Basic studies for process parameter developments for EP / HPR / Snow cleaning

Detlef Reschke

DESY

SRF conference, Berlin, Sep 20th – 25th

- Electro Polishing
  - Process, Electrolyte, Residues
  - Surface morphology
- High Pressure Water Rinsing
- CO_2 Snow Cleaning

Thanks to all colleagues who provided my their data, pictures and information !!!

All missing and faulty information is completely on my side.

Electro Polishing: Introduction

- More than 20 contributions about EP on this conference

  => major topic of this conference

- **EP without HF**:

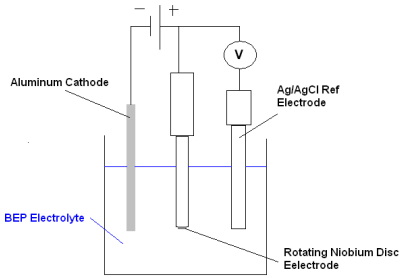
  => next talk by E. Palmieri

  - more activities at JLab (H_2SO_4 - methanol) and others

- no description of EP systems + EP cavity results !!!

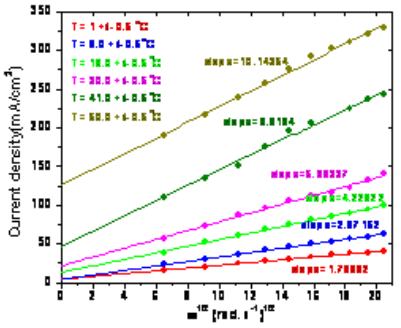
Fundamental analysis of EP process

- THPPO069 (F. Eozenou, CEA Saclay), THPPO060 (H. Tian, JLAB)



- analysis of diffusion coefficient of fluorine (applying a rotating disc set-up (RDE))

=> fluorine plays a key-role in the EP process



- Anodic polarization curves

=> determination of limiting current densities

- fluorine content measurement with EIS (El Spectroscopy)

=> aging of the electrolyte can be checked

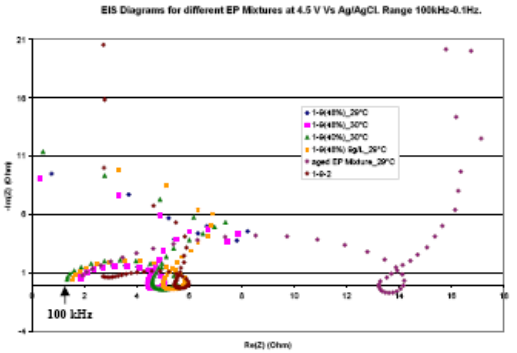


Figure 4: EIS diagrams for potentials on the plateau obtained for mixtures with different water concentrations. Features of the diagrams depend on the mixture's composition.

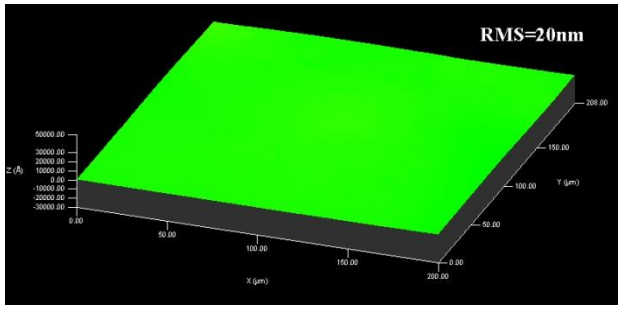
Fundamental analysis of EP process II

- "Surface Characterization…"; TUPPO086 (X. Zhao et al.)

