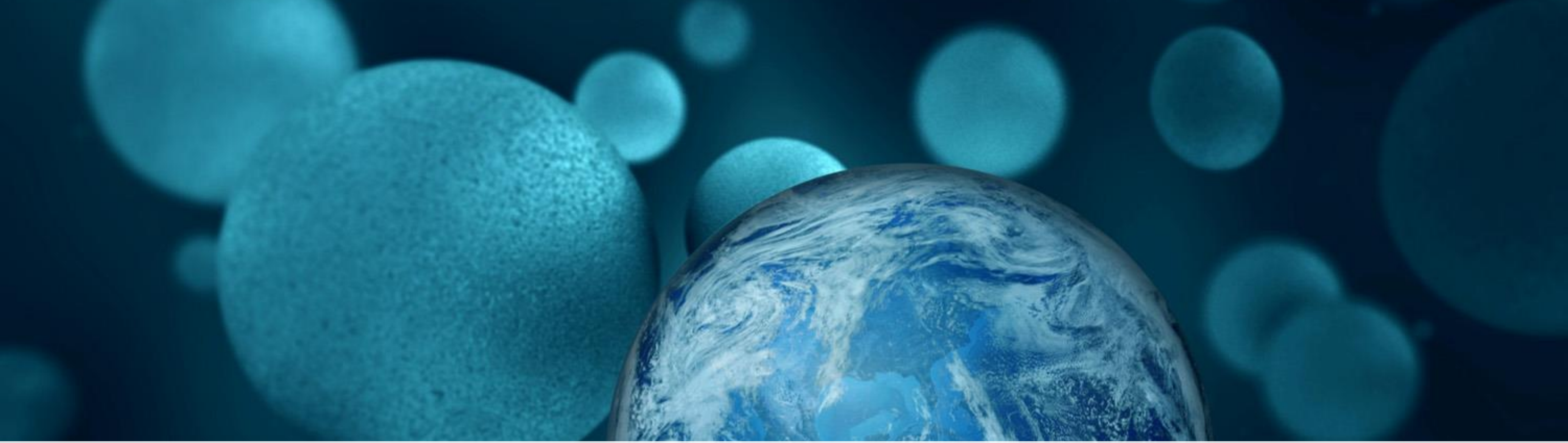


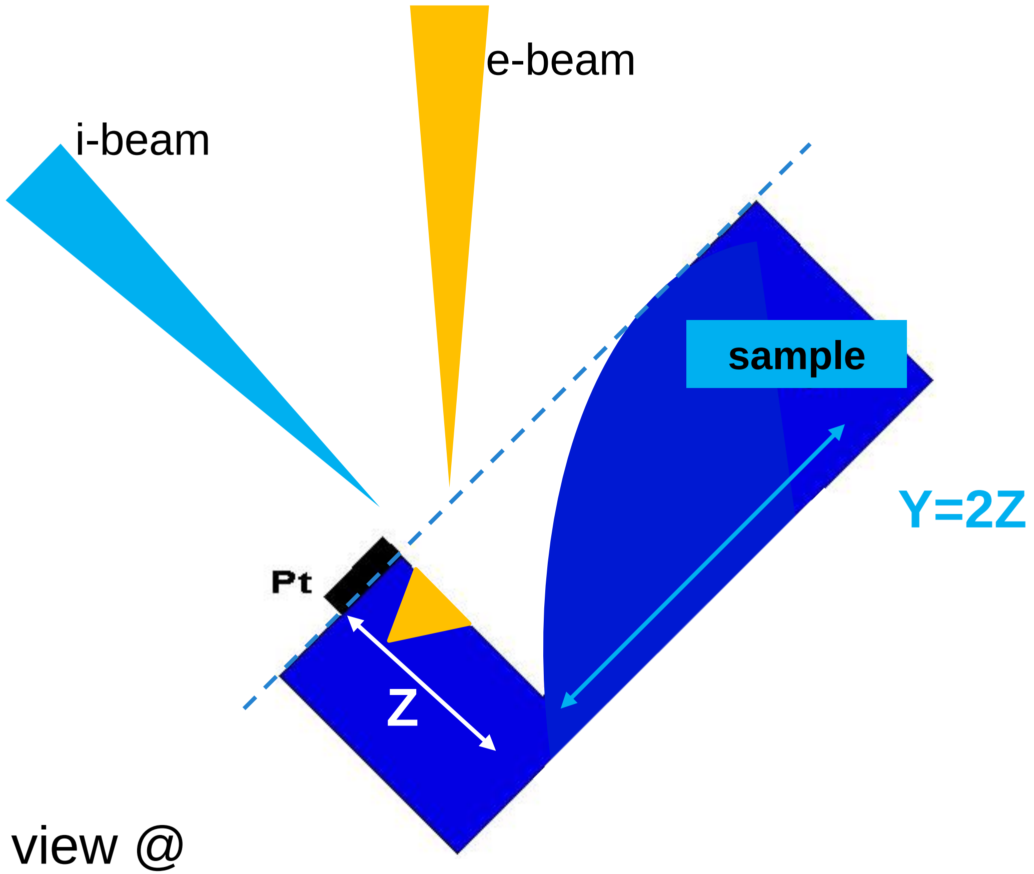
Scios
Cross Sectioning

Module 2

Scios : (small) dual beam

- DualBeam system unique functions
  - Slice with FIB and view with SEM (simultaneously=SPI)
  - In-situ cross sectioning, etching & coating
  - metal deposition for protection
  - Electron beam metal deposition for protection
  - Electron beam for charge neutralization
  - Thin TEM sample preparation (<100nm) with low kV cleaning
  - Site specific micro analysis

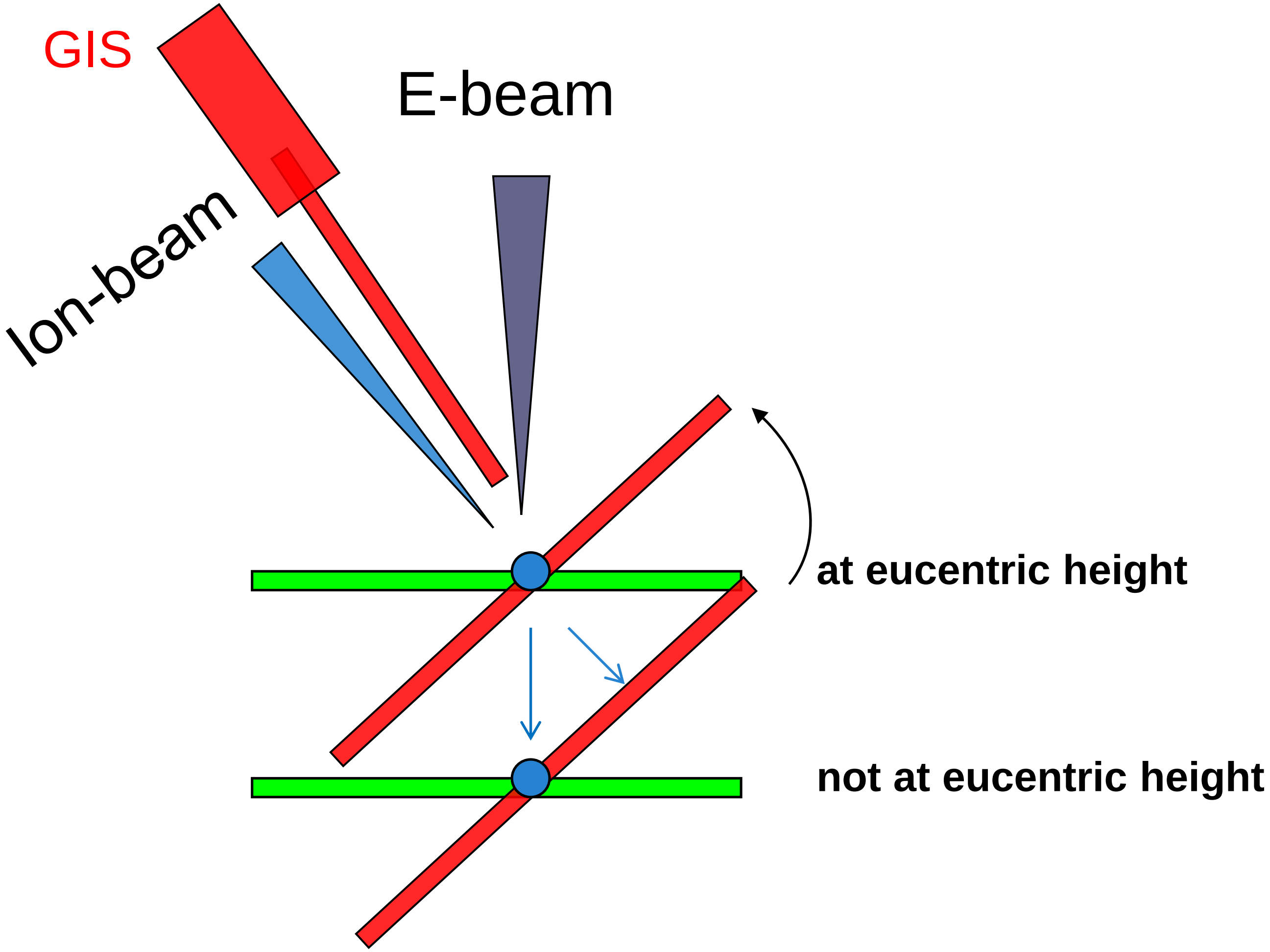
What is a Cross section ?



Side view @
52°tilt

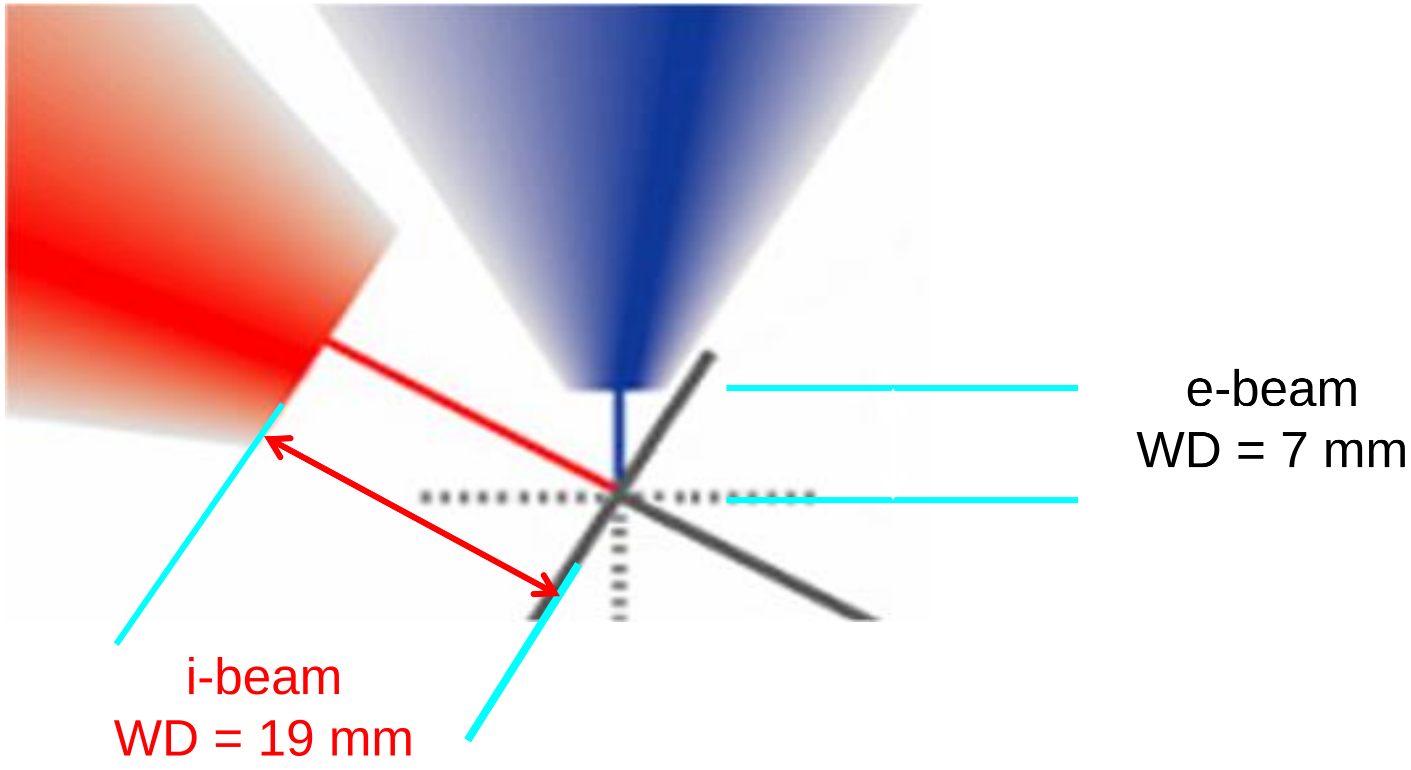
- FIB removes small amount of material leaving a perpendicular wall for imaging (with e-beam or i-beam)
- SEM takes image of revealed structures below the surface to image/measure the previously buried feature

Eucentric height and Coincidence Point



- Eucentric height is the coincident point of both beams and the tilt axis of the stage.
- It is the ‘magic’ distance from the pole piece to the sample surface.
- It is where typically all the dual beam work is done.

