

# Advances in Investigations of clean Nb surfaces

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- Motivation
- Enhanced field emission of Nb surfaces
- Preparation and measurement techniques
- Statistical distribution of field emitters
- FE properties and nature of emitters
- Conclusions and outlook



# Motivation

- Accelerating fields  $E_{acc}$  in SC Nb cavities are limited by **NC defects** and **protrusions** and surface impurities



**local quenches**      **electron loading due to field emission**

- Improved Nb purity and surface preparation techniques are required to achieve  $E_{acc} > 25$  MV/m at  $Q_0 > 10^{10}$  reliably
- Advanced surface investigation of clean Nb samples by **profilometry**, **scanning FE microscopy** and **SEM/EDX**



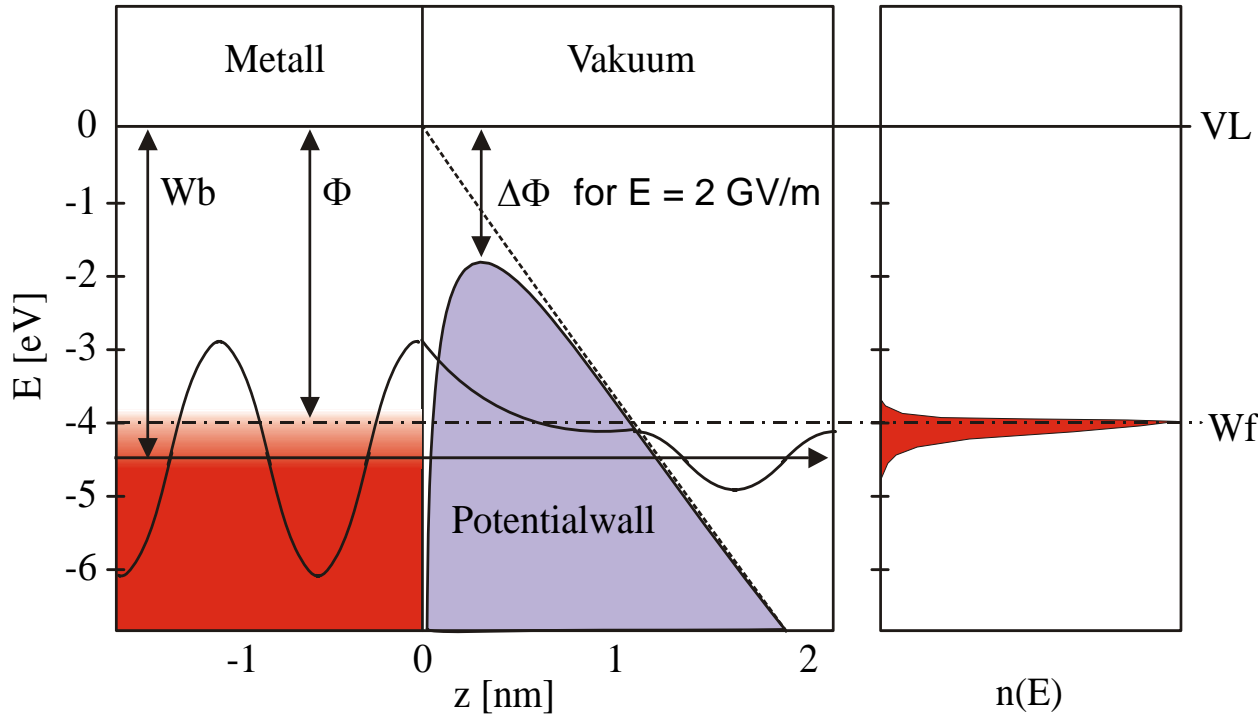
**Identification of relevant features for field limitation**

**Systematic improvement and control of surface quality**



# Field emission of electrons from flat metal surfaces

Electron waves of bound states in a metal can tunnel through the potential barrier  $V(z)$  at the solid surface into vacuum by means of the quantum mechanical tunnelling effect



$$V(z) = -e \cdot E \cdot z - \frac{e^2}{16\pi\epsilon_0 \cdot z}$$

work function  $\Phi$  of metal  
 applied field  $E$  on surface  
 image charge correction  
 $\Delta\Phi = \left(\frac{e^3 E}{4\pi\epsilon_0}\right)^{1/2}$

Calculation of the current density  $j(E)$  within the Fowler-Nordheim theory results in

$$j(E) = \frac{AE^2}{\Phi t^2(y)} \exp\left(-\frac{B\Phi^{3/2} v(y)}{E}\right)$$

with constants  $A=154$  and  $B=6830$  and slight correction functions  $t(y)$  and  $v(y)$

$$\Phi=4\text{eV at } E=2000 \text{ MV/m} \Rightarrow j=1 \text{ nA}/\mu\text{m}^2$$



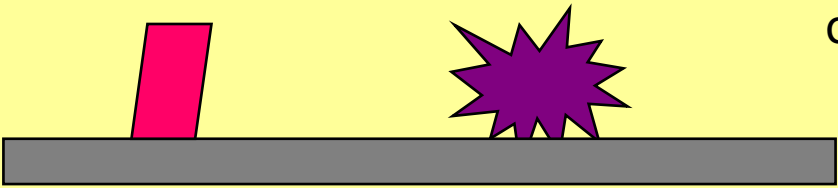
# Enhanced field emission of electrons from real surfaces

For real metal surfaces, i.e. **broad area cathodes with some roughness and pollution**, **nA currents occur at much lower fields (<100 MV/m)** than predicted by FN theory

