Cleanliness techniques

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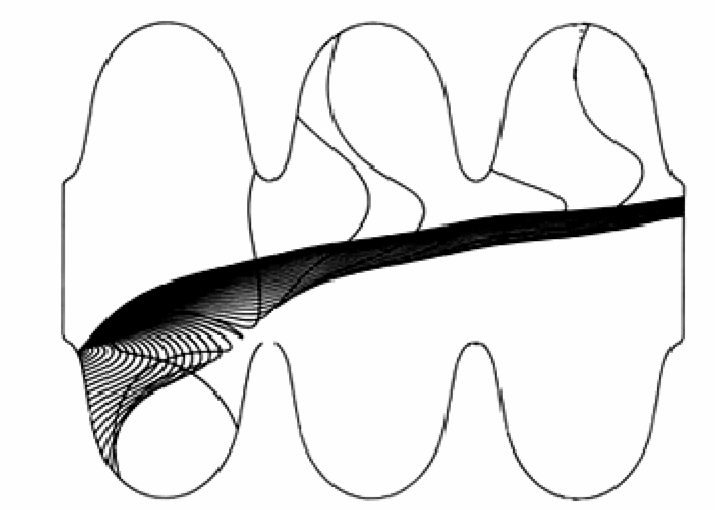
DESY

- Motivation: Field emission, particles, contaminations….
- Present picture of field emission
- Cleanroom technology: “Standard”
- Cleanroom technology for srf applications
- Alternative cleaning approaches
- Open questions + Summary

Motivation

- Major limitation of the last years in multi-cell cavities, especially in beam operation:



- **Field Emission!!**

- P. Kneisel + B. Lewis, SRF Workshop1995:

  „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."

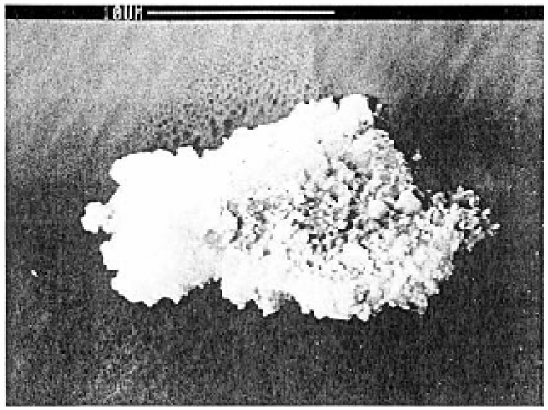
  "It is generally accepted that the field emission behavior of a niobium cavity reflects the level of cleanliness of the superconducting surfaces subject to the rf-fields."

Motivation

- Improved **clean** preparation techniques allowed an increased field emission onset:
- Typical (good) onset of field emission at 1.3 GHz
  - single-cell cavities: $E_{acc,onset} > 30$ MV/m
  - multi-cell cavities (vertical + horizontal): $E_{acc,onset} \approx (20 - 25)$ MV/m
- Rare multi-cell cavities with no field emission above $E_{acc,onset} > 30$ MV/m
- Cleanliness becomes more and more important for
  - i) cavity fabrication process
  - ii) all components of the beam vacuum system

Present picture of field emission: observations

- Metallic (conducting) particles of irregular shape; typical size: 0,5 - 20 µm
- Only 5% - 10% of the particles emit
- hydrocarbon contamination of the vacuum system



- 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

- No substantial difference in rf and dc behaviour

Present picture of field emission: model

- Protrusion-on-protrusion model explains the experimental observations
- Modifications of A_{FN} and β by adsorbed gases and oxide layers
- Activation of emitters between 200C and 800C by modification of the boundary layer
- Firing >1200C suppresses emission

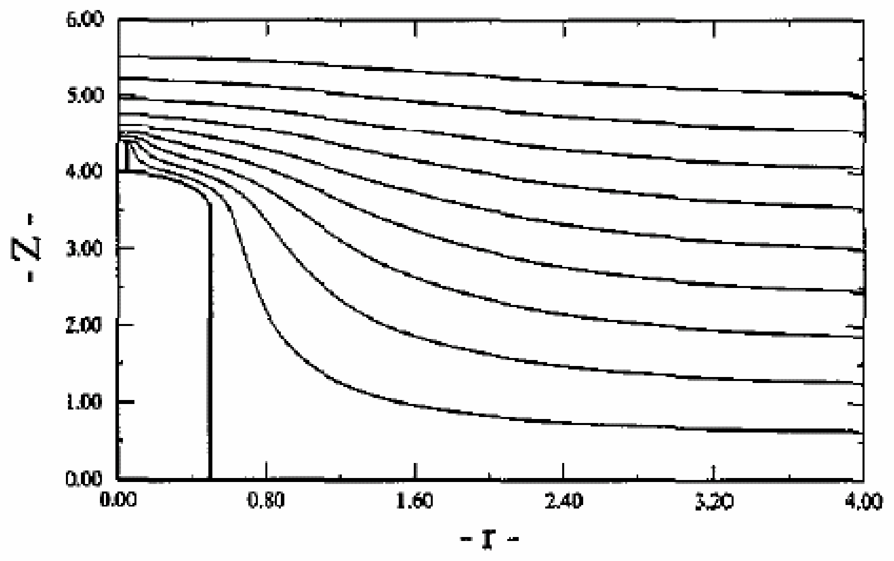


Figure 12. Calculated equipotentials for two superposed hemispherically capped cylindrical projections.

Few fundamentals of contamination

- Cleaning: Overcome adhesion forces of particles
- The adhesion forces depend on:
  - material, roughness, electrical charge, hardness of particle and 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, formation of potential, “zeta pot”
  - electrostatic

  (- chemical bonds)

Cleanroom technology: “Standard”

- **Specification of the contamination required !!**
- For cavity applications critical contaminations:
    - particles
    - hydro carbons
    - sulfur (EP process) ?
    - ???
- Task:
  Define and install the appropriate clean environment.
- Problem:
  Surface conditions (compared to semi-conductor industry) poorly known!
  => No investigations of the sensitive inner cavity surface possible !

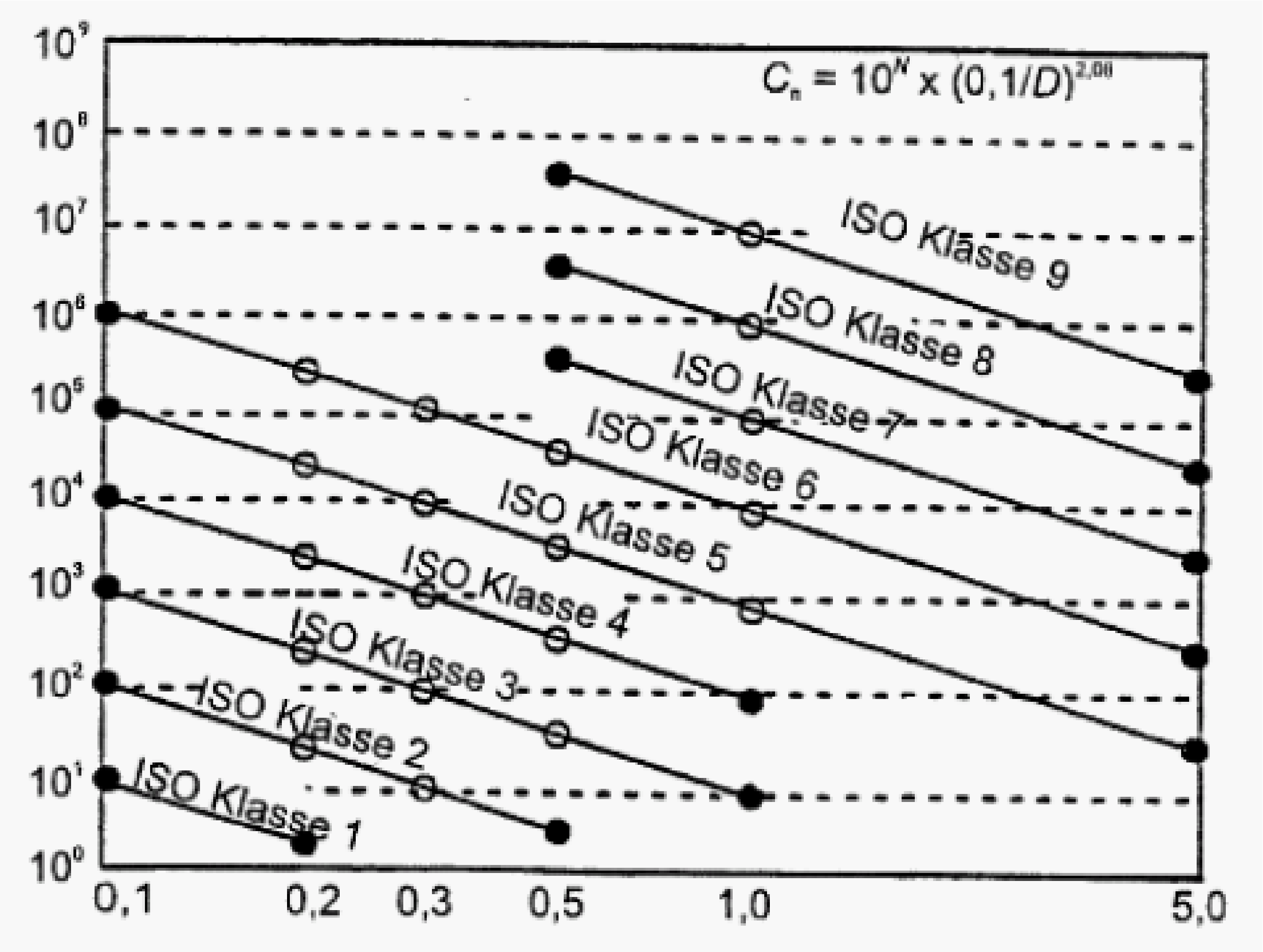
  - samples → very valuable, but bad statistics