Optimal specimen chamber and final lens design



Optimized e- and i- Beam
Working distance

FIB Cross-sectioning

Part 1

Cross section overview

- Find area of interest
- Set coincidence point
- Deposition of a metal layer
- Bulk Mill (Regular Cross Section)
  - Removes material in front of feature for viewing
- Intermediate Mill (Cleaning Cross Section)
  - Make face more perpendicular in a fast way
- Cleaning Mill Cleaning Cross Section)
  - Finely removes material to reveal feature
- Imaging (e-beam and i-beam, EDS analysis)

(general) Set-up

- Set i-beam current to 100pA (30kV)
- Set e-beam current to 400pA (10keV)
- Use e-beam to find a feature or area of interest
- Rotate stage to align horizontal axis of feature with tilt axis
- “Align feature” are aids to rotate the sample into position
- Set coincidence point (assumed after this)

Deposition of metal layer

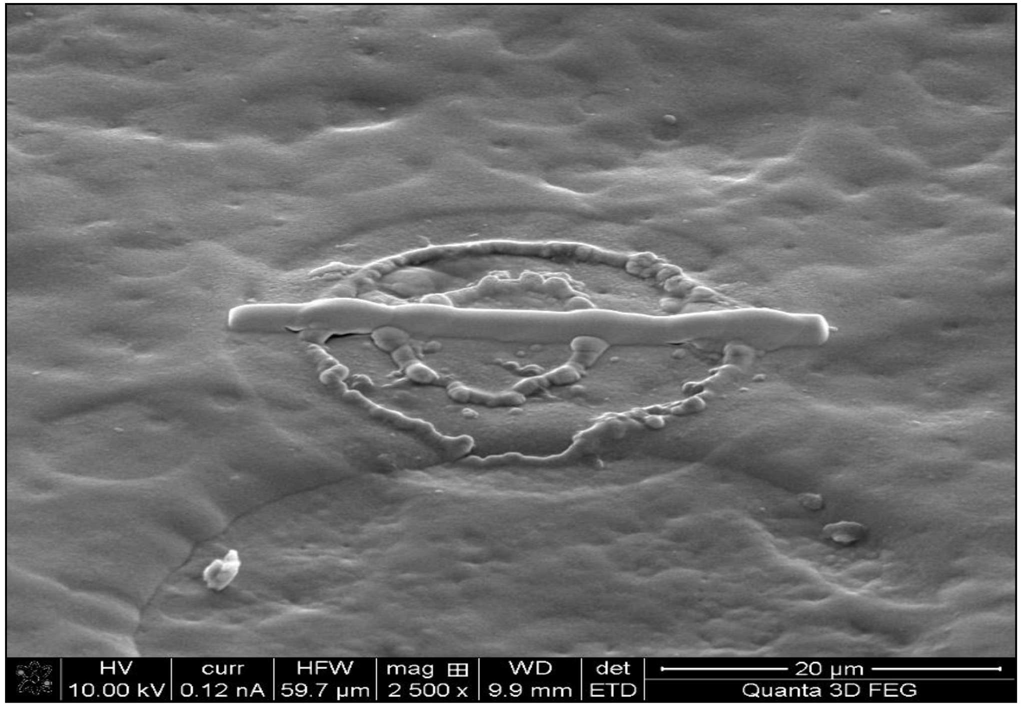
Why deposit (Pt, W or C) layer:

- For protection of area of interest
  - e-beam induced deposition (when top surface of sample is important) before
  - i-beam induced deposition
- Planarization of sample surface
- As a reference point

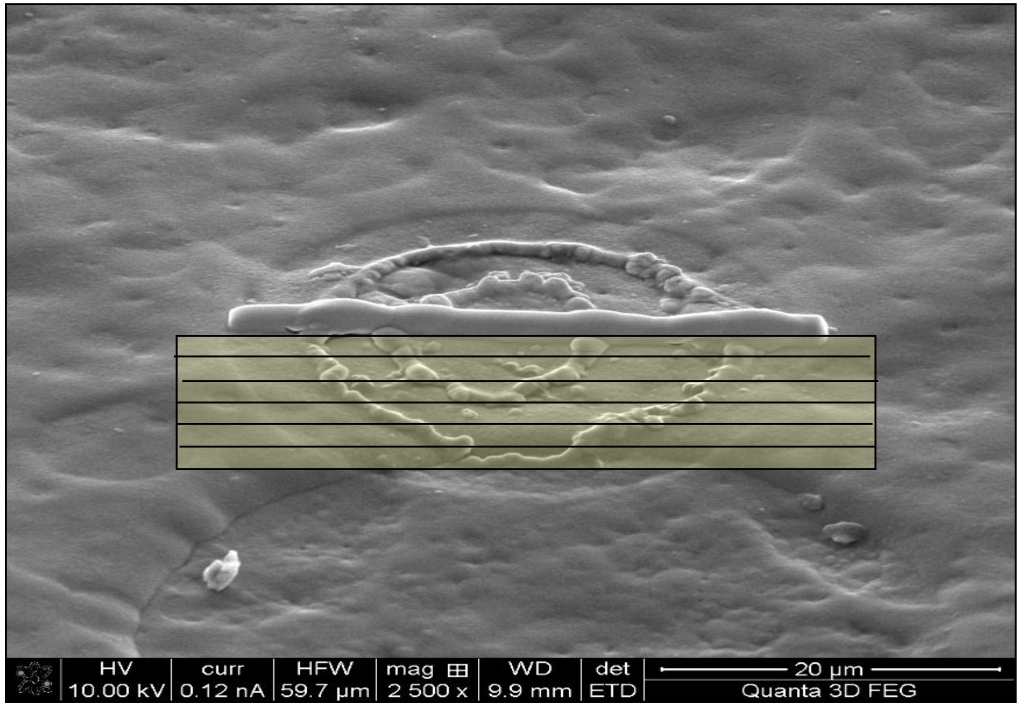
Bulk Mill

- Draw Regular Cross Section pattern
  - Align top edge ~ 1-2 µm from front of Pt layer
  - For X: Allow 3-4 microns on each side
  - For Z: The desired depth of the deepest part
  - For Y: 2 times the depth (Z)
- Set beam current 15-65 nA
- Use “Si multipass (new)” application
- Start milling

Pt deposition

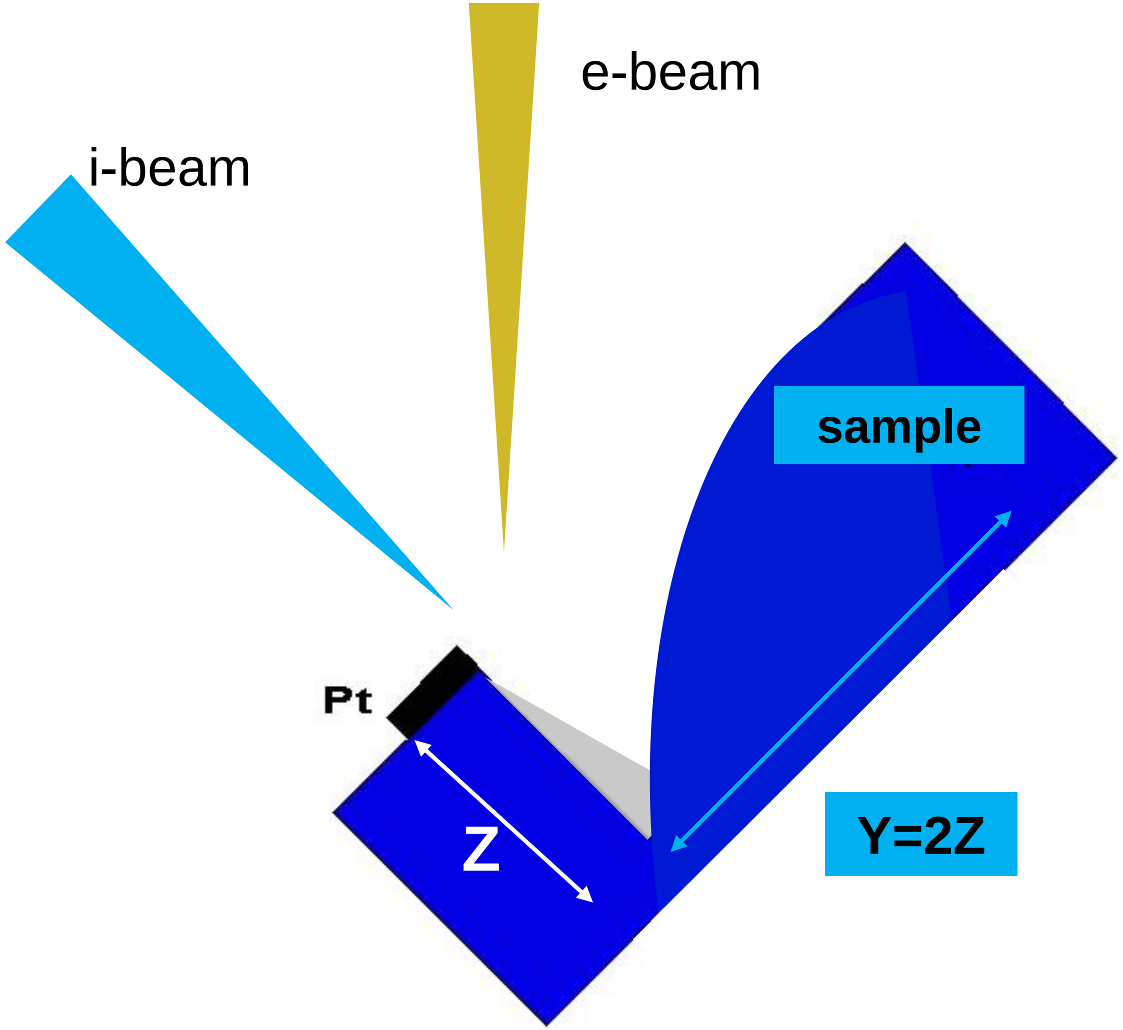


Pt deposition:
E-beam view @ 52°tilt



E-beam view @ 52°tilt:
material to be removed = bulk milling

Bulk milling with Regular Cross Section



Side view after removing material using **Si-multipass new** @ 52°tilt with high i-beam current

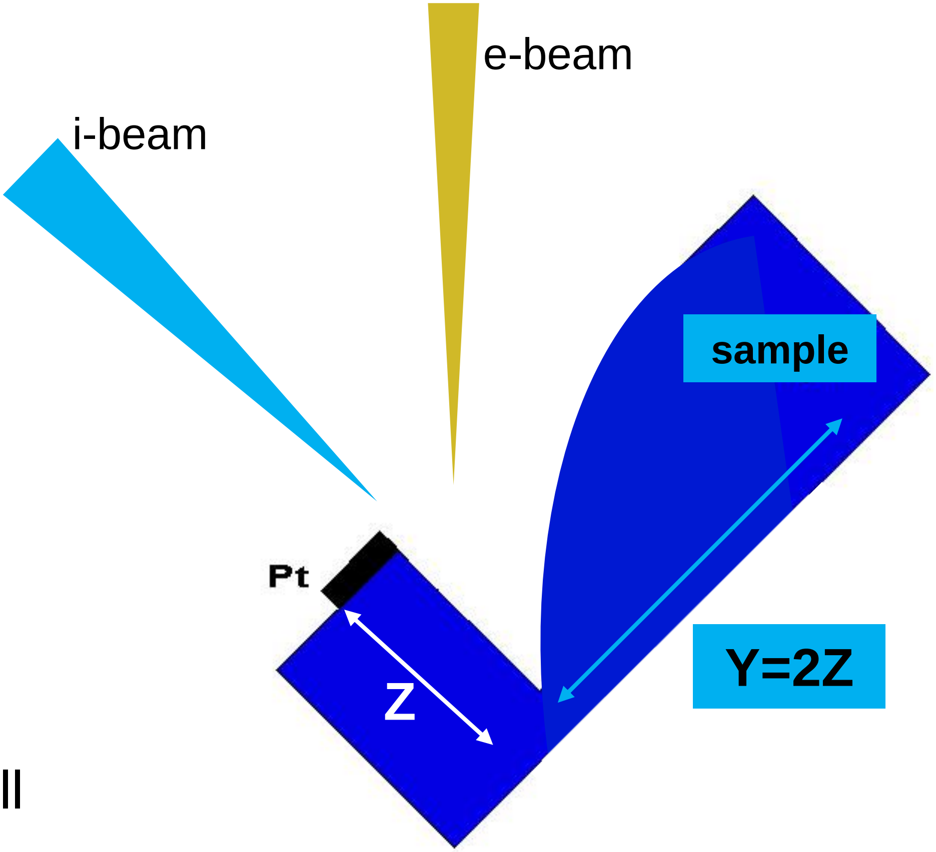
Intermediate Mill

- Select 1/4 - 1/2 the beam current of rough mill
- Tilt the sample +2° (extra tilt depends on BC and material)
- Remove previous pattern
- Draw Box pattern or Cleaning Cross Section (Si application file)
  - Adjust front close to Pt layer (~ 0.25 µm)
  - Adjust back to just overlap with just milled area
  - Set X to be about 1 µm smaller than previous mill
  - Set Z to about 1/4 to 1/2 of desired depth
- Ready, mill!

