Scanning Electron Microscope (SEM) in situ field emission measurement in Uppsala Univ.

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Background

Location: Developments & Experiments in Uppsala Univ. (UU).
Motivation: CLIC feasibility study / fundamental interest of understanding FE and BD phenomena, of material science.
Purpose: Find dependencies (field, time, gap, geometry, material, treatment, …, crystal orientation,…)
Speciality 1: Local filed emission and breakdown measurements inside an SEM.
Speciality 2: Post-breakdown surface & sub-surface analyses by Focused Ion Beam (FIB) and SEM.

Ideas – What we can do

Speciality 1: Local filed emission and breakdown measurements inside an SEM.

- Reproduce high-gradient electric field condition in μm range (1kV/μm = 1GV/m)
- 2D scan with controlled gap distance.
- Measurement and observations in one instrument.
- Compact & transportable experimental setup.
- Complementary work with Wuppertal Univ.

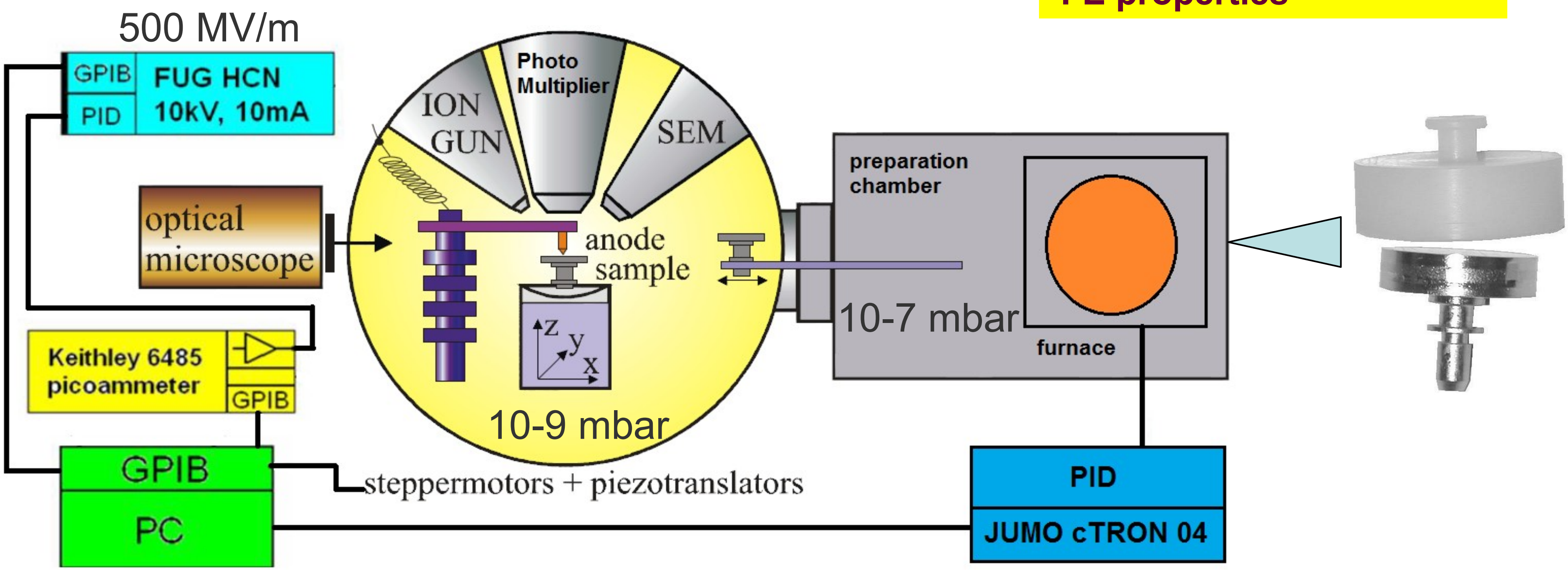
Courtesy of Stefan Lagotzky, Günter Müller, Berg. Universität Wuppertal

FE & SEM measurement techniques

Field emission scanning microscope (FESM):

- localisation of emitters

- FE properties



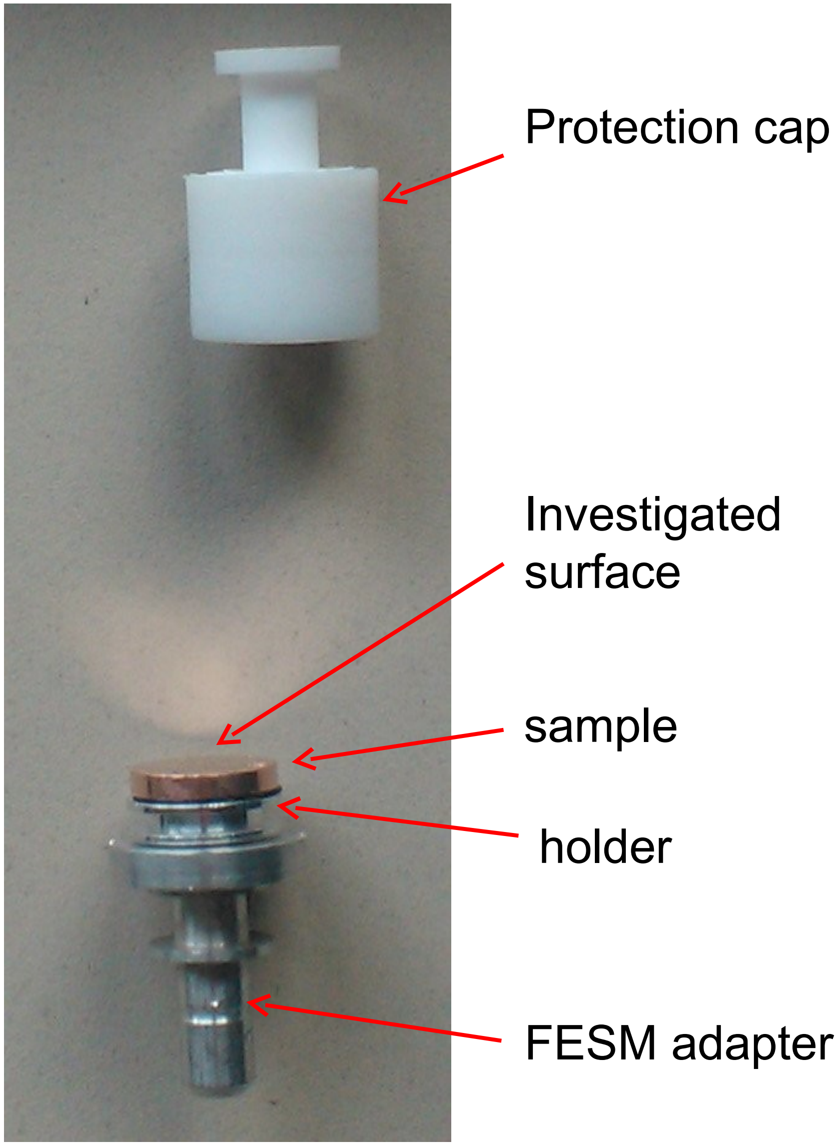
- Regulated V(x,y) scans for FE current I=1 nA & gap Δz Ŭ emitter density at E=U/Δz
- Spatially resolved I(E) measurements of single emitters Ŭ Eon, βFN, S
- Ion bombardment (**Ar,** Eion= 0 – 5 kV) and SEM (low res.)
- In-situ heat treatments up to 1000°C