Specification of Contamination

„Contamination has o be specified:

☐ Cleanliness class	Air – Surface (ISO – 14644-1)
☐ Temperature – Humidity	Air
☐ Noise Level	Room
☐ Vibration	Equipment - Building
☐ Molecular Contamination	Air (AMC) – Surface (SMC) (14644-8)
☐ Electrostatic Charge	Surface
☐ Magnetic field	Air

Cleanliness Classes according ISO14644-1



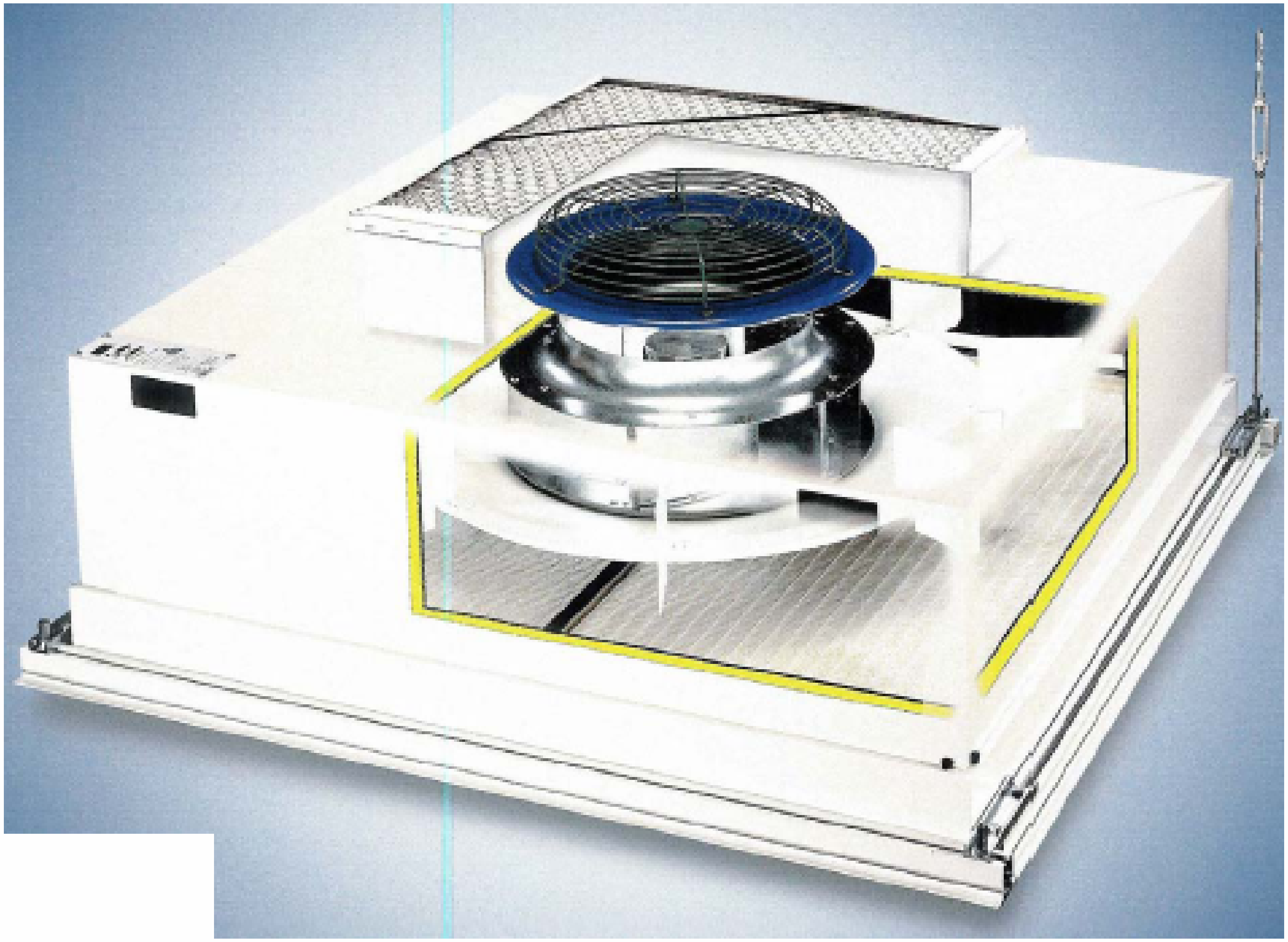
Particle Concentration per m^3

Particle Size in Micrometers

„Main – Topics“ in Cleanroom Technologies

- HVAC (Heating Ventilation Air Conditioning) – Systems
- Filtration Technology of Air – Gases and Liquids
- Processes under „clean“ – Conditions:
  - Vacuum–, Temperature- and Wetprocesses
- Personal – Clothing – Behavior in the Cleanroom
- Equipment which is suitable for Cleanroom use
- Cleaning – and Service processes of the Cleanroom and the Equipment

