

Surface Roughness Investigations on Nb Samples using Optical Profilometry

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- Motivation and strategy
- Surface roughness measurement techniques
- Results on polycrystalline Nb samples from DESY
- Results on single crystalline Nb samples from TJNAF
- Conclusion and outlook

Nb samples from: A. Matheisen, D. Reschke, X. Singer, J. Ziegler (DESY)
P. Kneisel (TJNAF)

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Motivation and strategy

- Enhanced field emission (EFE) is caused by **particulates** or **scratches** [1]
- **Quenches and high-field Q-drop** might depend on **surface roughness** [2]
- Number density of **particulates** can be much reduced by HPR [3], DIC [4] and clean room assembly, but influence of **surface irregularities** on EFE and quenches of poly/single crystalline EP/BCP Nb has been **less studied**

[1] A. Dangwal et al., Phys. Rev. ST Accel. Beams 12, 023501 (2009).

[2] J.Knobloch et al., Proc. 9th Workshop on SRF (1999), p.77.

[3] P. Kneisel et al., Proc. 7th Workshop on SRF (1995), p.311.

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

1-st step

Systematic measurements of **average surface roughness** and **local defect geometry** for typical Nb samples by means of optical profilometry and AFM

2-nd step

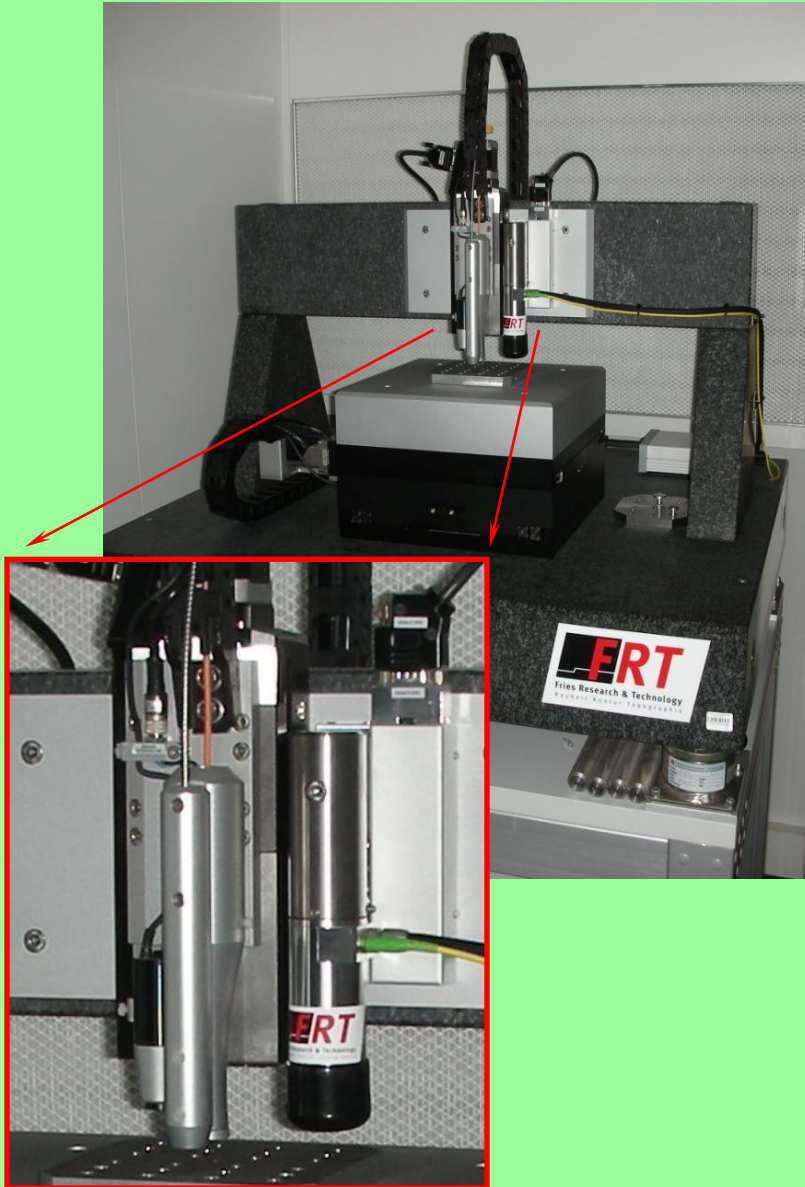
Localization and characterization of effective field emitters (E_{on} (1 nA), β , S) on the **same Nb samples** after HPR with FESM and in-situ SEM imaging

3-rd step

Ex-situ HRSEM/EDX identification of emitting defects and investigation of the **correlation between EFE parameters and geometry of defects**



Surface roughness measurement techniques



- **Optical profilometer (OP)**
 - white light irradiation and spectral reflection
 - fast scanning speed (100×100 pixel per min)
 - samples up to 20×20 cm² and 5 cm height
 - 2 μm lateral and 3 nm height resolution
- **atomic force microscope (AFM)**
 - operated in contact or non-contact mode
 - 2 μm positioning accuracy within OP scan
 - 34×34 μm² scanning range
 - 3 nm lateral and 1 nm height resolution
- **CCD camera** for positioning control
- **granite plate** with an active damping system for undisturbed measurement at nm scale
- **clean laminar air flow** from the back to reduce particulate contamination



Average surface roughness and electric field enhancement

Definition of **average surface roughness**

$$R_a = \frac{1}{n \cdot m} \sum_{i=1}^n \sum_{j=1}^m |z(x_i, y_j) - \bar{z}|$$

$$R_q = \sqrt{\frac{1}{n \cdot m} \sum_{i=1}^n \sum_{j=1}^m (z(x_i, y_j) - \bar{z})^2}$$

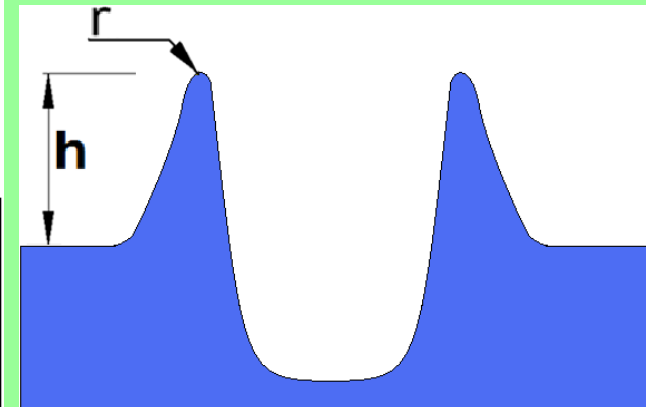
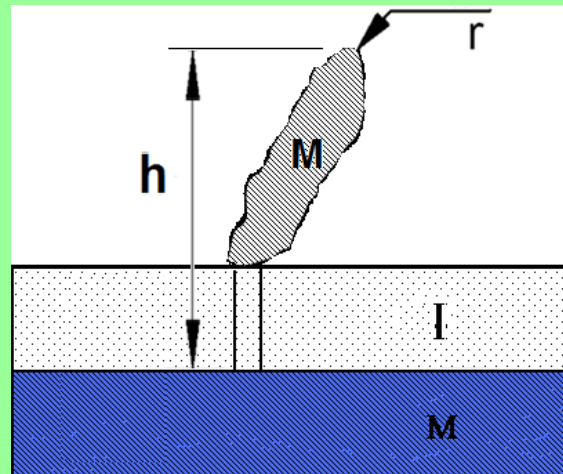
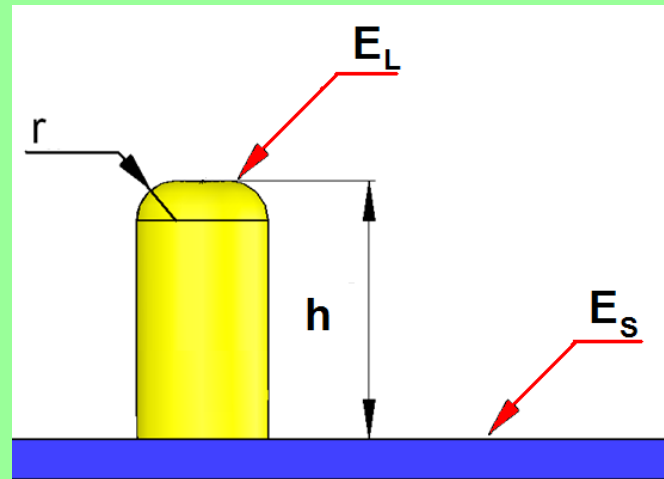
$z(x_i, y_j)$ = actual height value of profile
 n, m = pixel number in x and y direction
 \bar{z} = average height value