Final Polish

- Select 50 - 300 pA
- Draw Cleaning Cross-Section (Si appl file)
  - Adjust leading edge to go through feature
  - Adjust trailing edge just beyond previous mill
  - Set X to be about 1 µm smaller than previous mill
  - Set Z to about 1/4 to 1/2 of desired depth
- Start milling
- Grab frames periodically to check progress or use SPI

Cleaning Cross section

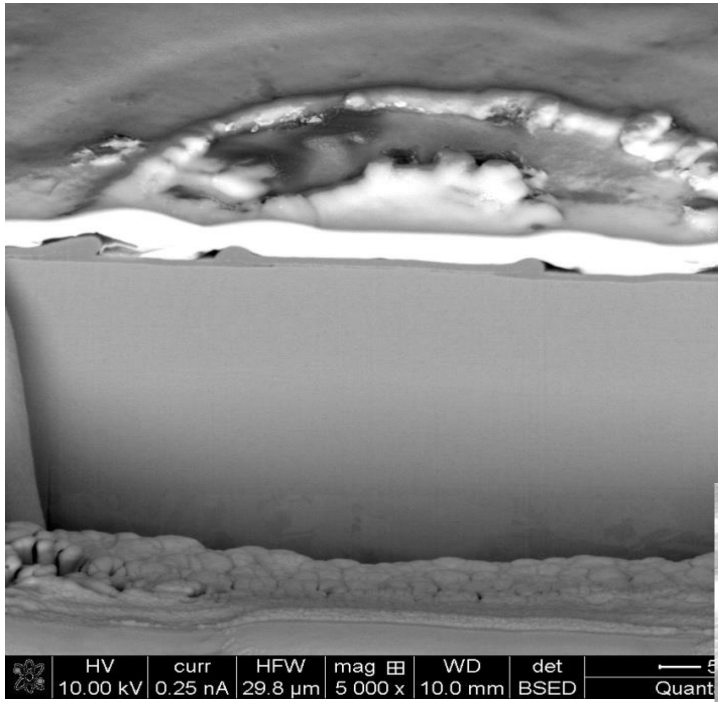


Side view after cleaning side wall using **Cleaning Cross Section** @ 52°tilt with medium high (and low) i-beam current

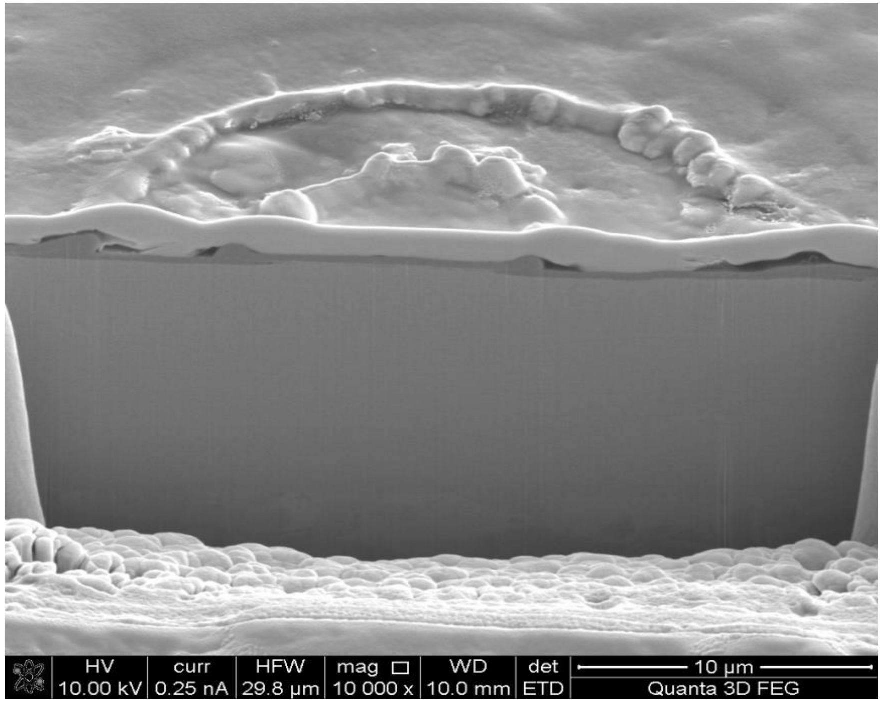
Imaging Cross section

- SEM; using OptiTilt in combination with T1 + T2
- FIB; using standard mode + ETD or ICE
  - Set beam current to 10-50pA
  - Tilt stage to 0° ; (compucentric) rotate 180°
    (+ scan rotate image 180°)
  - If needed focus + stigmate (outside area of interest)
  - Use beam shift and mag. to frame picture perfectly
  - Use a slow single scan ~40s to generate a nice photo

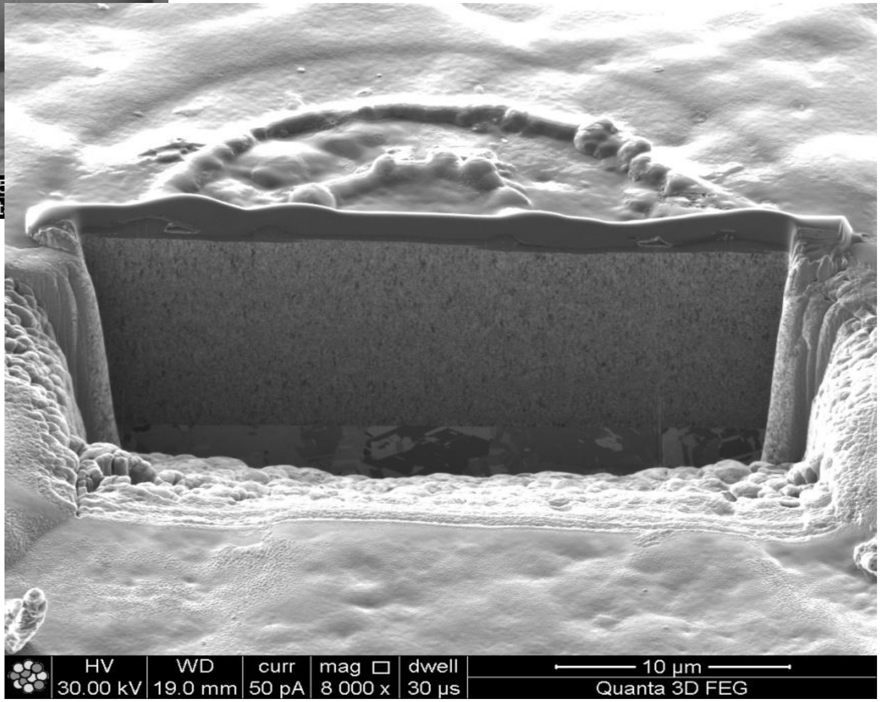
Imaging Cross section



BSE image:
compositional info



SE image:
topographical info

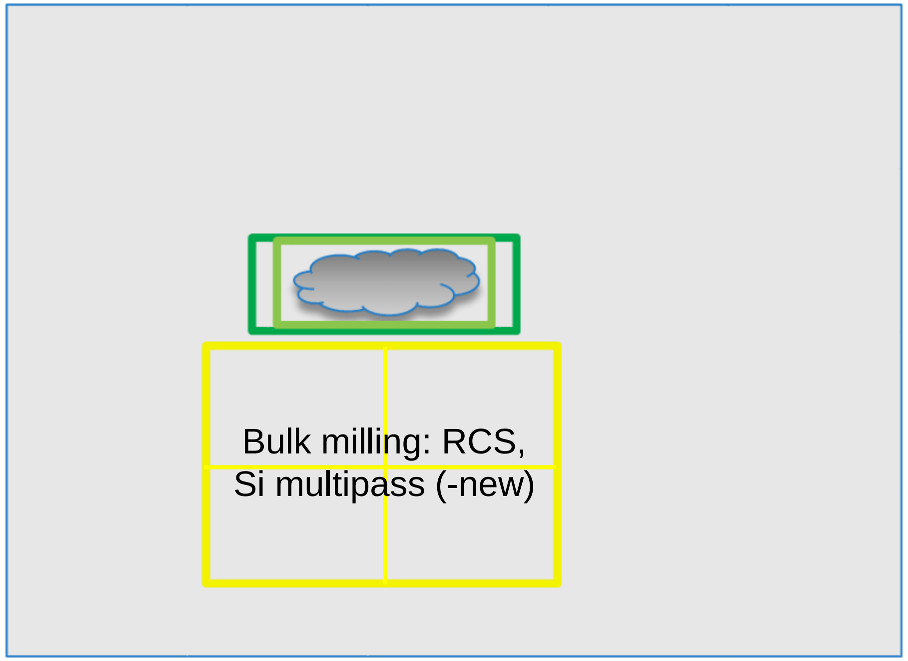


Ion induced SE image:
crystallographic orientation
contrast

Preparing cross section; step by step

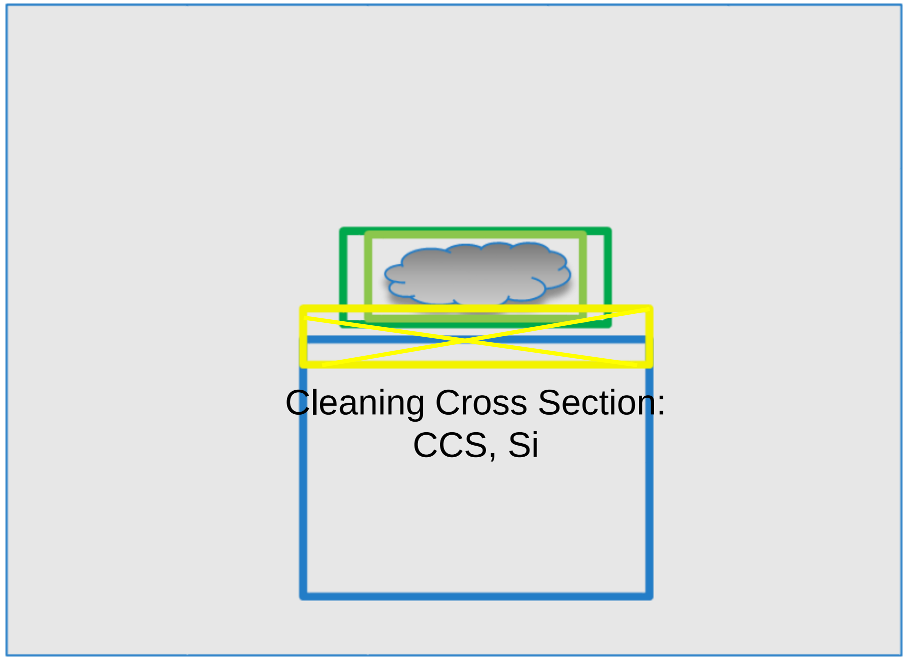
1. Find area of interest, link Z to WD, move sample to euc. Height.
2. Set beam coincidence point by using e-beam + Z (height)-adjustment
   (start with: zero beam shift and uncheck Z-Y link (compu tilt)
   - *<u>If needed</u> start with e-beam deposition in quad 1:*
     tilt back to zero; draw rectangle over area of interest.
     Choose Pt dep E str(uctures), change DT to 15us, change OL 75% (advance tab)
     set time to 300sec. E-beam 2kV >> 1nA ->Start
   - *Retract GIS*
3. Tilt to 52 and continue with ion beam Pt deposition in quad 2.
   Draw rectangle over E-beam dep. (increase X); Z=1um. Calculate the correct beam current, insert Pt GIS press F9.
   Place pattern over E-beam dep. When finished retract Pt GIS
4. Rough cut/bulk milling using Regular Cross Section + Si-multipass application file;
   RCS size of pattern: X slightly wider as Pt layer
   Set Z to required depth, Y=2Z. Choose a ion beam current according to pattern size and material.
   Leave space between end of pattern and the Pt layer (for high BC ∆>2um) start to mill front side.
5. Cleaning step: reduce ion beam current 2 steps. Apply an extra tilt according to beam current + use cleaning cross section (+ Si application); Z = ½ - ¼ depth of bulk milled depth.
   NOTE: instead of CCS; 5 boxes, Y=500nm + Z=8um (total milling time 3-5min.)
6. If needed repeat step 5 with a reduced BC.
7. Image cross section

Preparing cross section; step by step



Bulk milling: RCS,
Si multipass (-new)