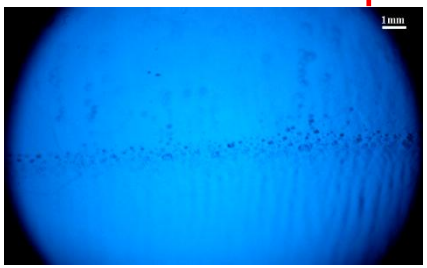
  => S and NbO found on witness samples

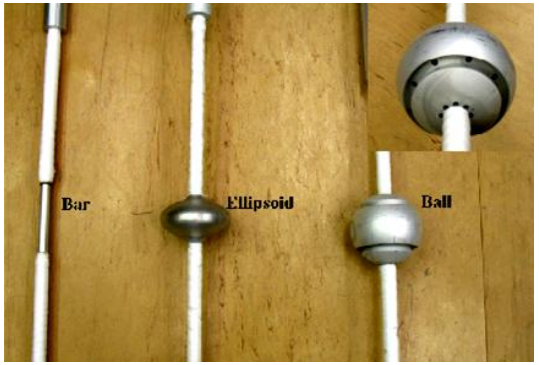
Buffered Electropolishing

- THPPO068 (F. Eozenou, CEA Saclay), THPPO097 (S. Jin, PKU/IHEP)
- proposed in the 1950th; further developed at JLab
- mixture of HF (conc. 48%), sulphuric (conc. 98%) + **lactic acids** (conc. 85%)
- fast removal rate (up to 4.6 µm/min)
- smoother surface than standard EP
- difference in EP mechanism => Fabien



- surface quality highly sensitive to cathode shape
- long term stability of acid ?





- in early stage of development for cavity application

Low Voltage EP

- THPPO070 (F. Eozenou, CEA Saclay)
- developed and under investigation + improvement at Saclay
- standard EP mixture, but low constant voltage of 5 V
- advantages:
  - less impurity contamination (probably Sulfur) after long EP
  - lower hydrogen contamination
  - higher life time of EP mixture
  - lower heat production
    => stable temperature w/o heat exchanger
    => vertical set-up for low frequency cavities possible
  - comparable single-cell results
- disadvantage
  - lower removal rate

Electrolyte Analysis

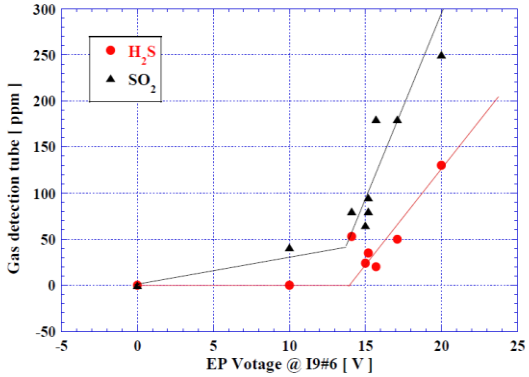
- THPPO081 (C. Hartmann, Henkel)
- Henkel, DESY (within CARE) in two studies since 2005
- acid quality control and quality management:
  - **Goal: Find a simple method and instrumentation**

- contaminations of pure elements (metallic) and organics (softener) can be identified by ICP-OES and TOC
- small changes of the electrolyte + distinction between warm / cold mixture of components not detectable
- mainly elaborate + expensive lab methods

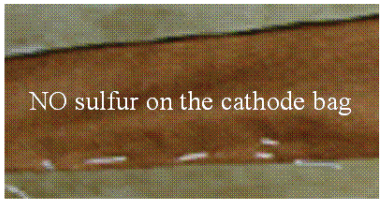
EP: Sulfur generation

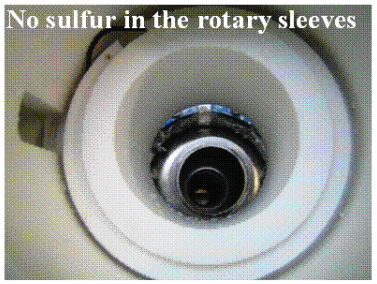
- "Sulfur Generation Mechanism" ; THPPO090 (K. Saito, KEK)

There is a threshold around 14V. When EP voltage reduced lower, S-contamination should be suppressed.



EP Voltage 17V





=>

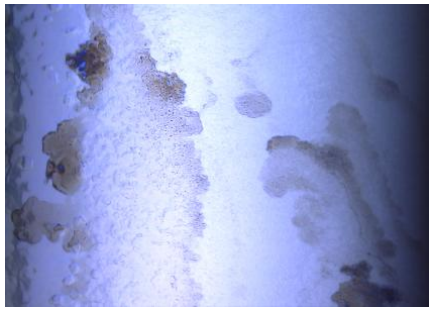
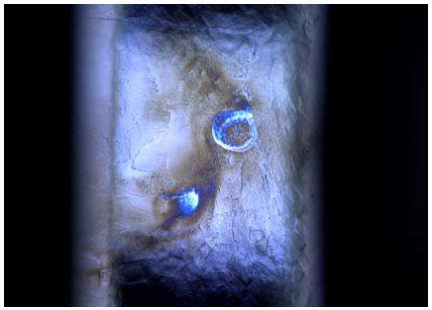
low voltage EP (< 14-17V) avoids S contamination

- Alternative Explanation: Sulfur due to highly Nb saturated acid (H.Hayano)