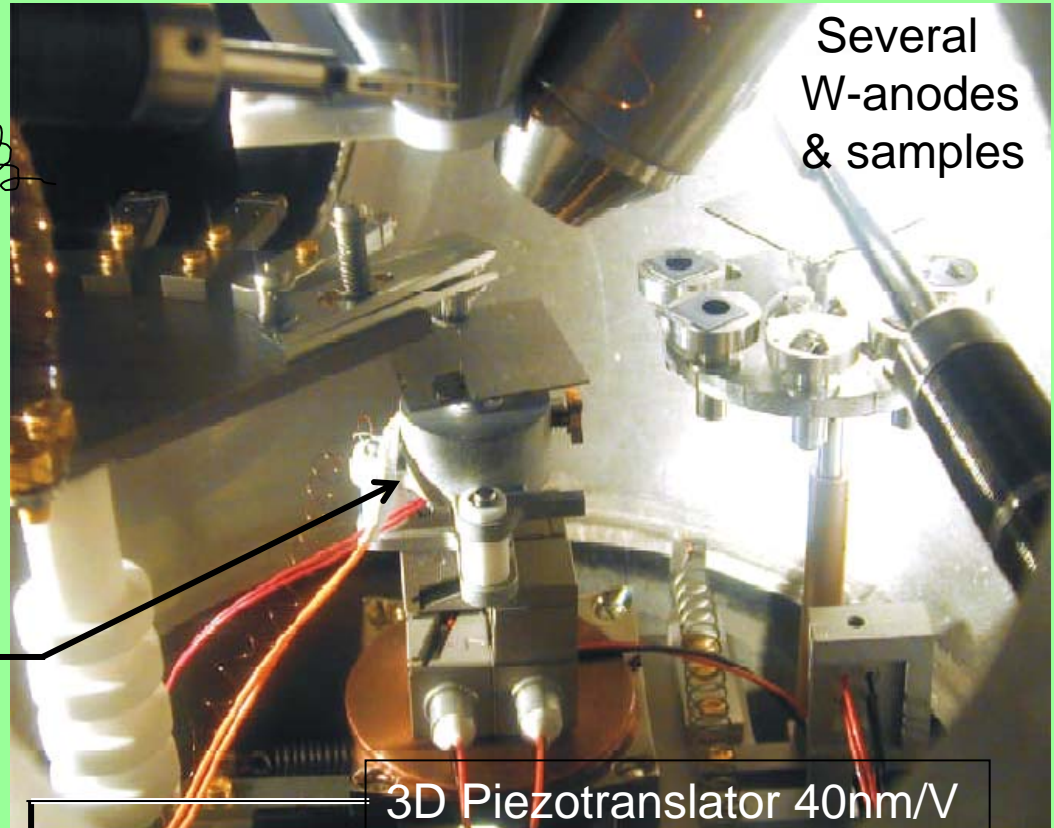
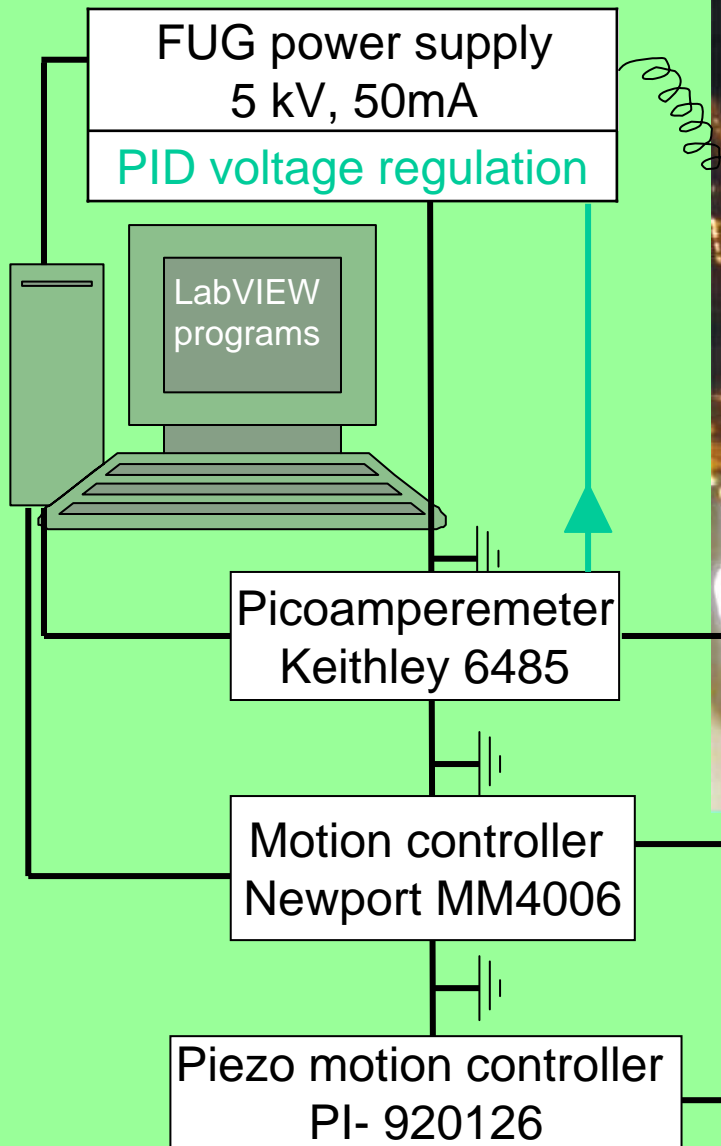
⇒ modified FN theory with **field enhancement factor  $\beta$**  describes at least the slope of **locally measured  $I(E)$  curves** quite well:

$$I(E) = S \frac{A(\beta \cdot E)^2}{\Phi} \exp\left(-\frac{B\Phi^{3/2}}{\beta \cdot E}\right) \quad \text{with emitting surface } S \text{ as fit parameter}$$

Theoretical models for **enhanced field emission** of real surfaces:

- **Geometric field enhancement** for **metallic protrusions/rough particulates** of height  $h$  and edge radius  $r_k$   
⇒  $\beta \approx h/r_k$   

- **Metal-Isolator-Vacuum** for metals with oxide layers ( $d < 10$  nm)  
⇒ irreversible creation of conducting channels ⇒ **switch-on effect**
- **Antenna or Metal-Isolator-Metal** for particles on oxidized metals  
after switch-on at  $\beta \approx h/d$  geometric field enhancement as above
- **Resonant tunneling through localized states** in adsorbates and oxides

# Field emission scanning microscope (FESM)

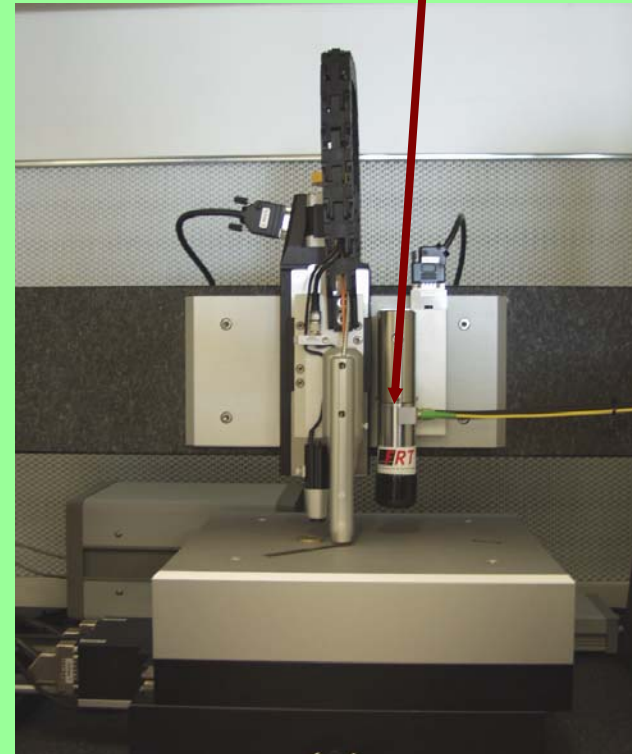


UHV system typically at  $2 \cdot 10^{-7}$  Pa  
LabVIEW automated scans of  $U(x,y)$  for 2 nA  
Scanning speed: (100×100) pixels in 1 hr  
I/V curves and localization of stable emitters



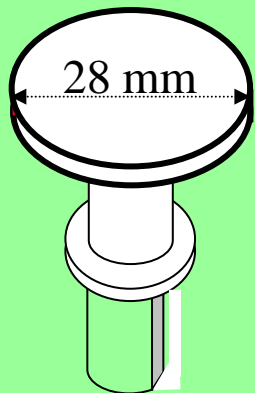
# Profilometer with AFM and SEM with EDX

Additional **surface analysis** of whole samples and **relocalized areas of enhanced FE**  
Optical profilometer with lateral resolution of 2  $\mu\text{m}$  and height resolution of 3 nm  
combined with **atomic force microscope AFM**  
Scanning speed: (100x100) pixels in 1 min

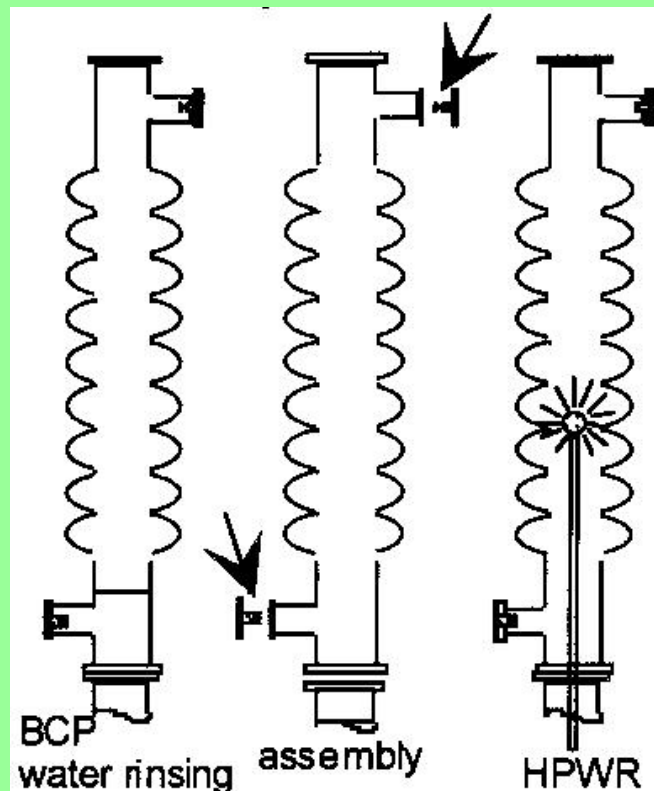


Scanning electron microscope SEM (XL-30)  
with energy dispersive X-ray analysis EDX