FFU - Filter Fan Unit - Technology



Daldrop + Dr.Ing.Huber

Measurement technology for Contamination

☐ Particles

in Air - available

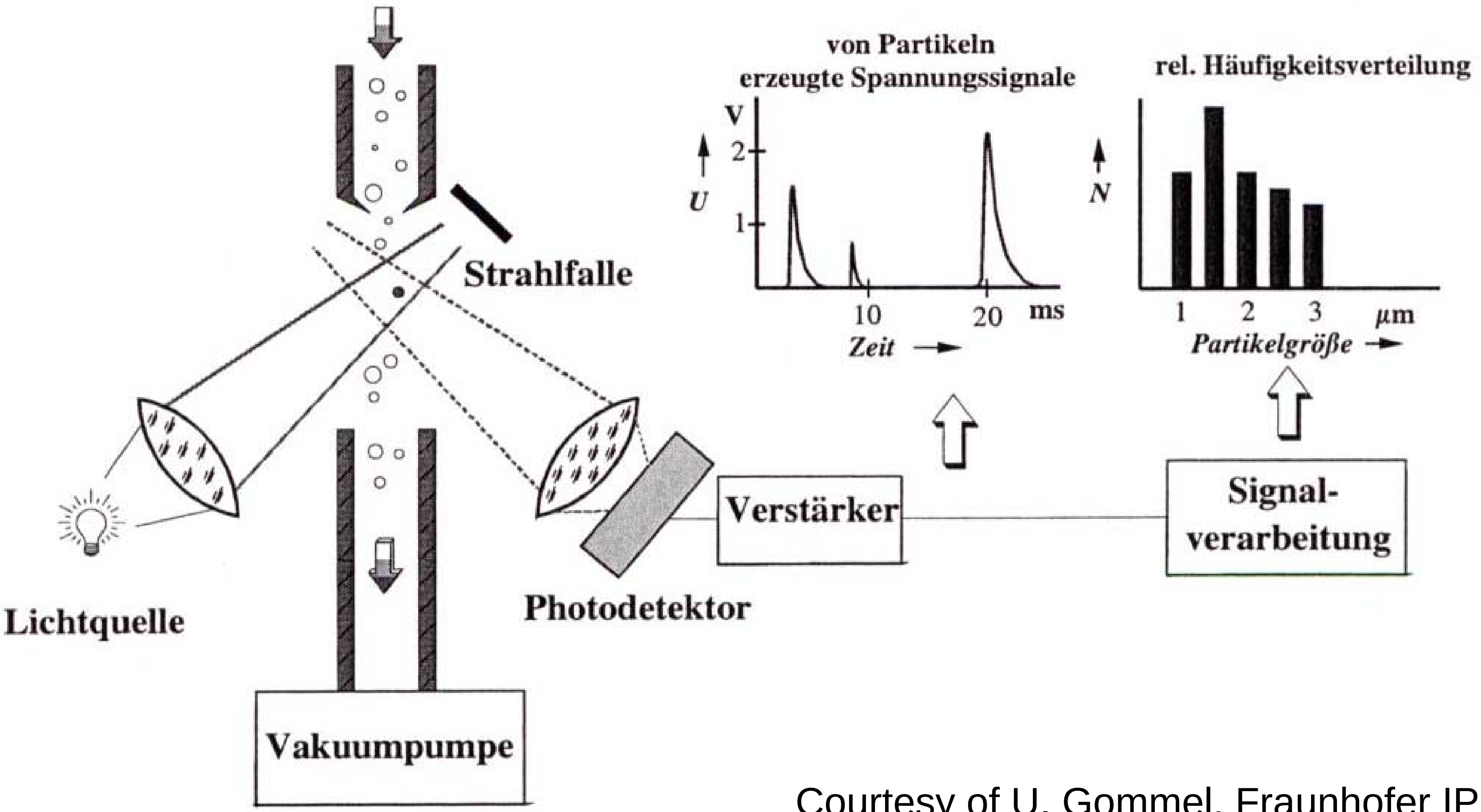
in Liquids - available

on surfaces - for big particles available

☐ AMC / SMC - partly available

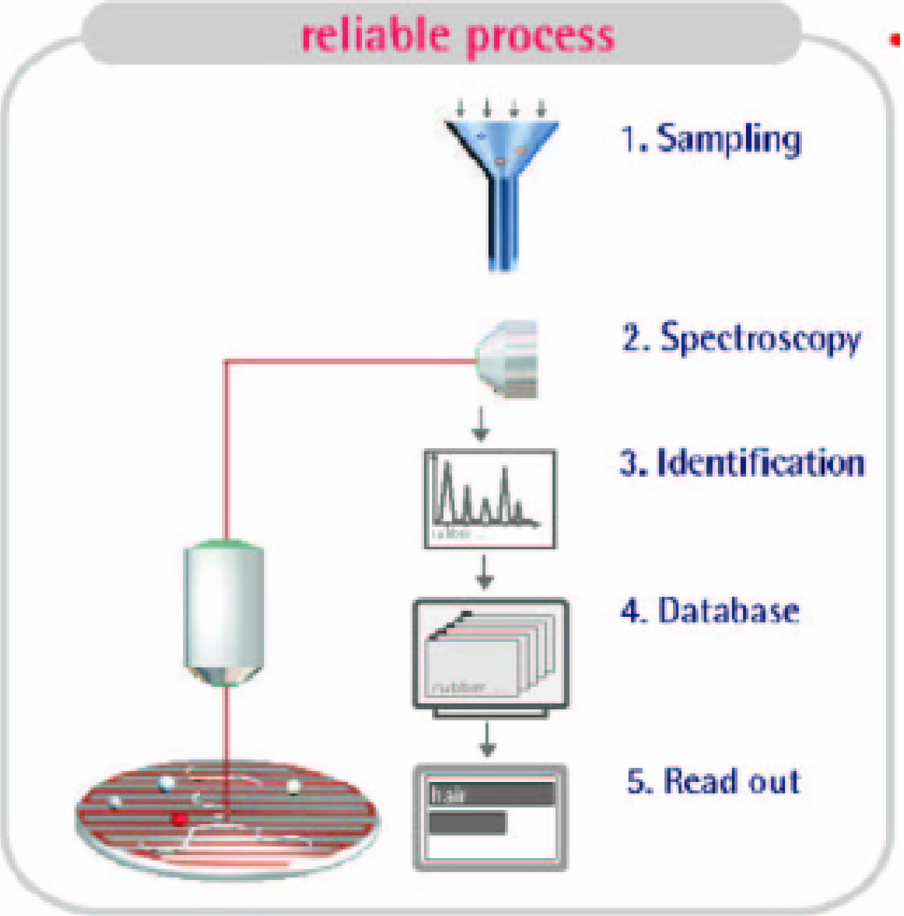
☐ Electrostatic Charge - available

Principle of optical particle counters



Courtesy of U. Gommel, Fraunhofer IPA

Particle – Measurement with Particle Analysis

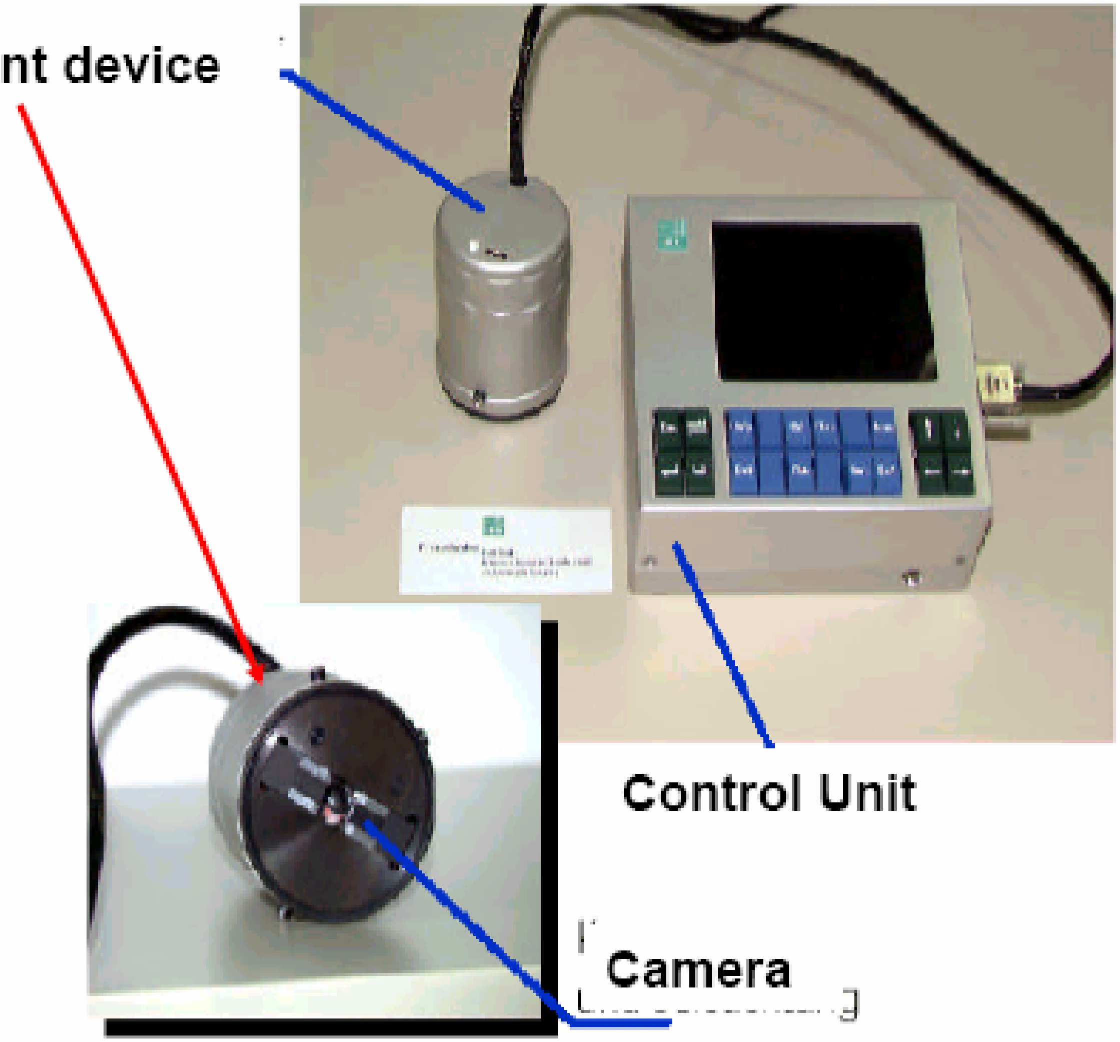


reliable process

1. Sampling
2. Spectroscopy
3. Identification
4. Database
5. Read out

Particle – Measurement on Surfaces

Measurement device

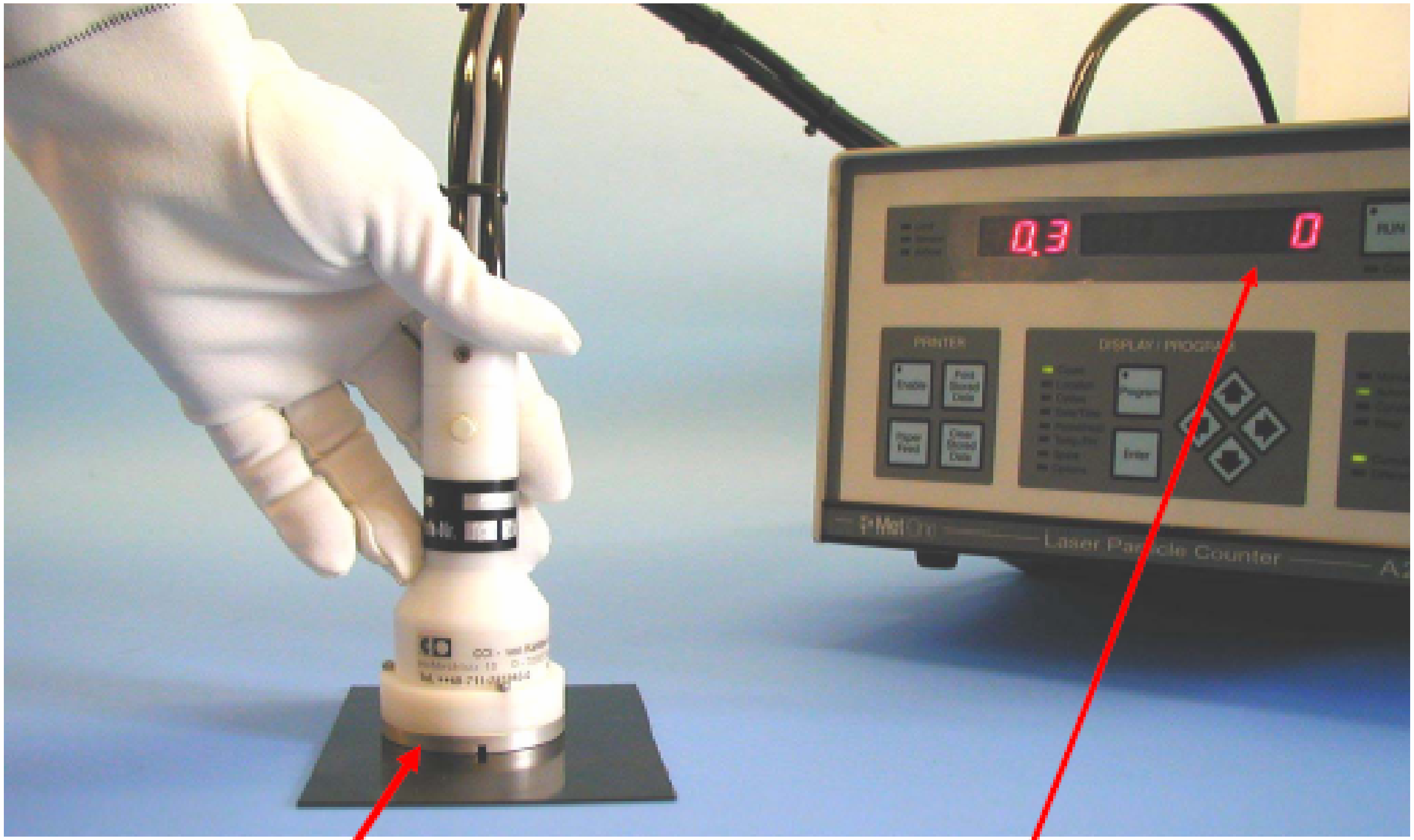


Control Unit

Camera

Fraunhofer Society

Particle – collection from surfaces and counting



Samplehead

Particlecounter

Flow Visualization around Equipment

“Standard” cleanroom technology: summary

- Contamination has to defined:
  - avoid and remove particles, hydro carbons, …
  - status of surface contamination often undefined