Ex-situ SEM + EDX **Identification of emitting defects**

Correlation of surface features to FE properties (positioning accuracy ~ ±100 µm)

Courtesy of Stefan Lagotzky, Günter Müller, Berg. Universität Wuppertal

Samples



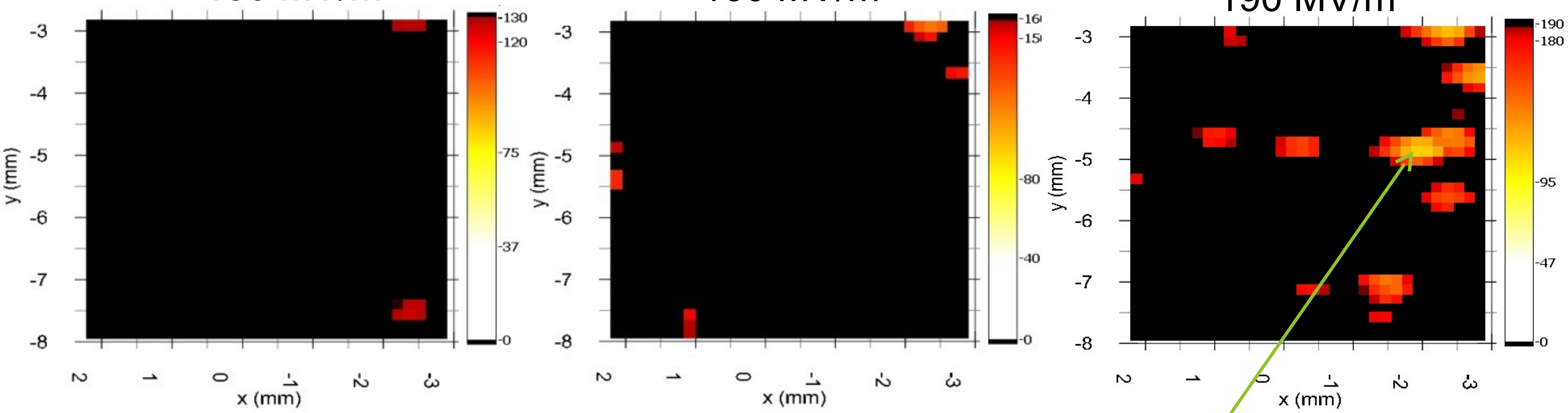
- Two flat **Cu** samples
- Diameter: ~11 mm
- One hole as mark to identify the position in different systems
- Diamond turned and glued to a sample holder at CERN
- Mounted to an adapter for the FESM at BUW
- Surface cleaned with **ionized N2**, cleanroom condition (class 100) with 5 bar pressure
- **Teflon protection cap** to avoid damage and contaminations after polishing and cleaning
- Only 1 sample measured yet