Estimated **electric field enhancement** for **protrusions**, **activated particulates** (initially MIM) and **scratches**:

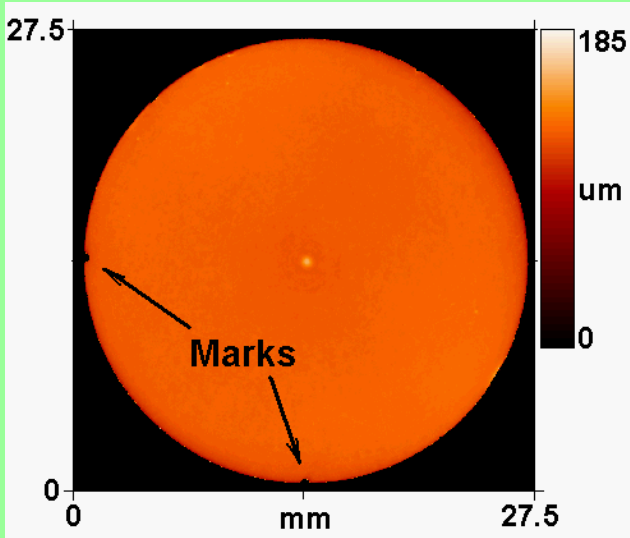
$$\beta_E = \frac{E_L}{E_S} \approx \frac{h}{r}$$

→ emission area $S \approx 2\pi r^2$

E_L = local electric field on defect
 E_S = electric field on flat surface
 h = height of defect
 r = curvature radius



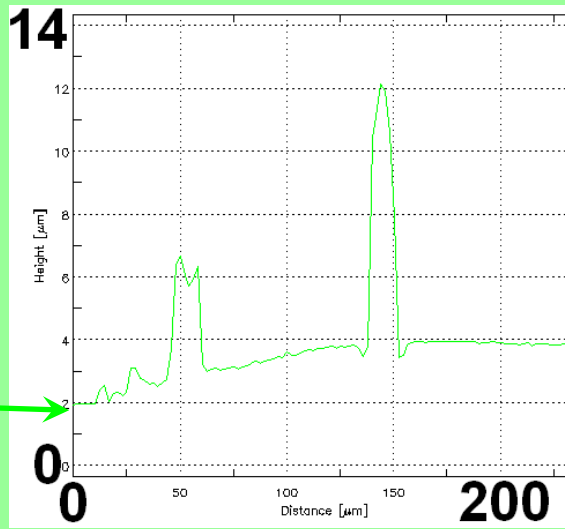
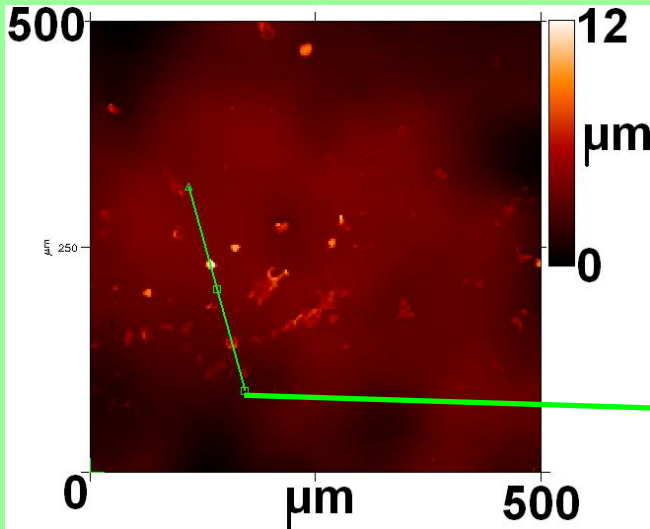
Results on polycrystalline Nb samples from DESY



Nb samples were assembled into the coupler port and BCP/EP/HPR processed with 9-cell cavities
 4 types of surface irregularities found with OP:

- particulates
- scratches
- grain boundaries
- round hills and holes

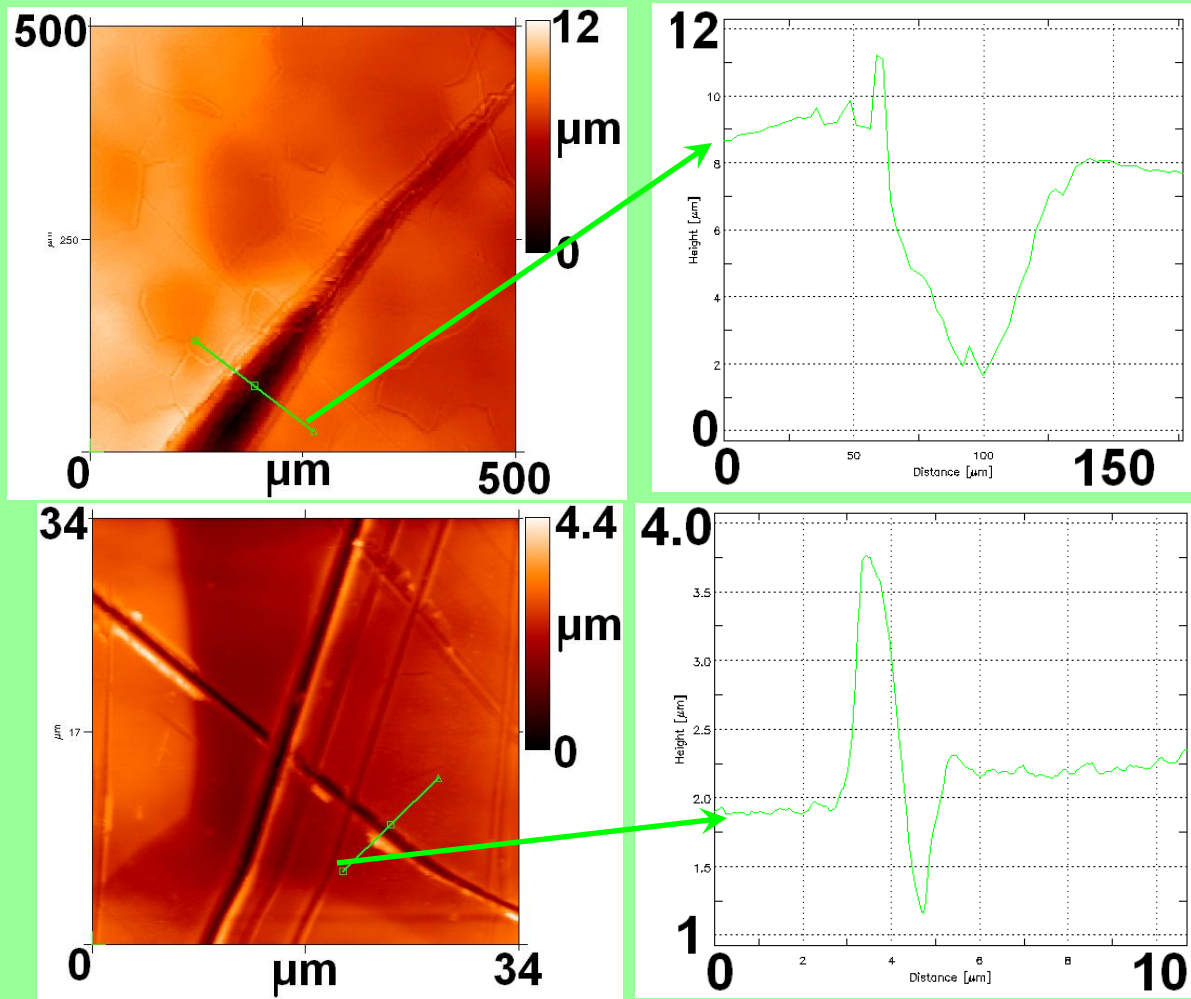
Particulates



< 5 μm	43 %
5 - 15 μm	48.4 %
15 - 25 μm	6.1 %
> 25 μm	2.5 %
$R_a = 0.276 \mu\text{m}$ $R_q = 0.548 \mu\text{m}$ $\beta_{E,max} = 15$	

OP results on polycrystalline Nb samples from DESY

Scratches



4 - 100 μm width
11 μm - 2.7 mm
length (on average
326 μm)
ridge height < 10 μm

$\beta_{E,\text{max}}$ = difficult

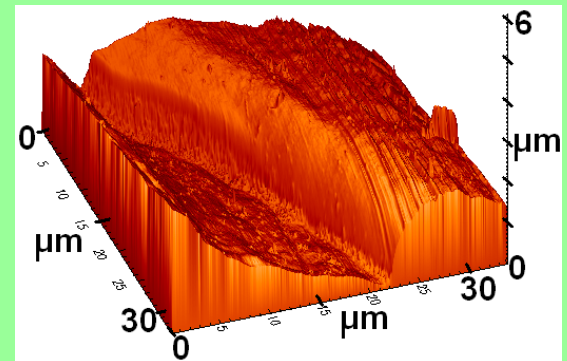
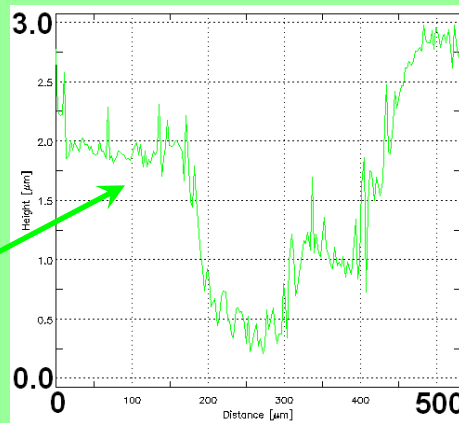
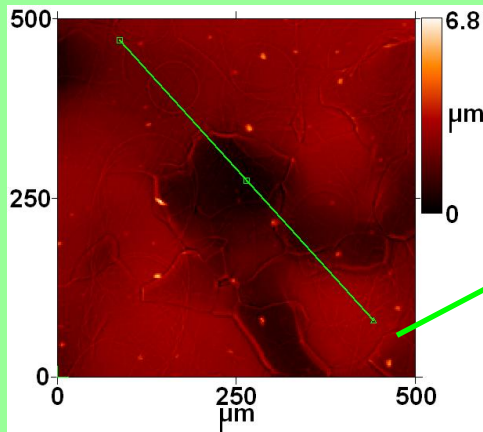
$R_a = 0.466 \mu\text{m}$
 $R_q = 0.646 \mu\text{m}$

$\beta_{E,\text{max}} = 13$



OP results on polycrystalline Nb samples from DESY

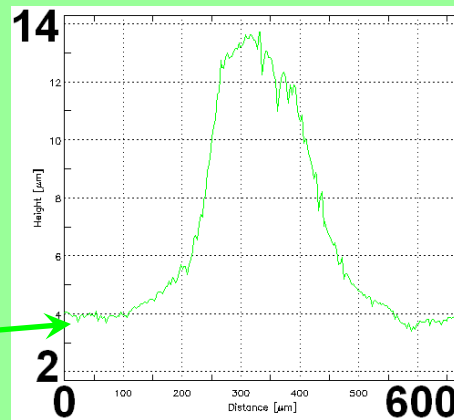
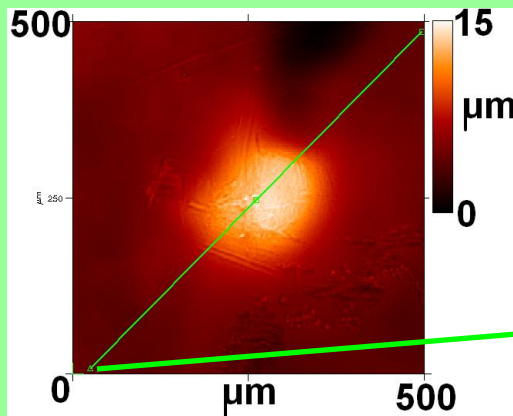
Grain boundaries