  => practical approach: *strategy of universal cleaning* applied
- Components for high-end cleanroom technology are available

  => adopt it for srf cavity applications!!

  (Cleanroom technology is more than a laminar flow bench)
- Measurement technology of *usual* contamination is available

  => adopt as much as possible

  => additional dedicated solutions necessary!

Cleanroom technology for srf applications

- Dedicated process equipment:
  - US: Ultrasonic cleaning (very important for pre-cleaning and components)
  - BCP/EP: etching/electropolishing
  - HPR: High pressure rinse
  - pumping systems, leak check and venting installations

  - tooling for handling and assembly

  - furnaces: 120 C “bake”,
    (800 C firing,)
    >1200 C postpurification with getter material
- Alternative cleaning approaches:
  - Megasonic
  - Dry-ice cleaning

Chemical etching and electropolishing

- Final chemical etching or electropolishing

  ↓ ↓

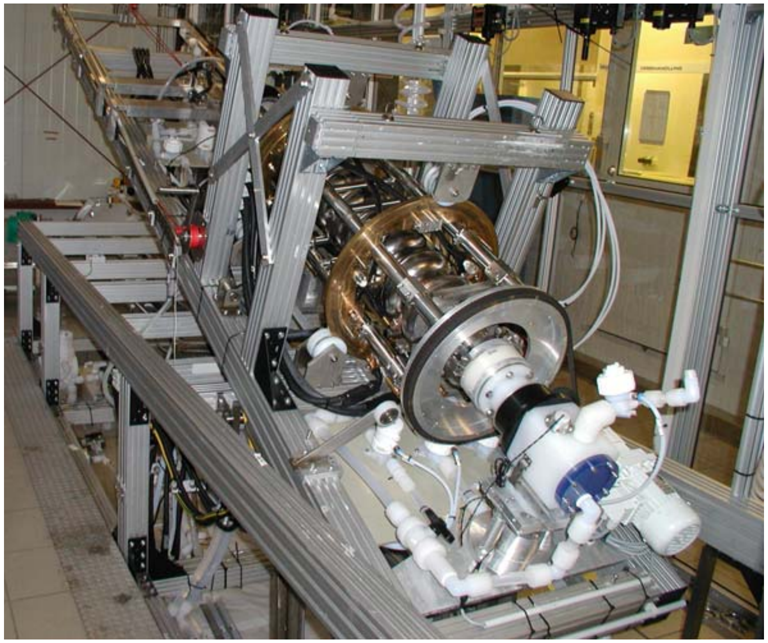
  BCP 1:1:2 HF : H_2SO_4 with volume ratio 1:9

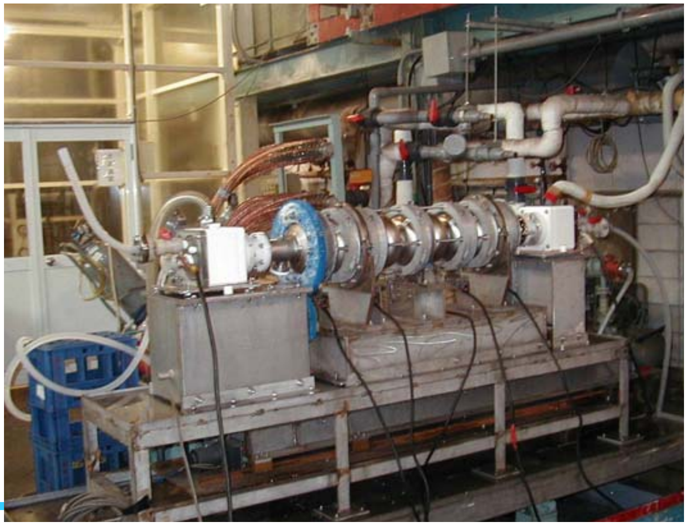
  - no cleaning, but surface removal (typically final 10 - 40 µm)
  - no (weak) removal of e.g. grease, plastics,
  - closed system with integrated DI-/pure water rinsing
  - acid quality: “pro analysi” or better

- Open Questions:
  - Which level of acid quality and particle filtration necessary?
  - Which “clean” environment necessary?
  - Alternative acid mixtures? Comparison of BCP 1:1:1 vs. 1:1:2 ?
  - stability of EP-mixture (HF degassing)

- hot water rinsing (better solubility, better drying)

BCP and EP facilities





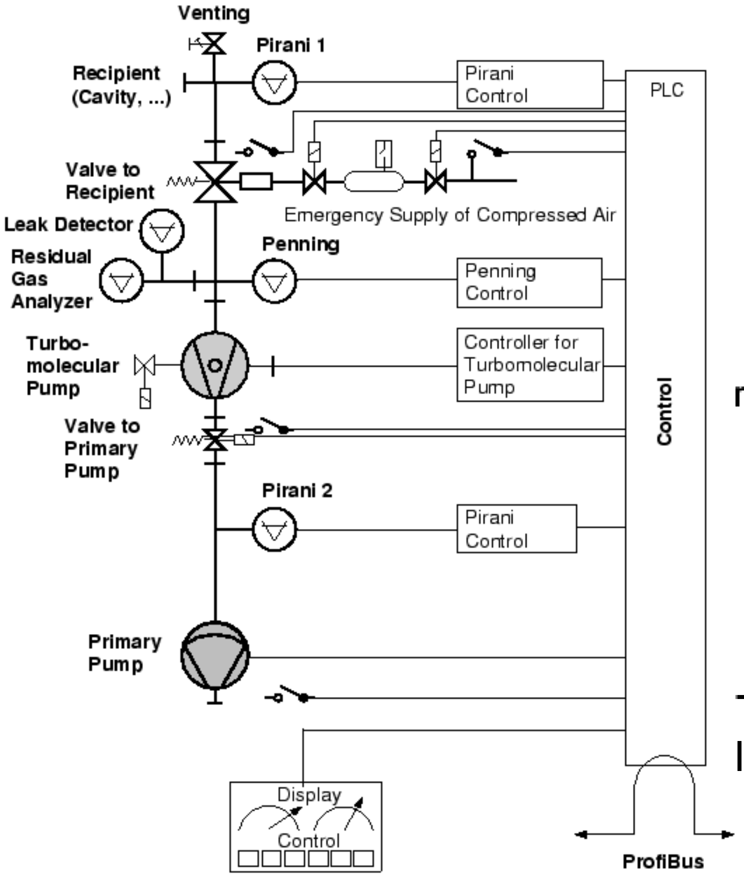
High pressure rinsing

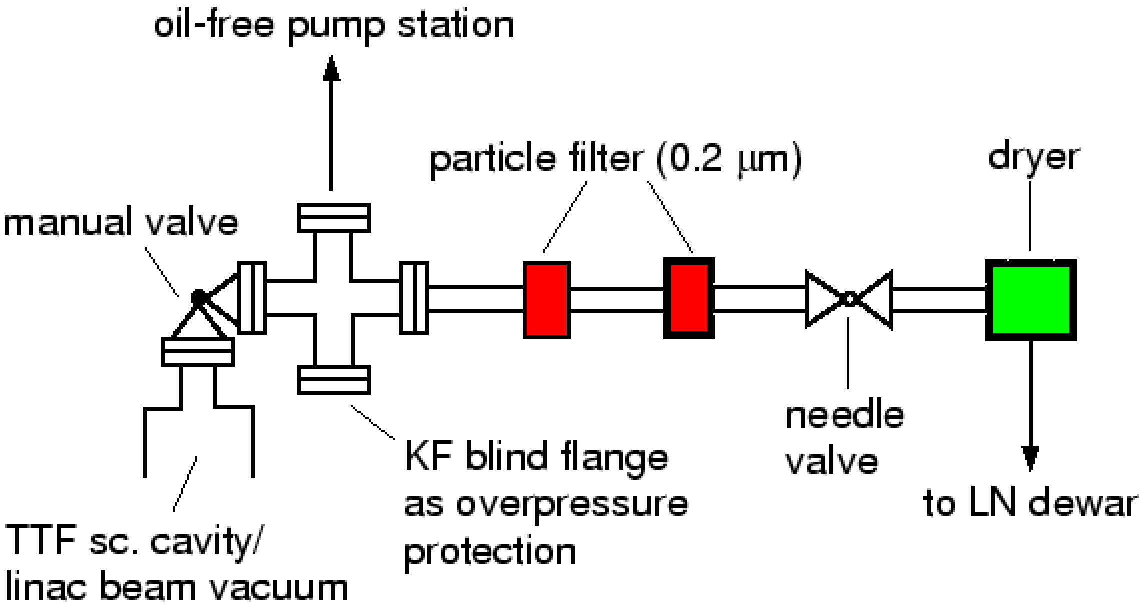
- High pressure rinsing (cleanroom cl.10 - 100)
  - inside rinsing
  - ultra pure water with p = (80 - 150) bar
  - outside rinsing maybe helpful to avoid transport of contamination into the assembly area

- improved high pressure rinsing systems:
  - no moving parts inside cavity
  - higher pressure
  - different jet shape
  - rinsing of longer units possible?
- check of particles (+ TOC) of HPR water
  check of drain water as QC of rinsing effect ?

