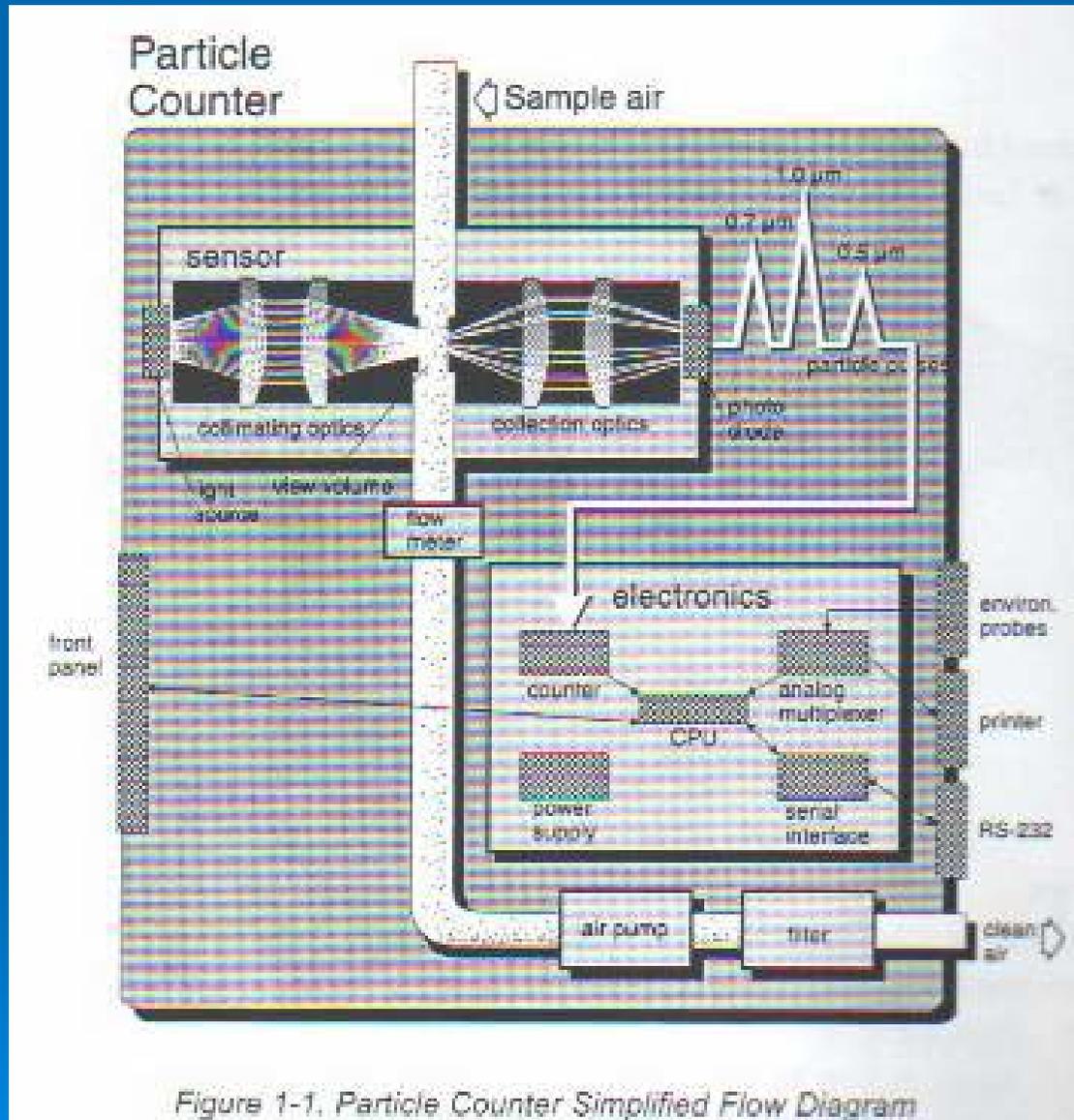
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But from where do we know that all this technology works as designed?

## Quality control during cavity preparation

- 1) Air; gasses; liquids
- 2) surfaces
- 3) Flow pattern
- 4) Degreasing and rinsing

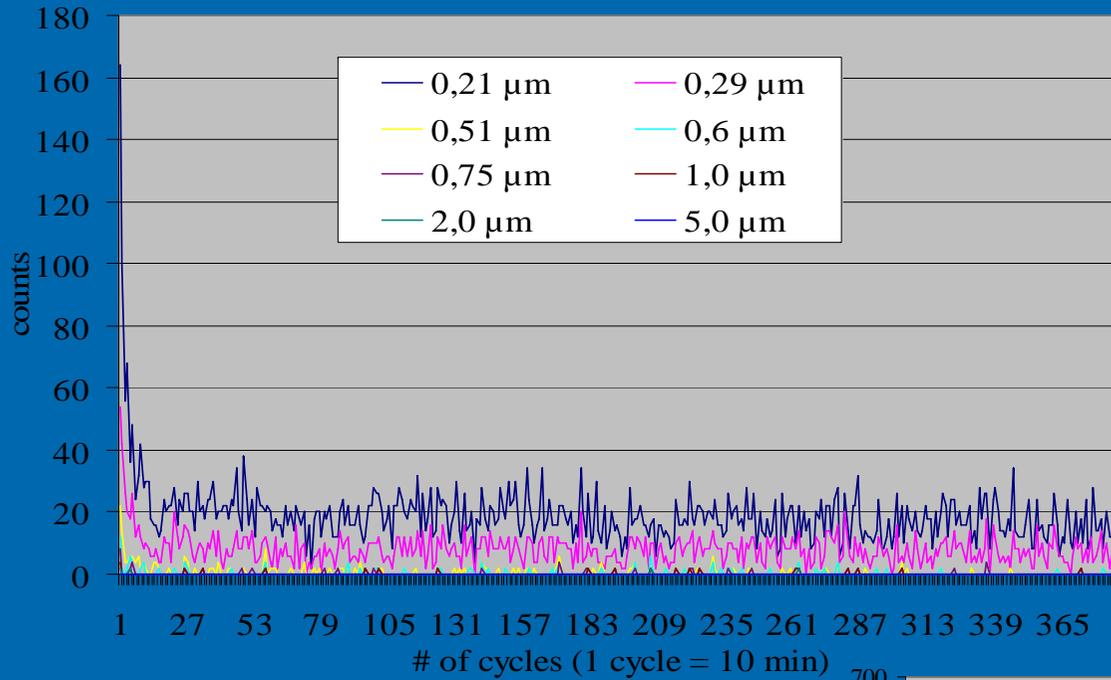
# Schematic of particle counters



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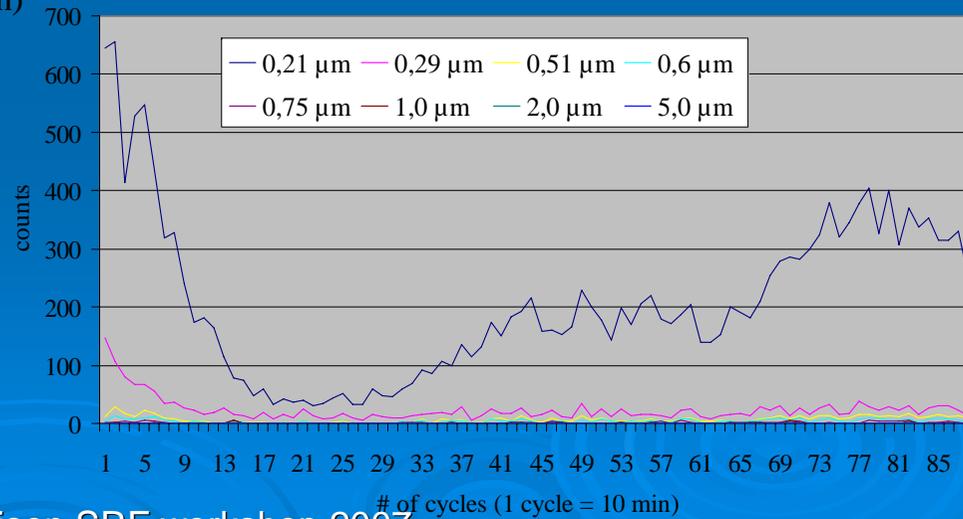
Air; gasses; liquids

## Some example on particle detection in ultra pure water



Particle concentration of  
the HPR filter after 72 h  
of rinsing

Particles found after  
installation of a new  
filter to the HPR stand,  
total sampling time 16  
hours



