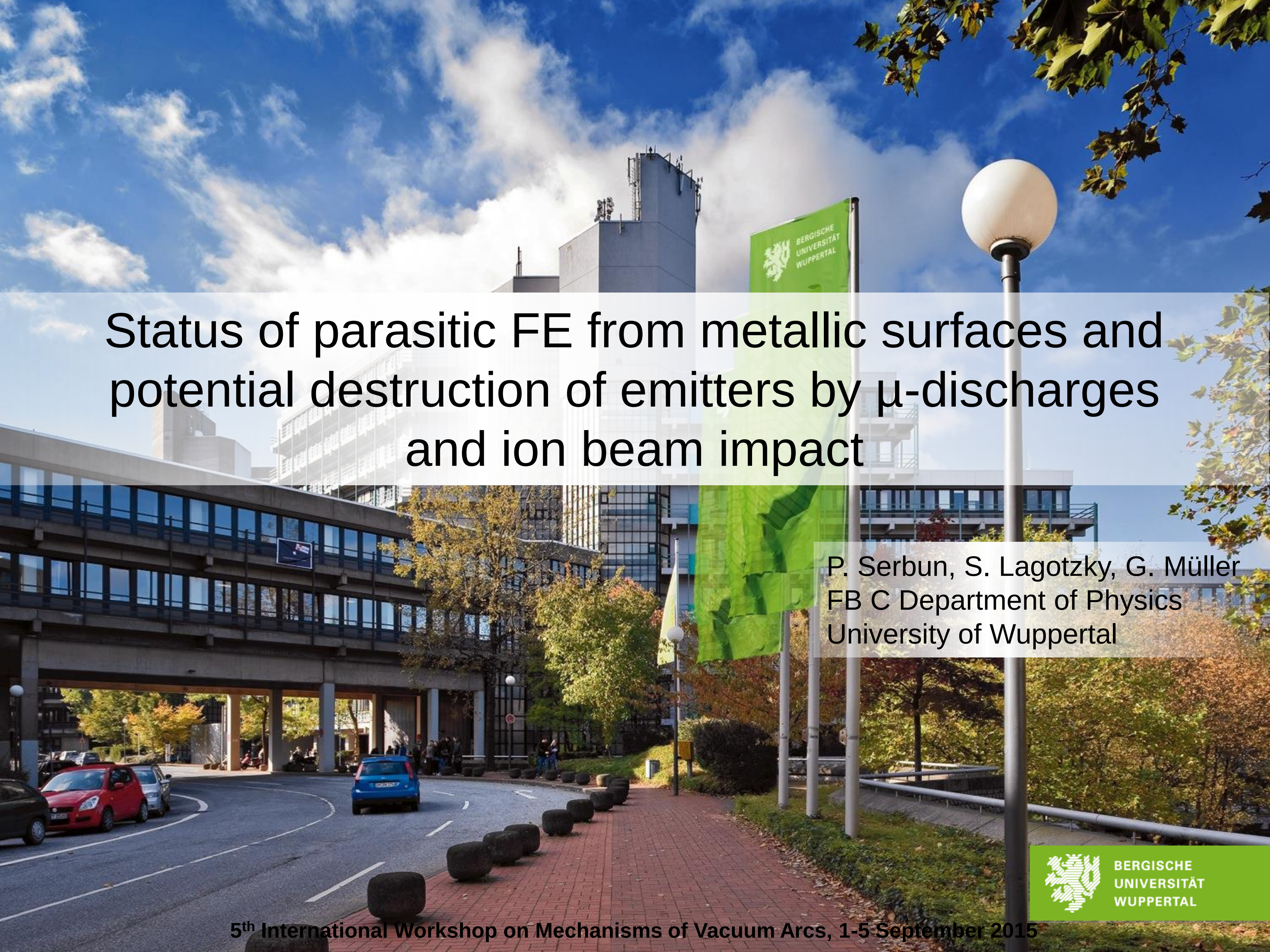

Status of parasitic FE from metallic surfaces and potential destruction of emitters by µ-discharges and ion beam impact

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5th International Workshop on Mechanisms of Vacuum Arcs, 1-5 September 2015

Outline

- Motivation
- Measurement techniques & samples
- Field emission results before and after cleaning
- Destruction of emitters by µ-dischages
- First results on Ar ion bombardment
- Conclusions and outlook

Motivation

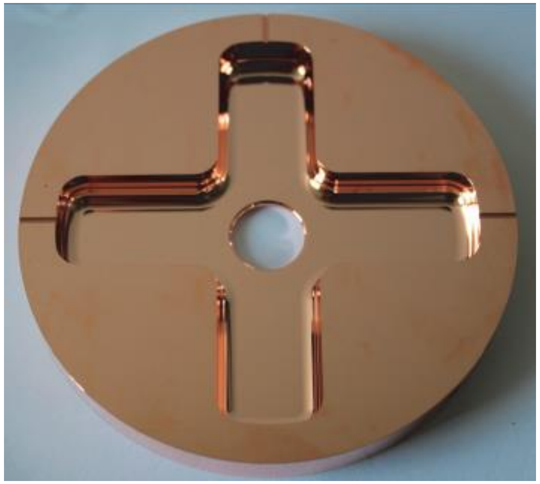
- Field emission (FE) creates "dark current" which absorbs rf energy, causes radiation, is seems to be the precursor of electric breakdown (BD);
- In turn, BD limits the operation of accelerators and can cause irreversible damage to their physical structure;
- The acceleration gradient for the present CLIC design is E_{acc}=100 MV/m (E_{peak}=243 MV/m) and achievable only after long conditioning of the structures [2];
- Deep and quantitative understanding of the origin of BD processes is important;

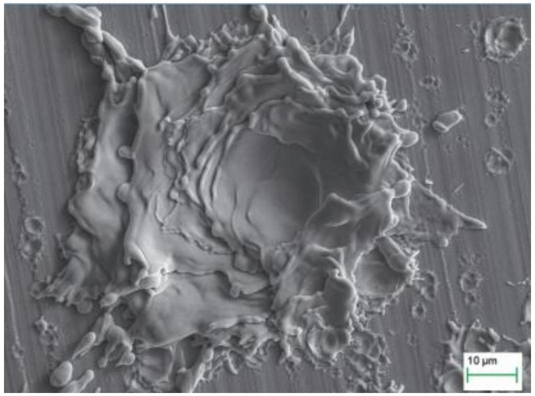
Goal: suppression of BD by using proper surface treatments.

Task: investigation of FE from flat Cu surfaces

- What causes FE from (relevant) Cu samples?
- How to reduce/avoid FE?

BMBF project 05H12PX6

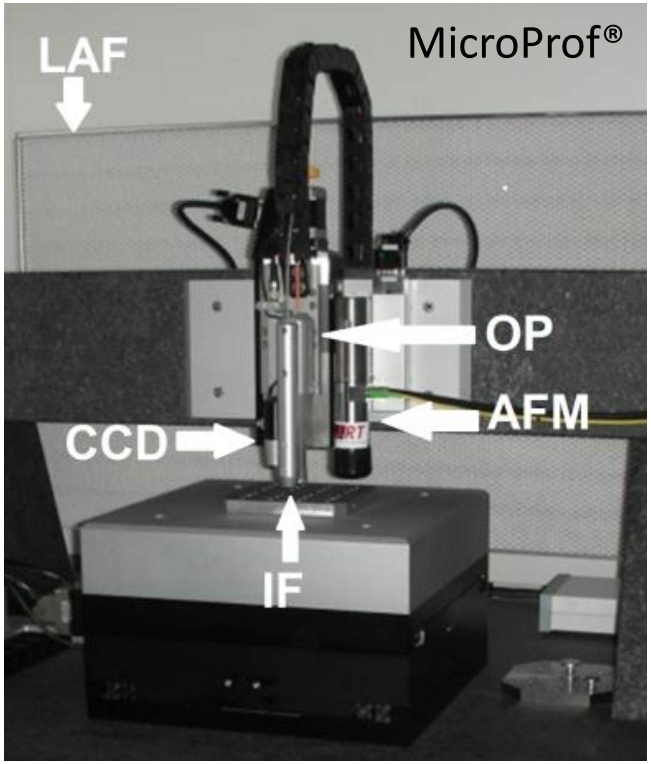

Cu disc of CLIC accel. structure [1]


Surface damage due to breakdown [1]

[1] A.T. Perez & G. Arnau, "*Determination of dislocations density in Cu-OFE for CLIC project by using EBSD*", Poster, MeVArc 2013
[2] W. Wuensch, "Study of the conditioning of RF structures", Presentation, MeVArc 2015.

