Recent results from in situ field emission measurements in the SEM at Uppsala University

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Outline

- Ideas in Uppsala: mesoscopic view of breakdown and field emission phenomena
- Experiment
- Setup in an scanning electron microscope (SEM)
- Emission stability from local emission site
- Emission dependence on grain orientations
- Summary and outlook
- Other activities: using focused ion beam (FIB)

Micro-, Meso-scopic study in SEMs at Uppsala

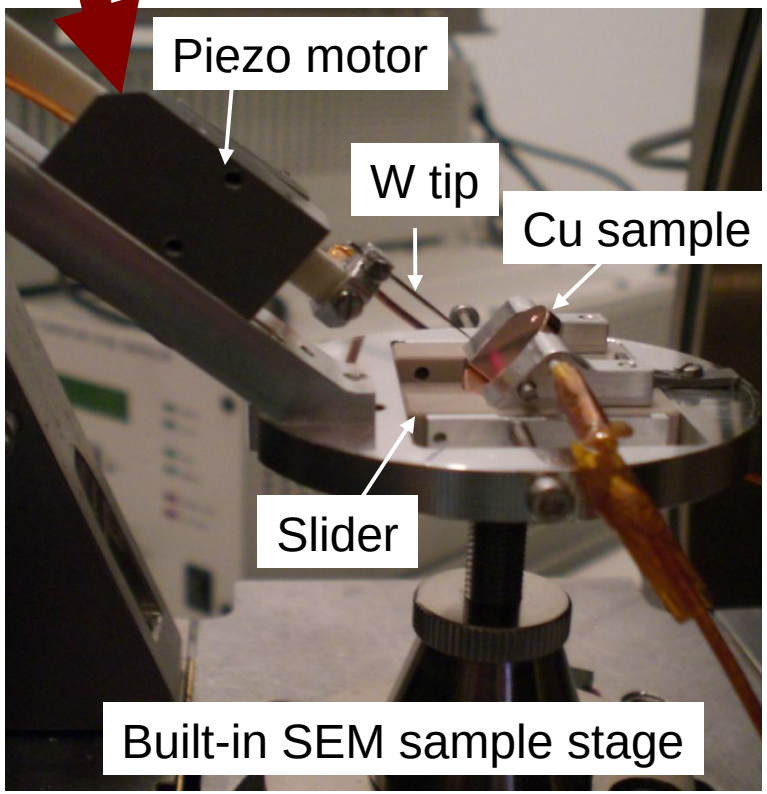
Scanning Electron Microscopes (SEM) open an opportunity to investigate local processes during breakdown

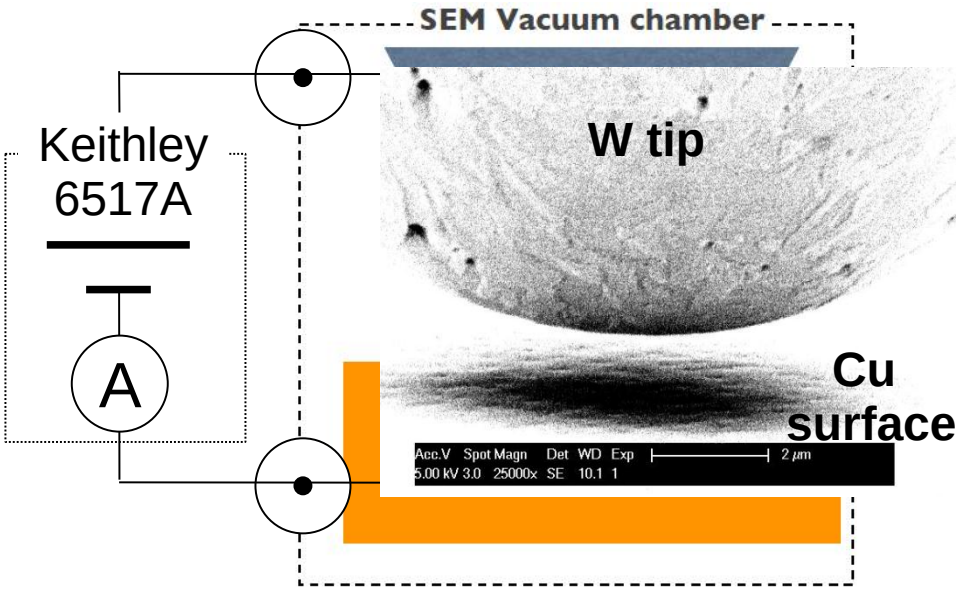
- Basic Idea is to reproduce high gradient electric field conditions in micro meter range: **100 MV/m = 100 V/µm**

- Localised BD phenomena can be measured and observed inside an SEM applying analysis techniques established in the field of material sciences

• Micro Structure Laboratory in the Angstrom Laboratory, Uppsala Univ. is one of Europe's most well equipped facilities for materials analysis with a wide range of equipment for surface imaging, atomic structure and composition analysis.

Experiment





Conditions of measurement:

- Cu samples provided by CERN, 12mm-D
- W tip commercially available, 5µm-R, cleaned by NaOH before measurements
- Gap accuracy 1.0 ± 0.1 µm
- Background current 0.2-0.3 pA

Local field emission measurement

Purposes:

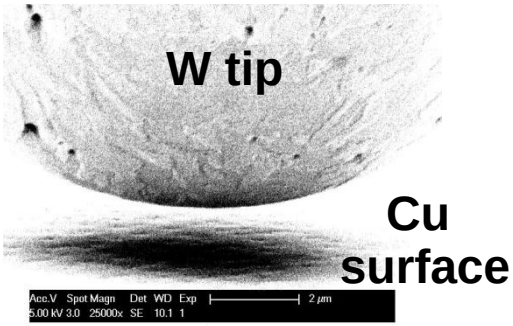
1. Property of pre-breakdown phase in (sub) μm range
   - **stability of electron emission**
- dependence on field, time, geometry
- surface modification due to emissions

2. “Weak point” of samples
- dependence on surface condition
- **effect of grain orientations**, boundary, size
- effect of surface treatment procedures