Courtesy of Stefan Lagotzky, Günter Müller, Berg. Universität Wuppertal

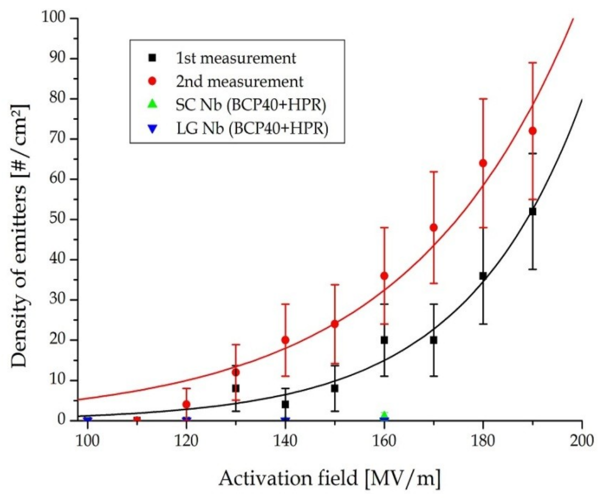
FESM results

Regulated E(x,y) maps for I = 1 nA , Δz ≈ 50 μm of the same area

130 MV/m 160 MV/m 190 MV/m



Eon = 120 MV/m

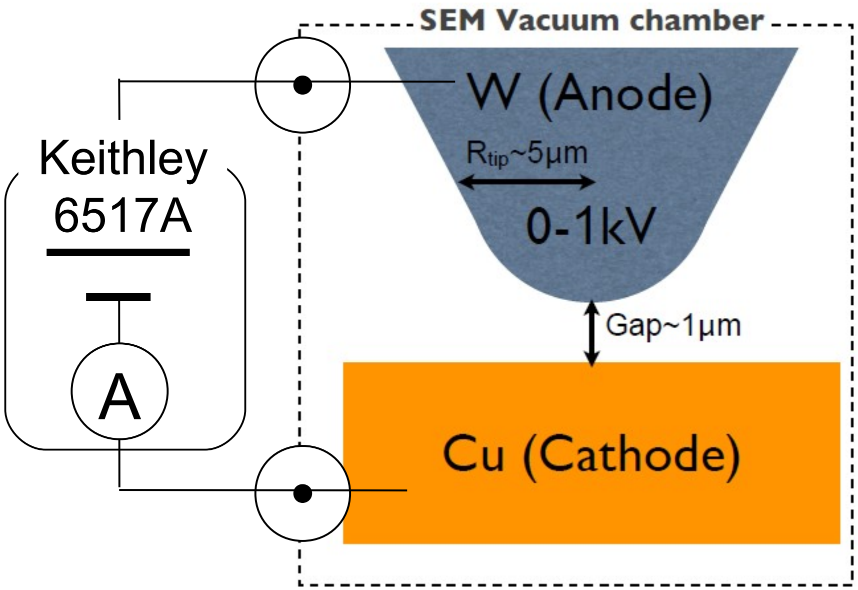


- **EFE starts at 130MV/m and not 500MV/m**
- Emitter density increases exponentially with field
- Activated emitters: Eact=(1,2 – 1,4)·Eon

→ 2nd measurement: shifted to lower fields

Possible explanations:
- Surface oxide
- adsorbates

Experiment setup in UU



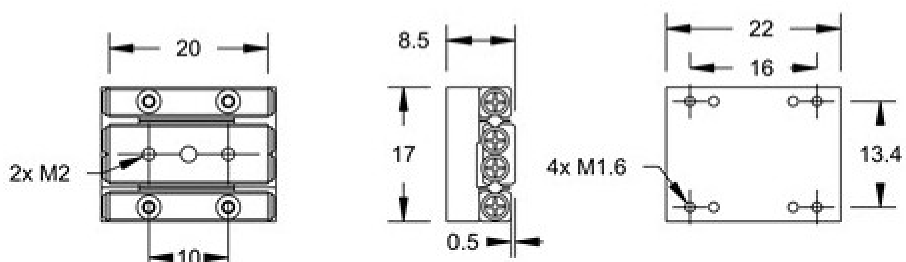
- Cathode: Cu samples provided by CERN, 12mm-D, XY-stage
- Anode: W tip commercially available, 5μm-R, Z-stage
- Gap: 1.0 ± 0.1 μm
- Background current ≈0.02pA

SmarAct Piezo positionner
http://www.smaract.de/

SLC-1720-S

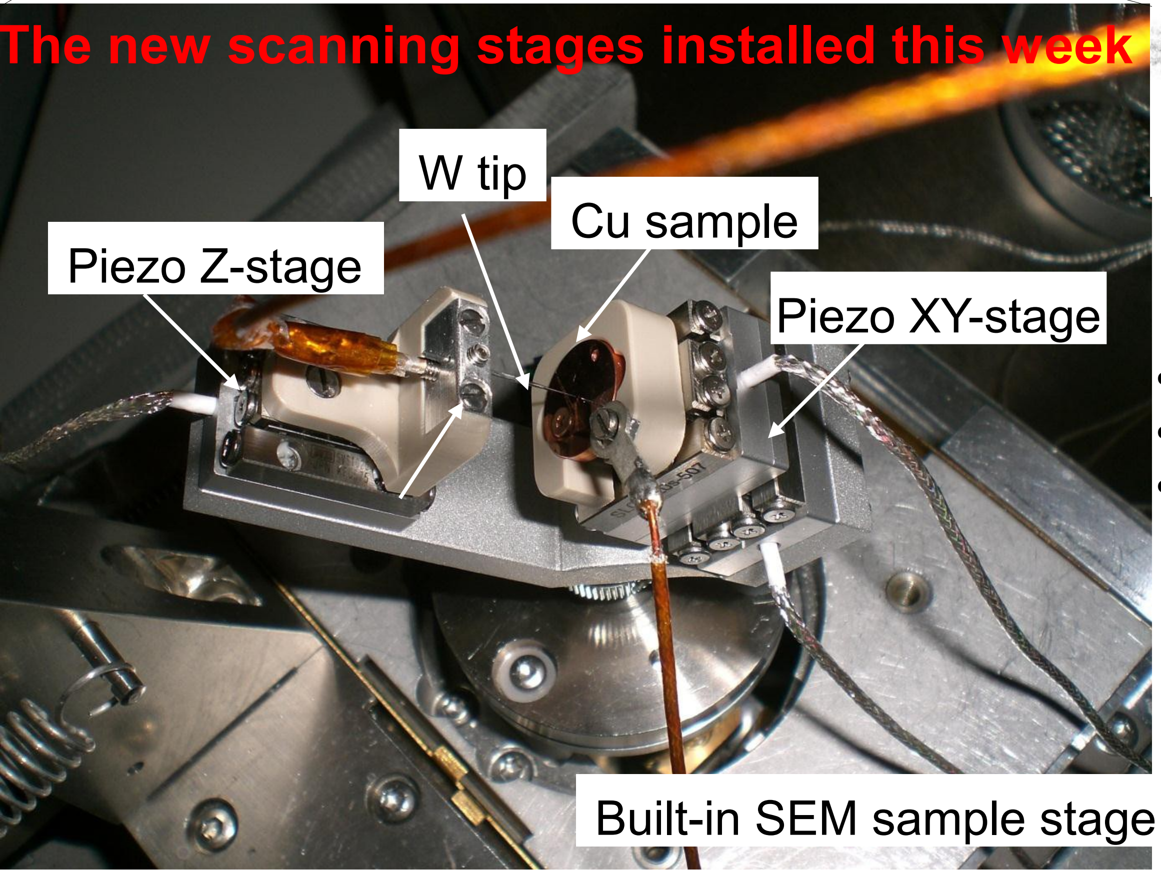
- Dimensions: 22 x 17 x 8.5 mm³
- Travel: about 12 mm
- Velocity: up to 13 mm/s
- Step width: 50 nm to 1000 nm
- Scanning range: about 1.4 µm
- Resolution: sub-nanometer
- Blocking force: up to 3 N
- Weight: about 13 g
- Allowable load: 40 N
- Integrated nanosensor
- Options:
  - Vacuum compatibility: HV, UHV
  - Non-magnetic

