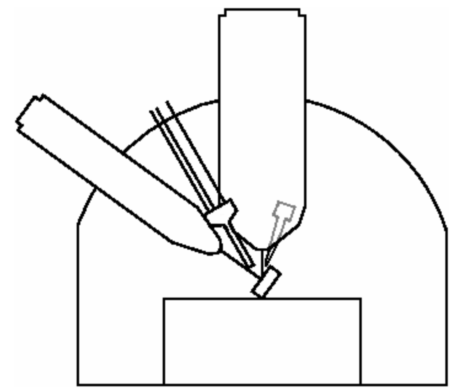
ETH
Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

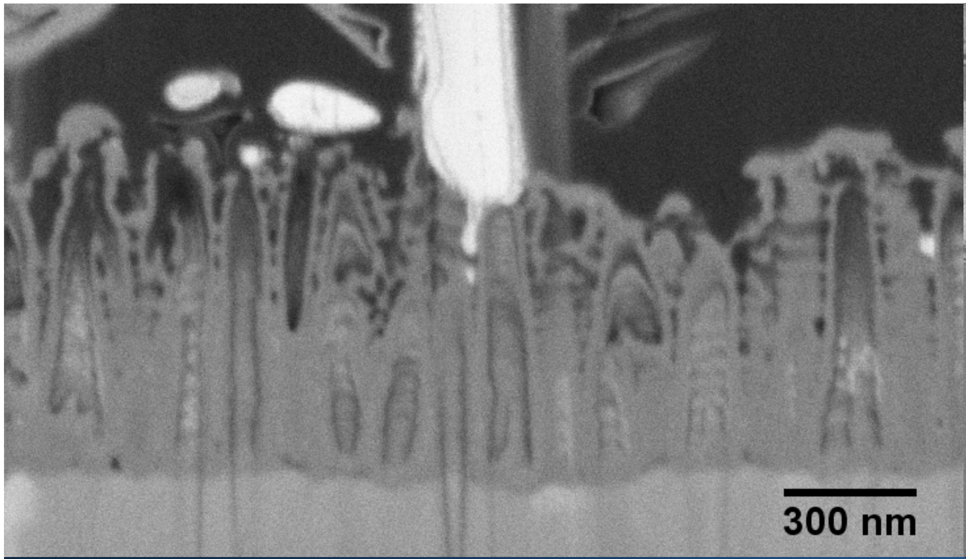
DMATL
Department of Materials

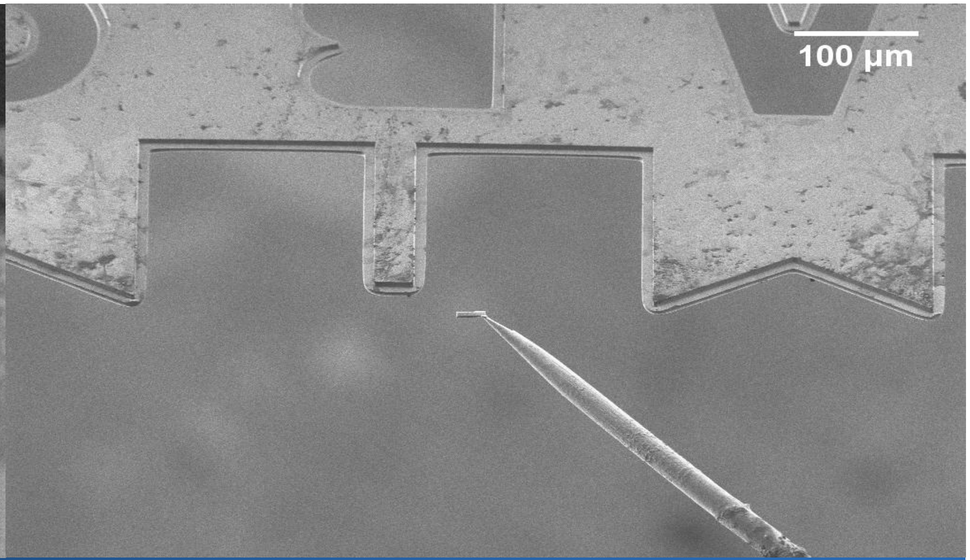
Introduction to FIB-SEM



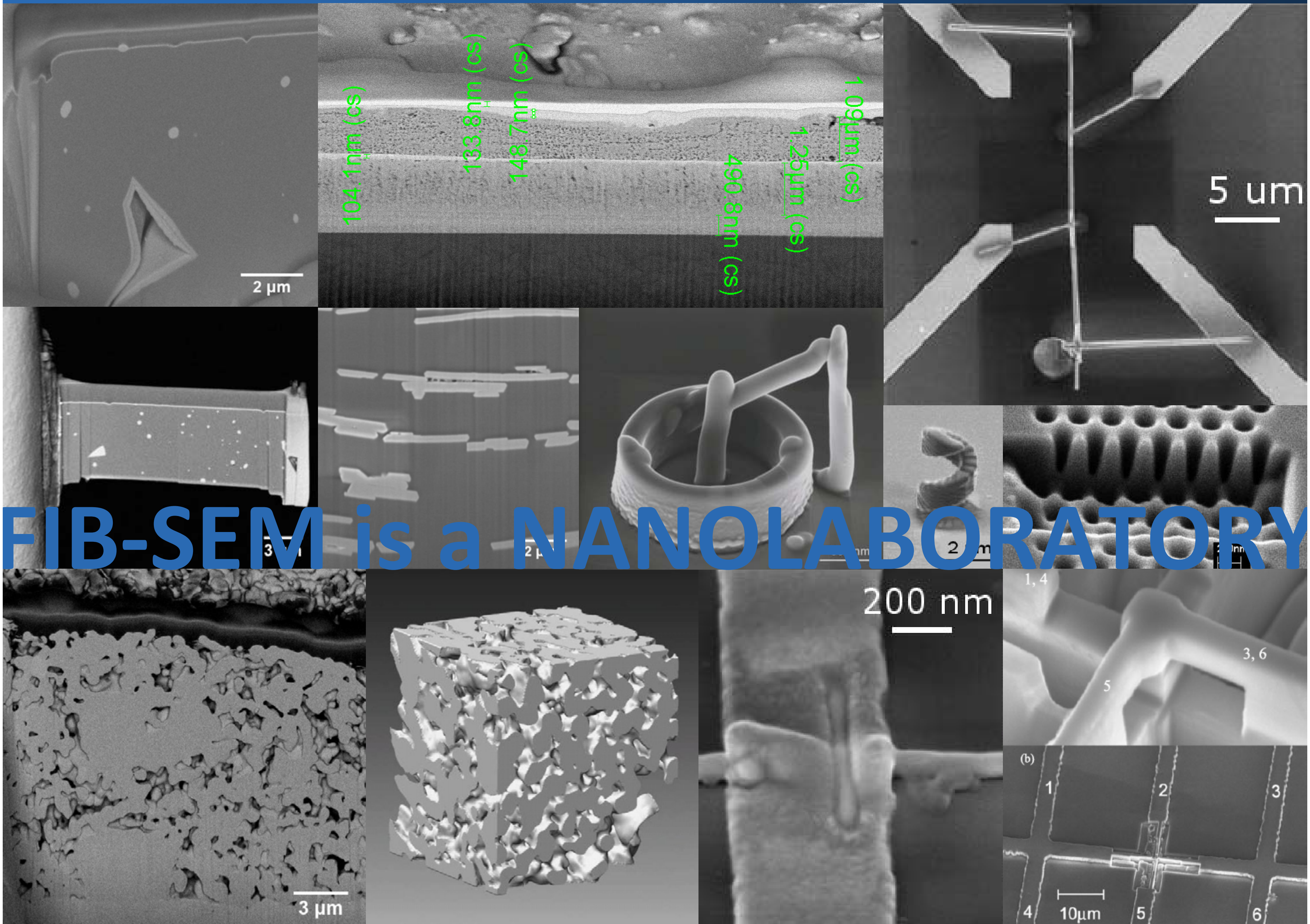
Basic Physics and Applications

Joakim Reuteler, Nonmetallic Inorganic Materials

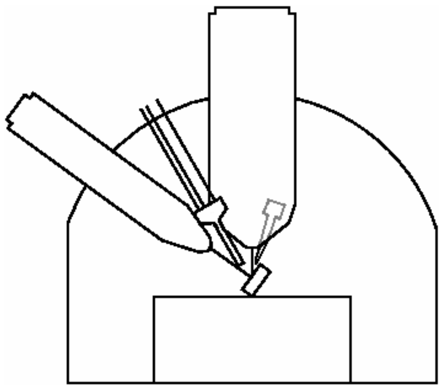




FIB-SEM is a NANOLABORATORY



Outline



1. Ga^+ source: LMIS
2. A combined microscope
   2.1. Focused Ion Beam (FIB)
   2.2. Scanning Electron Microscope (SEM)
   2.3. CrossBeam® NVision 40 from Carl Zeiss SMT
3. Micromanipulator MM3A from Kleindiek
4. Applications overview
   4.1. Cross Section (CS) – the basic skill!
   4.2. TEM lamella preparation
   4.3. Deposition and Milling with "feature mill"
5. Outlook and Literature

1. Ga^+ source: LMIS

Gilbert (1600)

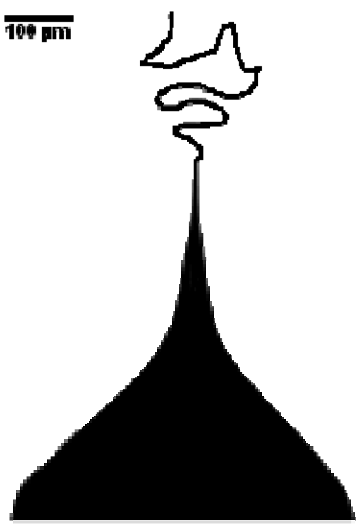
- fluid under high tension forms a cone → Gilbert cone
- even a thin thread can be sprayed out (jet)

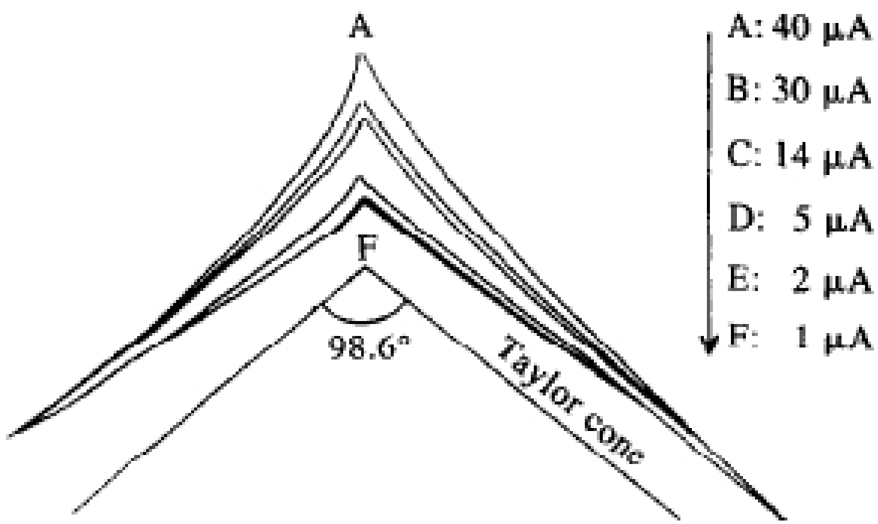
Taylor (1964)