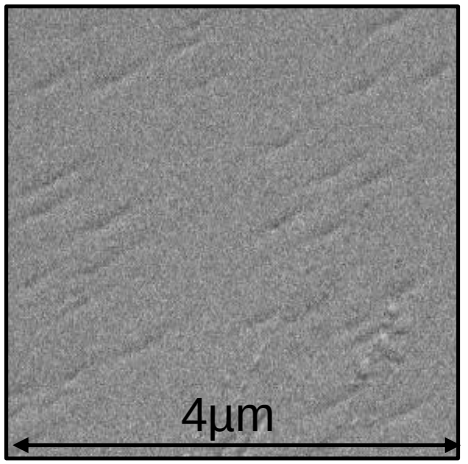
Emission stability measurement

Measurement Process

1. Approach W tip to the sample surface ~1μm



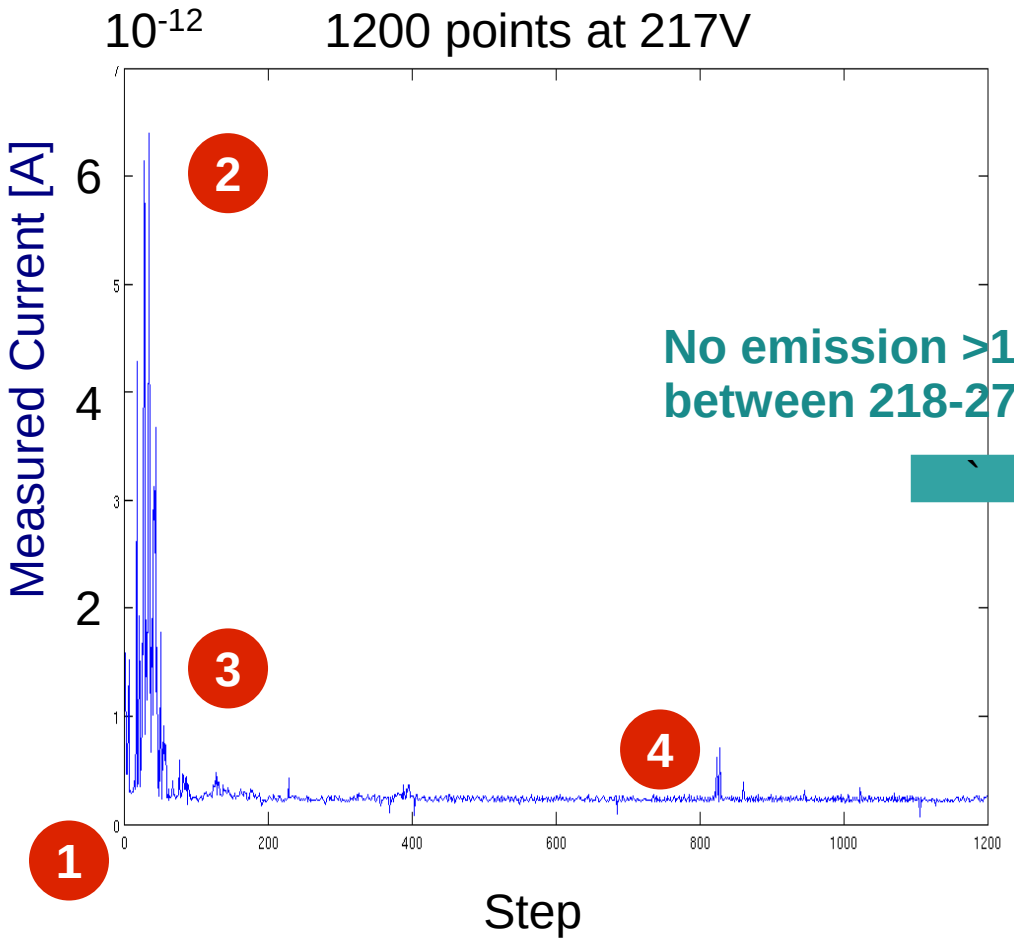
2. Take SEM images of target area



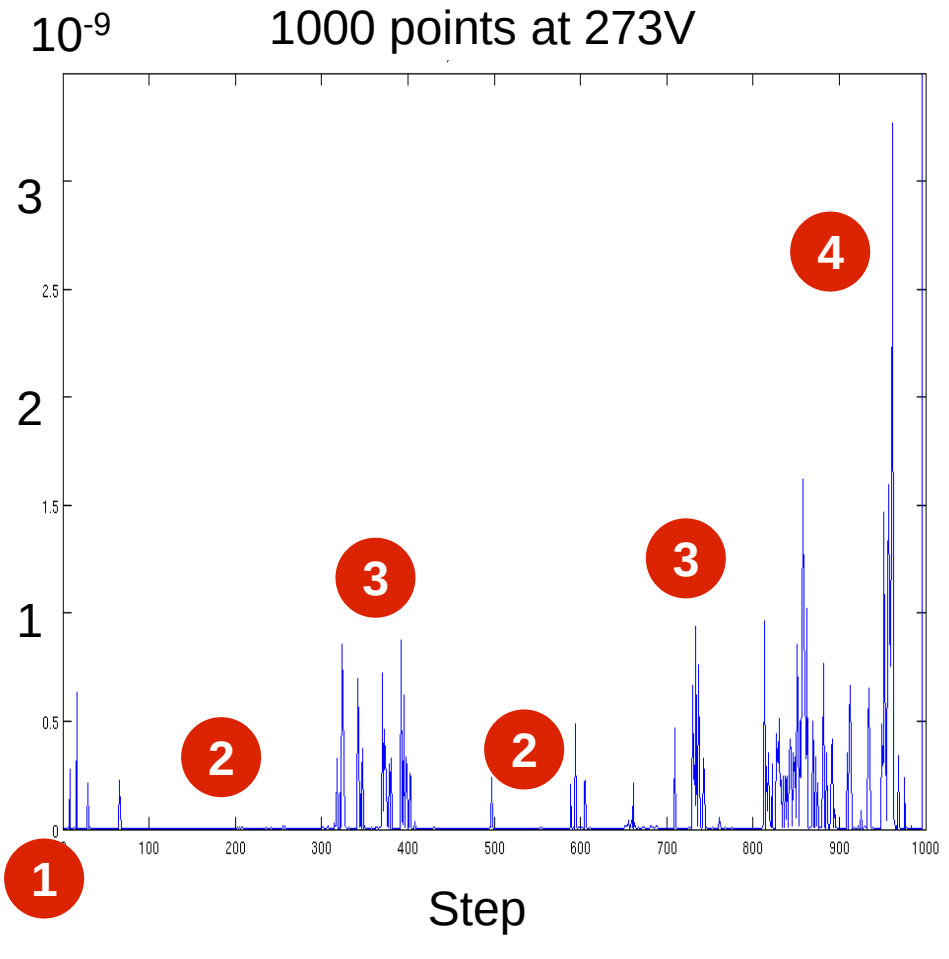
3. Apply HV on W tip from 0V up to 1kV with 1V step
4. Measure emission current from Cu sample

5. Once the measured current exceeds 1pA, keep the voltage and continue current measurement for 20 minutes
6. Repeat 3-5

Results 1: Emission stability



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

Discussion1: Emission stability

- Spikes appeared and disappeared: small (nm range) emitter growing and evaporating ?

- F-N would not be enough to explain what happens in the local emission site (In fact, even in macroscopic measurements “beautiful” F-N linear behavior is not always observed.)

- Still the tendency is that increasing the field, increasing the current (spikes are high and dense). → macroscopic emission current is an average of (unstable) local emissions ??

Local field emission measurement

Purposes:

1. Property of pre-breakdown phase in sub-μm range
   - **stability of electron emission**
- dependence on field, time, geometry
- surface modification due to emissions