step height < 1.55 μm
edge radius < 0.78 μm

$$\beta_{E,max} = 4$$

Round hills and holes



height < 17 μm
size 10 - 440 μm

$$R_a = 0.295 \mu\text{m}$$

$$R_q = 0.489 \mu\text{m}$$

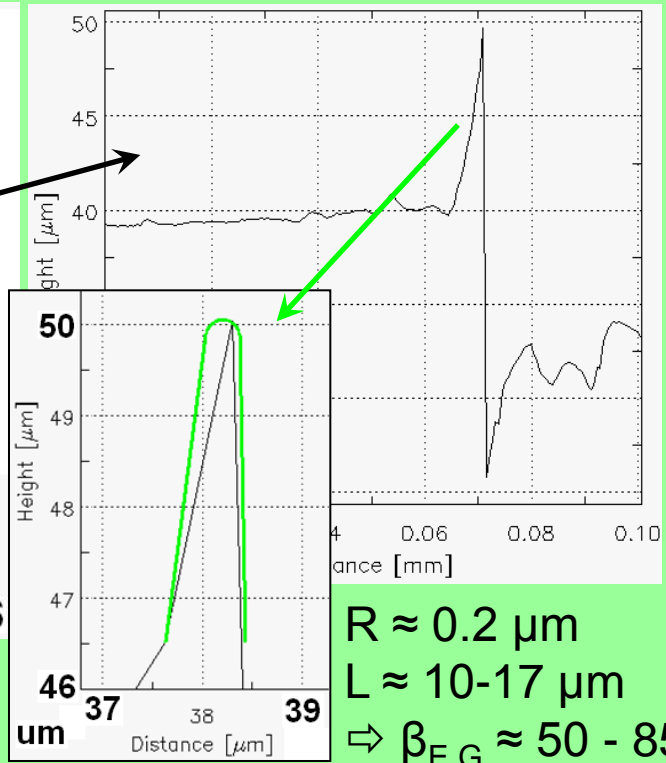
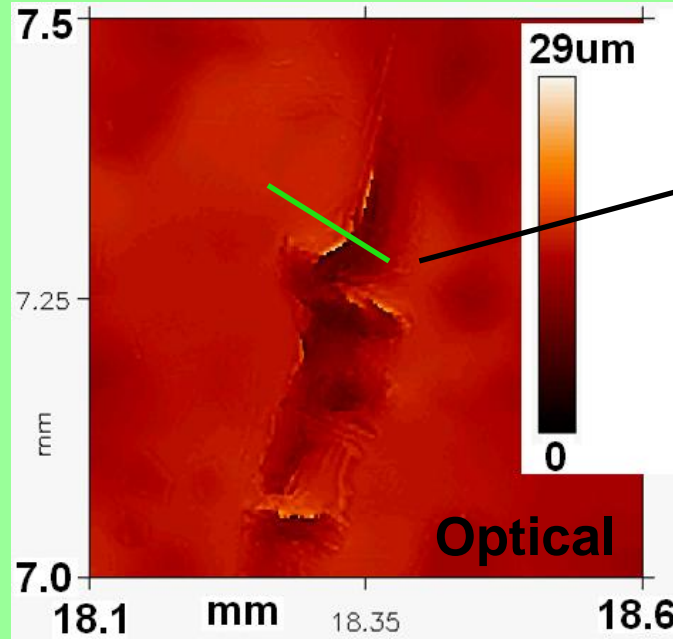
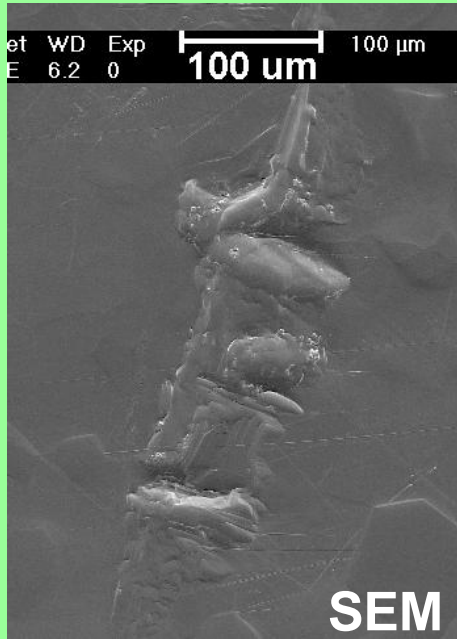
$$\beta_{E,max} < 4$$

Probably caused by foreign material inclusions and modified chemical reactions during EP

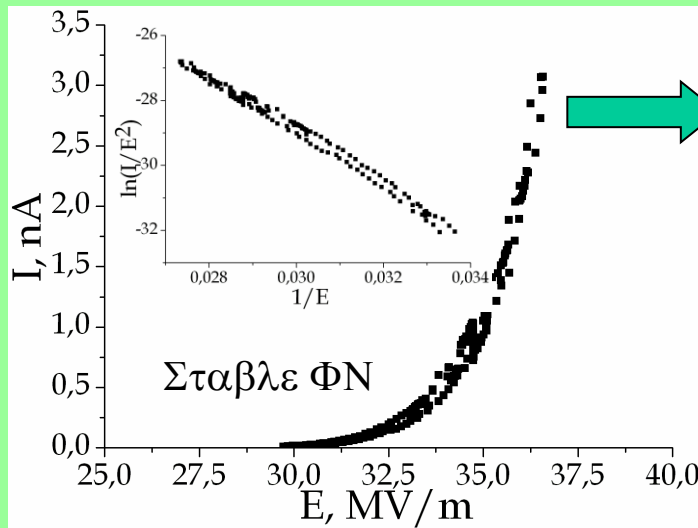
only **weak EFE** expected
but probably high magnetic field enhancement β_M ?



EFE/SEM/OP results on polycrystalline HPR-Nb samples



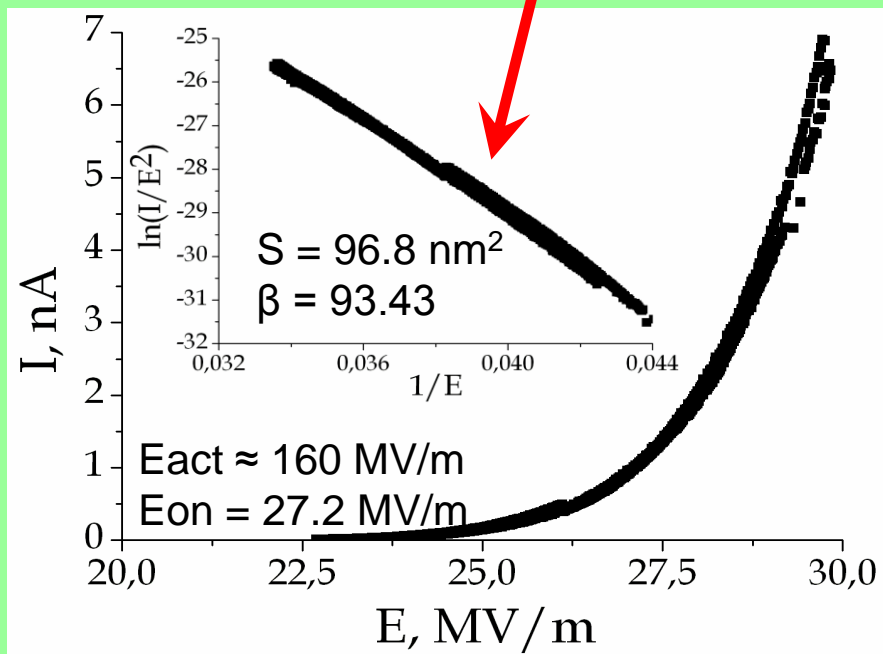
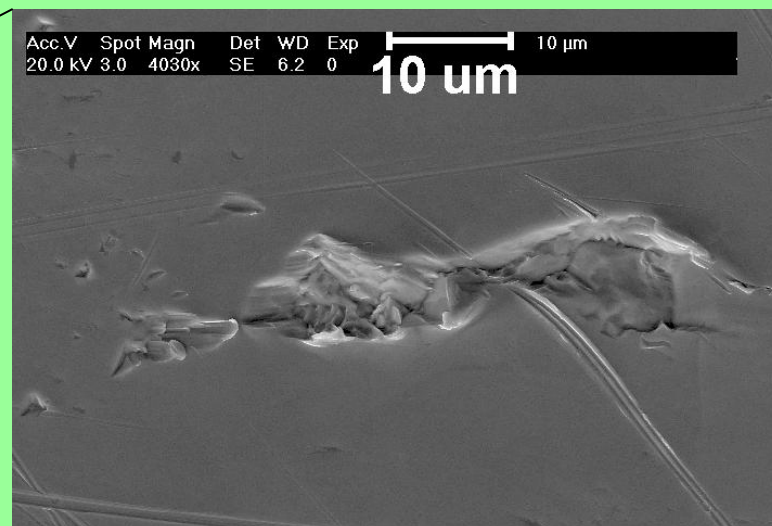
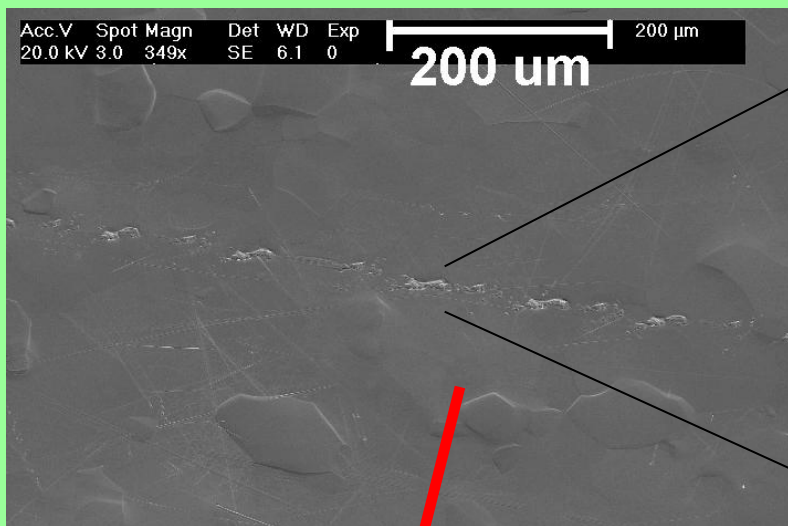
$R \approx 0.2 \mu\text{m}$
 $L \approx 10-17 \mu\text{m}$
 $\Rightarrow \beta_{E,G} \approx 50 - 85$