Surface quality control

- **Optical Profilometer (OP)**
  - white light irradiation and spectral reflection (chromatic aberration)
  - 20x20 cm^2 scanning range in 2 cm distance
  - Curved surface up to 5 cm height difference
  - 2 µm (3 nm) lateral (height) resolution
- **Further zooming by AFM**:
  - ±2 µm positioning relative to OP results
  - 98x98 μm^2 scanning range
  - 3 (1) nm lateral (height) resolution
  - contact or non-contact modes.
- Clean laminar air flow (LAF) from the back
- Granite plate with active damping system
- CCD camera for fast positioning
- interferometric film thickness sensor (IF)

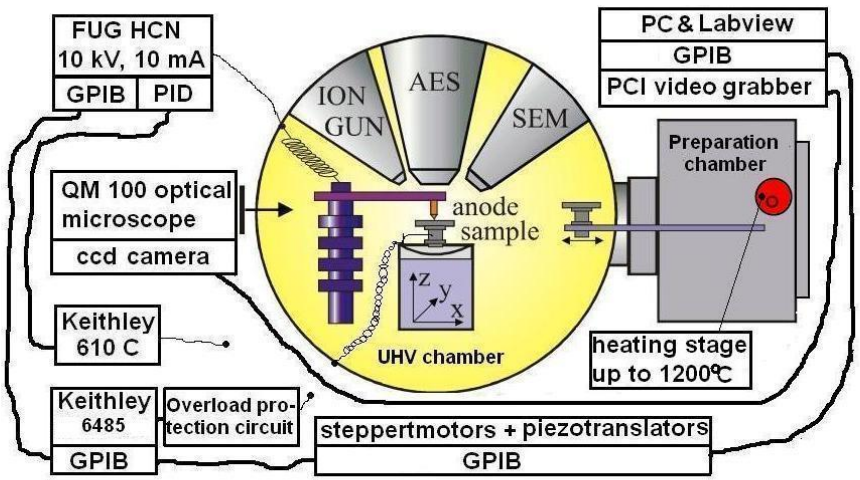


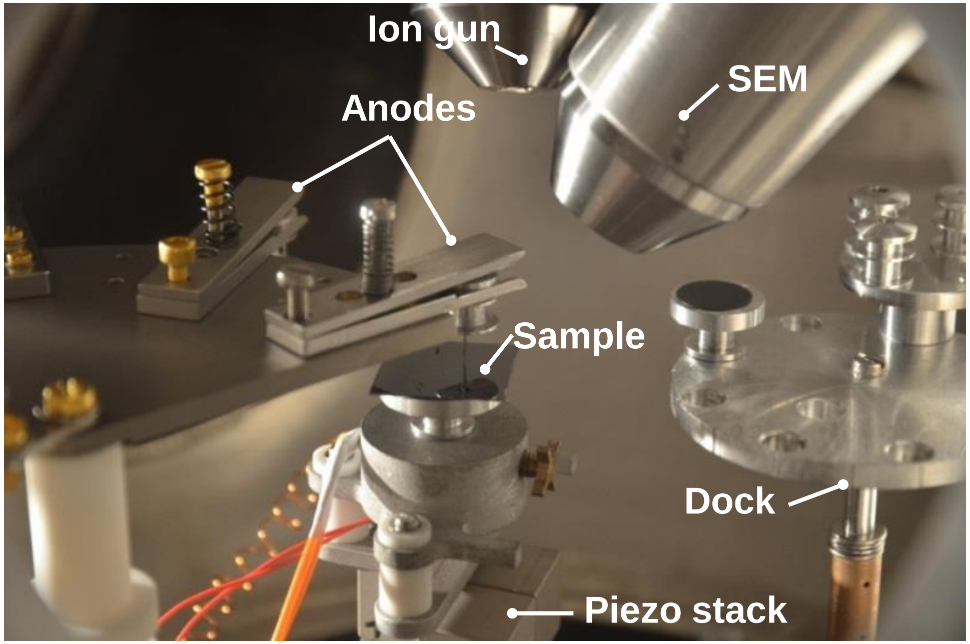
www.frt-gmbh.com

DC field emission scanning microscope (FESM)

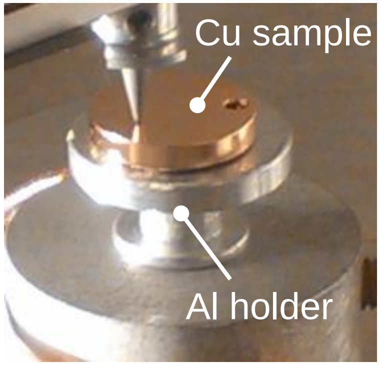
Provides PID-regulated **voltage scans** V(x,y) @ fixed current (typ. I=1nA): E_{act} (1nA)

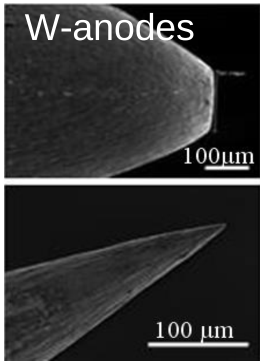
- **activation**, **localization** and **number density N** of emitters
- **local** U(z) and I(V) **measurements** of single emitters: E_{on}(1 nA), β_{FN}, S_{FN}

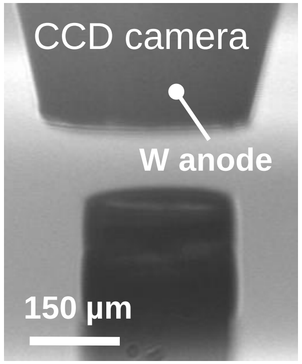




- base UHV at 10^{-7} Pa
- exchangeable W-anodes 3…330 µm
- cathodes up to 25x25 mm^2
- 3D drives:
  stepper motor/piezo-translator (100/40 nm/step)
- cathode tilt correction: ±1 µm within ±5 mm
- gap monitored via CCD camera (1 µm resolut.)
- heat treatments (< 1200° C)
- auger electron spectroscopy (AES): chemical state
- ion gun: for local emitter cleaning
- ex-situ SEM & EDX: identification of emitting defects
- clean laminar air flow around load-lock

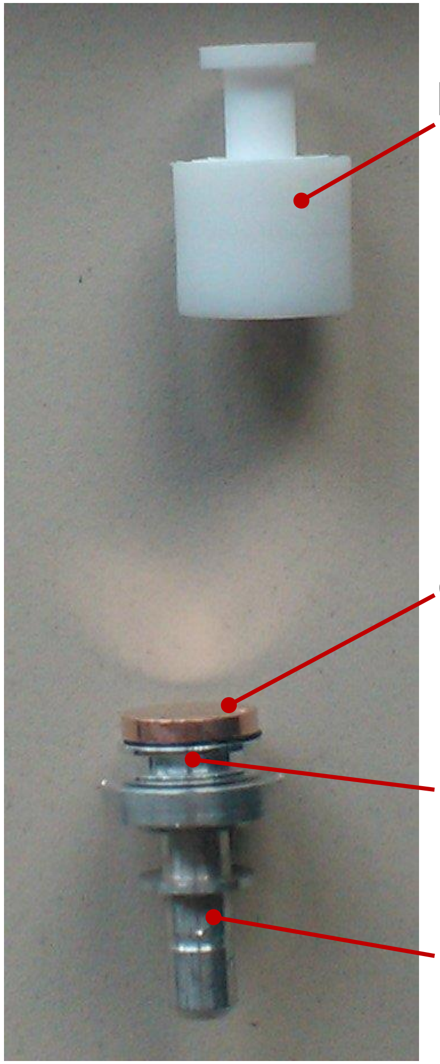






D. Lysenkov, G. Müller, *Int. J. of Nanotechnology* **2**, 2005.

Samples



Fabrication at CERN:

