Suppressing the Enhanced Field Emission from Nb Surfaces

-Arti Dangwal Pandey

- Motivation for field emission (FE) studies
- Field emission scanning microscope (FESM)
- FE Measurements on:

  Polycrystalline, single crystal, and large grain Nb surfaces

Bergische
universität
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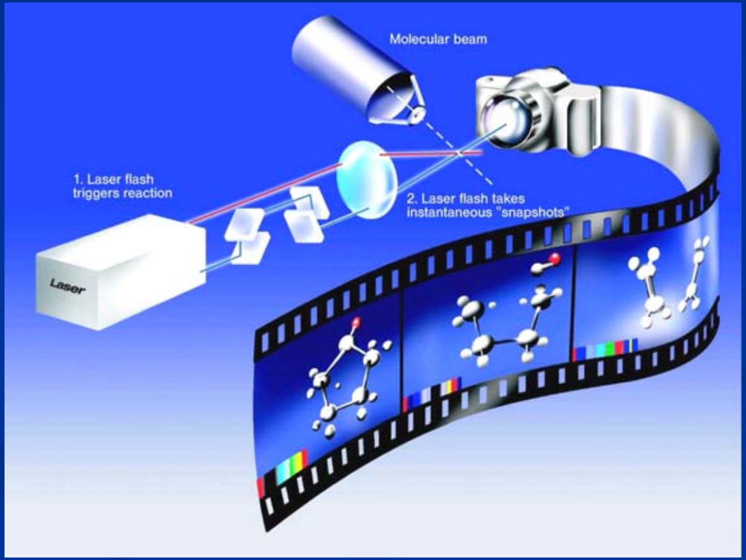
27th Jan 2009, DESY Hamburg

DESY

Motivation for field emission studies

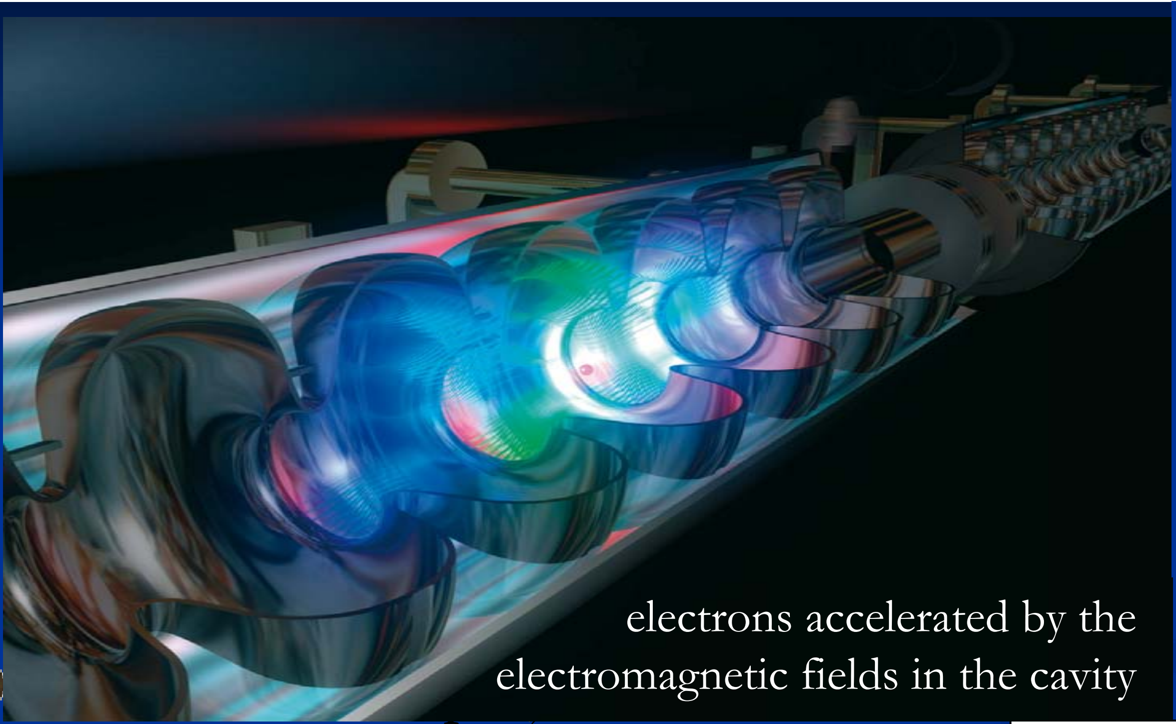
- Field emission from Nb/Cu surface:
  imposes limitation to high gradient linear accelerators
  ⇒ Challenges to future projects!



- The X-ray free electron laser (XFEL)
  - Ultrashort pulses (< 100 fs)
  - atomic wavelength (60 - < 1 Å)
  - extremely high intesity and brilliance
  - ⇒ to record films with atomic details

  e.g. Chemical process, formation of solids, studying plasma etc.
- TeV e^+e^- International Linear Collider (ILC)
  - 10 billion particles collide 4000 times/sec

FE: challenge to future linear accelerators



electrons accelerated by the electromagnetic fields in the cavity

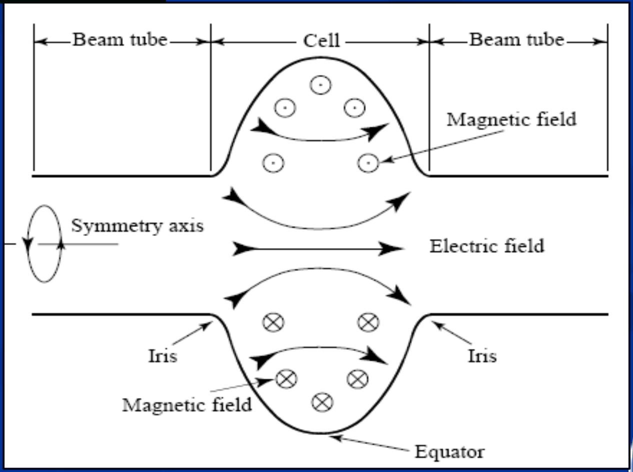
Structure of a 9-cell Nb cavity: component of linear accelerator

- Utilization of superconducting Nb cavities
- Limited by onset of Field emission (FE):

  @ $E_{acc} \approx$ 20-25 MV/m (typically)
- E_{acc} for XFEL (ILC): ~23.6 (31.5) MV/m,

  nr. of 9-cell cavities = 928 (16,000)

Avoid field emission!

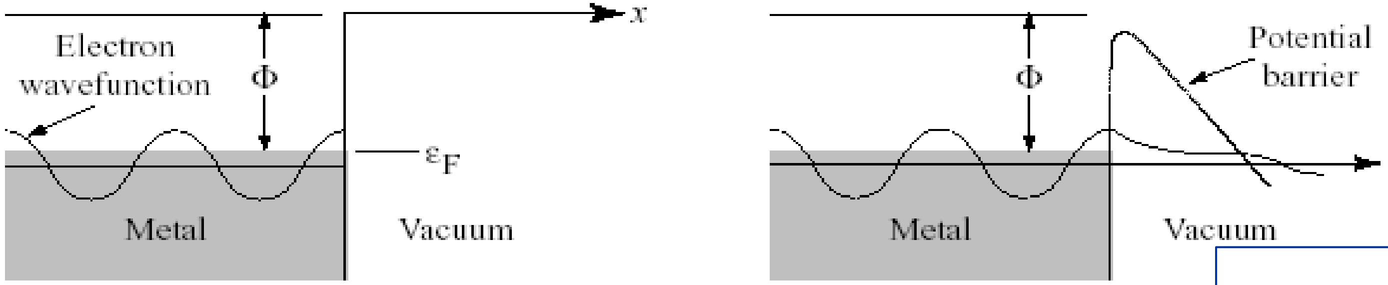


At iris of cavity: Esurface≈ 2 ·Eacc

Field Emission: an introduction

"Tunneling of electrons from a solid due to high electric fields"

Electrostatic potential of metal – vacuum interface



$$V(x) = -\frac{e^2}{16\pi\varepsilon_0 x} - eEx$$

(a) $E_{ext} = 0$
e^- confined by the potential barrier
(infinitely thick)

(b) $E_{ext} = E$
deformation of potential barrier
(finite thickness)

Fowler-Nordheim equation:

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

where, I in A, S in cm², E in MV/m, Φ in eV, then A= 154, B= 6830

e.g. for Nb surface: Φ= 4 eV, S = 10^{-14} m²

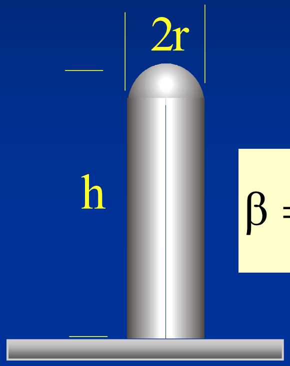
to get tunneling current of ≈ 1 µA, Required field ≈ 3GV/m

But… tunneling current is observed experimentally at ≈ 40 MV/m

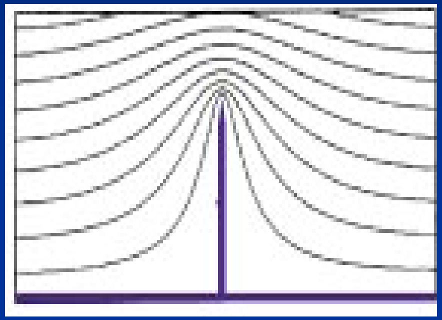
Enhanced Field Emission

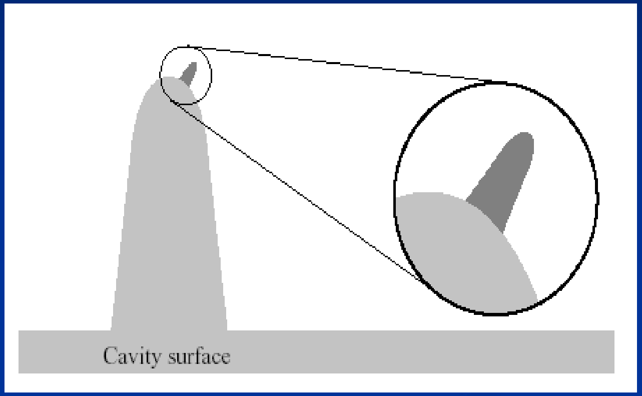
due to microparticles and irregularities on surface

⇒ Geometric field enhancement factor "β"   Range of β: 10 to 10000



$$\beta = \frac{h}{r} + 2$$





βeff = β1× β2

Modified FN equation for enhanced FE:

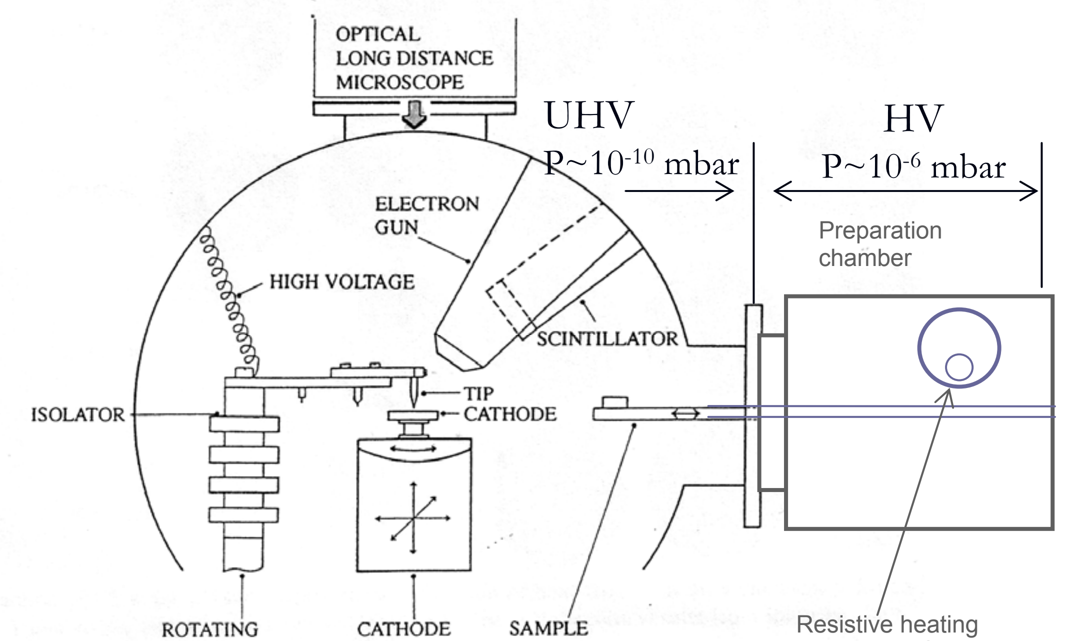
$$I = S.\frac{A(\beta \cdot E)^2}{\Phi} \exp\left(-\frac{B \cdot \Phi^{3/2}}{\beta \cdot E}\right)$$

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

(FN plots: straight line)

slope → β, intercept → S factor, for known Φ

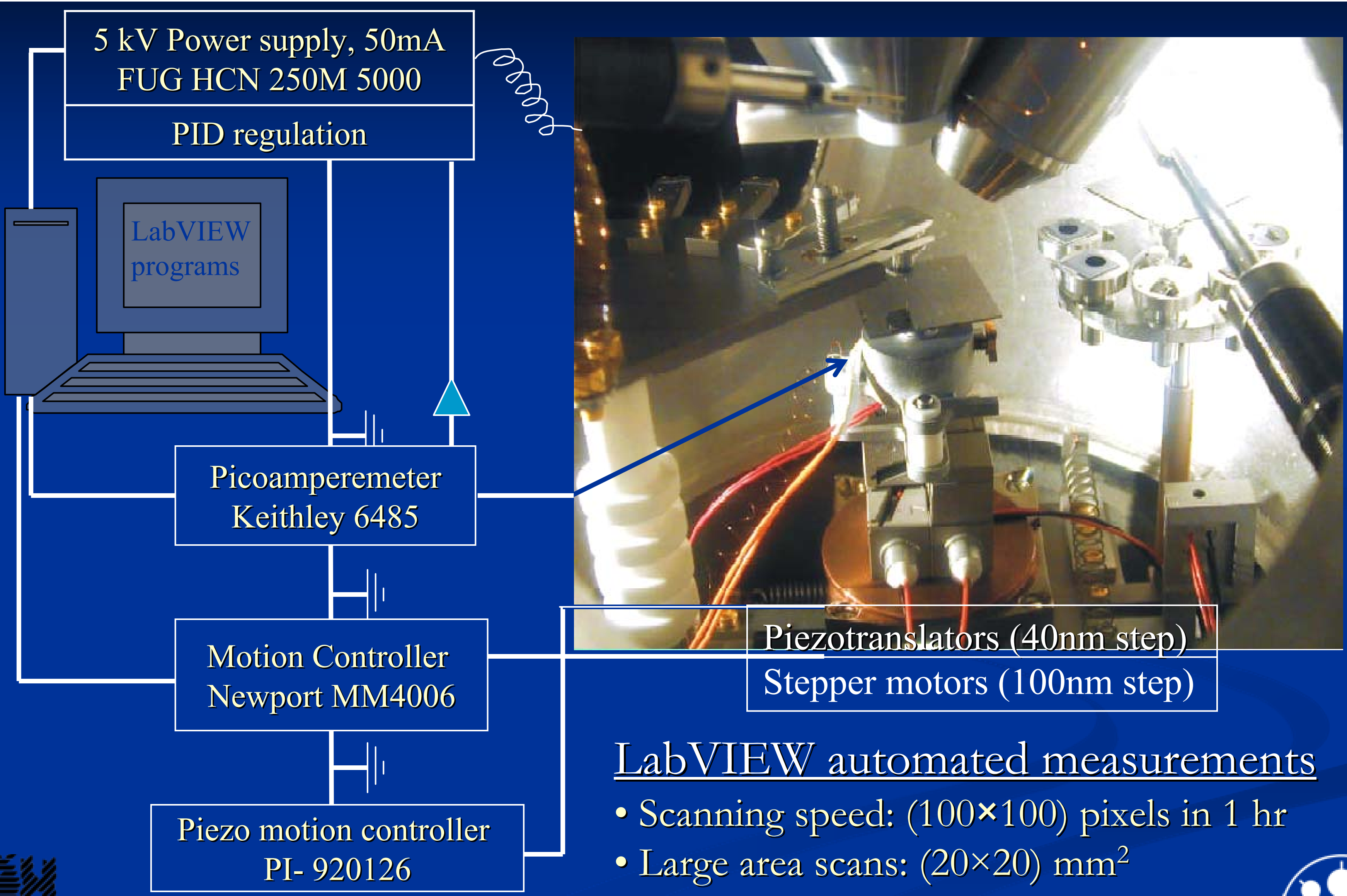
Field emission scanning Microscope (FESM)



Sample size up to Ø = 30 mm
Flat W-anodes Ø = 0.1-1 mm
exchangeable W- tip anodes
with apex $Ø_a$ = 20 nm - 20 µm

- Voltage scans V(x,y) for a given max. current at const. electrode distance ‘d’ with fast voltage regulation to prevent discharges
- Current scans I(x,y) for a given voltage at constant electrode distance
- High resolution measurements of the field emission properties of single emitters (I/V-curves, current limits, stability, fluctuations)
- High vacuum in-situ heat treatments

Field Emission Scanning Microscope (FESM)



5 kV Power supply, 50mA
FUG HCN 250M 5000

PID regulation

LabVIEW programs

Picoamperemeter
Keithley 6485

Motion Controller
Newport MM4006

Piezo motion controller
PI- 920126

Piezotranslators (40nm step)

Stepper motors (100nm step)

LabVIEW automated measurements

- Scanning speed: (100×100) pixels in 1 hr
- Large area scans: $(20\times20)\ \mathrm{mm}^2$

⇒ Better statistics

Enhanced FE: causes and remedies

- Field emitters on broad area cathode and cavity surface:
  - Micro particles of irregular shapes; typical size: 0.5-20 μm
  - ~ 10% of particles are emitters
  - Hydrocarbon contamination of vacuum system
  - Surface irregularities like scratches caused by massive tools
- Solutions:
  - Dustfree assembly
  - Careful fabrication and handling of the cavities
  - Using advanced cleaning techniques to avoid contamination

Cleaning techniques for Nb cavities

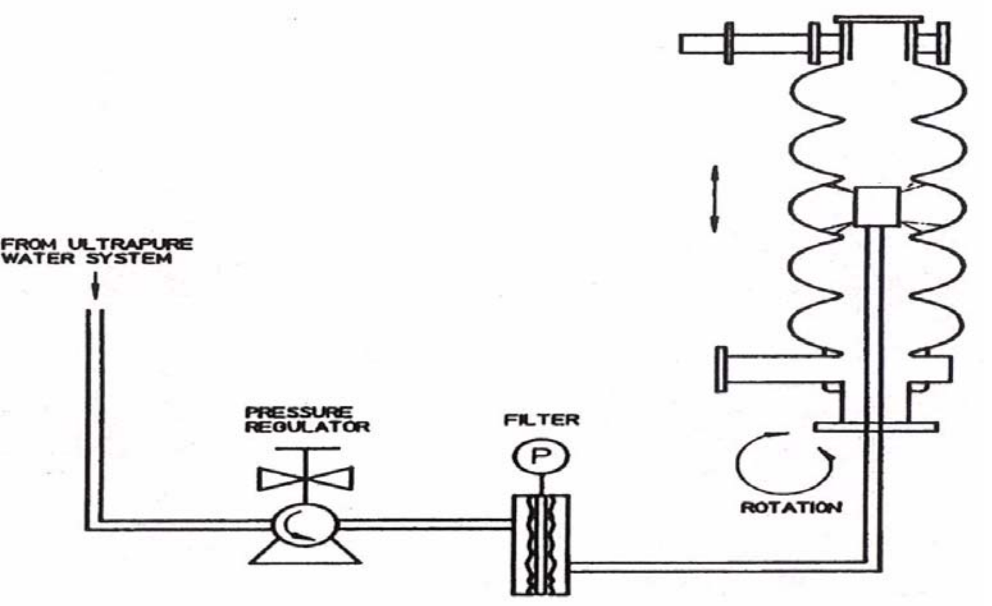
- Standard technique:

  High pressure water rinsing
  - particle removal down to 1-2 µm
- Proposed additional technique

  Dry ice cleaning
  - Particle removal down to < 100 nm
  - All applications unsuitable for water
  - No drying procedure necessary

Use of cleanroom during cavity cleaning and installation: already in practice now

High Pressure Rinsing (HPR)