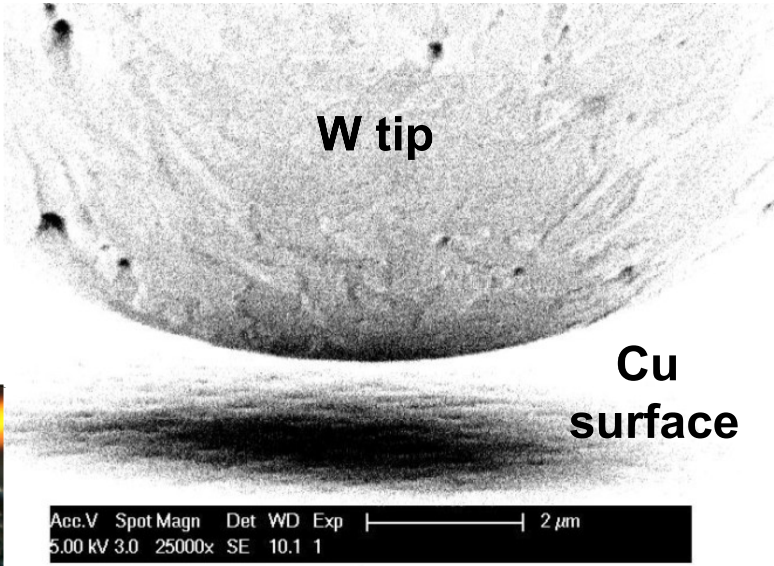
Drawing:



Linear dimensions are given in mm.