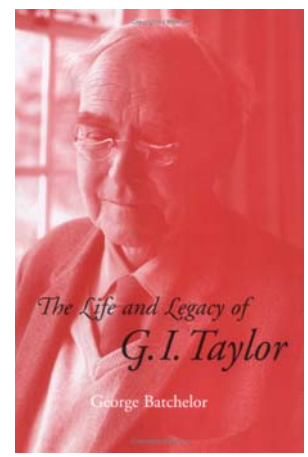
- exactly conical solution to equations of Electro Hydro Dynamics (EHD) → Taylor cone
- experimentally confirmed the conical shape

→ Taylor-Gilbert cone

Remark.
MRC Kolloquium on Dec 19, 2007
A.L. Yarin: "Electrospraying of Nanofibres and Nanotubes"
→ Gilbert was the scientist (and probably lover of) Queen Elisabeth I, she was very fond of physical phenomena.



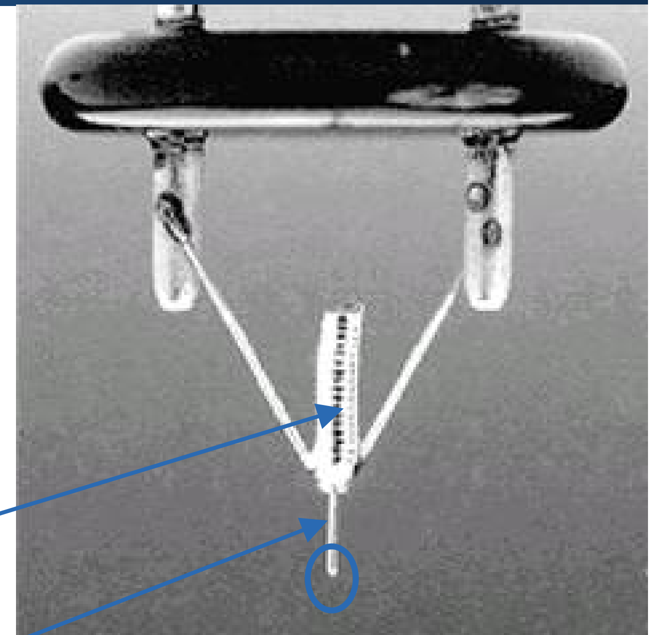




1. Ga^+ source: LMIS

Liquid Metal Ion Source (LMIS)

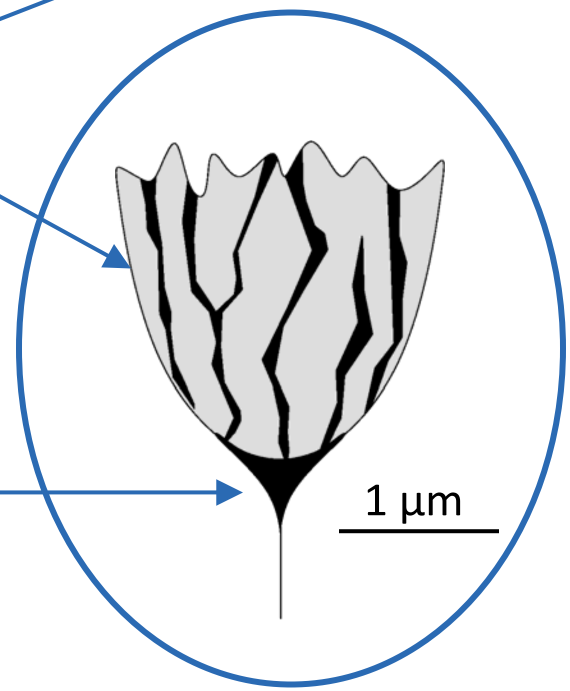
- Ga beam formed at the apex of Taylor-Gilbert cone
  → very high spacial coherence, i.e. focussable beam!



coil for heating, also serves as Ga reservoir

blunt W with grated surface for Ga transport

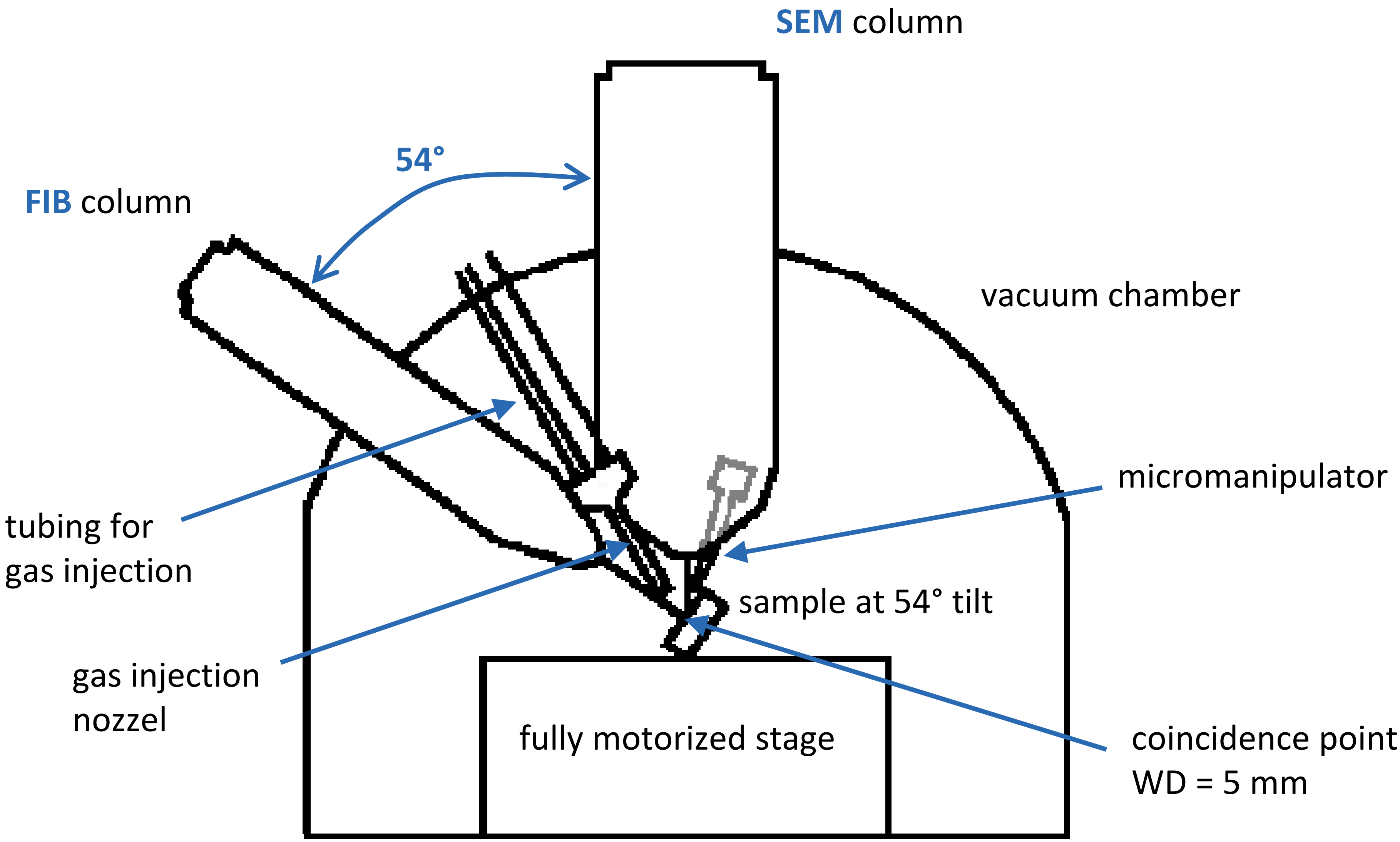
Ga forms a Taylor-Gilbert cone



Why Ga?

- melting point at 30 °C
  → liquid around room temperature
- low steam pressure
  → appliccable in HV
- $[Ga^{2+}]/[Ga^+] \sim 10^{-4}$ at 10 µm
  → narrow energy distribution

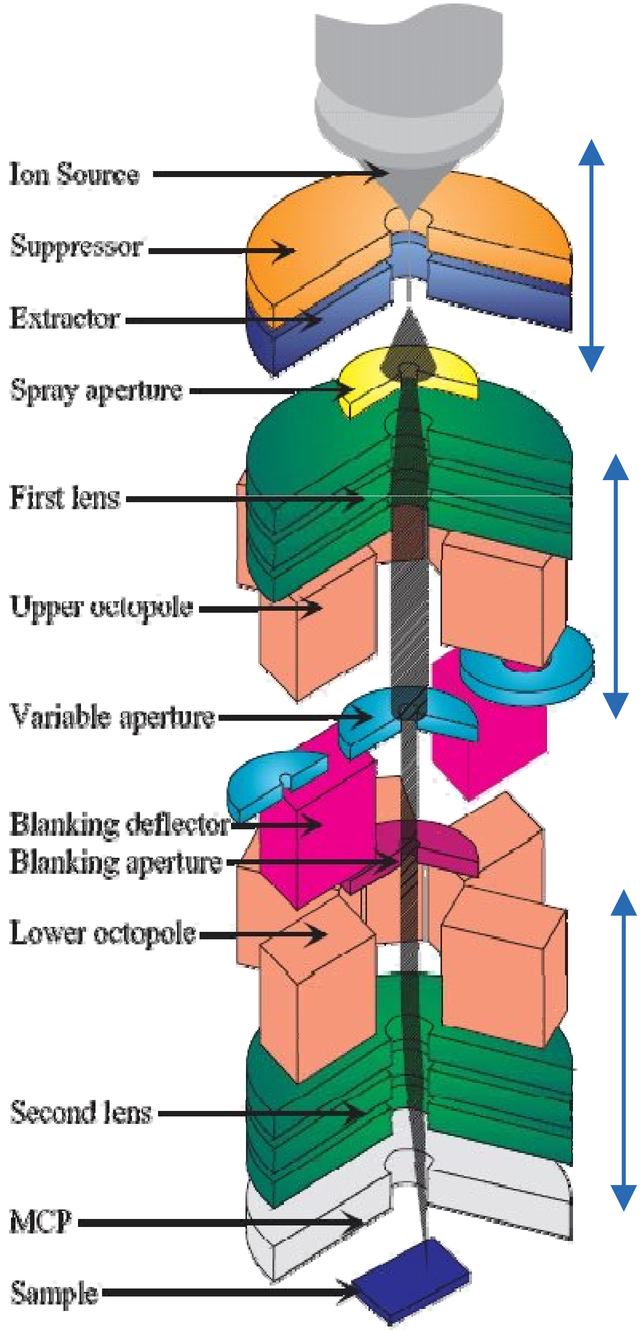
2. A combined microscope



2.1. Focused Ion Beam (FIB)

Ion column

- Ga^+ source (LMIS)
- ion optics (electrostatic lenses)
- fast beam blanker (electrostatic)
- different currents (aperture stripe)
- adjustable acceleration voltage