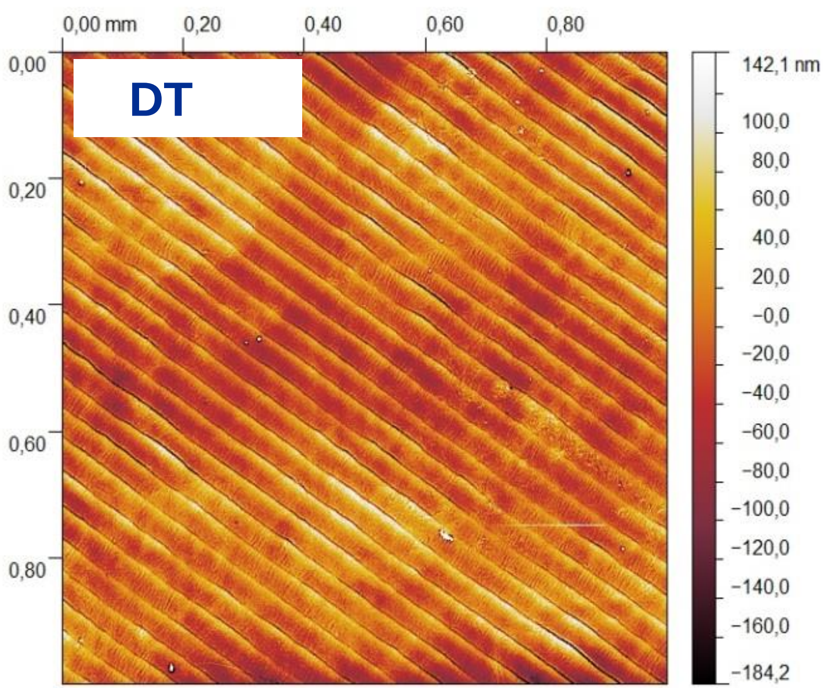
- Flat polycrystalline Cu samples of Ø =11 mm
- Small hole (0.5mm) as mark to identify the emitter position in different systems (SEM/FESM)
- Diamond turned (**DT**) samples
- Additional chemical etching (**SLAC** treatment: using H_3PO_4, HNO_3, acetic glacial acid and HCl, to remove a surface layer of 0.6 µm)

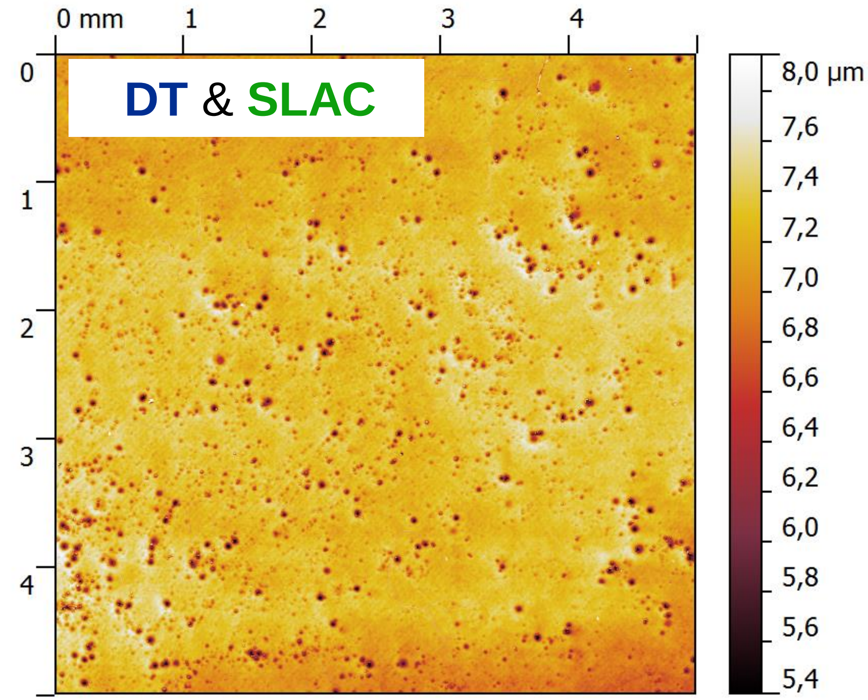
Investigation at BUW:

- Transport under Teflon® protection cap to avoid any surface damage and contaminations after polishing and cleaning
- Glued with SEM button on Al holder and mounted to an adapter for the FESM
- Caps opened under HV or clean room (ISO5)
- Final **cleaning** (N_2, DIC)
- **OP**+**AFM**/**FESM**/**SEM**+**EDX** measurements

Surface quality

Samples measured with optical profilometer (OP) in the area relevant for FESM



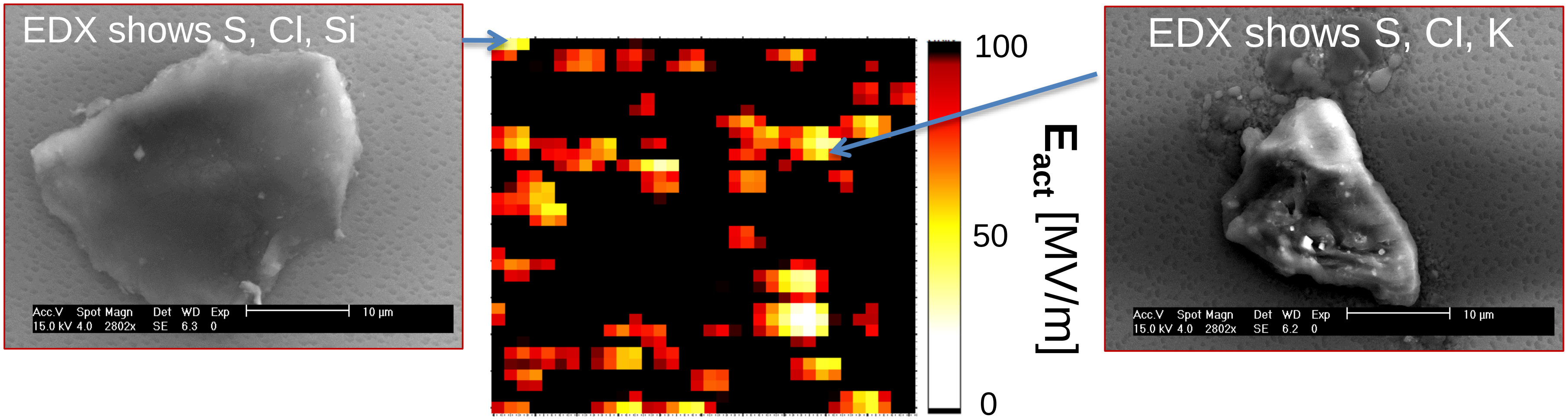


- Slightly waved surface (λ~ 0,5 – 1 mm)
- Many ridges from DT
- Damage layer?
- Average roughness: R_a/R_q = 126/145 nm

- Sample surface now very flat (±0.5 µm)
- Many pits (N < 18 mm^{-2}) due to etching
- Grain size: 1300 µm² - 5.3 mm²
- R_a/R_q = 150/230 nm slightly increased

FESM/SEM/EDX results

E-map for the sample DT+SLAC #1 before cleaning (1 nA, area 5x5 mm²)