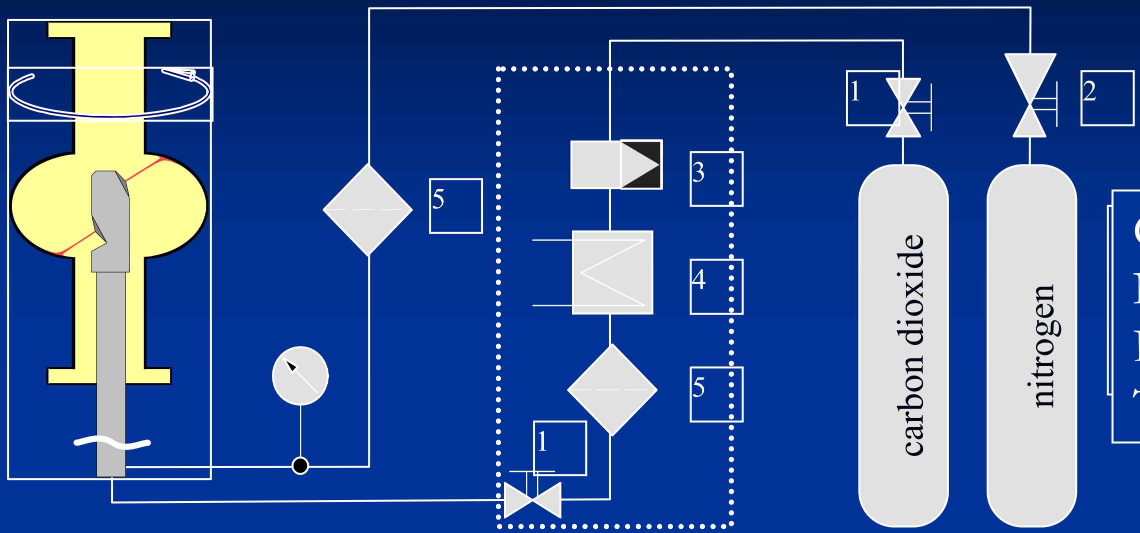


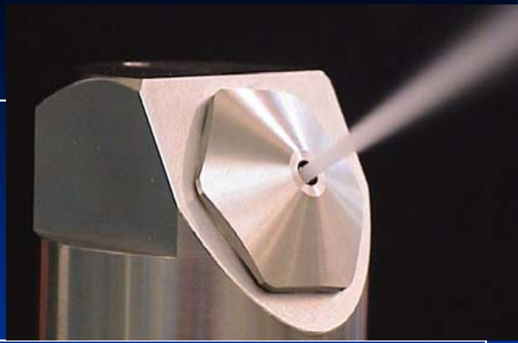
Ultra pure water	ρ >18MΩcm
Water flow	7 - 20 l/min
Water pressure	80-150 bar
Cavity rotation speed	4-5 rpm

Clean room: Class 10

Removal of μm size particles

Dry ice cleaning (DIC)





CO$_2$-pressure ~ 50 bar
N$_2$-pressure: 12 - 18 bar
Particle filtration < 0.05 µm
Temp. (liq. CO$_2$): -5° to - 40° C

1. Thermal effects

   Rapid cooling, increased volume after sublimation: power

2. Chemical effect

   liq. CO$_2$: good solvent for non polars (e.g. hydrocarbons)

3. Mechanical effect

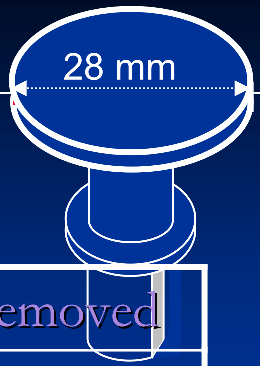
   High momentum snow crystals: shearing force

Specially constructed nozzle for cavity cleaning

Removal of particles down to < 100 nm
No drying procedure necessary ☺

Investigated Nb samples

for fundamental investigation of new cleaning techniques



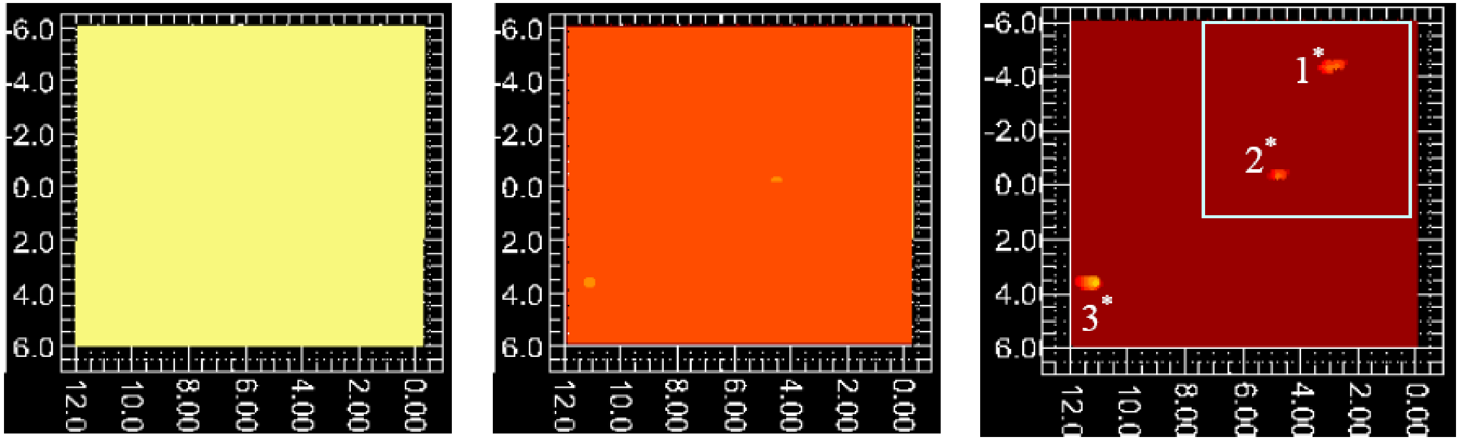
Sample names	Suface treatments	Surface damage layer removed
Polycrystalline Nb: PolyNb 1, 2, 3, 4	EP + HPR + DIC	150 ± 10 µm Nb
Polycrystalline Cu PolyCu 1, 2, 3, 4	MP + DIC	--
Crystalline Nb: SCNb1, 2 & LGNb1,2	BCP + HPR + DIC	30 µm
Crystalline Nb: SCNb1, 2,3 & LGNb3	BCP + HPR	100 µm

EP : Electropolishing
(HF: H_2SO_4 volume ratio 1:9)
BCP: Buffered cemical polishing
(HF: HNO_3: H_3PO_4 volume ratio 1:1:2)
MP: Mechanical polishing (diamond turned)

HPR: High Pressure water rinsing
DIC: Dry ice cleaning

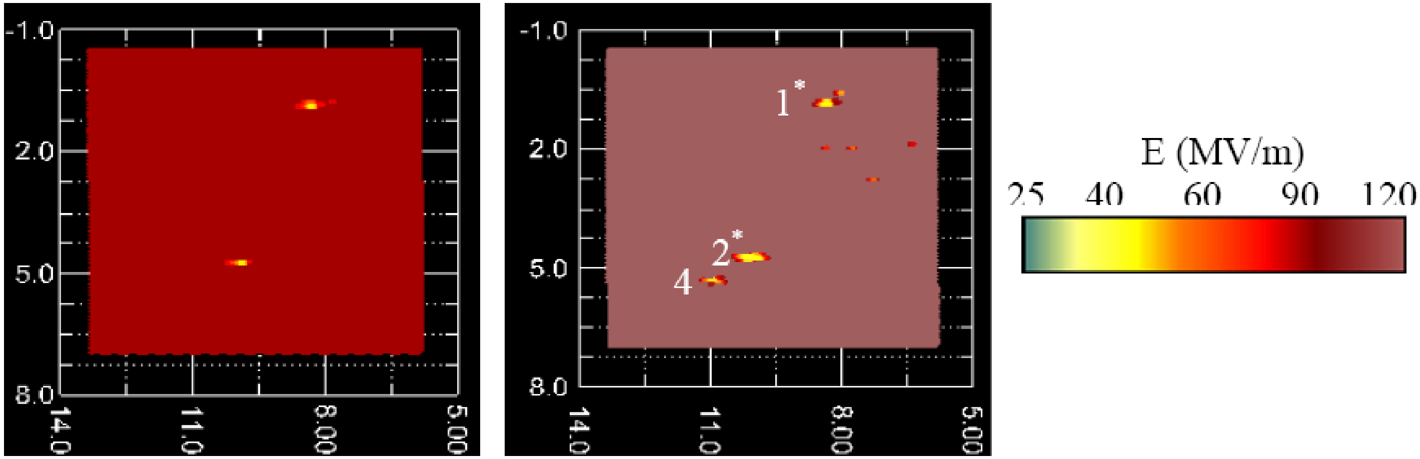
LGNb: Large grain Nb
SCNb: Single crystal Nb

Polycrystalline Nb: field emitters' distribution



(a) E = 40 MV/m, 0 emitter (b) E = 60 MV/m, 2 emitters (c) E = 90 MV/m, 3 emitters

1st series: $\varnothing_{\text{Anode}} = 300\ \mu m$, $d = 50\ \mu m\ (\pm 5\ \mu m)$, $A = (12 \times 12)\ mm^2$

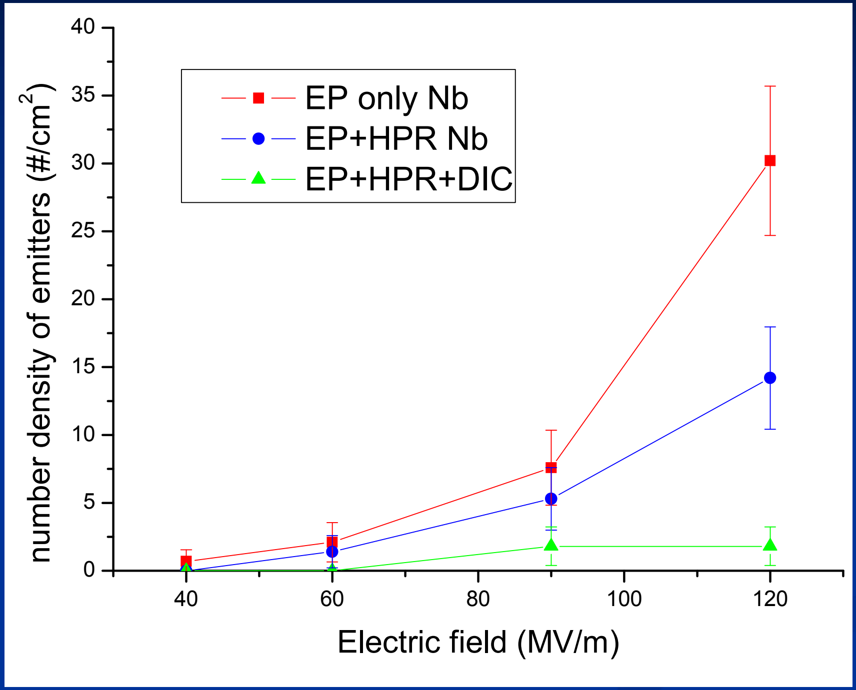


(d) E = 90 MV/m, 3 emitters (e) E = 120 MV/m, 8 emitters

2nd series: $\varnothing_{\text{Anode}} = 100\ \mu m$, $d = 40\ \mu m\ (\pm 5\ \mu m)$, $A = (7.5 \times 7.5)\ mm^2$

FE maps on EP Nb sample with volatege regulated for 1 nA current

Polycrystalline Nb: improvements by cleaning



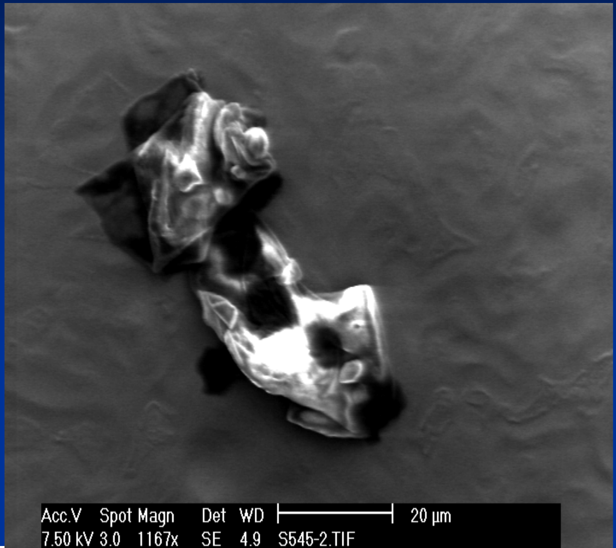
Surface treatments of Nb	EP	+ HPR	+ DIC
onset of FE @	40 MV/m	60 MV/m	90 MV/m
@120 MV/m, N (#/cm^2)	≈ 30	< 1/2 of EP Nb	< 1/7th of HPR Nb