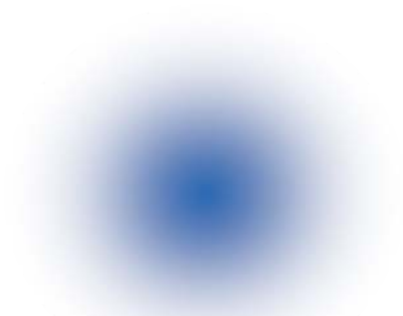


generation of beam (2 µA)

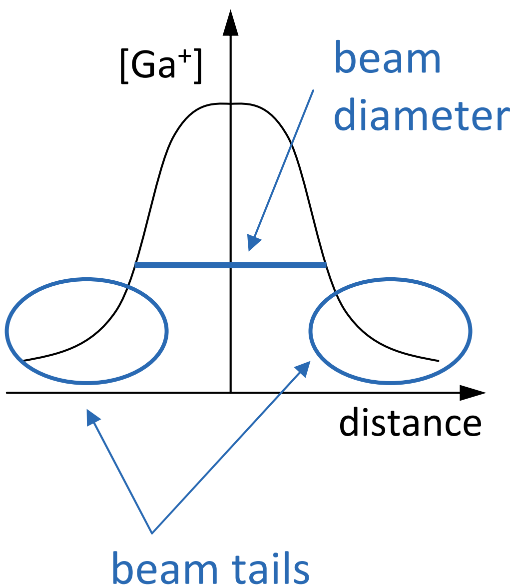
adjusting the current (typically pA to nA)

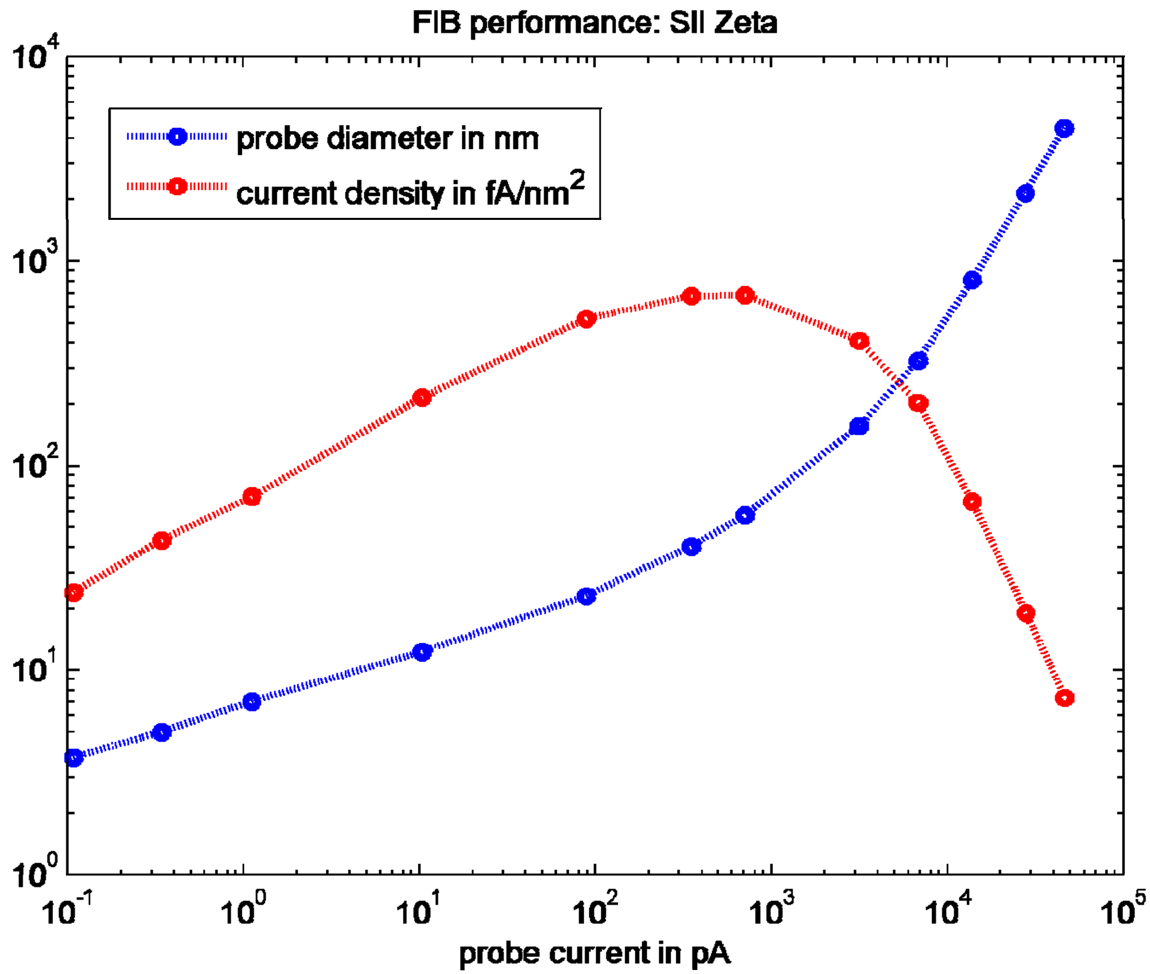
scanning the beam

2.1. Focused Ion Beam (FIB)



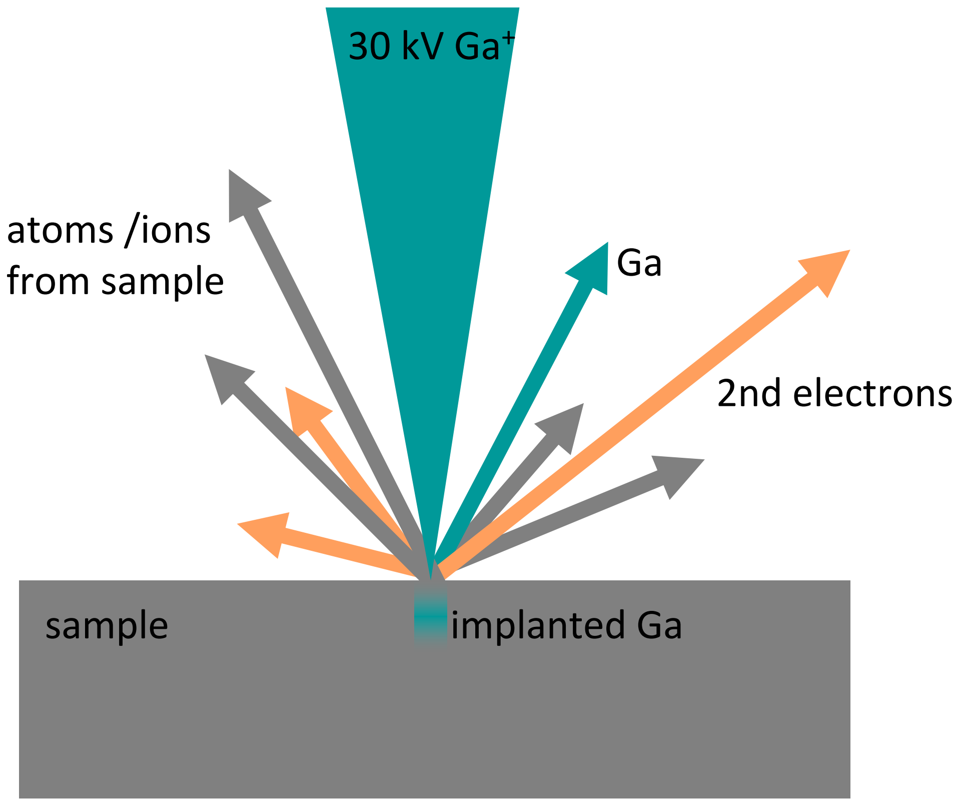
probe spot





- SMALL CURRENT → NARROW BEAM
- Beam tails can extend up to some µm

2.1. Focused Ion Beam (FIB): interaction with sample



Ga^+ beam hits substrate and yields

- secondary electrons
- sputterd atoms and ions
- implantation of Ga
- amorphisation /recrystallization

3 FIBing "modes":

- imaging, milling and deposition happen simultaneously
- ion current + atmosphere
  → pronounce one aspect!

Remarks.

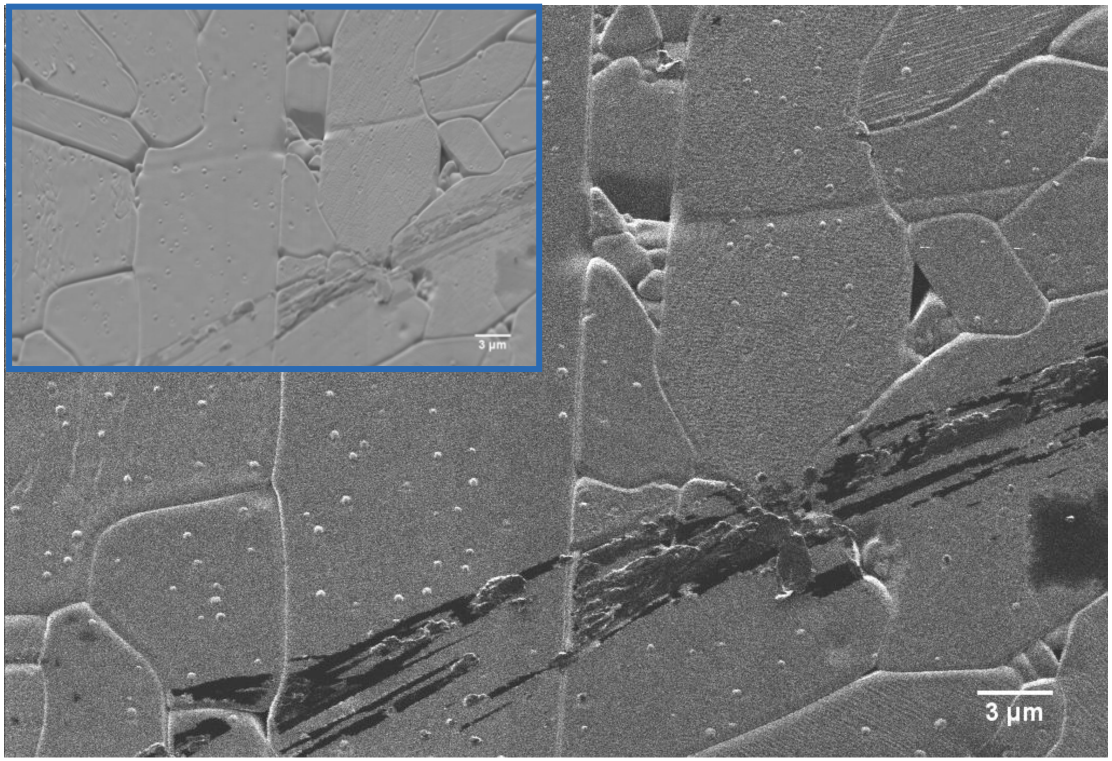
i) Implantation and amorphisation also occur at grazing incidence.

ii) Depth of damage layer depends also on energy of Ga^+.