Pumping systems, leak check and venting

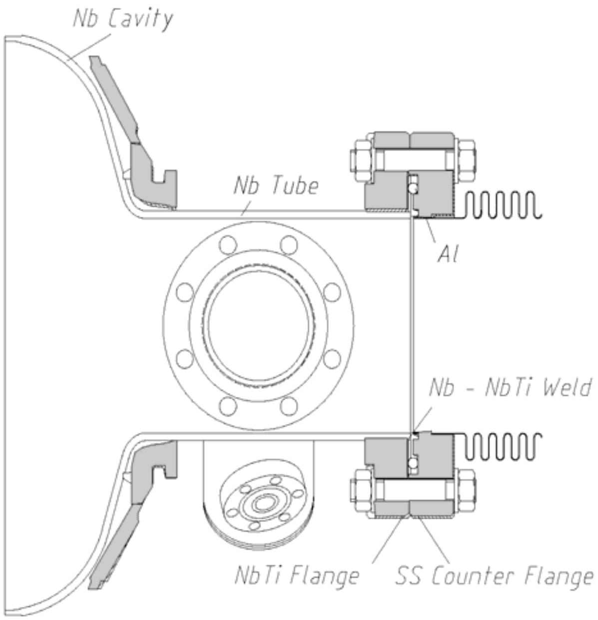
- leak check + venting (cleanroom cl.10)
  - oil-free pump stations with leak check and residual gas analyzer
  - laminar venting with pure, particle filtered N_2 or Ar



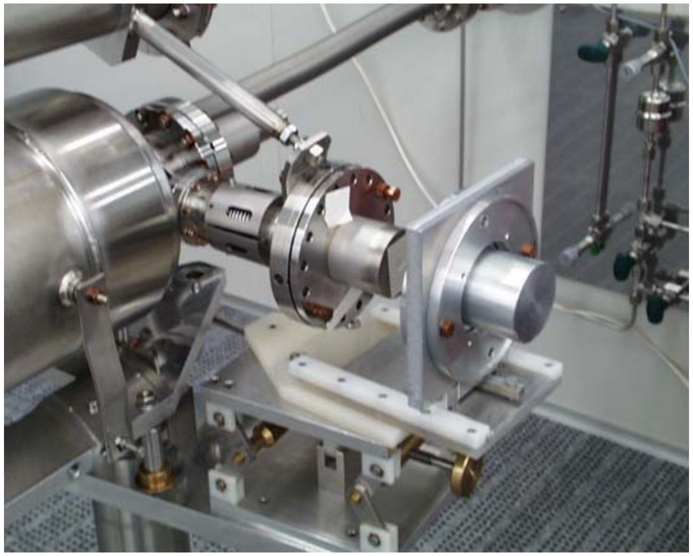


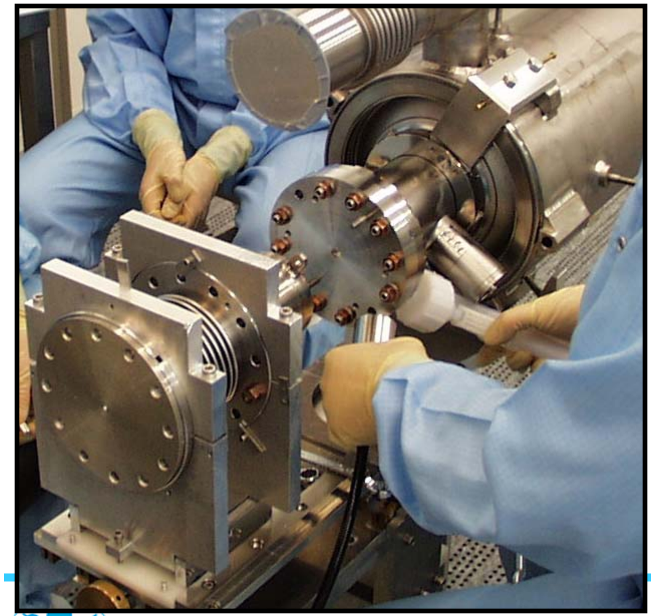
Handling and assembly

- assembly (cleanroom cl.10)
  - well cleaned components (flanges, power coupler, bolts, nuts)
  - well-trained and motivated personal
  - keep duration of actions at open cavity short
  - simple flange & gasket design e.g. NbTi-flange with Al-gasket
  - check of cleanliness?
- welding of flanges to avoid assembly work:
  - connecting cavities to a “super-structure”
  - e^- beam or Laser welding



Handling and assembly II





Furnaces / tuning

- Mostly not in cleanroom environment
- Avoid transport of contamination in ultra-clean area

- Future infrastructure:
  bring furnaces (800C / 120C) closer to the cleanroom

- Tuning:
  - often interrupts clean processing
  - tuning in cleanroom environment?

Alternative Cleaning Approaches

- Megasonic Rinsing (K.Saito et al.)
  - effective cleaning of sub-micron particles
  - development necessary:
    better transmission of power ⇒ (small) oscillator inside cavity
    transportation of particles ⇒ high flow rate
- Dry-Ice Cleaning (Poster of A. Brinkmann et al., DESY)
  - effective cleaning of sub-micron particles and film contamination
- Others:
  Laser, Plasma, UV light,
  hot steam etc.
  ⇒ no activities ??!

Next steps + major open questions:

- Quality control and assurance of HPR:
  i) check of particles and water quality of HPR supply water
  => ongoing work, good progress
  ii) practical approach, how to judge about the quality of final cleaning
  => e.g. Is particle counting of drain water useful?
  New clever ideas for sample experiments?
- simplify procedure and components with respect to cleanroom work
- cavity cleaning option before module assembly necessary/helpful
- optimal surface treatment with respect of field emission
  - BCP vs. EP; which mixture?; HF degassing of EP mixture
- (drying of cavities)
- ????

Summary

- Standard cleaning and assembly procedures allow high quality cavity performance, but:
  Field emission (= dark current) is still the main limitation, if usable gradients above 20 MV/m in multi-cell accelerator cavities are required
- Further improvements of standard techniques, quality control and development of alternative approaches necessary!

- Thanks to Th. von Kahlden, N. Krupka, U. Gommel and many other colleagues for their help!

Literature

- Some literature:
  - E. Ciapala et al., SRF Workshop 2001
  - W. Kern ed., Handbook of Semiconductor Cleaning Technology, 1993
  - 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
  - L.Gail, H.P.Hortig, Reinraumtechnik (in german), 2002
- In general:
  Proceedings of the SRF Workshops

What is manufactured under Cleanroom Conditions

- ☐ **Semiconductor 150 to 300 mm Wafer**
- ☐ **Pharmaceutical- and Foodindustry**