EP: stains

"Studies on EP process" ; THPPO086 (H. Hayano, T. Saeki et al., KEK)

=> analysis of brown "stains" for **fresh EP**


[Nb] of EP acid (at the end of EP)	Duration of exposure to the air. Stains?	Duration of light P.W. rinse	Duration of exposure to the air. Stains?
0.4 g/L	70 min. No stain	A few 10's sec.	**Stains appeared within a min.**
4.8 g/L	30 min. No stain	A few 10's sec.	6 min. **No stains**
8.6 g/L	30 min. No stain	A few 10's sec.	4 min. **No stains**

EP: Surface morphology

- TUPPO041 (A.T. Wu, Jlab), TUPPO045 (A. Navitzky et al., Wuppertal Univ.), TUPPO075 (Z.H. Sung et al., ASC), TUPPO081 (H. Tian et al., JLAB), TUPPO086 (X. Zhao et al., JLAB, KEK), THPPO091 (P.M. Michelato et al., INFN MIlano) + more

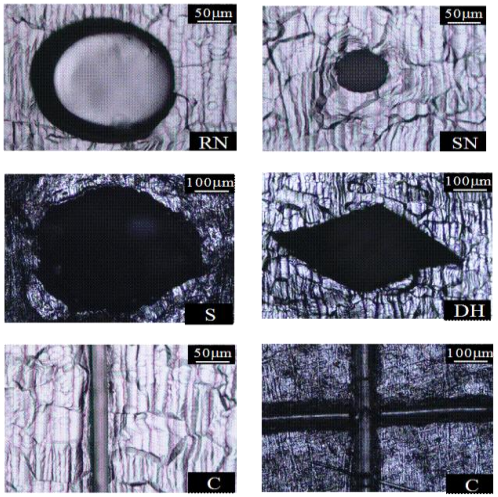
- many, many samples analyzed world wide
- natural and artificial defects

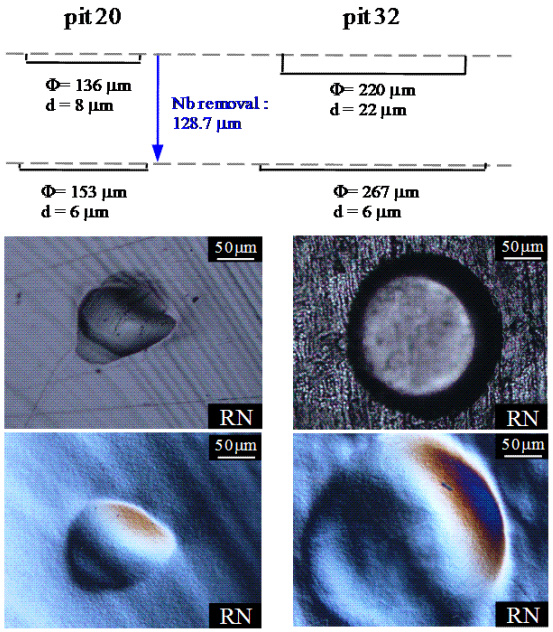
- optical microscope, profilometer, SEM, EDX, laser scanning microscopy, power spectral density, atomic force microscope

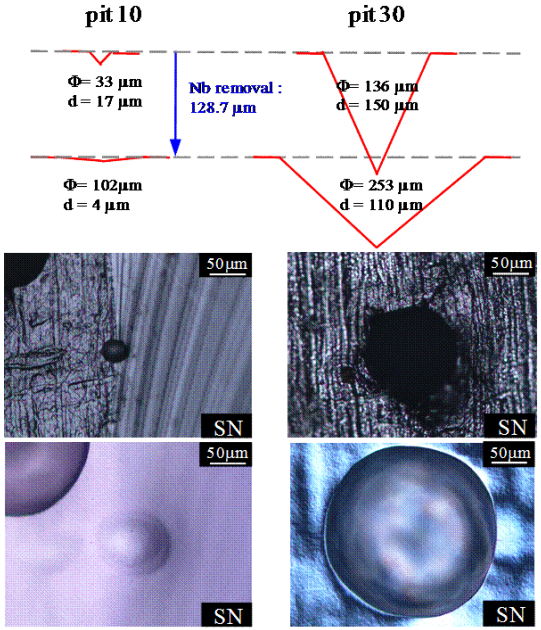
EP: Surface morphology II

- Defect evolution before and after EP

Artificial defects of different shape:







EP: more

- "Model of Hydrodynamic and Thermal Properties"; THPPO061 (C. Reece)
  => better understanding and control of the process

- "EP + mechanical polishing"; TUPPO071 (C. Antoine et al.)