- Impinging Ga^+ always mill a little bit and produce secondary electrons
- Sample surface is damaged more with increasing Ga^+ exposure

2.1. Focused Ion Beam (FIB): imaging

SE image from scanning with eBeam (3 kV, 120 um, hc)



SE image from scanning with **FIB** (30 kV, 40 pA)

FIB imaging:

- impinging Ga^+ produce secondary electrons
  → ET or InLens detector
- typically at 30 kV, 40 pA
  → optimal resolution and signal
- other currents and energies
  → different contrast

Advantages:

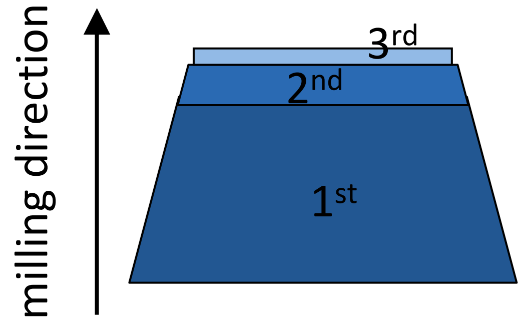
- channeling contrast
- removal of oxide layer

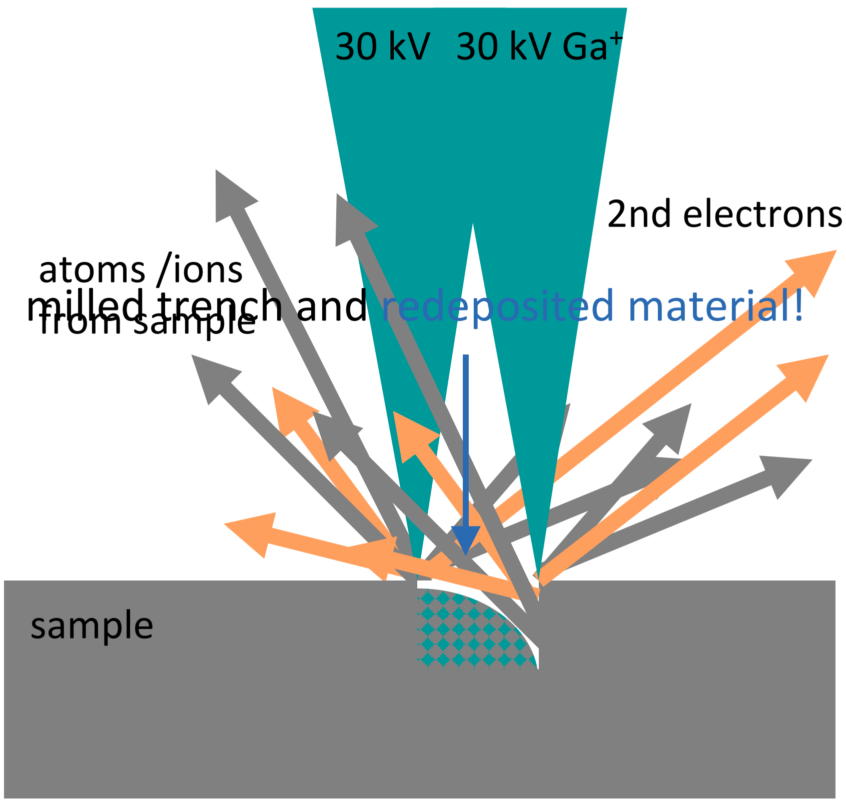
Disadvantages:

- damage of surface

Remark.
Now a days there is scanning He microscopy
→ high contrast
→ highest resolution

2.1. Focused Ion Beam (FIB): milling

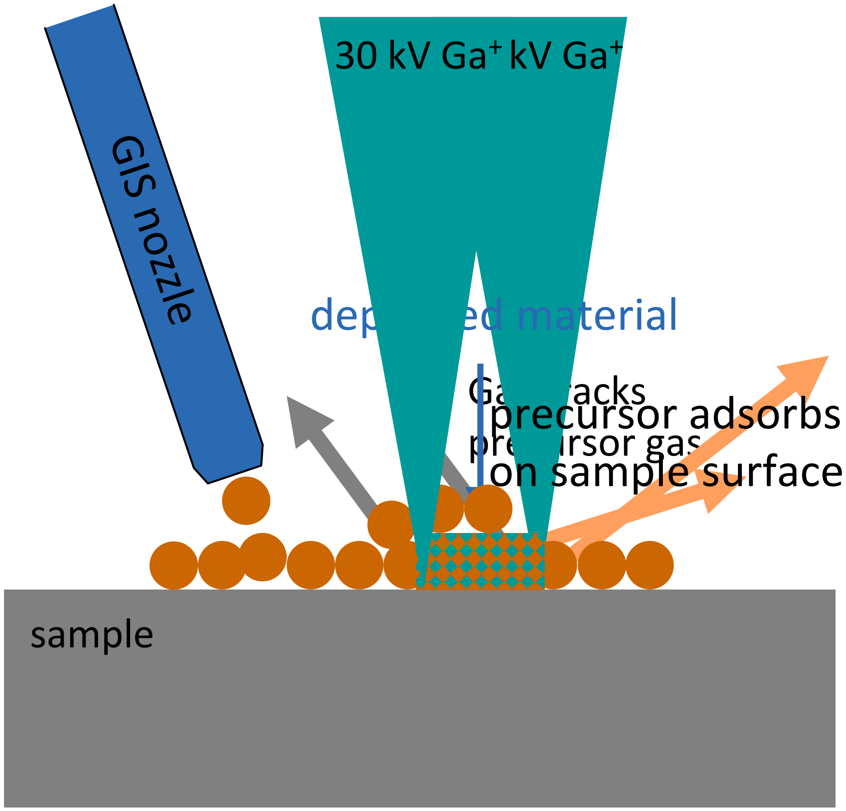




FIB as a nano scalpell: **milling**

- sputter process
- less damage at cutting surface for small currents
- resolution better for small current but high currents mill faster
  → use series of decreasing currents
- redeposition (all in one layer gives a wedge) → milling strategy is important (“milling mode” / “deposition mode”)
- dwell time! (↑ for ceramics)
- pixel fill factor ≥ 100 % is OK

2.1. Focused Ion Beam (FIB): depostion



Deposition of material

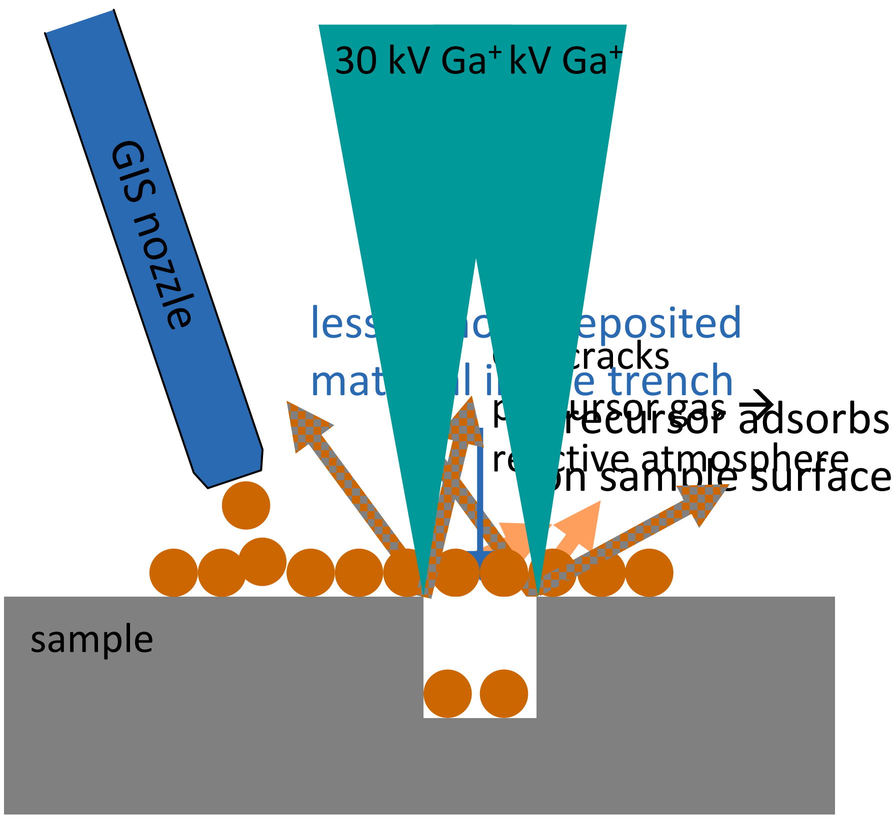
- nozzel → local gas atmosphere
- decomposition of precursor gas → CVD
- ion current = Area x 5 pA/µm^2
- pixel fill factor = 40 - 70 %!
- short dwell time
  (0.4 µs for C, 0.2 µs for Pt)
- also possible to use e-beam for depo
  → 1 kV & slow scanning speed!
- deposited material: mixture of Ga, C and the depo species

Examples of species that can be deposited:

C, Pt, W, Pd, SiO_2

- Deposited species is not pure: lots of Ga and C
- Deposition without surface damage only by eBeam deposition

2.1. Focused Ion Beam (FIB): enhanced milling



Enhanced milling (etching)

- impinging Ga^+ knocks out atoms and ions from sample
- redeposition is prevented by chemical reaction with the adsorbed gas
  → formation of volatile species
- etching gases that react only with certain species
  → selective milling