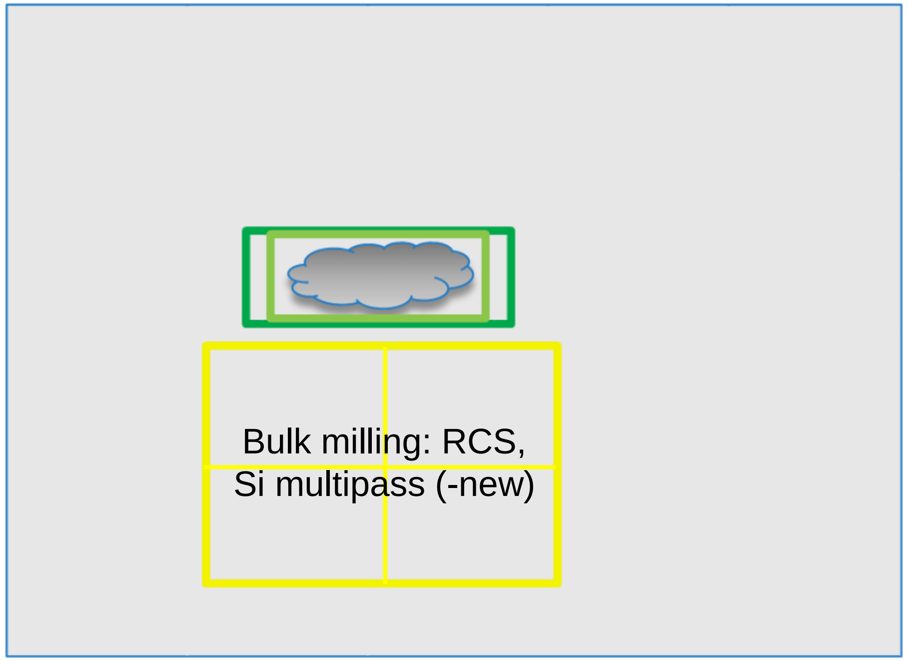
Step 1-4



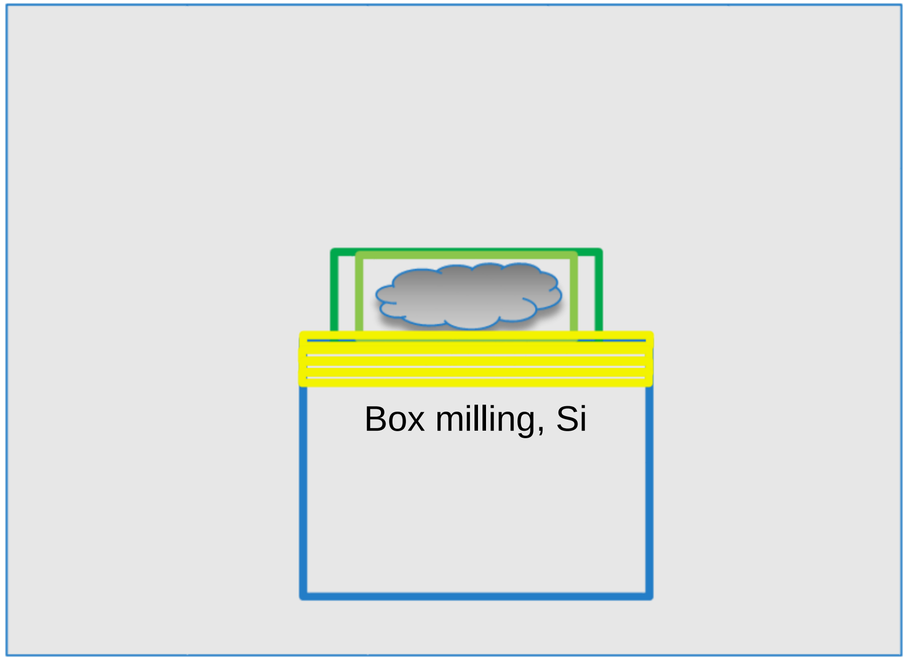
Cleaning Cross Section:
CCS, Si

Step 5+6

Preparing cross section; step by step



Step 1-4

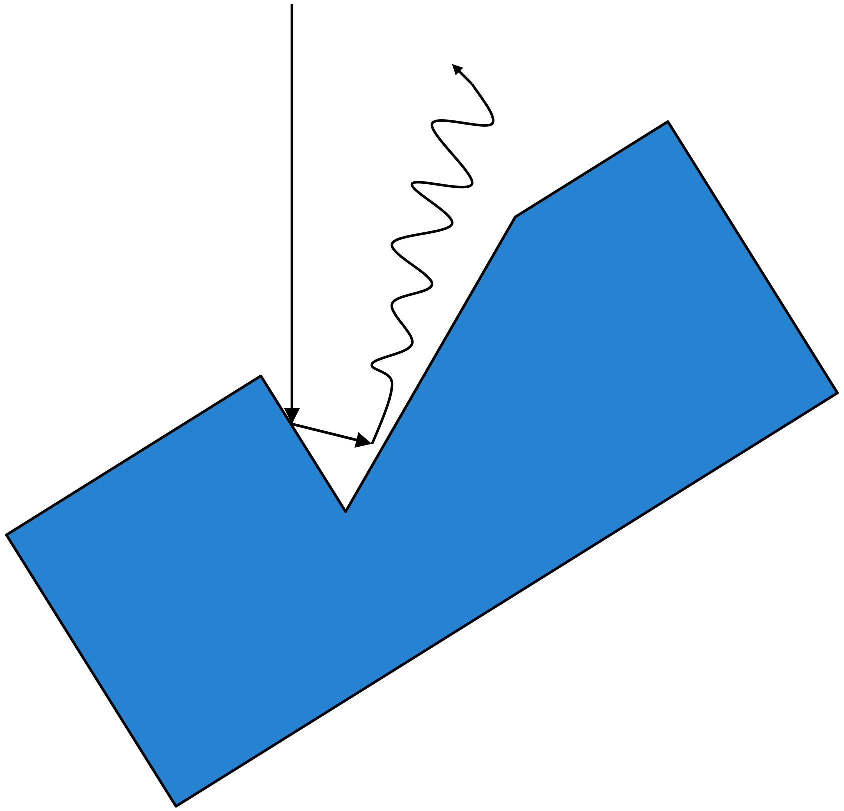


Step 5+6

FIB Cross-sectioning
Part 2

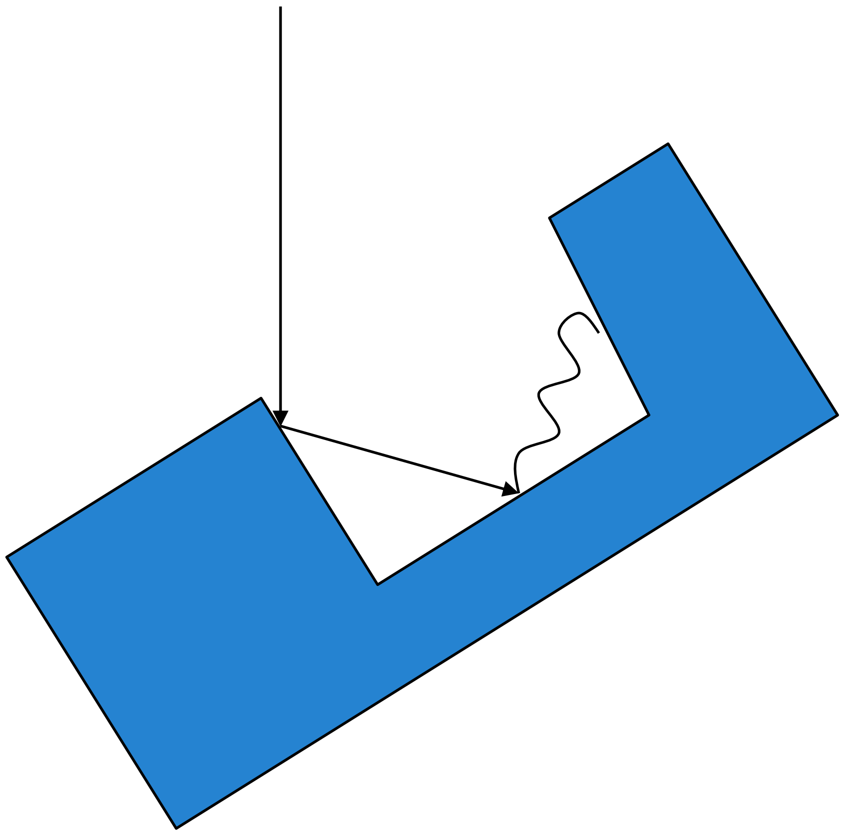
Preparing for EDS analysis

- For a clear x-ray signal that is only derived from the cross-section face
- The back of a typical cross-section will reflect rays
- These rays obscure the real signal
- 2 ways to prepare



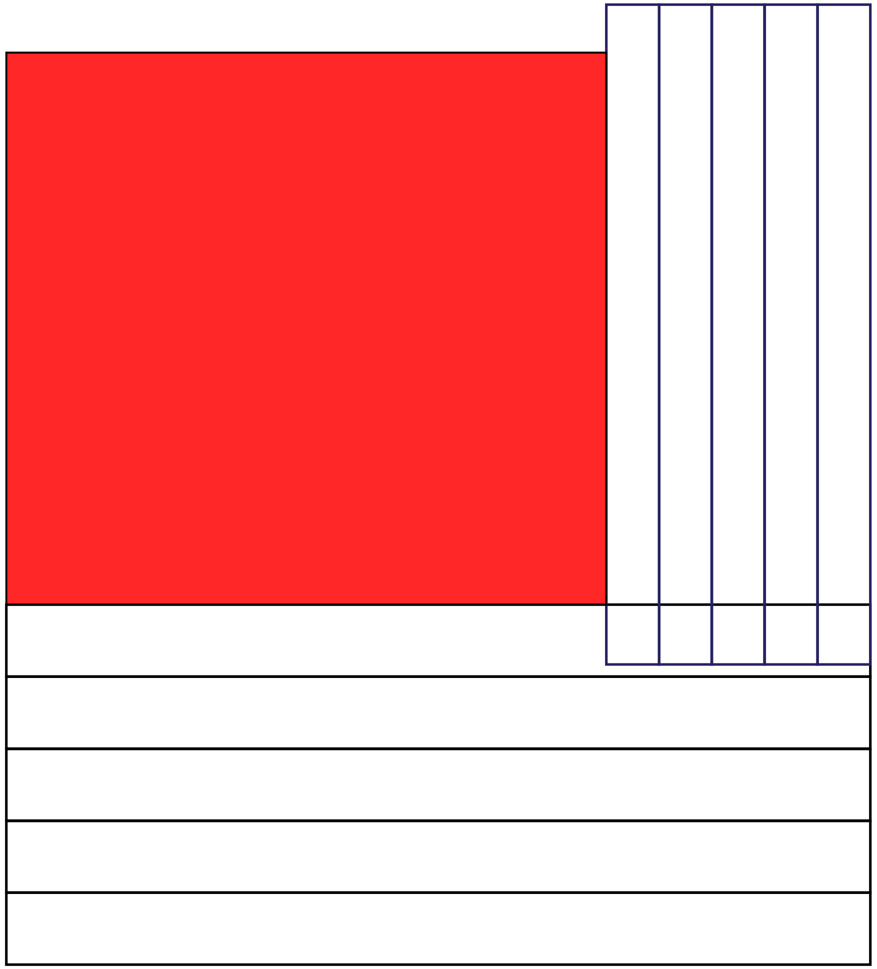
Preparing for EDS analysis

- So make a big box, also deeper than before
- X-ray signal just from cross-section
- Typical size would be 20 µm by 20 µm by 20 µm
- Use largest beam current available for bulk mill