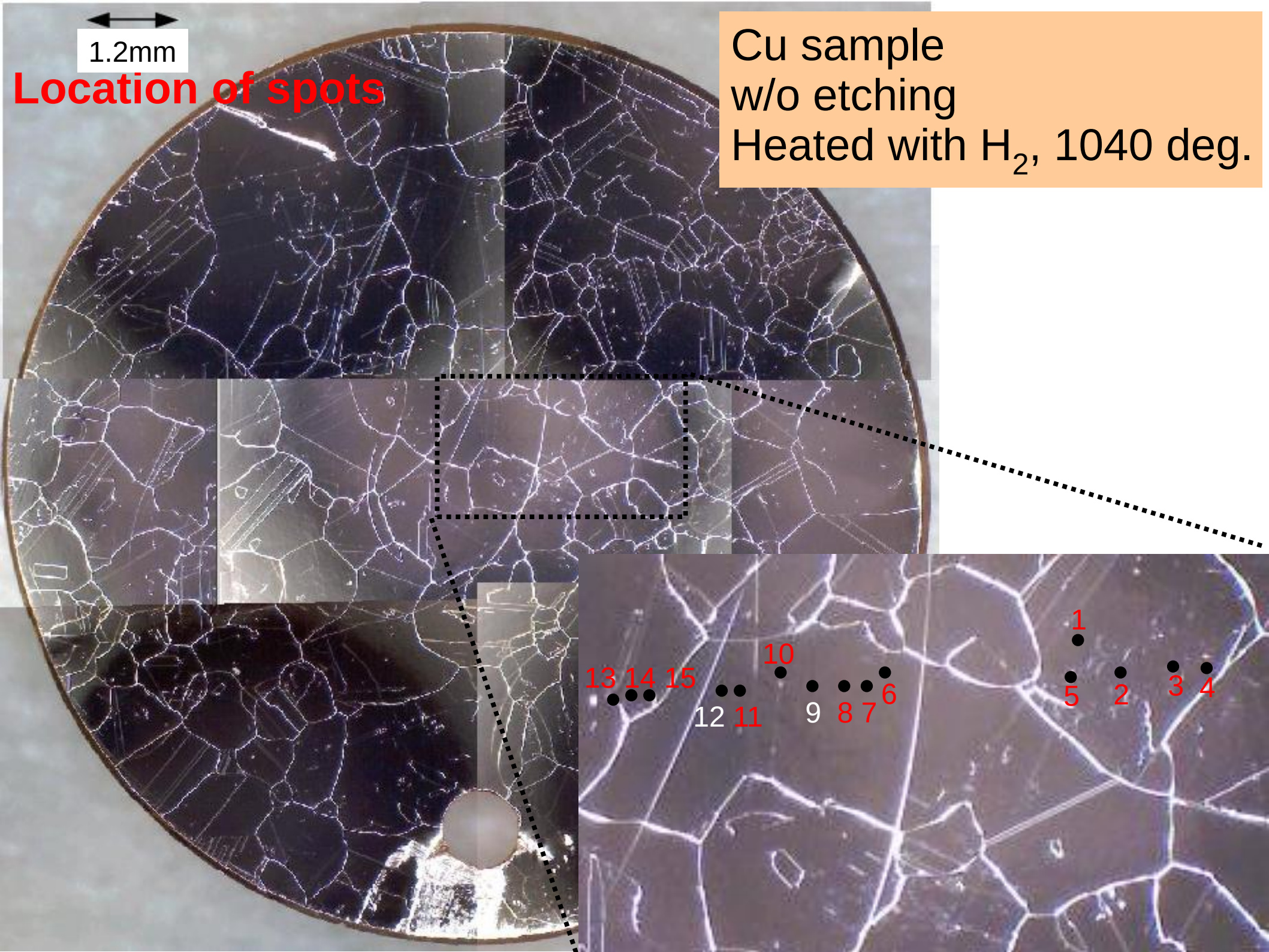
2. “Weak point” of samples
- dependence on surface condition
- **effect of grain orientations**, boundary, size
- effect of surface treatment procedures

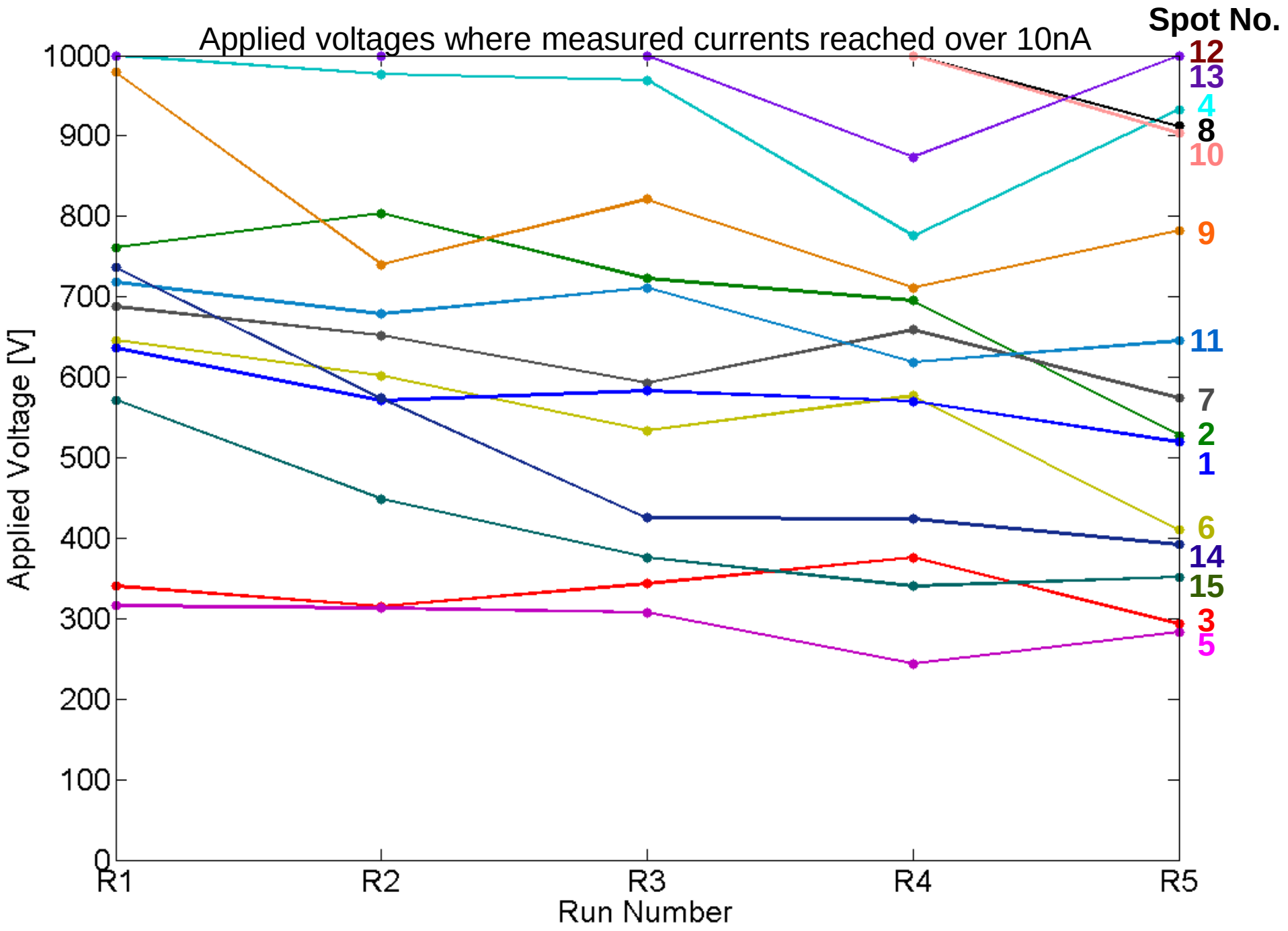
Variation in onset voltage

Measurement Process
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

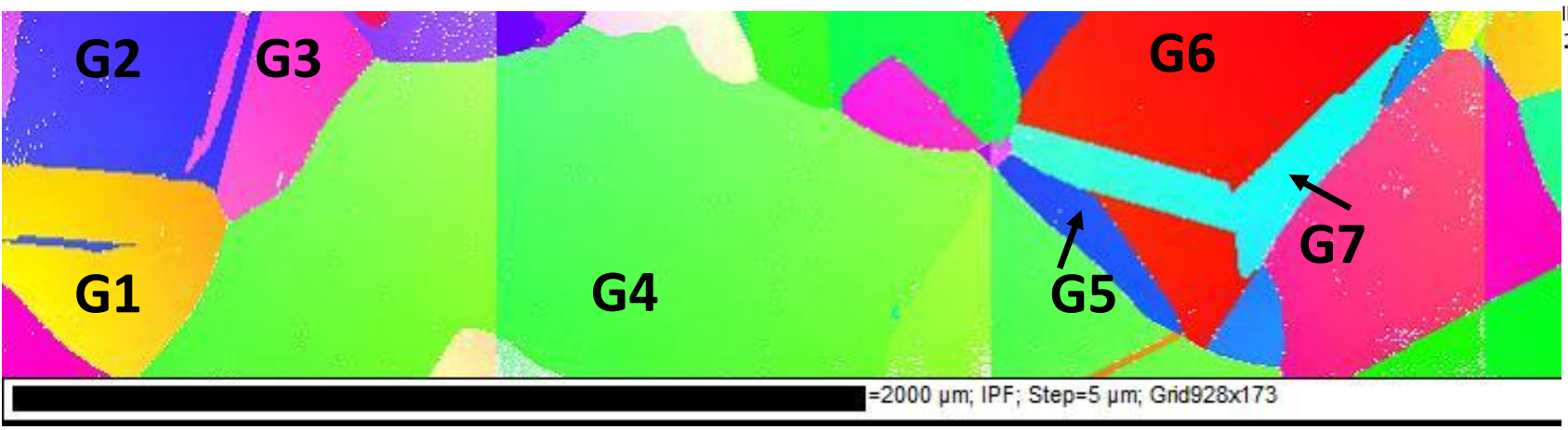
5. Stop HV supply once the current exceeds 10 nA (onset voltage)
6. Repeat 5 runs at same spot
7. Move sliders to the new spot (repeat 15 times)