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Air; gasses; liquids

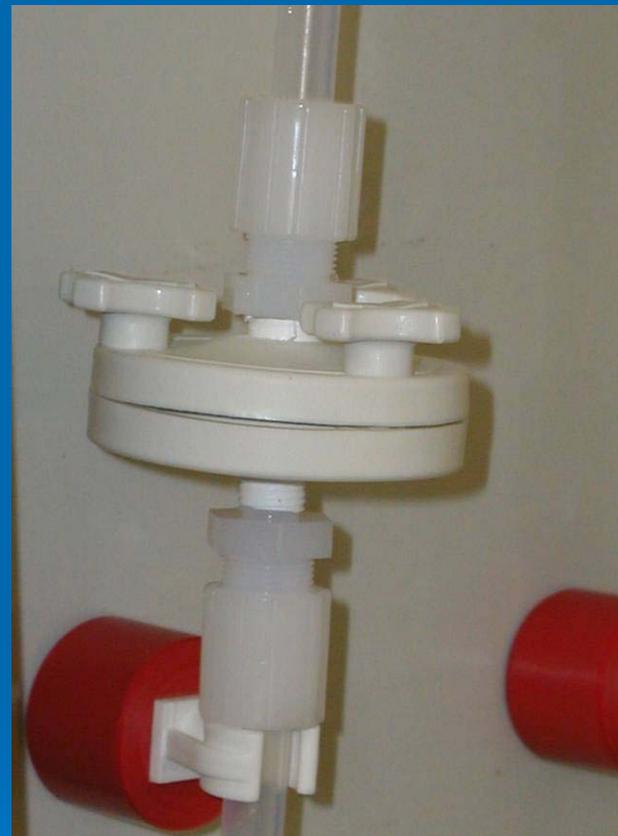
| unit                            | hardness<br>[Deutsche<br>Härte] | Restivity<br>[Mohm<br>cm] | Bacteria colonies<br>[per liter]      | TOC<br>[ppb]   | Particulates<br>[Counts<br>>0,3 µm/ liter] |
|---------------------------------|---------------------------------|---------------------------|---------------------------------------|----------------|--|
| Tap water                       | 7-10                            | << 0,0001                 | >= 100<br>( @DESY tap<br>connection ) | Not<br>defined | Not defined                                |
| Decalcification of<br>tap water | <=1                             | << 0,0001                 | 50-100                                | <10            | Not defined                                |
| Reverse<br>osmosis              | <=1                             | R<= 0,2                   | 50-100                                | < 10           | Not defined                                |
| UV light                        | <=1                             | R = 18,2                  | 1-5                                   | 1-3            | Behind filter<br>20- 100                   |
| Ion exchanger /<br>Polisher     | <=1                             | R = 18,2                  | 1-5                                   | <10            | Behind filter<br>20-100                    |
| UV light                        | <=1                             | R = 18,2                  | 1-5                                   | 1-3            | Behind filter<br>20- 100                   |
| Point of use<br>filtration      | <=1                             | R = 18,2                  | 1-5                                   | 1-3            | <=10                                       |

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## Particle detection on rinsing water of the HPR system



HPR ejection nozzles and QC funnel



Particle filter  $\leq 2 \mu\text{m}$   
in HPR draining line

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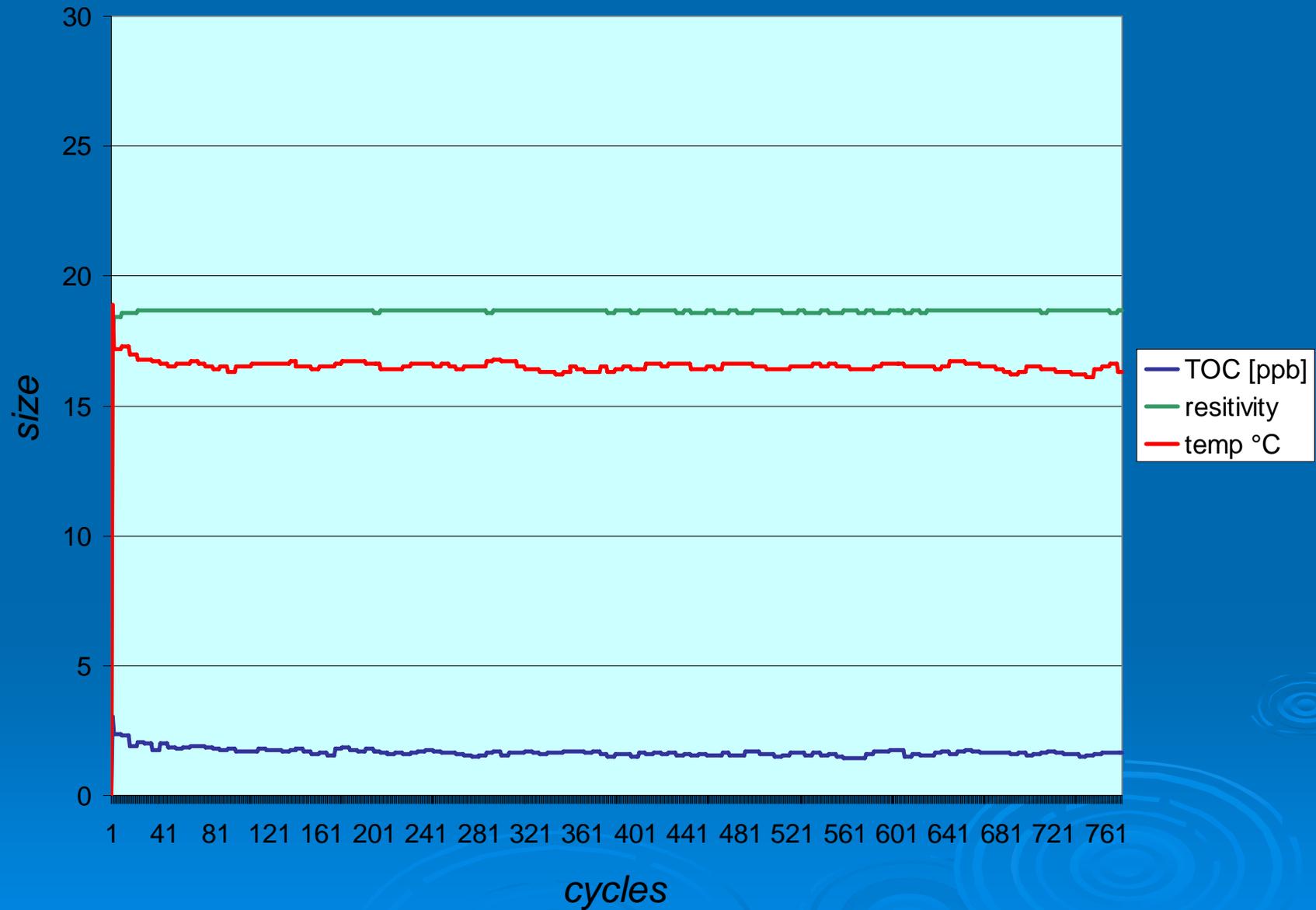
Air; gasses; liquids



Scanning microscope for particle filter counting and visual analysis

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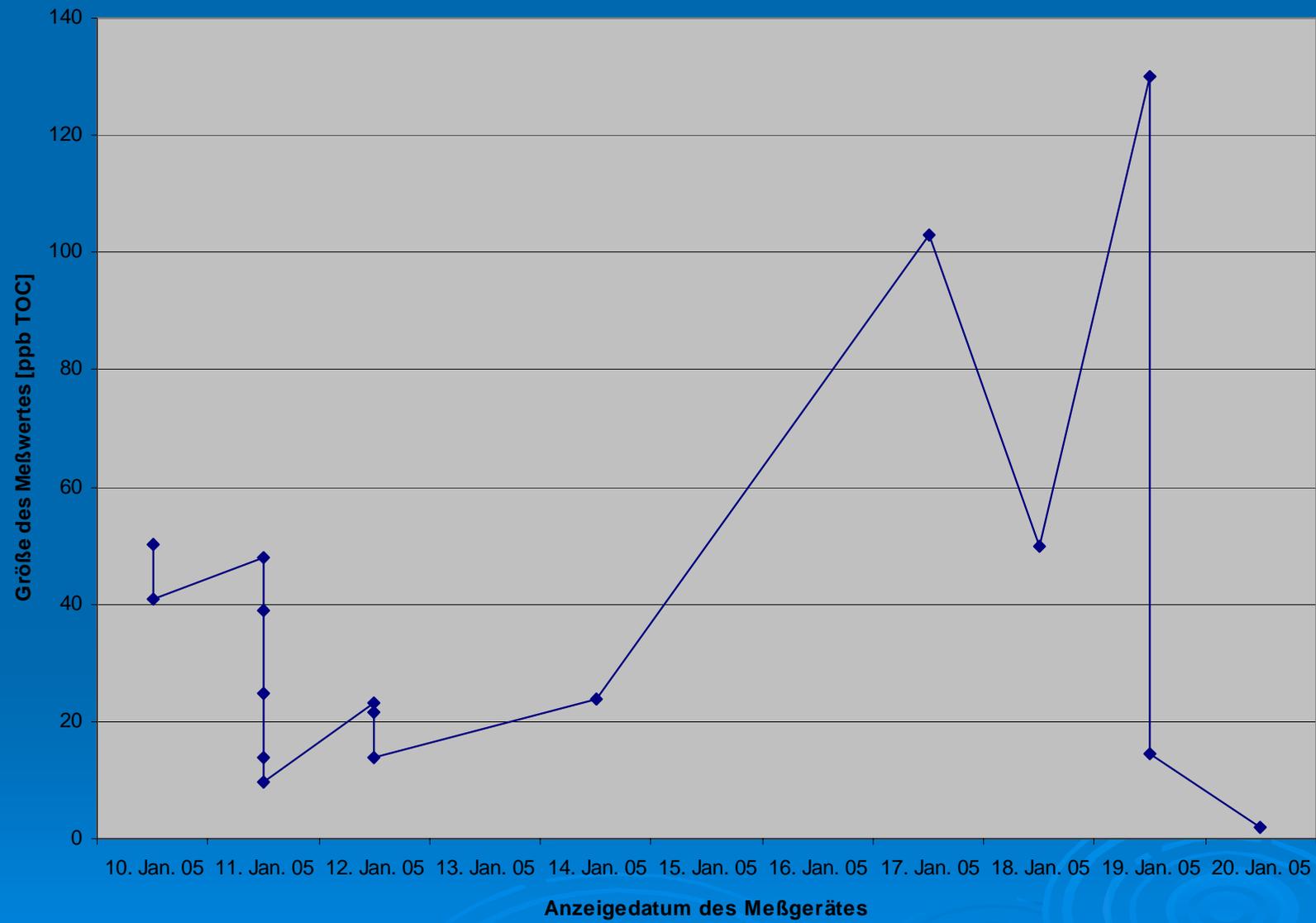
# Monitoring of the total oxydable carbon (TOC ) in ultra pure water