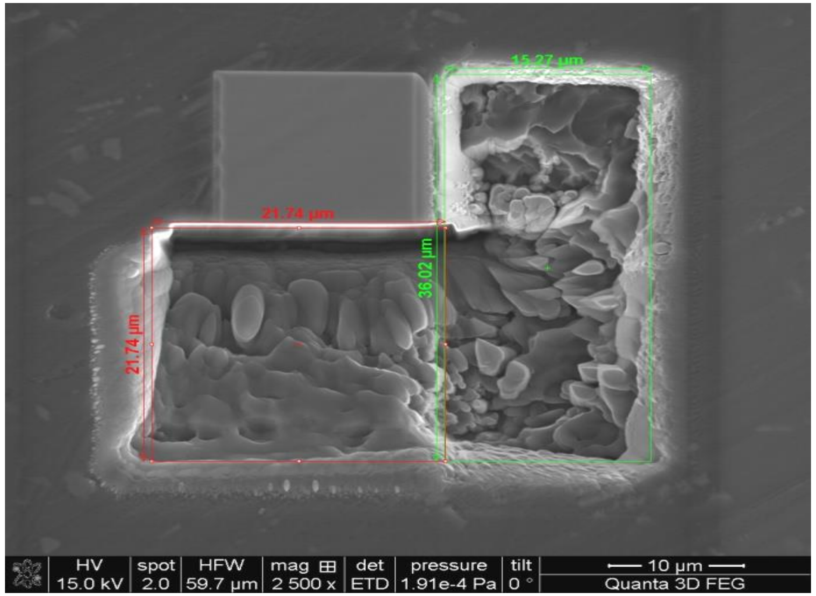


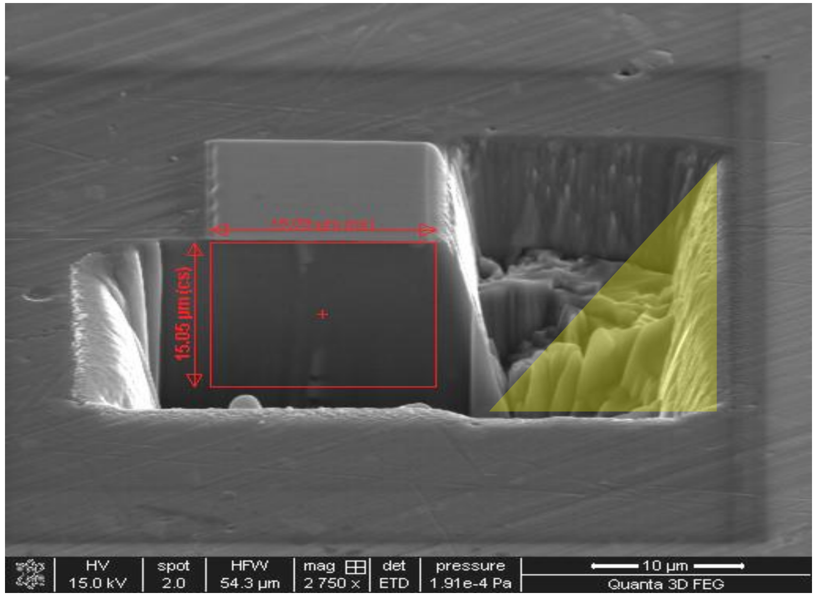
Preparing for EDS analysis

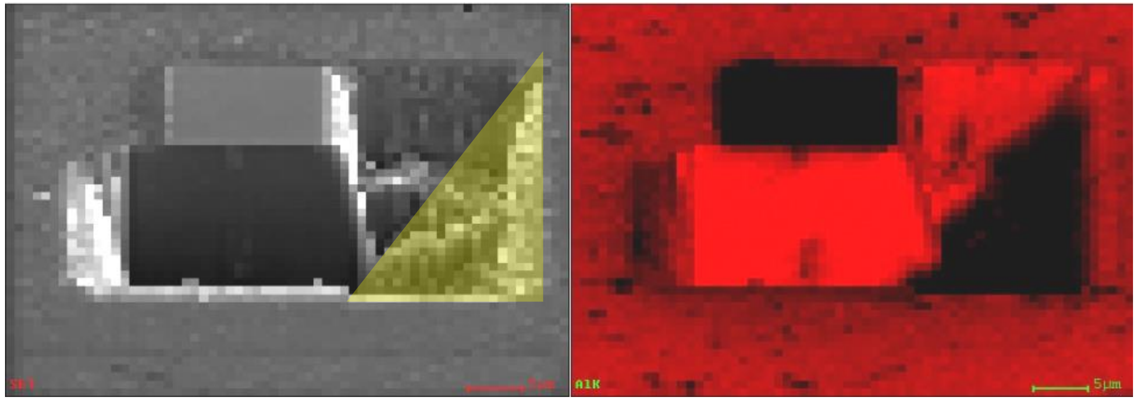
- Deposit a Pt-square
- Make a cross-section pattern bigger than face to expose
- Make anti-shadow cross section at right hand side
- Use a large current to reduce milling time



Preparing for EDS analysis

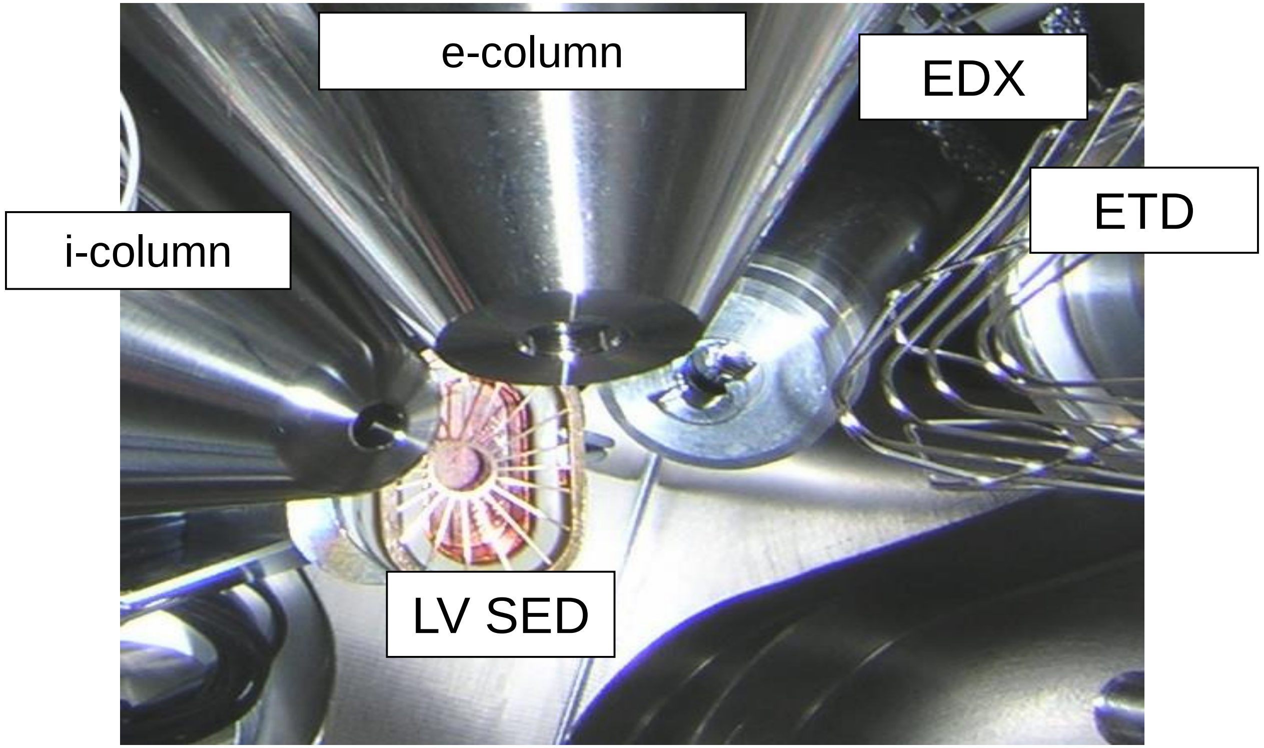




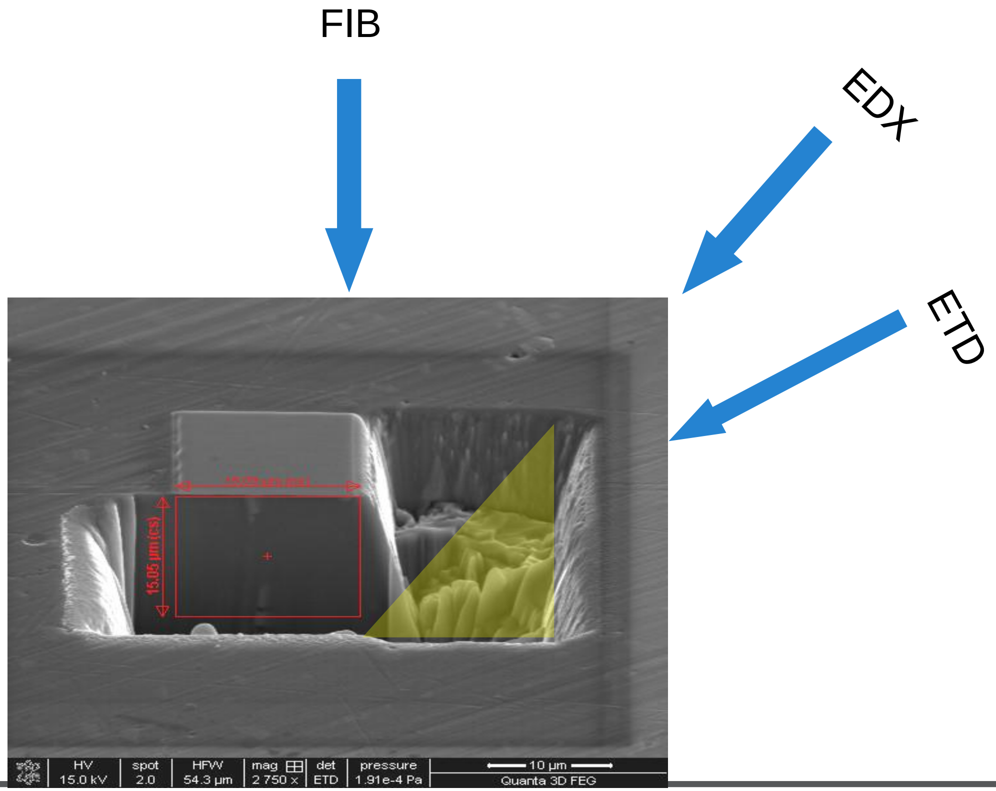


- To prevent shadowing: blocking the X-rays on their way to the detector
- Material at the right hand side of the cross section needs to be removed

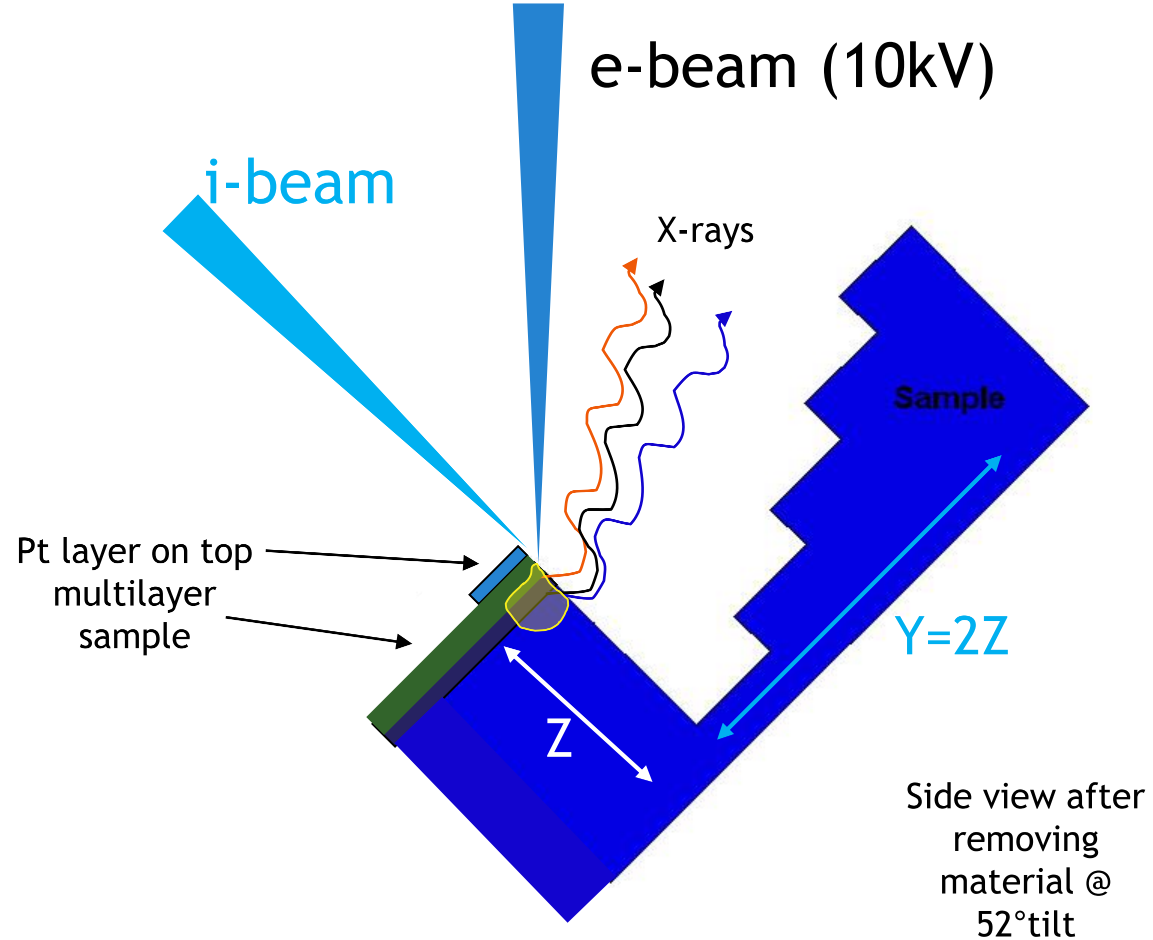
Preparing for EDS analysis



- To prevent shadowing:
  blocking the X-rays on their way to the detector
- Material at the right hand side of the cross section needs to be removed