$E_{\text{act}} \approx 160 \text{ MV/m}$
 $E_{\text{on}} = 35 \text{ MV/m}$
 $S \approx 70 \text{ nm}^2$
 $\beta_{E,\text{FN}} \approx 60$

Large scratches \Rightarrow good correlation
 $\beta_{E,\text{FN}} \approx \beta_{E,G}$

EFE/SEM results on polycrystalline Nb samples

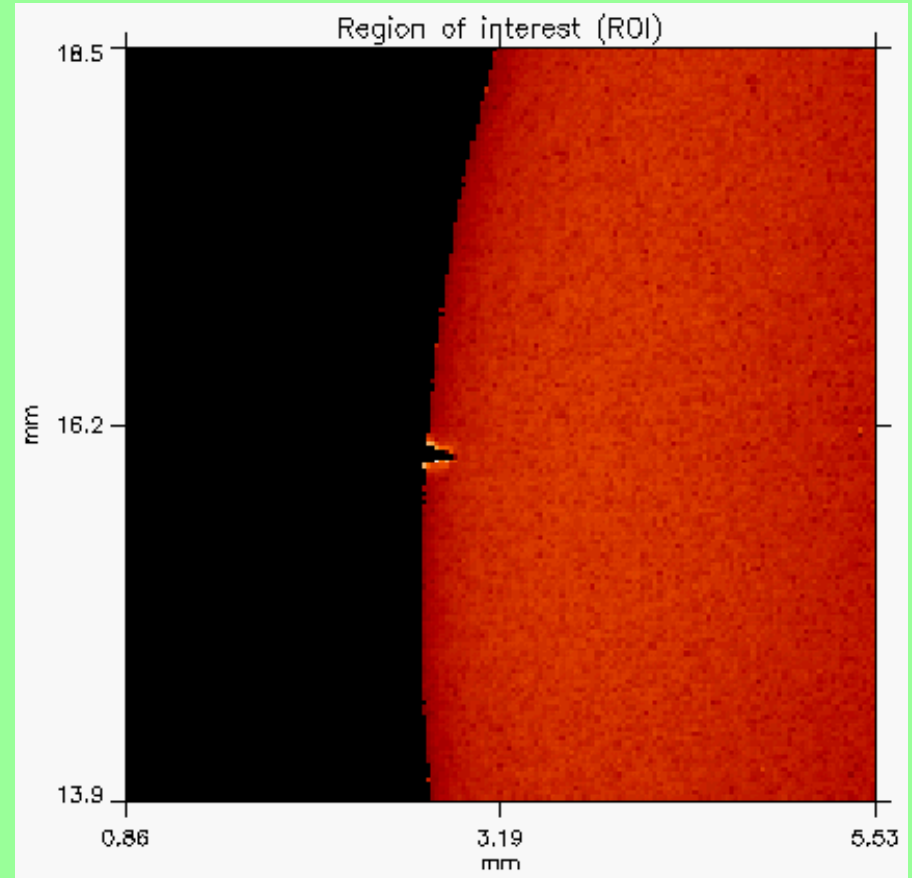
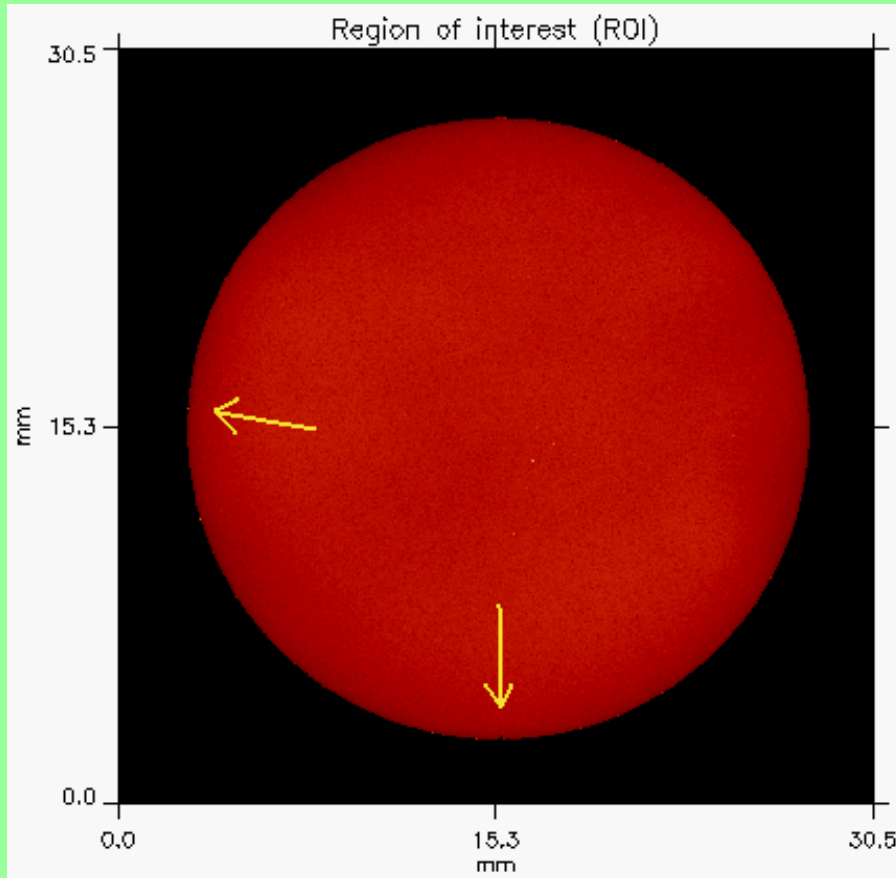


