



## Update NOVALIS

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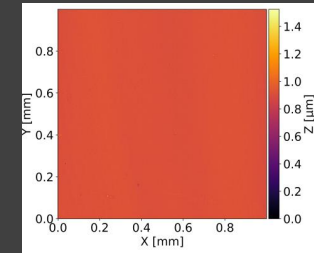
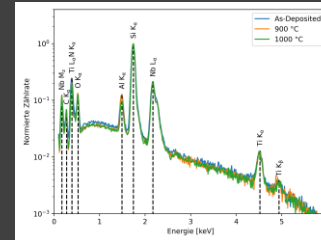
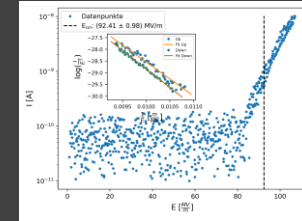
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WUPPERTAL

# NbTiN Samples

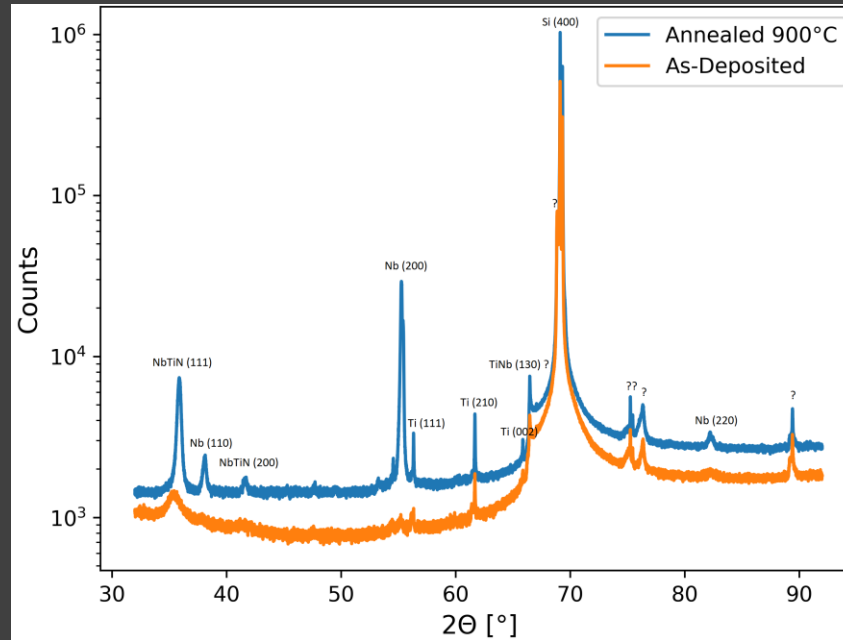
- Samples system
  - Si(100)-Wafer
  - 15 nm AlN
  - 60 nm Nb<sub>0.75</sub>Ti<sub>0.25</sub>N
- Three samples
  - As-Deposited
  - Annealed at 900 °C (1 h)
  - Annealed at 1000 °C (1 h)