EDS analyses on cross section

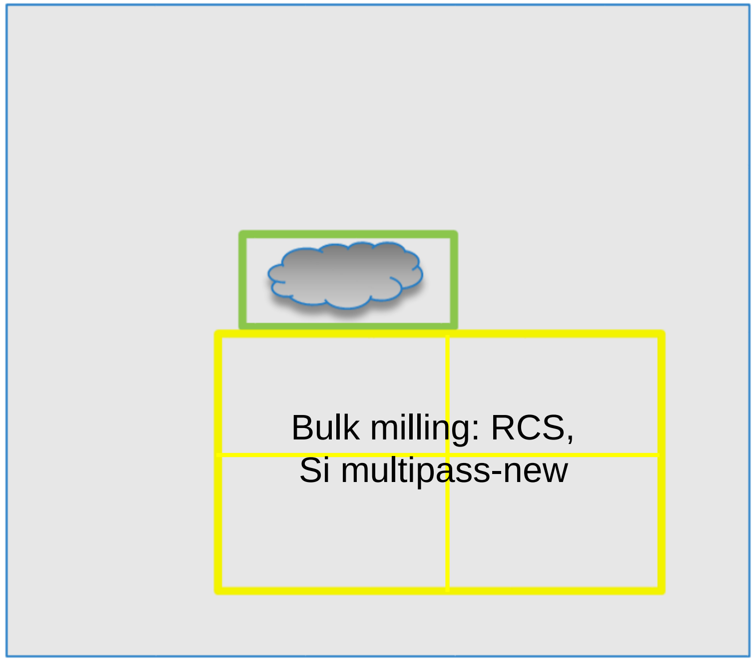


Side view after removing material @ 52°tilt

Preparation of EDX-Cross-Section step by step

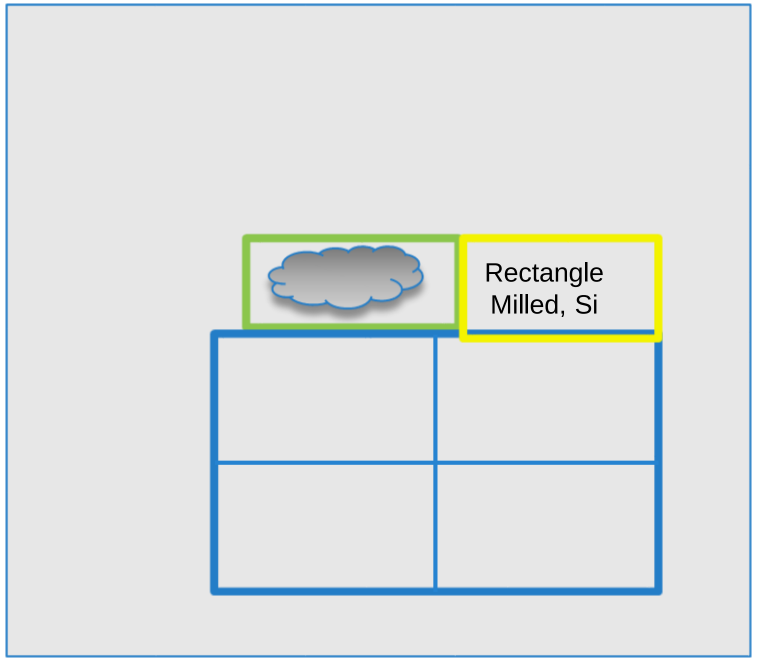
1. Find area of interest
2. Set coincidence point using e-beam + Z-adjustment (start with: zero beam shift and uncheck compu tilt)
   - If needed start with e-beam deposition in quad 1:
     tilt back to zero; draw rectangle over area of interest.
     Choose Pt dep E str, change DT to 15us, change OL 75% (advance tab)
     set time to 300sec. E-beam 2kV >> 1nA ->Start
   - Retract GIS
3. Tilt to 52 and continue with ion beam Pt deposition in quad 2.
   Overlay rectangle on E-beam dep. (increase X); Z=1um. Calculate the correct beam current
4. Rough cut/bulk milling using Regular Cross Section + Si multipass new application file;
   RCS size of pattern: X = 10-15um wider as Pt layer at right hand side
   Set Z to required depth , Y=2Z. Choose a ion beam current according to size and material.
   Put pattern not too close to the Pt layer (for high BC Δ>2um) start to mill front side.
5. Mill rectangle at right hand side of Pt layer
6. Cleaning step: reduce ion beam current 2 steps.
   According to beam current apply an extra tilt + using cleaning cross section (Si application):
   Z = ½ depth of bulk milled depth.
7. If needed repeat step 6 with a reduced BC.

Preparation of EDX-Cross-Section step by step



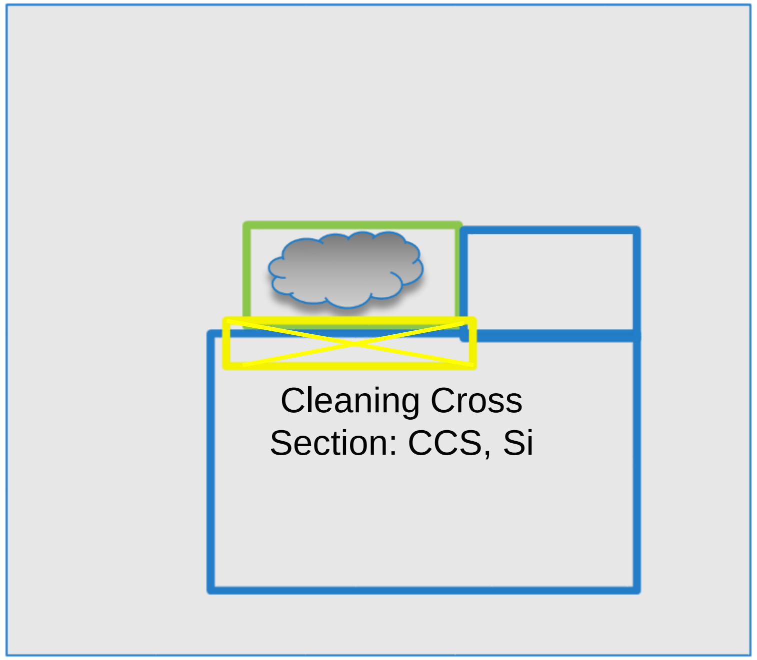
Bulk milling: RCS,
Si multipass-new

Step 1-4



Rectangle
Milled, Si

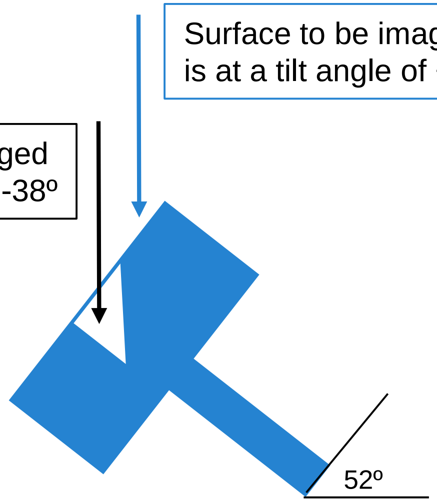
Step 5



Cleaning Cross
Section: CCS, Si

Step 6+7

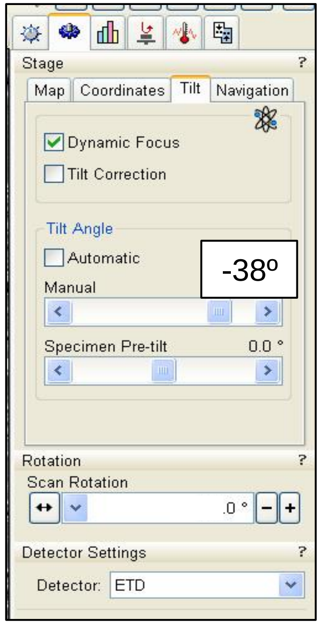
Imaging cross section using dynamic focus



Surface to be imaged with e-beam is at a tilt angle of +52º

Cross section to be imaged with e-beam is at an angle of -38º

sample 52º tilted



☑ Dynamic Focus

☐ Tilt Correction

Tilt Angle

☐ Automatic

Manual: -38º

Specimen Pre-tilt: 0.0 °

Rotation

Scan Rotation: 0 °

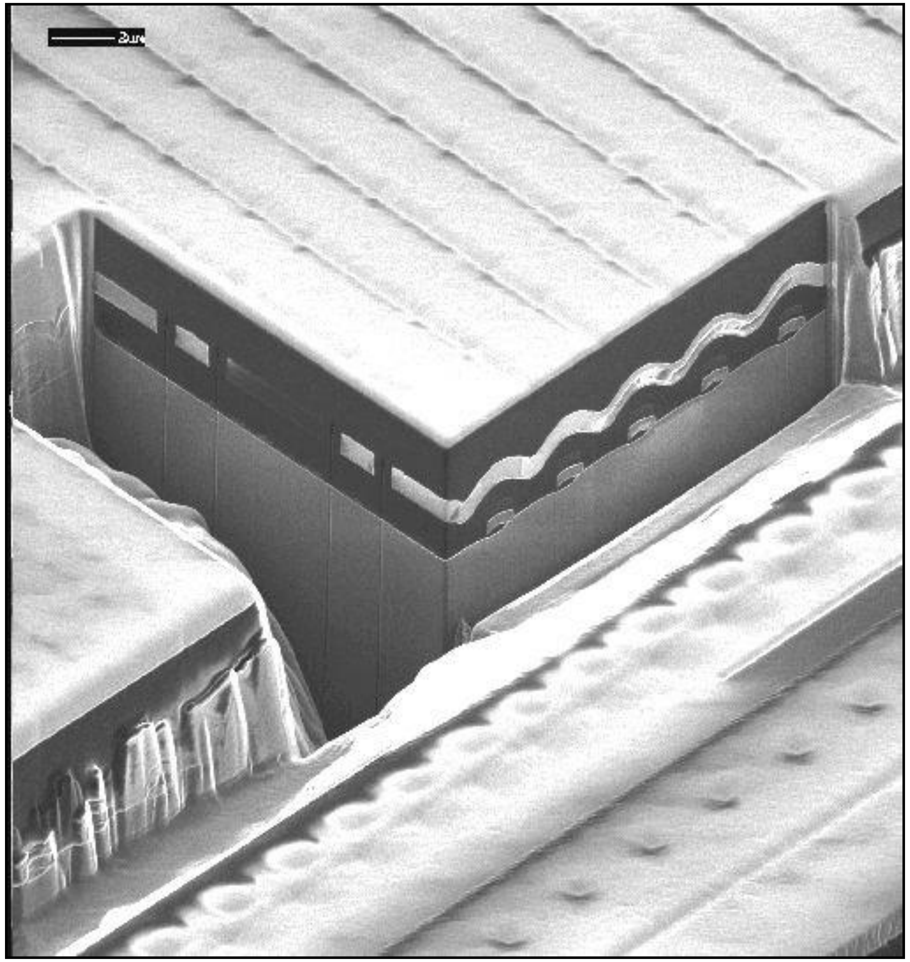
Detector Settings

Detector: ETD

NOTE: the dynamic focus has to be set manually to -38º

Corner cross section

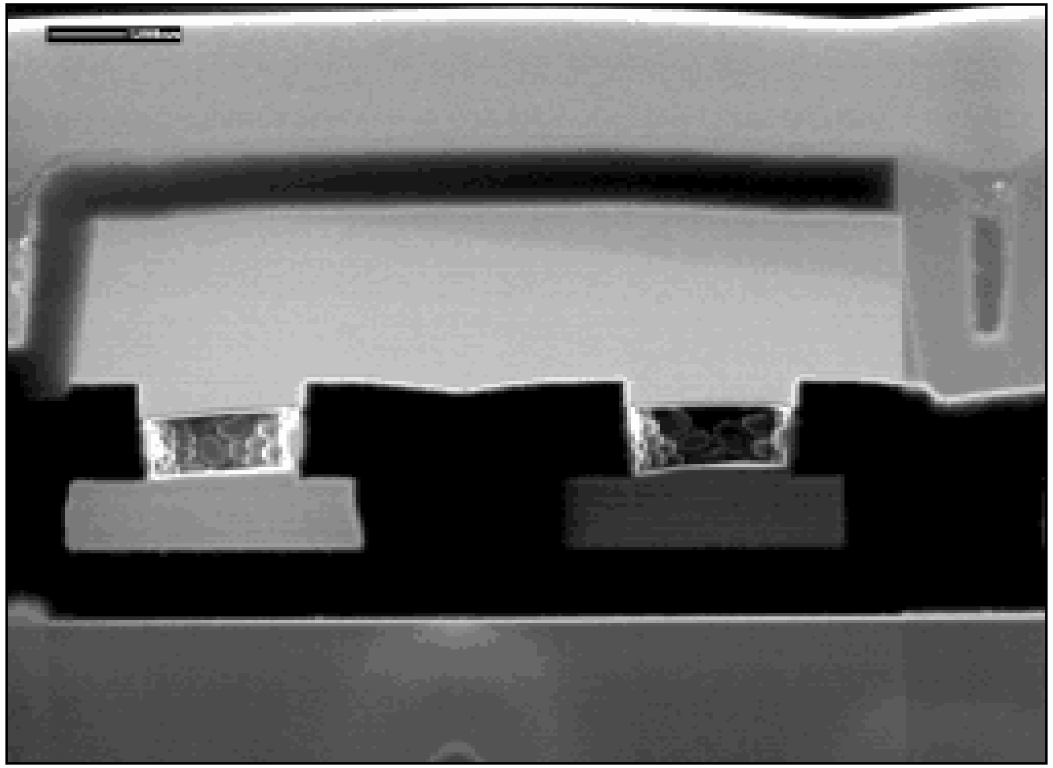
- Good for showing row and column structures
- Can see horizontal and vertical structure in one image