Real life



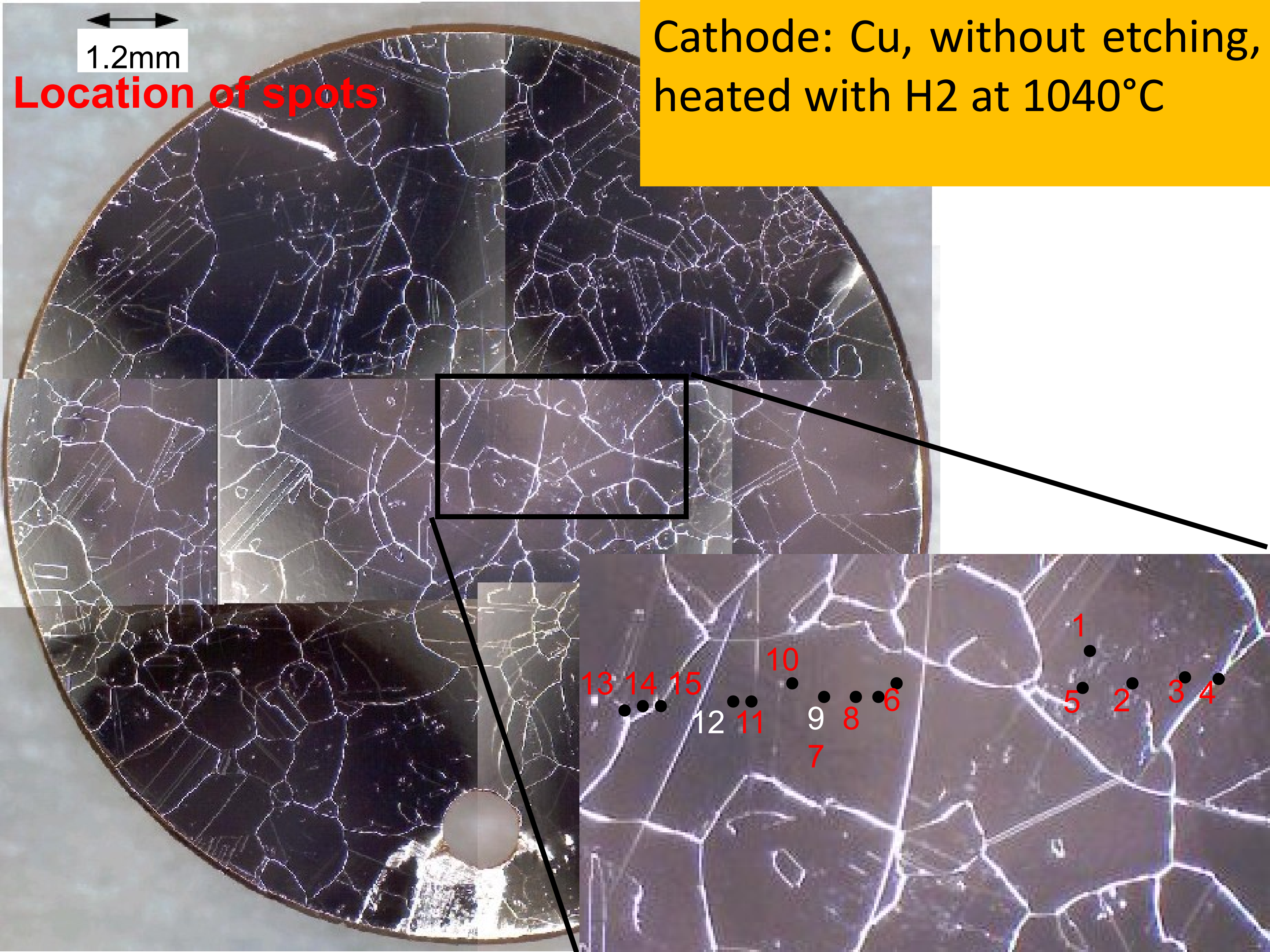


- Targeted scan
- Emission “SPOT” (≠area)
- Surface observation before&after

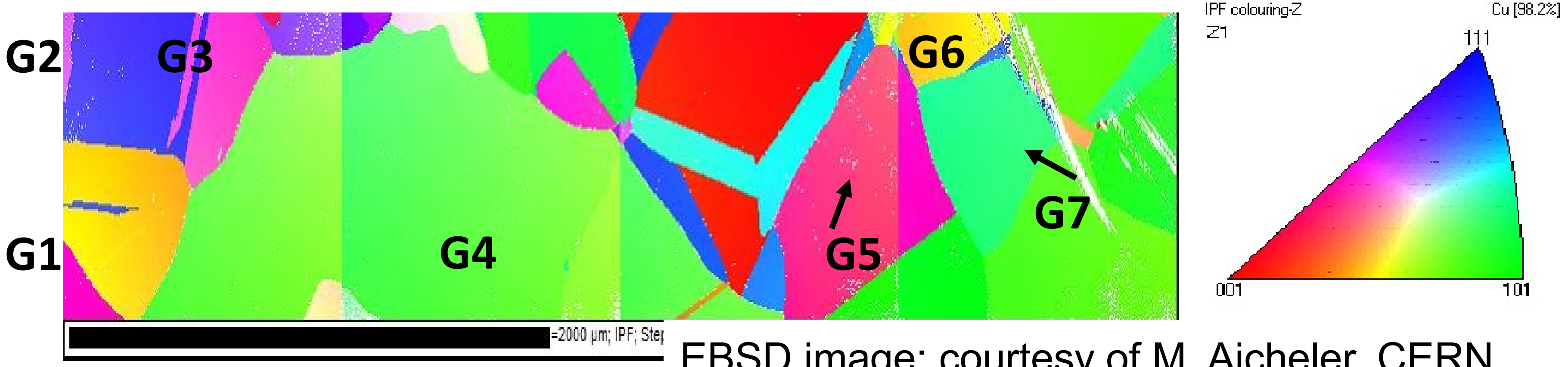
Example:
Grain orientation dependence
(done with the old setup)

1. Approach W tip to the sample surface ~1μm
2. Take SEM images of target area
3. Apply HV on W tip from 0V up to 1kV with 1V step
4. Measure emission current from Cu sample
5. Stop HV supply once the current exceeds 10 nA (onset voltage)
6. Repeat 5 runs at the same spot
7. Move sliders to a new spot (blind)
8. Analyses: Comparison of onset voltage of each spots, Electron Backscatter Diffraction (EBSD) in order to identify grain orientations of spots.

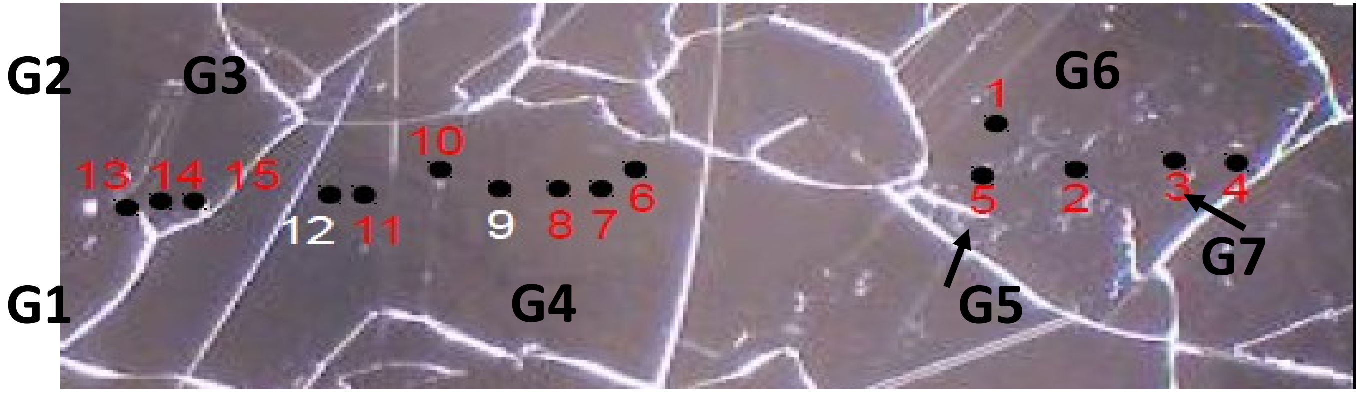
Cathode: Cu, without etching, heated with H2 at 1040°C