# Preparation techniques for Nb samples



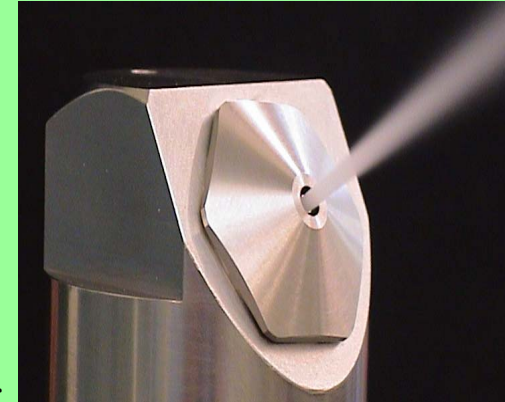
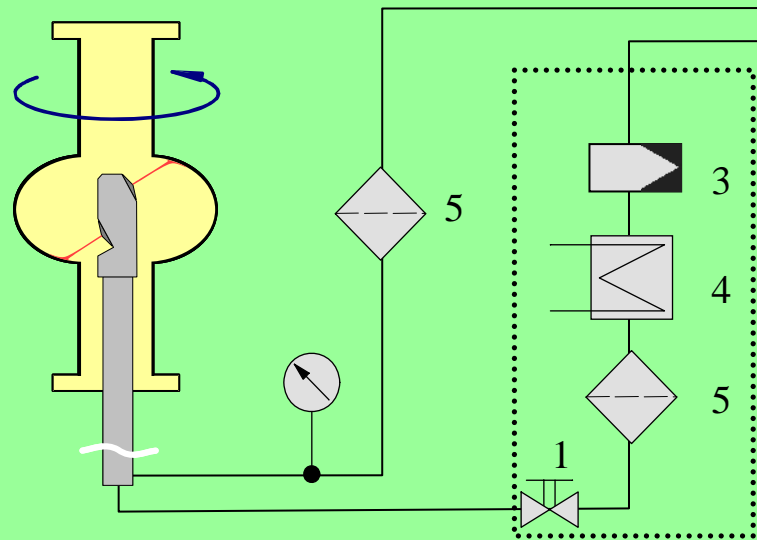
Nb samples prepared like cavities at DESY  
Buffered chem. BCP or electropolished EP  
and **high pressure rinsed HPR** with water  
mostly in single cells, few in 9-cell cavities



Ultra pure water	$\rho > 18\text{M}\Omega\text{cm}$
Water flow	7 - 20 l/min
Water pressure	80-150 bar
Rotation speed	4-5 rpm

# Dry ice cleaning of Nb samples (DIC)

Process developed at FH Stuttgart and adapted for cavities at DESY

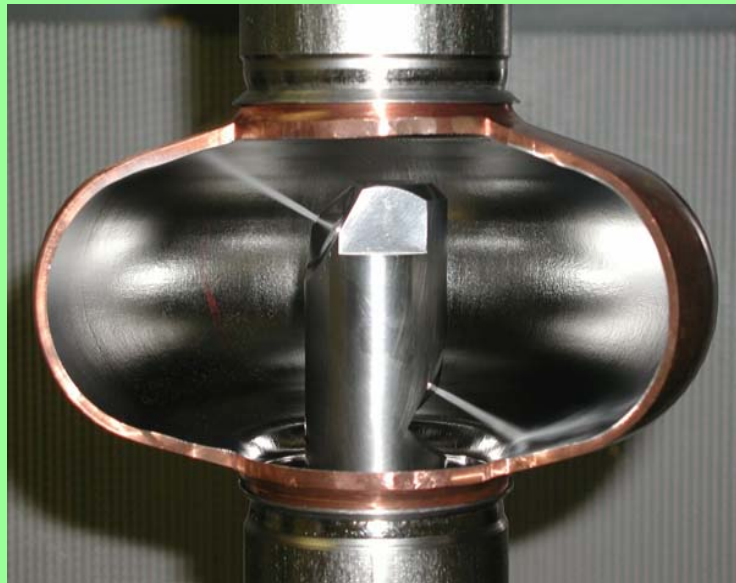


- 1 valve
- 2 pressure reducer
- 3 gas purifier
- 4 chiller
- 5 filter

CO<sub>2</sub> pressure ~ 50 bar  
N<sub>2</sub>-pressure: 12 - 18 bar  
Particle filter < 0.05 μm  
CO<sub>2</sub> temperature > -40°C

3 cleaning effects:

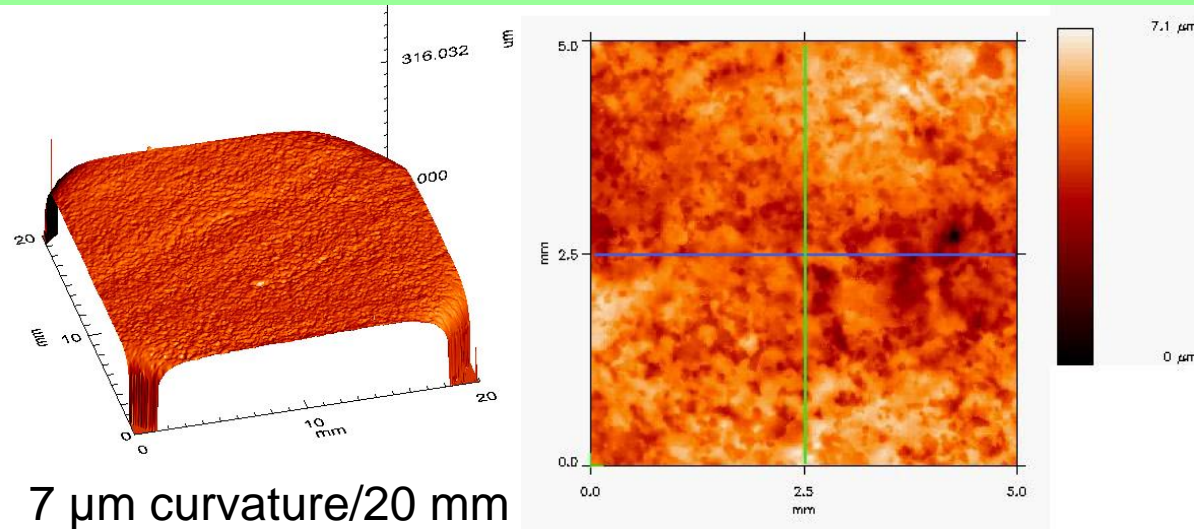
- Mechanically
  - impact of snow crystals ⇒ **shearing forces**
- Chemically
  - liquid CO<sub>2</sub> is good **solvent for hydrocarbons**
- Thermally
  - rapid cooling ⇒ **brittling of contaminants**
  - sublimated volume increase × 500



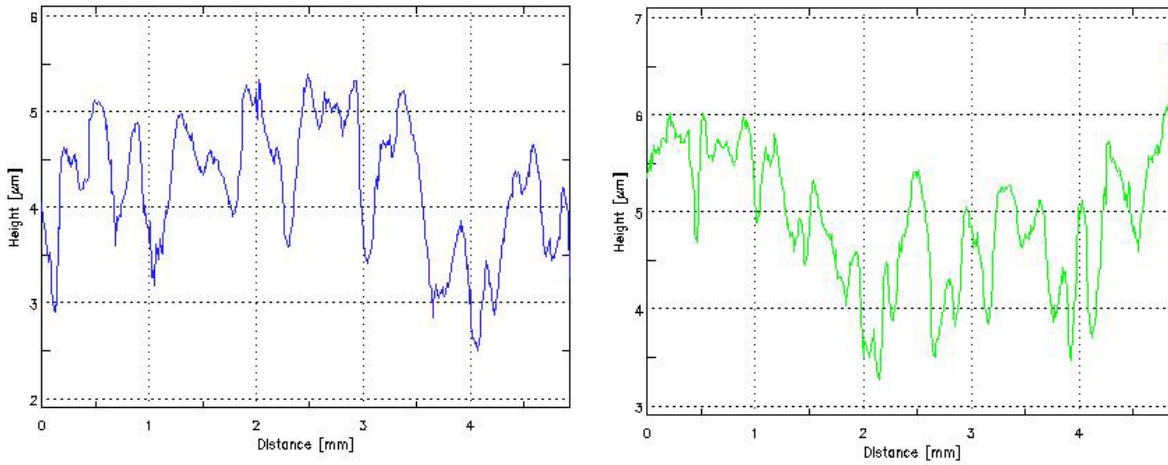


# Quality control scans of EP/HPR-Nb prepared in 9-cell cavity

Profiles of whole sample and central part of sample  
scanned area  $20 \times 20 \text{ mm}^2$   $5 \times 5 \text{ mm}^2$