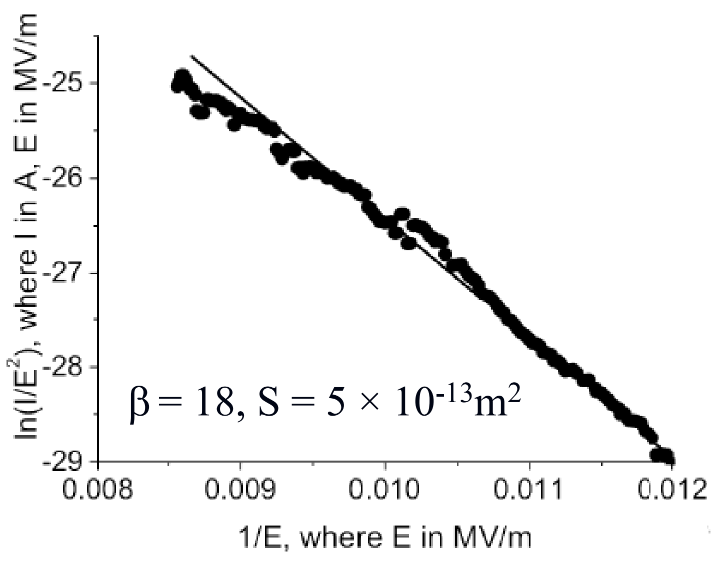
- Improvements after HPR
- Huge suppression of FE after DIC

[A. Dangwal et al , Physica C 441 (2006) 83-88]

Cleaning potential of DIC

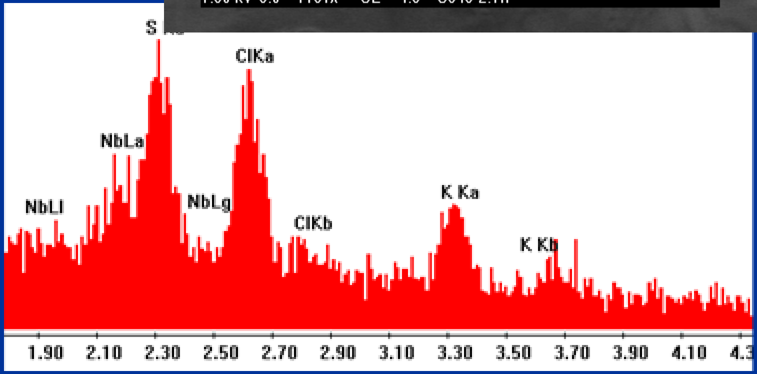
for HPR resistant field emitting particulates





$\beta = 18$, $S = 5 \times 10^{-13} m^2$

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



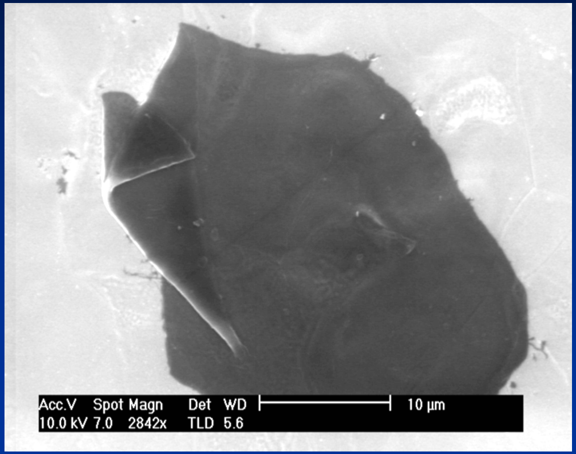
EDX spectrum: S, Cl, K contents

<u>‘S’ content:</u> most probably due to some remnant from chemical bath (EP)

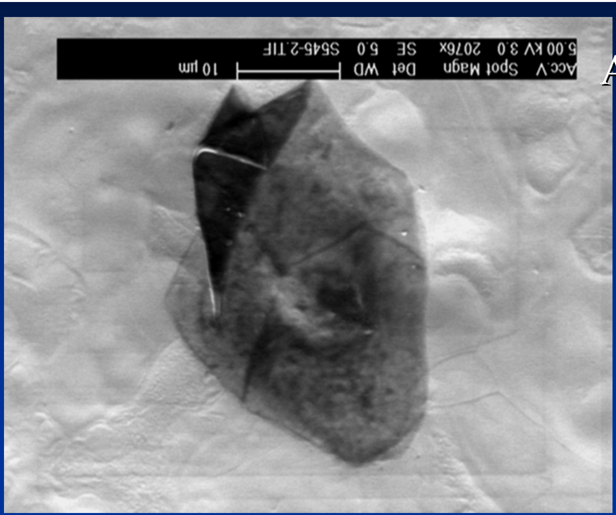
Emitter disappeared after DIC

Flake like microparticulate

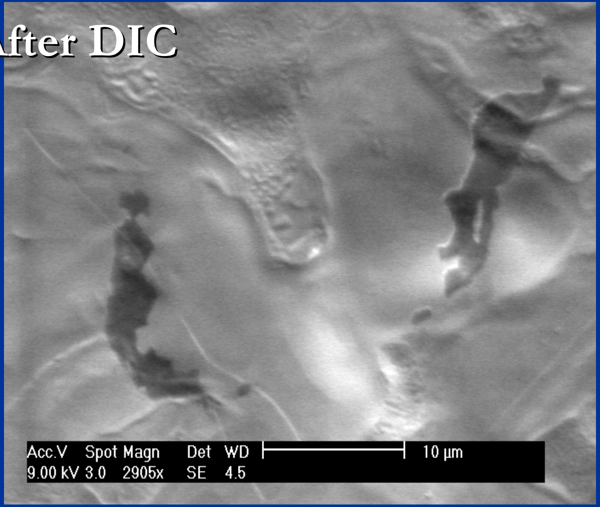
Before HPR



After HPR



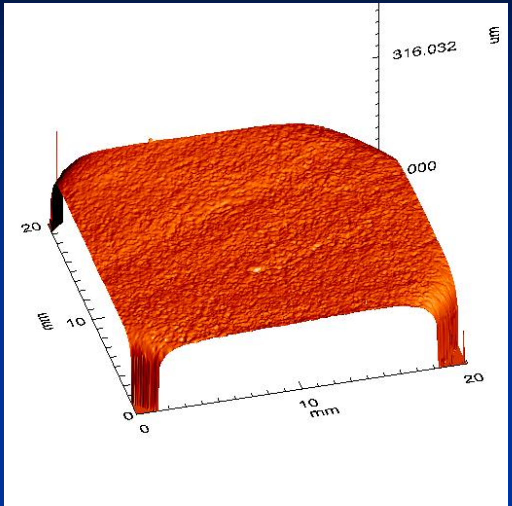
After DIC

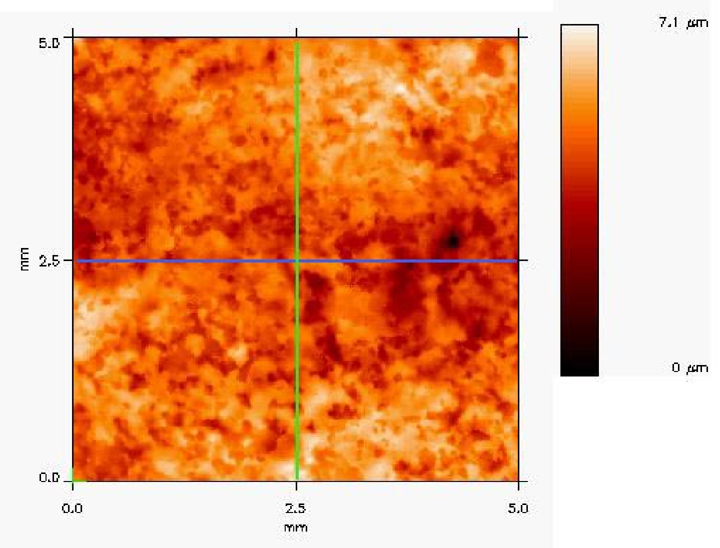


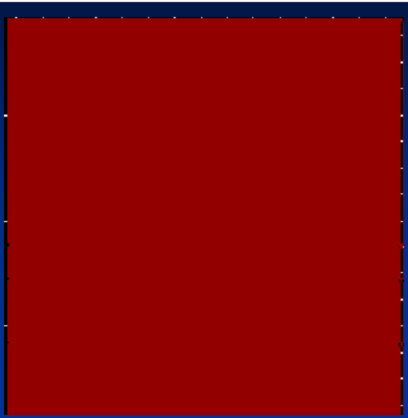
emitter	HPR	HPR+DIC
E_{on} (MV/m)	54.3	62.8
$\beta_\uparrow$	67.4	35.4
$\beta_\downarrow$	51.2	38.0
$S_\uparrow$ (m^2)	2×10^{-17}	8.3×10^{-13}
$S_\downarrow$ (m^2)	1.2×10^{-15}	2.4×10^{-13}

Emitter of ~ 20 µm size destroyed by DIC
Remnants emitting at higher E_{on}!