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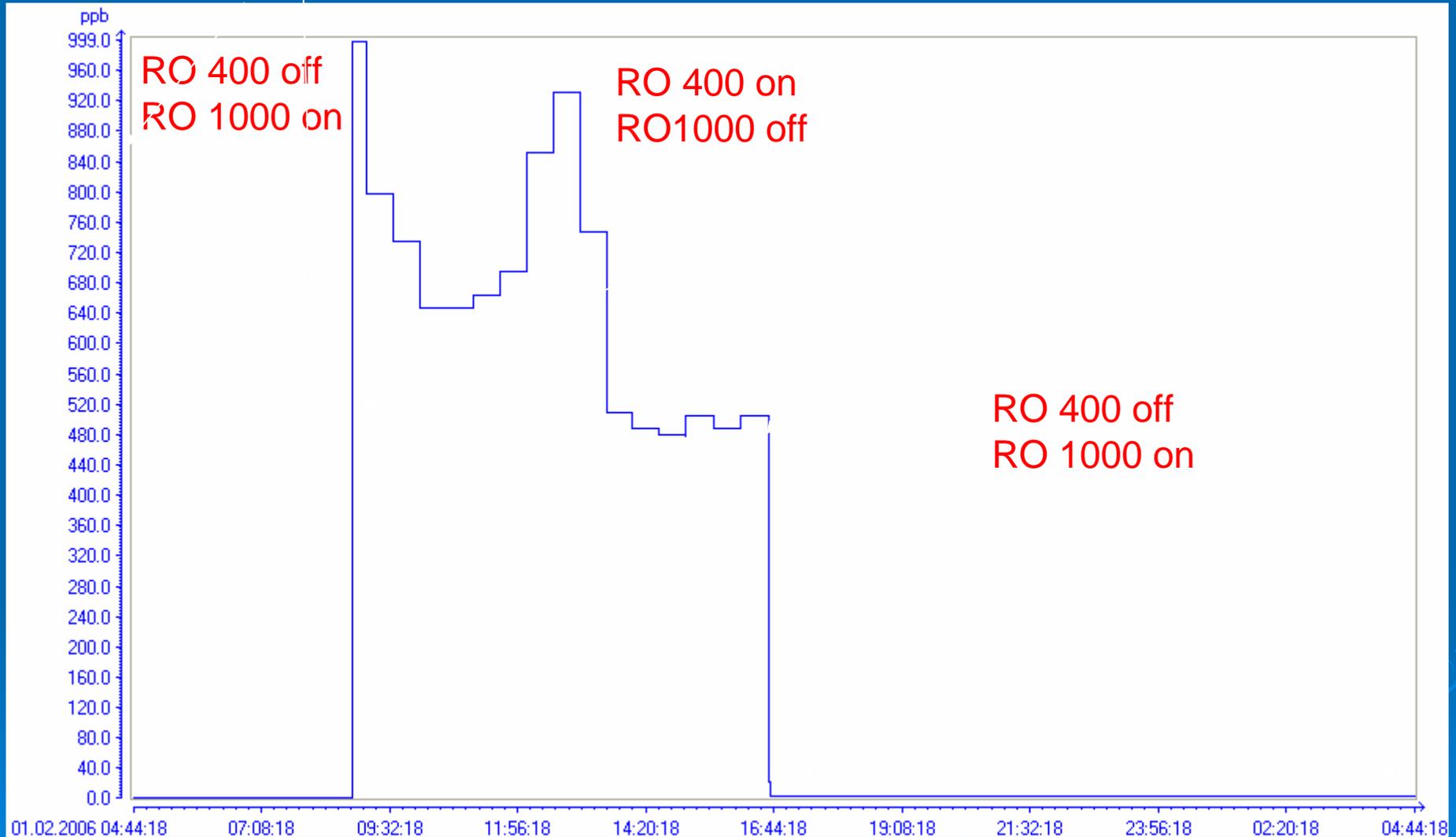
Air; gasses; liquids

### TOC-Meßwerte vor RWA Shut down am 20. Januar 2005, ca. 14:00



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## Example on bacteria contamination in one of the two reverse osmosis unit of the cleanwater plant



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Air; gasses; liquids

# Quality control on particulates removed from a surface by ionized air (top gun)



QC of studs before  
installation to a cavity



QC of washers before  
installation to a cavity

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Air; gasses; liquids

# Quality control on particle concentration on surfaces



Air particle counter modified for measurement on surfaces



Particle detection on a cleanroom wall



Particle detection on a cleanroom overall

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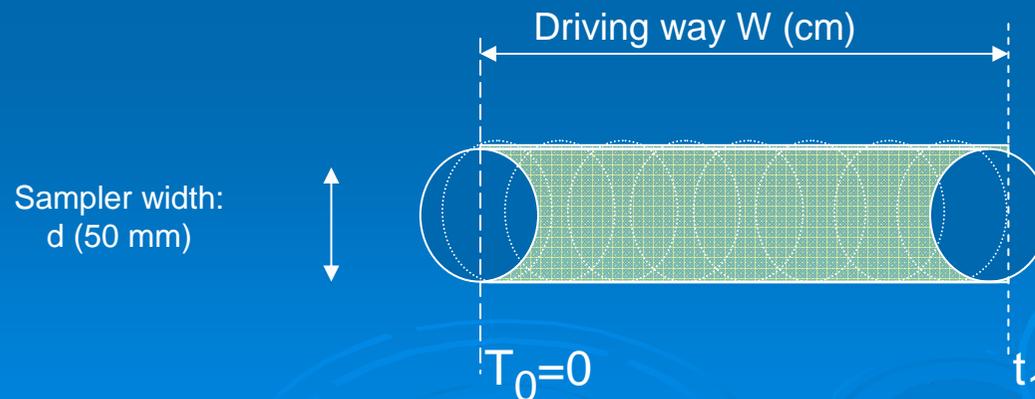