## Analysing with

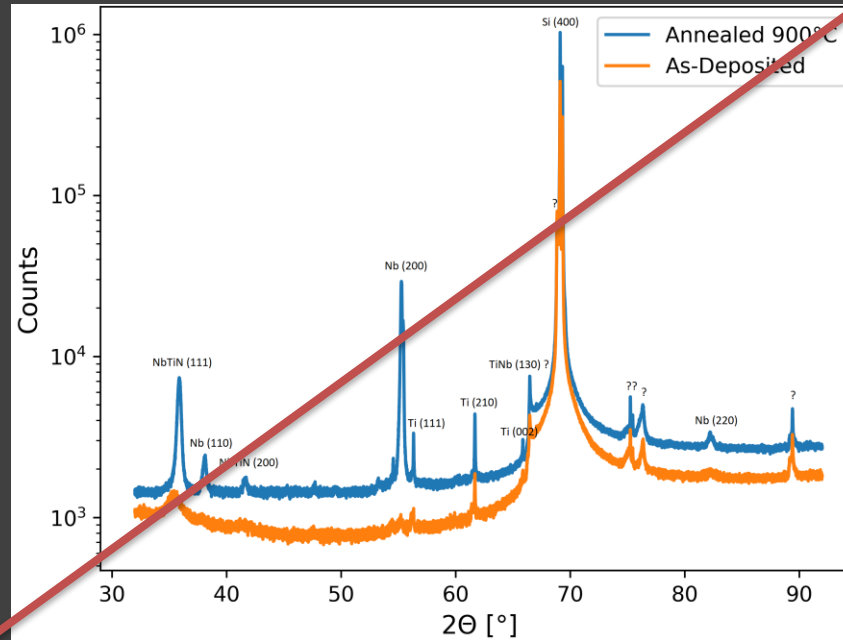
- FESM
- XRD
- EDX
- SEM
- OP



# Results of XRD Measurement

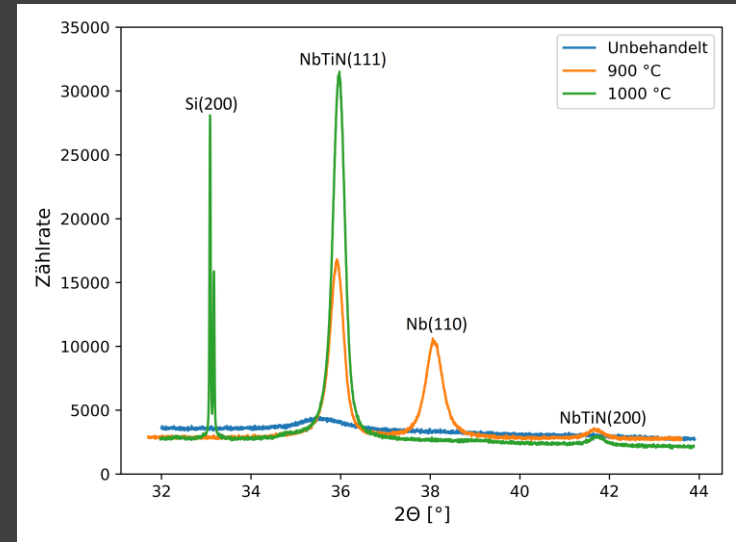


## Results of XRD Measurement

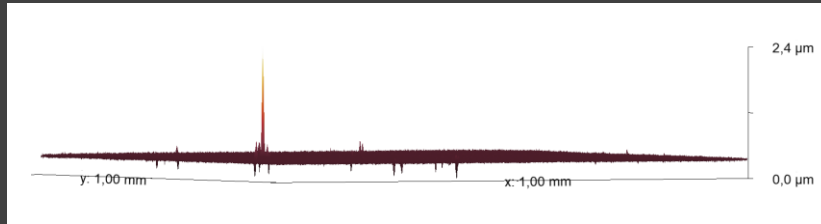


## Results of XRD Measurement

- Only NbTiN and Si Peaks visible
    - Nb(110) belongs to sample holder
  - As-Deposited NbTiN under heavy microstrain
    - high fwhm and bragg angle is lower
  - Bragg-angle of NbTiN(111) increases
    - As-Deposited:  $35.565 \pm 0.013^\circ$
    - 900 °C:  $35.916 \pm 0.001^\circ$
    - 1000 °C:  $35.960 \pm 0.001^\circ$
  - Grainsize of NbTiN(111) increases
    - As-Deposited:  $75.297 \pm 3.480 \text{ \AA}$
    - 900 °C:  $272.182 \pm 1.532 \text{ \AA}$
    - 1000 °C:  $319.084 \pm 1.631 \text{ \AA}$
- Annealing releases stress in the film
- NbTiN(200) is formed by annealing

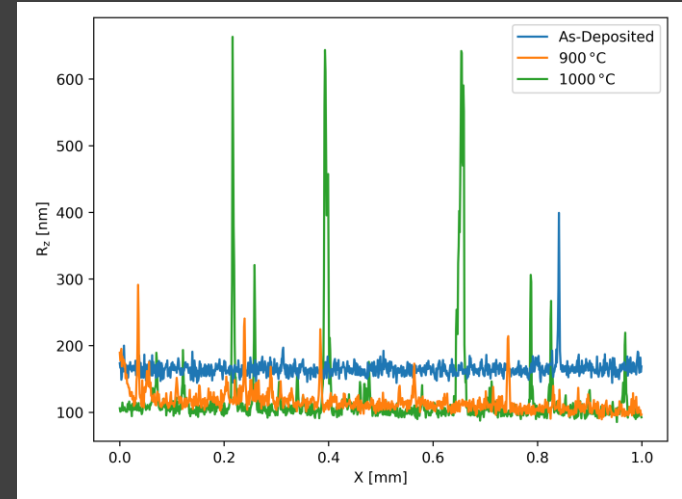


## Results for the surface roughness

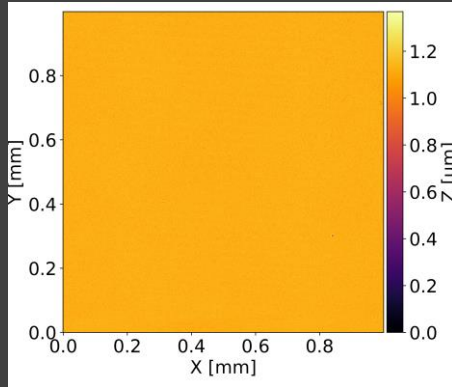


1000 °C

- A few deeper defects on the surface
- 3D-view shows distinction between defect types
  - Annealing promotes formation of defects
  - Wafer defects become visible
- Determination of  $R_z$  over a few subareas