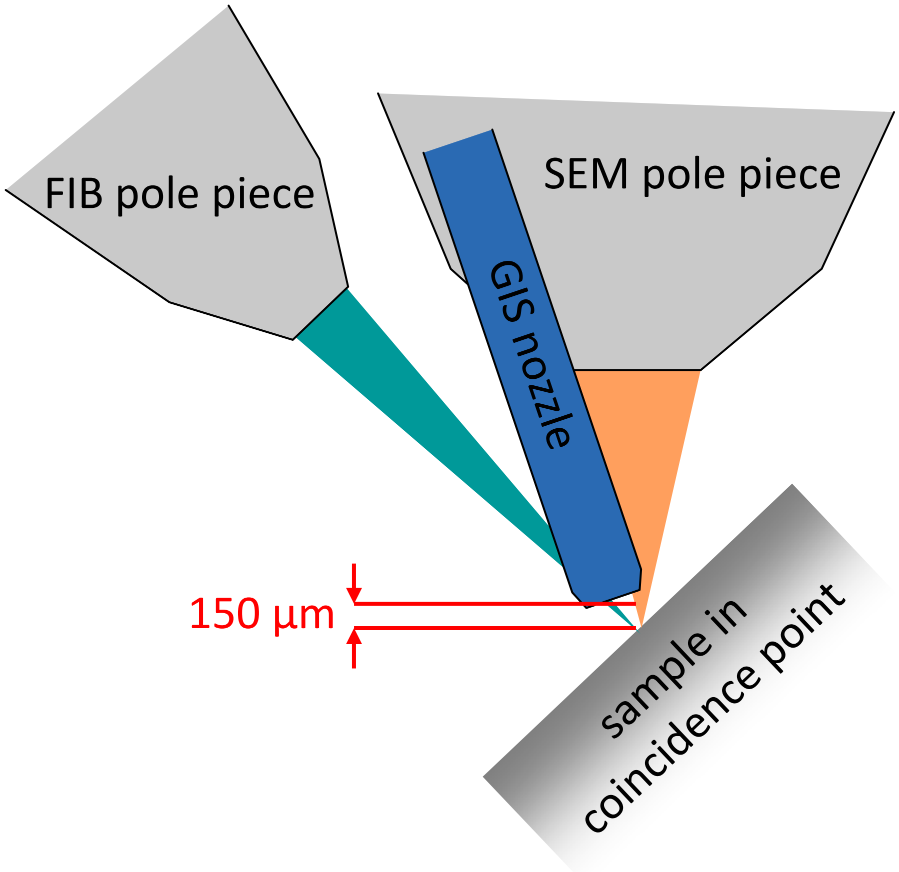
Examples of etching gases

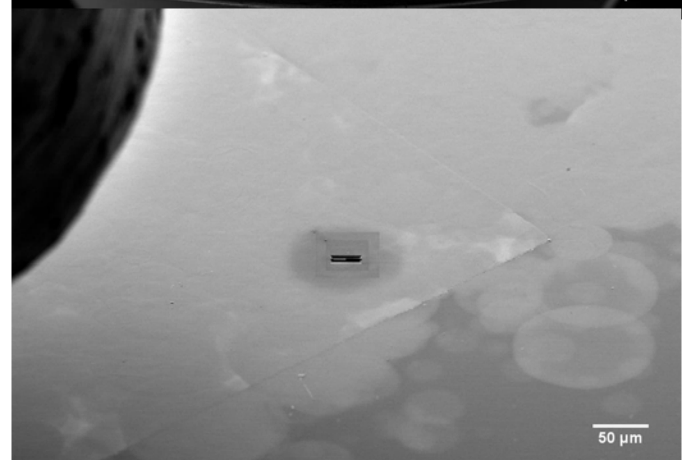
XeF_2 → enhanced Si and insulator milling

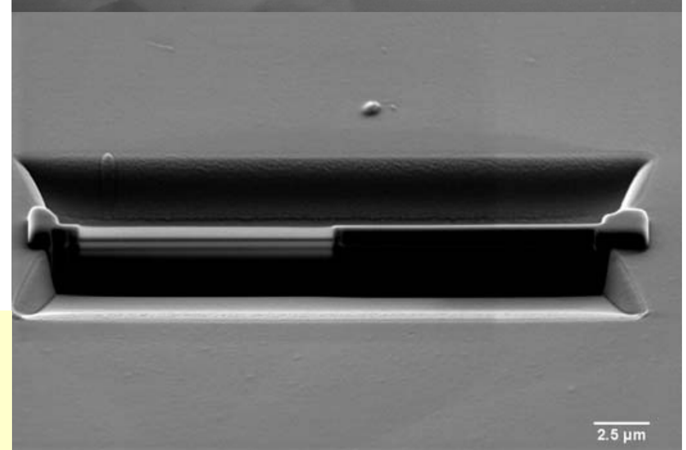
I_2 → enhanced metal milling

H_2O → enhanced carbon (polymers, ...) milling

2.1. Focused Ion Beam (FIB): GIS nozzel

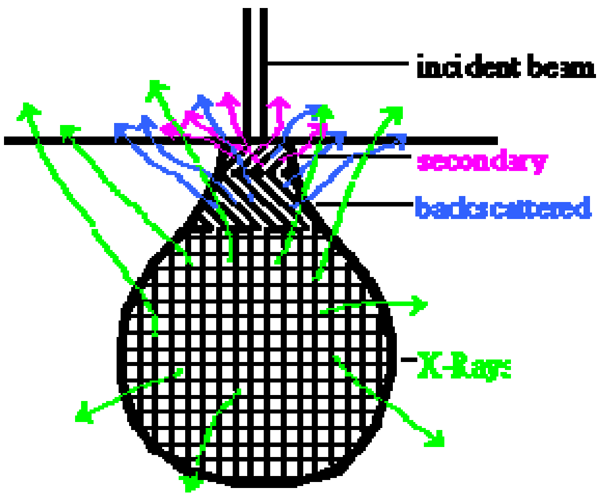






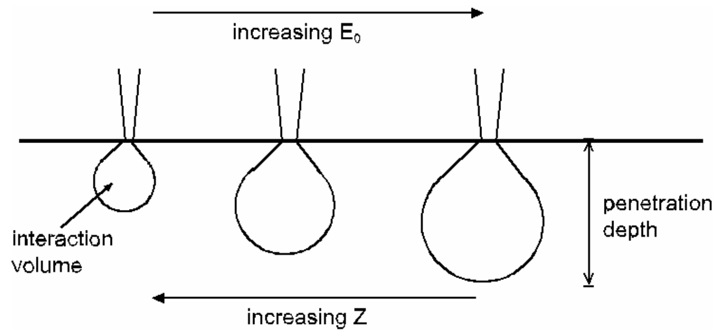
Rule: Only insert GIS nozzel, when sample is in coincidence point!

2.2. Scanning Electron Microscope (SEM)



SEM

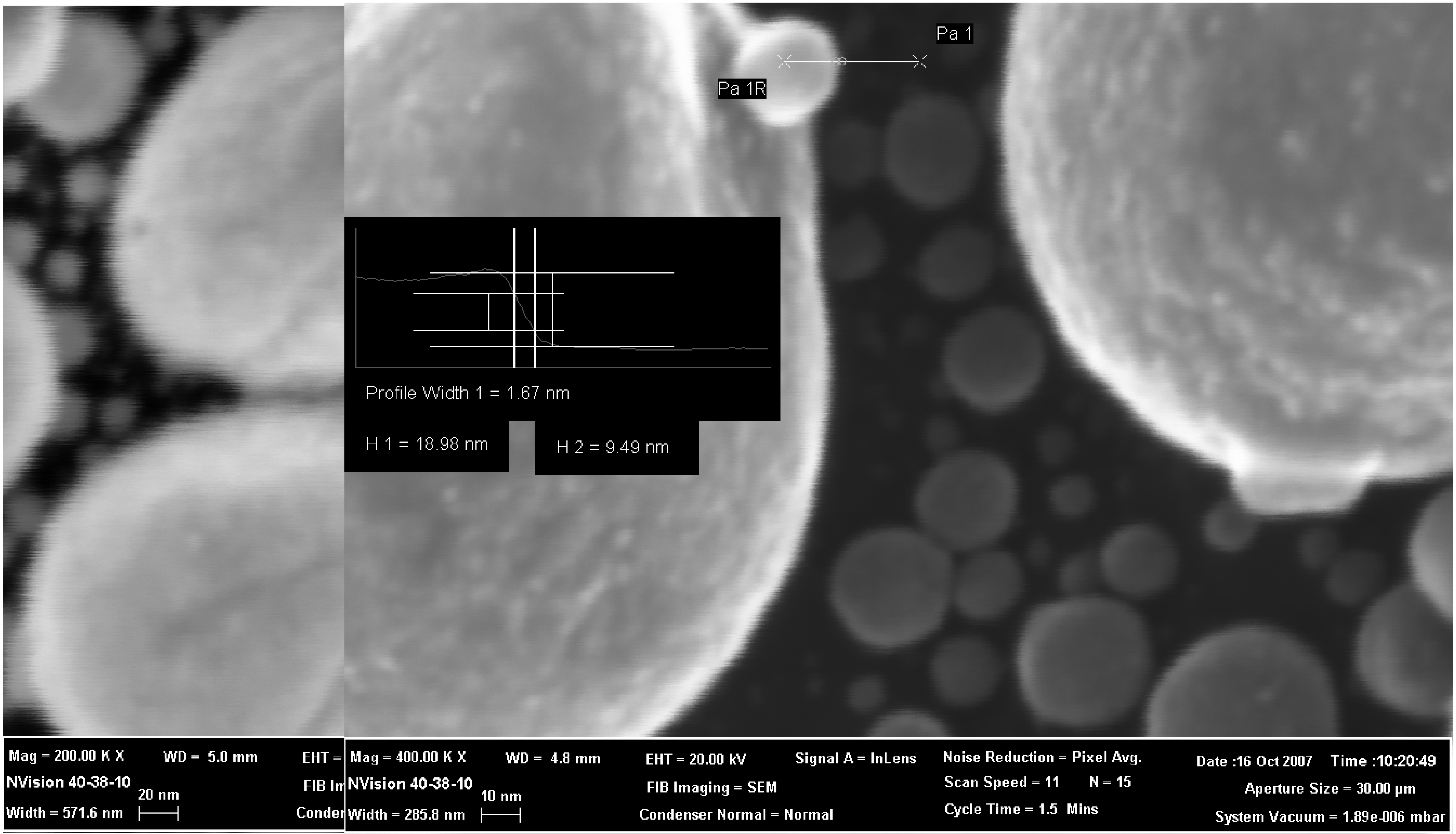
- probesize vs **interaction volume** → resolution
- imaging using several different signals → information



Size of interaction volume depends on eBeam energy and atomic number of material

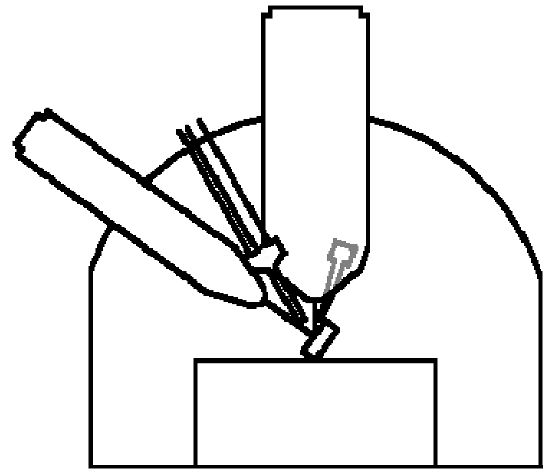
Signal	*Use*	*Resolution (typical)*	*Detector*
SE	Best surface sensibility	3 nm	SE, InLens
BSE	Z-contrast	15 nm	EsB
BSE	cristallographic information	15 nm	EBSD
X-Ray	semi-quantitative chemical information	500 nm	EDX

2.2. Scanning Electron Microscope (SEM)



Best resolution at 1 kV effectively 10 nm, for 20 kV maybe 5 nm. → Don't expect too much!