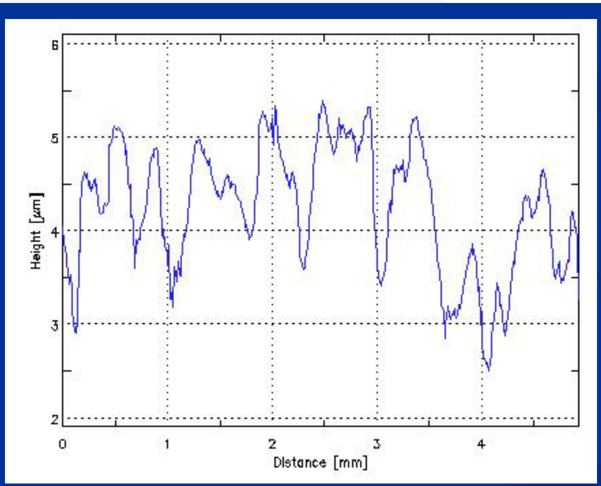
Nb sample EP+HPR inside a cavity at DESY

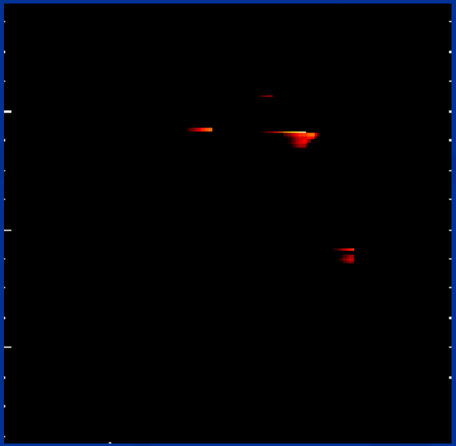






No FE @120 MV/m





5 emitters @ 150MV/m

Surface profile:
- o Grain boundaries visible
- o GB step height ~ 1-2 µm
- o Micro roughness: < 0.2 µm

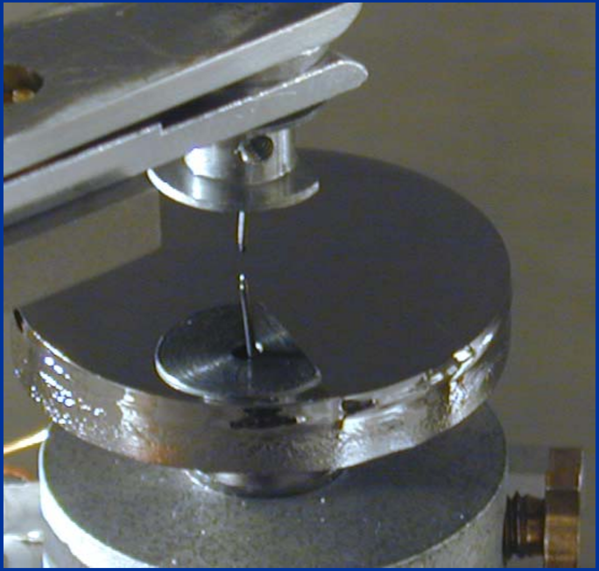
⇨Excellent Nb surface by EP

No field emission upto 120 MV/m: best measured EP'd sample

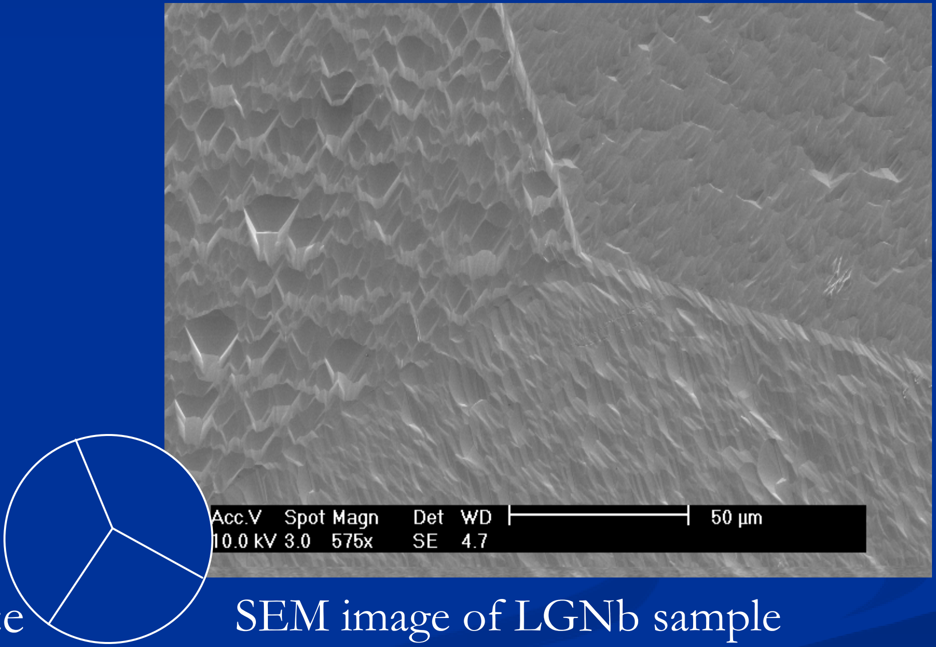
Crystalline Nb

Motivation to investigate crystalline Nb (single crystal/ large grain):

- First test with single cell cavity of large grain Nb yielded E_{acc} up to 45 MV/m
- Replacement of EP'd polycrystalline Nb with BCP'd large grain Nb ??
- To study 150°C heat treatment effects on single crystal and large grain Nb ??
- To obtain intrinsic FE properties of smooth Nb



Visual appearance: mirror like surface
⇒ Surface roughness ~ 6 nm

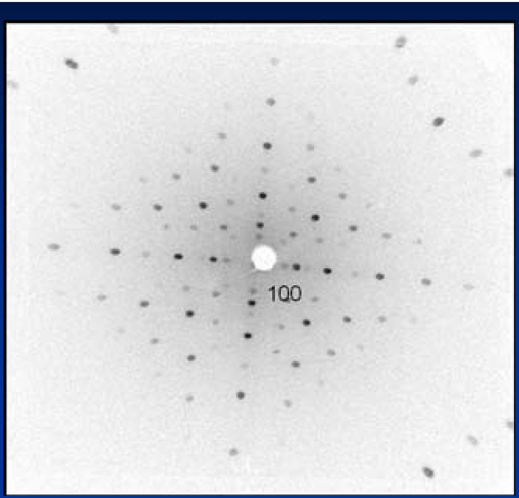


SEM image of LGNb sample containing three grains of ~ cm size

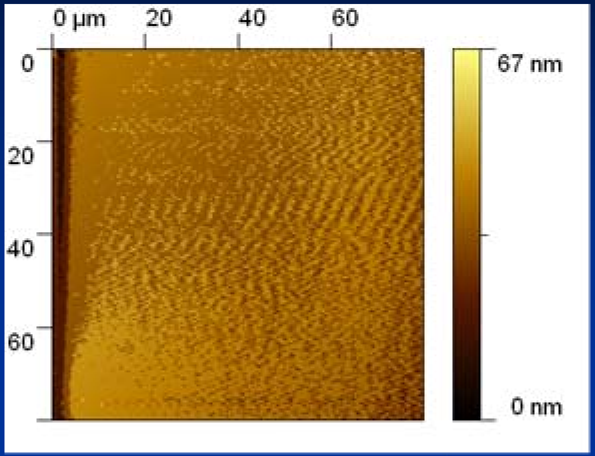
BCP'd crytsalline Nb: superior quality surfaces

Crystalline Nb: superior quality surfaces

HR optical microscopic image



XRD micrograph
(100) plane

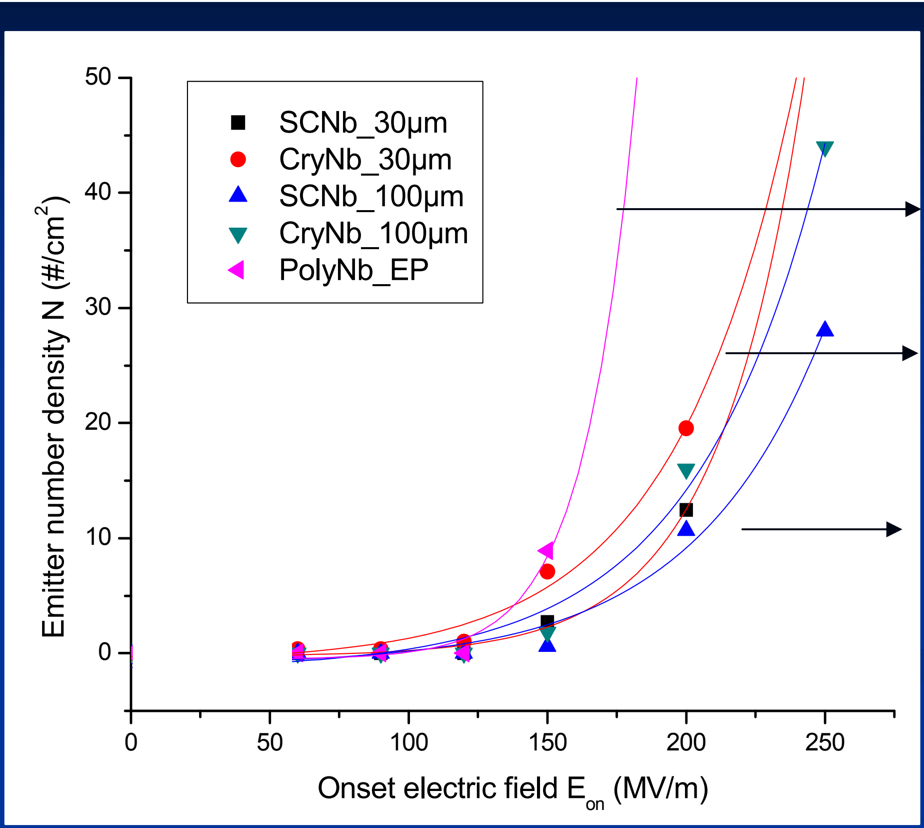


AFM scan over (80×80) μm^2
surface roughness = 7.5 nm

Sample	Removed damage layer using BCP	Orientation	Surface roughness
SCNb1	30µm	(110)	11.7 nm
SCNb2	30µm	(110)	17.6 nm
SCNb3	100 µm	(110)	7.0 nm
SCNb4	100 µm	(111)	6.2 nm
SCNb5	100 µm	(100)	7.5 nm
LGNb1	30µm	(110), (111), (110)	100, 110.5,62.7
LGNb2	30µm	nm	nm
LGNb3	100 µm	(100), (110), (111)	8.8, 6.9, 6.8

FE onset @ 150 MV/m (SCNb), @ 120 MV/m (LGNb)

FE properties wrt material removed



Roughness:

grain boundary height ~1.5 µm

~ 60- 110 nm
~ 12- 18 nm

~ 6 nm
~ 8 nm

FE onset for BCP+HPR Nb:
SCNb: @ 150 MV/m
LGNb: @ 120 MV/m

For more material removal:

- Tendency of N(E) exp fit lines shift to the right, i.e. higher E_{onset}
- Evidence for reduced slopes, i.e. less N at a given field E