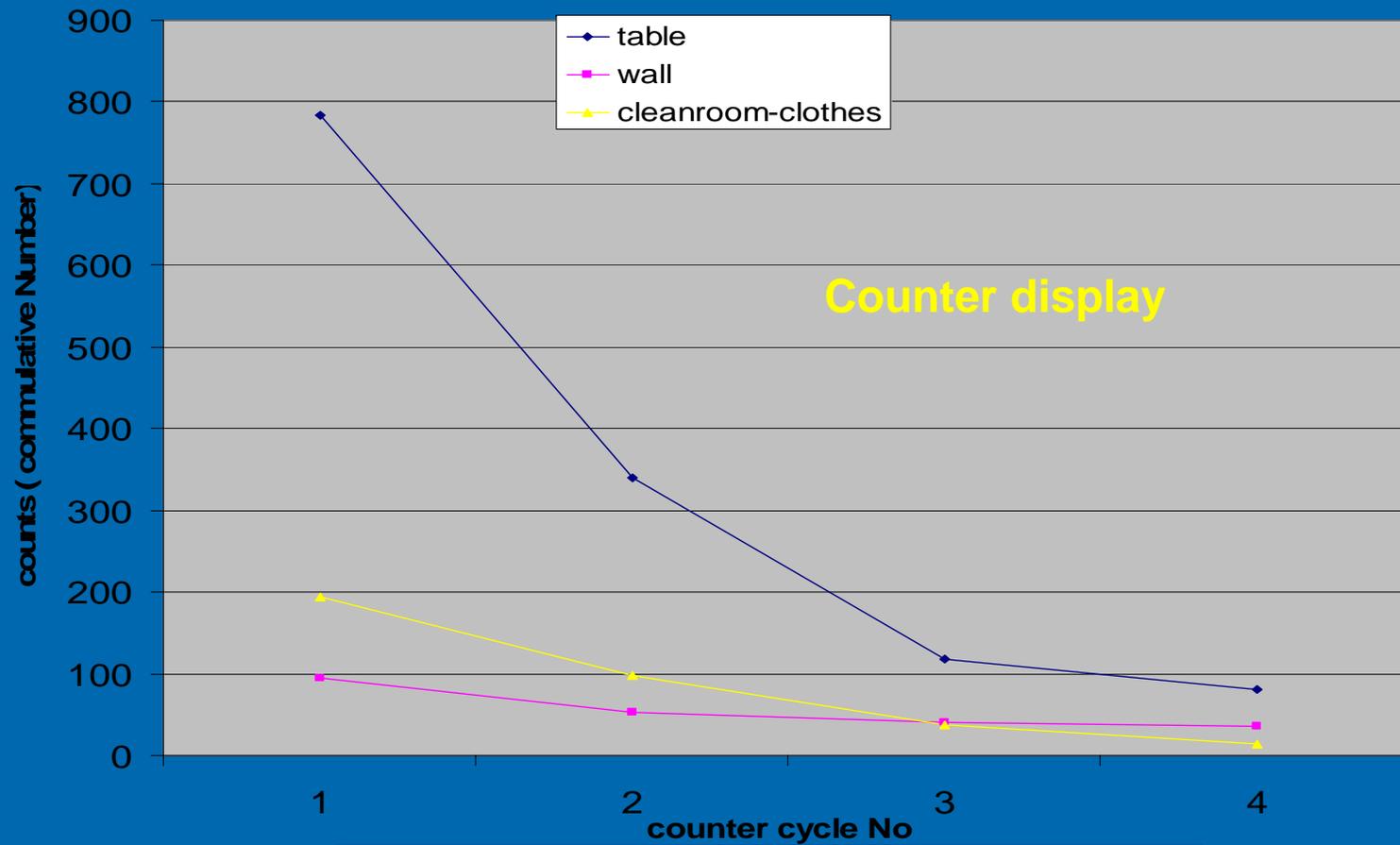
Particle detection on a cleanroom table

Instrument : modified Air Particle Counter  
Add on surface instrumentation: designed by CCI von Kahlden GmbH ®<sub>1</sub>

## Definition of particle density on surface

Particle density :  $P = C/A$  [counts per cm<sup>2</sup>]  
Static application :  $P = C / A$  counts /  $(\pi d^2)/4$   
Dynamic application:  $P = C/Ax$   
Scanned surface:  $Ax = A_1 + A_2$   
 $A_1 = (\pi d^2)/4$  [mm<sup>2</sup>]  
 $A_2 = W*d$  [mm<sup>2</sup>]  
Sampler speed:  $V = W / t_1$   
setting of counter: Sampling time t [sec]  
Dynamic application:  $P = Ct / (V*t_1*d + 1964)$

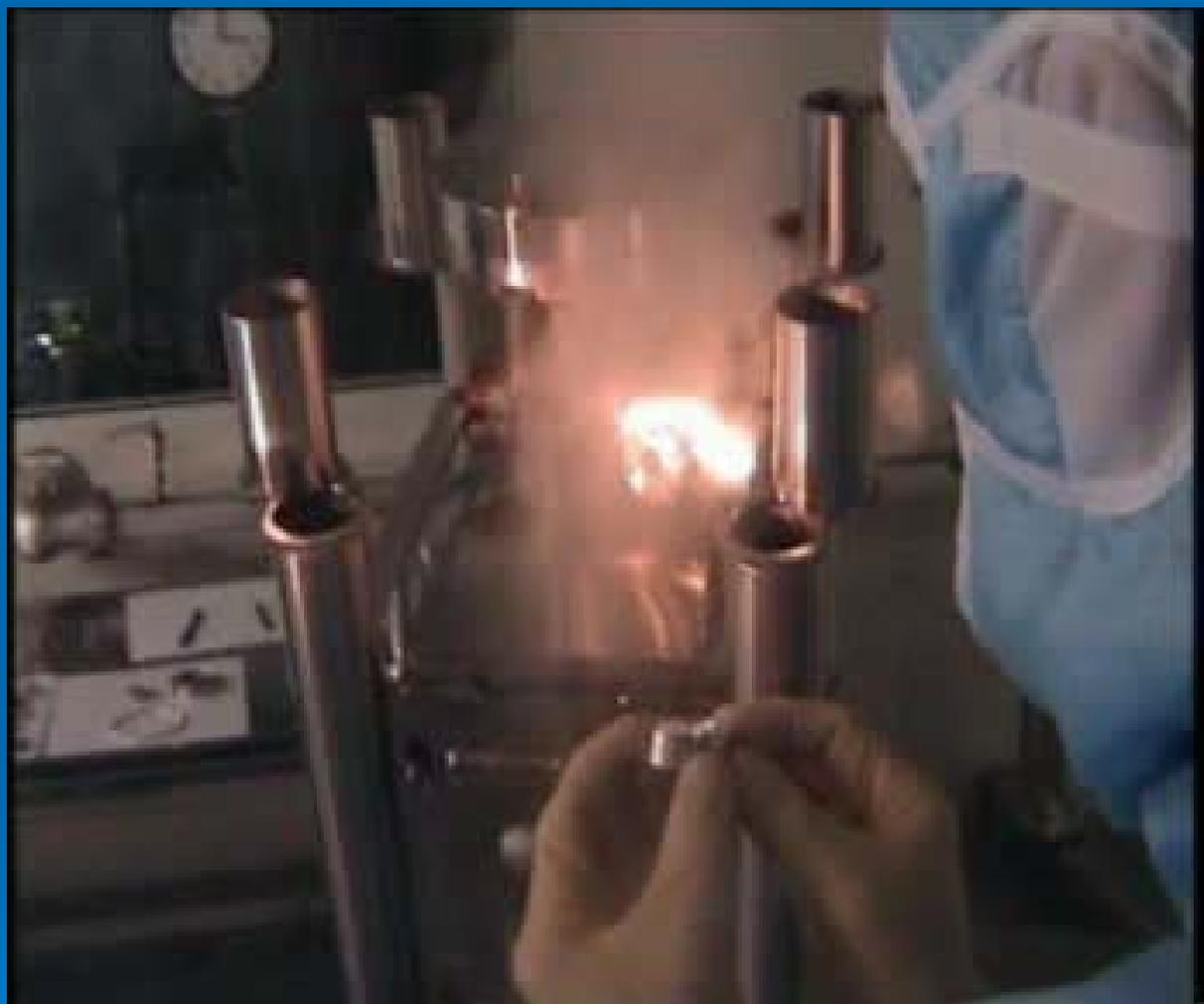




Particle concentration on:

|                   |                                     |
|-------------------|-------------------------------------|
| Table             | = 67,3 [Particles/mm <sup>2</sup> ] |
| Wall              | = 11,5 [Particles/mm <sup>2</sup> ] |
| Cleanroom overall | = 17,4 [Particles/mm <sup>2</sup> ] |

related numbers of particle density



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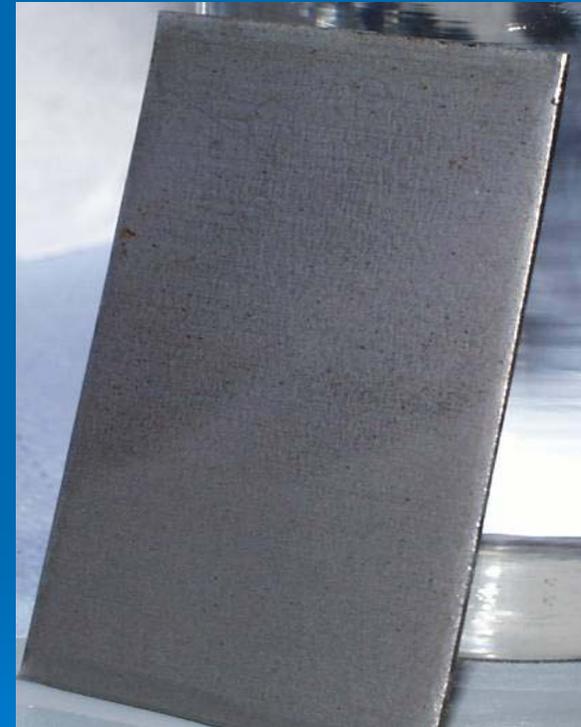
Flow pattern

# Degreasing and Rinsing

Test on cleaning procedure/ detergent  
Nb sample polluted with grease and oil



Not efficient cleaning



After Ultrasonic cleaning with  
sufficient detergent and procedure

## New method to control the High pressure rinsing jet (INFN Milano)



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## One example of Cavity preparation

Preparation of superconducting cavities at DESY

Cavities arrive from Industry and have undergone incoming inspection  
After that

A) Main EP

- 1) Degreasing and rinsing
- 2) Prepare for EP
- 3) EP treatment
- 4) Clean cavity and rinse for cleanroom
- 5) outside etching for 800 C annealing
- 6) 800 C annealing
- 7) Tuning for vertical test