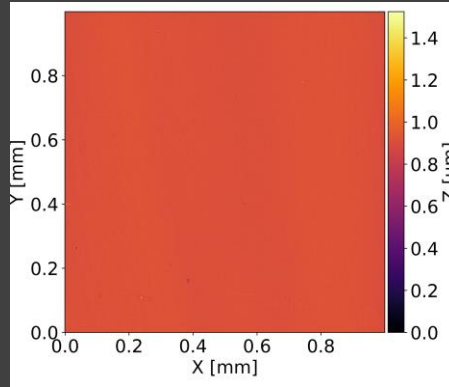


## Results for the surface roughness



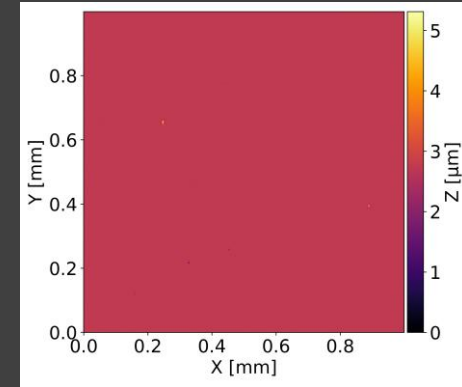
As-Deposited

- $R_a = (24.11 \pm 0.76) \text{ nm}$
  - $R_z = (141.67 \pm 3.06) \text{ nm}$
- over  $10 \times 10 \text{ mm}^2$



Annealed at 900 °C

- $R_a = (15.93 \pm 0.33) \text{ nm}$
  - $R_z = (94.59 \pm 2.00) \text{ nm}$
- over  $10 \times 10 \text{ mm}^2$

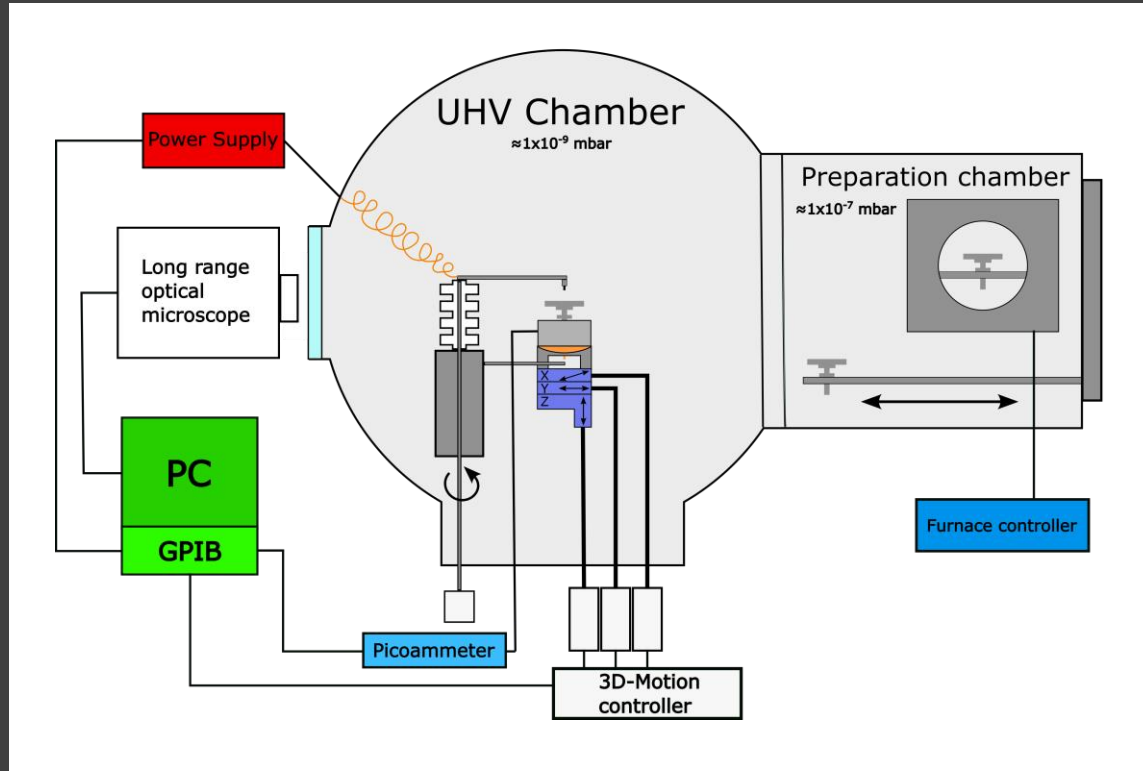


Annealed at 1000 °C

- $R_a = (14.15 \pm 0.82) \text{ nm}$
  - $R_z = (80.34 \pm 0.13) \text{ nm}$
- over  $10 \times 10 \text{ mm}^2$

→ Annealing reduces  $R_a$  by 41.30 % and  $R_z$  by 43.29 %

# Experimental Setup



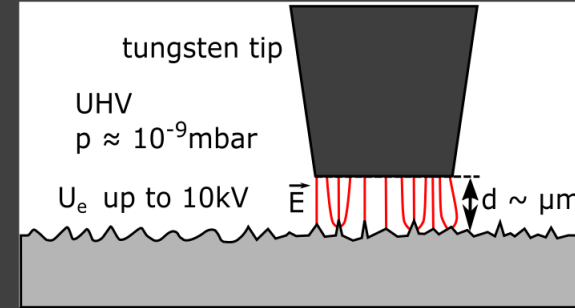


# Working principle

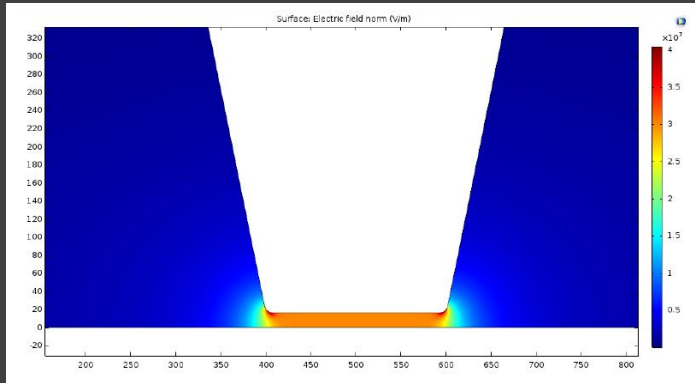
Current flows after Fowler-Nordheim:

$$I(E) = \frac{AS\beta^2 E^2}{\Phi} \exp\left(-\frac{B\Phi^{\frac{3}{2}}}{\beta E}\right)$$