In most **EFE locations** it is difficult to identify the exact emitter position due to **complex geometry of defects**

AFM measurements are required for β_{geo} estimation of small structures

Results on single crystalline Nb samples from TJNAF

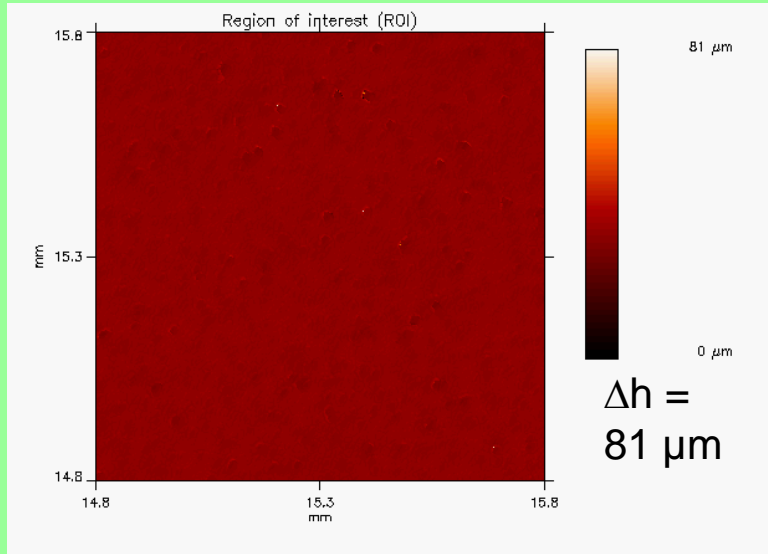
In order to investigate the possible advantages of **single crystalline Nb** for SRF, 4 round samples with **varying BCP damage layer removal** (20, 40, 80, 120 μm) and **two marks at the edge** for clear orientation have been measured with OP.



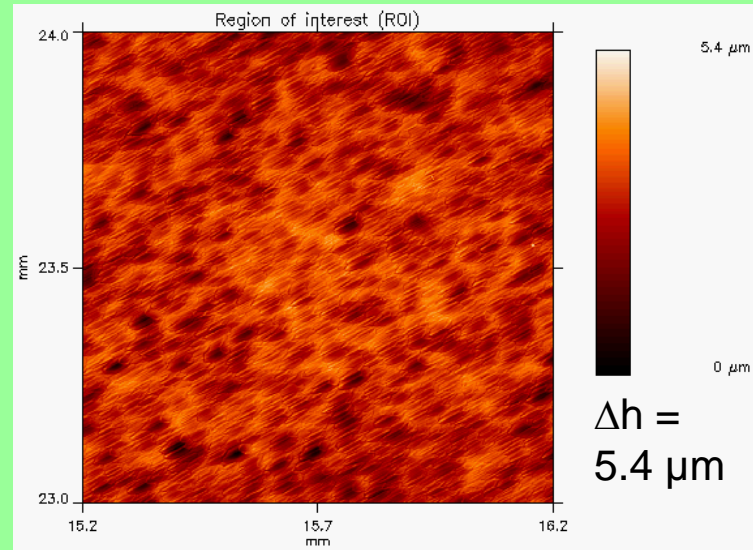
OP of single crystal Nb for different BCP layer removal