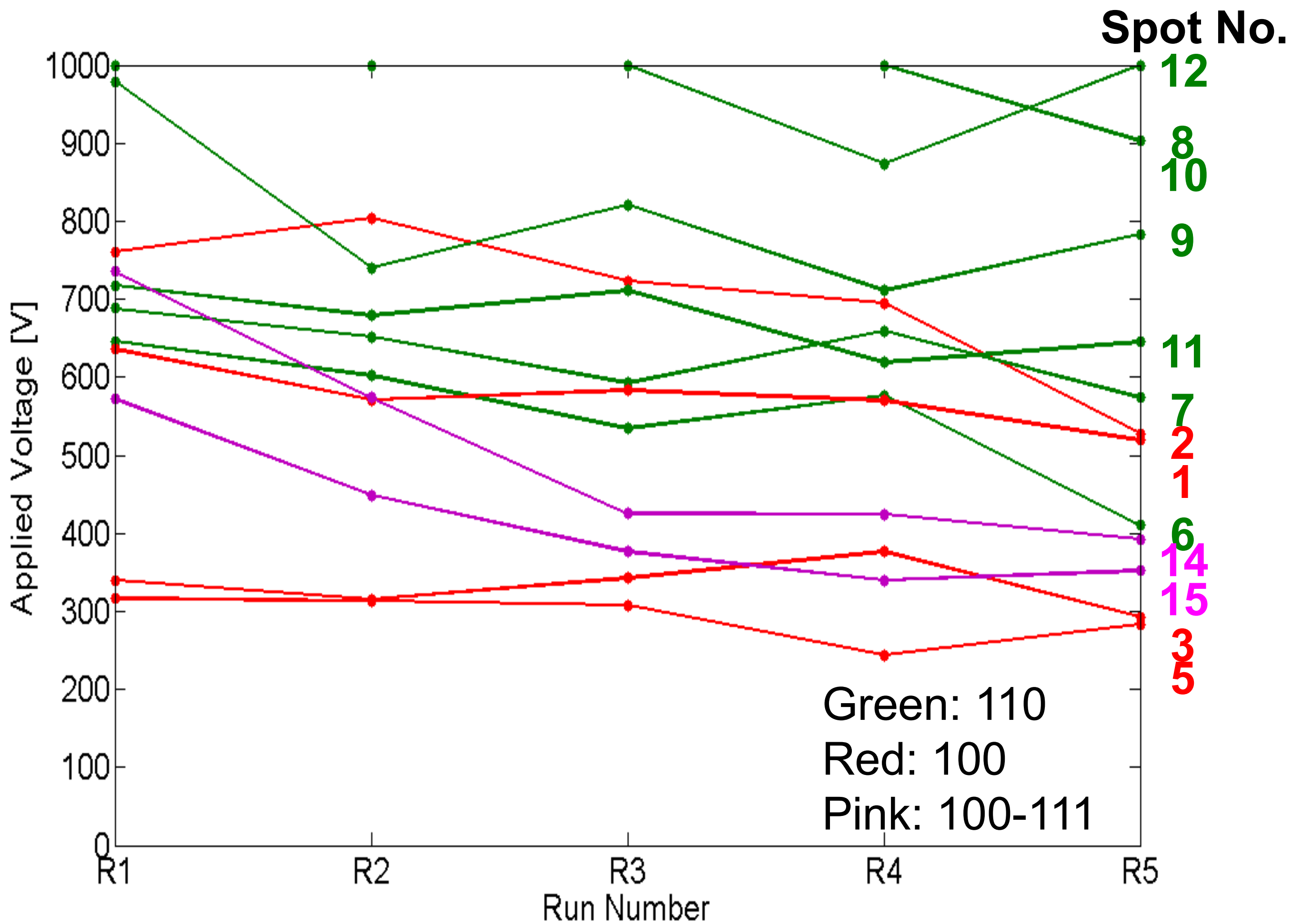
Grain orientations of measured area

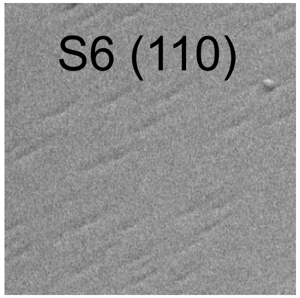


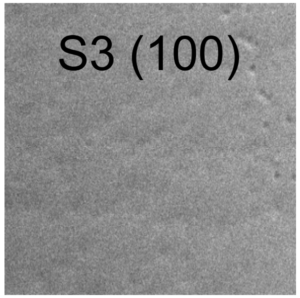
EBSD image: courtesy of M. Aicheler, CERN

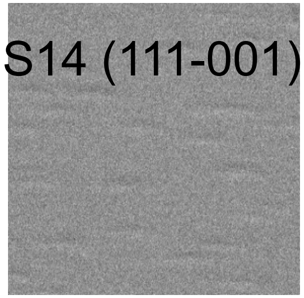


Grain orientation dependence









Summary

- The onset voltages where the measured current exceeded 10 nA were varied from 300 V to over 1000 V.
- The onset voltages were decreased at subsequent runs at most of spots (cf. Wuppertal's result).
- In order to study any dependences of emission behavior, more statistics are required.
- **We are now almost ready to perform local field emission measurements with piezo scanners.**