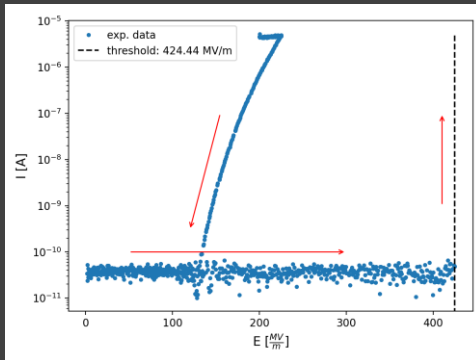
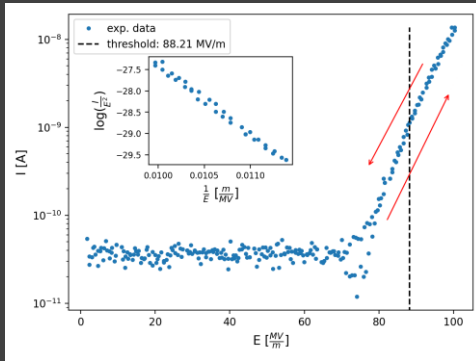
With  $A = 1.54 \cdot 10^6 \frac{\text{AeV}}{(\text{MV})^2}$ ,  $B = 6830 \frac{eV^{\frac{3}{2}}\text{MV}}{m}$ ,  $\Phi$  as work function,  $\beta$  as geometric factor of the tip/surface and  $S$  as effective area of emittance



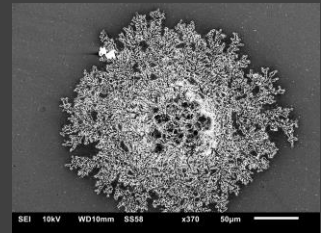
- Surface roughness leads to enhanced field emission → lowers onset-field
- Tip radius and shape can vary heavily
  - Between a few nm and 1 mm
  - Sharper tips yield higher resolution
  - Truncated cone tips are more mechanical stable



# Current-Voltage-Curves (IV-Kurven)



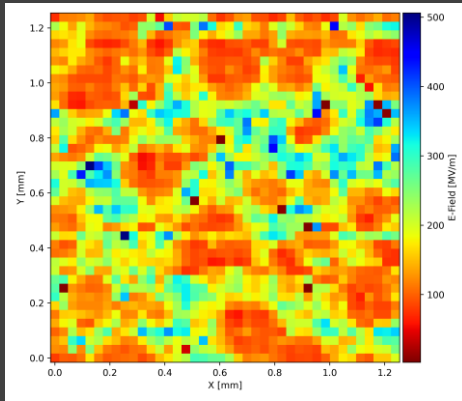
- Increasing voltage until selected current, then decreasing voltage
- Onset-field threshold set to 1 nA
- Plotting and fitting  $\text{Log}\left(\frac{I}{E^2}\right)$  vs.  $\frac{1}{E}$  yields  $\beta$ ,  $S$  and  $\Phi$
- On some measurements sudden jump to high currents
  - called **activation of surface**
  - activation irreversibly changes surface
- Onset-field threshold set at the highest field strength



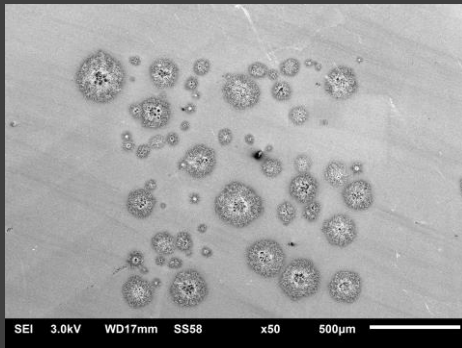
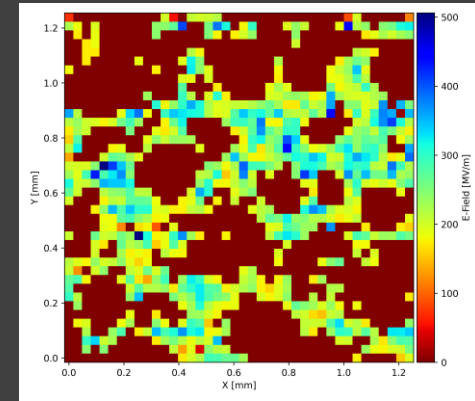
# Measurement programm for the FESM