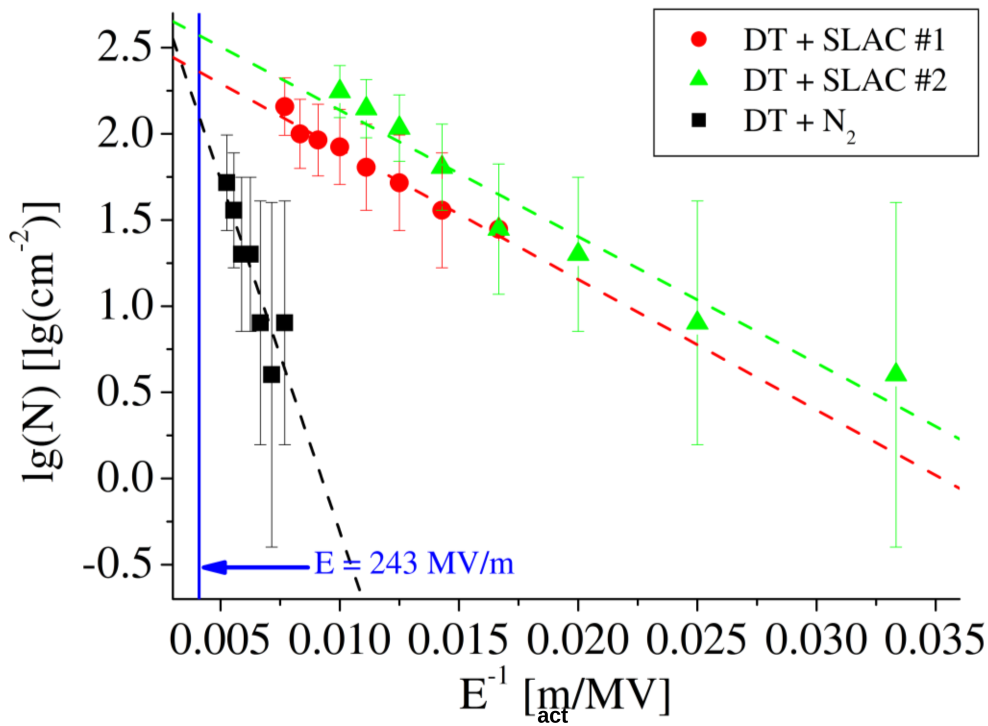
SEM/EDX analysis of Cu surface:

- 60% particulates (Al, Cl, S, Si, K)
- 10% surface defects
- 30% emission sites: unknown origin

- EFE is dominated by foreign particulates: cleaning the surface to reduce FE

FE activation statistics without/with N_2 cleaning

for two types of samples



- Emission from surfaces without any cleaning starts at 30 MV/m
- Cleaning with N_2 shows FE starting at 130 MV/m
- N @ E_{peak} = 243 MV/m reduces from 229/372 cm^{-2} to 124 cm^{-2}
- N increases exponentially [3]:

  $N \sim exp(1-/E_{act})$

[3] S. Lagotzky, G. Müller, submitted to Nucl. Instr. Methods Phys. Res. Sect. A (2015).

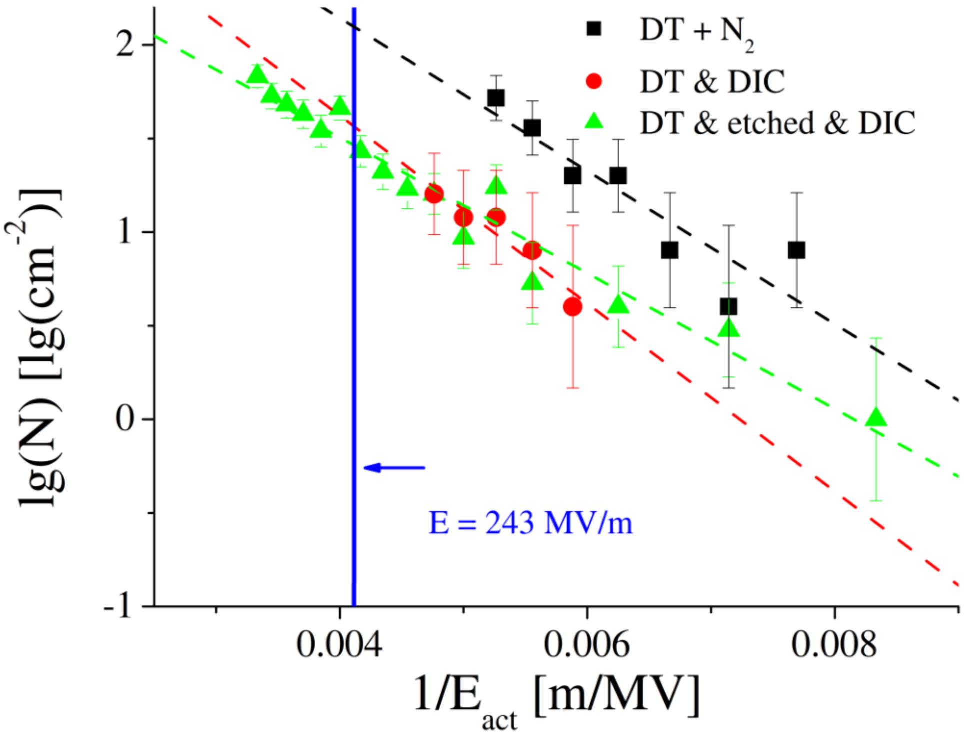
Dry ice cleaning (DIC) system

www.cryosnow.com

- Commercial DIC system (SJ-10, CryoSnow) installed in cleanroom (class iso 5)
- Non-abrasive blasting where dry ice is accelerated in a pressurized air stream and directed at the surface
- Samples/caps cleaned for 2.5 min under 90° /45° and 3 x rotated in 90° steps
- Most particulates (> 100 nm) are removed

FE activation statistics after DIC cleaning

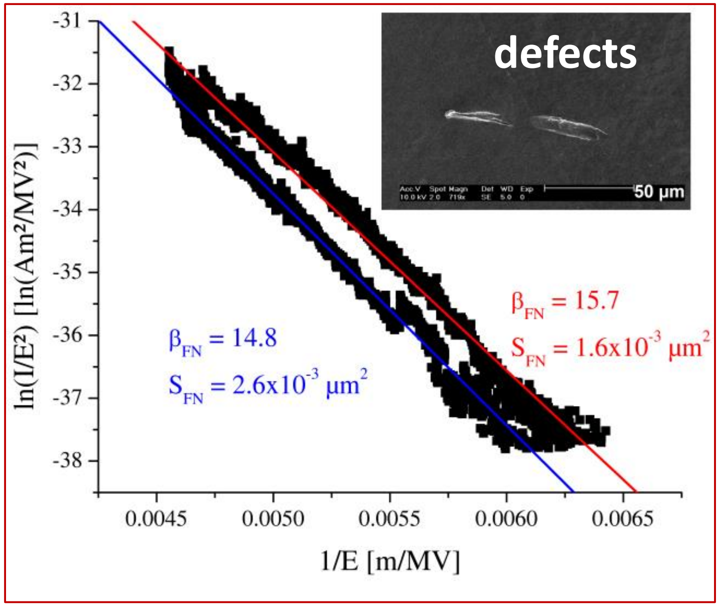
for three types of samples

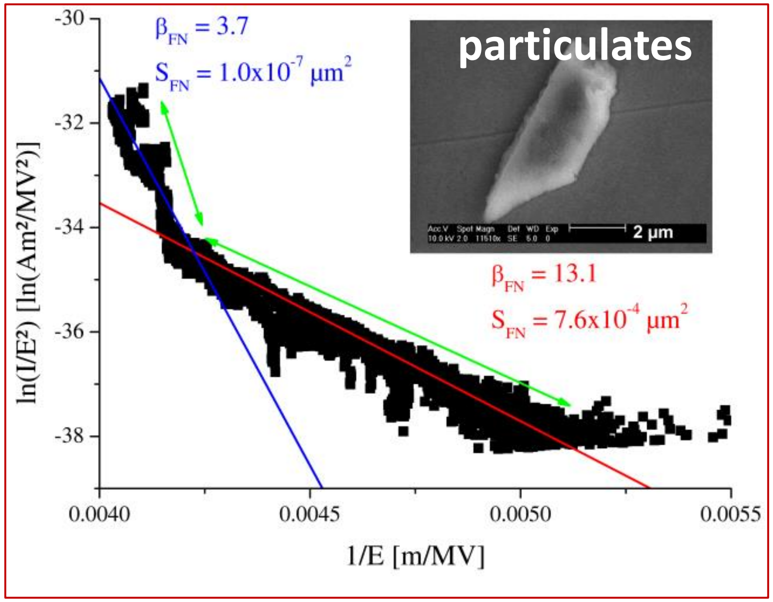


- DIC reduces N significantly
- N @ E_{peak} = 243 MV/m reduces from 124 cm^{-2} to 29 cm^{-2}
- Chemical etching didn't reduce N