High Pressure Water Rinsing

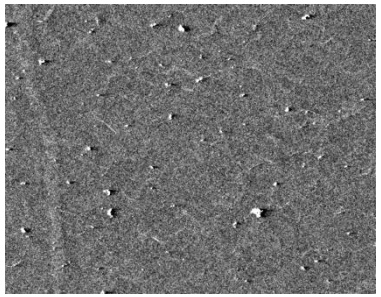
- Well established with systems at all labs
- But, only three (!) dedicated posters !!!
  - C. Reece et al., JLAB: “Particulate Cleaning”; THPPO062
  - A.M. Rowe et al., Fermilab: “Microbiologically Influenced Corrosion”; poster withdrawn
  - S. Mitsunobu et al., KEK: “Horizontal HPR for KEKB”; THPPO087
  - A. Matheisen et al., DESY: “ New HPR System at DESY”; THPPO073

=> My feeling: **HPR has still a lot of a Black Box !**

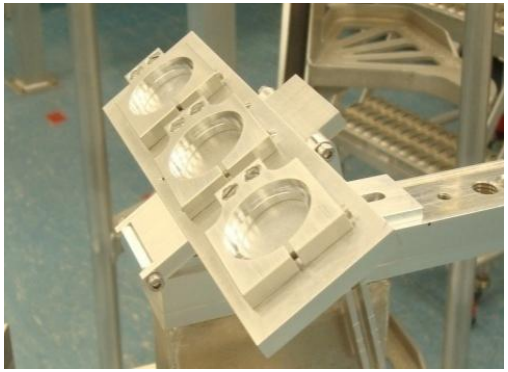
HPR: Analysis I

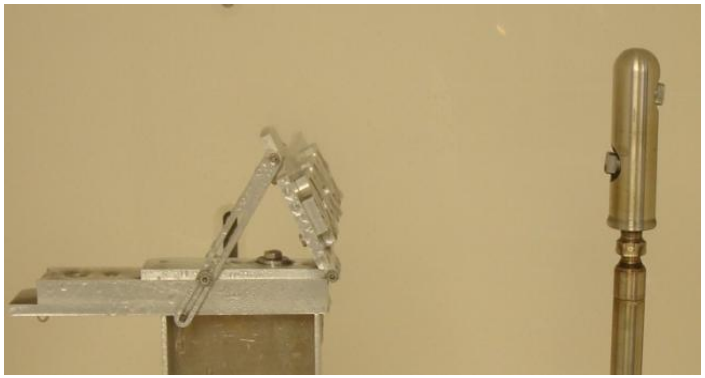
- Study on “Effectiveness of Particulate Cleaning…” by C. Reece et al.

=> Cleaning of SS316 (8 - 50 µm with ~13 particles/mm^2)
+ Nb_2O_5 (0.3 - 31 µm- particles with ~110 particles/mm^2)



Nb_2O_5 particles (100x)





Fixture for holding three samples during HPR

=> all SS316 particles removed for all tested angles and distances

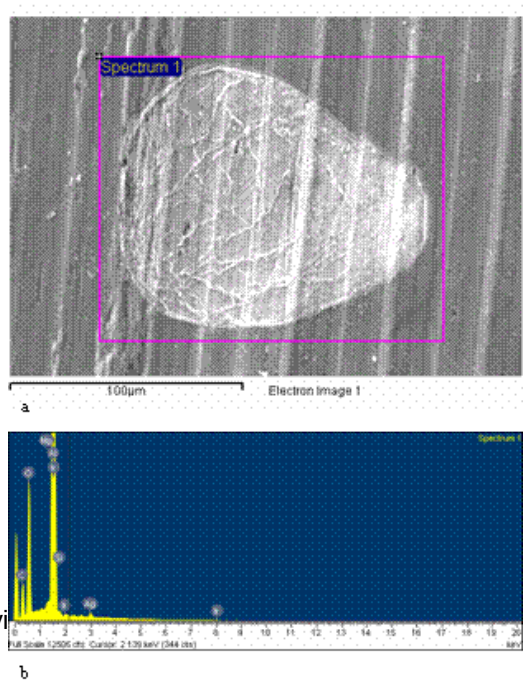
=> ~ 90% of Nb_2O_5 particles removed, but angle dependence requires more tests

HPR: Analysis II

- "Microbiologically Influenced Corrosion in the ANL/FNAL Ultra Pure Water High Pressure Rinse System" by A.M. Rowe et al.