- Measuring I-V-Curves over  $6.5 \times 6.5 \text{ mm}^2$ 
  - 440 I-V-Curves in total
  - Max. current 10 nA
- Stepsize  $317.65 \text{ }\mu\text{m}$ 
  - No overlap with  $250 \text{ }\mu\text{m}$  tip
- Evaluation of I-V-Curves with
  - Constant Current Map
    - Determination of  $E_{\text{on}}$
  - $\beta$ ,  $\phi$  und  $S$

## Constant Current Map – filtering of damaged areas

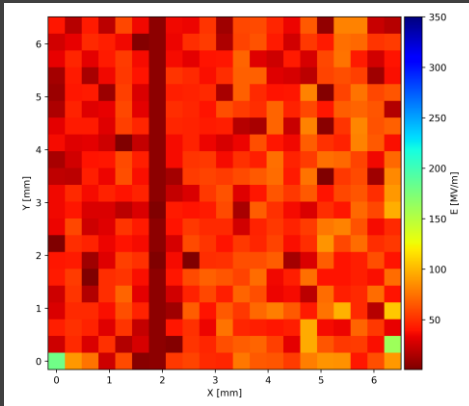


Damage threshold:  $140 \frac{\text{MV}}{\text{m}}$

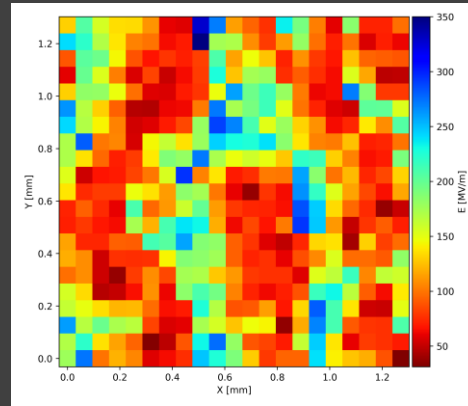
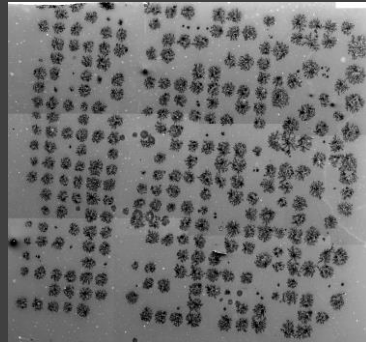


- Damages has to be filtered out
- ➔ Setting individual threshold for each map

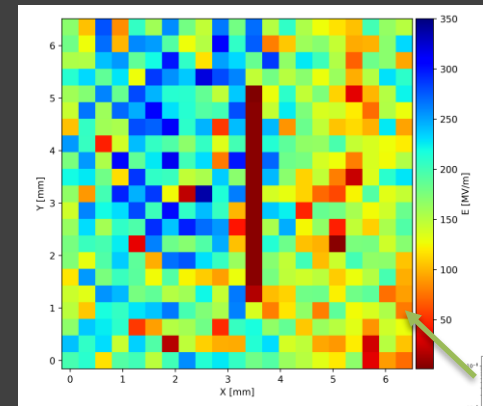
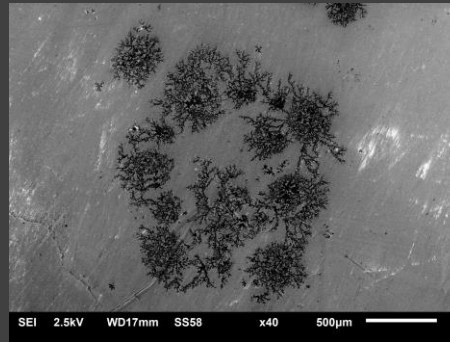
# Results of Constant Current Map



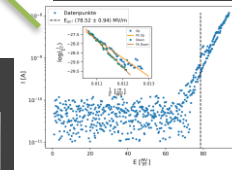
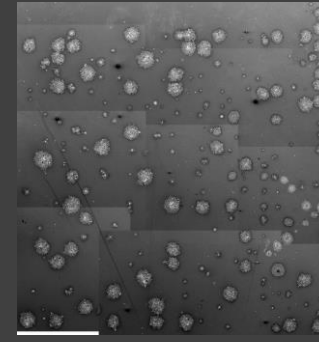
As - Deposited