Single emitter characteristics on DIC-cleaned samples

SEM/EDX : 57% surface defects; 12% particulates (Al, Si, W); 31% unidentified;

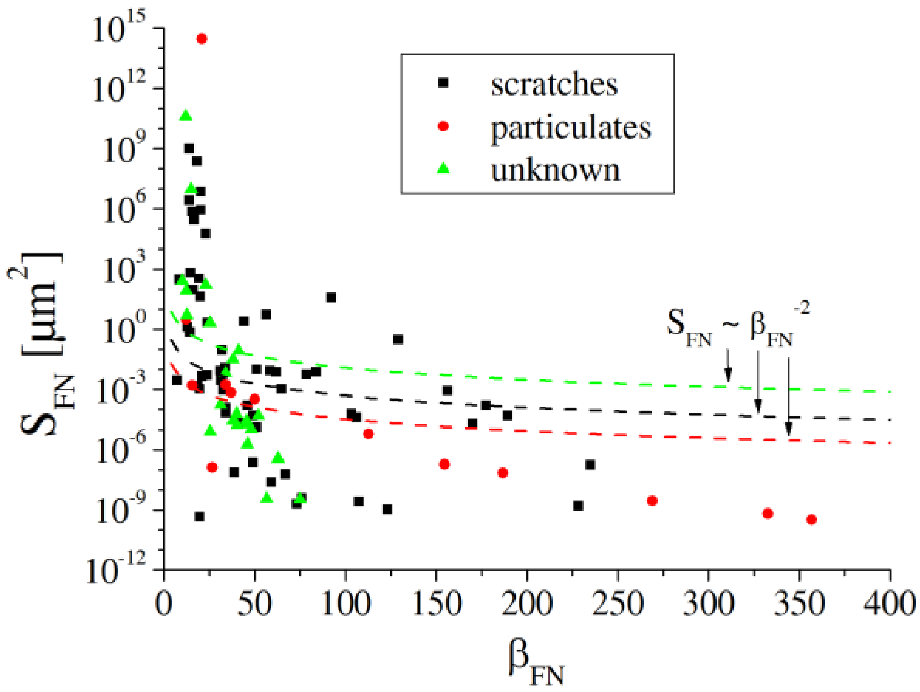




- Rather stable FE
- Slight jumps probably due to melting of micro-tips

- More unstable FE
- Changed slope at high fields due to bad electrical contact to bulk

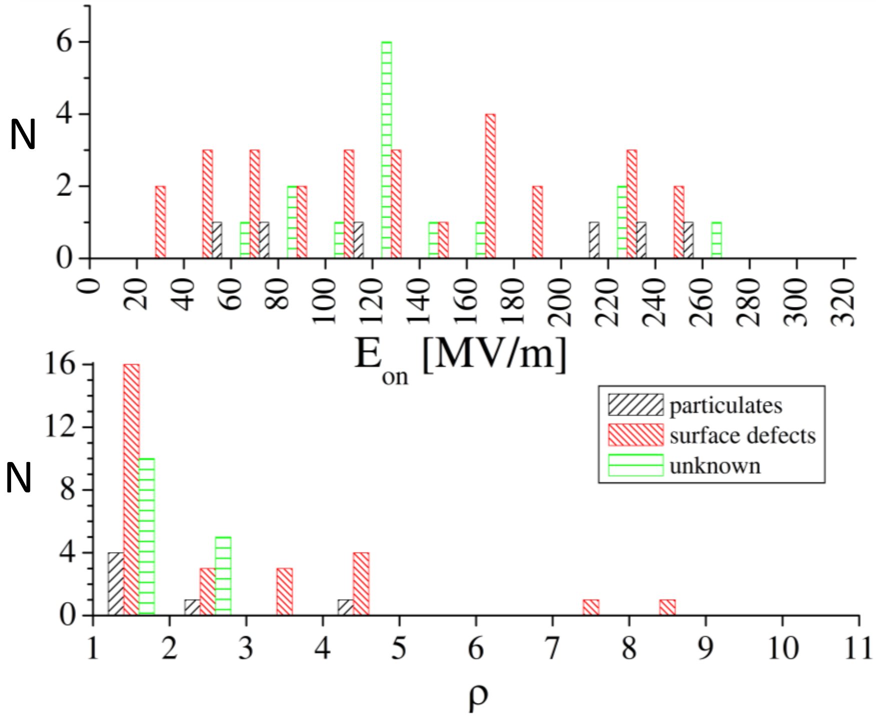
Correlation between S_{FN} and β_{FN}



- Highest β_{FN} are caused by particulates
- Most S_{FN} and β_{FN} are in a reasonable range for all types of emitters
- Most data are correlated: $S_{FN} \sim \beta_{FN}^{-2}$
- No correlation at low β_{FN}: high S_{FN} values with respect to the anode size hint at other FE mechanisms like MIV- and MIM-emission [4]

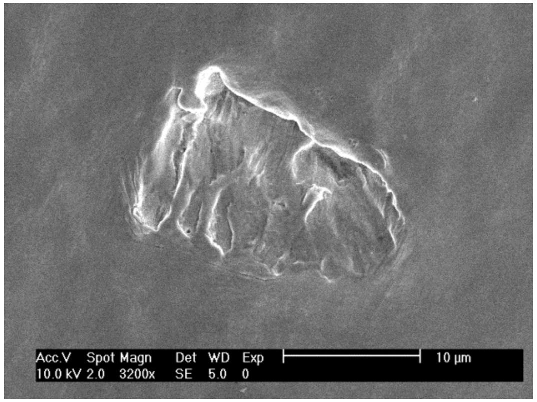
[4] R. V. Latham, High voltage vacuum insulation, Academic Press, London (1995).

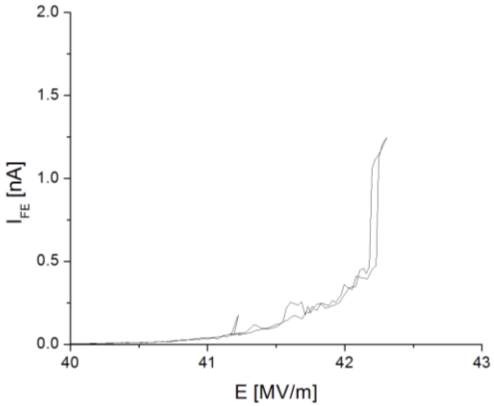
Possible origin of breakdowns in FR structures

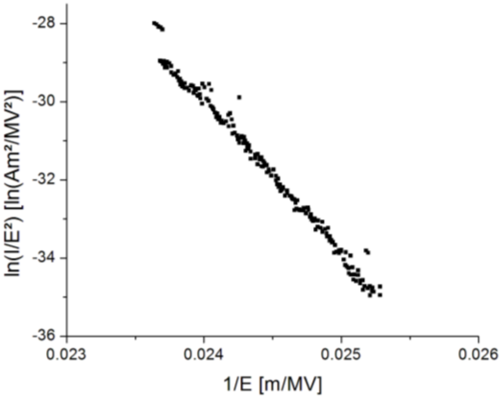


- All emitters yielded a reduced $E_{on} < E_{act}$ hence, emitters can lead to strong e^- loading of accelerating structures if activated
- Activation strength described by field reduction function $\rho = E_{act}/E_{on}$
- 20% of emitters show ρ > 3 (surface defects)
- High-p emitters would cause an emitter explosion and a BD of the cavity field