2.2. Scanning Electron Microscope (SEM)



Steve Reyntjens:

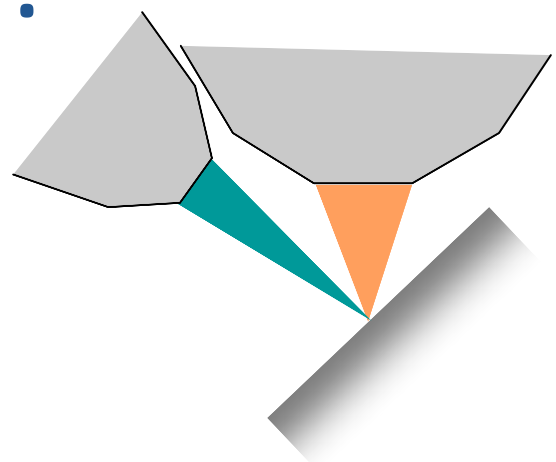
"Half of the rent of working with a Dual Beam is understanding the geometry!"

Pecularities of working in a combined microscope

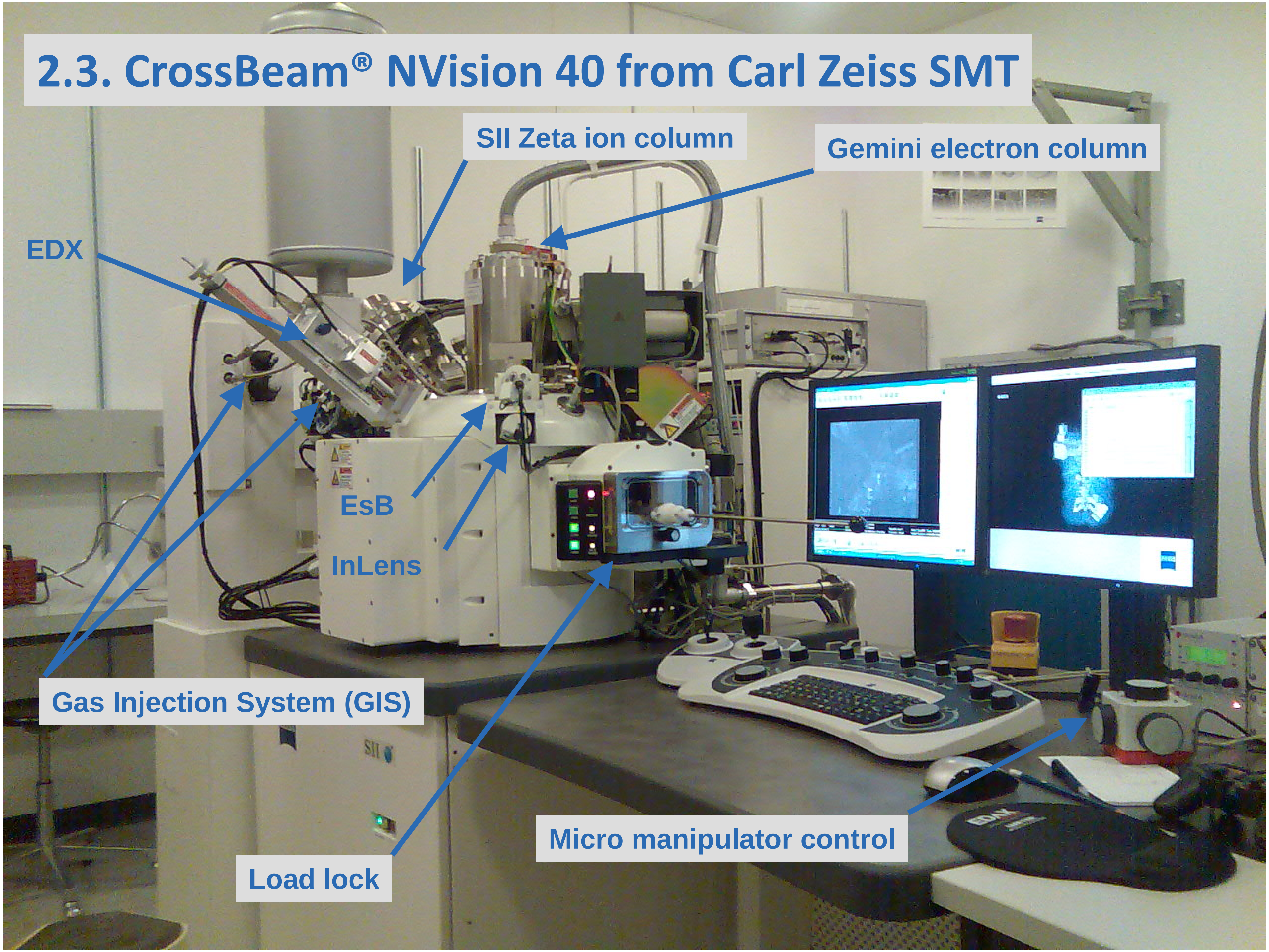
- working in **coincidence point, i.e. WD 5 mm**
  → eBeam is less stable for low EHT
- working at **54° tilt**
  → samples look different than from top view!
- working with **120 µm apperture** and **high current** mode
  → really strong contrast, again samples look different than at „normal" settings

Why all this?

- **see same spot with SEM and FIB**
- **normal incidence for FIB**
- **simultaneous FIBing and SEMing**



2.3. CrossBeam® NVision 40 from Carl Zeiss SMT



2.3. CrossBeam® NVision 40 from Carl Zeiss SMT

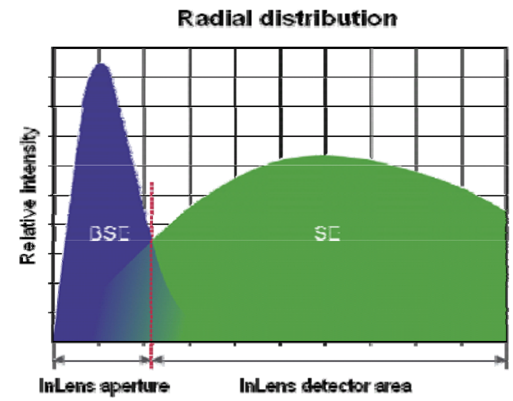
SEM: **Gemini column**

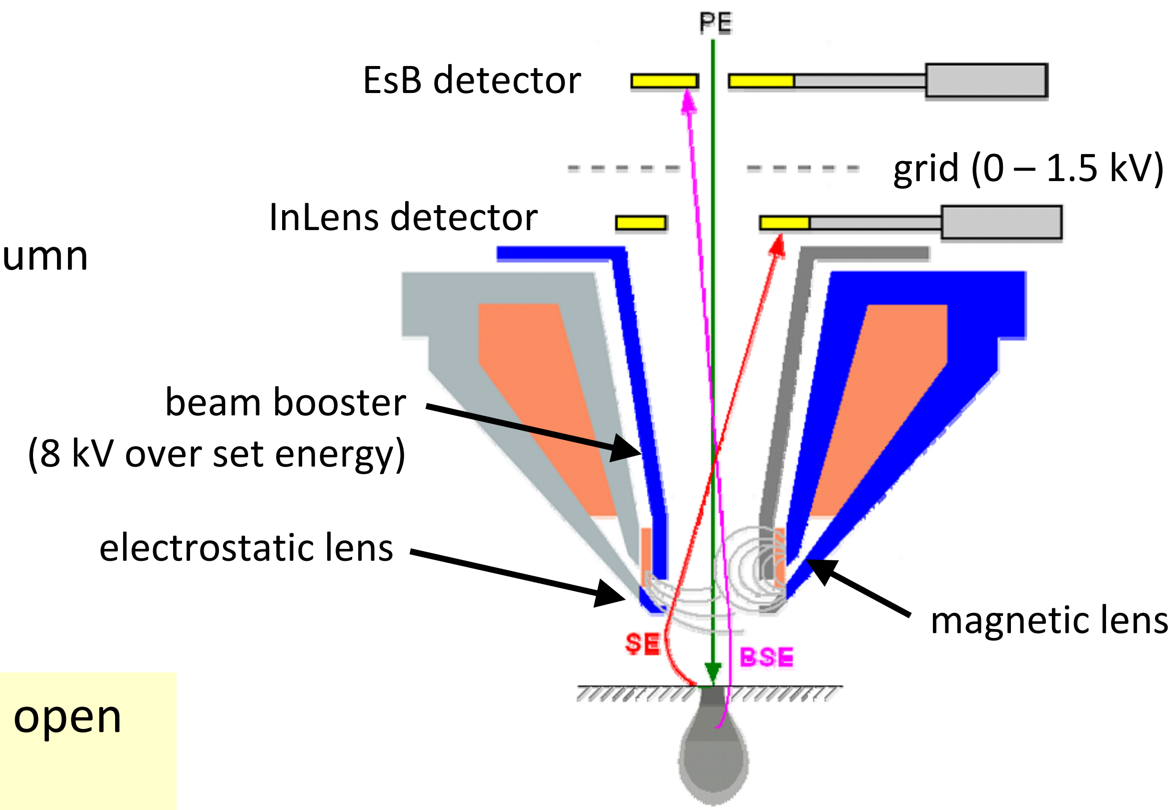
→ decceleration field (beam booster)

→ inLens and EsB geometry selects electron energy

→ typically adjust a lot for stigmatism

→ construction problem: column valve position not ideal





Rule: Leave microscope with open SEM colum valve!