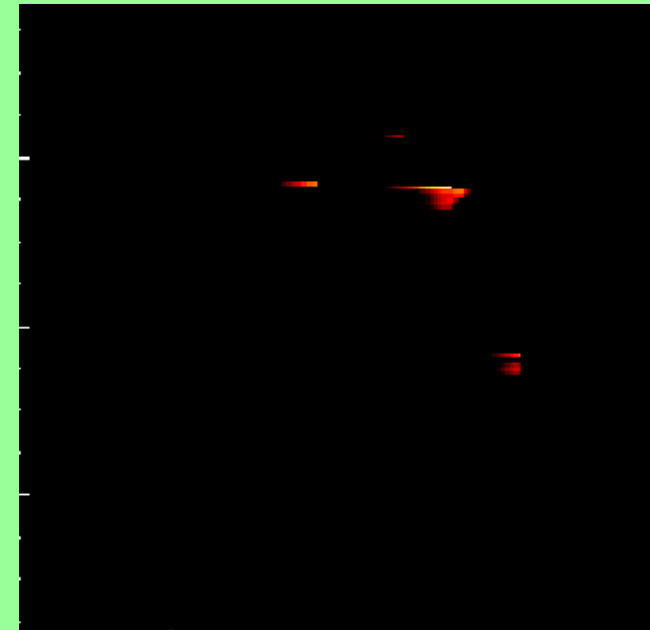


7 μm curvature/20 mm



Line scans  $\Rightarrow$  grooves  $< 2 \text{ μm}$ , roughness  $< 0.2 \text{ μm}$

PID-regulated  $U(x,y)$  for 1 nA  
scanned area =  $7.5 \times 7.5 \text{ mm}^2$   
flat W-anode  $\varnothing_a = 100 \text{ μm}$   
anode voltage  $U = 4800 \text{ V}$   
electrode spacing  $\Delta z = 32 \text{ μm}$



no emission @ 120MV/m  
5 emitters @ 150MV/m



best EP/HPR sample yet



# Emitter distribution on single crystal Nb after BCP/HPR

Alternative approach for **mirror-like surfaces**: large crystal Nb+BCP30 $\mu$ m/HPR

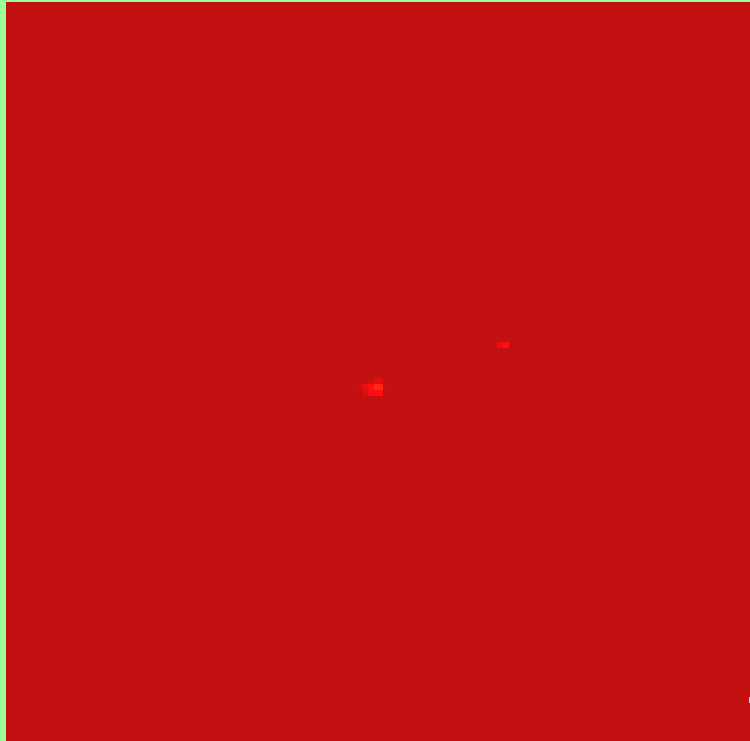
PID-regulated **voltage maps**  $U(x,y)$  for 1 nA scanned area = 7.5 $\times$ 7.5 mm<sup>2</sup>

flat W-anode  $\varnothing_a = 100 \mu\text{m}$

electrode spacing  $\Delta z = 32 \mu\text{m}$

anode voltage  $U = 4800 \text{ V}$

$\Delta z = 24 \mu\text{m}$



no emission @ 120MV/m

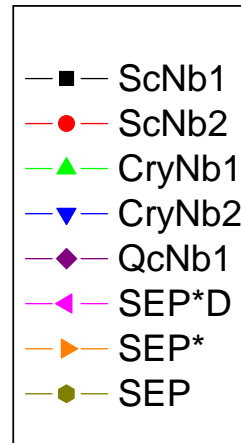
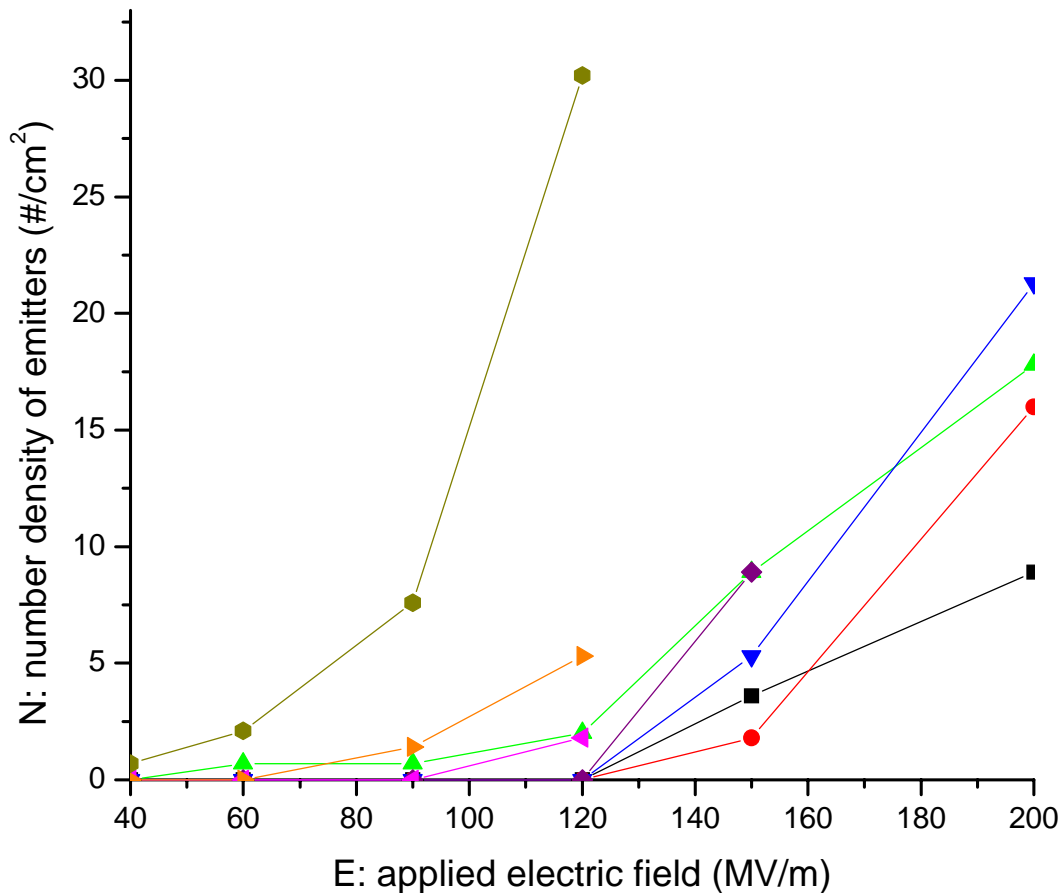
2 emitters @ 150MV/m