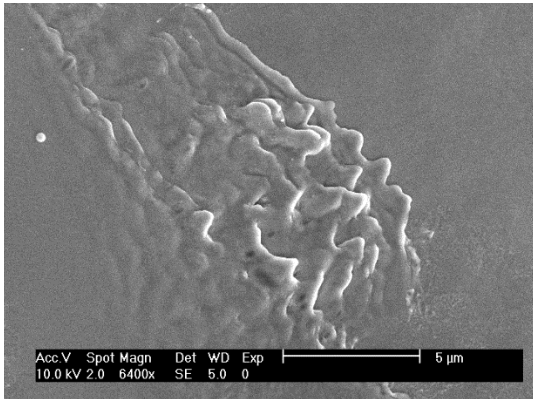
Examples: two candidates for breakdowns

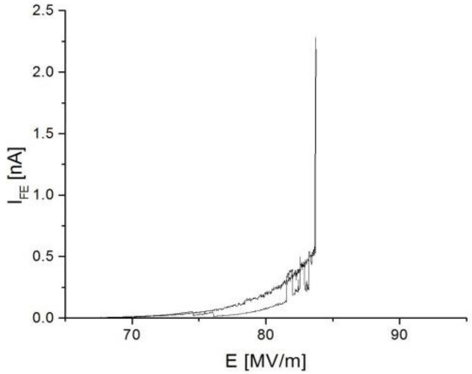


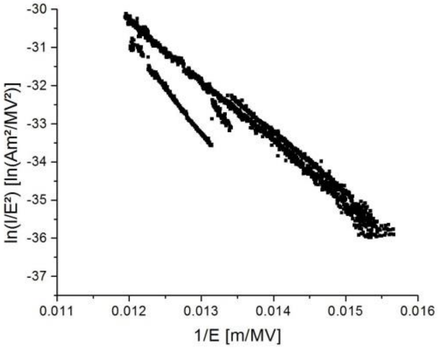




- Activated between E_{act} = 240-250 MV/m, E_{on} = 54 MV/m → ρ = 4.62
  → calculated current @ 243 MV/m: $I_{FN} \sim 10^{22}$ A!







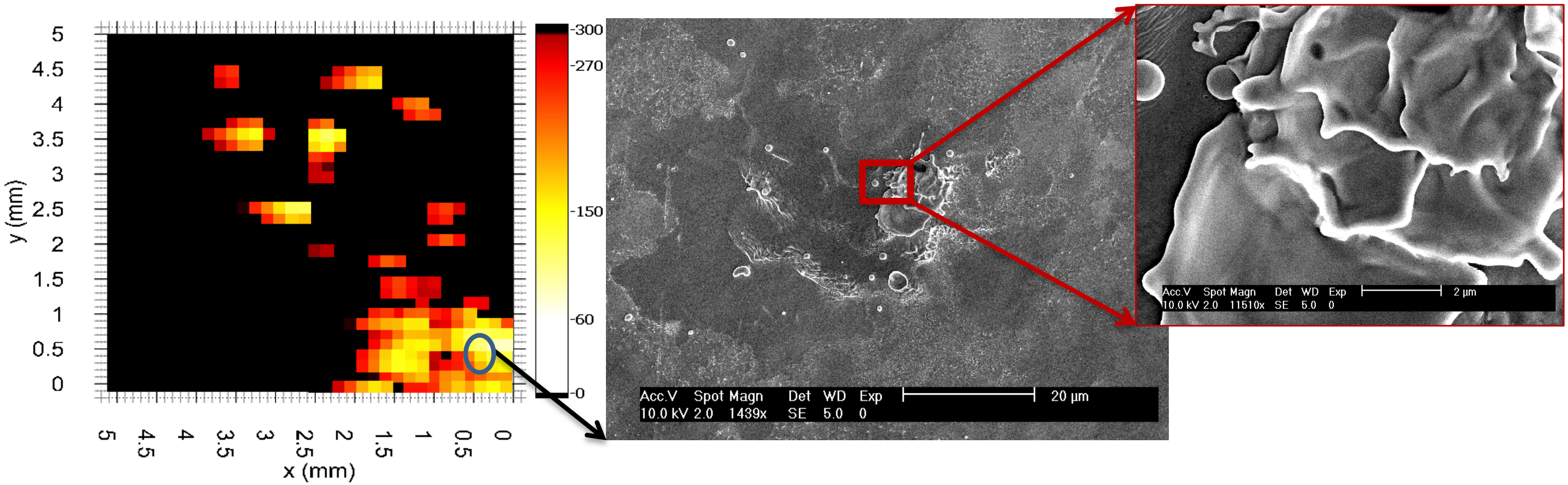
- Activated between E_{act} = 130-140 MV/m, E_{on} = 80 MV/m → ρ = 1.75
  → calculated current @ 243 MV/m: $I_{FN} \sim 3$ mA

Accidental discharches in the FESM on Cu

Usually discharges are prevented during FESM scans by the PID voltage regulation

Nevertheless, some unwanted discharges happen:

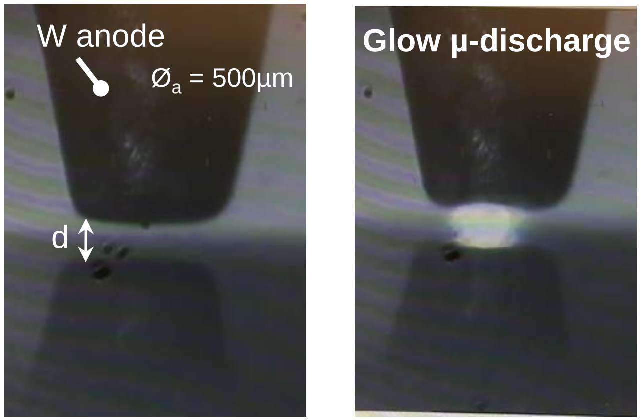
- during scans (if the current jump is faster than ~2 ms)
- during local measurements (because of activation effects)

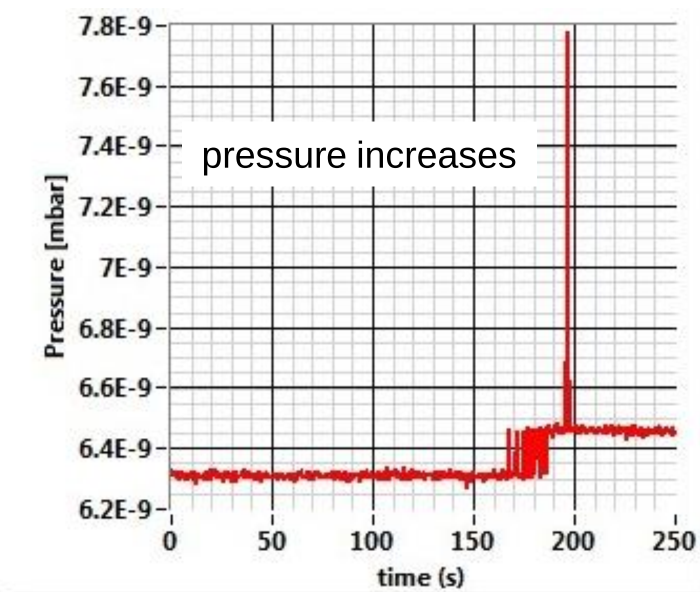


- Discharges (most-likely caused by high-ρ emitters) destroy the surface and lead to the formation of new stable and strong emitters, probably similar to BDs in cavities
- In accelerating structures such a new emitter triggers the next BD, which forms another emitter, that ignite a BD etc.