Ultra sonic rinsing (up to 4KW Power )  
Rinsing by ultra pure water

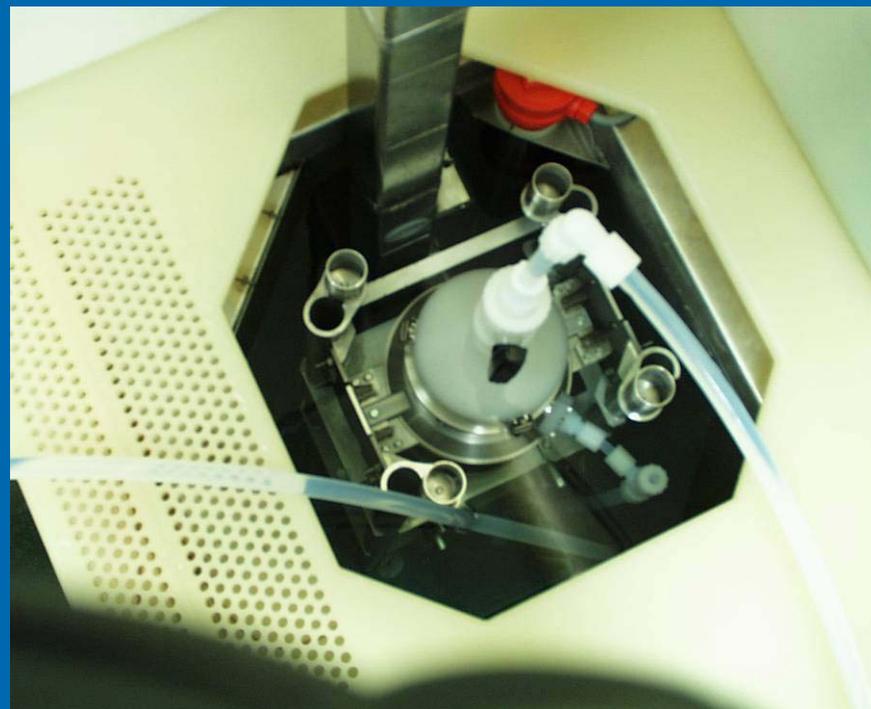
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Degreasing and rinsing



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Degreasing and rinsing



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Degreasing and rinsing



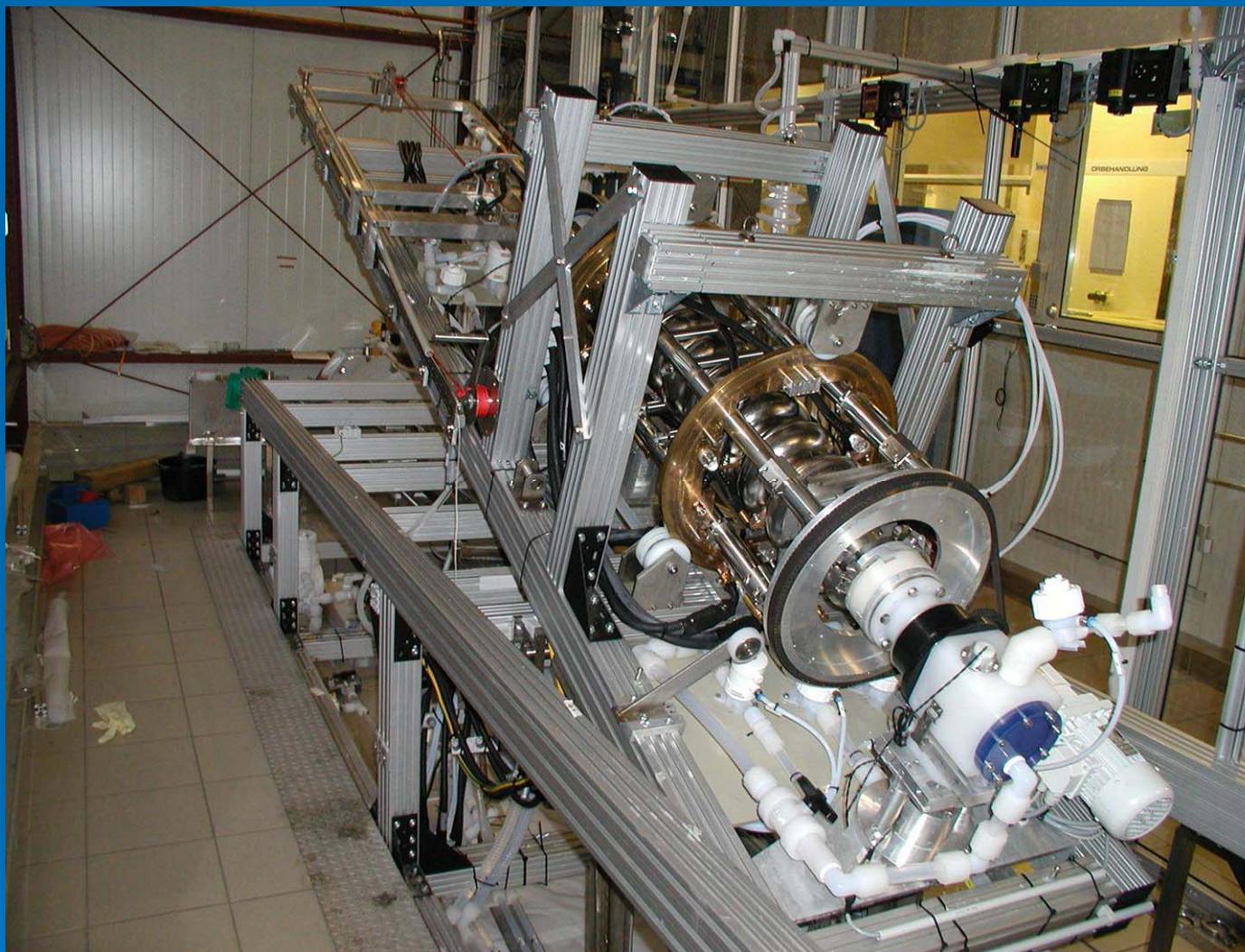
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Prepare for EP



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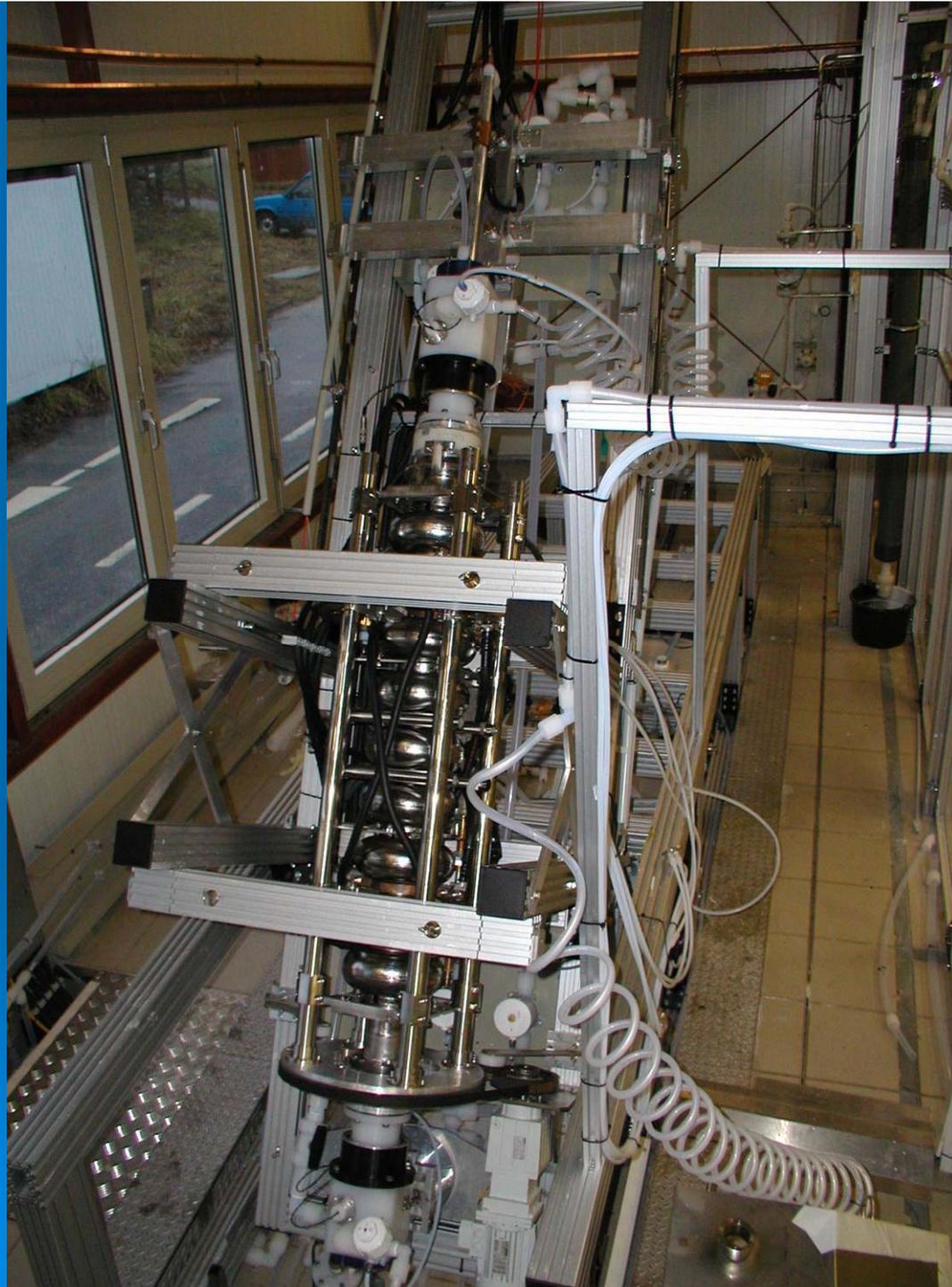
EP treatment