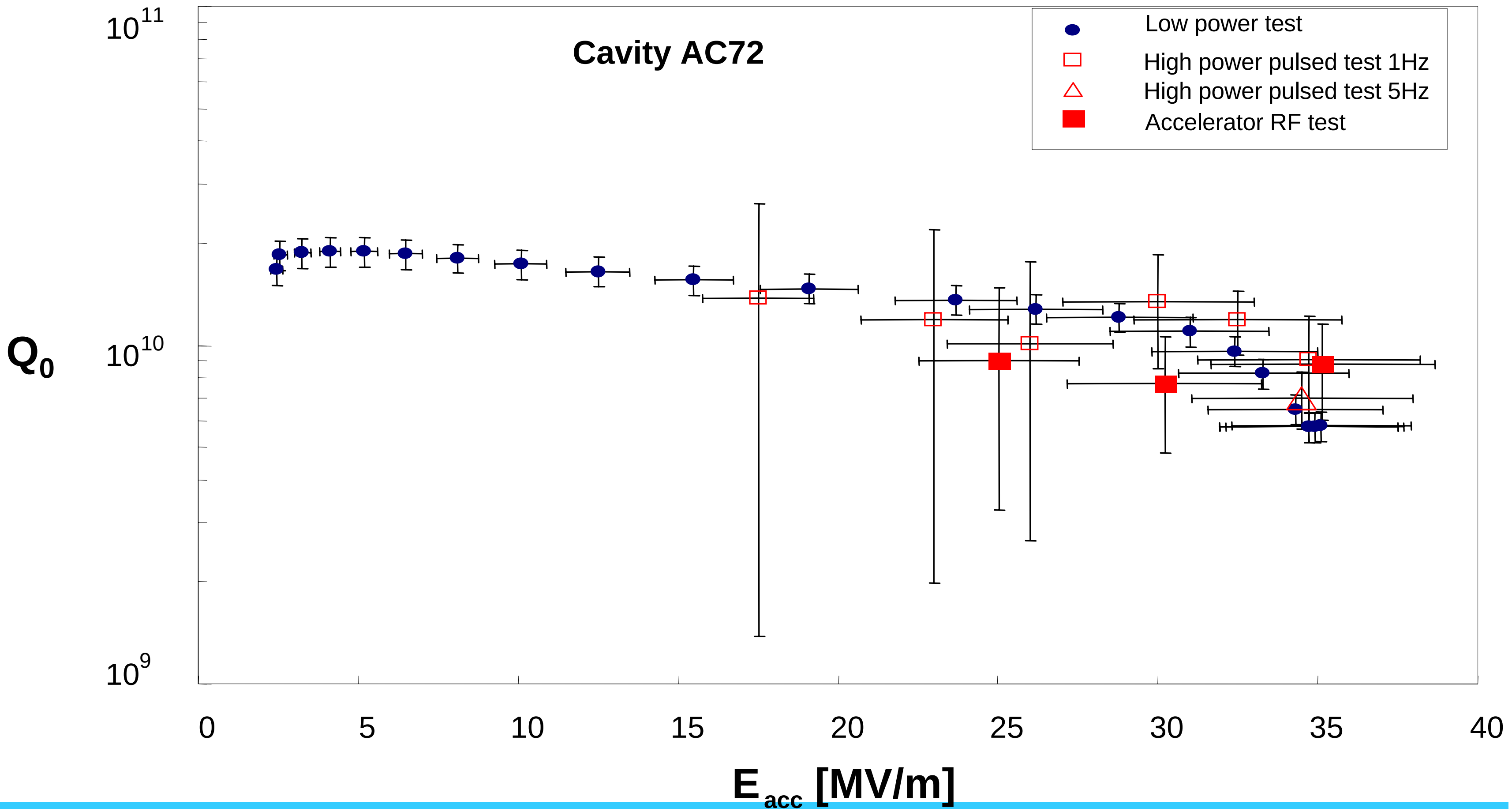
  Sterile areas in filling lines up to class 5 (Grade A)
  Medical devices in Grade C and D (class 6 to 7)
- ☐ **Biotechnology**
- ☐ **Materials**

  pure / clean plastics, Gases and liquids for the Semiconductor Industry
- ☐ **Others**

  Micromechanic; Coating Technology (Lenses); Circuit Boards; Spacetechnology; Carindustry (Carpainting, Windshilds, Breakesystems); Cleaning Technology

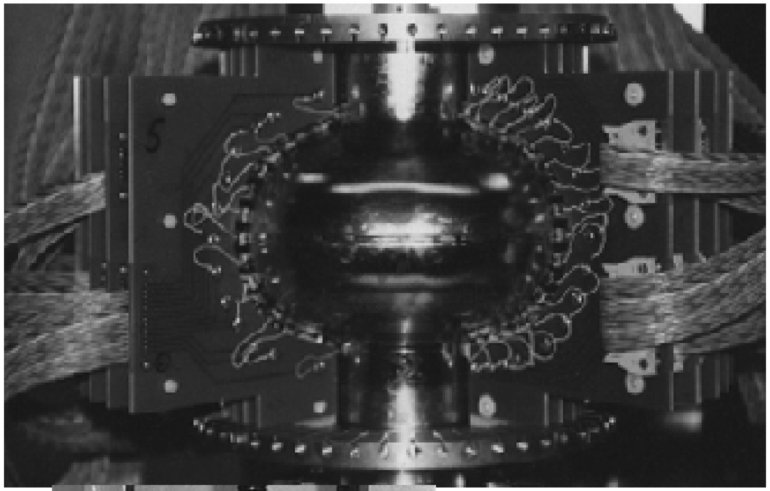
Introduction

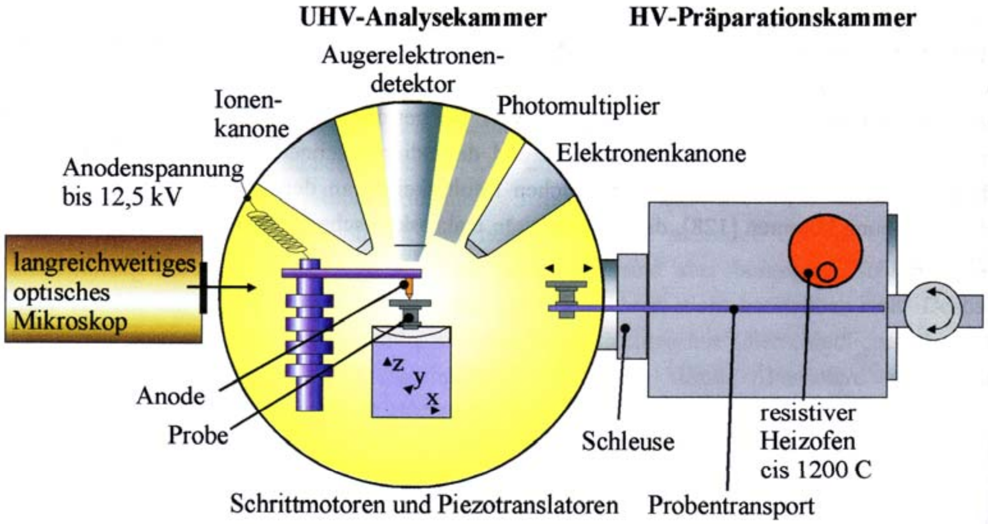
- 35 MV/m without field emission in e$^-$ - beam operation is possible !!

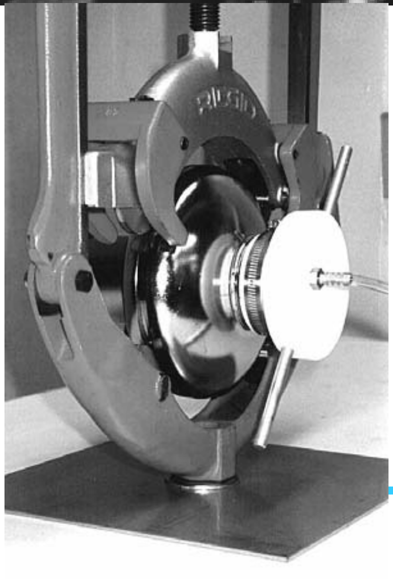


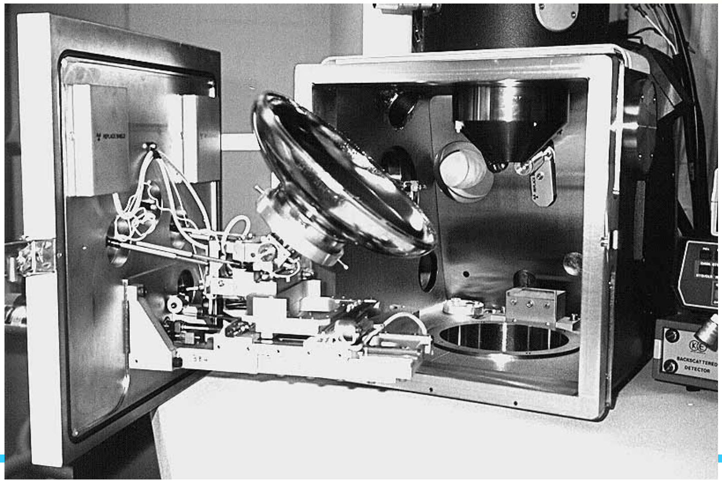
Present picture of field emission: instruments