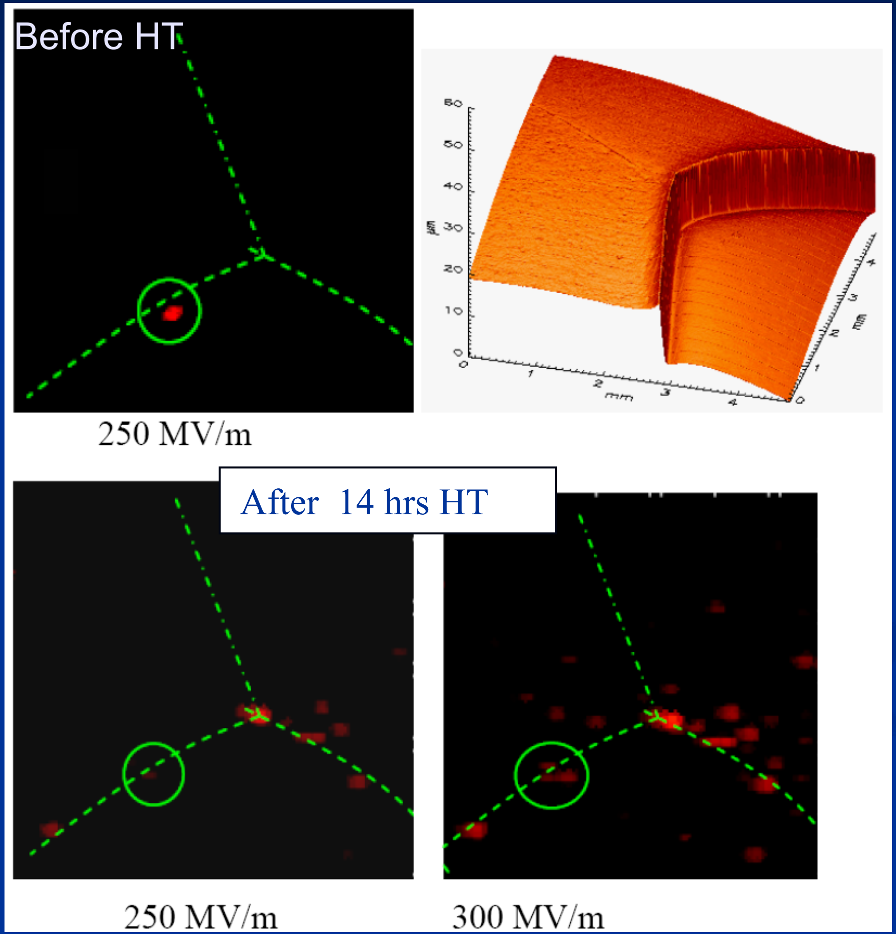
Hint for higher emitter # for higher surface roughness

BCP‘d crystalline Nb samples better than EP‘d polyNb

[A. Dangwal et al, communicated with PRST-AB]

In-situ heat treatments (HT) @ 150 °C

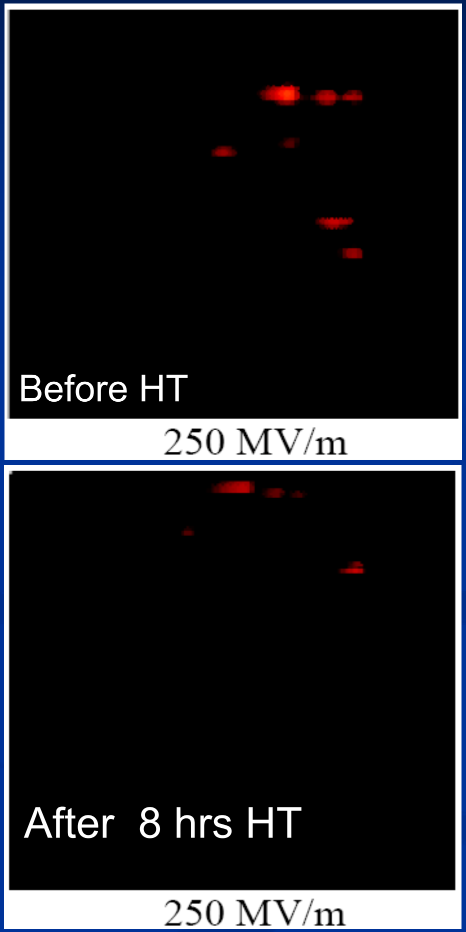
Large grain Nb



250 MV/m

250 MV/m 300 MV/m

Single crystal Nb



250 MV/m

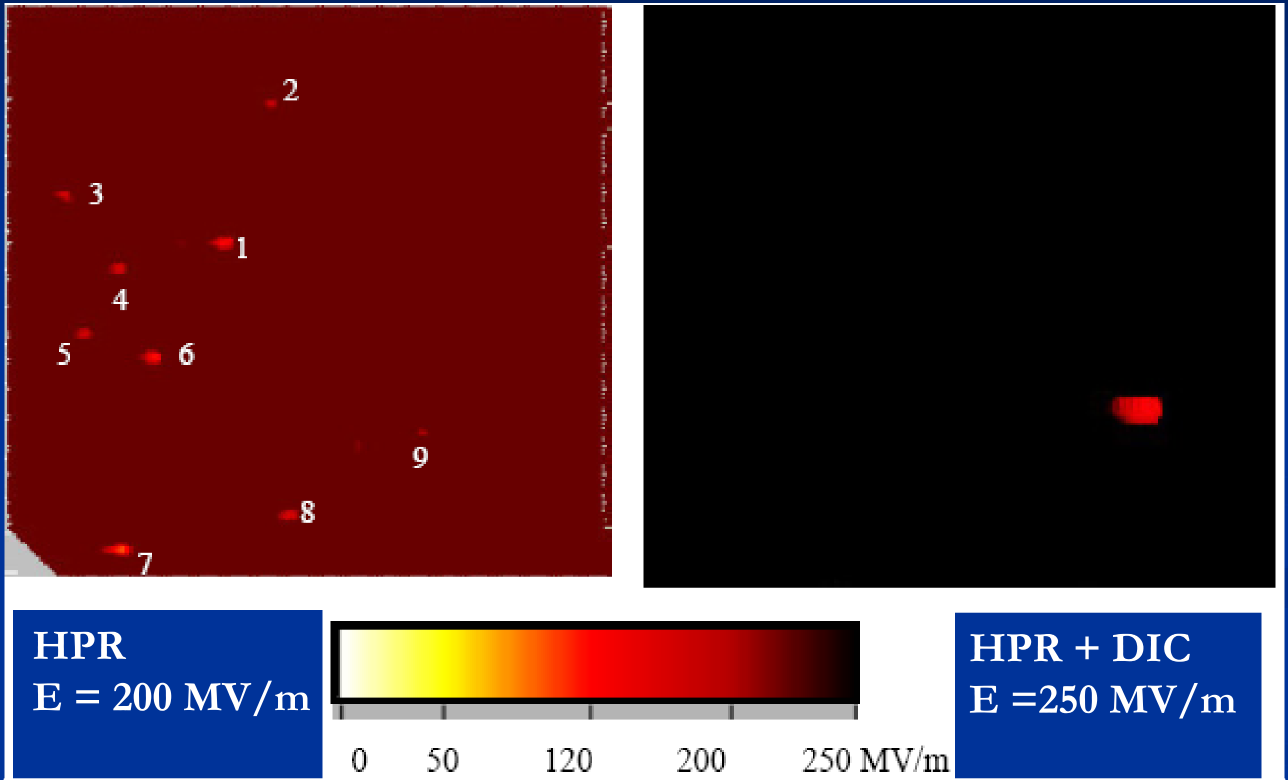
250 MV/m

After HT: more emitters appear on LGNb near grain boundaries @ E> 250 MV/m; no change for SCNb, no features observed in SEM

Ist evidence for impurity segregation in grain boundaries

Cleaning potential of DIC

for HPR resistant field emitters

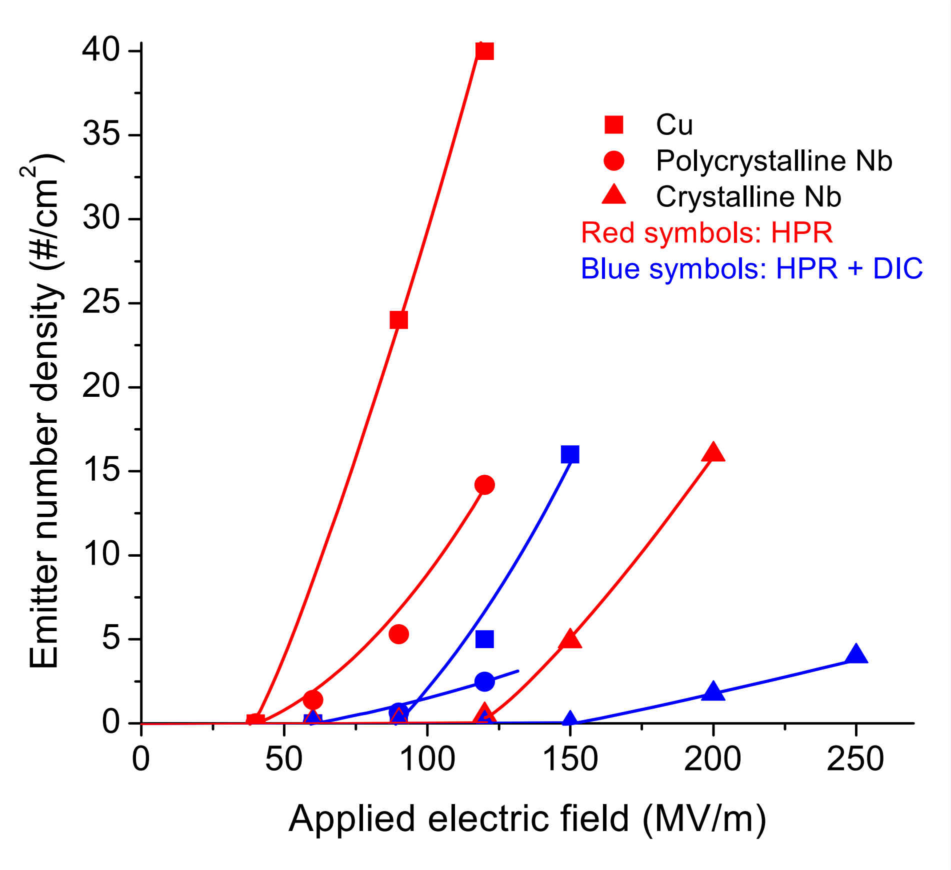


E-map for 2 nA current on a SCNb sample before and after DIC over (5×5) mm^2 area, $Ø_{anode}$= 100 µm

Appreciable improvement by DIC
on high quality single crystal Nb surface

Cleaning potential of DIC

Statistical overview: N(E) curves for different materials



<u>Best case:</u>
No FE @ 250 MV/m
(sample SCNB4)

⇒ (i) Onset of FE shift to higher E; (ii) Huge reduction of N @ particular E

DIC suppresses EFE effectively on Cu and Nb surfaces

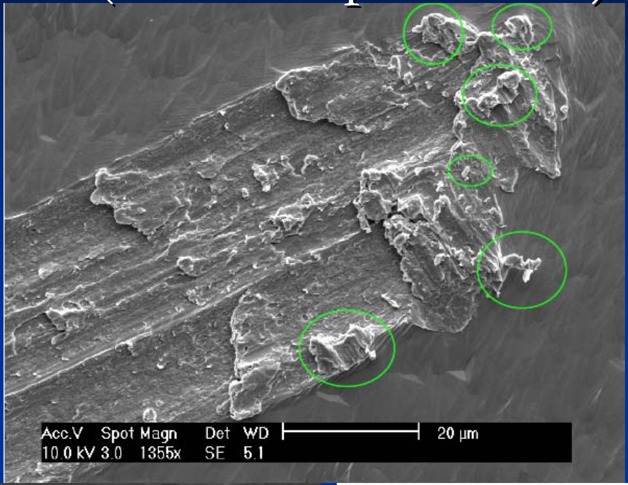
[A. Dangwal et al., J. Appl. Phys. 102, 044903 (2007)