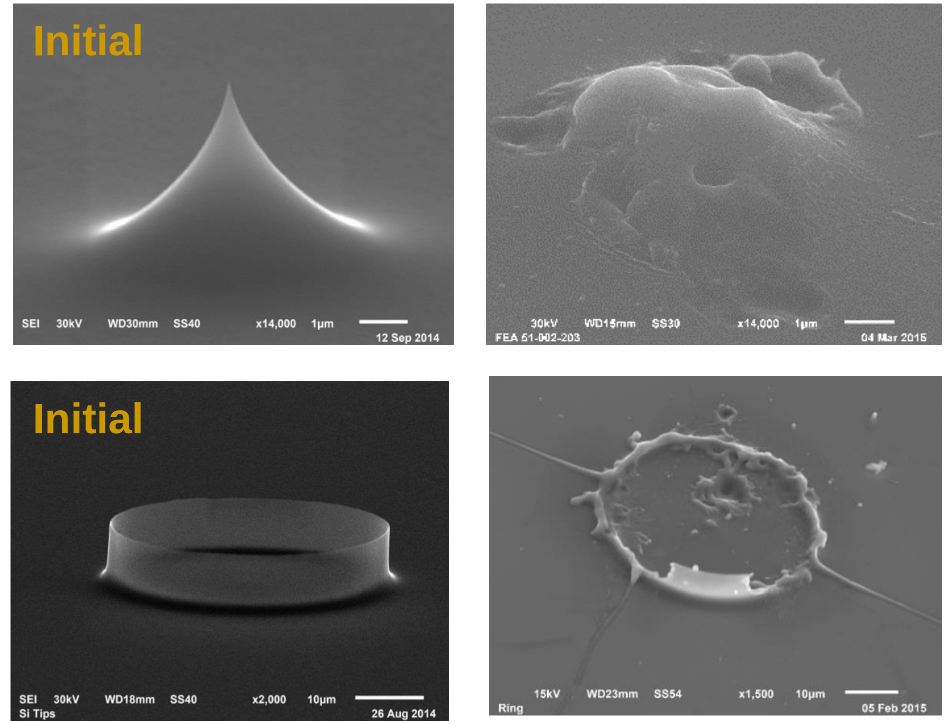
µ-discharges in the FESM on Si samples

CCD camera images of vacuum gap and glow µ-discharge





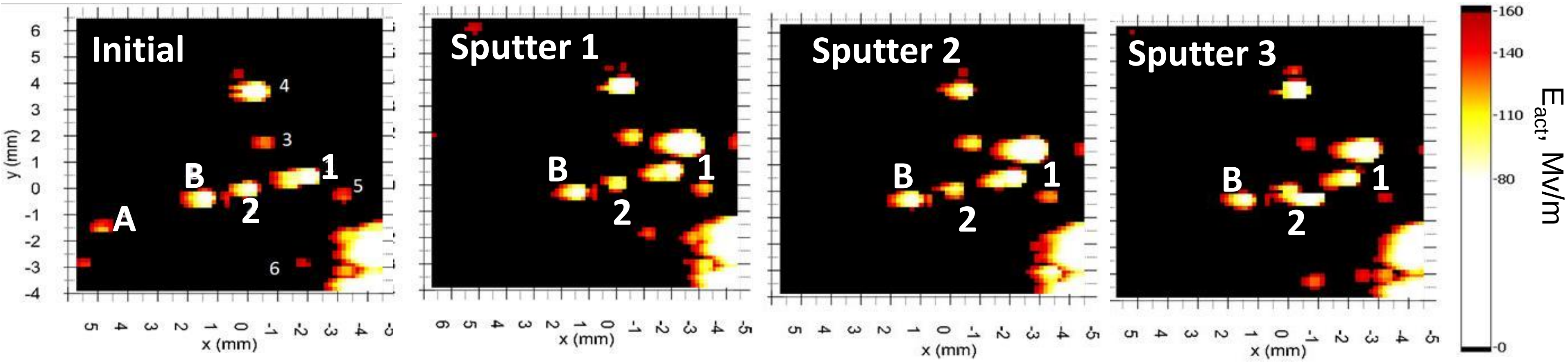
SEM images of different emitters before/after



Discharges lead to

- morphological changes (significantly shortened, partially/completely removed emitters)
- create craters;

First results on Ar ion bombardment (local sputtering) of Nb



- Emitter A was deactivated;
- Emitters become weaker even at moderate ion energy (max. 2keV);
- Formation of new emitters?

Emitter No.	initial	Sputter 1 E_{ion} = 1keV, t=1h	Sputter 2 E_{ion} = 1keV, t=1h	Sputter 3 E_{ion} = 2keV, t=1h
	β_{FN}	β_{FN}	β_{FN}	β_{FN}
A	not meas.	gone	-	-
B	100	100	57	69
1	114	85	85	85
2	58	58	47	32

Conclusions & outlook

- Actual surface quality of DT+SLAC etched Cu not sufficient for CLIC structures
  - N = 229 & 370 cm^{-2} at E = 243 MV/m, mainly caused by particulates
- Cleaning with N_2 (DIC) decreases N significantly by a factor ~1.8-2.8 (7.7-12.3)
  - N = 124 (29) cm^{-2} at E = 243 MV/m, mainly caused by surface defects
- Geometrical field enhancement not sufficient to explain FE of Cu surfaces
  - Alternative emission processes like the MIV/MIM-model or field-induced protrusion growth
  - Activated emitters with high ρ are candidates for BDs in accelerating structures
- µ-discharges destroy emitters but often create new emitters/craters
- Low energy Ar ion bombardment is more reliable way to weaken/remove emitters
- Current and ion conditioning of emitters on Nb will be studied in the next project

Acknowledgements:

Funding by BMBF projects 05H12PX6 (completed) and 05H15PXRB1 (just started)

Thank you for your attention

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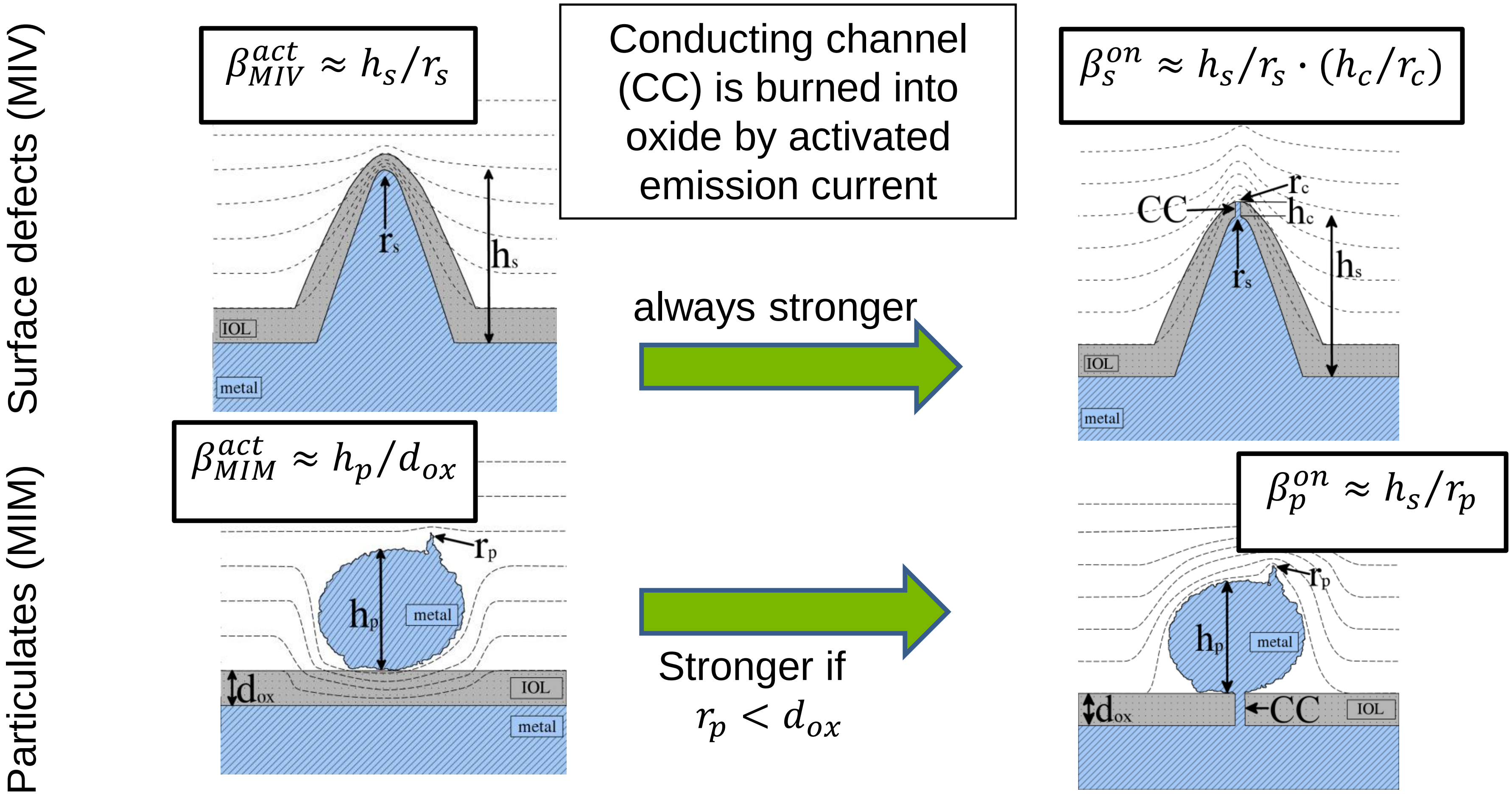
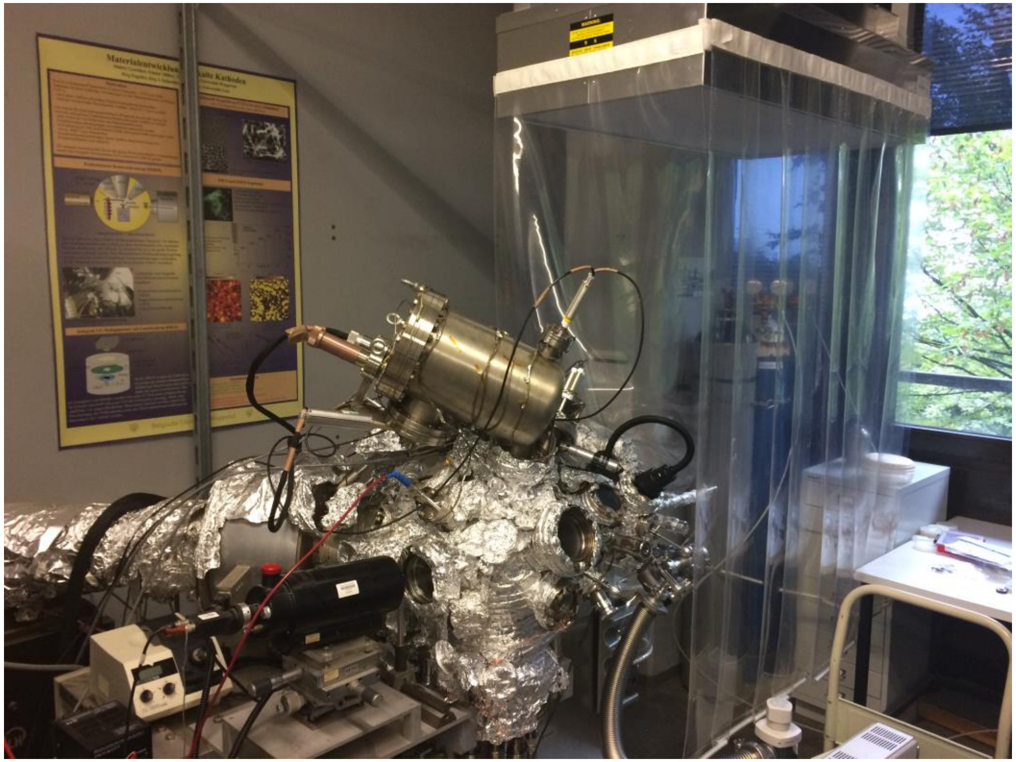
Activation of emitters