=> brown, biomass nodules + brownish red patches found and analysed

=> "… microbiologically influenced corrosion in 316L stainless steel manifolds attached to the high pressure diaphragm pump was identified …"



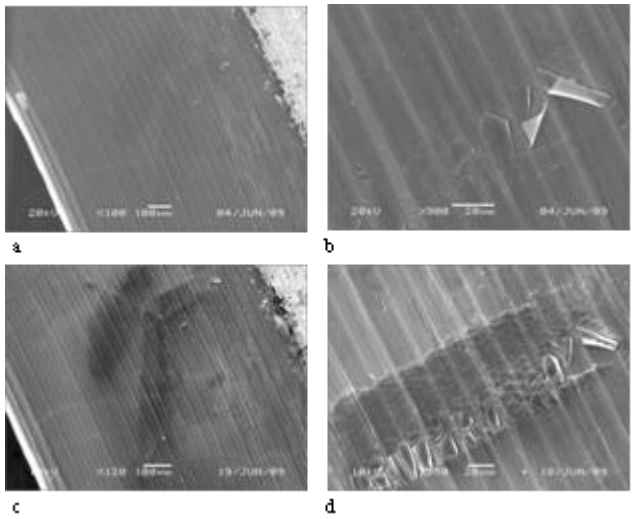


Figure 5: (a) Fibrous nodule at magnification of 650x,
(b) EDS spectrum of fibrous nodule (Al, Mg, C, O, Si, and Ag)

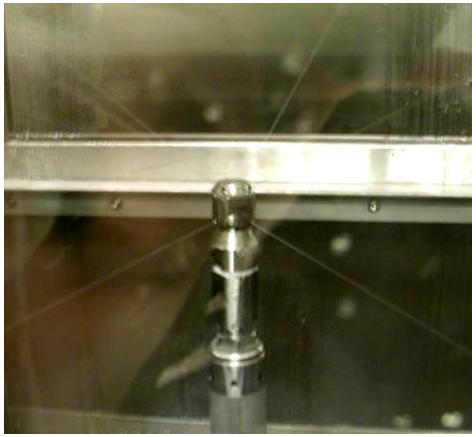
HPR: New Systems

- “Horizontal High pressure Water Rinsing for KEKB Superconducting Cavity” by S. Mitsunobu et al.

=> horizontal HPR with 60 bar on KEKB prototype cavity with first promising results

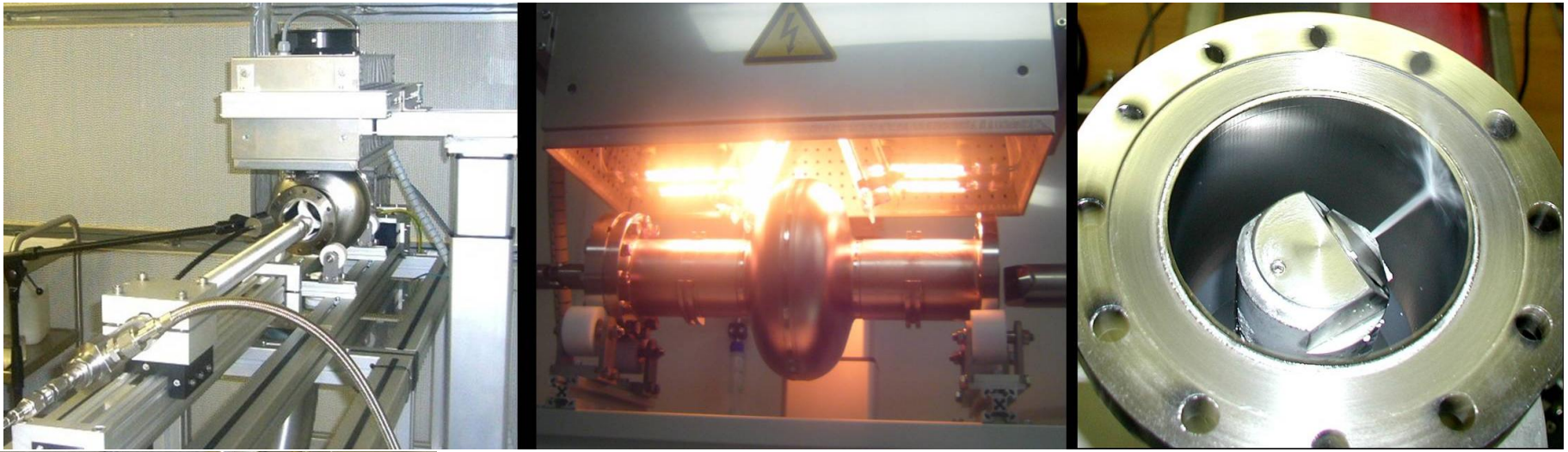
- “A New High Pressure Rinsing System Established at DESY” by A. Matheisen et al.