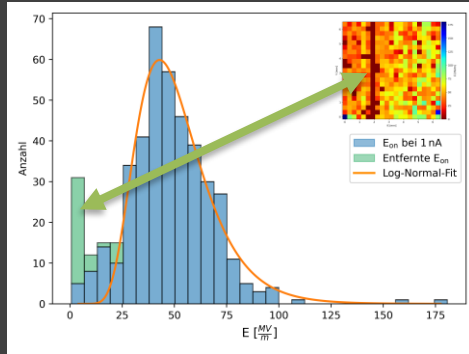
900 °C



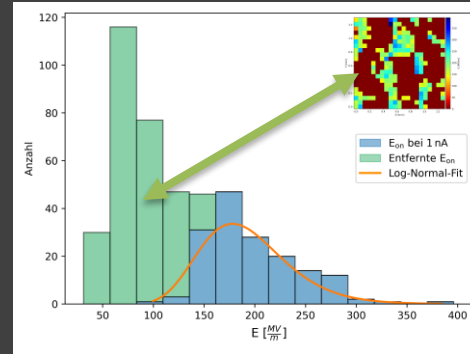
1000 °C



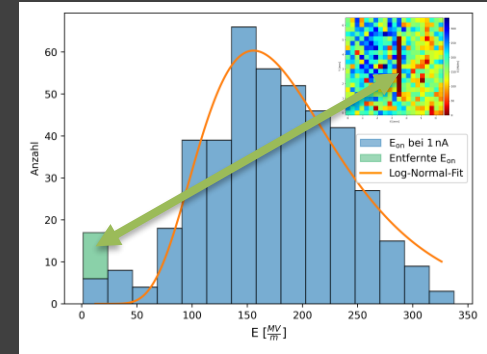
# Results Constant Current Map



$$\bar{E}_{on}: 51.53 \pm 1.03 \frac{\text{MV}}{\text{m}}$$



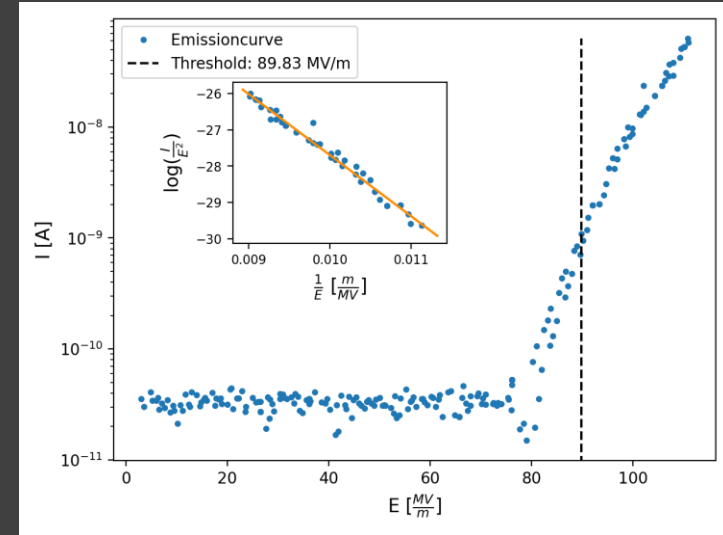
$$\bar{E}_{on}: 192.37 \pm 2.87 \frac{\text{MV}}{\text{m}}$$



$$\bar{E}_{on}: 195.71 \pm 6.43 \frac{\text{MV}}{\text{m}}$$

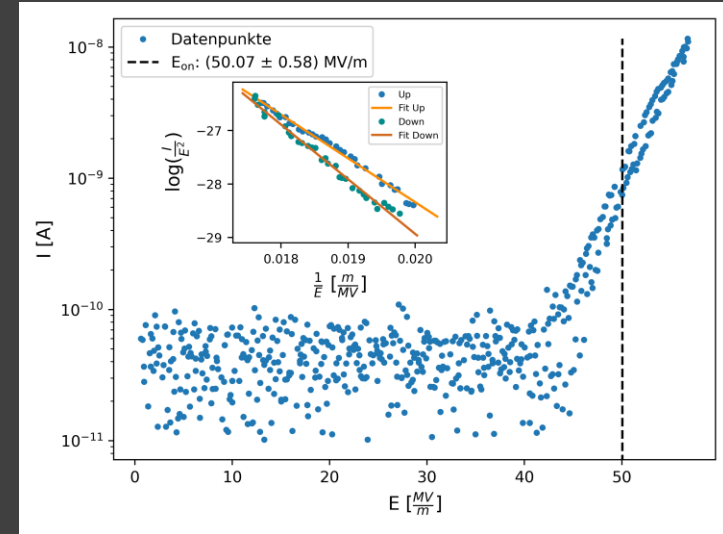
# Analysis of I-V-Curves

- Determination of  $\beta$ ,  $\phi$  and  $S$  separate over Up and Down direction
  - Altering the surface through FE
    - $\beta$  and  $S$  change
- Fits are done with varying  $\beta$  and keeping  $S$  and  $\phi$  fix for several values of  $S$  and  $\phi$ 
  - Setting damage threshold
  - Work function between 2.5 eV and 5.5 eV
    - Stepsize 0.05 eV
  - $S$  between  $10^{-23} \text{ m}^2$  and  $10^{-8} \text{ m}^2$ 
    - Stepsize  $0.1 \cdot 10^x$
  - Damaged areas will not be taken into account