Impact of DIC on surface protrusions

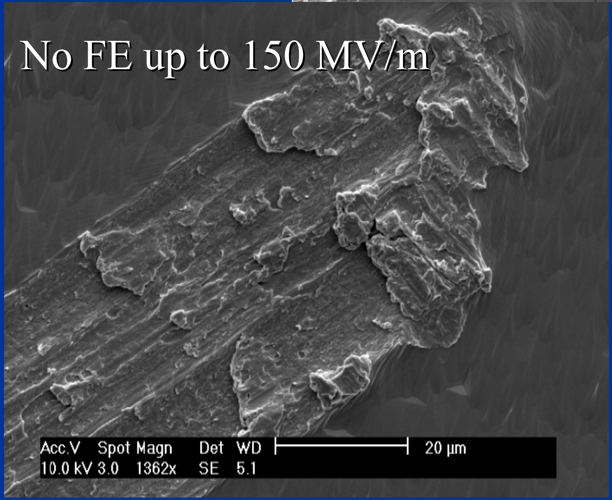
(Microscopic results)

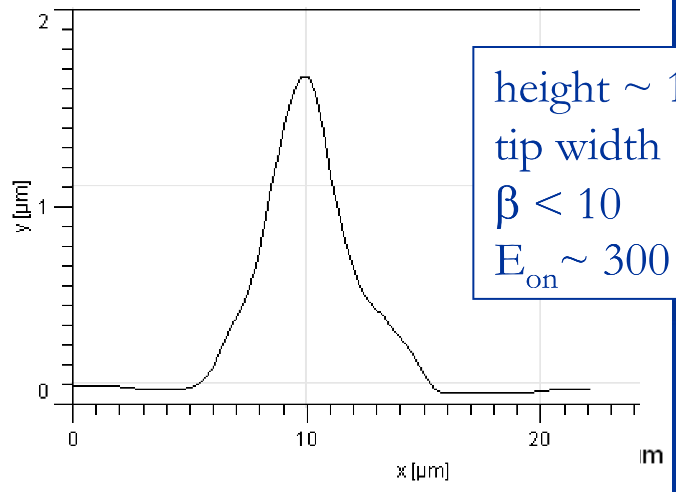
Before DIC

E_{onset}= 60 MV/m



After DIC


No FE up to 150 MV/m

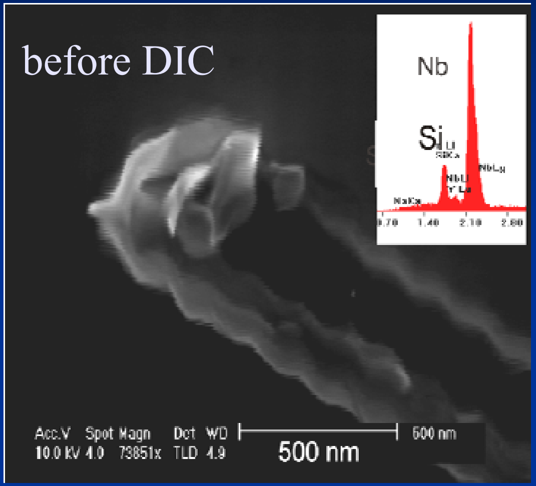


height ~ 1.6 µm
tip width ~ 0.5 µm
$\beta < 10$
E_{on}~ 300 MV/m

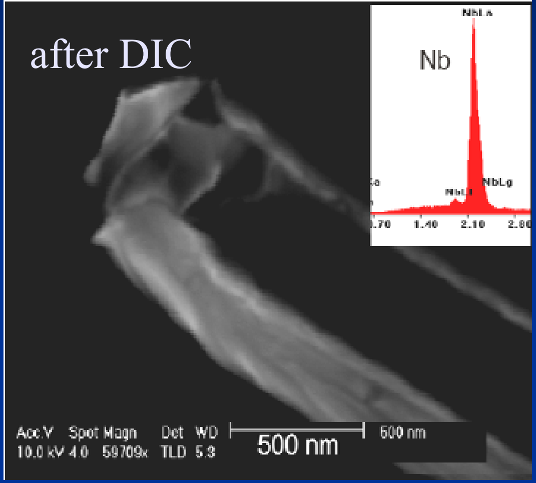
Removal of delaminations + partial smoothing of surface protrusions by DIC

Impact of DIC on surface protrusions

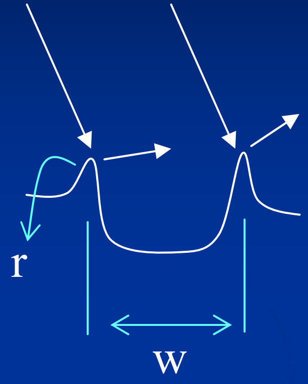
(Microscopic results)



Onset of FE @ 150 MV/m



No FE up to 250 MV/m



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

$S \sim r^2$

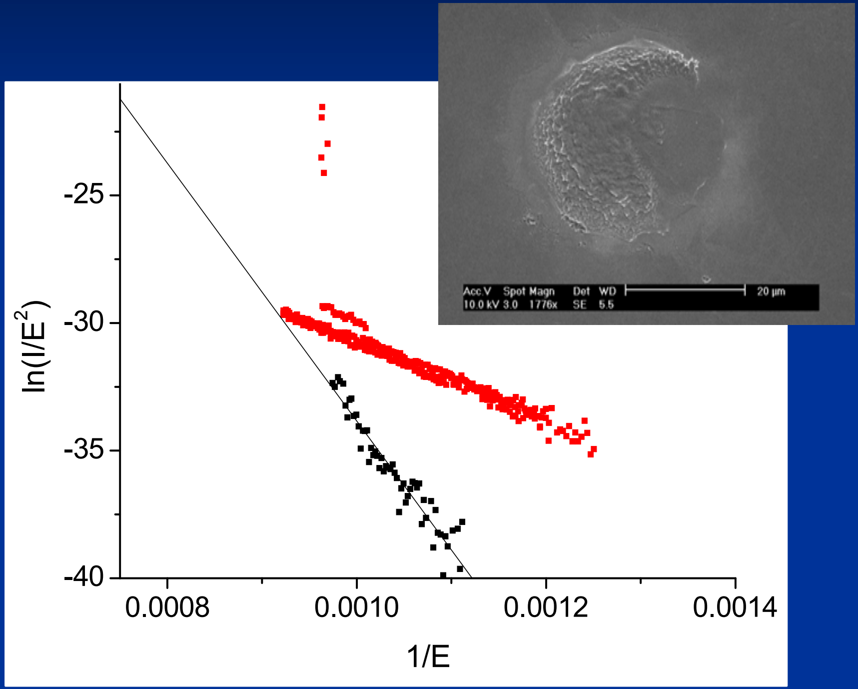
dry ice particles → impinging forcefully on the surface

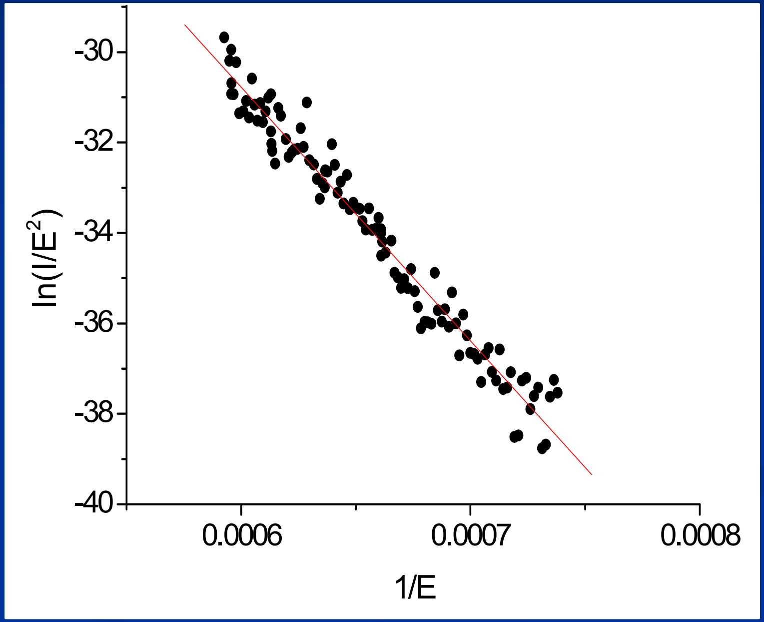
$\Rightarrow$ smoothen the sharp edges of scratches

- partial smoothing of surface irregularities < 100 nm
- removal of contaminants within irregularities

Intrinsic FE measurements: single crystal Nb

measured with W-tip anodes of $\varnothing_a$ = 5-20 µm d > 2 µm





Initially intrinsic field emission of Nb (111)

slope β = 1 ⇒ Φ = 3.8 eV

creation of an emitter at ~ 1 GV/m by a microdischarge ⇒ crater in Nb

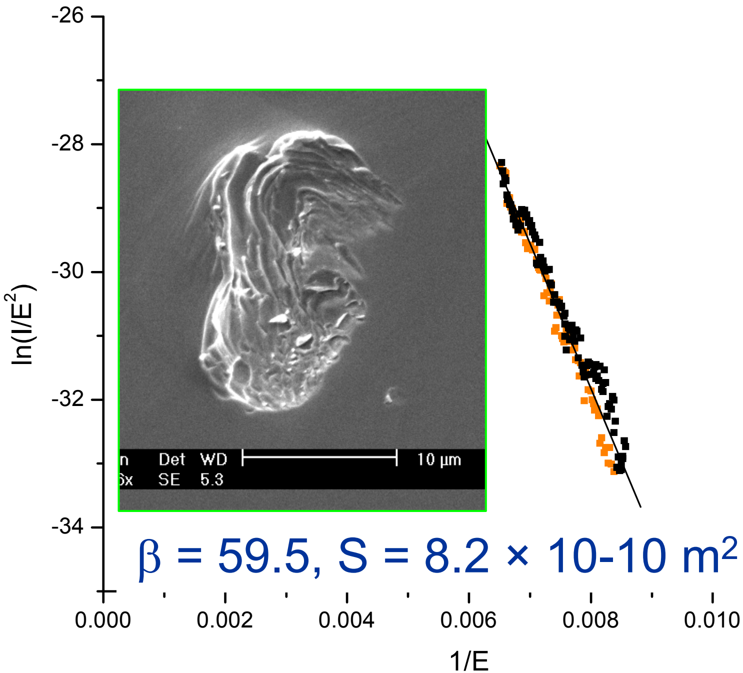
Intrinsic field emission of Nb(100)

slope β = 1 ⇒ Φ = 4.05 eV

SCNb samples reveal anisotropy of work function Φ

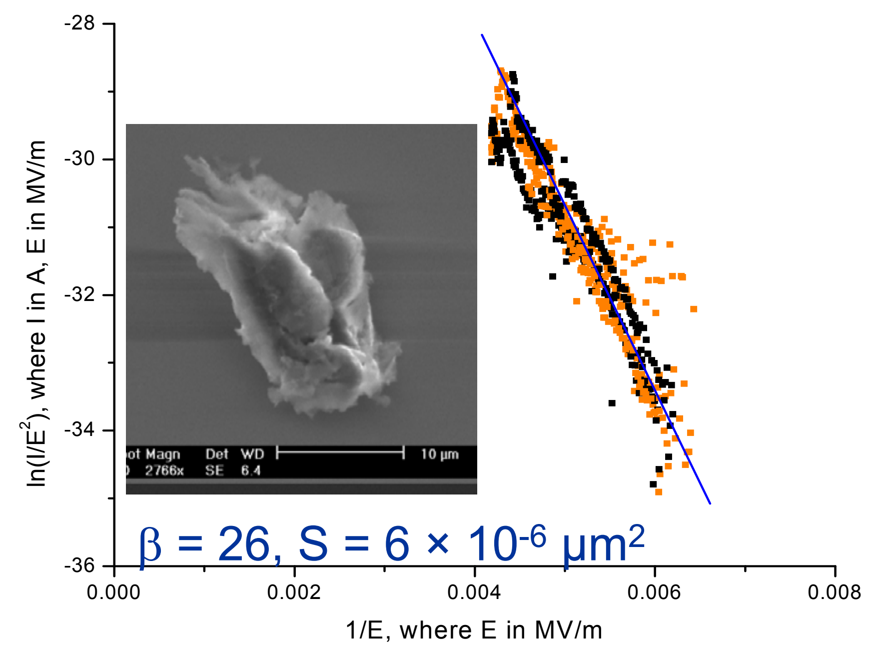
[A. Dangwal et al, communicated with PRST-AB]