5 emitters @ 200MV/m

$\Rightarrow$  **best FE performance of all Nb samples yet**

# Emitter statistics for various types of Nb samples



single crystal #1  
 & #2, BCP30HPR  
 3 large grains #2  
 & #1, BCP30HPR  
 EP+HPR at DESY  
 EP+HPR+DIC  
 EP+HPR  
 EP at Saclay

$$E_{\text{peak}} = 2 \times E_{\text{acc}}$$

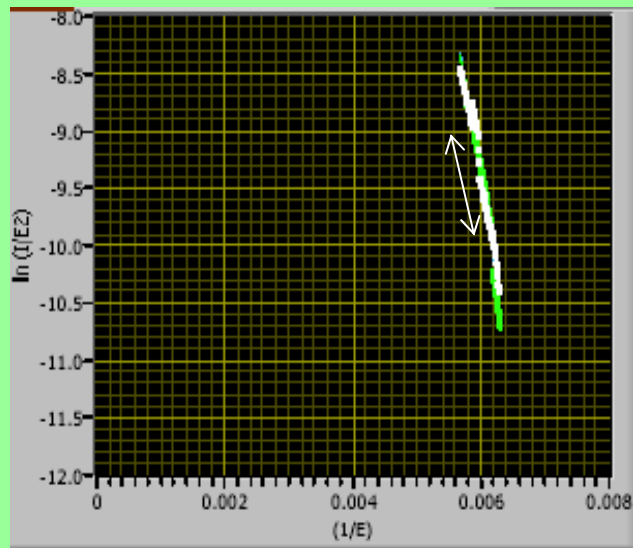
Systematically reduced FE by EP+HPR, DIC and large crystal Nb  
 BCP+HPR of large crystal Nb is probably sufficient



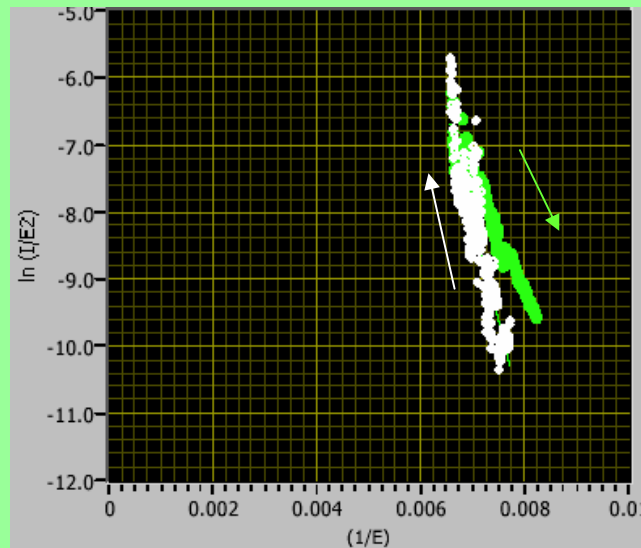
# Locally measured I/V-curves and FN-Analysis of emitters

$$\ln\left(I(E)/E^2\right) = \ln\frac{S \cdot A \cdot \beta^2}{\Phi} - \frac{B \cdot \Phi^{3/2}}{\beta} \frac{1}{E}$$

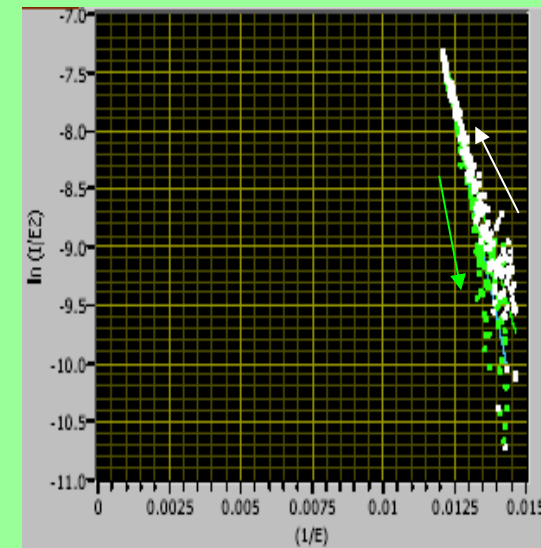
Typical FN-plots of a stable



activated



deactivated emitter



$E_{on}(1 \text{ nA}) = 76.9 \text{ MV/m}$

$\beta_{\uparrow} = 19.3 \quad S_{\uparrow} = 1 \times 10^{-13} \text{ m}^2$

$\beta_{\downarrow} = 17.9 \quad S_{\downarrow} = 5 \times 10^{-13} \text{ m}^2$

$E_{on}(1 \text{ nA}) = 103.3 \text{ MV/m}$

$\beta_{\uparrow} = 17.4 \quad S_{\uparrow} = 1 \times 10^{-11} \text{ m}^2$

$\beta_{\downarrow} = 31.2 \quad S_{\downarrow} = 3 \times 10^{-16} \text{ m}^2$

$E_{on}(1 \text{ nA}) = 54.3 \text{ MV/m}$

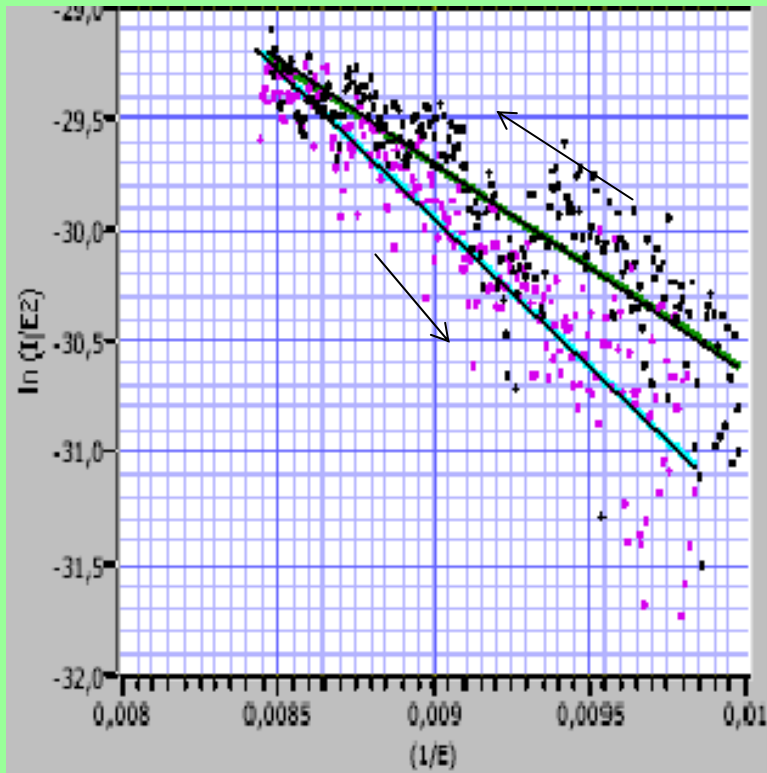
$\beta_{\uparrow} = 67.4 \quad S_{\uparrow} = 2 \times 10^{-17} \text{ m}^2$

$\beta_{\downarrow} = 61.2 \quad S_{\downarrow} = 1 \times 10^{-15} \text{ m}^2$

After first processing, most emitters are stable up to 100 nA



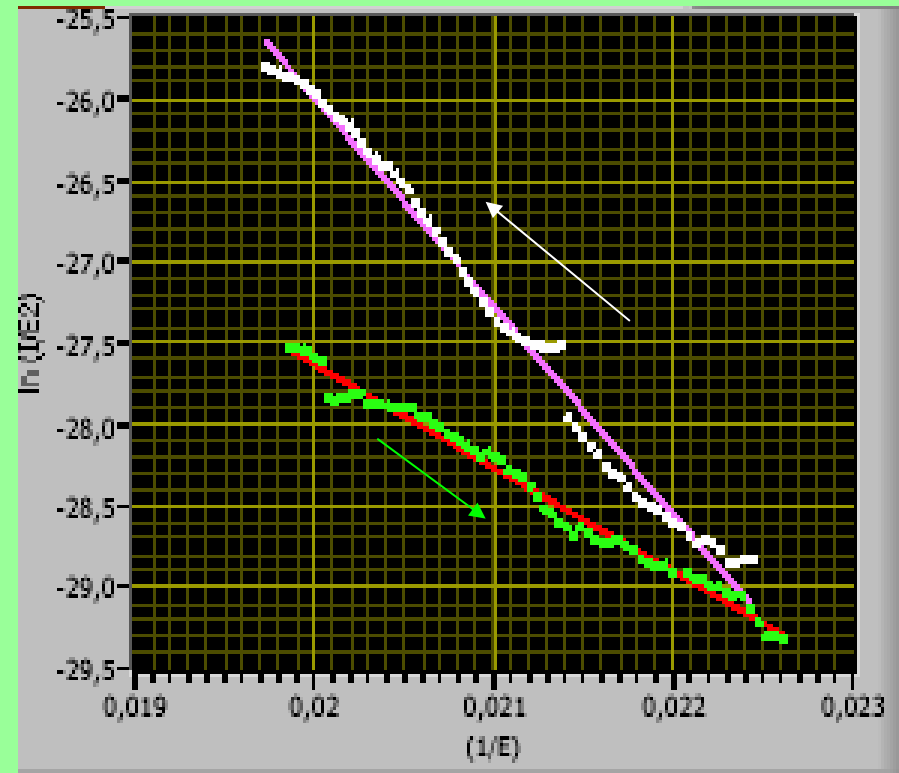
# Current processing of instable emitters