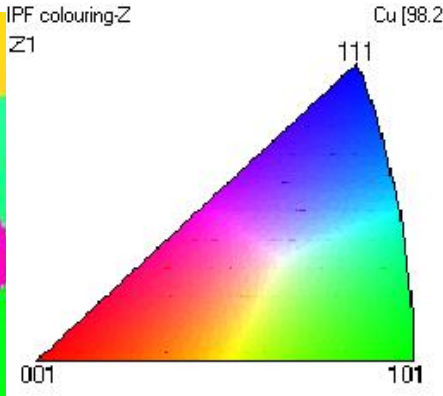
Sample: Cu, without etching, heated in H_2 atmosphere at 1040°C
Analyses: Comparison of onset voltage of each spots
Electron Backscatter Diffraction (EBSD) in order to check grain orientation dependence

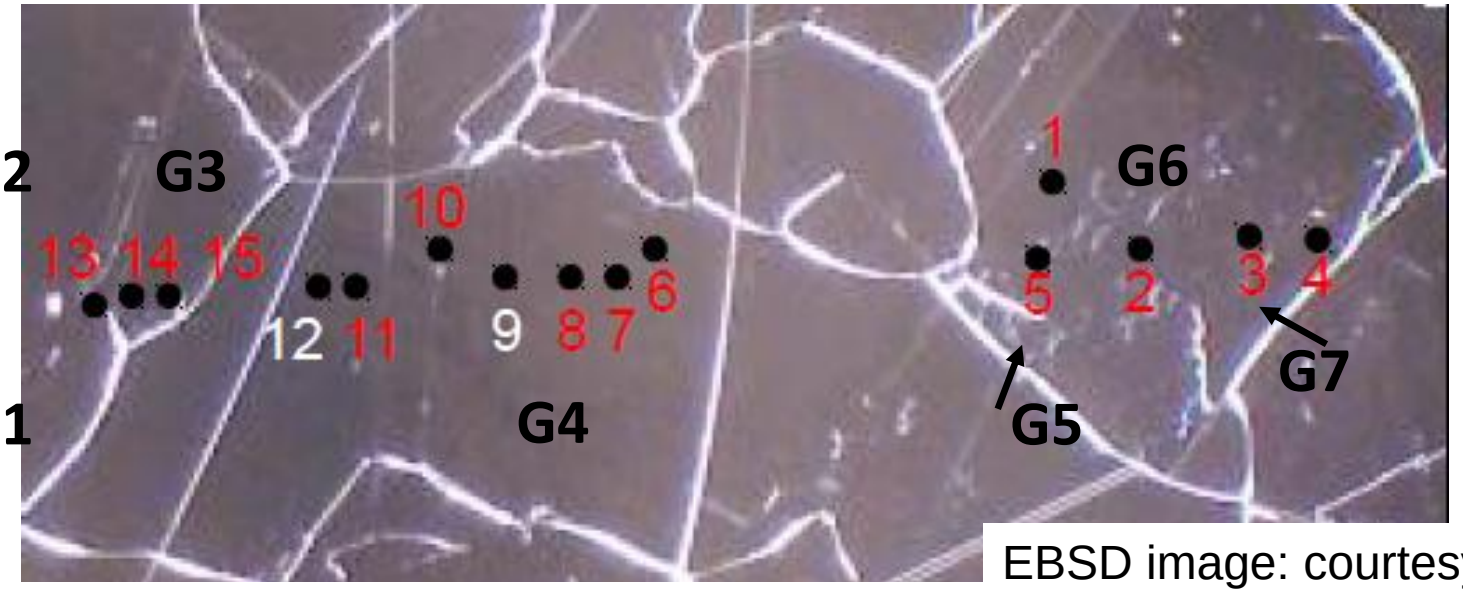

1.2mm
Location of spots
Cu sample
w/o etching
Heated with H_2, 1040 deg.
1
10
13 14 15
12 11
9 8 7 6
5 2 3 4


Applied voltages where measured currents reached over 10nA
Spot No.
12
13
4
8
10
9
11
7
2
1
6
14
15
3
5
Applied Voltage [V]
1000
900
800
700
600
500
400
300
200
100
0
R1
R2
R3
R4
R5
Run Number