Co-workers

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Mechanical drawing and manufacture
Masih Noor
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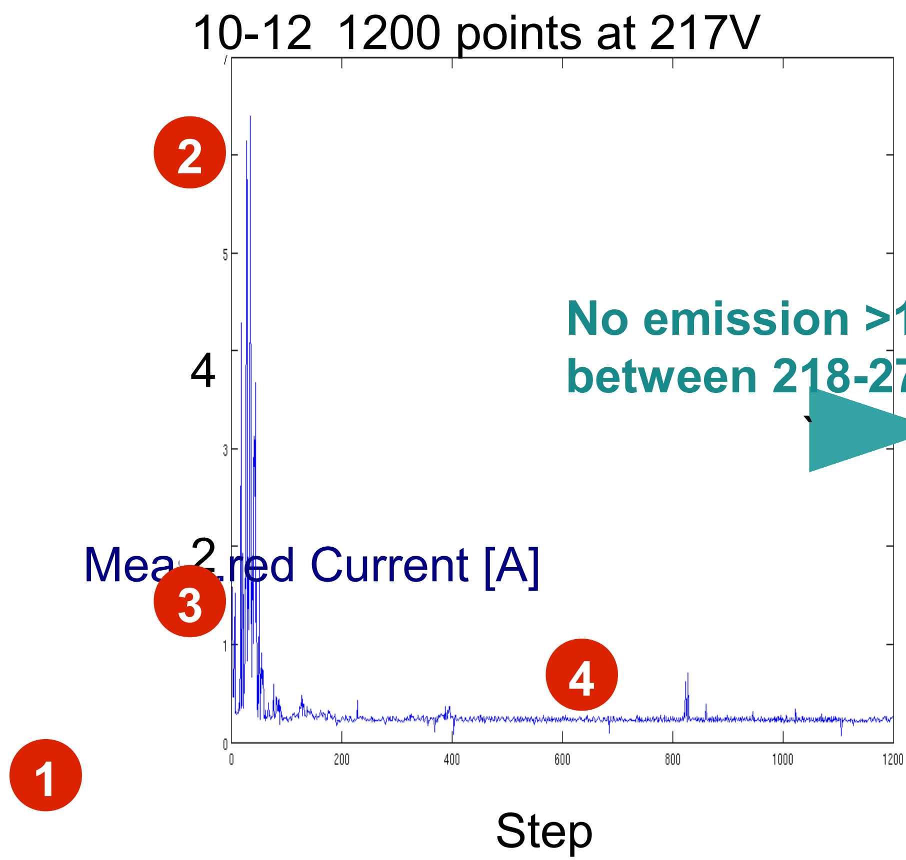
25 Feb. 2012, Abisko, Sweden

Backups

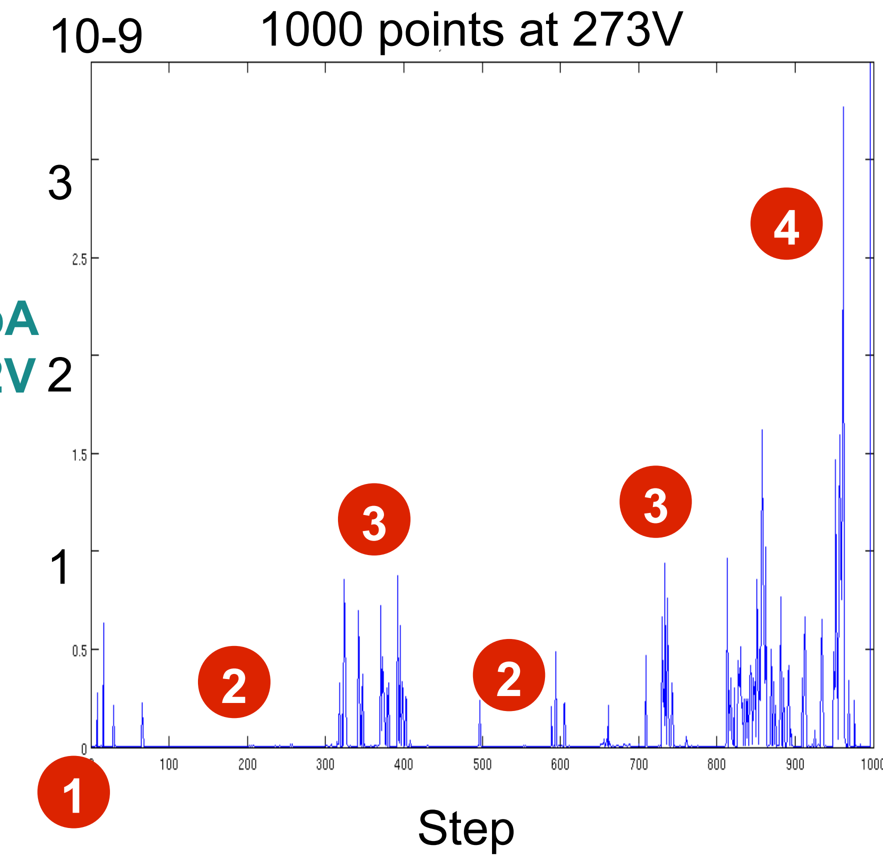
Emission stability measurement

1. Approach W tip to the sample surface ~1μm
2. Take SEM images of target area
3. Apply HV on W tip from 0V up to 1kV with 1V step
4. Measure emission current from Cu sample
5. Once the measured current exceeds 1pA, keep the voltage and continue current measurement for 20 minutes
6. Repeat 3-5
7. Stop measurement once the measured current exceeds 10nA.

Emission stability measurement



No emission >1pA between 218-272V

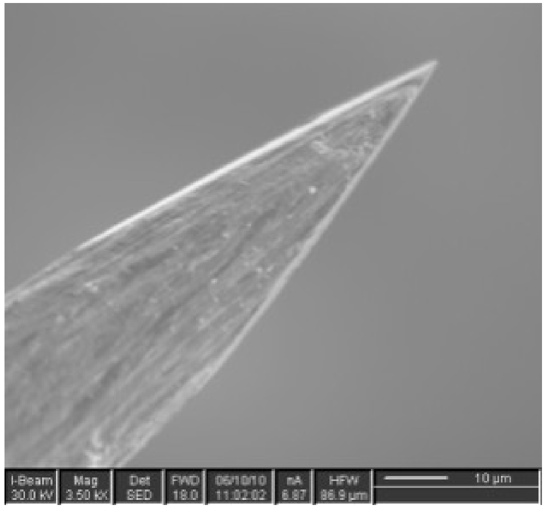


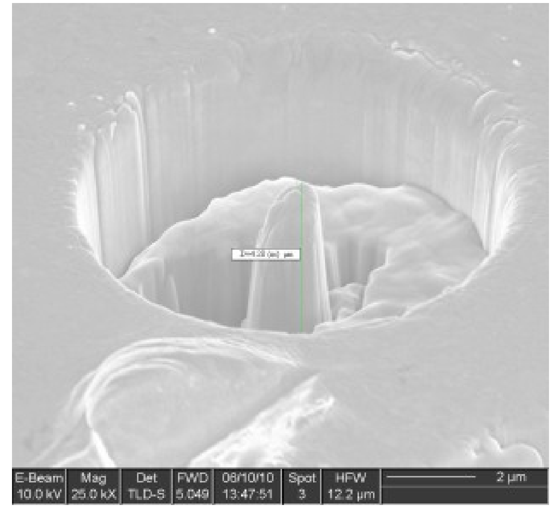
1. Measured current exceeded 1pA
2. Up to 6 pA
3. Decreased to the bg-level
4. Stayed at the bg-level