Cavity installed in electro polishing apparatus

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1) EP treatment



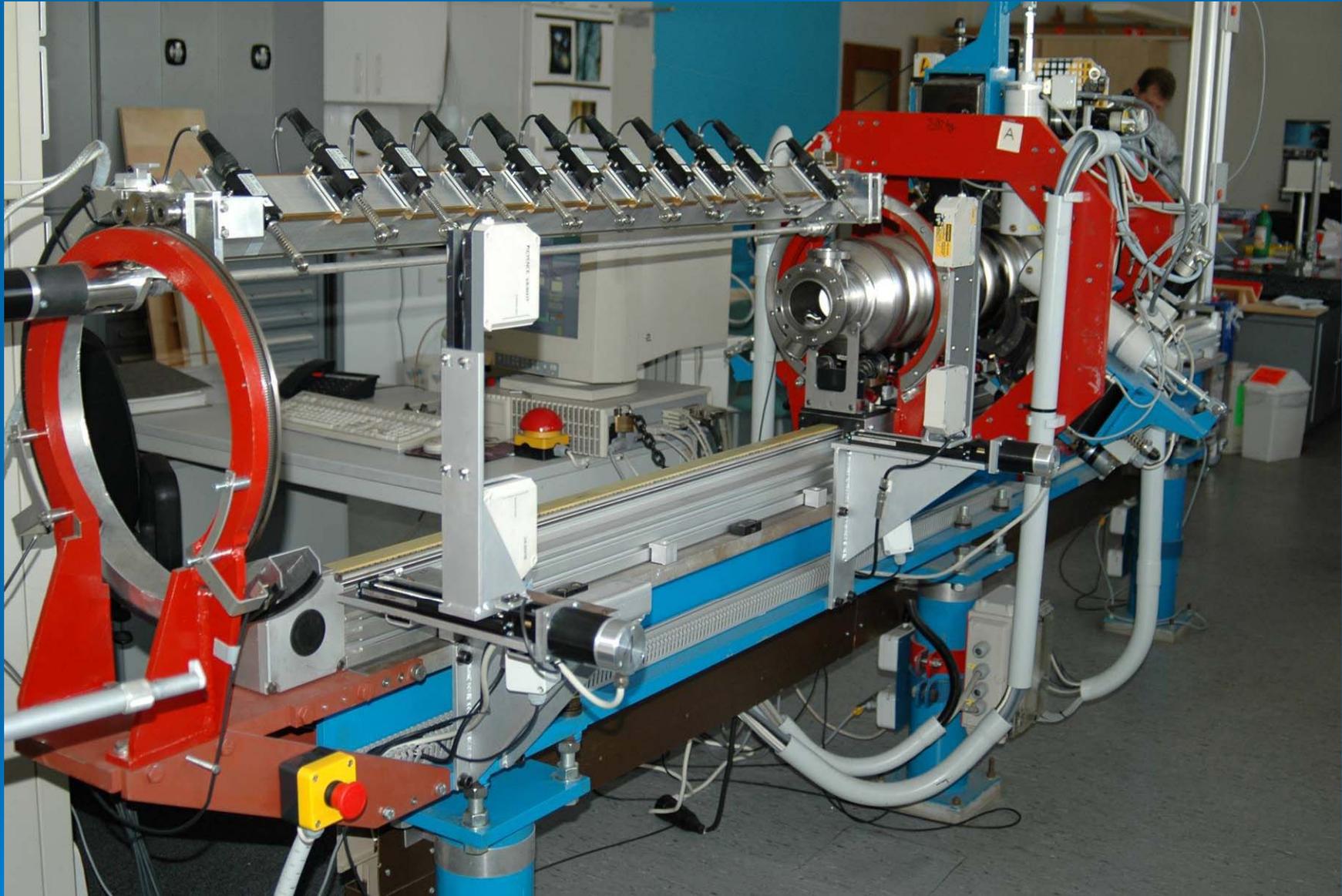
Ep apparatus  
during  
draining of acid

EP treatment



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800 C annealing



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Tuning for vertical test

After that

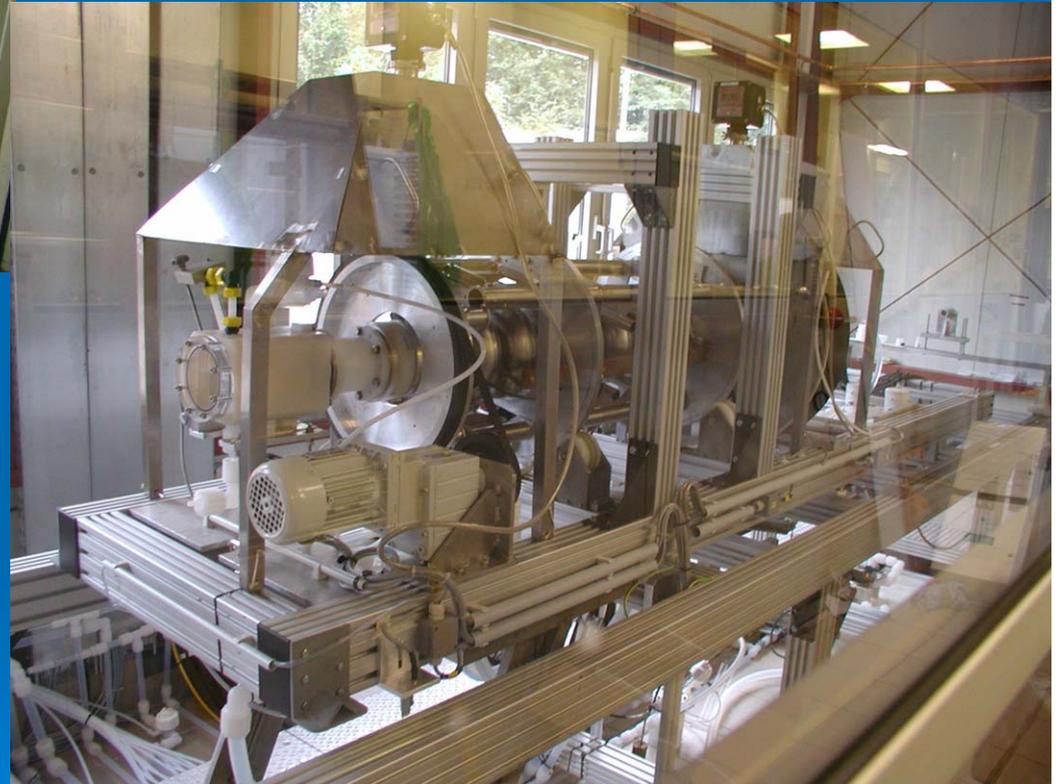
B) Preparation for vertical test at 2K

- 1) "Car wash"
  - 2) Degreasing and rinsing
  - 2a) Prepare for EP and EP treatment
  - 2b) or BCP treatment
  - 4) Rinsing and 1st high pressure rinsing
  - 5) Drying in class 10 (ASTM)
  - 6) Assembly of accessories
  - 7) Alcohol rinsing
  - 8) Six times high pressure rinsing
  - 9) Drying in class 10 (ASTM)
  - 10) Assembly of test antenna
  - 11) 120 C baking
- vertical test



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“Car wash”



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Prepare for EP  
and EP treatment