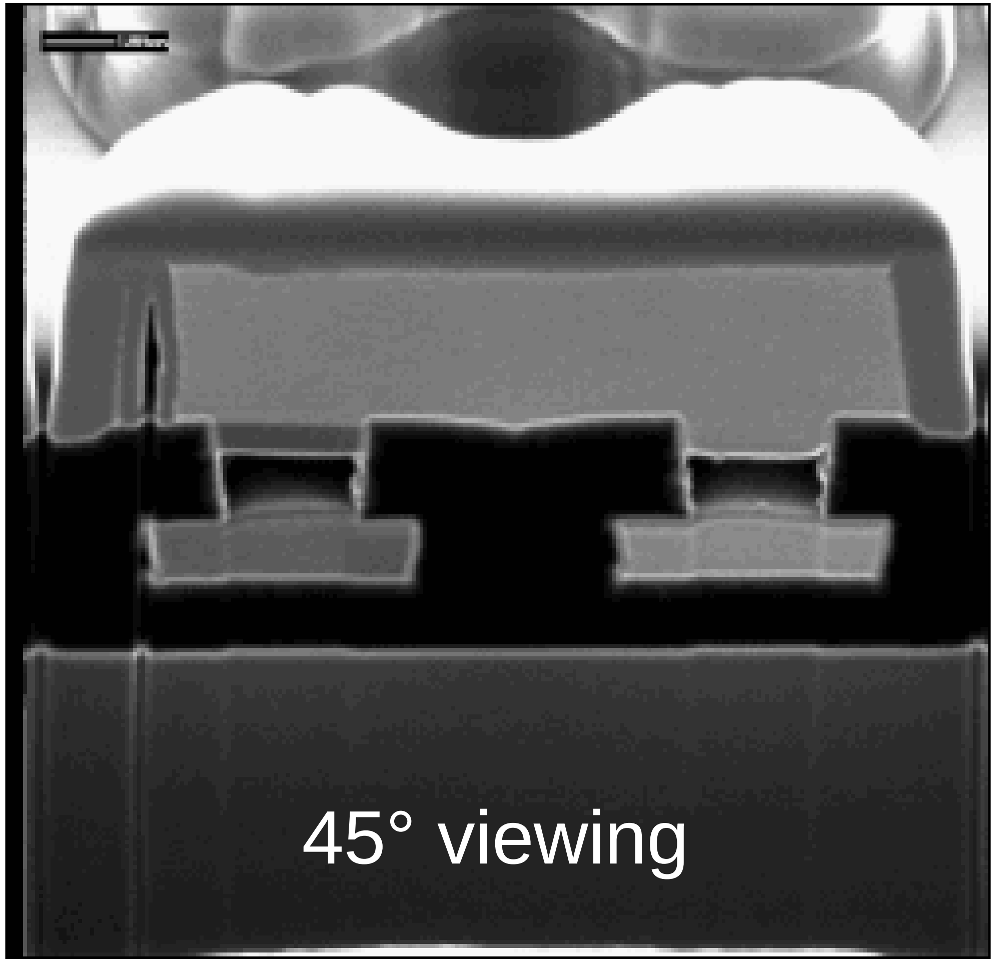
0° to 90° Viewing

- Direct observation for thickness measurements without tilt correction
- Yet allows milling so can find exact location
- Excellent signal to detector since feature not in a hole

Comparing 90° to 45°

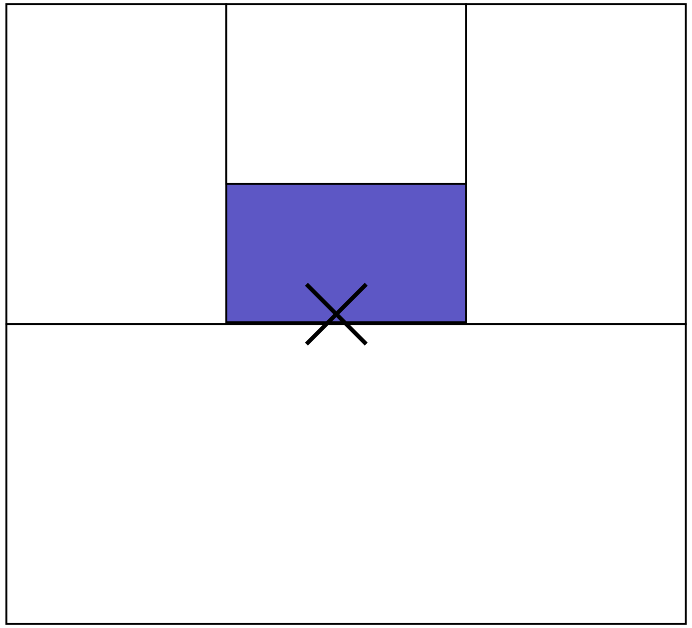


90° viewing



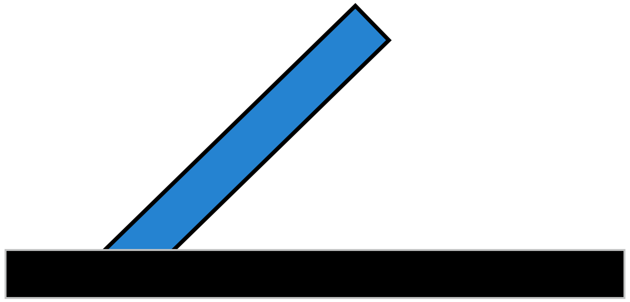
45° viewing

Sample pre-preparation

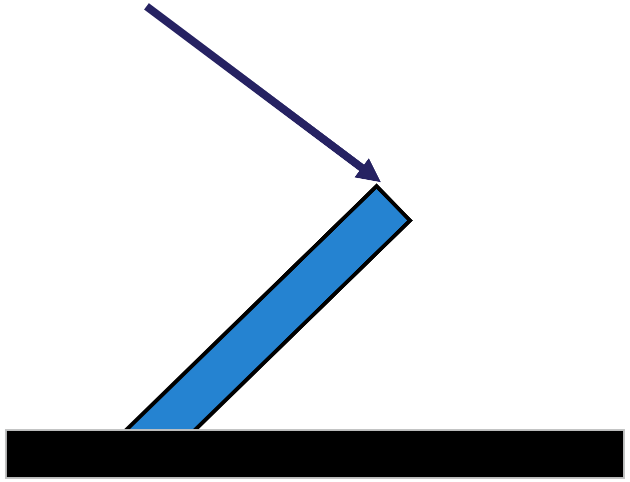


- Scribe or cleave sample close to feature
- Mount sample on pre-tilted holder

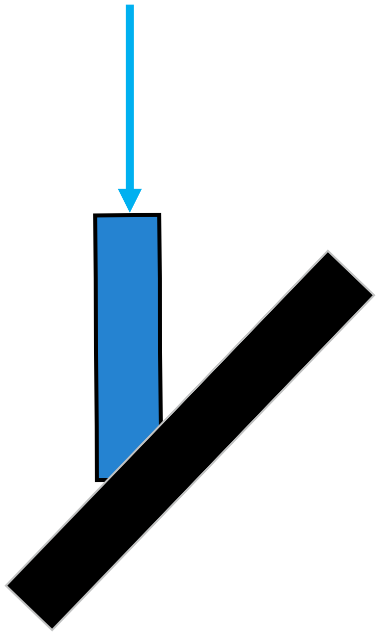
Stage movement for 0° to 90° Viewing



Start pre tilted...



milling on sidewall



Extra tilt for imaging

0° to 90° Viewing Procedure

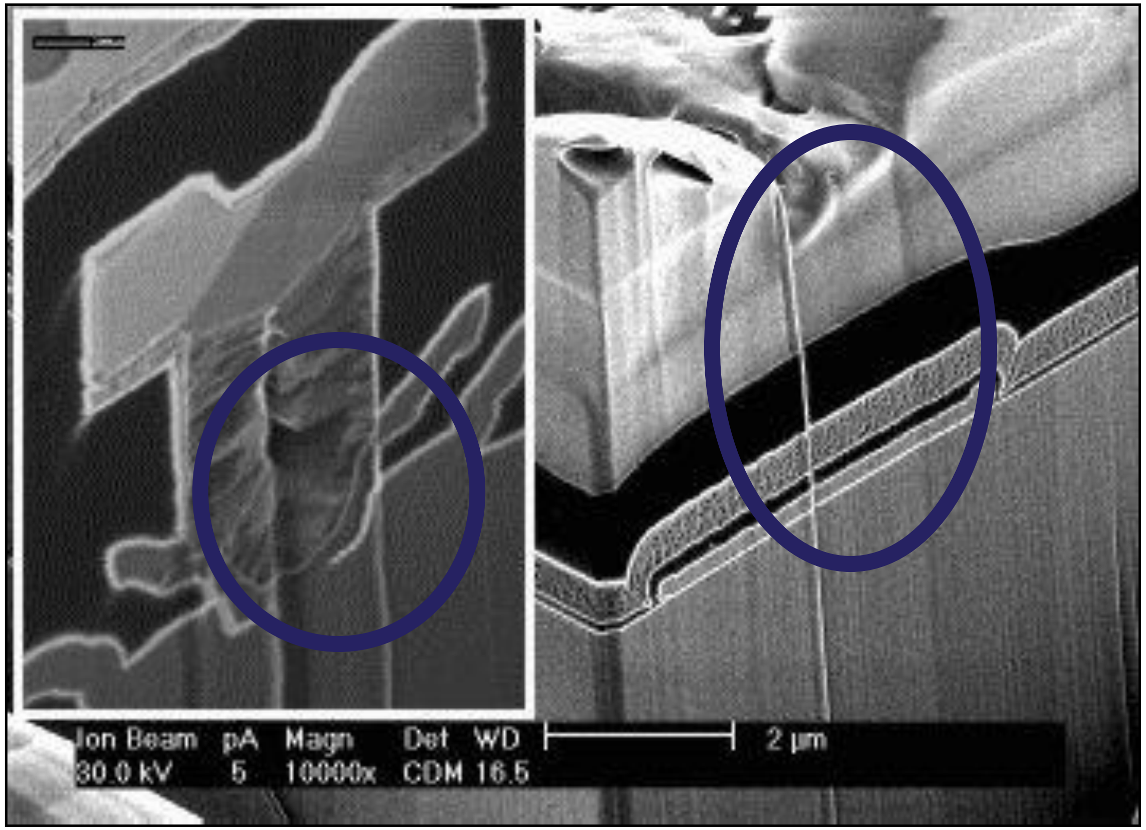
1. Scribe or cleave sample close to feature
2. Mount sample pre-tilted 45°
3. Rotate sample edge parallel to tilt axis (down toward user)
4. Set feature to eucentric height
5. Milling at 0 deg stage tilt
6. Stage tilt 45° for plan view
7. Navigate to feature

FIB Cross-sectioning
Part 3

Avoiding Curtains

Curtaining

- **Caused by:**
  - Surface topography
  - Sputter rate differences: fast next to slow
  - Pores
  - Crystallographic orientation
- **Solutions:**
  - Planarize with metal deposition
  - Use correct beam current
  - Angled cut

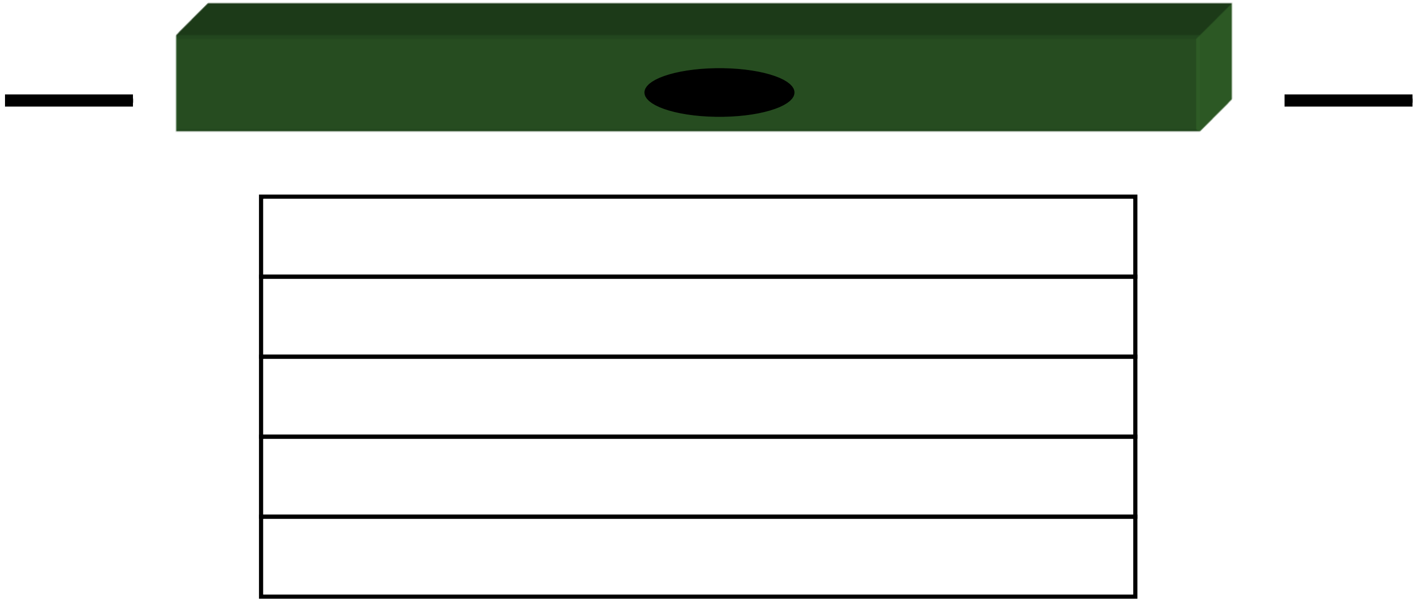


Planarize with FIB Deposition

Evens out the surface topography

1. Make fiducial marks
    - put in line with final edge of cross-section
    - and outside where cross-section will be milled
    - use line scans, ~2 µm long
    - one on each side of the cross-section
2. Deposit Platinum bar
    - x = 1-2 µm wider than cross-section
    - y = ~2 microns
    - z = height of step of 1 µm
    - BC = x * y * 1 * 6 pA /µm^2
    - application file = Pt deposition
3. Mill as usual, with fiducial as a guide for where to stop milling