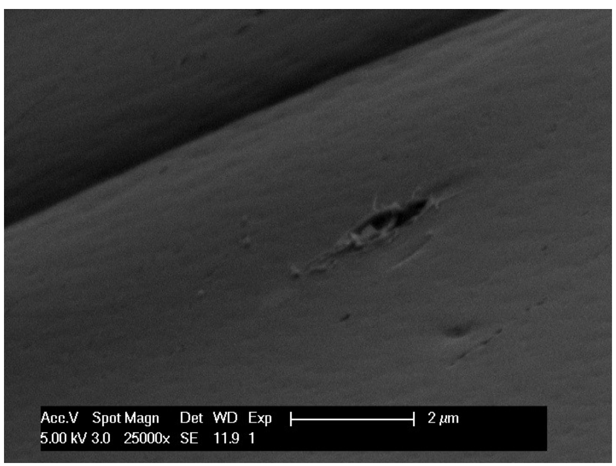
1. Measured current exceeded 1pA
2. Decreased to the bg-level
3. Spikes ~1nA
5. Emissions > nA then exceeded 10nA

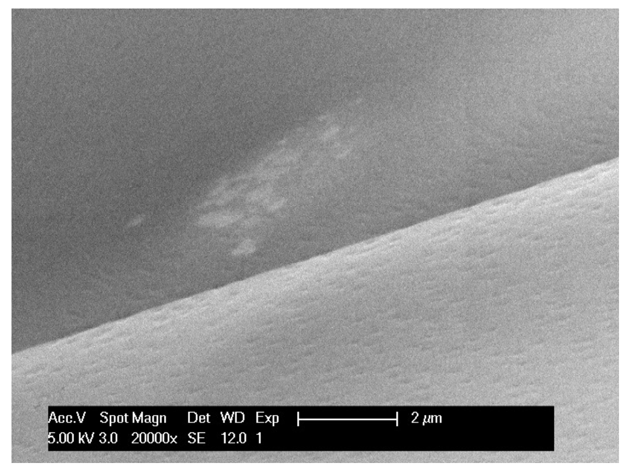
Summary: Emission stability

Spikes appeared and disappeared as if field emitters were growing and evaporating.
Still the tendency is that higher the E-field, higher the current.
Macroscopic emission current that follows Fowler-Nordheim might be an average of (unstable) local emissions.
The assumption above could be tested by statistical measurements and/or measurements with anodes of various sizes.


I-Beam 30.0 kV
Mag 3.50 kX
Det SED
FWD 18.0
11:02:02
nA 6.87
HFW 86.9 μm
10 μm

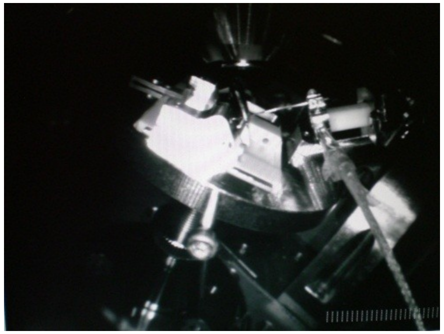

E-Beam 10.0 kV
Mag 25.0 kX
Det TLD-S
FWD 5.049
08/10/10 13:47:51
Spot 3
HFW 12.2 μm
2 μm


Acc.V Spot Magn Det WD Exp
5.00 kV 3.0 25000x SE 11.9 1
2 μm

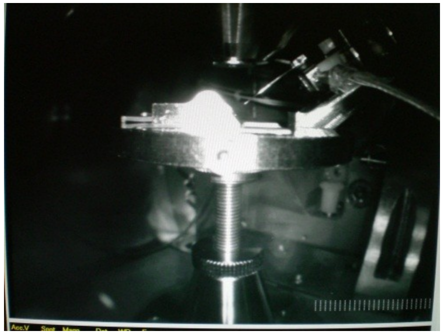

Acc.V Spot Magn Det WD Exp
5.00 kV 3.0 20000x SE 12.0 1
2 μm

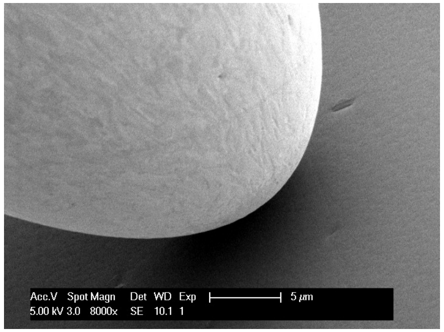
Gap determination

1. Find a target site by approaching the tip to the surface with the SEM stage tilted 30 degrees.



2. Take the target site surface images at 0 degree. Low magnification images are used for finding the position on the surface and high magnification images are for comparing surface condition before/after measurements. (the tip should be retracted in order to avoid shadows)





3. Set the tip on the measurement position. The stage is tilted 30 degrees and rotated to be in a plane perpendicular to the SEM detector.
4. Comparing with a reference image and a marked transparent sheet, the gap can be set in less than 10% accuracy.