2.3. CrossBeam® NVision 40 from Carl Zeiss SMT

FIB column: **SII Zeta**

- fixed number of appertures: 13
- condensor allows to adjust current for each apperture
  → in principle different sets of currents are possible
- adjustable acceleration voltage
  → sets of currents for different voltages
- problem with stability of LMIS → often need to heat

In Smart SEM: **FIB control**

- FIB imaging only with imaging current (typically 40 pA, 30 kV)
- FIB milling box is set in FIB image, column optics values are computed for the milling current from a list
  → well aligned currents needed to avoid shifts!
- new program “daily align”
  → adjust those currents that you will use that day

Rule: Do not click auto extractor!

2.3. CrossBeam® NVision 40 from Carl Zeiss SMT, **“Our” FIB-SEM**

Organization

- System is part of EMEZ → acknowledge support in publications
- System was partially financed by SNF → acknowledge support
- Room: HPM A66 (33312)
- Technician: Philippe Gasser, HPT C 104 (36541)

Booking

- no booking more than 2 weeks in advance
- 25 % from 8 am to 5 pm is reserved for EMEZ
- NMW, LNM, LMPT, MICRO have special access rights for the first 3 years (i.e. until June 2010)
  → 75 % from 8 am to 5 pm together
- please don’t waste the precious beam time!

2.3. CrossBeam® NVision 40 from Carl Zeiss SMT, **“Our” FIB-SEM**

Configuration

- fully motorized 6-axes stage (m-axis: adjust tilting axis)
- GIS:
  - 2 solid state precursors: Pt, C (soon Pd, W)
  - insulator deposition: SiO_2
  - etching gases: H_2O (soon XeF_2)
- EDX and EBSD detectors: EDAX Pegasus XM 2 System
  (mounting positions do not allow for simultaneous data acquisition)
- Lithography kit: Raith ELPHY Quantum
  → external high precision control of eBeam and FIB
  → import of GDS2 files
- Micromanipulators: Kleindiek MM3A
  - 1 for TEM lamella lift-out
  - 4 for special purposes
- load lock

3. Micromanipulator MM3A from Kleindiek

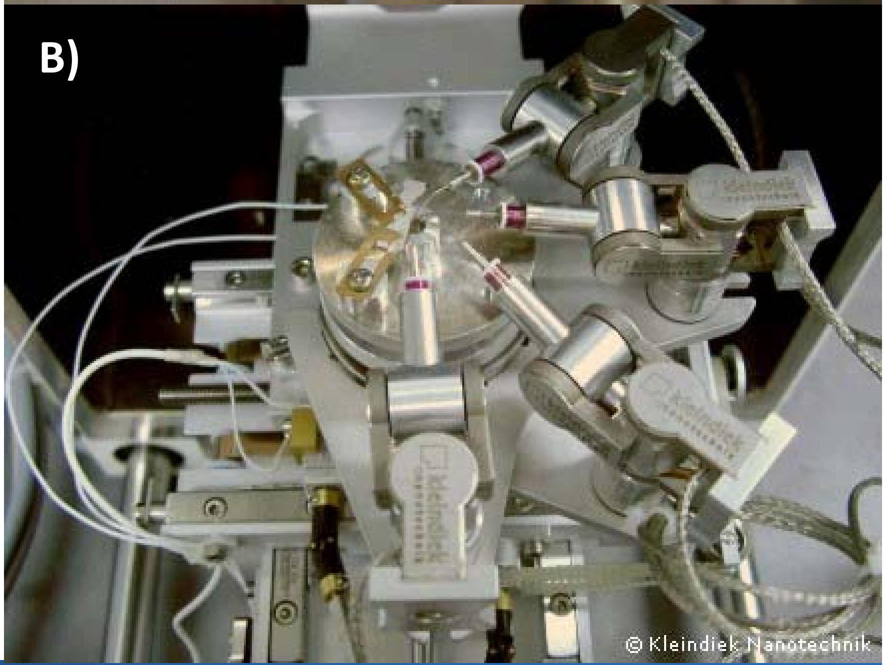
MM3A: MicroManipulator with 3 Axes

A) roof mounted MM3A
→ for TEM lamella lift-out

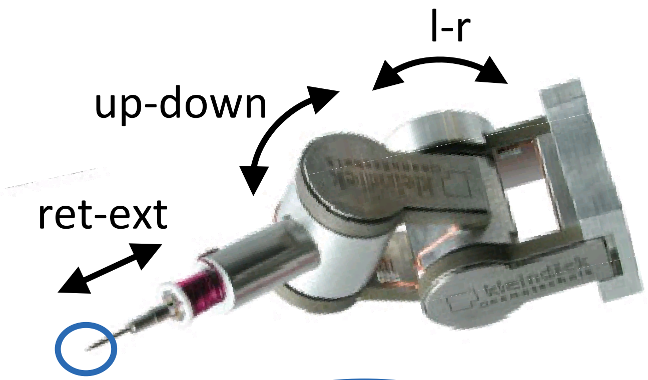
B) stage or **door mounted** MM3A
→ multi purpose:
e.g. electrical measurements,
manipulation of nano objects,
force measuremnts

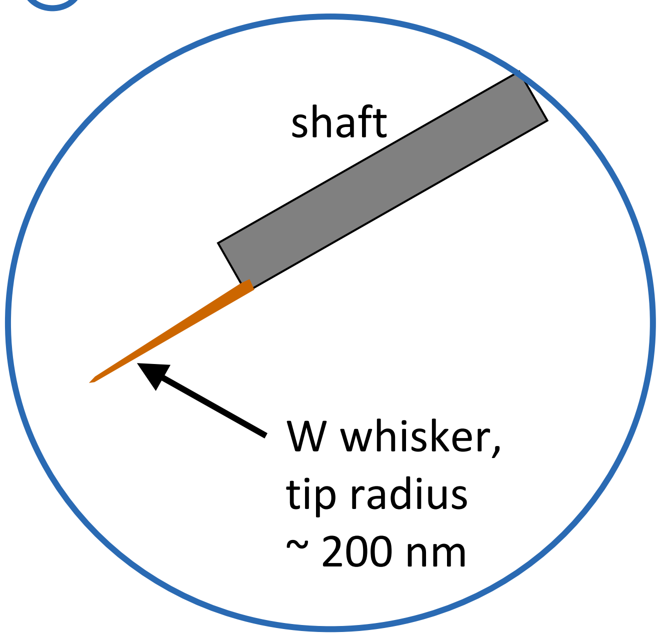
add-ons:

- gripper: like tweezers
- rotip: rotatable tip
- force measurement sensor (→ LNM)



3. Micromanipulator MM3A from Kleindiek: steering



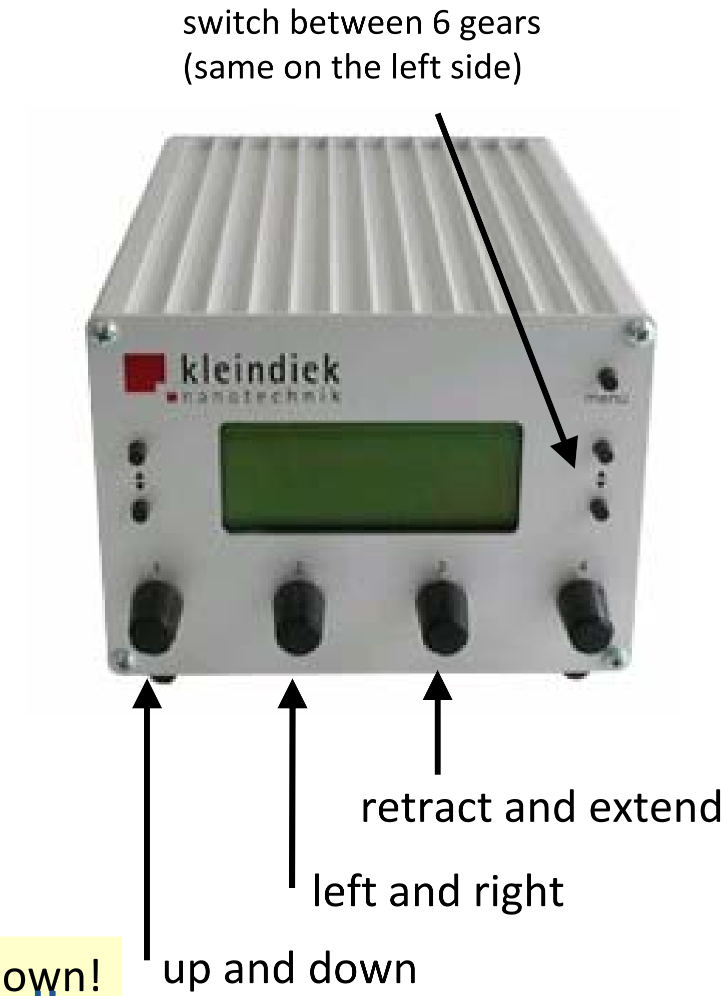


Piezo motors

→ **continuous** (f) or **slip-stick** (c) mode

Programmable speeds example:

1 → f1: finest continuous movement
2 → f4: 4x f1
3 → hybrid f4 + -c1
4 → c1: one slip-stick step
5 → c32: 32x c1
6 → c64: 64x c1



Rule: Switch on Specimen Current Monitor before touch down!

Manipulator tip is electrically connected to ground!