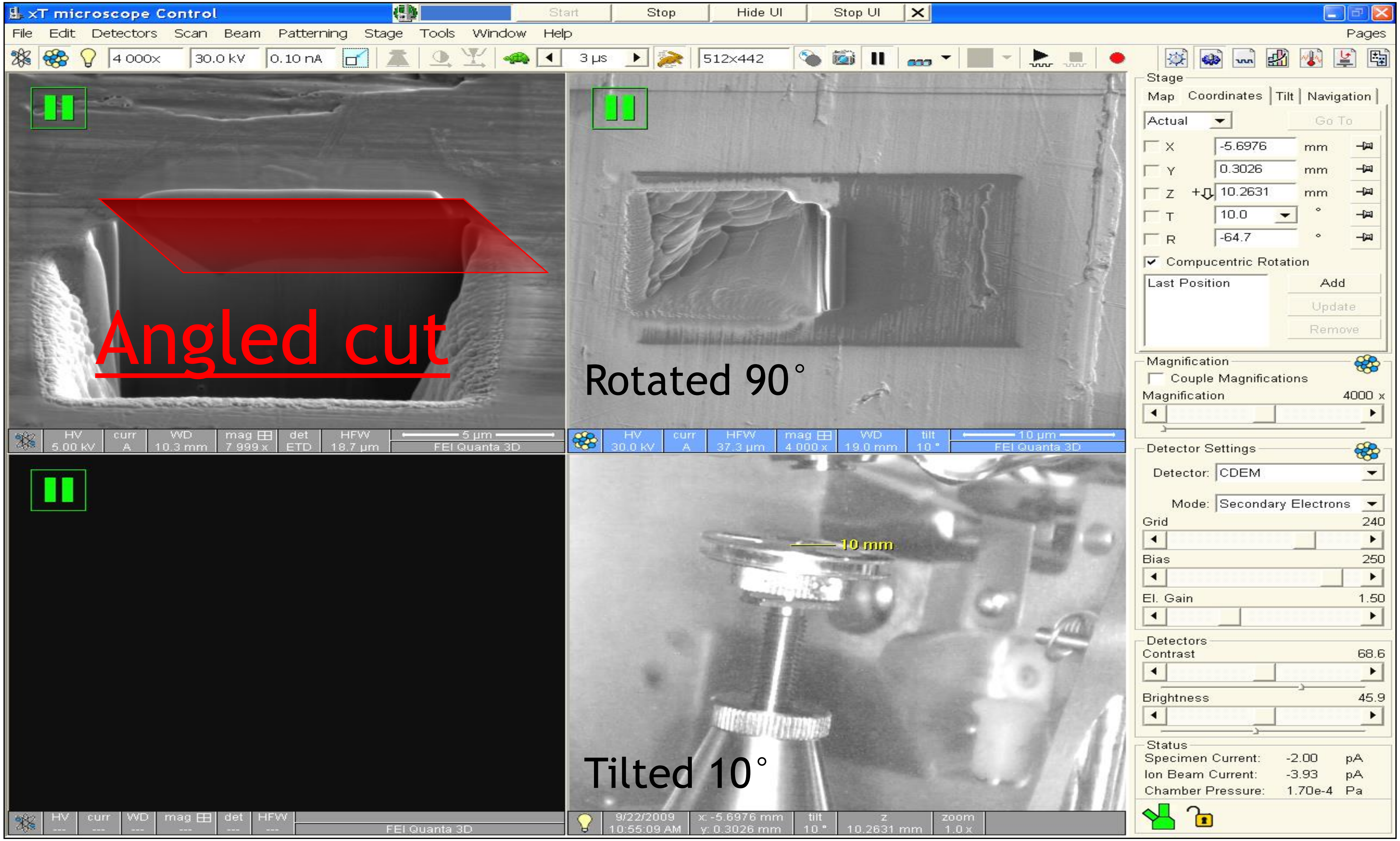
Planarize with FIB Deposition



Angled Cut

Reduces cumulative effects of stacked tungsten plugs.
Voids and edges transfer to lower levels

1. Bulk mill as usual an extra 3 µm wider
2. Save stage location - “normal”
3. Stage rotate 90°
4. Tilt stage to 8° - 10°
5. Scan rotate -90° (optional)
6. Save position -“angled”
7. Polish as usual
8. To check progress, go to stage location “normal”
9. Return to milling at stage location “angled”
10. Return to normal with “normal”



Angled cut

Rotated 90°

Tilted 10°

4 000x
30.0 kV
0.10 nA
3 µs
512x442

Stage
☐ X -5.6976 mm
☐ Y 0.3026 mm
☐ Z 10.2631 mm
☐ T 10.0 °
☐ R -64.7 °
☑ Compucentric Rotation

Magnification
☐ Couple Magnifications
Magnification 4000 x

Detector Settings
Detector: CDEM
Mode: Secondary Electrons
Grid 240
Bias 250
El. Gain 1.50

Detectors
Contrast 68.6
Brightness 45.9

Status
Specimen Current: -2.00 pA
Ion Beam Current: -3.93 pA
Chamber Pressure: 1.70e-4 Pa

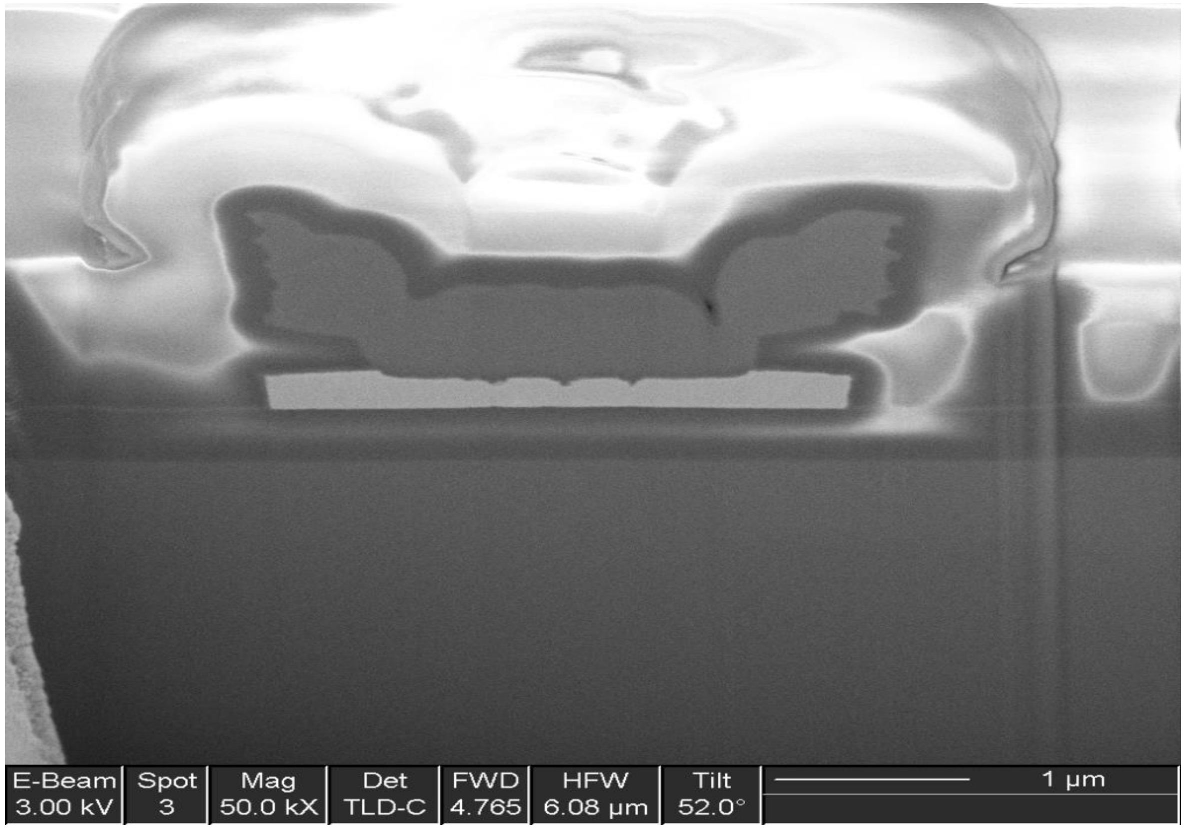
HV	curr	WD	mag	det	HFW
5.00 kV	A	10.3 mm	7 999 x	ETD	18.7 µm

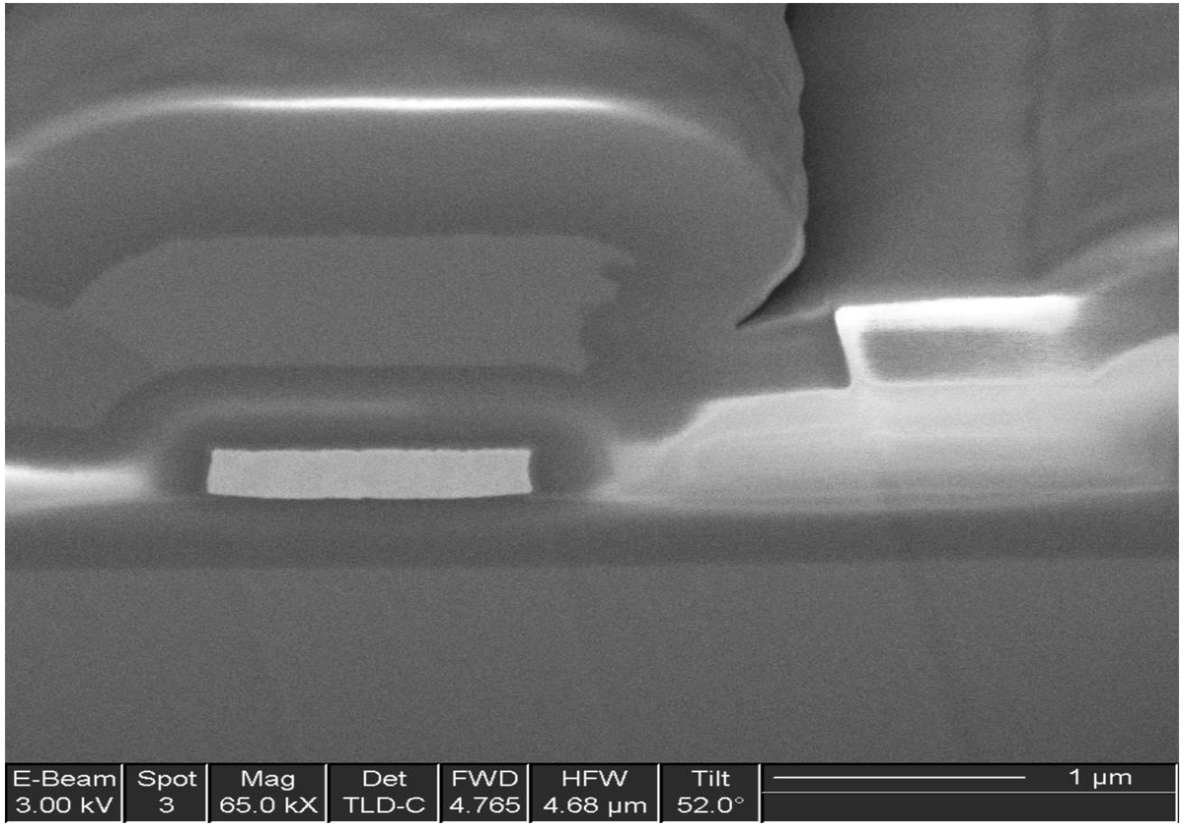
HV	curr	HFW	mag	WD	tilt
30.0 kV	A	37.3 µm	4 000 x	19.0 mm	10 °

HV	curr	WD	mag	det	HFW
---	---	---	---	---	---

9/22/2009 10:55:09 AM
x: -5.6976 mm y: 0.3026 mm
tilt 10 °
z 10.2631 mm
zoom 1.0 x

Angled Cut


E-Beam
3.00 kV
Spot
3
Mag
50.0 kX
Det
TLD-C
FWD
4.765
HFW
6.08 µm
Tilt
52.0°
1 µm


E-Beam
3.00 kV
Spot
3
Mag
65.0 kX
Det
TLD-C
FWD
4.765
HFW
4.68 µm
Tilt
52.0°
1 µm

DB Cross Section Geometry

Key

- **Metal deposition**
- **SEM**
- **FIB**
- **Sample plane**
- **Cross section**
- **Projection of sample plane to SEM image plane**
- **Projection of cross section plane to SEM plane**
- **Cross section image surface**
- **Stage at 0°**

Cos 52°

Sin 52°

38° from 0°