- Some tools developed for field emission investigation



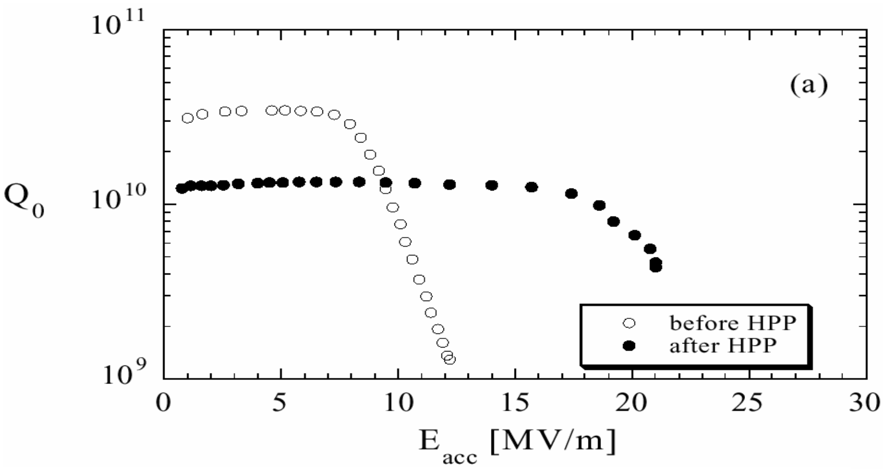


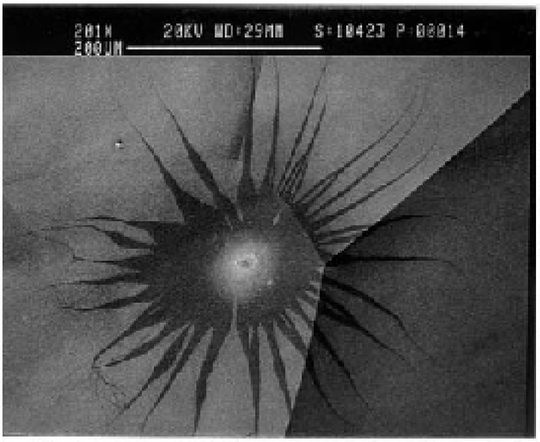


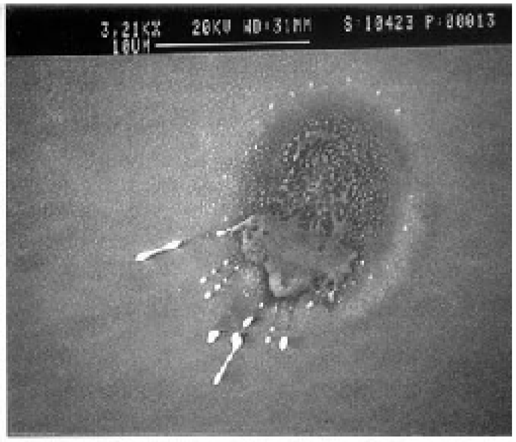


Present picture of field emission: processing

- Processing of emitters (“conditioning”) possible
  - i) rf and helium proc. with moderate rf power and cw-like operation
  - ii) high peak power processing with high rf power and short pulses
- Helium processing: i) modification of the adsorbed gases (≈ seconds)
  ii) explosive destruction (≈ subseconds; rare)
- High peak power processing (HPP): local melting leads to formation of a plasma and finally to the explosion of the emitter (model by J. Knobloch) → “star bursts” (Lichtenberg figures) caused by the plasma

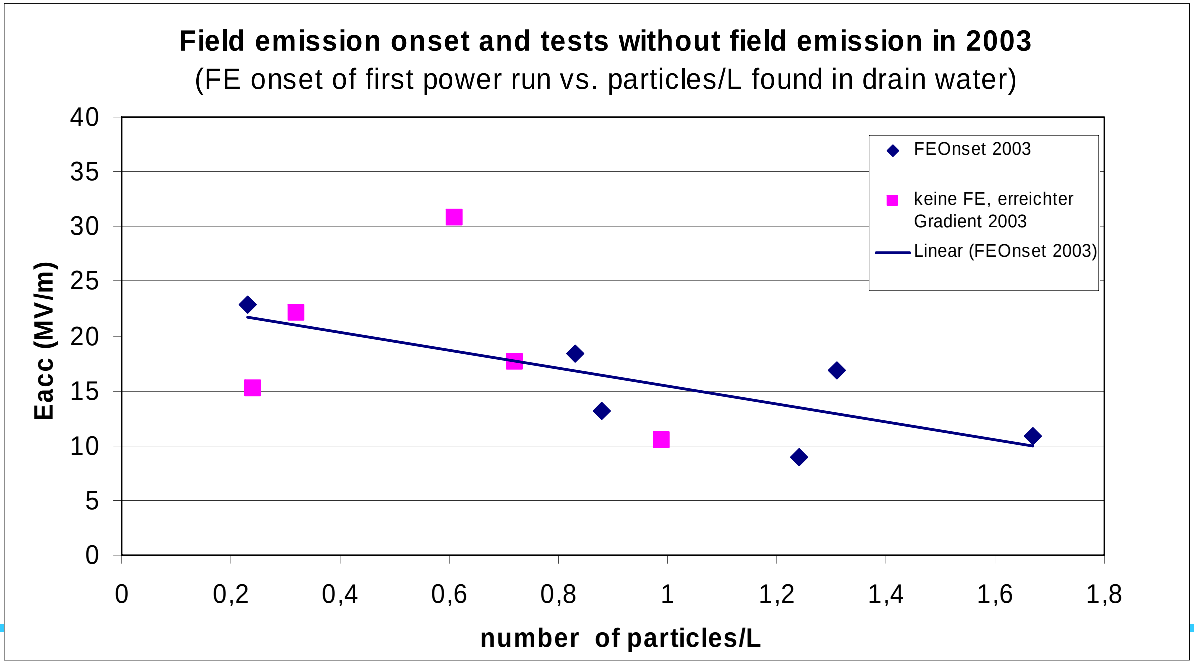






Standard procedures (ctd.)

- N-times high pressure rinsing (cleanroom cl.10 - 100)
  - check of particles (+ TOC) of HPR water
  - check of drain water as quality control of rinsing effect

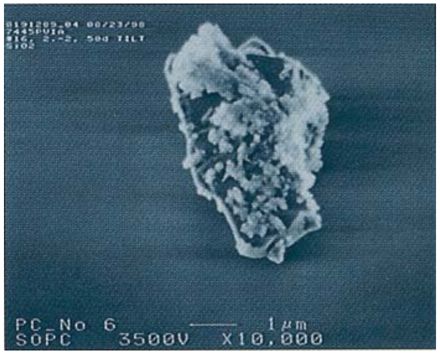


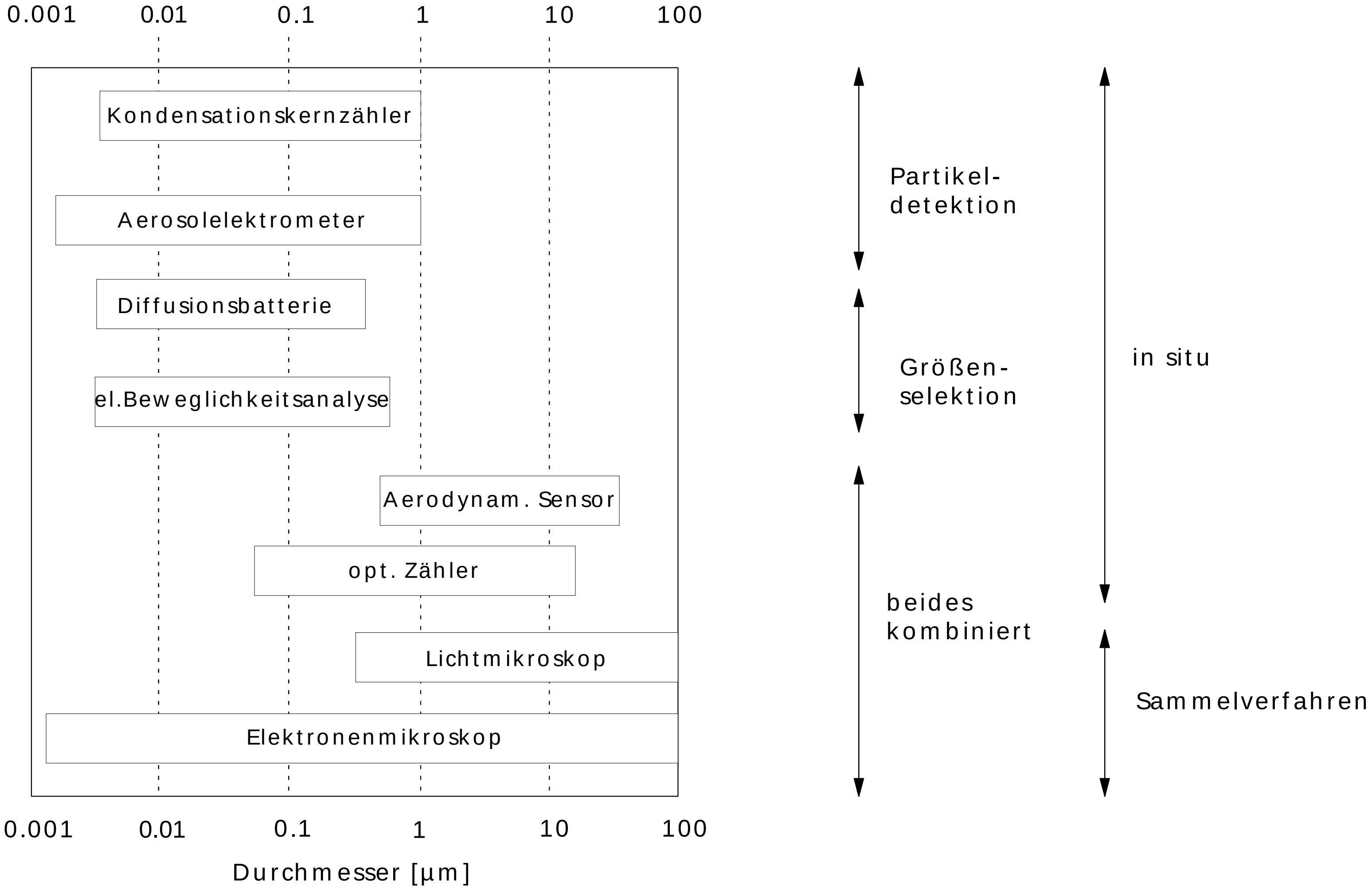
particles > 6 µm

Standard procedures: risk analysis

- Assembly:
  - TTF: 3 of 10 assemblies + 3 of 4 disassemblies after final HPR
  ☠ risk of contamination with particles
  reminder: most particles are created during opening bolt-nut connections!
- String assembly:
  ☠ no further cleaning of inner cavity surface possible
  ☠ risk of improper cleaning due to complex structure
- Venting:
  - TTF: 3 - 5 times vented between final BCP/EP and beam operation
  ☠ risk of contamination with particles? => no negative experience