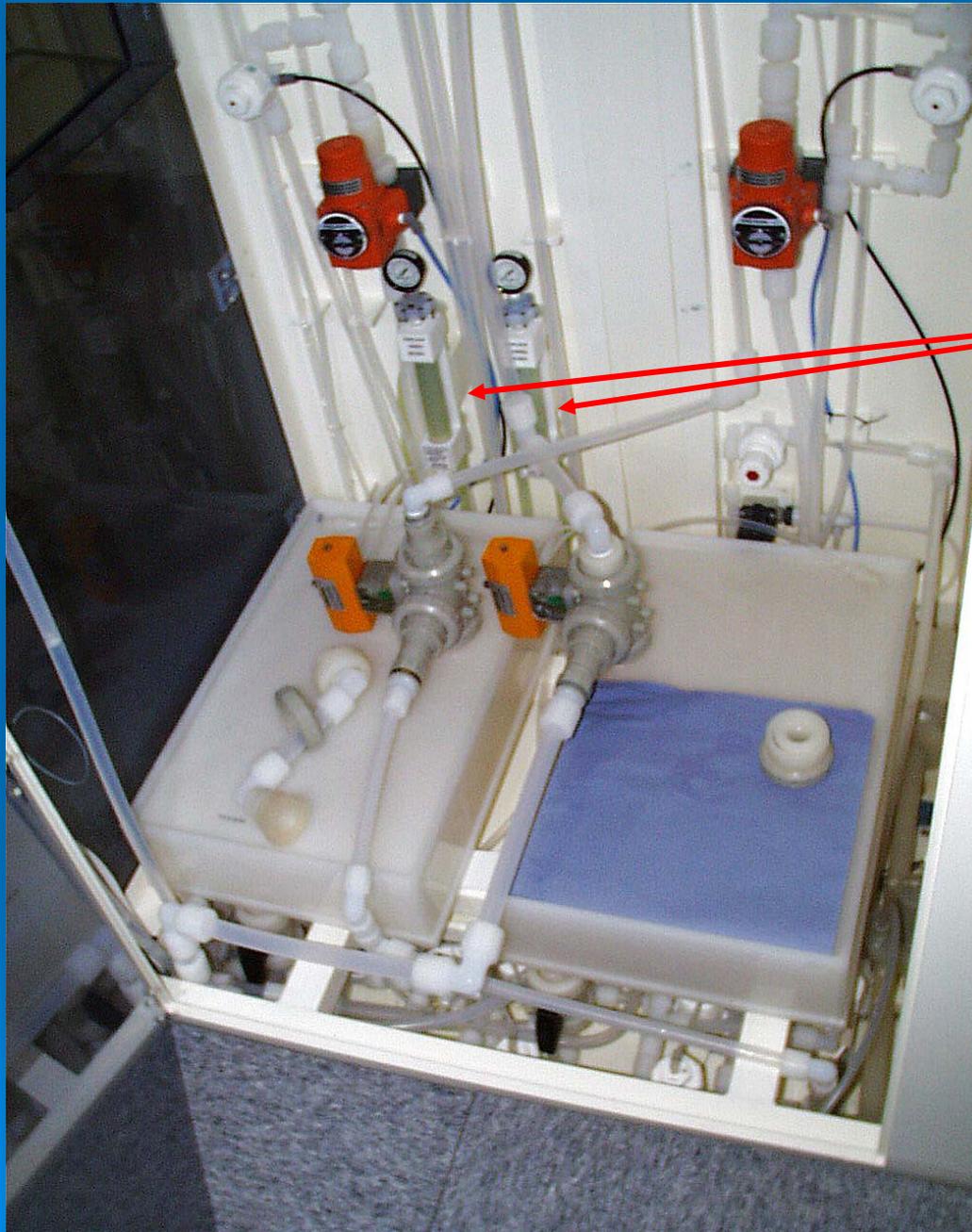


Cavity installed in BCP bench  
(Buffered chemical polishing

07

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or BCP treatment



**Säure!!!**

Valve box for BCP  
treatment

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or BCP treatment



View into class 10000 cleanroom  
"Wet preparation area"



Rinsing and 1st high  
pressure rinsing

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Drying in class 10 (ASTM)



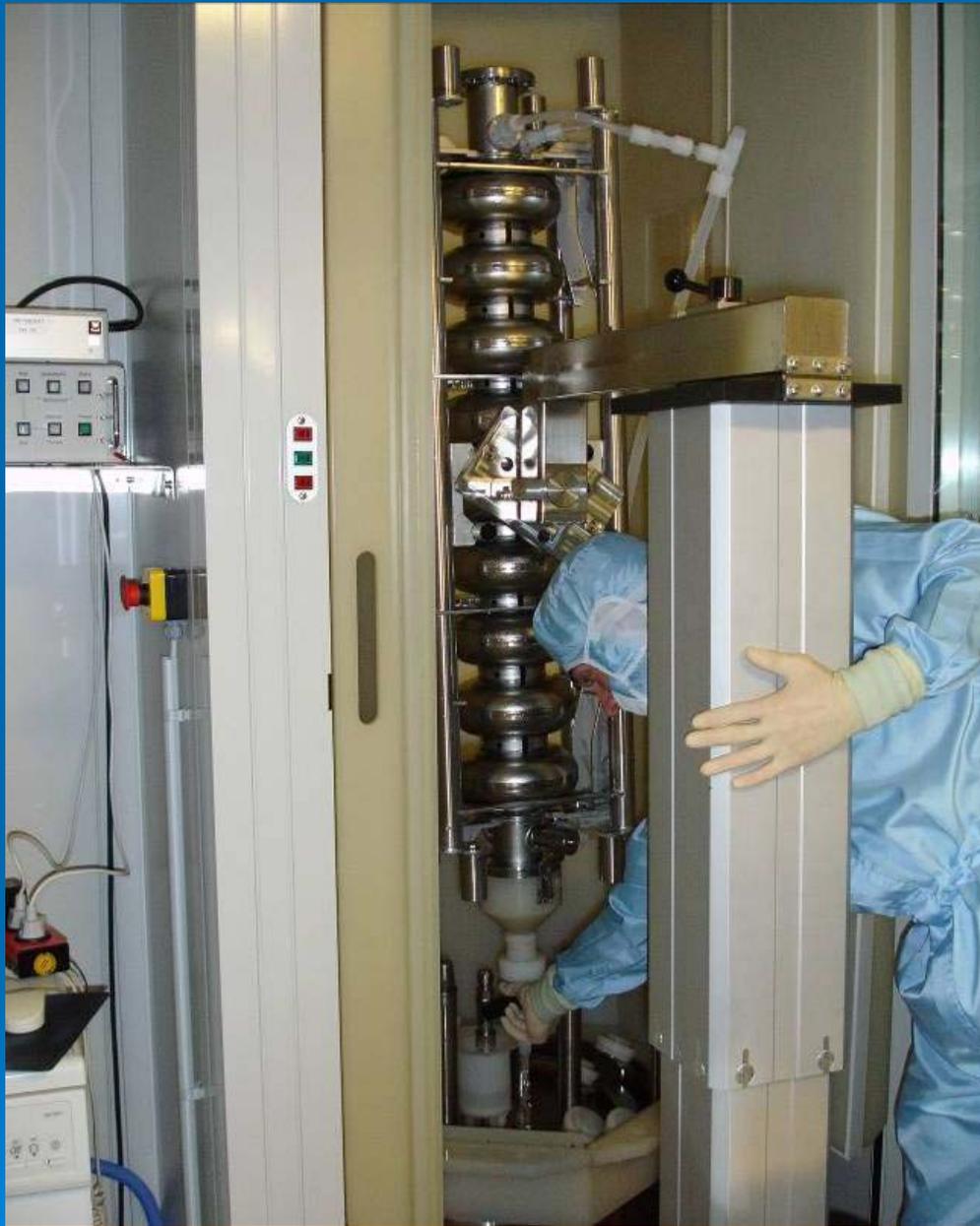
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Assembly of accessories



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Alcohol rinsing



HPR Stand inside the cleanroom

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Six times high pressure rinsing



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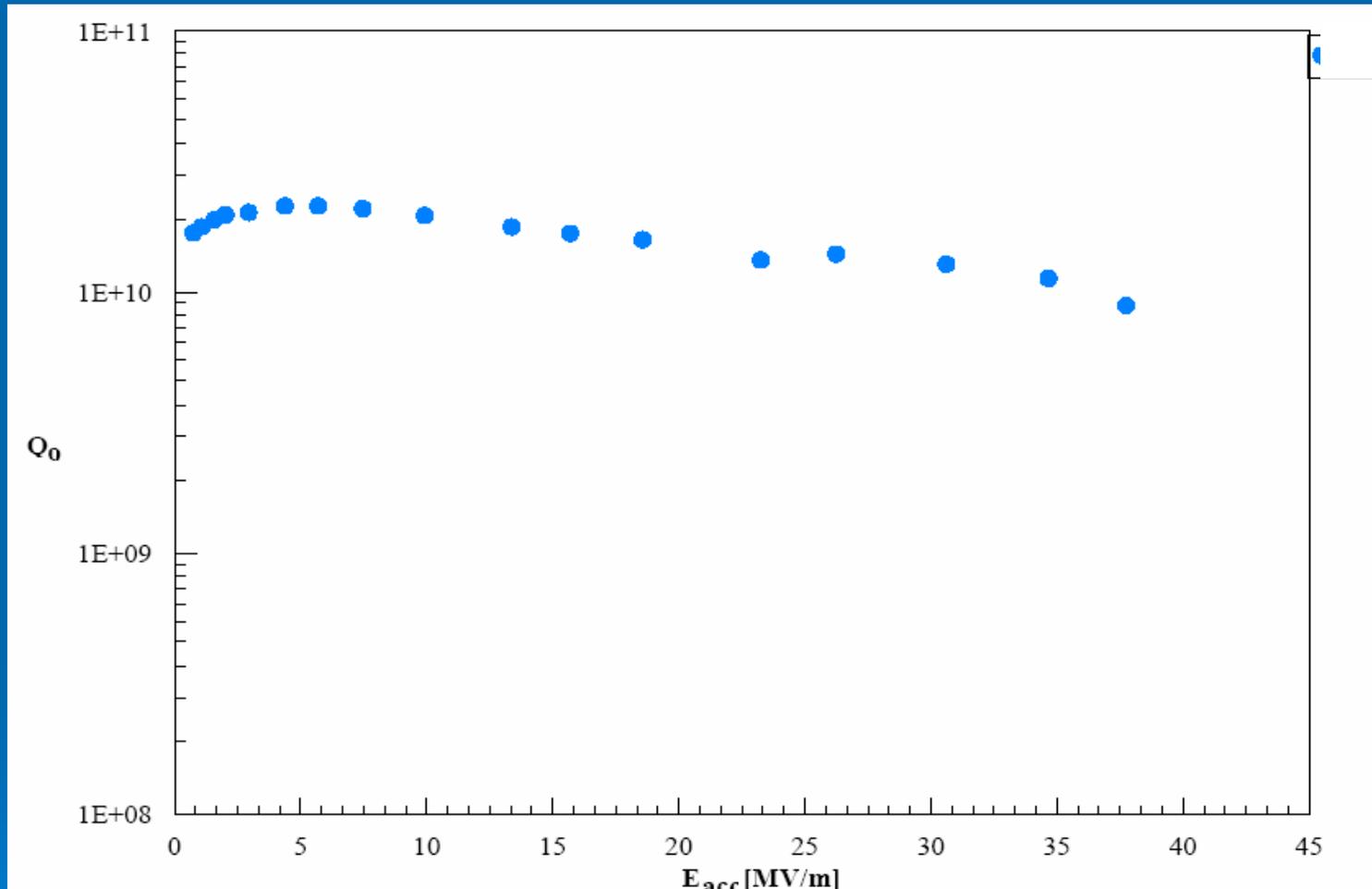
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120 C baking