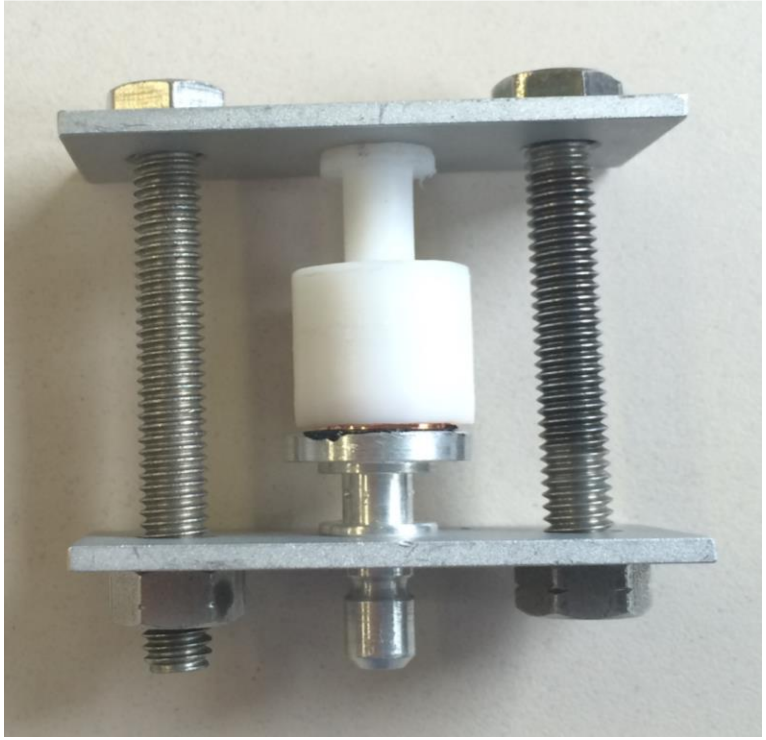
- Insulating oxide layer (IOL, thickness d_{ox} ~ few nm) on metallic surfaces
- Question: How are emitters activated?



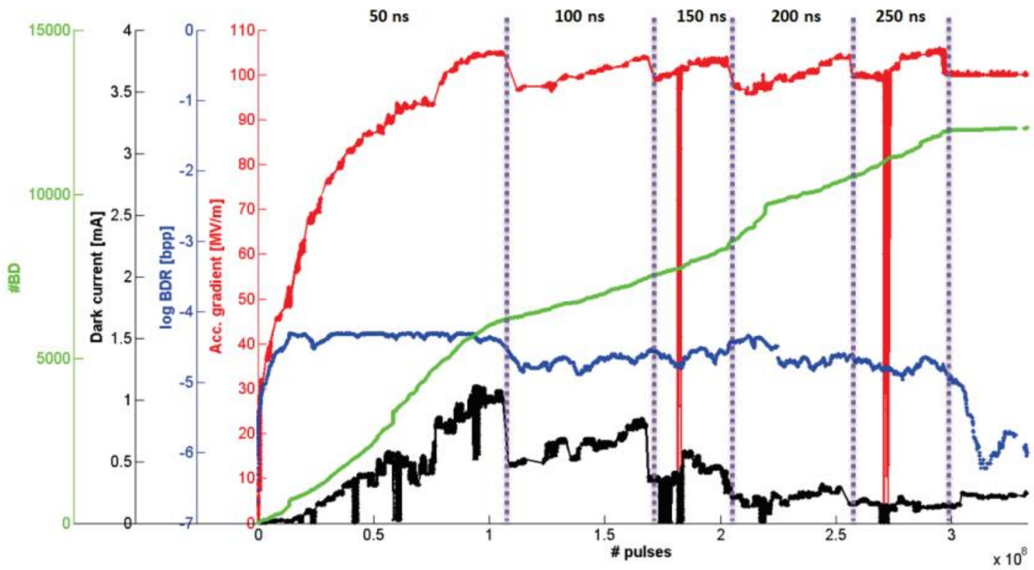
Avoiding particulate contaminations

- A cleanroom environment (class ISO 3) was installed around the load-lock of the FESM to avoid particulate contaminations during installation of samples
- Protection cap mechanically fixed until sample reaches cleanroom environment
- Protection cap loosened under laminar air flow
- Final removement of cap in preparation chamber at p ~ 10^{-7} mbar





Consequences of EFE-results for structure conditionning



- E_{peak}/E_{acc} = 2.43
- After 1,800 h ~12,000 BDs in 28 cells → ~430 BDs per cell [2]
- BD rate still too high (factor 10)
- **Goal: BD rate < 10^{-7} BD/pulse/m**
- EFE reduced but still present

- Actual cleaning: Ultrasonic baths with de-ionized water and clean alcohol
- Potential improvements due to EFE results:
  - N_2-cleaning → factor 1.8 – 2.8 → BD rate still too high
  - DIC → factor 7.7 – 12.3 → goal for BD rate achievable
- Conditioning results of a N_2/DIC-cleaned accel. structure still pending

[2] A. Degiovanni et al., WEPME015, Proc. IPAC2014, http://jacow.org/.

Cleaning process

- Cleaning of (grounded) samples with handgun (d ~ 5 cm) typically for 5 min
- Liquid CO_2 (10 bar) and N_2 (8 - 10 bar, propellant gas)
  → Flat (12x3 mm) or round (Ø = 5 - 10 mm) jet of CO_2 snow particles
- Samples are treated 2.5 min under 90° / 45° and 3 x rotated in 90° steps
- Teflon protection caps are cleaned as well