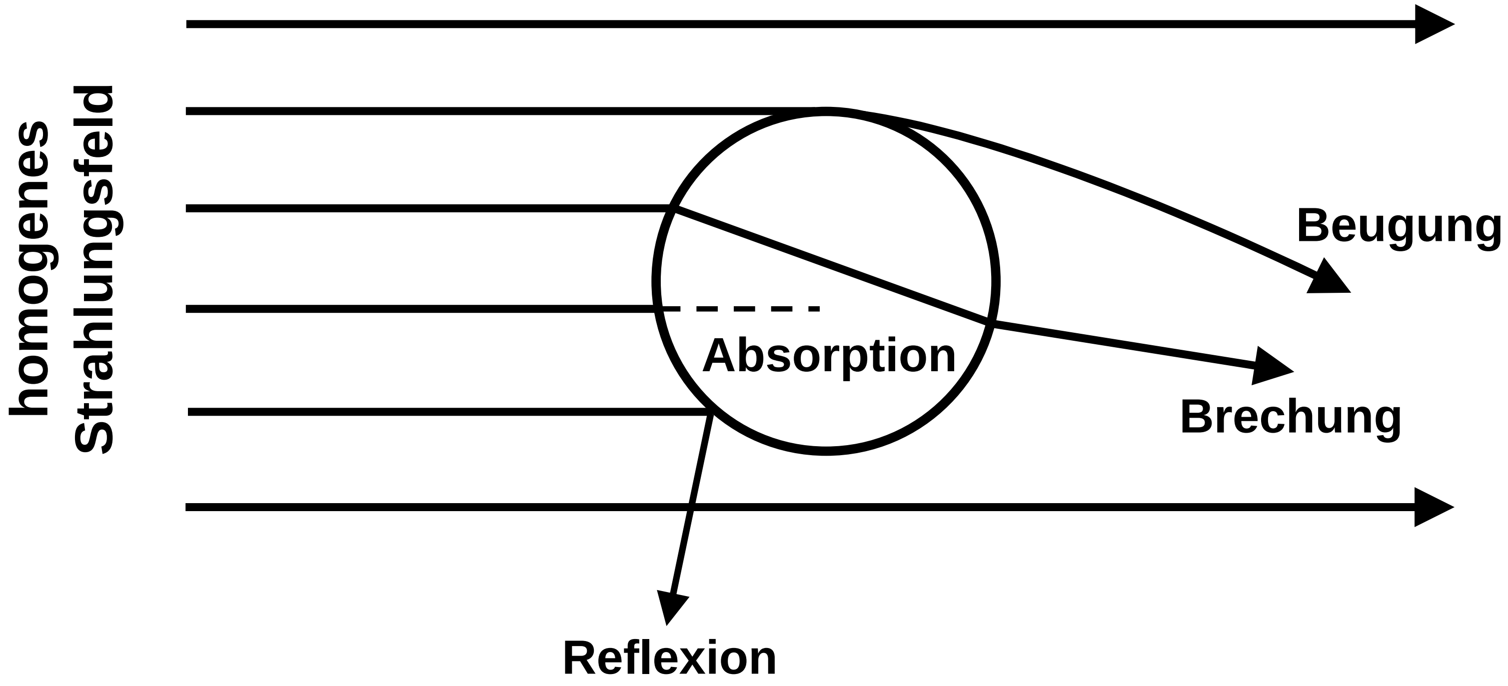
Principles of particle counting





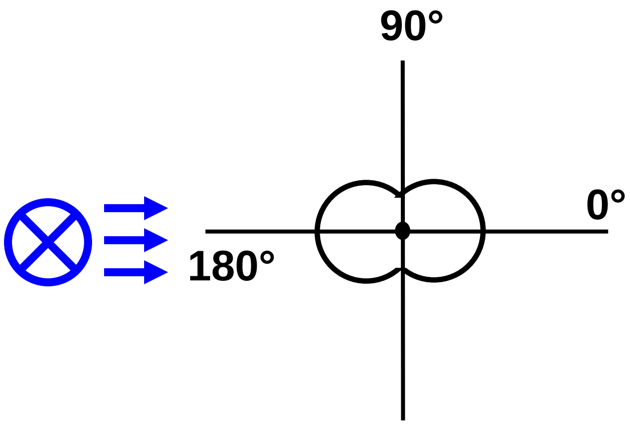
Theory of scattering at particles

- Kugelkörper



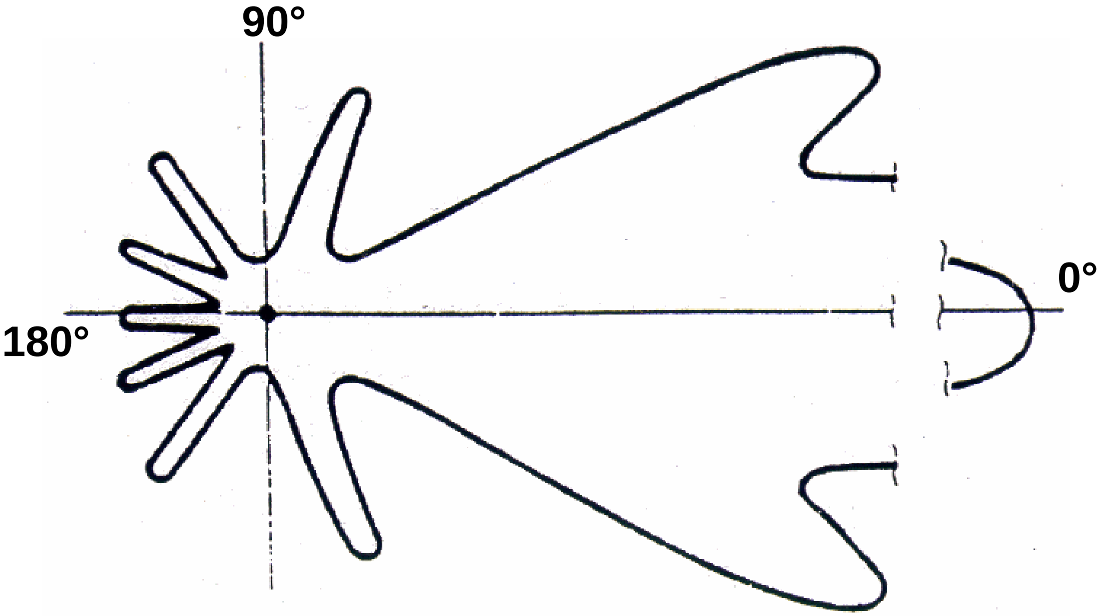
Scattered light – Rayleigh-/Mie-scattering

- Teilchen << λ



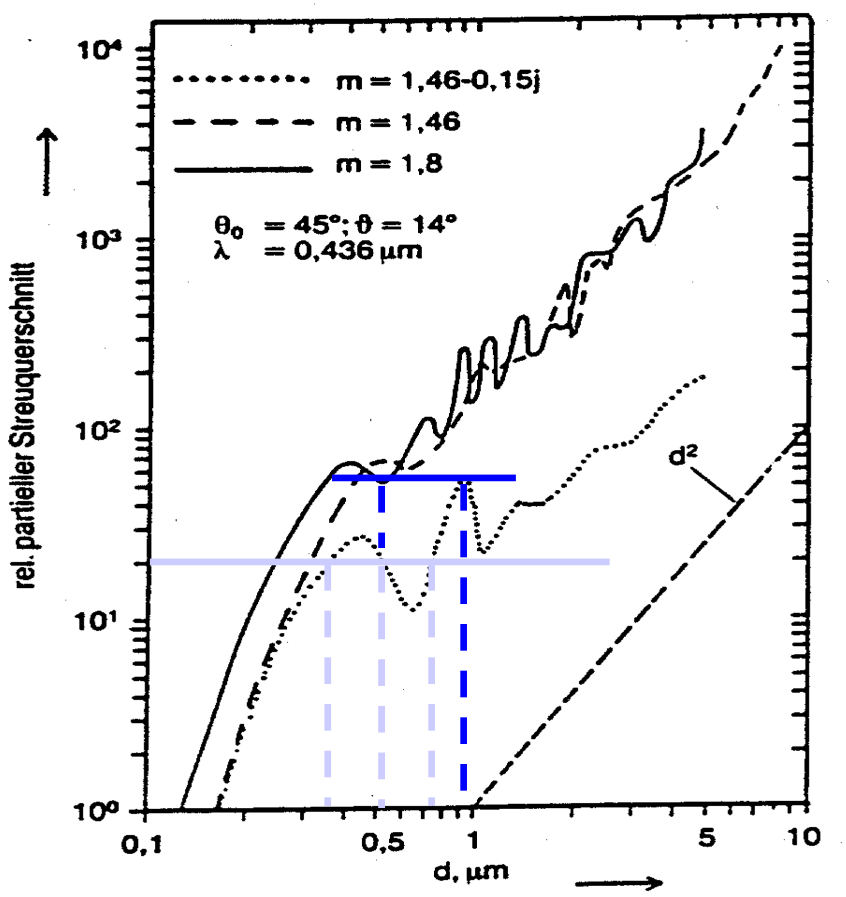
Rayleigh-Streuung

- Teilchen ≥ λ



Mie-Streuung

T & T: Streulichttheorie – rel. Streulichtintensität



- Mehrdeutigkeit
  - 0,35 µm ≠ 0,55 µm ≠ 0,75 µm
- Brechungsindex (Materialeigenschaft)
  - 0,55 µm ≠ 0,95 µm

Principle of optical particel counters – 90° set up