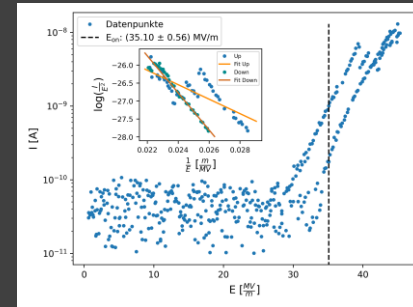
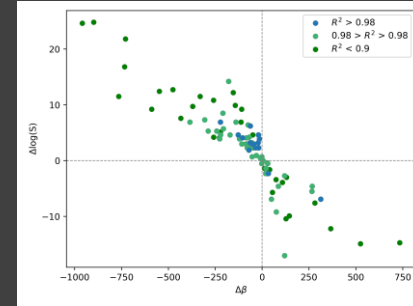
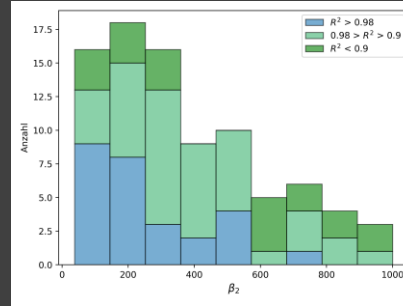
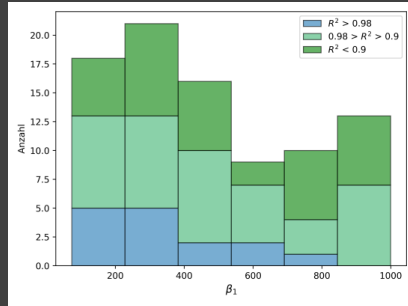
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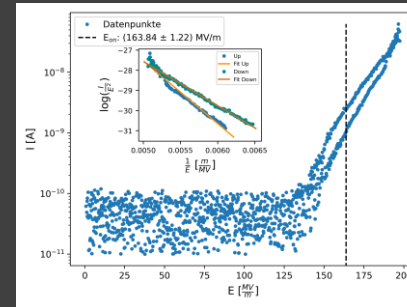
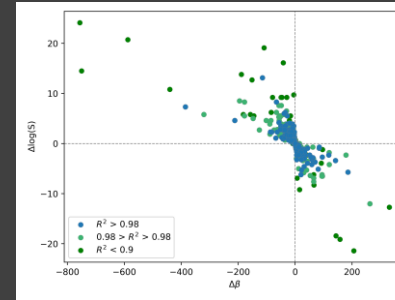
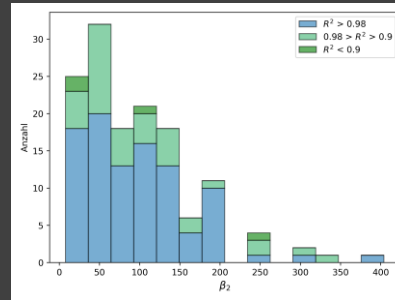
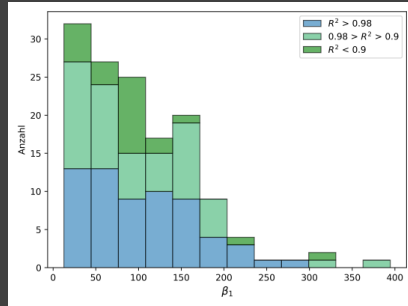


# Results of the geometric factor – As - Deposited



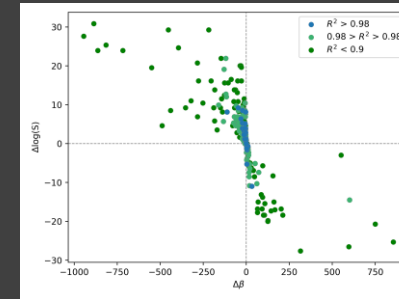
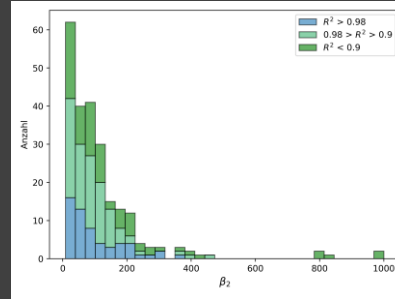
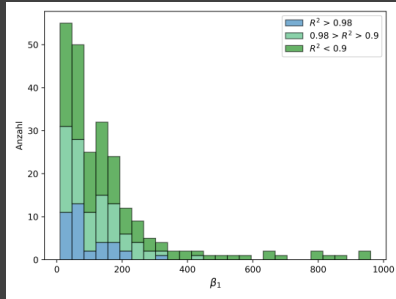
- Out of 440 Measurements only 92 were suitable for fitting
  - Data from activation of the surface is not suitable for fitting
- Quality of fits increases for  $\beta_2$
- Comparison between  $\beta_1$  and  $\beta_2$ 
  - In 61 cases  $\beta_1 > \beta_2$
  - In 26 cases  $\beta_1 < \beta_2$
  - In 5 cases no change
- Microtips are destroyed
- Problem: damaged surface → bigger  $\beta_1$ 
  - Film destruction during Up-Direction at currents as low as  $I \approx 1$  nA
- Negative correlation between  $\log(S)$  and  $\beta_1$

# Results of the geometric factor– 900 °C Probe



- Lots of damaged areas reduces data suitable for fitting
- Quality of fits increases for  $\beta_2$
- No real changes between  $\beta_1$  and  $\beta_2$
- Negative correlation between  $\log(S)$  and  $\beta_1$

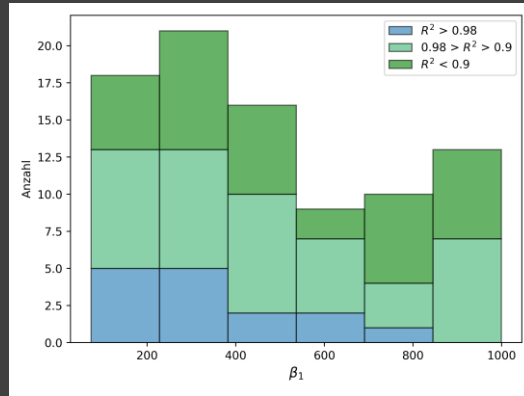
# Results of the geometric factor – 1000 °C Probe