$$E_{\text{on}}(1 \text{ nA}) = 33 \text{ MV/m}$$

$$\beta_{\uparrow} = 231 \quad S_{\uparrow} = 3 \times 10^{-19} \text{ m}^2$$

$$\beta_{\downarrow} = 160 \quad S_{\downarrow} = 2 \times 10^{-17} \text{ m}^2$$



$$E_{\text{on}}(1 \text{ nA}) = 44.6 \text{ MV/m}$$

$$\beta_{\uparrow} = 59.5 \quad S_{\uparrow} = 8 \times 10^{-10} \text{ m}^2$$

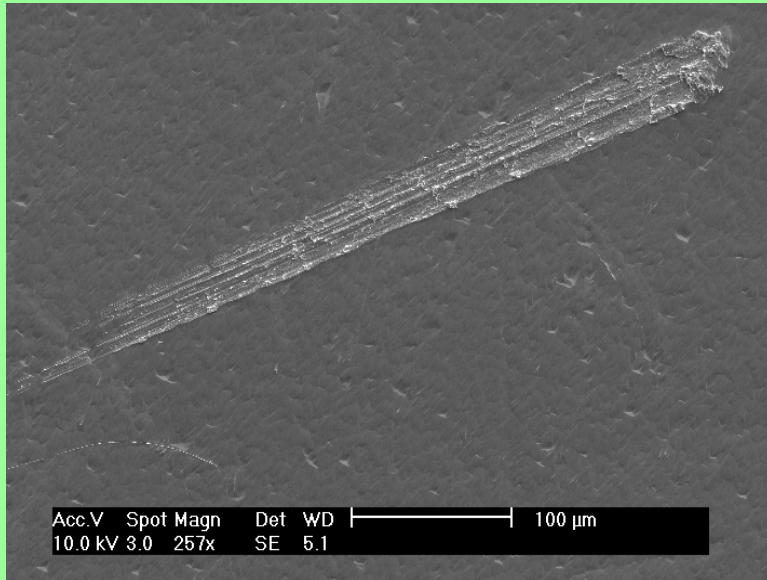
$$\beta_{\downarrow} = 119 \quad S_{\downarrow} = 1 \times 10^{-16} \text{ m}^2$$

Fluctuations / oscillations most probably caused by adsorbates

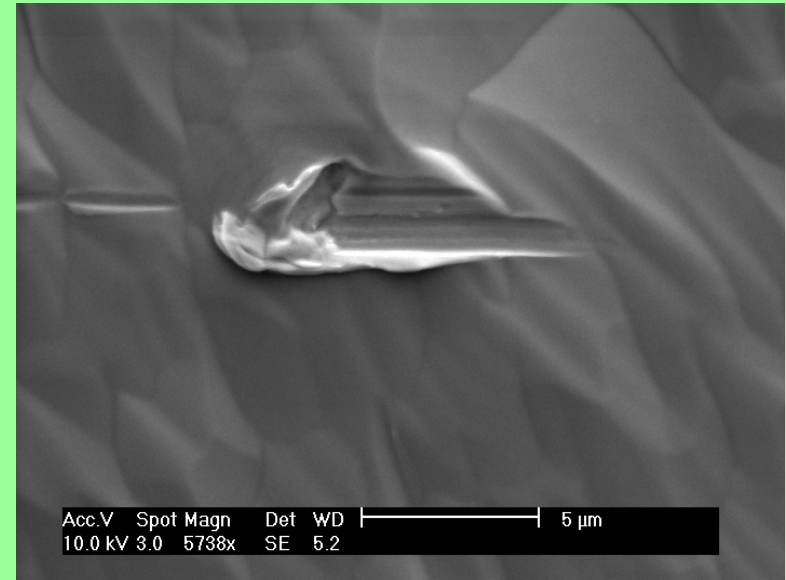
Understanding of instabilities and nature of emitters very difficult



# Typical protrusion emitters containing only Nb (+ O?)



$E_{on}(2nA) < 60$  MV/m  
~500 μm long scratch  
(mishandling of sample)

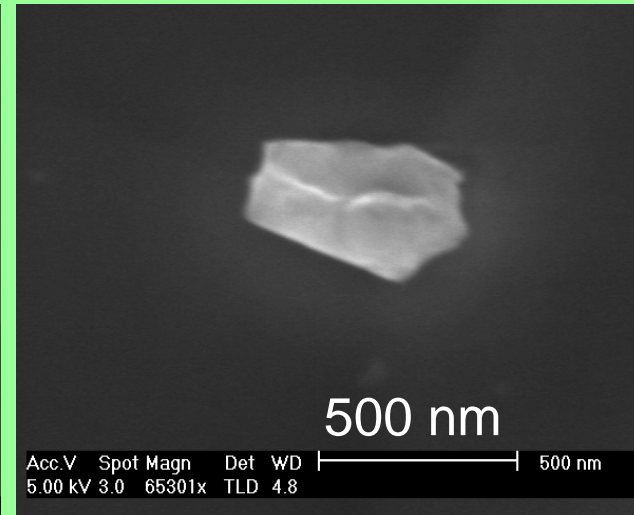
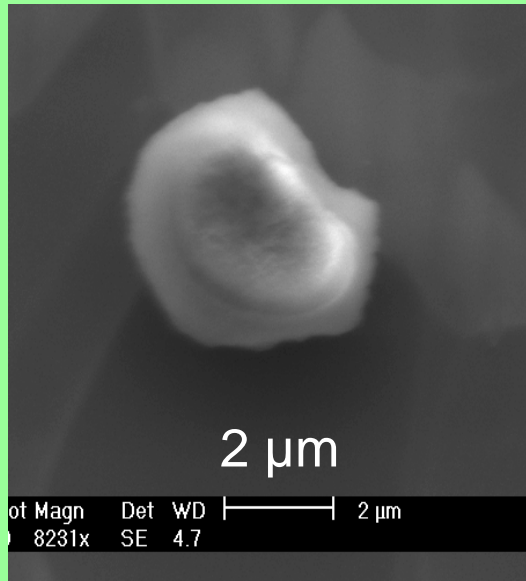
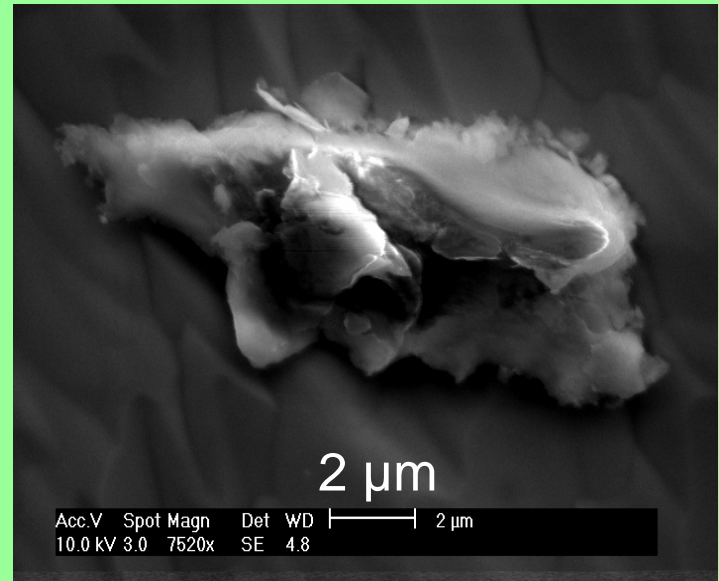