4. Applications overview

Cross Section: cut flat section for looking into the material

- width x depth ≈ 2 x 1 μm^2 to 50 x 20 μm^2
- typically 1 - 2 h work

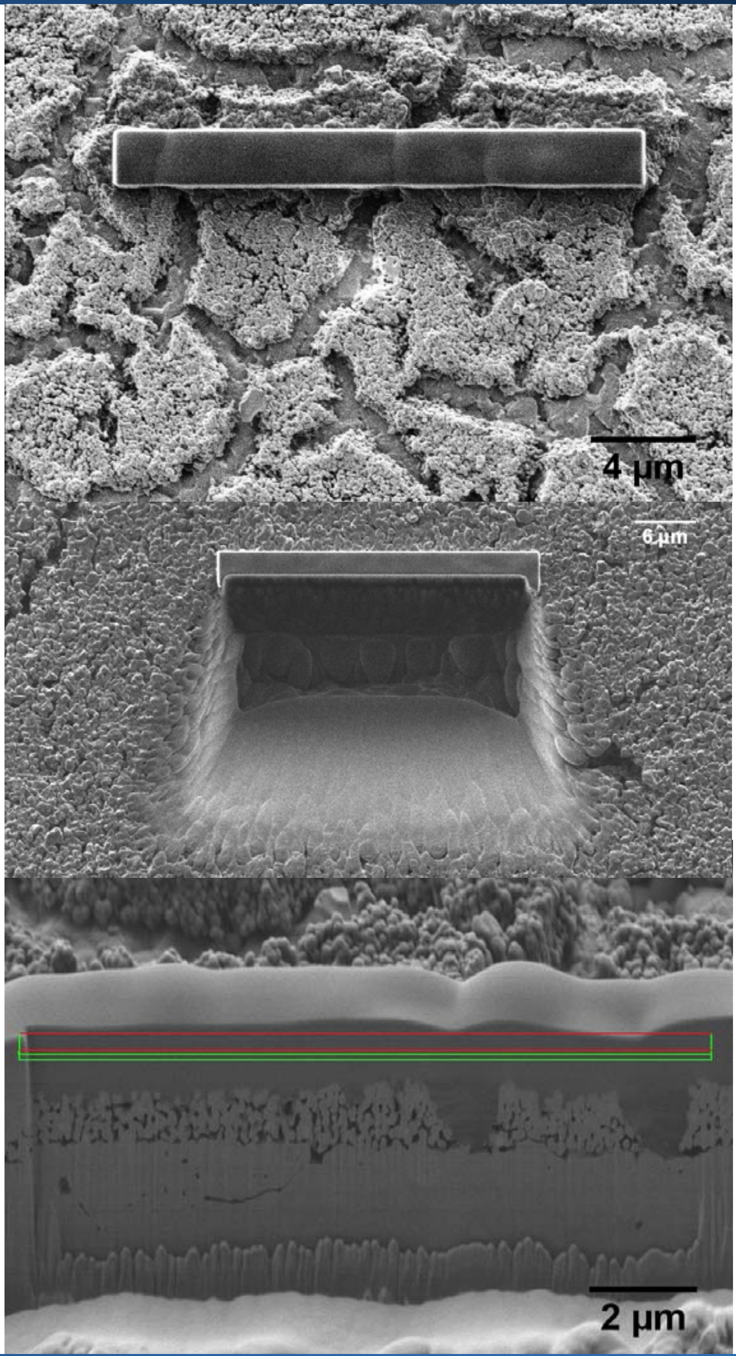
TEM lamella preparation: cut a thin lamella for inspection in TEM

- width x depth ≈ 10 x 5 μm^2
- thickness ≈ 60 to 200 nm
- typically 6 – 12 h work

Deposition /milling with "feature mill": complex patterns, special milling strategies

- load bit maps with prepared patterns
- maybe 30 min to 1 h of work

4.1. Cross Section (CS) – the basic skill



Bring sample into coincidence

- adjust eucentricity (M axis) → 54° tilt
- set WD to 5.25 mm and drive into focus (Z axis)
- switch to FIB and fine tune Z,
  adjust Y beam shift

Three steps for preparation of a Cross Section (CS)

- deposition of protective layer
- milling a large trapezoid trench
- polishing the CS

Why is this “the basic skill”?

- If you can do properly a cross section, then you can do everything else as well as well.

4.1. Cross Section (CS) – the basic skill

Detailed receipe:

deposition of protective layer: RECTANGULAR BOX

- typically area A = 2 x 15 μm^2
- deposition with thickness, typically 2 µm
- current = A · 5 pA/μm^2 (e.g.) 30x5 pA = 150 pA)
- pixel fill factor 40 – 75 % (adjust milling resolution)
- dwell time (0.4 µs for C, 0.2 µs Pt)

milling a large trench: COARSE TRAPEZOID

- depth ≈
- height ≈ depth
- current ≈ 15 nA
- dwell time large for hard material

polishing CS: FINE TRAPEZOID

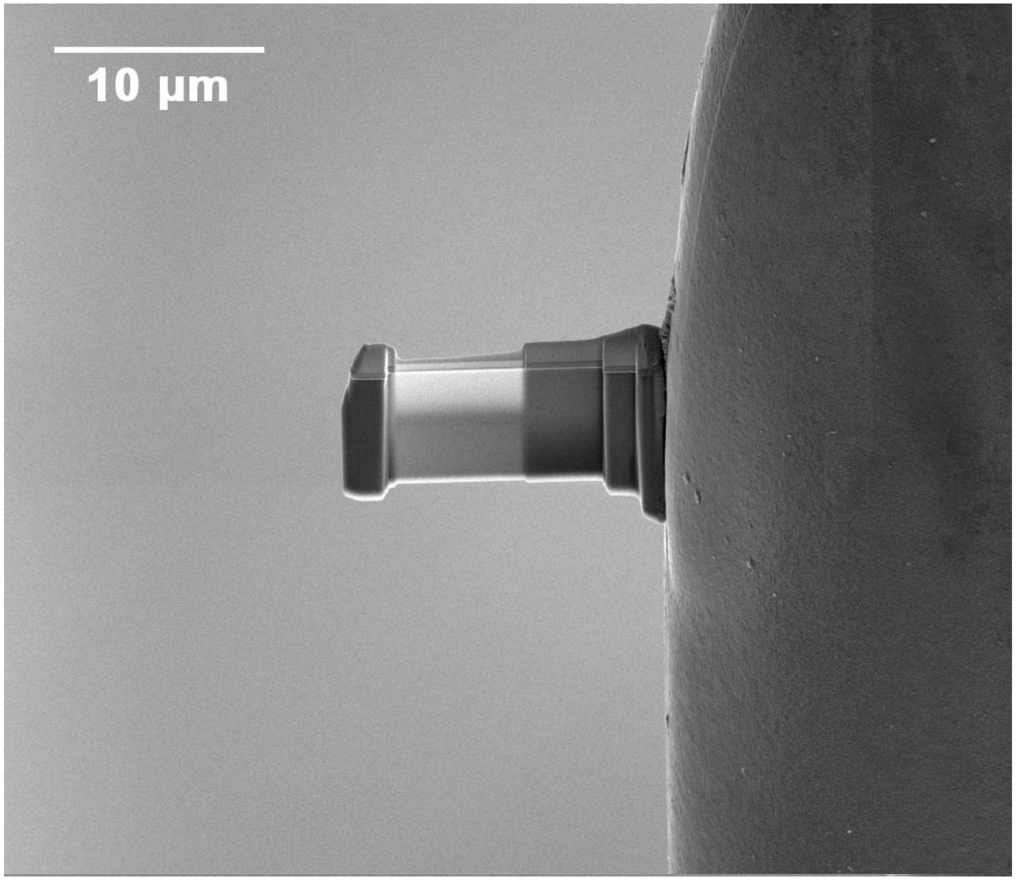
- for each current down to 40 pA do a milling

imaging CS: SEM MODE

- adjust tilt correction angle to 36°
- take micrographs from overview to detail view

TEM lamella preparation:

- protection layer
- cut two big trenches
- cut free lamella partially
- solder it to a manipulator tip
- cut free lamella from sample
- lift-out the lamella
- bring TEM grid to coincidence point
- solder lamella to TEM grid
- cut free manipulator tip
- final thinning of lamella



4.2. TEM Lamella preparation

Details of in-situ lift-out:

big trenches milled, lamella is 1 µm thick

→ change stage tilt to 10°, cut free lamella partially
use deposition mode for milling

→ insert micromanipulator, bring tip to lamella and at
Switch on SCM!
Do not press on lamella!

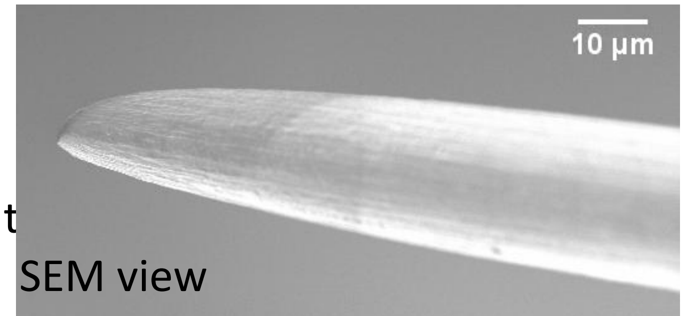
→ cut free lamella
Move stage down for “lift-out”!

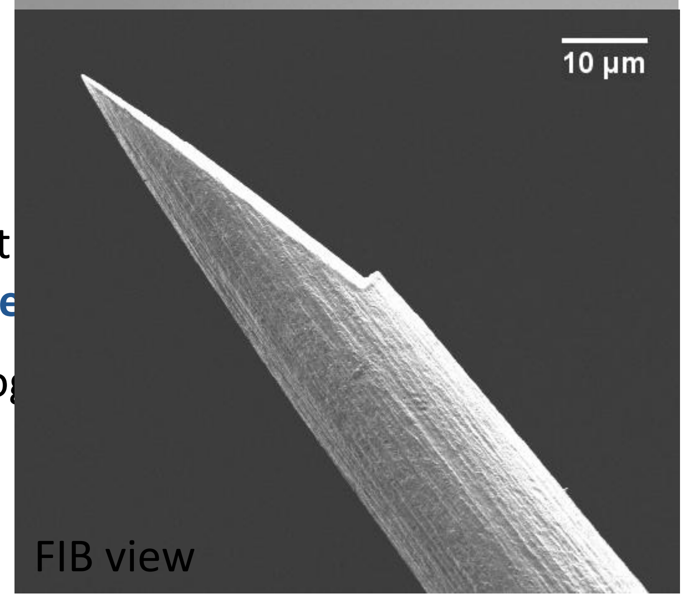
→ bring TEM grid to WD 6 mm at 10° stage tilt, adjust
Switch between FIB and SEM imageing in TV mode

→ bring lamella in touch with grid and solder them to
Check that SCM is on!
Do not press lamella to grid!

→ drive away lamella, then sharpen the tip

→ bring lamella in coincidence at 54+Δ° and polish





4.3. Feature mill

Example:

Milling or deposition of regular dots

- prepare a bitmap using your favourite graphics program
  and compute the area of the pattern
- bring sample into coincidence point
- select region on which the pattern shall be
- open feauture mill and draw in an image
- load your bitmap and select a rectangle
- compute the dose from magnification and area
  remember high currents have large beam diameter → resolution limit!
- start

Outlook

Many more things (applications) can be done with a FIB-SEM!

Examples are:

- contacting of small structures by metal deposition
- non perpendicular sections
- EDX linescan of mapping on a section
- EBSD mapping on a section
- automated repeated cross sectioning → 3D image
- ....

Literature

- R. G. Forbes, Understanding how the liquid-metal ion source works, *Vacuum,* Vol 48, no 1, pg 85-97 (1997)
- S. Reyntjens, R. Puers, A review of focused ion beam applications in microsystem technology, *J. Micromech. Microeng*. **11** (2001), 287-300