scanned area 1×1 mm²

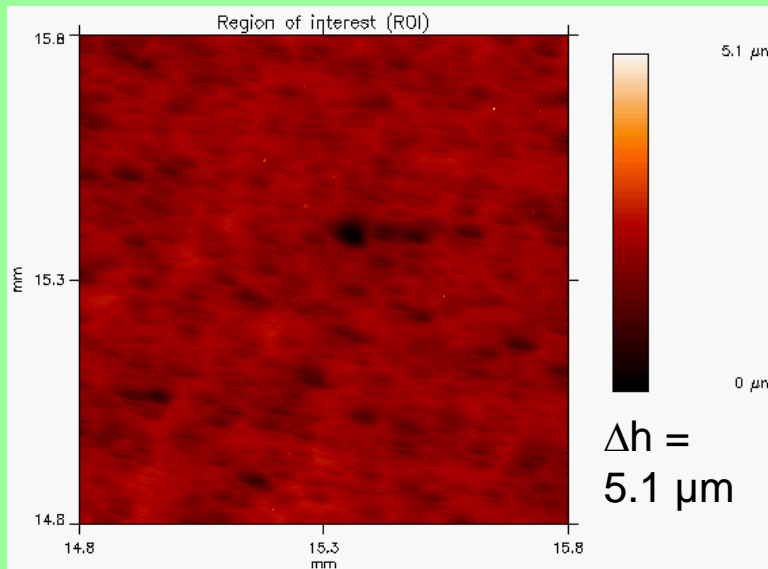
BCP
20 μm



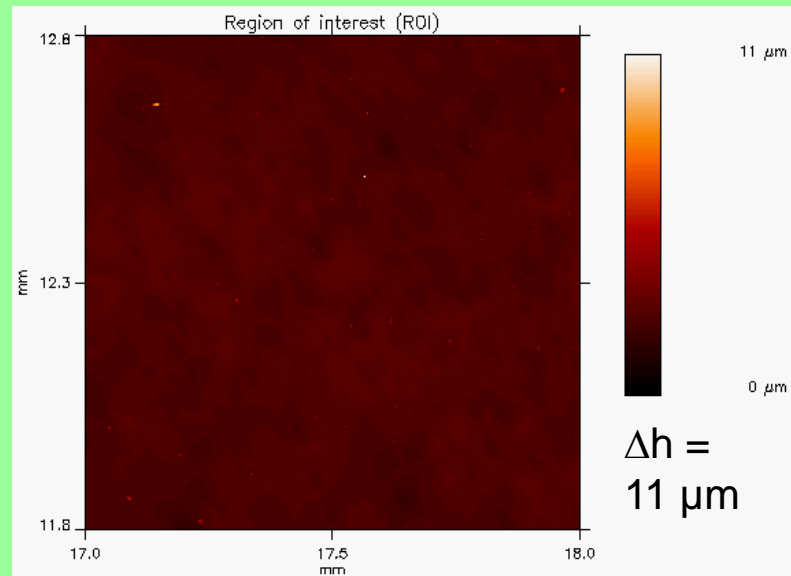
BCP
40 μm



BCP
80 μm

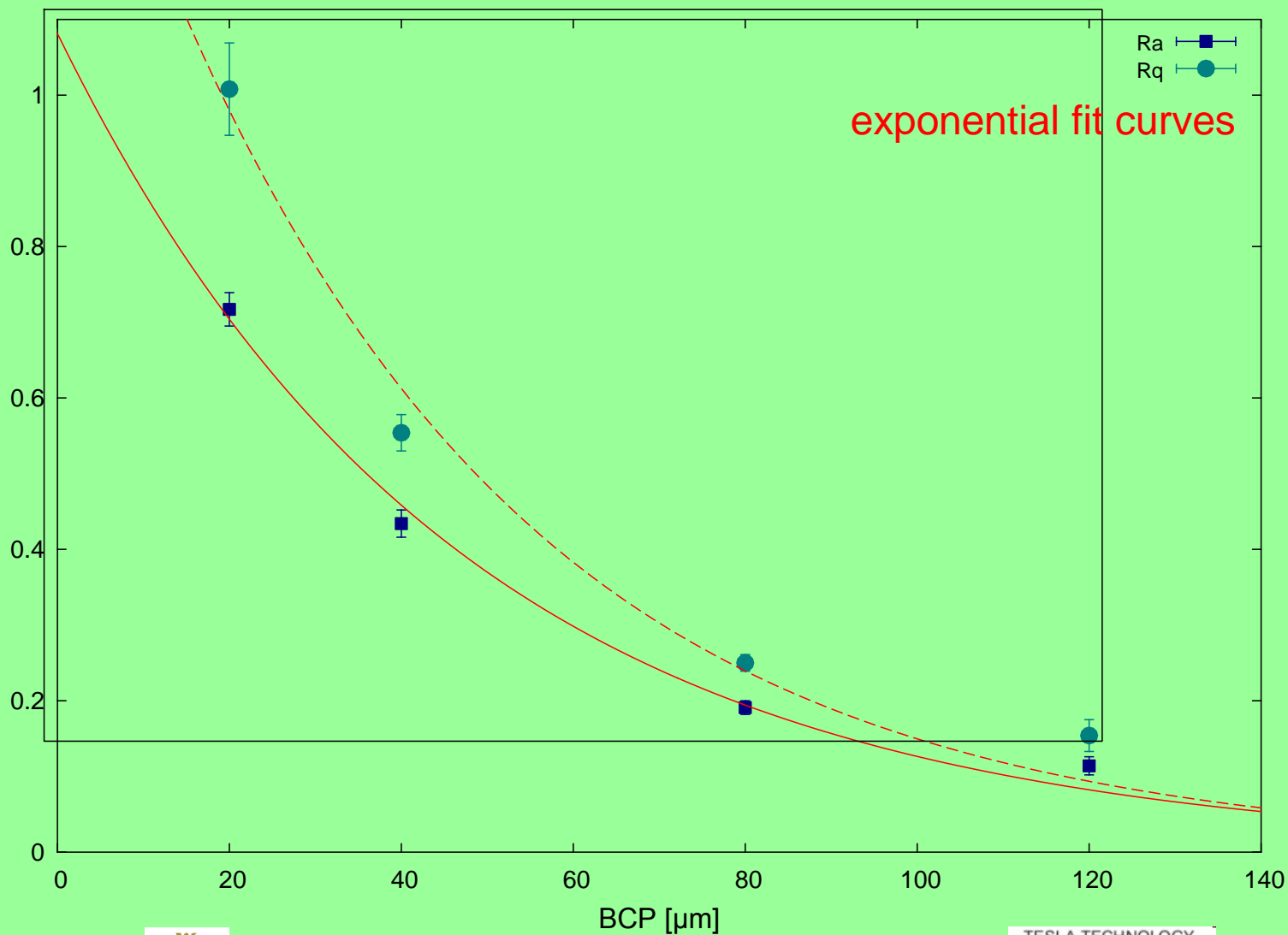


BCP
120 μm



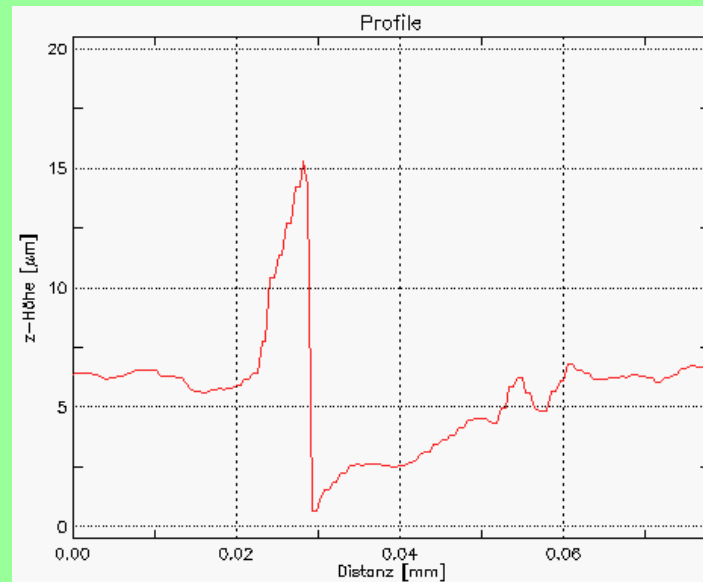
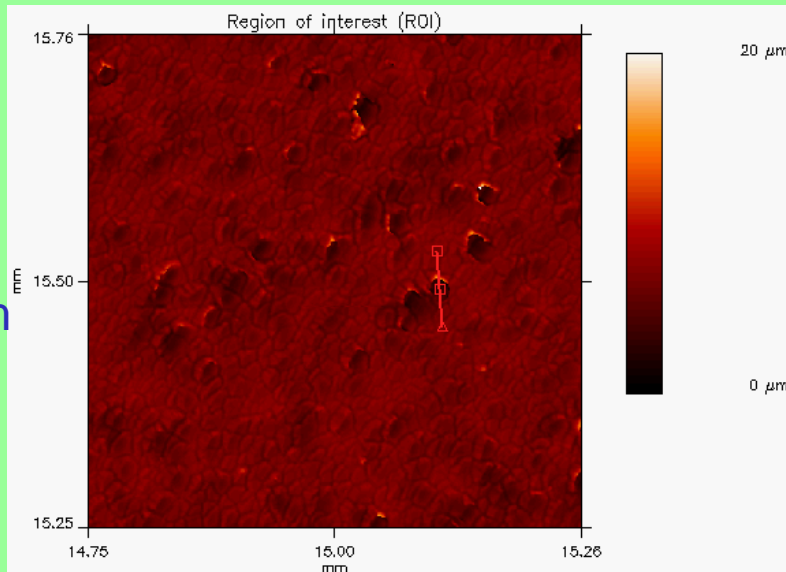
Average surface roughness of single crystal BCP-Nb

Each point based on profiles in 10 different defect-free areas of 1 mm²
BCP roughness



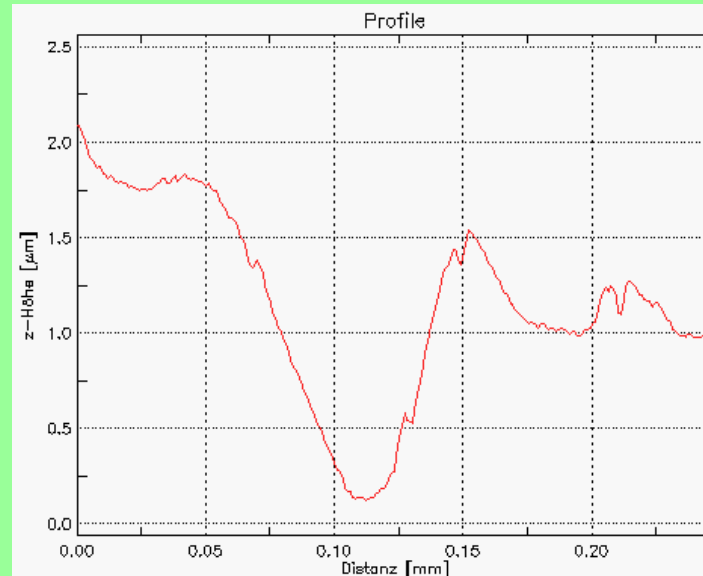
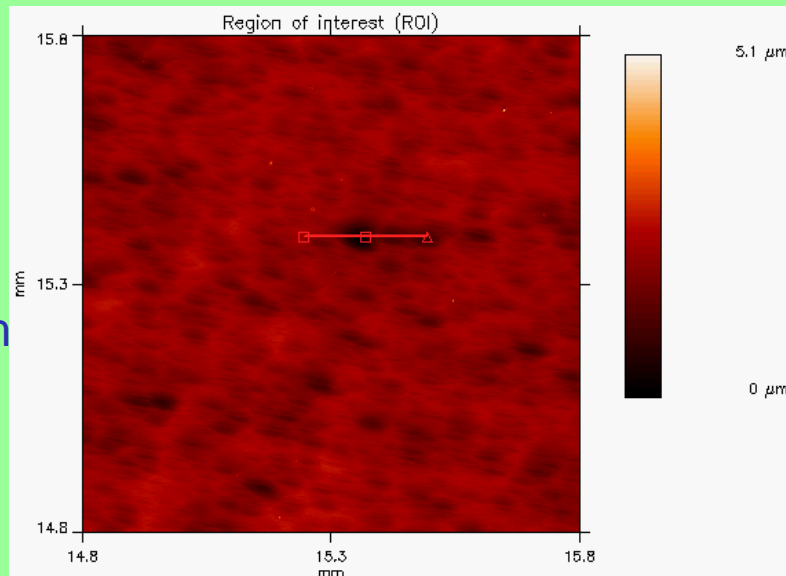
Regular pit-like features on single crystal BCP-Nb