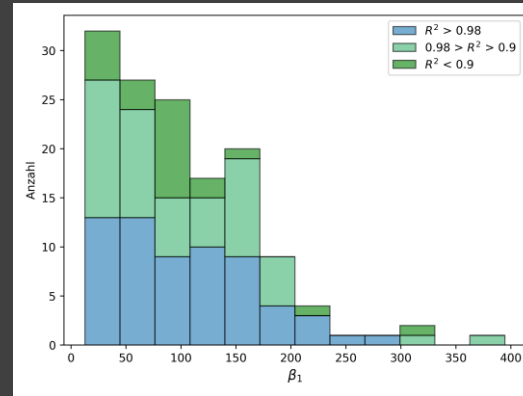
- Out of 440 Measurements 235 were suitable for fitting
  - Noticeable less surface damage
- Comparison between  $\beta_1$  and  $\beta_2$ 
  - In 111 cases  $\beta_1 > \beta_2$
  - In 44 cases  $\beta_1 < \beta_2$
  - In 80 cases no change→ Microtips are destroyed
- Negative correlation between  $\log(S)$  and  $\beta_1$

# Results of the geometric factor – comparison

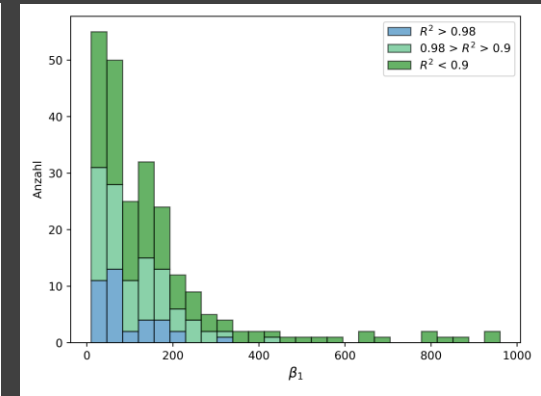
Annealed samples show lower  $\beta_1$ -values  
→ Reduces surface roughness / less surface damage



As-Deposited

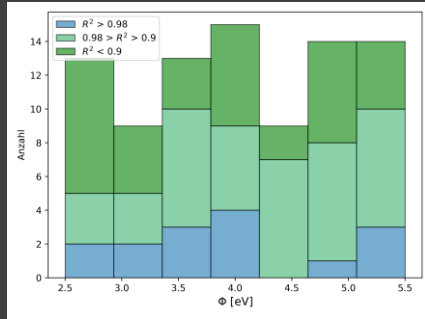


900 °C

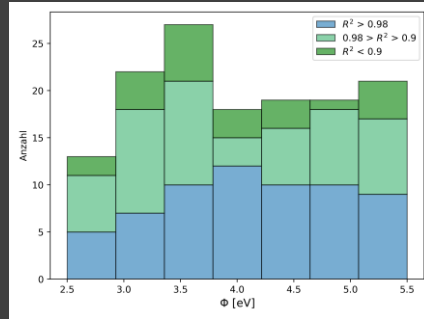


1000 °C

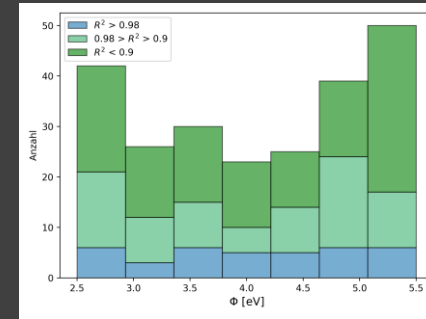
## Results for the work function



As-Deposited



900 °C

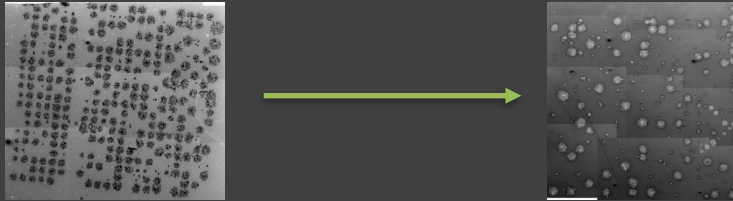


1000 °C

- Damage threshold is taken into account
- No clear conclusion about the work function can be drawn
  - Damage can influence the results for the work function
- UPS / Kelvin probe measurements will be done to determine  $\Phi$

# Conclusion

- Annealing NbTiN sample has lots of advantages for FE
  - Increasing grainsize of NbTiN
    - from  $\approx 75 \text{ \AA}$  to  $\approx 320 \text{ \AA}$
  - Releasing stress in the film
  - Reducing  $R_z$  by 43.29 %
    - Less/smaller microtips lowers  $\beta$
  - Increasing onset-field significantly
    - from  $\approx 50 \frac{\text{MV}}{\text{m}}$  to  $\approx 200 \frac{\text{MV}}{\text{m}}$
  - Improved film adhesion due to reduced stress
    - Significantly reduced surface destruction after FE



Thank you for your  
Attention