$E_{on}(2nA) = 90$  MV/m  
~5 μm long groove  
 $\beta = 71$ ,  $S = 2.3 \cdot 10^{-6} \mu\text{m}^2$



$E_{on}(2nA) > 140$  MV/m  
~1 μm small defect  
 $\beta = 59$ ,  $S = 7 \cdot 10^{-8} \mu\text{m}^2$

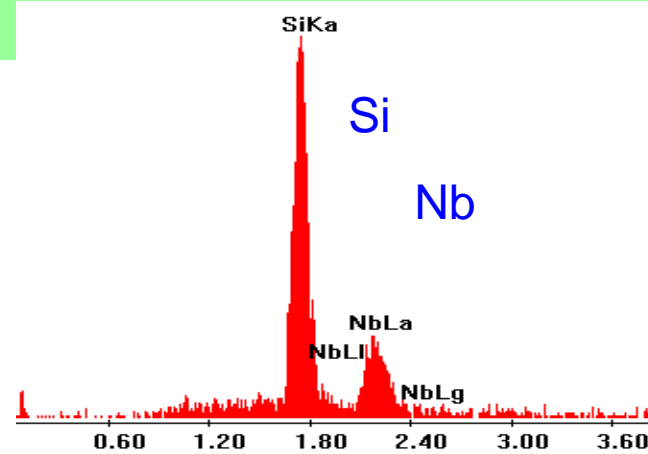
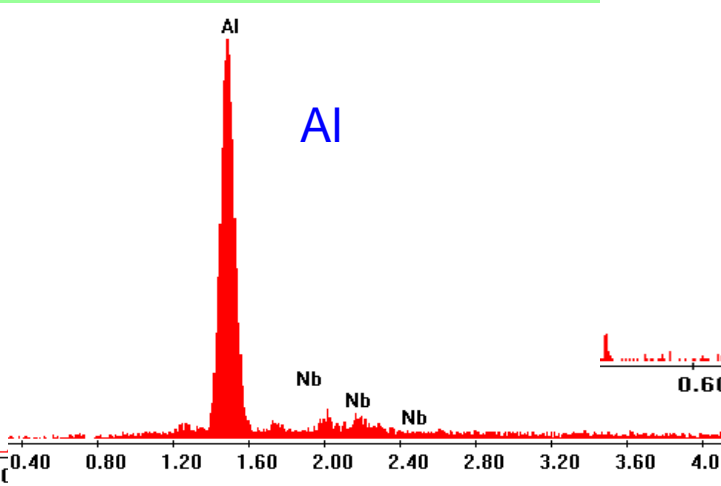
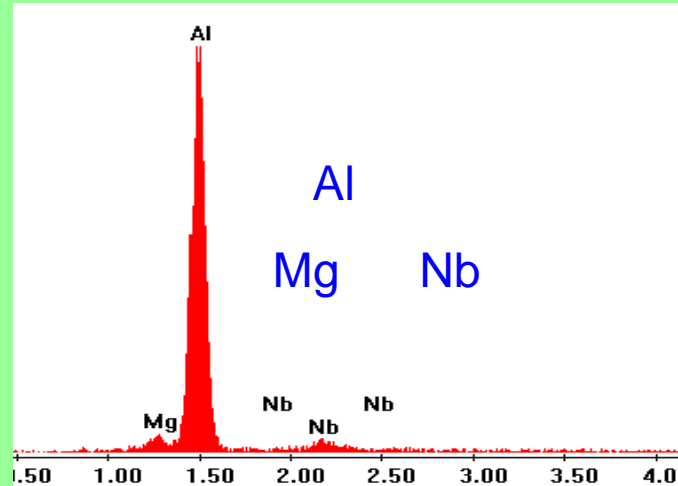
# Typical particulate emitters containing impurities



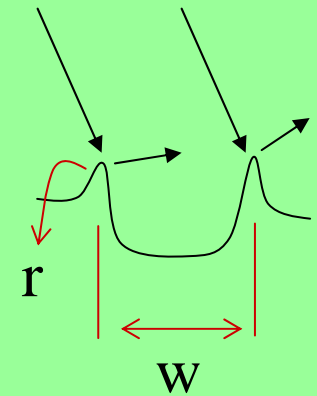
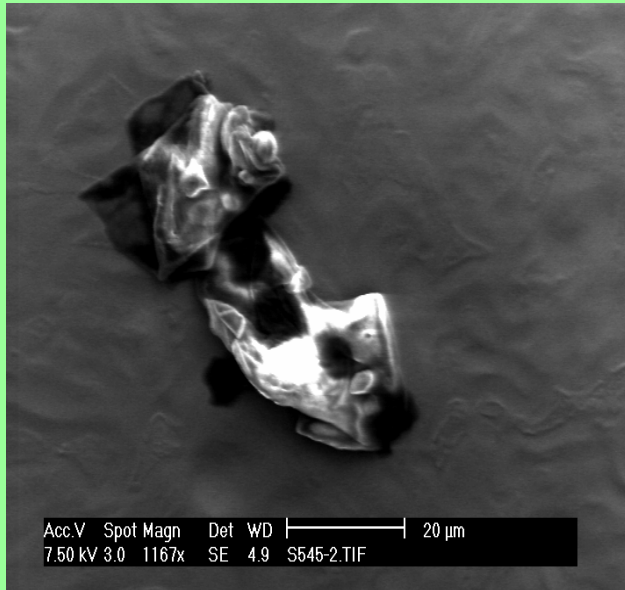
$E_{on}(2nA) = 140 \text{ MV/m}$   
 $\beta = 31, S = 6.8 \cdot 10^{-6} \mu\text{m}^2$

$E_{on}(2nA) = 132 \text{ MV/m}$   
 $\beta = 27, S = 7 \cdot 10^{-5} \mu\text{m}^2$

$E_{on}(2nA) > 120 \text{ MV/m}$   
 $\beta = 46, S = 6 \cdot 10^{-7} \mu\text{m}^2$