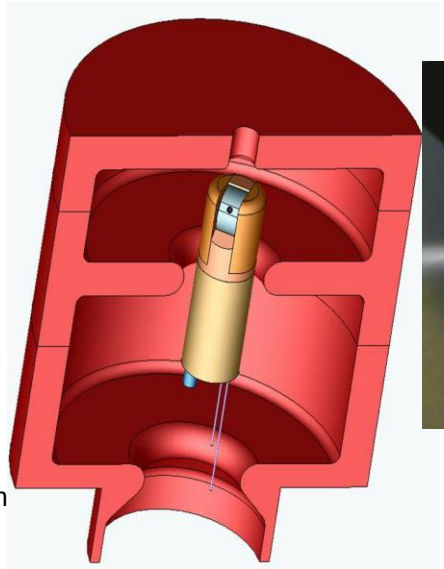
=> successful operational since 2008; prototype for industrialization

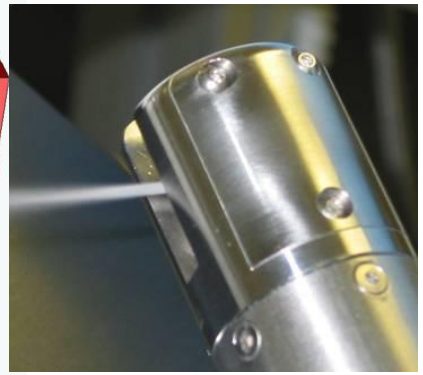


CO_2 Snow Cleaning

- Reminder: only one cleaning system operational
  => DESY: horizontal + vertical cleaning stands (presented at SRF 2007)







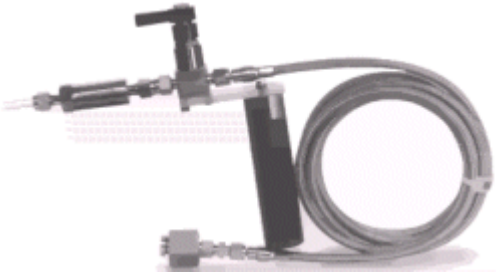
CO_2 Snow Cleaning

only one paper:

- Study on "Effectiveness of Particulate Cleaning…" by C. Reece et al. (THPPO062; as for HPR)

=> Cleaning of SS316 (8-50 µm) + Nb_2O_5 (0.3-31 µm)- particles

=> hand-held dual gas (CO_2 + N_2) system



courtesy Applied Surface Technology

- Preliminary cleaning tests:

=> all SS316 particles removed for all tested angles and distances;

=> effective for Nb_2O_5 particles < 3 µm, but angle + distance dependent

- **"Cleaning standard samples with CO_2 snow proved to be more effective than either US or HPR, …"**

Summary

- **Electropolishing**
  - one major topic here at Berlin
  - lots of substantial work
  - my high-light: Defect evolution + correlation to optical inspection, T-mapping, cavity results, …
- **HPR**
  - works well, but we do not really know why
- **Snow Cleaning**
  - highly efficient **additional** cleaning tool
  - still undervalued for clean room + pre - clean room applications

CO_2 Snow Cleaning: Working principle

- dry-ice “snow”: mechanical, thermal + chemical cleaning forces
  - mechanical: momentum of dry-ice jet
  - thermo-mechanical: i) embrittlement by shock-freezing
    ii) shearing forces by high momentum
    iii) drastic volume increase by sublimation
  - chemical: liquid CO2 acts as solvent for hydrocarbons + silicone
- removal of particles down to < 100nm => local, dry, without residues
- simple checks with air and surface particle counters possible
- nozzle designs: i) mixture of CO_2 and N_2
  ii) CO_2 jet surrounded by N_2

- **Additional cleaning option** => no replacement of HPR
- Effective removal of particulate and film contamination
- Dry cleaning process => horizontal + moisture sensitive applications