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If ever things!!!! works perfect you will get



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## From vertical test to module

( Shortened version there are more steps than shown here necessary

Back to clean room

Install FEM (in situ bead pull measurement)

Tank welding

Remove FEM

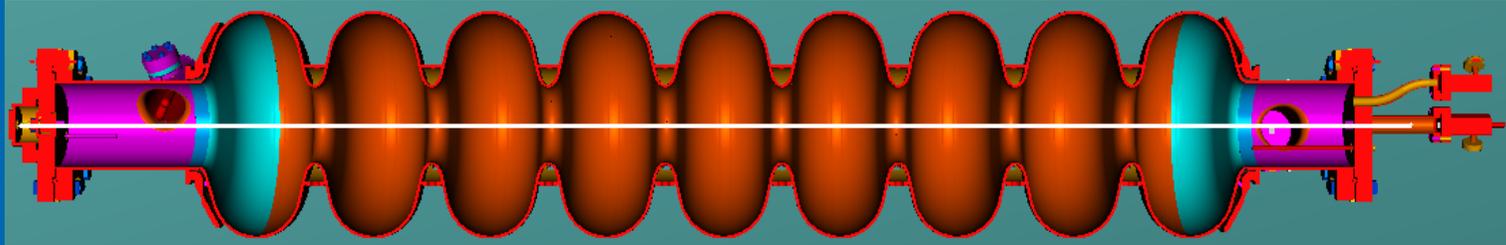
Install Antennas

High pressure rinse cavity

Install power coupler

Assemble of module

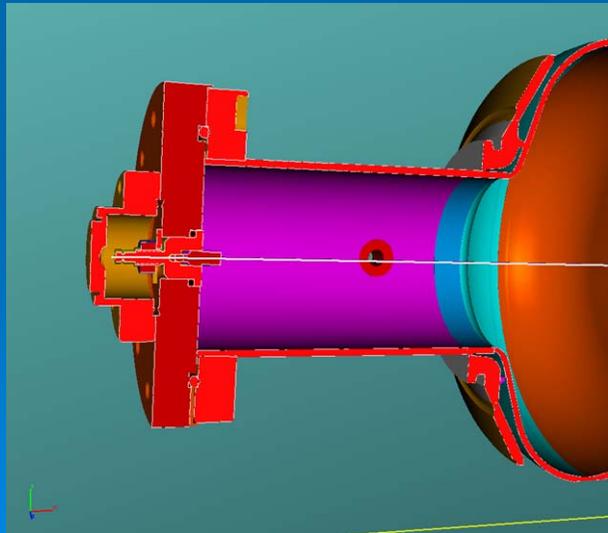
# 1) Cavity assembly for vessel weld



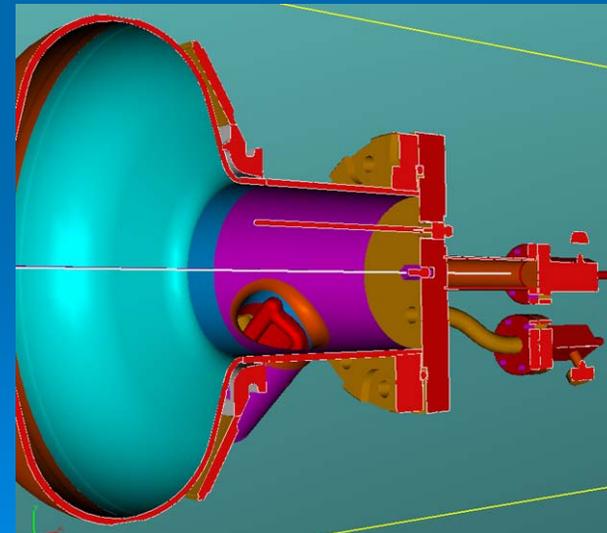
Cavity with FMS, Teflon tube on cavity axis

## Requirements to the FMS:

- Cleanroom class ISO 4 (ASTM: RK10)
- Protect inner surface of cavity from ambient air
- No desorption from FMS (Teflon tube and clamping system)
- Vacuum tightness

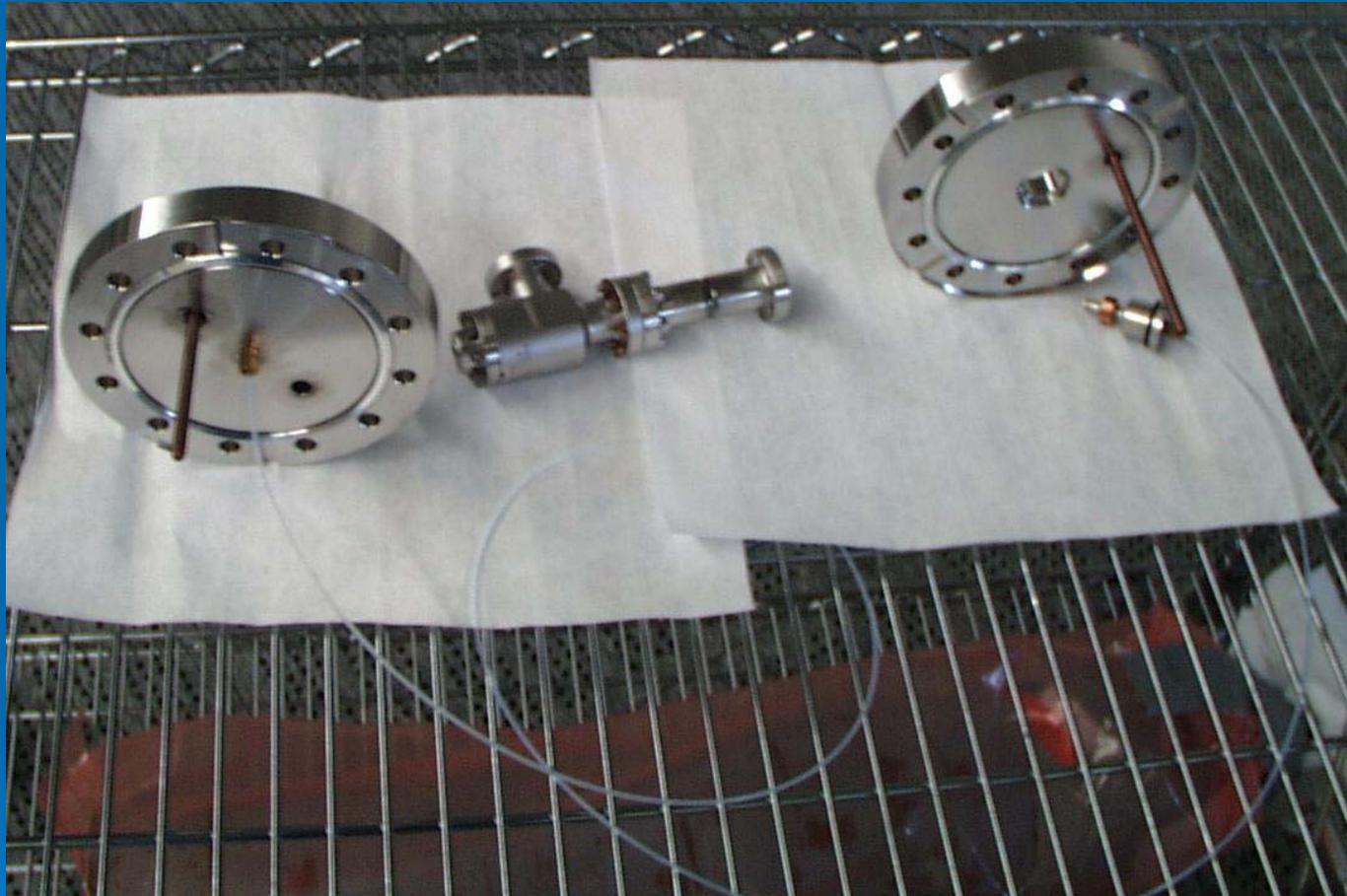


Cavity long end group



Cavity short end group

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谢谢大家

会议愉快

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