Grain orientations of measured area



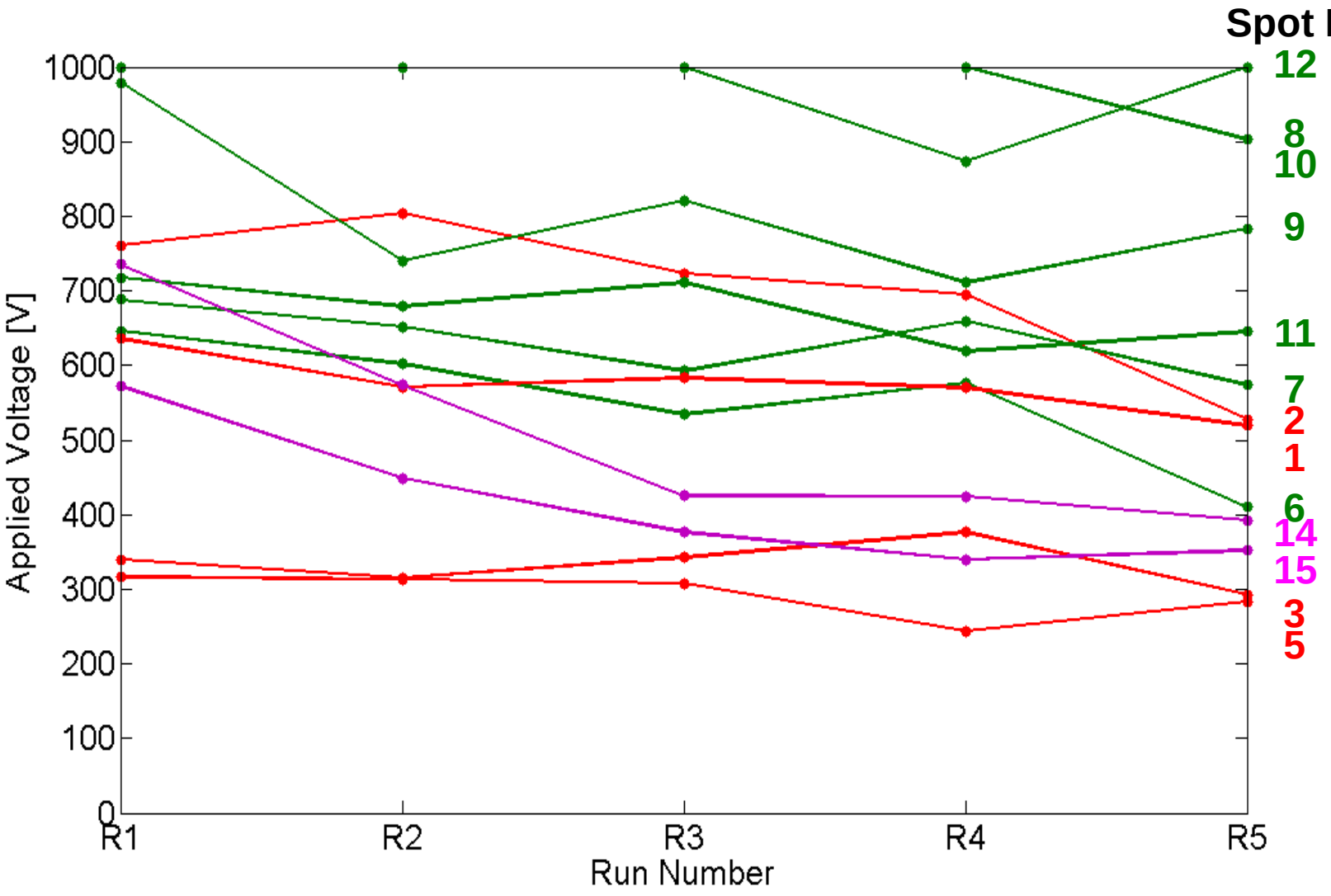




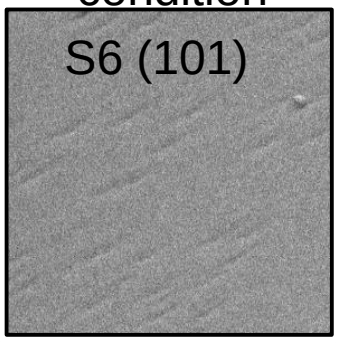
EBSD image: courtesy of M. Aicheler, CERN

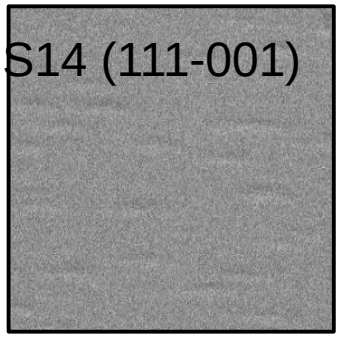
Grain	G2	G3	G4	G6	G7
Spot	13	14, 15	6-12	1-3,5	4

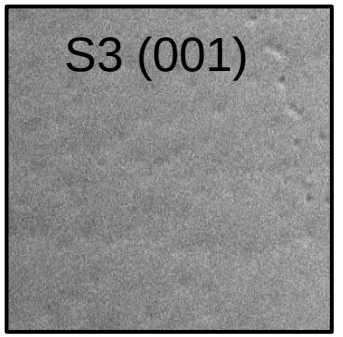
Grain orientation dependence



Surface condition







Colour	Grain	Spot	V_{on_R1}	V_{on_R5}
Red	001	1-3,5	300-750	300-520
Green	101	6-12	650->1000	410-1000
Pink	111-001	14,15	580-730	360-400

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Discussion2: variation in onset voltage

- The onset voltages where the measured current exceeded 10 nA were quite variable from 300 V to over 1000 V.

- The onset voltages decreased at subsequent runs at most of spots

- Grain boundary dependence of onset voltage was investigated.
  - Spread at 101 surface was larger than others.
  - Average V_{onset} didn't correlate to the work function of each grain orientations.

Grain	V_{on_av}	Work function
001	410V	4.430eV
101	750V	4.375eV
111	-	4.878eV

- More statistics is required (we'll order soon a 3D stage for the sample for remotely controlled scanning!)

Summary and outlook

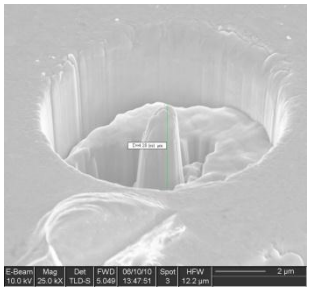
- We are now finally able to perform local field emission measurement, because we have - SEM in situ setup
  - gap determination method
- Experimental results shows that local emission behavior couldn't be completely described by F-N equation.
- The emission behavior are quite variable. In order to study any dependence of emission behavior, more statistics are required.

What to do next

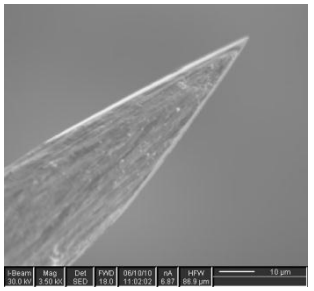
- Surface analyses (grain orientation, grain size, nano size degradation measurements etc) should be performed before measurements.
- More statistics for more samples in order to define “week points” of the ACS.
- Remotely controlled sample stage is strongly required.

Other activities using Focused Ion Beam (FIB)

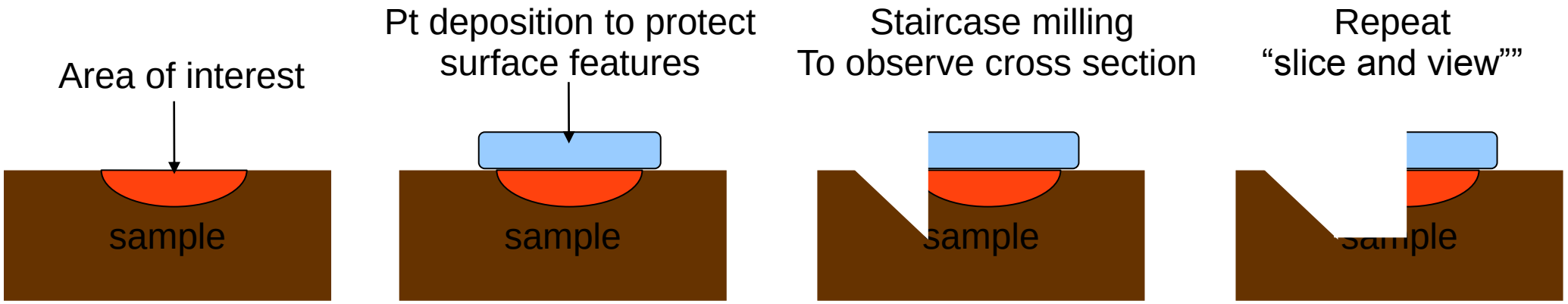
- FIB combined with high-res SEM provides
- surface modification (“milling”)



- tip polishing



- cross section observation (surface damage analysis, sample characterisation, see movies)



Co-workers

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Mechanical drawing and manufacture
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Lars-Erik Lindquist

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Back-up

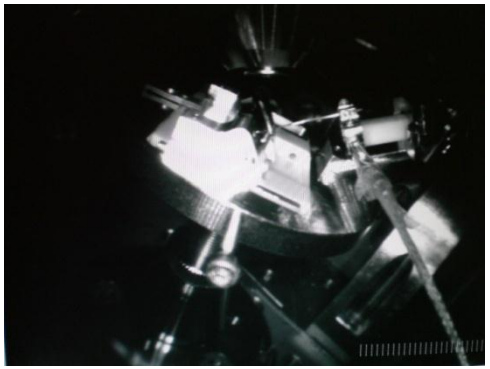
Gap determination (critical parameter!)