Single emitter investigations



β = 59.5, S = 8.2 × 10-10 m²

Surface defect of ~ 12 µm mean size



β = 26, S = 6 × 10^{-6} µm²

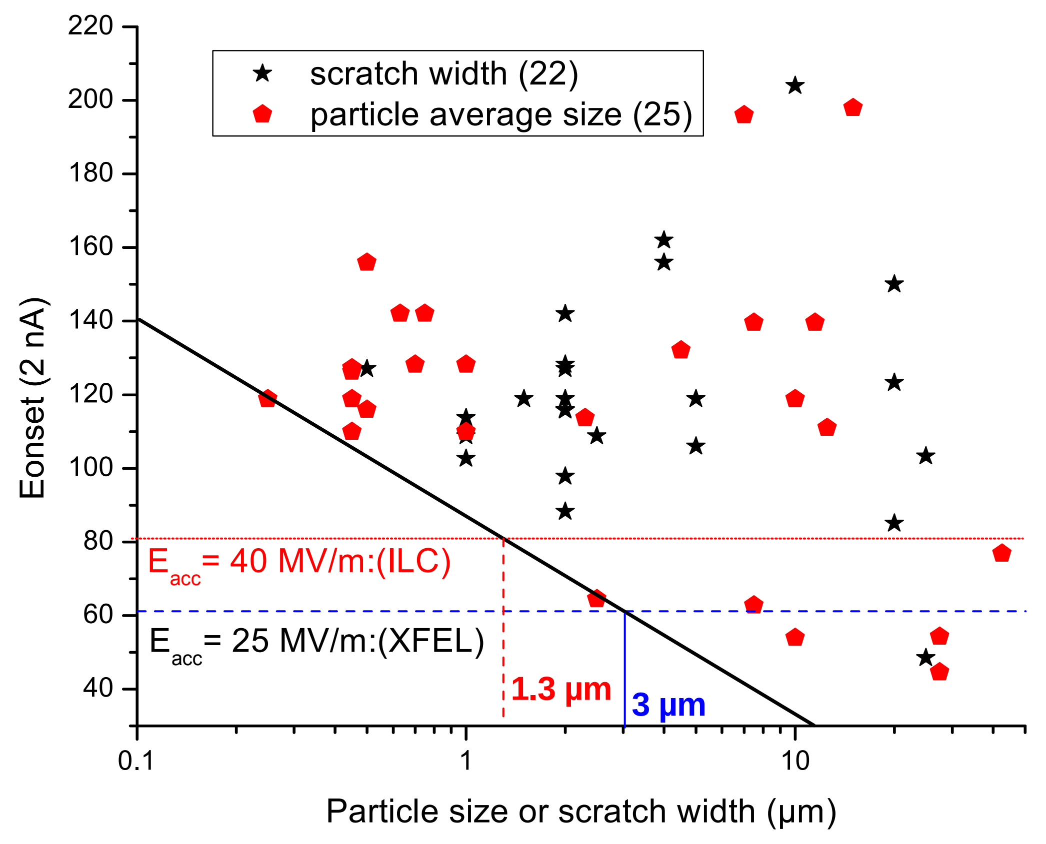
Al particulate of ~ 14 µm mean size

Field emitters identified in SEM as:

Surface irregularities 67%, generally showed **stable FN like emission**

Particulates 33 %, generally observed **unstable FN like emission**.

Correlation between E_{onset} and emitter size ?



Evidence for correlation ⇒ fast FE quality control by defect size

[A. Dangwal et al, Proceeding of 13th International Workshop on RF Superconductivity 2007]

Conclusions

- DIC effectively removes FE particulates and weakens protrusions
  ⇒ in situ repair cleaning of FE cavities
- Large grain/single crystal Nb samples treated with BCP-HPR show better FE results than EP-HPR polycrystalline Nb samples
  ⇒ reliable alternative for SRF cavities with less FE !
- Evidence for impurity segregation to grain boundaries in large grain Nb after in-situ heat treatment at 150°C
- Intrinsic FE on SCNb with $\beta = 1$ and $\Phi = 4$ eV partially obtained
  ⇒ surface roughness enhances FE of particulates !
- Evidence for a correlation between onset field and emitter size
  ⇒ fast FE quality control on samples for XFEL !

Acknowledgements

University of Wuppertal

Prof. Dr. G. Müller
C. S. Pandey,
J. Pouryamout

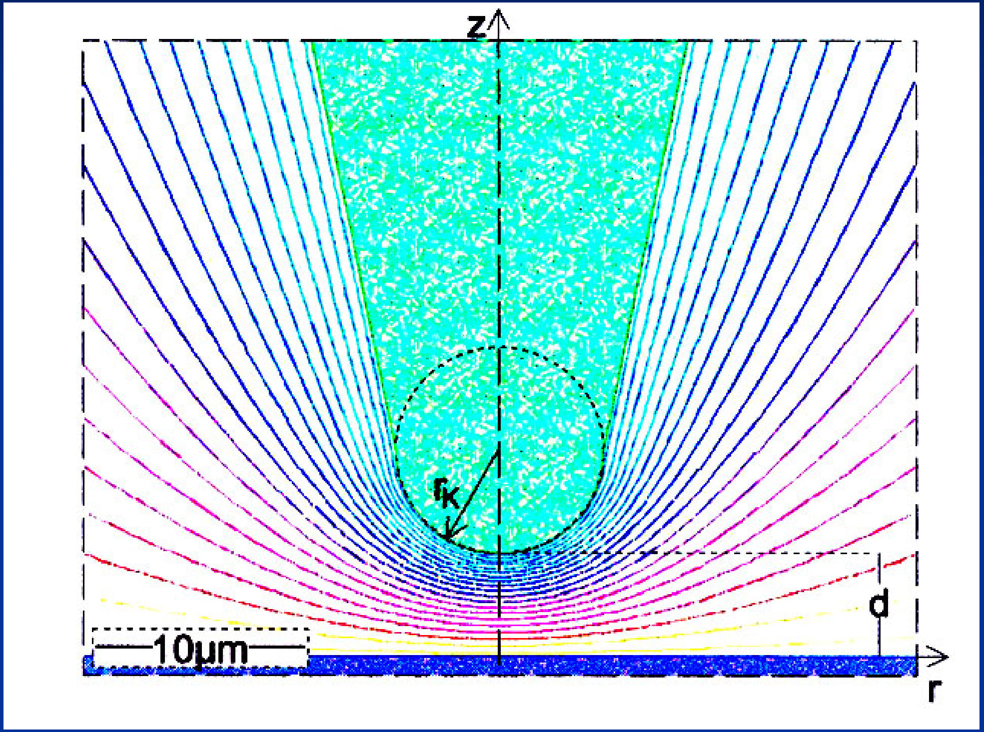
DESY

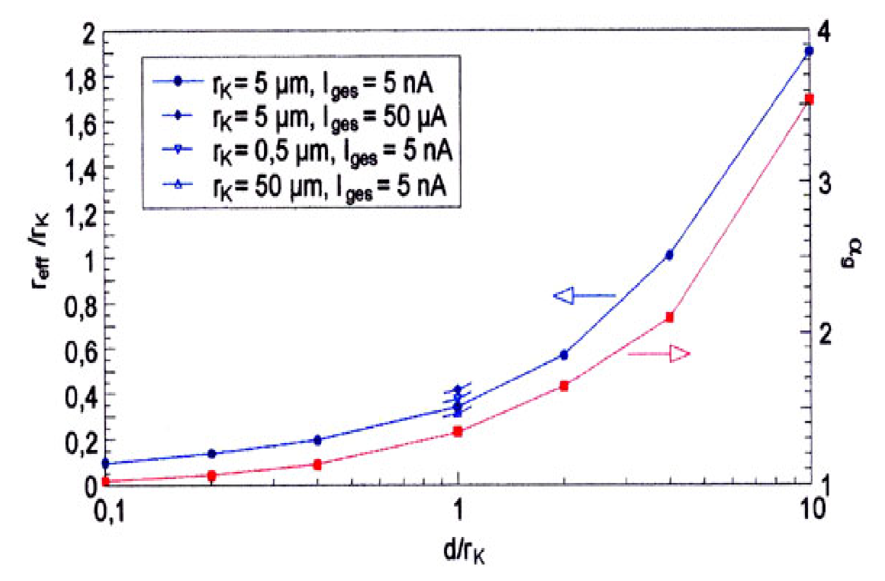
Prof. Dr. D. Proch,
Dr. K. Floettmann,
A. Matheisen,
Dr. D. Reschke,
Dr. X. Singer,
Dr. W. Singer

Funded by DESY and European FP6 program CARE

Thank you for your kind attention.

Correction factor for the measurements with W tip as anode





Electric field below the tip is reduced.

The electric field should be modified with correction factor α

$$E_{max} = U/(\alpha d)$$

$$I = S.J = S.\frac{e^3}{8\pi h}\cdot\frac{E^2}{t^2(y)\cdot\Phi}\exp\left(-\frac{8\pi\sqrt{2m_e}}{3he}\cdot\frac{v(y)\cdot\Phi^{3/2}}{E}\right)$$

where $y = \sqrt{(e^3E/4\pi\varepsilon_0\Phi^2}$; $t^2(y) \approx 1.1$, $v(y) \approx 0.95 - y^2$

t(y) and v(y) : tabulated functions for Image charge effect

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

where, I in A, S in cm^2, E in MV/m, Φ in eV, then A= 154, B= 6830

Example: Nb surface: Φ= 4 eV, S = 10^{-14}m^2

to get tunneling current of ≈ 1 µA, required field ≈ 3GV/m

But… tunneling current is observed experimentally at ≈ 40 MV/m

⇒ **Enhnaced field emission (EFE)**