BCP
20 μm



~ circular
sharp rim
average
 \varnothing 30 μm
 Δh 5 μm

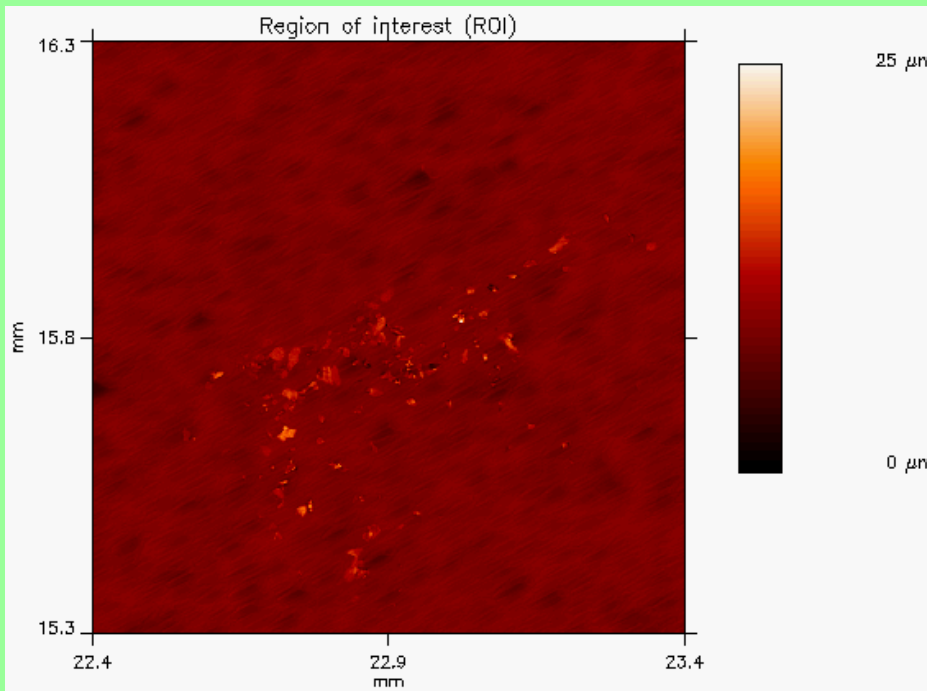
BCP
80 μm



elliptic
no rims
average
 \varnothing 60 μm
 Δh 1.5 μm

Few local defects found on single crystal BCP-Nb

All samples showed a few local defects ($> 5 \mu\text{m}$) which might cause EFE

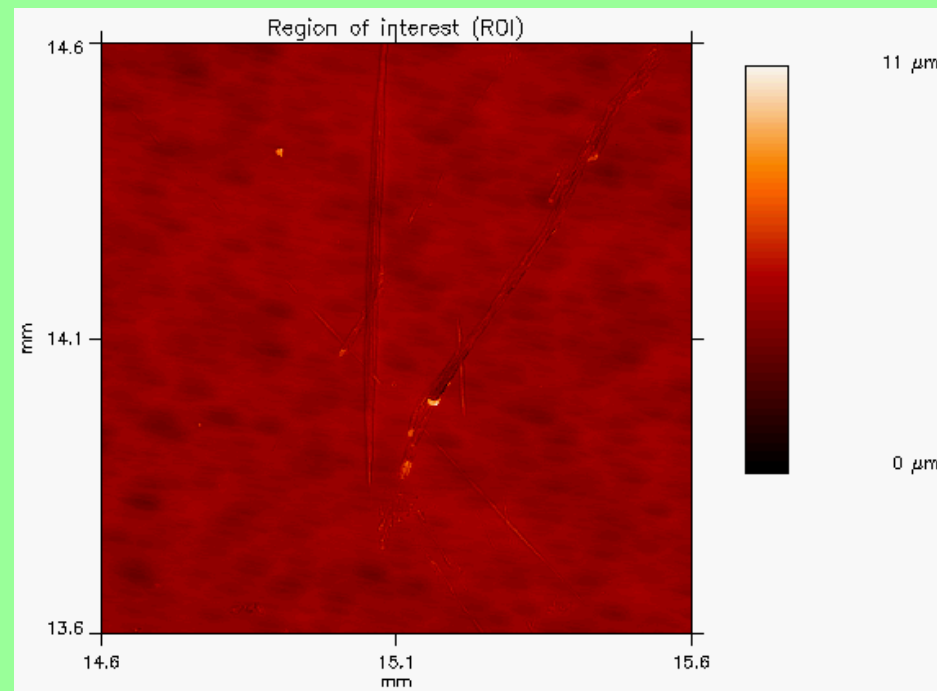


BCP 40 μm

accumulated **particulates**

average size $\sim 22 \mu\text{m}$, $\Delta h 5 = \mu\text{m}$

resistant against nitrogen blow



BCP 80 μm

long **scratch** ($\sim 0.8 \text{ mm}$)

mean width $100 \mu\text{m}$, depth $1-2 \mu\text{m}$

at one end peak $\Delta h = 10 \mu\text{m}$

Conclusion and outlook

- OP of Nb samples ($> \mu\text{m}$) is suitable for fast quality control of processes

Results for polycrystalline samples:

- Particulates with $\beta_{E,\text{max}} > 15$ must be removed by HPR and DIC
- Scratches with $\beta_{E,\text{max}} > 13$ must be prevented by a more careful handling
- Grain boundaries ($\Delta h \approx \mu\text{m}$), hills and holes are not harmful for EFE but probably cause magnetic field enhancement and limitation?
- Emitter density of EP/HPR Nb samples increases exponentially with field
- Correlation between EFE, SEM and OP of localized emitters difficult

Results and outlook for single crystalline samples:

- Mean surface roughness decreases exponentially with BCP layer removal
- Regular pit-like features and few defects found, influence on EFE ?
- Correlation between EFE, SEM and OP of localized emitters will be easier
- Activation of various types of emitters by heating will be investigated soon