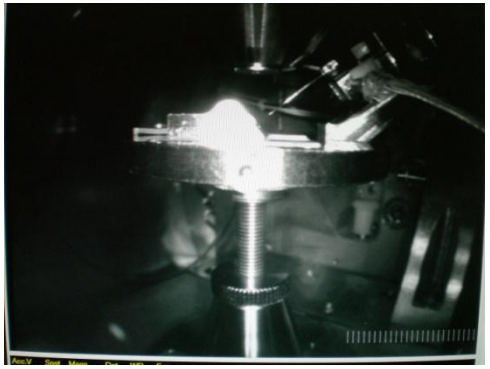
1.Find a target site by approaching the tip to the surface with the SEM stage tilted 30 degrees.

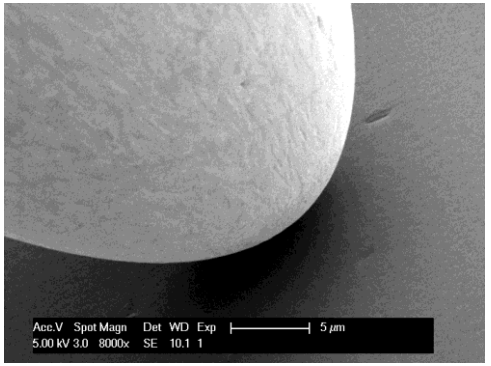
2.Take the target site surface images at 0 degree. Low magnification images are used for finding the position on the surface and high magnification images are for comparing surface condition before/after measurements. (the tip should be retracted in order to avoid shadows)

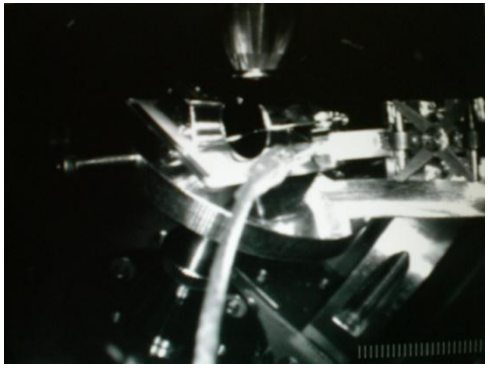
3.Set the tip on the measurement position. The stage is tilted 30 degrees and rotated to be in a plane perpendicular to the SEM detector.

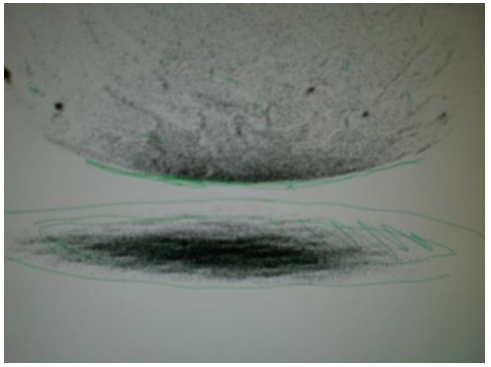
4. Comparing with a reference image and a marked transparent sheet, the gap can be set in less than 10% accuracy.