# Effect of DIC on particulate and protrusion emitters



$$\Rightarrow \beta = h/r \sim w/r$$

$$S \sim r^2$$

$E_{on}(1nA) = 77 \text{ MV/m}$

S particulate removed by DIC

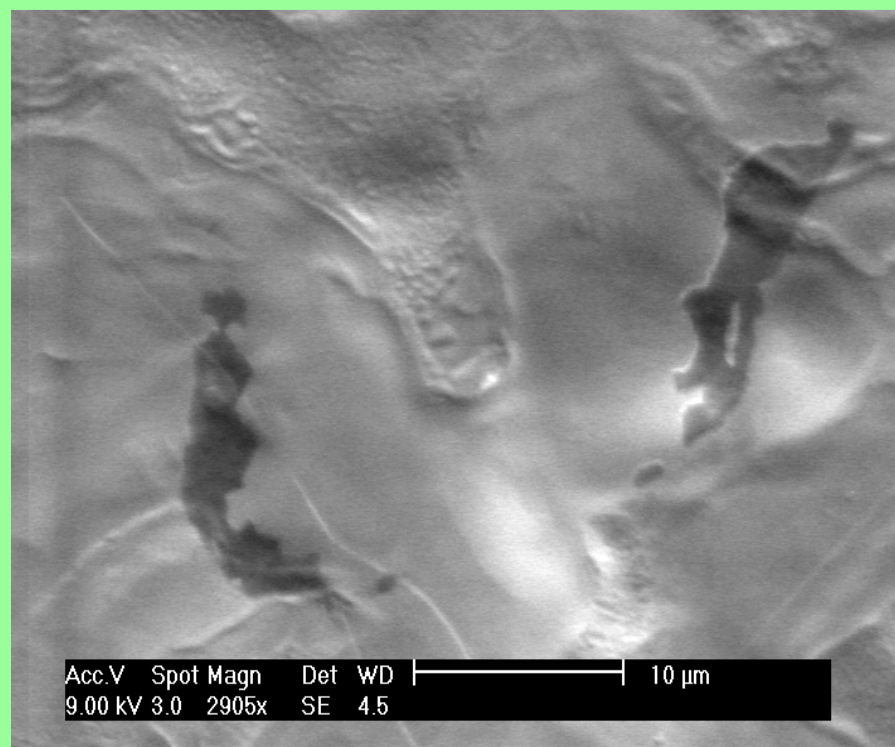
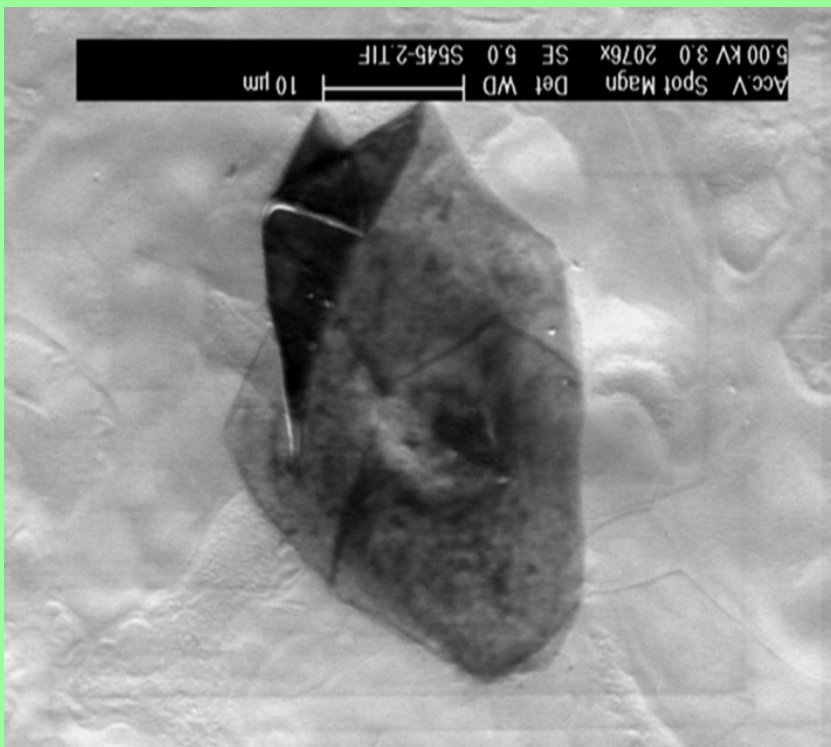
Protrusion	HPR	HPR+DIC
$E_{on}$ (MV/m)	48.5	103.3
$\beta_{\uparrow}$	166.7	17.4
$\beta_{\downarrow}$	147	31.2
$S_{\uparrow}$ (m <sup>2</sup> )	$1.6 \times 10^{-20}$	$9.6 \times 10^{-12}$
$S_{\downarrow}$ (m <sup>2</sup> )	$7.2 \times 10^{-20}$	$3.3 \times 10^{-16}$

FE of protrusion much reduced by DIC





# Effect of DIC on a flake-like emitter with exposed edge



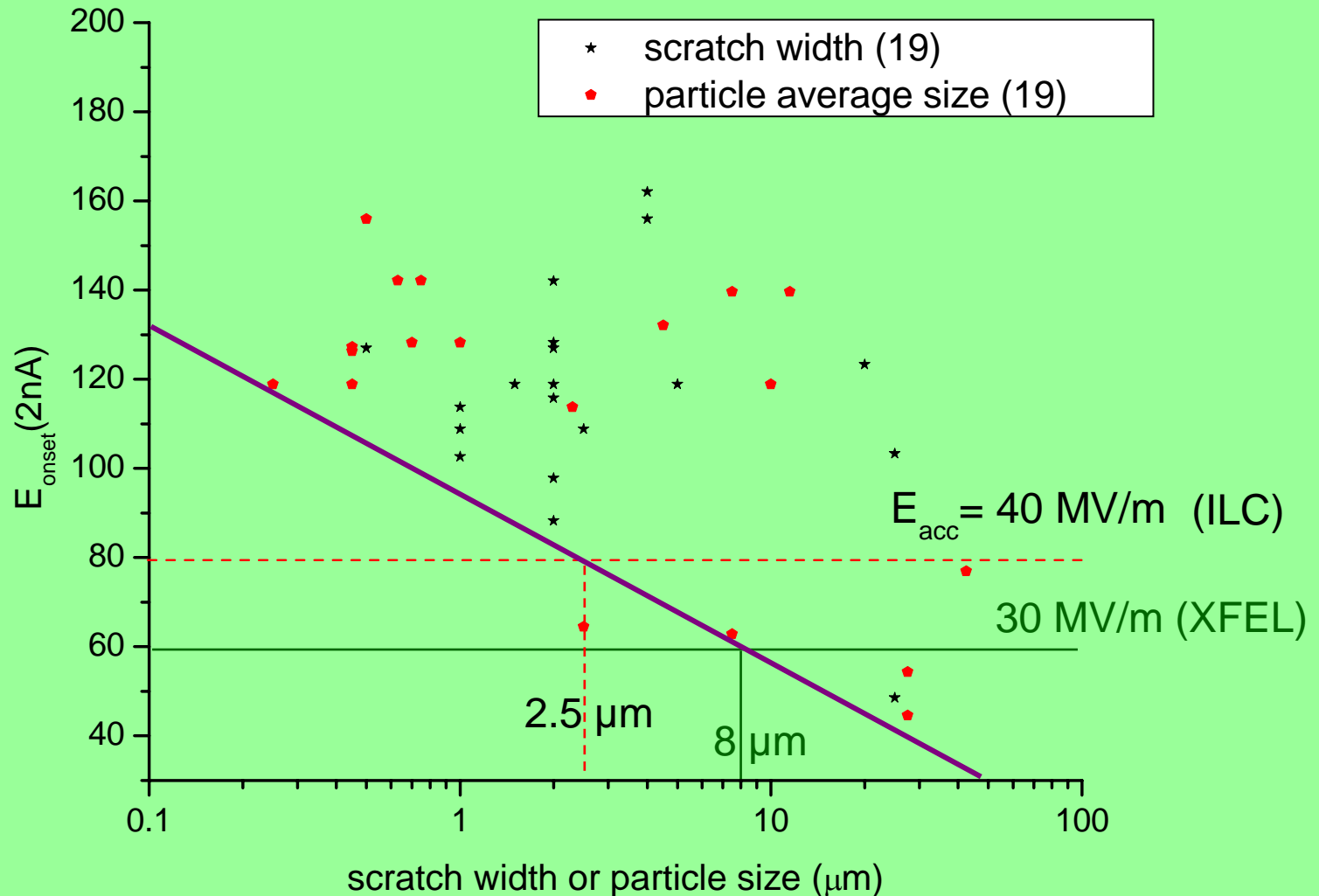
emitter of ~ 20 μm size destroyed by DIC  
remnants emitting at higher  $E_{on}$ !

EDX: no foreign element detected  
(probably oxide of Nb)

emitter	HPR	HPR+DIC
$E_{on}$ (MV/m)	54.3	62.8
$\beta_{\uparrow}$	67.4	35.4
$\beta_{\downarrow}$	51.2	38.0
$S_{\uparrow}$ (m <sup>2</sup> )	$2 \times 10^{-17}$	$8.3 \times 10^{-13}$
$S_{\downarrow}$ (m <sup>2</sup> )	$1.2 \times 10^{-15}$	$2.4 \times 10^{-13}$

# Correlation between FE onset field and emitter size ?

based on FE measurements and SEM analysis of 38 field emitters



Evidence for correlation  $\Rightarrow$  fast FE quality control by emitter size



## Conclusions and outlook !

- Standard EP+HPR Nb sample provides good FE performance  
no field emission up to  $E_{on} = 120 \text{ MV/m} \Rightarrow E_{acc} = 60 \text{ MV/m}$
- Large Nb crystal BCP+HPR samples show best FE results  
 $\Rightarrow$  interesting alternative for cavity fabrication !
- Particulates and protrusions identified as relevant emitters
- DIC effectively removes particulates and weakens protrusions
- After first processing, most emitters are stable up to 100 nA  
 $\Rightarrow$  instabilities and nature of emitters challenging !
- Evidence for correlation between onset field and emitter size  
 $\Rightarrow$  fast FE quality control on samples for XFEL !



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