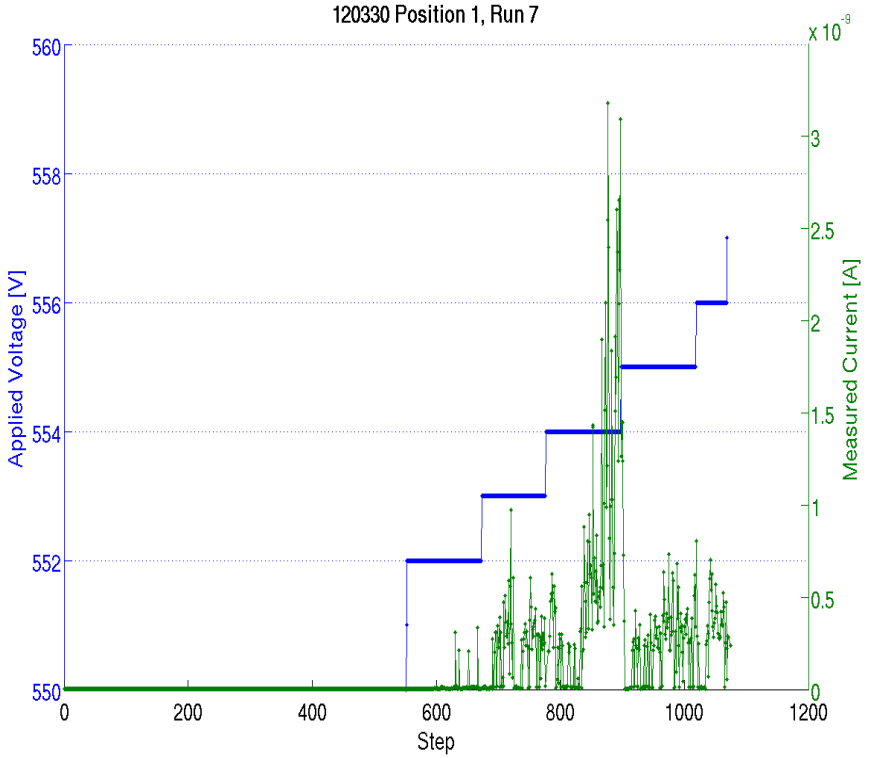


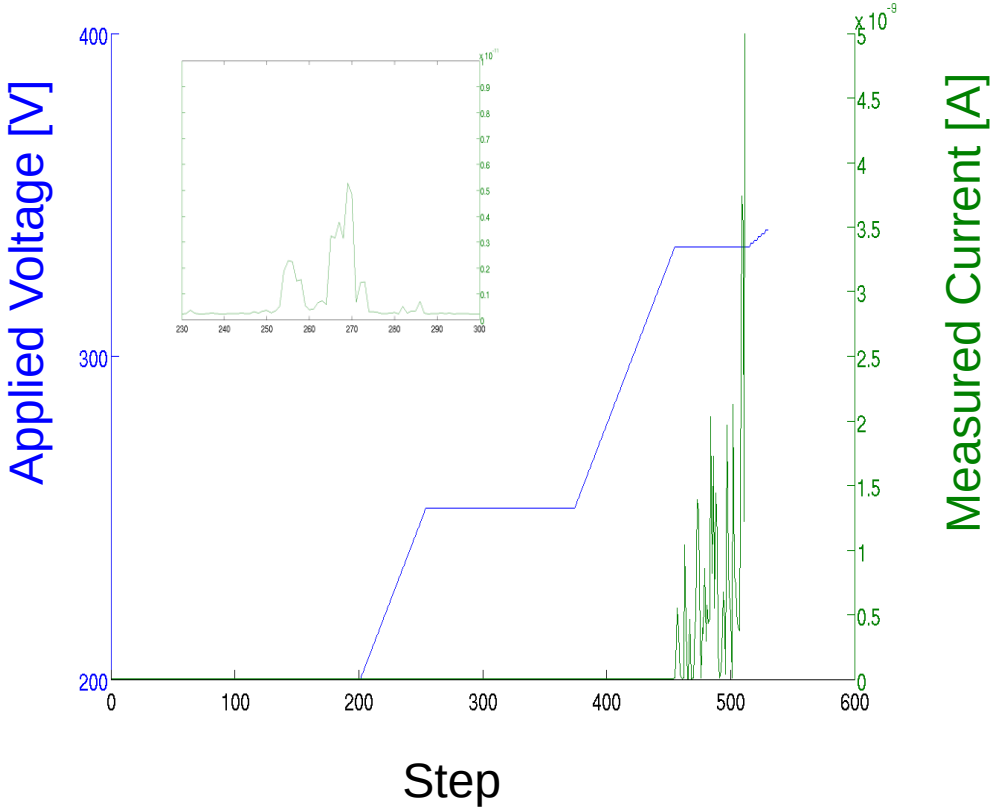




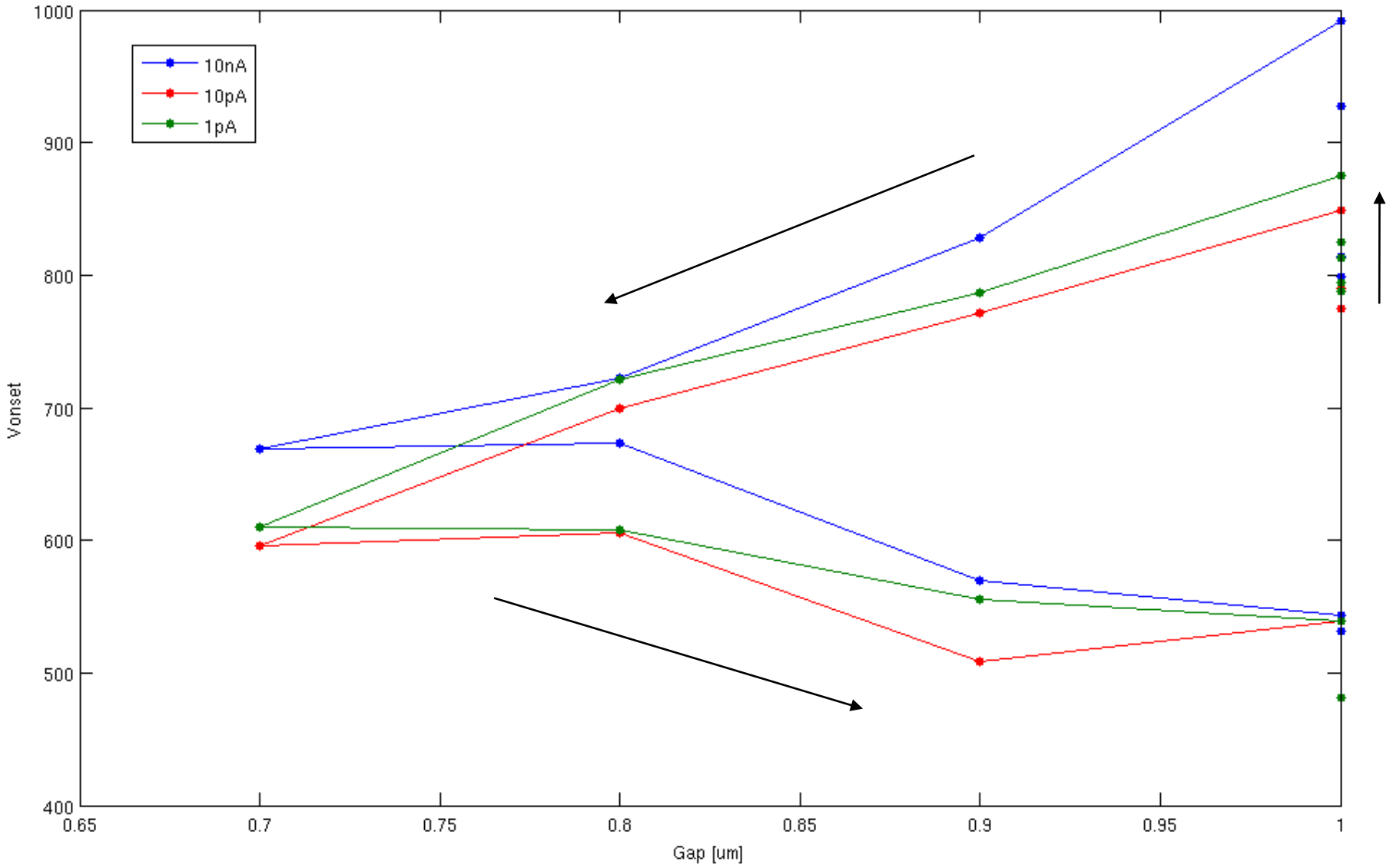


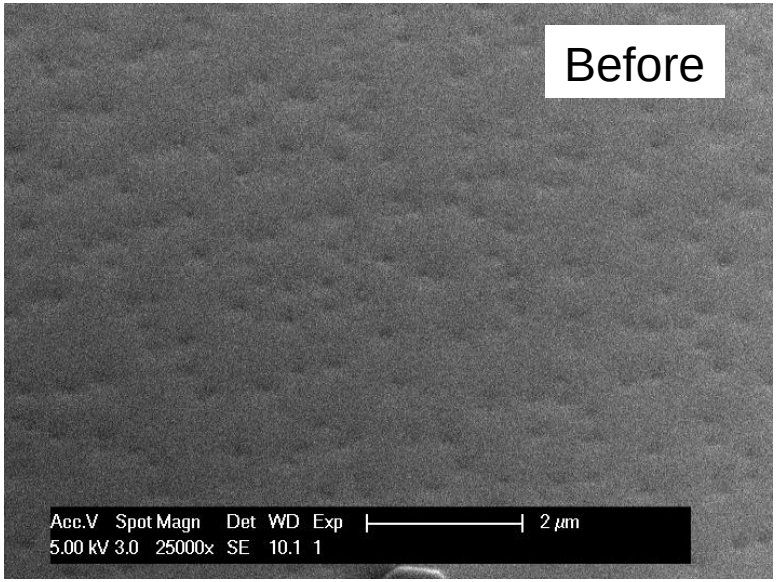
Results 2: Emission stability

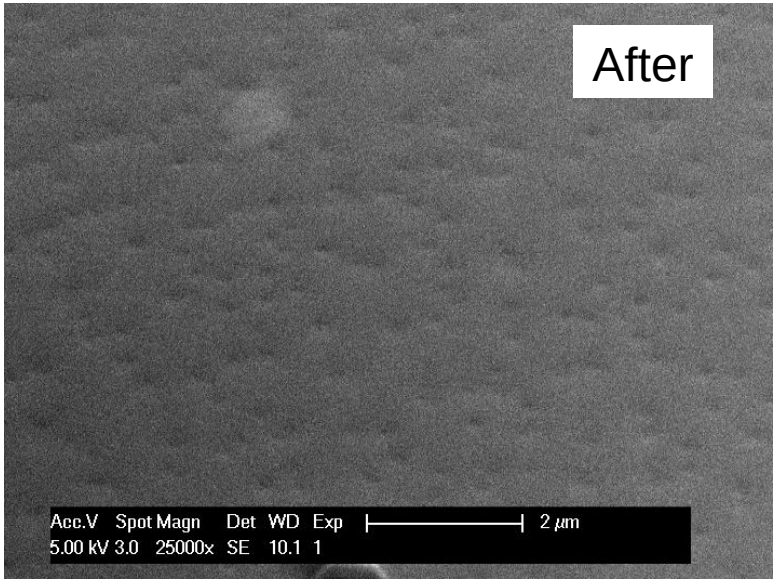




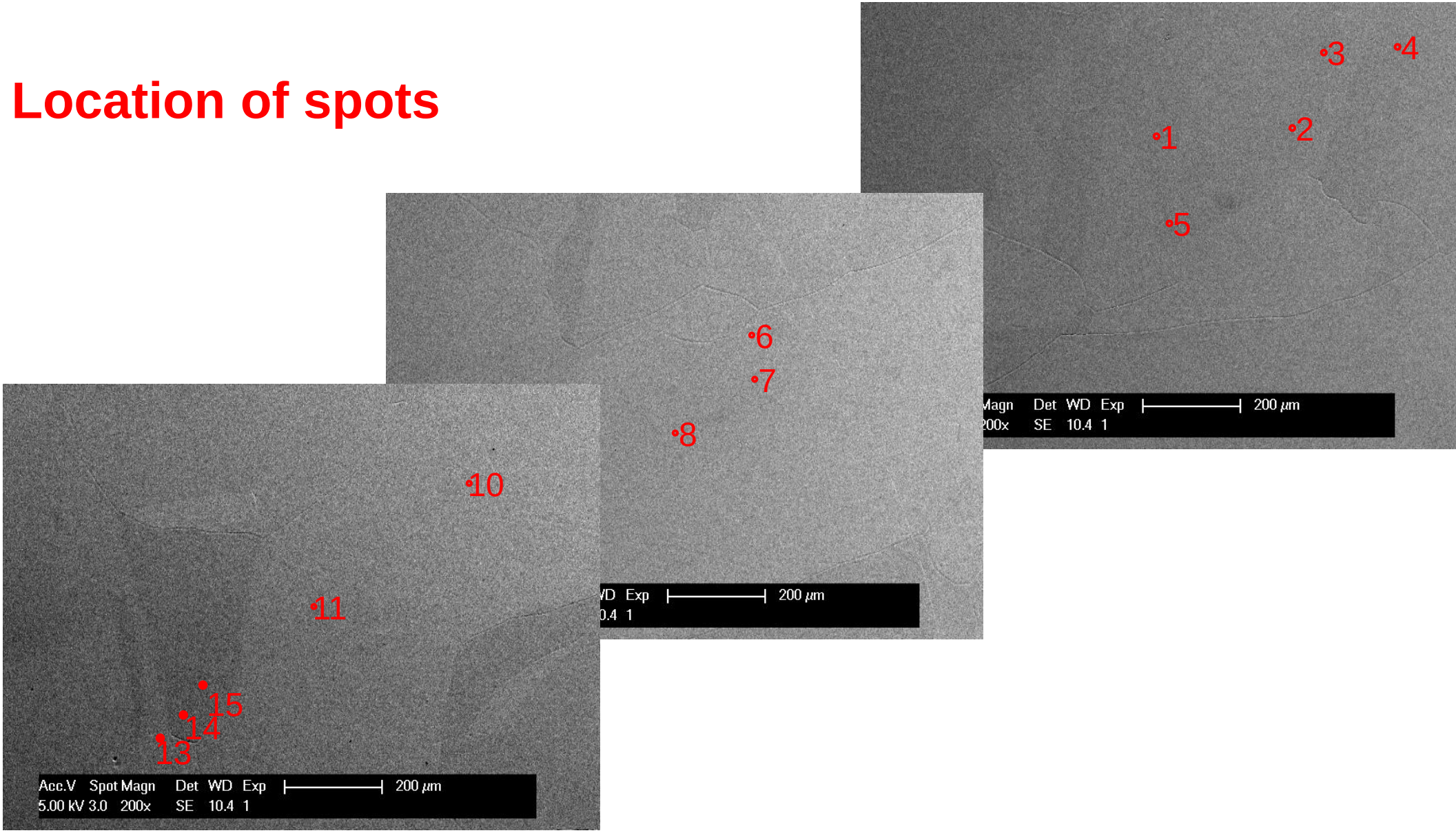
Results 2: Field dependence



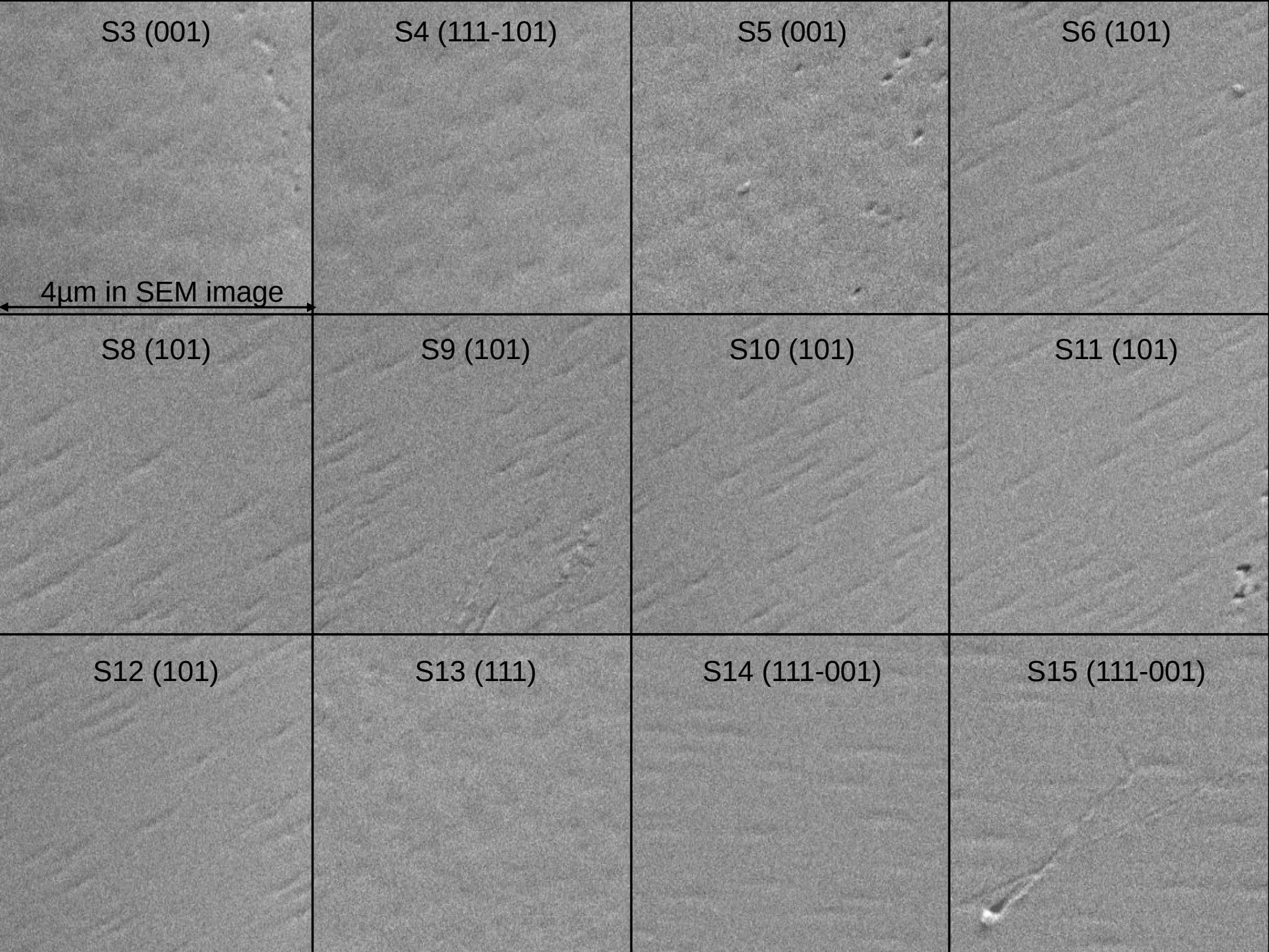

Before
Acc.V Spot Magn Det WD Exp 2 μm
5.00 kV 3.0 25000x SE 10.1 1


After
Acc.V Spot Magn Det WD Exp 2 μm
5.00 kV 3.0 25000x SE 10.1 1

Location of spots



Contrasts on SEM images indicates that surface conditions are varied.


S3 (001)
S4 (111-101)
S5 (001)
S6 (101)
4μm in SEM image
S8 (101)
S9 (101)
S10 (101)
S11 (101)
S12 (101)
S13 (111)
S14 (111-001)
S15 (111-001)

What's happening: Field emission from copper

"Typical" field emission properties are seen in figures below. Those are described in Fowler-Nordheim equation which gives field emission area and field enhancement factor.

Is that still true in a local site?